IUCrData

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15-(4-Chlorophenyl)-6b-hydroxy-17-methyl-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 methanol hemisolvate

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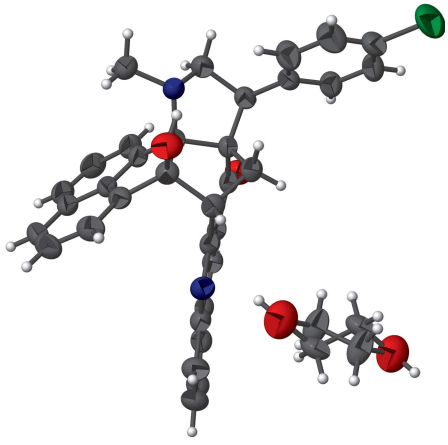
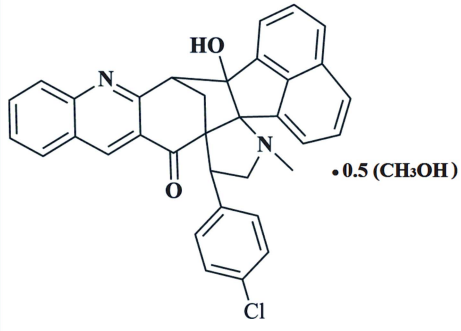
Structural data: full structural data are available from iucrdata.iucr.org

In the title solvate, $C_{35}H_{25}ClN_2O_3\cdot0.5CH_3OH$, the conformation of the central 1-methylpyrrolidine ring is best described as an envelope 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 makes dihedral angles of 40.87 (12), 67.89 (11), 81.86 (9) and 70.86 (12)° with the mean planes of the cyclopentane, cyclohexane, acenaphthylene and chlorobenzene rings, respectively. The quinoline ring system is inclined to the naphthalene ring system by 54.14 (6)°. There is a short O—H···N contact in the molecule making an *S*(5) ring motif. In the crystal, there is a disordered and partially occupied ethan-1,2-diol solvent molecule present, located about an inversion centre, which links the title molecules *via* O—H···O and C—H···O hydrogen bonds, forming chains propagating along the *a*-axis direction.

3D view



Chemical scheme



Structure description

The pyrrolidine ring system is found in a vast variety of compounds displaying an impressive range of biological activities (Babu *et al.*, 2012). Optically active pyrrolidines have been used as intermediates, chiral ligands or auxiliaries in controlled asymmetric synthesis (Savithri *et al.*, 2014). Pyrrolidine compounds are reported to exhibit anti-microbial, antifungal (Govind *et al.*,2003), anti-influenza virus A (Stylianakis *et al.*, 2003), anti-inflammatory and antitumor (Li & Xu, 2004) activities. They also inhibit retroviral reverse transcriptases [*i.e.*, human immunodeficiency virus type 1 (HIV-1)], cellular DNA polymerases, protein kinases (Bellina & Rossi, 2006). They also act as antidepressant

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3O···N1	0.82	2.28	2.984 (4)	145
O2—H2O···N2	0.87 (3)	2.20 (3)	2.695 (2)	116 (2)
O2—H2O···O3^{i}	0.87 (3)	2.26 (3)	3.021 (5)	146 (2)
C33—H33···O3^{i}	0.93	2.53	3.408 (6)	157

Symmetry code: (i) $-x+1, -y+1, -z+1$.

agents (Wróbel *et al.*, 2013), antibiotics (Nirmala *et al.*, 2009), anticonvulsant, sphingosine-1-phosphate (S1P) receptor agonists, malic enzyme inhibitors, ketoamide-based cathepsin K inhibitors and human melanocortin-4 receptor agonists (Babu *et al.*, 2012). Quinoline derivatives have been synthesized and explored for their analgesic activity, anti-allergetic properties and in treating Alzheimer's disease (AD). They are also reported for their anticancer, antitinephritic, antitumor, anti-inflammatory (Shivaraj *et al.*, 2013), antimalarial, antibacterial and antifungal activities (Marella *et al.*, 2013).

The molecular structure of the title compound is shown in Fig. 1. In the molecule there is a short intramolecular O—H···N contact forming an $S(5)$ ring motif (Fig. 1 and Table 1). The conformation of the central 1-methyl pyrrolidine ring (C12/C14/N2/C26/C27) is best described as an envelope with atom N2 as the flap and displaced by 0.585 (2) Å from the plane through the other four atoms. The cyclopentane ring adopts a twist conformation on the C10—C11 bond and the cyclohexane ring has an envelope conformation with atom C11 as the flap and displaced by 0.854 (2) Å from the mean plane through the other five atoms. The quinoline ring system (C1–C9/N1) and the acenaphthylene ring system (C14–C25) adopt almost planar conformations, with the maximum deviations being 0.069 (2) Å for atom C8 in the quinoline ring system and 0.128 (2) Å for atom C14 in the acenaphthylene ring system. Chlorine atom Cl1 is displaced from the benzene ring (C28–C33) to which it is attached by 0.0164 (8) Å. The mean plane of the pyrrolidine ring makes dihedral angles of 40.87 (12), 67.89 (11), 81.86 (9) and 70.86 (12)° with the mean

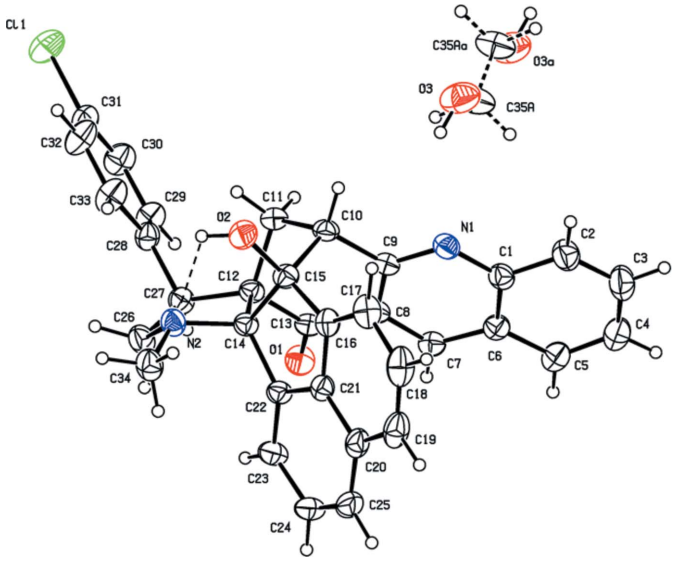


Figure 1
The molecular structure of the title compound, showing the atom labelling and displacement ellipsoids drawn at the 30% probability level. The O—H···N contact is shown as a thin dashed line (see Table 1).

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{34}H_{25}ClN_2O_2{\cdot}0.5CH_4O$
M_r	544.53
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	293
a, b, c (Å)	10.3848 (3), 10.4306 (3), 14.4513 (4)
α, β, γ (°)	69.394 (1), 72.388 (1), 70.871 (1)
V (Å^3)	1352.92 (7)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.18
Crystal size (mm)	0.22 × 0.16 × 0.12
Data collection	
Diffractometer	Bruker SMART APEXII area-detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2008)
T_{min}, T_{max}	0.966, 0.978
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	20173, 5563, 4425
R_{int}	0.019
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.629
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.052, 0.178, 1.05
No. of reflections	5563
No. of parameters	386
No. of restraints	3
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.66, −0.44

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

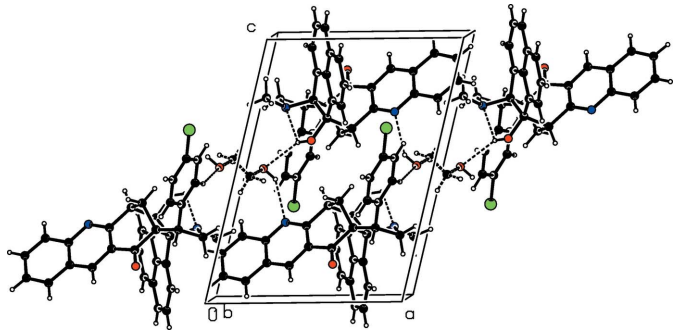


Figure 2
The crystal packing of the title compound viewed along the b axis. Hydrogen bonds are shown as dashed lines (see Table 1). For clarity, only one position of the disordered and partially occupied ethan-1,2-diol solvent molecule is shown.

planes of the cyclopentane, cyclohexane, acenaphthylene and chlorobenzene rings, respectively. The quinoline ring system is inclined to the naphthalene ring system by 54.14 (6)°.

In the crystal, there is a disordered and partially occupied ethan-1,2-diol solvent molecule present, located about an inversion centre, which links the title molecules *via* O—H···O and C—H···O hydrogen bonds, forming chains propagating along the *a*-axis direction. (Table 1 and Fig. 2).

Synthesis and crystallization

A mixture of (*E*)-2-(4-chlorobenzylidene)-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 and concentrated under reduced pressure. It was then subjected to column chromatography using petroleum ether–AcOEt (5:1 *v*/*v*) as eluent to obtain the title compound as a pure product. Colourless block-like crystals were prepared by slow evaporation of a solution of the title compound in ethanol at room temperature.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The partially occupied methanol solvent molecule is disordered and located about an inversion centre (Fig. 1). The solvate molecule shows a high degree of disorder in such a way that the methyl carbon atom assumes four positions. Two are within the asymmetric unit, each with 0.25 occupancy, and the counterparts occupy symmetry-related positions, each with 0.25 occupancy. Similarly, the hydroxyl group assumes two positions with 0.5 occupancy. The hydrogen atoms could not be fixed on the carbon atom with agreeable geometry.

Acknowledgements

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full crystallographic data

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15-(4-Chlorophenyl)-6b-hydroxy-17-methyl-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 methanol hemisolvate

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15-(4-Chlorophenyl)-6b-hydroxy-17-methyl-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 methanol hemisolvate

Crystal data

$C_{34}H_{25}ClN_2O_2·0.5CH_4O$
M_r = 544.53
Triclinic, $P\overline{1}$
a = 10.3848 (3) Å
b = 10.4306 (3) Å
c = 14.4513 (4) Å
α = 69.394 (1)°
β = 72.388 (1)°
γ = 70.871 (1)°
V = 1352.92 (7) Å^3
Z = 2
$F(000)$ = 569
D_x = 1.337 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5568 reflections
θ = 1.5–26.5°
μ = 0.18 mm^{-1}
T = 293 K
Block, colourless
0.22 × 0.16 × 0.12 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} = 0.966, T_{max} = 0.978
20173 measured reflections
5563 independent reflections
4425 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 26.5°, θ_{min} = 1.5°
h = −13→13
k = −13→12
l = −18→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.178
S = 1.05
5563 reflections
386 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1116P)^2 + 0.2946P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.019 (4)

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)
Cl1	0.72400 (8)	−0.21816 (7)	0.66553 (5)	0.0903 (3)	
O1	0.58561 (15)	0.35178 (15)	0.13530 (11)	0.0563 (4)	
O2	0.67317 (16)	0.59399 (18)	0.39571 (11)	0.0570 (4)	
H2O	0.743 (3)	0.520 (3)	0.405 (2)	0.092 (10)*	
N1	0.27834 (16)	0.70993 (17)	0.28663 (12)	0.0470 (4)	
N2	0.84974 (15)	0.43617 (16)	0.27419 (13)	0.0472 (4)	
C1	0.19626 (18)	0.75391 (19)	0.21738 (14)	0.0463 (4)	
C2	0.0684 (2)	0.8558 (2)	0.23152 (19)	0.0626 (6)	
H2	0.0427	0.8932	0.2860	0.075*	
C3	−0.0175 (2)	0.8992 (3)	0.1653 (2)	0.0702 (6)	
H3	−0.1023	0.9648	0.1760	0.084*	
C4	0.0197 (2)	0.8469 (2)	0.08168 (19)	0.0634 (6)	
H4	−0.0402	0.8778	0.0374	0.076*	
C5	0.1428 (2)	0.7514 (2)	0.06473 (17)	0.0549 (5)	
H5	0.1686	0.7199	0.0074	0.066*	
C6	0.23238 (19)	0.69925 (19)	0.13379 (14)	0.0447 (4)	
C7	0.35429 (19)	0.58980 (19)	0.12598 (14)	0.0440 (4)	
H7	0.3811	0.5501	0.0725	0.053*	
C8	0.43319 (17)	0.54205 (18)	0.19758 (13)	0.0391 (4)	
C9	0.39312 (17)	0.60925 (18)	0.27596 (13)	0.0401 (4)	
C10	0.48274 (18)	0.55891 (19)	0.35269 (13)	0.0423 (4)	
H10	0.4294	0.5842	0.4151	0.051*	
C11	0.54119 (19)	0.39953 (19)	0.37358 (13)	0.0448 (4)	
H11A	0.4683	0.3517	0.3885	0.054*	
H11B	0.5893	0.3612	0.4292	0.054*	
C12	0.64345 (17)	0.38709 (17)	0.27249 (13)	0.0393 (4)	
C13	0.55758 (18)	0.41925 (18)	0.19547 (13)	0.0402 (4)	
C14	0.71568 (17)	0.51111 (17)	0.24384 (13)	0.0388 (4)	
C15	0.61918 (18)	0.61159 (19)	0.31166 (13)	0.0414 (4)	
C16	0.60670 (18)	0.75928 (19)	0.24087 (14)	0.0434 (4)	
C17	0.5485 (2)	0.8893 (2)	0.25675 (18)	0.0569 (5)	
H17	0.5065	0.8983	0.3216	0.068*	
C18	0.5533 (3)	1.0098 (2)	0.1728 (2)	0.0694 (7)	
H18	0.5162	1.0987	0.1837	0.083*	
C19	0.6103 (2)	1.0011 (2)	0.0763 (2)	0.0663 (6)	
H19	0.6108	1.0833	0.0230	0.080*	
C20	0.66844 (19)	0.8680 (2)	0.05661 (16)	0.0525 (5)	
C21	0.66716 (17)	0.74950 (18)	0.14204 (14)	0.0412 (4)	
C22	0.72521 (17)	0.60975 (18)	0.13645 (13)	0.0410 (4)	

C23	0.7808 (2)	0.5866 (2)	0.04288 (15)	0.0538 (5)	
H23	0.8193	0.4952	0.0367	0.065*	
C24	0.7782 (2)	0.7046 (3)	−0.04387 (16)	0.0638 (6)	
H24	0.8132	0.6890	−0.1074	0.077*	
C25	0.7264 (2)	0.8401 (3)	−0.03804 (17)	0.0638 (6)	
H25	0.7294	0.9148	−0.0969	0.077*	
C26	0.8953 (2)	0.3053 (2)	0.24478 (17)	0.0559 (5)	
H26A	0.9666	0.2373	0.2800	0.067*	
H26B	0.9325	0.3234	0.1725	0.067*	
C27	0.76447 (19)	0.25010 (19)	0.27486 (15)	0.0463 (4)	
H27	0.7655	0.2117	0.2217	0.056*	
C28	0.75497 (19)	0.13475 (19)	0.37432 (15)	0.0458 (4)	
C29	0.7023 (2)	0.0238 (2)	0.38486 (16)	0.0543 (5)	
H29	0.6719	0.0224	0.3310	0.065*	
C30	0.6933 (2)	−0.0857 (2)	0.47347 (18)	0.0632 (6)	
H30	0.6583	−0.1601	0.4788	0.076*	
C31	0.7367 (2)	−0.0827 (2)	0.55281 (17)	0.0589 (5)	
C32	0.7896 (3)	0.0258 (3)	0.5450 (2)	0.0720 (7)	
H32	0.8186	0.0273	0.5994	0.086*	
C33	0.7997 (3)	0.1334 (2)	0.45517 (19)	0.0653 (6)	
H33	0.8373	0.2060	0.4495	0.078*	
C34	0.9561 (2)	0.5167 (2)	0.23428 (18)	0.0600 (5)	
H34A	0.9804	0.5378	0.1619	0.090*	
H34B	1.0375	0.4619	0.2615	0.090*	
H34C	0.9203	0.6033	0.2533	0.090*	
O3	0.1064 (5)	0.6190 (5)	0.4934 (3)	0.0885 (11)	0.5
H3O	0.1791	0.6313	0.4529	0.133*	0.5
C35A	0.0517 (15)	0.5293 (19)	0.4674 (10)	0.067 (3)	0.25
H35A	0.0192	0.5837	0.4053	0.080*	0.25
H35B	0.1295	0.4524	0.4515	0.080*	0.25
C35B	−0.0043 (14)	0.5733 (13)	0.4846 (11)	0.058 (3)	0.25
H35C	−0.0904	0.6180	0.5235	0.069*	0.25
H35D	−0.0121	0.6094	0.4143	0.069*	0.25

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1073 (6)	0.0674 (4)	0.0697 (4)	−0.0169 (4)	−0.0261 (4)	0.0138 (3)
O1	0.0633 (8)	0.0546 (8)	0.0575 (8)	−0.0072 (6)	−0.0138 (7)	−0.0295 (7)
O2	0.0588 (8)	0.0722 (10)	0.0482 (8)	−0.0098 (8)	−0.0204 (6)	−0.0250 (7)
N1	0.0430 (8)	0.0512 (9)	0.0457 (9)	−0.0099 (7)	−0.0042 (6)	−0.0181 (7)
N2	0.0390 (7)	0.0422 (8)	0.0555 (9)	−0.0108 (6)	−0.0091 (6)	−0.0082 (7)
C1	0.0415 (9)	0.0444 (10)	0.0504 (10)	−0.0140 (7)	−0.0059 (8)	−0.0103 (8)
C2	0.0523 (11)	0.0584 (13)	0.0701 (14)	−0.0041 (9)	−0.0108 (10)	−0.0203 (11)
C3	0.0510 (11)	0.0571 (13)	0.0899 (18)	−0.0011 (10)	−0.0205 (11)	−0.0124 (12)
C4	0.0590 (12)	0.0553 (12)	0.0738 (15)	−0.0175 (10)	−0.0283 (11)	−0.0005 (11)
C5	0.0596 (11)	0.0518 (11)	0.0570 (12)	−0.0212 (9)	−0.0208 (9)	−0.0056 (9)
C6	0.0456 (9)	0.0437 (9)	0.0459 (10)	−0.0189 (7)	−0.0098 (7)	−0.0066 (7)

C7	0.0478 (9)	0.0475 (10)	0.0411 (9)	−0.0172 (8)	−0.0078 (7)	−0.0142 (8)
C8	0.0394 (8)	0.0415 (9)	0.0371 (9)	−0.0151 (7)	−0.0025 (7)	−0.0119 (7)
C9	0.0389 (8)	0.0440 (9)	0.0370 (8)	−0.0149 (7)	−0.0007 (7)	−0.0129 (7)
C10	0.0433 (9)	0.0513 (10)	0.0318 (8)	−0.0129 (7)	−0.0017 (7)	−0.0152 (7)
C11	0.0482 (9)	0.0481 (10)	0.0351 (9)	−0.0165 (8)	−0.0033 (7)	−0.0084 (7)
C12	0.0410 (8)	0.0356 (8)	0.0389 (9)	−0.0117 (7)	−0.0038 (7)	−0.0098 (7)
C13	0.0445 (9)	0.0410 (9)	0.0362 (8)	−0.0172 (7)	−0.0010 (7)	−0.0124 (7)
C14	0.0397 (8)	0.0374 (9)	0.0382 (9)	−0.0111 (7)	−0.0056 (7)	−0.0103 (7)
C15	0.0450 (9)	0.0469 (10)	0.0377 (9)	−0.0126 (7)	−0.0100 (7)	−0.0161 (7)
C16	0.0414 (9)	0.0432 (9)	0.0534 (10)	−0.0119 (7)	−0.0157 (7)	−0.0166 (8)
C17	0.0564 (11)	0.0514 (11)	0.0766 (14)	−0.0086 (9)	−0.0226 (10)	−0.0311 (10)
C18	0.0670 (13)	0.0395 (11)	0.111 (2)	−0.0067 (9)	−0.0369 (14)	−0.0227 (12)
C19	0.0595 (12)	0.0427 (11)	0.0920 (18)	−0.0165 (9)	−0.0333 (12)	0.0046 (11)
C20	0.0419 (9)	0.0486 (10)	0.0616 (12)	−0.0155 (8)	−0.0189 (8)	0.0015 (9)
C21	0.0379 (8)	0.0404 (9)	0.0475 (10)	−0.0138 (7)	−0.0127 (7)	−0.0080 (7)
C22	0.0396 (8)	0.0430 (9)	0.0384 (9)	−0.0147 (7)	−0.0039 (7)	−0.0087 (7)
C23	0.0514 (10)	0.0591 (12)	0.0447 (10)	−0.0176 (9)	0.0028 (8)	−0.0147 (9)
C24	0.0538 (11)	0.0867 (17)	0.0384 (10)	−0.0223 (11)	0.0010 (8)	−0.0075 (10)
C25	0.0507 (11)	0.0717 (15)	0.0512 (12)	−0.0220 (10)	−0.0124 (9)	0.0112 (10)
C26	0.0446 (10)	0.0438 (10)	0.0668 (13)	−0.0077 (8)	−0.0038 (9)	−0.0108 (9)
C27	0.0471 (9)	0.0386 (9)	0.0506 (10)	−0.0090 (7)	−0.0086 (8)	−0.0123 (8)
C28	0.0450 (9)	0.0375 (9)	0.0540 (10)	−0.0081 (7)	−0.0149 (8)	−0.0102 (8)
C29	0.0618 (11)	0.0492 (11)	0.0576 (12)	−0.0204 (9)	−0.0198 (9)	−0.0096 (9)
C30	0.0726 (14)	0.0496 (11)	0.0693 (14)	−0.0265 (10)	−0.0207 (11)	−0.0036 (10)
C31	0.0583 (12)	0.0464 (11)	0.0597 (12)	−0.0074 (9)	−0.0179 (10)	−0.0012 (9)
C32	0.0918 (17)	0.0656 (14)	0.0678 (15)	−0.0188 (13)	−0.0444 (13)	−0.0069 (11)
C33	0.0827 (15)	0.0525 (12)	0.0742 (15)	−0.0258 (11)	−0.0381 (12)	−0.0072 (10)
C34	0.0461 (10)	0.0555 (12)	0.0762 (15)	−0.0183 (9)	−0.0143 (10)	−0.0096 (10)
O3	0.105 (3)	0.113 (3)	0.058 (2)	−0.045 (3)	−0.0189 (19)	−0.018 (2)
C35A	0.076 (8)	0.089 (11)	0.057 (6)	−0.049 (8)	0.009 (7)	−0.039 (7)
C35B	0.061 (7)	0.045 (7)	0.060 (8)	0.005 (5)	−0.026 (6)	−0.012 (5)

Geometric parameters (Å, º)

Cl1—C31	1.743 (2)	C18—C19	1.364 (4)
O1—C13	1.216 (2)	C18—H18	0.9300
O2—C15	1.418 (2)	C19—C20	1.413 (3)
O2—H2O	0.87 (3)	C19—H19	0.9300
N1—C9	1.315 (2)	C20—C21	1.407 (3)
N1—C1	1.369 (2)	C20—C25	1.409 (3)
N2—C26	1.460 (3)	C21—C22	1.404 (2)
N2—C34	1.463 (2)	C22—C23	1.373 (3)
N2—C14	1.469 (2)	C23—C24	1.414 (3)
C1—C6	1.413 (3)	C23—H23	0.9300
C1—C2	1.414 (3)	C24—C25	1.361 (4)
C2—C3	1.365 (3)	C24—H24	0.9300
C2—H2	0.9300	C25—H25	0.9300
C3—C4	1.396 (4)	C26—C27	1.531 (3)

C3—H3	0.9300	C26—H26A	0.9700
C4—C5	1.354 (3)	C26—H26B	0.9700
C4—H4	0.9300	C27—C28	1.516 (3)
C5—C6	1.416 (3)	C27—H27	0.9800
C5—H5	0.9300	C28—C33	1.376 (3)
C6—C7	1.406 (3)	C28—C29	1.379 (3)
C7—C8	1.369 (3)	C29—C30	1.386 (3)
C7—H7	0.9300	C29—H29	0.9300
C8—C9	1.425 (2)	C30—C31	1.366 (3)
C8—C13	1.493 (2)	C30—H30	0.9300
C9—C10	1.505 (2)	C31—C32	1.370 (3)
C10—C11	1.523 (3)	C32—C33	1.387 (3)
C10—C15	1.560 (2)	C32—H32	0.9300
C10—H10	0.9800	C33—H33	0.9300
C11—C12	1.544 (2)	C34—H34A	0.9600
C11—H11A	0.9700	C34—H34B	0.9600
C11—H11B	0.9700	C34—H34C	0.9600
C12—C13	1.513 (2)	O3—C35B	1.434 (15)
C12—C27	1.561 (2)	O3—C35A	1.437 (13)
C12—C14	1.574 (2)	O3—H3O	0.8200
C14—C22	1.525 (2)	C35A—C35A^{i}	1.35 (3)
C14—C15	1.582 (2)	C35A—H35A	0.9700
C15—C16	1.509 (2)	C35A—H35B	0.9700
C16—C17	1.362 (3)	C35B—C35B^{i}	1.41 (3)
C16—C21	1.401 (3)	C35B—H35C	0.9700
C17—C18	1.409 (3)	C35B—H35D	0.9700
C17—H17	0.9300		

C15—O2—H2O	112 (2)	C19—C18—H18	118.8
C9—N1—C1	118.06 (15)	C17—C18—H18	118.8
C26—N2—C34	112.47 (16)	C18—C19—C20	120.5 (2)
C26—N2—C14	105.83 (15)	C18—C19—H19	119.7
C34—N2—C14	115.37 (15)	C20—C19—H19	119.7
N1—C1—C6	122.77 (17)	C21—C20—C25	116.39 (19)
N1—C1—C2	118.60 (18)	C21—C20—C19	115.8 (2)
C6—C1—C2	118.62 (19)	C25—C20—C19	127.8 (2)
C3—C2—C1	120.1 (2)	C16—C21—C22	113.43 (16)
C3—C2—H2	120.0	C16—C21—C20	123.31 (17)
C1—C2—H2	120.0	C22—C21—C20	123.25 (18)
C2—C3—C4	121.2 (2)	C23—C22—C21	118.69 (17)
C2—C3—H3	119.4	C23—C22—C14	132.84 (17)
C4—C3—H3	119.4	C21—C22—C14	108.43 (15)
C5—C4—C3	120.3 (2)	C22—C23—C24	118.6 (2)
C5—C4—H4	119.9	C22—C23—H23	120.7
C3—C4—H4	119.9	C24—C23—H23	120.7
C4—C5—C6	120.3 (2)	C25—C24—C23	122.5 (2)
C4—C5—H5	119.8	C25—C24—H24	118.7
C6—C5—H5	119.8	C23—C24—H24	118.7

C7—C6—C1	117.53 (17)	C24—C25—C20	120.43 (19)
C7—C6—C5	122.98 (18)	C24—C25—H25	119.8
C1—C6—C5	119.43 (18)	C20—C25—H25	119.8
C8—C7—C6	119.54 (17)	N2—C26—C27	105.35 (15)
C8—C7—H7	120.2	N2—C26—H26A	110.7
C6—C7—H7	120.2	C27—C26—H26A	110.7
C7—C8—C9	119.00 (16)	N2—C26—H26B	110.7
C7—C8—C13	120.79 (16)	C27—C26—H26B	110.7
C9—C8—C13	120.20 (15)	H26A—C26—H26B	108.8
N1—C9—C8	122.92 (16)	C28—C27—C26	113.54 (16)
N1—C9—C10	118.33 (15)	C28—C27—C12	115.08 (15)
C8—C9—C10	118.69 (15)	C26—C27—C12	103.16 (14)
C9—C10—C11	108.97 (14)	C28—C27—H27	108.2
C9—C10—C15	113.30 (14)	C26—C27—H27	108.2
C11—C10—C15	101.39 (14)	C12—C27—H27	108.2
C9—C10—H10	110.9	C33—C28—C29	117.68 (18)
C11—C10—H10	110.9	C33—C28—C27	122.91 (17)
C15—C10—H10	110.9	C29—C28—C27	119.39 (17)
C10—C11—C12	101.90 (13)	C28—C29—C30	121.9 (2)
C10—C11—H11A	111.4	C28—C29—H29	119.1
C12—C11—H11A	111.4	C30—C29—H29	119.1
C10—C11—H11B	111.4	C31—C30—C29	118.96 (19)
C12—C11—H11B	111.4	C31—C30—H30	120.5
H11A—C11—H11B	109.3	C29—C30—H30	120.5
C13—C12—C11	107.57 (14)	C30—C31—C32	120.8 (2)
C13—C12—C27	113.75 (15)	C30—C31—Cl1	119.69 (17)
C11—C12—C27	117.37 (15)	C32—C31—Cl1	119.56 (18)
C13—C12—C14	108.77 (13)	C31—C32—C33	119.4 (2)
C11—C12—C14	102.87 (13)	C31—C32—H32	120.3
C27—C12—C14	105.68 (13)	C33—C32—H32	120.3
O1—C13—C8	121.20 (16)	C28—C33—C32	121.3 (2)
O1—C13—C12	123.65 (16)	C28—C33—H33	119.4
C8—C13—C12	115.15 (14)	C32—C33—H33	119.4
N2—C14—C22	115.32 (13)	N2—C34—H34A	109.5
N2—C14—C12	102.19 (13)	N2—C34—H34B	109.5
C22—C14—C12	117.84 (14)	H34A—C34—H34B	109.5
N2—C14—C15	112.19 (14)	N2—C34—H34C	109.5
C22—C14—C15	103.50 (13)	H34A—C34—H34C	109.5
C12—C14—C15	105.66 (13)	H34B—C34—H34C	109.5
O2—C15—C16	112.06 (14)	C35A—O3—H3O	109.5
O2—C15—C10	107.96 (14)	C35A^{i}—C35A—O3	121.1 (13)
C16—C15—C10	115.87 (14)	C35A^{i}—C35A—H35A	107.1
O2—C15—C14	112.86 (14)	O3—C35A—H35A	107.1
C16—C15—C14	104.98 (13)	C35A^{i}—C35A—H35B	107.1
C10—C15—C14	102.84 (13)	O3—C35A—H35B	107.1
C17—C16—C21	119.23 (18)	H35A—C35A—H35B	106.8
C17—C16—C15	132.34 (19)	C35B^{i}—C35B—O3	118.6 (12)
C21—C16—C15	108.42 (15)	C35B^{i}—C35B—H35C	107.7

C16—C17—C18	118.6 (2)	O3—C35B—H35C	107.7
C16—C17—H17	120.7	C35B^{i}—C35B—H35D	107.7
C18—C17—H17	120.7	O3—C35B—H35D	107.7
C19—C18—C17	122.5 (2)	H35C—C35B—H35D	107.1
C9—N1—C1—C6	−2.3 (3)	N2—C14—C15—C16	114.11 (14)
C9—N1—C1—C2	176.14 (17)	C22—C14—C15—C16	−10.84 (16)
N1—C1—C2—C3	−178.35 (19)	C12—C14—C15—C16	−135.32 (13)
C6—C1—C2—C3	0.1 (3)	N2—C14—C15—C10	−124.27 (14)
C1—C2—C3—C4	−1.4 (4)	C22—C14—C15—C10	110.77 (14)
C2—C3—C4—C5	0.0 (3)	C12—C14—C15—C10	−13.71 (17)
C3—C4—C5—C6	2.6 (3)	O2—C15—C16—C17	−50.2 (3)
N1—C1—C6—C7	3.5 (3)	C10—C15—C16—C17	74.3 (2)
C2—C1—C6—C7	−174.87 (17)	C14—C15—C16—C17	−173.05 (19)
N1—C1—C6—C5	−179.23 (16)	O2—C15—C16—C21	131.07 (15)
C2—C1—C6—C5	2.4 (3)	C10—C15—C16—C21	−104.43 (16)
C4—C5—C6—C7	173.33 (18)	C14—C15—C16—C21	8.23 (17)
C4—C5—C6—C1	−3.7 (3)	C21—C16—C17—C18	−1.0 (3)
C1—C6—C7—C8	−0.8 (2)	C15—C16—C17—C18	−179.61 (18)
C5—C6—C7—C8	−177.90 (16)	C16—C17—C18—C19	2.0 (3)
C6—C7—C8—C9	−2.9 (2)	C17—C18—C19—C20	−0.3 (3)
C6—C7—C8—C13	175.65 (15)	C18—C19—C20—C21	−2.1 (3)
C1—N1—C9—C8	−1.7 (2)	C18—C19—C20—C25	177.5 (2)
C1—N1—C9—C10	−179.11 (15)	C17—C16—C21—C22	179.04 (16)
C7—C8—C9—N1	4.4 (2)	C15—C16—C21—C22	−2.0 (2)
C13—C8—C9—N1	−174.18 (15)	C17—C16—C21—C20	−1.5 (3)
C7—C8—C9—C10	−178.25 (15)	C15—C16—C21—C20	177.38 (15)
C13—C8—C9—C10	3.2 (2)	C25—C20—C21—C16	−176.57 (16)
N1—C9—C10—C11	142.05 (15)	C19—C20—C21—C16	3.0 (3)
C8—C9—C10—C11	−35.4 (2)	C25—C20—C21—C22	2.8 (3)
N1—C9—C10—C15	−105.91 (17)	C19—C20—C21—C22	−177.60 (17)
C8—C9—C10—C15	76.61 (19)	C16—C21—C22—C23	176.50 (16)
C9—C10—C11—C12	68.81 (16)	C20—C21—C22—C23	−2.9 (3)
C15—C10—C11—C12	−50.91 (16)	C16—C21—C22—C14	−5.43 (19)
C10—C11—C12—C13	−73.10 (16)	C20—C21—C22—C14	175.16 (15)
C10—C11—C12—C27	157.13 (15)	N2—C14—C22—C23	64.8 (3)
C10—C11—C12—C14	41.64 (17)	C12—C14—C22—C23	−56.1 (3)
C7—C8—C13—O1	−5.8 (2)	C15—C14—C22—C23	−172.29 (19)
C9—C8—C13—O1	172.71 (16)	N2—C14—C22—C21	−112.90 (16)
C7—C8—C13—C12	174.08 (14)	C12—C14—C22—C21	126.16 (15)
C9—C8—C13—C12	−7.4 (2)	C15—C14—C22—C21	10.01 (17)
C11—C12—C13—O1	−137.17 (17)	C21—C22—C23—C24	0.5 (3)
C27—C12—C13—O1	−5.4 (2)	C14—C22—C23—C24	−176.98 (18)
C14—C12—C13—O1	112.08 (18)	C22—C23—C24—C25	1.9 (3)
C11—C12—C13—C8	42.91 (18)	C23—C24—C25—C20	−2.0 (3)
C27—C12—C13—C8	174.68 (13)	C21—C20—C25—C24	−0.3 (3)
C14—C12—C13—C8	−67.84 (17)	C19—C20—C25—C24	−179.9 (2)
C26—N2—C14—C22	−89.00 (17)	C34—N2—C26—C27	−169.53 (17)

C34—N2—C14—C22	36.0 (2)	C14—N2—C26—C27	−42.71 (19)
C26—N2—C14—C12	40.12 (17)	N2—C26—C27—C28	−99.30 (18)
C34—N2—C14—C12	165.16 (16)	N2—C26—C27—C12	25.9 (2)
C26—N2—C14—C15	152.85 (15)	C13—C12—C27—C28	−118.14 (16)
C34—N2—C14—C15	−82.11 (19)	C11—C12—C27—C28	8.7 (2)
C13—C12—C14—N2	−145.22 (14)	C14—C12—C27—C28	122.61 (16)
C11—C12—C14—N2	100.90 (15)	C13—C12—C27—C26	117.64 (17)
C27—C12—C14—N2	−22.73 (17)	C11—C12—C27—C26	−115.54 (18)
C13—C12—C14—C22	−17.71 (19)	C14—C12—C27—C26	−1.61 (19)
C11—C12—C14—C22	−131.58 (15)	C26—C27—C28—C33	36.1 (3)
C27—C12—C14—C22	104.78 (17)	C12—C27—C28—C33	−82.5 (2)
C13—C12—C14—C15	97.27 (15)	C26—C27—C28—C29	−142.47 (19)
C11—C12—C14—C15	−16.61 (17)	C12—C27—C28—C29	99.0 (2)
C27—C12—C14—C15	−140.24 (14)	C33—C28—C29—C30	0.3 (3)
C9—C10—C15—O2	163.29 (14)	C27—C28—C29—C30	178.93 (19)
C11—C10—C15—O2	−80.11 (16)	C28—C29—C30—C31	0.6 (3)
C9—C10—C15—C16	36.7 (2)	C29—C30—C31—C32	−0.7 (4)
C11—C10—C15—C16	153.30 (15)	C29—C30—C31—Cl1	179.10 (17)
C9—C10—C15—C14	−77.19 (17)	C30—C31—C32—C33	−0.3 (4)
C11—C10—C15—C14	39.40 (16)	Cl1—C31—C32—C33	179.97 (19)
N2—C14—C15—O2	−8.21 (19)	C29—C28—C33—C32	−1.3 (3)
C22—C14—C15—O2	−133.17 (15)	C27—C28—C33—C32	−179.8 (2)
C12—C14—C15—O2	102.35 (16)	C31—C32—C33—C28	1.3 (4)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*O*···N2	0.87 (3)	2.20 (3)	2.695 (2)	116 (2)
O3—H3*O*···N1	0.82	2.28	2.984 (4)	145
O2—H2*O*···O3^{ii}	0.87 (3)	2.26 (3)	3.021 (5)	146 (2)
C33—H33···O3^{ii}	0.93	2.53	3.408 (6)	157

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