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1,4,9,12-Tetramethoxy-14-octyl-5,8-dihydro-diindolo[3,2-*b*;2′,3′-*h*]carbazole with an unknown solvent

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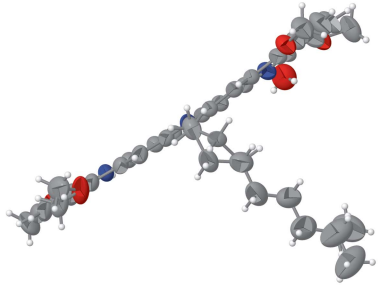
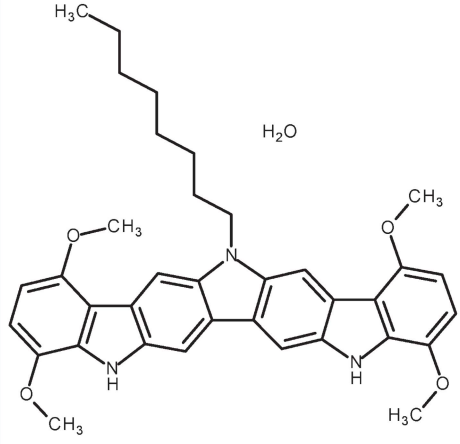
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $2C_{36}H_{39}N_3O_4{\cdot}H_2O$, is a linear π-conjugated ladder oligomer with an alkyl chain on the central nitrogen atom. This diindolocarbazole, prepared *via* a twofold Cadogan reaction, adopts a sligthly convex shape, *anti* to the disordered octyl group. The unit cell contains nine molecules of the title compound and half a water molecule per main molecule. The water molecule forms hydrogen bridges, connecting the carbazole-NH and methoxy groups of different molecules. The crystal contains solvent molecules which are located in a channel parallel to the *c* axis. It was not possible to determine the position and nature of the solvent (a mixure of choroform, *n*-pentane and DMSO). The SQUEEZE [Spek (2015). *Acta Cryst.* C**71**, 9–18] option of *PLATON* was used to model the missing electron density. The given chemical formula and other crystal data do not take into account these solvent molecules.

3D view



Chemical scheme



Structure description

Indolocarbazoles are currently investigated as materials for optical (Nemkovich *et al.*, 2009) and electronic applications (Wakim *et al.*, 2004; Zheng *et al.*, 2015). Among the different synthetic routes (Vlasselaer & Dehaen, 2016), the twofold Cadogan reaction (Cadogan *et al.*, 1965) is a very successful route to indolocarbazoles (Kistenmacher & Müllen, 1992; Wrobel *et al.*, 2013) and is also suitable for the preparation of higher oligomers (Srour *et al.*, 2016). In a continuation of our studies on indolo-annulated heterocycles (Dassonneville *et al.*, 2011; Nissen & Detert, 2011; Letessier & Detert, 2013), we present here the first X-ray structure of a linear diindolocarbazole (Fig. 1).

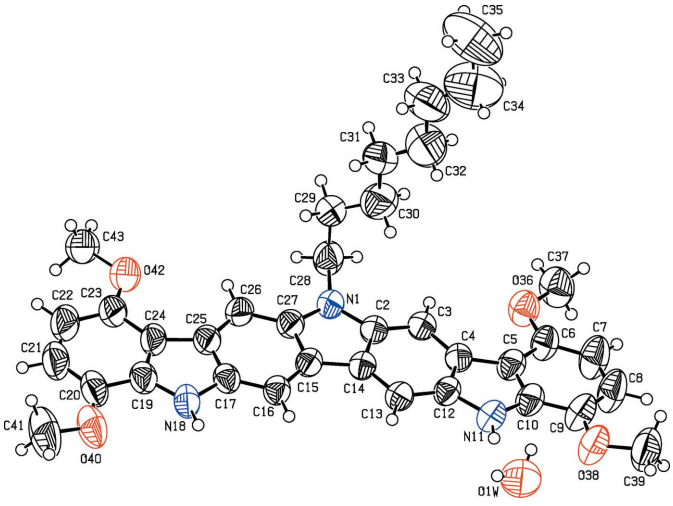


Figure 1
The crystal structure of the title compound, with the atom labelling and displacement ellipsoids drawn at the 50% probability level. Only the major occupancy sites of the disordered atoms are shown.

The unit cell contains nine molecules of the title compound and half a water molecule per title molecule. The water forms hydrogen bridges, connecting the carbazole-NH and methoxy groups of three molecules (Table 1, Fig. 2). The π-conjugated segment is slightly convex, with the alkyl chain on top. Two nearly planar carbazole units [maximum deviations of 0.074 (4) Å for C9 from the mean plane through N11/C2–C10/C12–C14 and 0.017 (3) Å for C27 of the mean plane through N18/C15–C17/C19–C27) enclose an angle of 8.17 (7)°, *anti* to the alkyl chain. Two modes of disorder are present in the octyl chain: the terminal methyl group adopts an *anti* and a *gauche* conformation (occupancy ratio 0.70:0.30), with torsion angles of 172.8 (6)° (*anti*, C312—C33—C34—C35) and −60.1 (12)° (*gauche*, C35*A*—C34—C33–C32). Similarly, the disorder at C29 (0.85:0.15 occupancy ratio *anti*/*gauche*) is characterized by torsion angles of −176.3 (3)° for C28—C28—C29—C31 and −121.9 (13)° for C28—C29*A*—C30—C23.

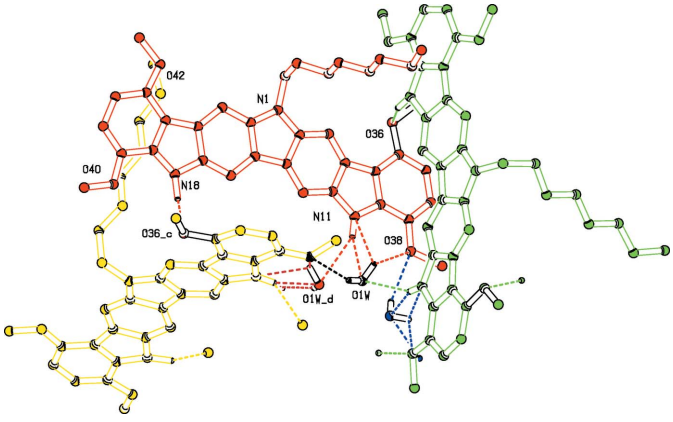


Figure 2
A partial packing diagram, showing the hydrogen-bonding network. View along the *c* axis. Most of the H atoms omitted for clarity. Symmetry-equivalent molecules are drawn with different colours.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···O1*W*	0.88	2.03	2.879 (5)	162
N11—H11···O1*W*^{i}	0.88	2.45	3.238 (6)	149
N18—H18···O36^{i}	1.09	2.03	3.068 (3)	157
O1*W*—H1*W*···N11	0.84	2.43	2.879 (5)	114
O1*W*—H1*W*···O38	0.84	2.41	2.900 (6)	118
O1*W*—H2*W*···O38^{i}	0.75	2.48	3.181 (6)	158

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

Synthesis and crystallization

200 mg of 9-octyl-2,7-bis(2,5-dimethoxyphenyl)-3,6-dinitro-9-*H*-carbazole was mixed with triethyl phosphite (4 ml) and heated for 30 min in a microwave oven (483 K, 300 W). The mixture was dissolved in ethyl acetate, hydrochloric acid (6 *M*, 30 ml) was added and stirred at 353 K for 3 h. The product was isolated by extraction with dichloromethane from an aqueous solution followed by column chromatography (SiO_2, petroleum ether:ethyl acetate 5:1). Yield 37 mg (20%), m.p. 392 K. Single crystals were obtained from a solution in DMSO and chloroform.

^{1}H NMR: (400 MHz, DMSO-δ_6): *d* = 11.16 (*s*, 2 H, N—H), 8.10 (*s*, 2 H. 6-H, 7-H), 6.89 (*d*, *J* = 8.5 Hz, 2 H, 3-H, 10-H), 6.55 (*d*, *J* = 8.7 Hz, 2 H, 2-H, 11-H), 4.45 (*t*, *J* = 6.9 Hz, 2 H, OCH_2), 4.03 (*s*, 6 H, 1-OCH_3, 12-OCH_3), 3.95 (*s*, 6 H, 4-OCH_3, 9-OCH_3), 1.91 (*m*, 2 H), 1.39–1.22 (*m*, 10 H), 0.78 (*t*, *J* = 7 Hz, 3 H, CH_3). ^{1}C NMR: (400 MHz, DMSO-δ_6): *d* = 149.9 (C-1, C-12), 139.9 (C-4, C-9), 136.9 (C-13a, C-14a), 133.9 (C-5a, C-7a), 131.8 (C-4a, C-8a), 121.9 (C-15*a*, C-12*b*), 121.5 (C-6a, C-6 b), 112.7 (C12a, C15b), 106.4 (C-3, C-10), 100.7 (C-6, C-7),

Table 2
Experimental details.

Crystal data	
Chemical formula	$2C_{36}H_{39}N_3O_4{\cdot}H_2O$
M_r	1173.42
Crystal system, space group	Trigonal, $R\overline{3}$:*H*
Temperature (K)	193
a, *c* (Å)	26.3932 (5), 24.8270 (5)
V (Å^3)	14977.5 (6)
Z	9
Radiation type	Mo *K*α
μ (mm^{-1})	0.08
Crystal size (mm)	0.27 × 0.17 × 0.14
Data collection	
Diffractometer	Bruker SMART APEXII
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	131773, 7932, 2895
R_{int}	0.145
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.064, 0.222, 0.85
No. of reflections	7932
No. of parameters	429
No. of restraints	24
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.39, −0.24

Computer programs: *SMART*, *APEX2* and *SAINT* (Bruker, 1997), *SIR2004* (Altomare *et al.*, 1999), *SHELXL2016* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

97.9 (C-2, C-11), 55.7 (4-OCH_3, 9-OCH_3), 55.5 (1-OCH_3, 12-OCH_3), 42.4 (OCH_2), 31.2, 28.6 (2 C), 27.8, 26.5, 22.0, 13.9 (CH_3); IR (ATR): 3424, 3064, 2992, 2925, 2850, 1624, 1597, 1515, 1460, 1420, 1378, 1346, 1305, 1280, 1252, 1223, 1161, 1108, 1089, 1016, 970, 844 cm^{-1}; MS: (FD): 578 ($M^{\cdot+*}$), HR–ESI: 578.3019, calculated for $C_{36}H_{40}N_3O_4^+$: 578.3011

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Two modes of disorder are present in the octyl chain: the terminal methyl group (occupancy ratio 0.70:0.30) and atom C29 (occupancy ratio 0.85:0.15). The s.o.f. for the disordered carbon atoms were kept fixed, while the anisotropic thermal parameters were refined using the ISOR instruction. The water molecule was refined with an s.o.f of 0.5. The crystal contains solvent molecules which are located in a channel parallel to the *c* axis. It was not possible to determine the position and nature of the solvent (mixure of choroform, *n*-pentan and DMSO). The SQUEEZE (Spek, 2015) option of *PLATON* (Spek, 2009) was used to model the missing electron density.

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full crystallographic data

IUCrData (2017). **2**, x170462 [https://doi.org/10.1107/S241431461700462X]

1,4,9,12-Tetramethoxy-14-octyl-5,8-dihydrodiindolo[3,2-*b*;2′,3′-*h*]carbazole with an unknown solvent

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1,4,9,12-Tetramethoxy-14-octyl-5,8-dihydrodiindolo[3,2-*b*;2′,3′-*h*]carbazole

Crystal data

$2C_{36}H_{39}N_3O_4{\cdot}H_2O$
M_r = 1173.42
Trigonal, $R\bar{3}$:*H*
a = 26.3932 (5) Å
c = 24.8270 (5) Å
V = 14977.5 (6) Å^3
Z = 9
F(000) = 5634
D_x = 1.171 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 8684 reflections
θ = 2.4–19.1°
μ = 0.08 mm^{-1}
T = 193 K
Block, colourless
0.27 × 0.17 × 0.14 mm

Data collection

Bruker SMART APEXII diffractometer
Radiation source: sealed tube
CCD scan
131773 measured reflections
7932 independent reflections
2895 reflections with $I > 2\sigma(I)$
R_{int} = 0.145
θ_{max} = 27.9°, θ_{min} = 1.5°
h = −33→34
k = −34→33
l = −32→32

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.222
S = 0.85
7932 reflections
429 parameters
24 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1266P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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.

Refinement. Hydrogen atoms attached to carbons were placed at calculated positions with C—H = 0.95 Å (aromatic) or 0.98–0.99 Å (sp^3 C-atom). All H atoms were refined in the riding-model approximation with isotropic displacement parameters (set at 1.2–1.5 times of the U_{eq} of the parent atom).

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
N1	0.39718 (9)	0.15387 (9)	−0.05021 (9)	0.0616 (6)	
C2	0.33803 (11)	0.11326 (11)	−0.05643 (10)	0.0569 (7)	
C3	0.31146 (11)	0.06711 (11)	−0.09279 (10)	0.0577 (7)	
H3	0.333786	0.058309	−0.117113	0.069*	
C4	0.25060 (11)	0.03444 (11)	−0.09185 (10)	0.0568 (7)	
C5	0.20813 (12)	−0.01553 (12)	−0.12255 (11)	0.0663 (7)	
C6	0.21232 (14)	−0.04878 (13)	−0.16476 (12)	0.0738 (8)	
C7	0.16204 (16)	−0.09611 (16)	−0.18386 (14)	0.1020 (11)	
H7	0.164166	−0.117596	−0.213625	0.122*	
C8	0.10804 (16)	−0.11305 (17)	−0.16021 (14)	0.1040 (12)	
H8	0.074218	−0.146831	−0.173361	0.125*	
C9	0.10238 (14)	−0.08218 (15)	−0.11835 (13)	0.0875 (10)	
C10	0.15277 (13)	−0.03232 (13)	−0.10074 (11)	0.0696 (8)	
N11	0.15846 (9)	0.00550 (10)	−0.05968 (9)	0.0687 (6)	
H11	0.129679	0.003742	−0.040337	0.082*	
C12	0.21804 (11)	0.04723 (11)	−0.05408 (10)	0.0590 (7)	
C13	0.24413 (12)	0.09410 (11)	−0.01950 (10)	0.0591 (7)	
H13	0.221748	0.102997	0.004654	0.071*	
C14	0.30485 (11)	0.12791 (11)	−0.02150 (9)	0.0554 (7)	
C15	0.34586 (11)	0.18072 (11)	0.00642 (10)	0.0578 (7)	
C16	0.33799 (12)	0.21639 (11)	0.04236 (10)	0.0596 (7)	
H16	0.300219	0.207086	0.054655	0.072*	
C17	0.38772 (13)	0.26599 (11)	0.05931 (10)	0.0610 (7)	
N18	0.39149 (11)	0.30915 (9)	0.09462 (9)	0.0695 (6)	
H18	0.351821	0.296759	0.117984	0.083*	
C19	0.44947 (15)	0.35066 (12)	0.09997 (12)	0.0738 (8)	
C20	0.47299 (18)	0.40164 (13)	0.13206 (14)	0.0839 (10)	
C21	0.5324 (2)	0.43785 (14)	0.13098 (17)	0.1015 (12)	
H21	0.549631	0.472860	0.151597	0.122*	
C22	0.56788 (18)	0.42417 (15)	0.10023 (19)	0.1097 (14)	
H22	0.608978	0.450052	0.101049	0.132*	
C23	0.54594 (16)	0.37413 (14)	0.06817 (16)	0.0923 (11)	
C24	0.48467 (14)	0.33606 (12)	0.06833 (13)	0.0750 (9)	
C25	0.44519 (13)	0.28154 (11)	0.04191 (12)	0.0676 (8)	
C26	0.45295 (12)	0.24538 (12)	0.00583 (12)	0.0678 (8)	
H26	0.490808	0.254411	−0.005931	0.081*	
C27	0.40278 (12)	0.19545 (11)	−0.01222 (11)	0.0611 (7)	
C28	0.44510 (13)	0.14724 (14)	−0.06915 (13)	0.0830 (9)	
H28A	0.479014	0.186180	−0.077441	0.100*	0.85
H28B	0.433502	0.123775	−0.102669	0.100*	0.85
H28C	0.448498	0.156016	−0.108184	0.100*	0.15
H28D	0.480695	0.179279	−0.052218	0.100*	0.15
C29	0.46330 (18)	0.11551 (18)	−0.0245 (2)	0.0924 (13)	0.85
H29A	0.501773	0.120371	−0.034567	0.111*	0.85
H29B	0.468401	0.135942	0.010268	0.111*	0.85

C29A	0.4496 (8)	0.0993 (9)	−0.0641 (8)	0.058 (5)	0.15
H29C	0.491994	0.112866	−0.064435	0.070*	0.15
H29D	0.433341	0.076631	−0.097653	0.070*	0.15
C30	0.42270 (17)	0.05359 (18)	−0.01624 (15)	0.1057 (12)	
H30A	0.419618	0.032706	−0.050292	0.127*	0.85
H30B	0.383581	0.048371	−0.008451	0.127*	0.85
H30C	0.401214	0.069270	0.003999	0.127*	0.15
H30D	0.391820	0.018897	−0.035301	0.127*	0.15
C31	0.4386 (2)	0.02484 (19)	0.02872 (19)	0.1261 (15)	
H31A	0.478014	0.031055	0.020650	0.151*	
H31B	0.441973	0.046529	0.062317	0.151*	
C32	0.4015 (2)	−0.0360 (2)	0.03997 (16)	0.1324 (16)	
H32A	0.396167	−0.057599	0.005916	0.159*	
H32B	0.362699	−0.041886	0.050309	0.159*	
C33	0.4192 (2)	−0.0638 (2)	0.0815 (2)	0.1427 (17)	
H33A	0.458966	−0.055696	0.071930	0.171*	
H33B	0.422937	−0.042822	0.115696	0.171*	
C34	0.3843 (4)	−0.1264 (3)	0.0933 (3)	0.192 (3)	
H34A	0.346215	−0.134099	0.107849	0.230*	0.7
H34B	0.376296	−0.147749	0.058667	0.230*	0.7
H34C	0.383354	−0.147994	0.060296	0.230*	0.3
H34D	0.405145	−0.135721	0.121238	0.230*	0.3
C35	0.4083 (5)	−0.1519 (4)	0.1304 (3)	0.202 (4)	0.7
H35A	0.380554	−0.193783	0.134472	0.304*	0.7
H35B	0.445381	−0.146321	0.116116	0.304*	0.7
H35C	0.415159	−0.132607	0.165531	0.304*	0.7
C35A	0.3246 (8)	−0.1491 (10)	0.1112 (8)	0.179 (7)	0.3
H35D	0.306388	−0.191287	0.117537	0.269*	0.3
H35E	0.324431	−0.129526	0.144661	0.269*	0.3
H35F	0.302544	−0.141854	0.083450	0.269*	0.3
O36	0.26787 (9)	−0.03024 (9)	−0.18319 (8)	0.0809 (6)	
C37	0.27329 (16)	−0.06189 (16)	−0.22806 (13)	0.1004 (11)	
H37A	0.259234	−0.102542	−0.217425	0.151*	
H37B	0.314429	−0.043590	−0.238912	0.151*	
H37C	0.249893	−0.061055	−0.258331	0.151*	
O38	0.05177 (9)	−0.09506 (10)	−0.09089 (9)	0.0999 (7)	
C39	0.00197 (15)	−0.15248 (16)	−0.09840 (17)	0.1205 (14)	
H39A	−0.012199	−0.156443	−0.135507	0.181*	
H39B	−0.029162	−0.158138	−0.073385	0.181*	
H39C	0.013321	−0.182006	−0.091425	0.181*	
O40	0.43208 (12)	0.40842 (9)	0.15991 (9)	0.1011 (8)	
C41	0.4547 (2)	0.45374 (17)	0.19893 (16)	0.1433 (18)	
H41A	0.485908	0.452607	0.219340	0.215*	
H41B	0.423350	0.448265	0.223561	0.215*	
H41C	0.470514	0.491679	0.180841	0.215*	
O42	0.57773 (10)	0.35734 (9)	0.03698 (13)	0.1135 (9)	
C43	0.6389 (5)	0.3968 (5)	0.0414 (4)	0.102 (3)	0.57
H43A	0.646779	0.437003	0.036787	0.153*	0.57

H43B	0.659522	0.387936	0.013464	0.153*	0.57
H43C	0.652498	0.392724	0.076988	0.153*	0.57
C43A	0.6370 (7)	0.3986 (8)	0.0112 (5)	0.114 (5)	0.43
H43D	0.668387	0.405174	0.036311	0.171*	0.43
H43E	0.639392	0.436013	0.002852	0.171*	0.43
H43F	0.641233	0.381021	−0.022029	0.171*	0.43
O1W	0.0541 (2)	−0.0271 (2)	0.0018 (2)	0.1203 (16)	0.5
H1W	0.070937	−0.046260	−0.004863	0.180*	0.5
H2W	0.068337	−0.007660	0.025637	0.180*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0533 (14)	0.0561 (13)	0.0704 (14)	0.0236 (11)	−0.0004 (11)	−0.0020 (11)
C2	0.0586 (17)	0.0549 (16)	0.0546 (15)	0.0264 (14)	−0.0025 (13)	0.0037 (13)
C3	0.0597 (17)	0.0578 (16)	0.0545 (15)	0.0284 (14)	0.0005 (13)	0.0043 (13)
C4	0.0626 (17)	0.0525 (15)	0.0518 (15)	0.0262 (14)	−0.0066 (13)	0.0023 (12)
C5	0.0637 (19)	0.0687 (18)	0.0593 (17)	0.0278 (15)	−0.0099 (14)	−0.0012 (15)
C6	0.070 (2)	0.074 (2)	0.0631 (18)	0.0250 (17)	−0.0109 (16)	−0.0156 (15)
C7	0.085 (3)	0.105 (3)	0.083 (2)	0.023 (2)	−0.007 (2)	−0.033 (2)
C8	0.081 (3)	0.107 (3)	0.089 (2)	0.021 (2)	−0.017 (2)	−0.041 (2)
C9	0.056 (2)	0.098 (2)	0.084 (2)	0.0200 (18)	−0.0081 (17)	−0.011 (2)
C10	0.067 (2)	0.0739 (19)	0.0572 (17)	0.0276 (17)	−0.0144 (15)	−0.0058 (15)
N11	0.0555 (15)	0.0737 (15)	0.0665 (15)	0.0244 (13)	−0.0043 (11)	−0.0029 (12)
C12	0.0547 (16)	0.0584 (16)	0.0561 (16)	0.0225 (14)	−0.0064 (13)	0.0032 (13)
C13	0.0599 (17)	0.0625 (17)	0.0525 (15)	0.0288 (15)	−0.0015 (13)	0.0000 (13)
C14	0.0579 (17)	0.0553 (16)	0.0482 (14)	0.0248 (14)	−0.0033 (13)	0.0041 (12)
C15	0.0551 (16)	0.0541 (16)	0.0570 (15)	0.0220 (13)	−0.0045 (13)	0.0051 (13)
C16	0.0604 (17)	0.0574 (16)	0.0564 (15)	0.0260 (14)	−0.0057 (13)	−0.0025 (13)
C17	0.074 (2)	0.0516 (16)	0.0562 (16)	0.0302 (15)	−0.0093 (14)	−0.0010 (13)
N18	0.0786 (17)	0.0510 (13)	0.0654 (14)	0.0222 (13)	−0.0132 (12)	−0.0035 (11)
C19	0.086 (2)	0.0523 (18)	0.076 (2)	0.0290 (18)	−0.0213 (17)	0.0022 (15)
C20	0.116 (3)	0.0511 (19)	0.077 (2)	0.036 (2)	−0.032 (2)	−0.0049 (16)
C21	0.115 (3)	0.049 (2)	0.119 (3)	0.025 (2)	−0.046 (3)	−0.0009 (19)
C22	0.092 (3)	0.051 (2)	0.160 (4)	0.017 (2)	−0.041 (3)	0.013 (2)
C23	0.080 (2)	0.050 (2)	0.138 (3)	0.0252 (19)	−0.029 (2)	0.004 (2)
C24	0.074 (2)	0.0458 (17)	0.091 (2)	0.0194 (16)	−0.0242 (17)	0.0043 (15)
C25	0.069 (2)	0.0514 (17)	0.0766 (19)	0.0257 (15)	−0.0139 (16)	0.0057 (15)
C26	0.0552 (17)	0.0559 (17)	0.086 (2)	0.0230 (15)	−0.0039 (15)	0.0083 (15)
C27	0.0595 (18)	0.0532 (16)	0.0657 (17)	0.0246 (14)	−0.0030 (14)	0.0061 (14)
C28	0.0632 (19)	0.077 (2)	0.099 (2)	0.0273 (17)	0.0086 (17)	−0.0136 (18)
C29	0.077 (3)	0.080 (3)	0.127 (4)	0.043 (2)	−0.021 (3)	−0.024 (3)
C29A	0.056 (11)	0.076 (14)	0.060 (11)	0.047 (11)	−0.010 (10)	−0.011 (11)
C30	0.110 (3)	0.117 (3)	0.097 (3)	0.062 (3)	−0.020 (2)	−0.012 (2)
C31	0.128 (3)	0.103 (3)	0.170 (4)	0.074 (3)	−0.061 (3)	−0.038 (3)
C32	0.174 (4)	0.128 (4)	0.101 (3)	0.080 (4)	−0.034 (3)	−0.008 (3)
C33	0.203 (5)	0.139 (4)	0.130 (4)	0.118 (4)	−0.025 (4)	−0.014 (3)
C34	0.233 (7)	0.215 (7)	0.164 (5)	0.139 (6)	0.025 (5)	0.061 (5)

C35	0.319 (11)	0.184 (7)	0.160 (7)	0.167 (8)	0.067 (7)	0.051 (6)
C35A	0.138 (12)	0.225 (16)	0.156 (13)	0.077 (11)	−0.011 (11)	−0.022 (11)
O36	0.0801 (15)	0.0857 (14)	0.0682 (13)	0.0350 (12)	−0.0015 (11)	−0.0177 (11)
C37	0.113 (3)	0.106 (3)	0.078 (2)	0.052 (2)	0.0006 (19)	−0.026 (2)
O38	0.0648 (14)	0.0994 (17)	0.1058 (17)	0.0189 (13)	−0.0138 (13)	−0.0196 (13)
C39	0.075 (2)	0.096 (3)	0.150 (4)	0.013 (2)	−0.013 (2)	−0.026 (3)
O40	0.145 (2)	0.0707 (15)	0.0760 (14)	0.0449 (15)	−0.0241 (15)	−0.0223 (12)
C41	0.207 (5)	0.093 (3)	0.102 (3)	0.055 (3)	−0.042 (3)	−0.046 (2)
O42	0.0677 (16)	0.0602 (14)	0.195 (3)	0.0189 (13)	−0.0109 (16)	0.0058 (15)
C43	0.069 (5)	0.067 (4)	0.157 (9)	0.024 (4)	0.006 (7)	0.023 (7)
C43A	0.074 (6)	0.098 (8)	0.127 (10)	0.011 (5)	0.002 (9)	0.030 (8)
O1W	0.109 (4)	0.125 (4)	0.135 (4)	0.065 (3)	0.005 (3)	−0.010 (3)

Geometric parameters (Å, º)

N1—C2	1.392 (3)	C29—C30	1.453 (5)
N1—C27	1.398 (3)	C29—H29A	0.9900
N1—C28	1.440 (3)	C29—H29B	0.9900
C2—C3	1.392 (3)	C29A—C30	1.586 (19)
C2—C14	1.416 (3)	C29A—H29C	0.9900
C3—C4	1.393 (3)	C29A—H29D	0.9900
C3—H3	0.9500	C30—C31	1.521 (5)
C4—C12	1.422 (4)	C30—H30A	0.9900
C4—C5	1.449 (4)	C30—H30B	0.9900
C5—C10	1.406 (4)	C30—H30C	0.9900
C5—C6	1.406 (4)	C30—H30D	0.9900
C6—O36	1.372 (3)	C31—C32	1.429 (6)
C6—C7	1.374 (4)	C31—H31A	0.9900
C7—C8	1.393 (5)	C31—H31B	0.9900
C7—H7	0.9500	C32—C33	1.470 (5)
C8—C9	1.374 (5)	C32—H32A	0.9900
C8—H8	0.9500	C32—H32B	0.9900
C9—O38	1.382 (4)	C33—C34	1.464 (7)
C9—C10	1.394 (4)	C33—H33A	0.9900
C10—N11	1.381 (3)	C33—H33B	0.9900
N11—C12	1.405 (3)	C34—C35A	1.448 (18)
N11—H11	0.8800	C34—C35	1.459 (9)
C12—C13	1.375 (3)	C34—H34A	0.9900
C13—C14	1.392 (3)	C34—H34B	0.9900
C13—H13	0.9500	C34—H34C	0.9900
C14—C15	1.444 (3)	C34—H34D	0.9900
C15—C16	1.386 (4)	C35—H35A	0.9800
C15—C27	1.428 (4)	C35—H35B	0.9800
C16—C17	1.377 (4)	C35—H35C	0.9800
C16—H16	0.9500	C35A—H35D	0.9800
C17—N18	1.401 (3)	C35A—H35E	0.9800
C17—C25	1.426 (4)	C35A—H35F	0.9800
N18—C19	1.372 (4)	O36—C37	1.442 (3)

N18—H18	1.0943	C37—H37A	0.9800
C19—C24	1.410 (4)	C37—H37B	0.9800
C19—C20	1.412 (4)	C37—H37C	0.9800
C20—O40	1.367 (4)	O38—C39	1.438 (4)
C20—C21	1.369 (5)	C39—H39A	0.9800
C21—C22	1.388 (5)	C39—H39B	0.9800
C21—H21	0.9500	C39—H39C	0.9800
C22—C23	1.396 (5)	O40—C41	1.418 (4)
C22—H22	0.9500	C41—H41A	0.9800
C23—O42	1.368 (4)	C41—H41B	0.9800
C23—C24	1.414 (4)	C41—H41C	0.9800
C24—C25	1.445 (4)	O42—C43	1.422 (12)
C25—C26	1.397 (4)	O42—C43A	1.529 (16)
C26—C27	1.395 (4)	C43—H43A	0.9800
C26—H26	0.9500	C43—H43B	0.9800
C28—C29A	1.334 (18)	C43—H43C	0.9800
C28—C29	1.601 (5)	C43A—H43D	0.9800
C28—H28A	0.9900	C43A—H43E	0.9800
C28—H28B	0.9900	C43A—H43F	0.9800
C28—H28C	0.9900	O1W—H1W	0.8406
C28—H28D	0.9900	O1W—H2W	0.7487

C2—N1—C27	108.5 (2)	H29A—C29—H29B	107.4
C2—N1—C28	125.9 (2)	C28—C29A—C30	125.0 (13)
C27—N1—C28	124.2 (2)	C28—C29A—H29C	106.1
C3—C2—N1	128.8 (2)	C30—C29A—H29C	106.1
C3—C2—C14	121.8 (2)	C28—C29A—H29D	106.1
N1—C2—C14	109.3 (2)	C30—C29A—H29D	106.1
C2—C3—C4	116.8 (2)	H29C—C29A—H29D	106.3
C2—C3—H3	121.6	C29—C30—C31	115.6 (4)
C4—C3—H3	121.6	C31—C30—C29A	143.0 (7)
C3—C4—C12	120.7 (2)	C29—C30—H30A	108.4
C3—C4—C5	133.1 (3)	C31—C30—H30A	108.4
C12—C4—C5	106.2 (2)	C29—C30—H30B	108.4
C10—C5—C6	118.9 (3)	C31—C30—H30B	108.4
C10—C5—C4	107.1 (2)	H30A—C30—H30B	107.4
C6—C5—C4	133.9 (3)	C31—C30—H30C	101.2
O36—C6—C7	125.7 (3)	C29A—C30—H30C	101.2
O36—C6—C5	115.4 (2)	C31—C30—H30D	101.2
C7—C6—C5	118.9 (3)	C29A—C30—H30D	101.2
C6—C7—C8	121.0 (3)	H30C—C30—H30D	104.5
C6—C7—H7	119.5	C32—C31—C30	119.9 (4)
C8—C7—H7	119.5	C32—C31—H31A	107.3
C9—C8—C7	121.7 (3)	C30—C31—H31A	107.3
C9—C8—H8	119.1	C32—C31—H31B	107.3
C7—C8—H8	119.1	C30—C31—H31B	107.3
C8—C9—O38	127.3 (3)	H31A—C31—H31B	106.9
C8—C9—C10	117.5 (3)	C31—C32—C33	119.2 (4)

O38—C9—C10	115.2 (3)	C31—C32—H32A	107.5
N11—C10—C9	128.4 (3)	C33—C32—H32A	107.5
N11—C10—C5	109.6 (2)	C31—C32—H32B	107.5
C9—C10—C5	121.8 (3)	C33—C32—H32B	107.5
C10—N11—C12	108.3 (2)	H32A—C32—H32B	107.0
C10—N11—H11	125.8	C34—C33—C32	121.7 (5)
C12—N11—H11	125.8	C34—C33—H33A	106.9
C13—C12—N11	128.9 (3)	C32—C33—H33A	106.9
C13—C12—C4	122.3 (2)	C34—C33—H33B	106.9
N11—C12—C4	108.7 (2)	C32—C33—H33B	106.9
C12—C13—C14	116.9 (2)	H33A—C33—H33B	106.7
C12—C13—H13	121.5	C35A—C34—C33	117.9 (11)
C14—C13—H13	121.5	C35—C34—C33	118.2 (7)
C13—C14—C2	121.2 (2)	C35—C34—H34A	107.7
C13—C14—C15	132.0 (2)	C33—C34—H34A	107.7
C2—C14—C15	106.9 (2)	C35—C34—H34B	107.7
C16—C15—C27	121.2 (2)	C33—C34—H34B	107.8
C16—C15—C14	132.1 (2)	H34A—C34—H34B	107.1
C27—C15—C14	106.6 (2)	C35A—C34—H34C	107.8
C17—C16—C15	116.6 (3)	C33—C34—H34C	107.8
C17—C16—H16	121.7	C35A—C34—H34D	107.8
C15—C16—H16	121.7	C33—C34—H34D	107.8
C16—C17—N18	127.7 (3)	H34C—C34—H34D	107.2
C16—C17—C25	123.5 (3)	C34—C35—H35A	109.5
N18—C17—C25	108.8 (2)	C34—C35—H35B	109.5
C19—N18—C17	107.9 (3)	H35A—C35—H35B	109.5
C19—N18—H18	134.9	C34—C35—H35C	109.5
C17—N18—H18	114.9	H35A—C35—H35C	109.5
N18—C19—C24	110.6 (3)	H35B—C35—H35C	109.5
N18—C19—C20	126.8 (3)	C34—C35A—H35D	109.5
C24—C19—C20	122.6 (3)	C34—C35A—H35E	109.5
O40—C20—C21	128.5 (3)	H35D—C35A—H35E	109.5
O40—C20—C19	114.2 (3)	C34—C35A—H35F	109.5
C21—C20—C19	117.3 (4)	H35D—C35A—H35F	109.5
C20—C21—C22	121.0 (4)	H35E—C35A—H35F	109.5
C20—C21—H21	119.5	C6—O36—C37	116.7 (2)
C22—C21—H21	119.5	O36—C37—H37A	109.5
C21—C22—C23	123.0 (4)	O36—C37—H37B	109.5
C21—C22—H22	118.5	H37A—C37—H37B	109.5
C23—C22—H22	118.5	O36—C37—H37C	109.5
O42—C23—C22	126.7 (4)	H37A—C37—H37C	109.5
O42—C23—C24	116.1 (3)	H37B—C37—H37C	109.5
C22—C23—C24	117.2 (4)	C9—O38—C39	117.2 (3)
C19—C24—C23	118.9 (3)	O38—C39—H39A	109.5
C19—C24—C25	106.2 (3)	O38—C39—H39B	109.5
C23—C24—C25	134.9 (4)	H39A—C39—H39B	109.5
C26—C25—C17	119.7 (2)	O38—C39—H39C	109.5
C26—C25—C24	133.8 (3)	H39A—C39—H39C	109.5

C17—C25—C24	106.4 (3)	H39B—C39—H39C	109.5
C27—C26—C25	117.2 (3)	C20—O40—C41	115.4 (3)
C27—C26—H26	121.4	O40—C41—H41A	109.5
C25—C26—H26	121.4	O40—C41—H41B	109.5
C26—C27—N1	129.5 (3)	H41A—C41—H41B	109.5
C26—C27—C15	121.7 (3)	O40—C41—H41C	109.5
N1—C27—C15	108.7 (2)	H41A—C41—H41C	109.5
C29A—C28—N1	125.8 (8)	H41B—C41—H41C	109.5
N1—C28—C29	110.4 (3)	C23—O42—C43	112.0 (6)
N1—C28—H28A	109.6	C23—O42—C43A	125.5 (8)
C29—C28—H28A	109.6	O42—C43—H43A	109.5
N1—C28—H28B	109.6	O42—C43—H43B	109.5
C29—C28—H28B	109.6	H43A—C43—H43B	109.5
H28A—C28—H28B	108.1	O42—C43—H43C	109.5
C29A—C28—H28C	105.9	H43A—C43—H43C	109.5
N1—C28—H28C	105.9	H43B—C43—H43C	109.5
C29A—C28—H28D	105.9	O42—C43A—H43D	109.5
N1—C28—H28D	105.9	O42—C43A—H43E	109.5
H28C—C28—H28D	106.2	H43D—C43A—H43E	109.5
C30—C29—C28	116.1 (3)	O42—C43A—H43F	109.5
C30—C29—H29A	108.3	H43D—C43A—H43F	109.5
C28—C29—H29A	108.3	H43E—C43A—H43F	109.5
C30—C29—H29B	108.3	H1W—O1W—H2W	109.4
C28—C29—H29B	108.3		

C27—N1—C2—C3	174.3 (2)	N18—C19—C20—C21	−179.1 (3)
C28—N1—C2—C3	−19.3 (4)	C24—C19—C20—C21	0.9 (4)
C27—N1—C2—C14	−1.0 (3)	O40—C20—C21—C22	179.9 (3)
C28—N1—C2—C14	165.3 (3)	C19—C20—C21—C22	−1.1 (5)
N1—C2—C3—C4	−177.3 (2)	C20—C21—C22—C23	1.1 (6)
C14—C2—C3—C4	−2.4 (4)	C21—C22—C23—O42	−179.7 (3)
C2—C3—C4—C12	−1.9 (3)	C21—C22—C23—C24	−0.8 (5)
C2—C3—C4—C5	180.0 (3)	N18—C19—C24—C23	179.4 (3)
C3—C4—C5—C10	175.7 (3)	C20—C19—C24—C23	−0.7 (4)
C12—C4—C5—C10	−2.7 (3)	N18—C19—C24—C25	−0.2 (3)
C3—C4—C5—C6	−1.1 (5)	C20—C19—C24—C25	179.8 (2)
C12—C4—C5—C6	−179.5 (3)	O42—C23—C24—C19	179.6 (3)
C10—C5—C6—O36	−178.9 (2)	C22—C23—C24—C19	0.6 (4)
C4—C5—C6—O36	−2.4 (5)	O42—C23—C24—C25	−1.1 (5)
C10—C5—C6—C7	1.0 (4)	C22—C23—C24—C25	179.9 (3)
C4—C5—C6—C7	177.4 (3)	C16—C17—C25—C26	−0.1 (4)
O36—C6—C7—C8	176.4 (3)	N18—C17—C25—C26	−179.4 (2)
C5—C6—C7—C8	−3.4 (5)	C16—C17—C25—C24	179.7 (2)
C6—C7—C8—C9	2.4 (6)	N18—C17—C25—C24	0.4 (3)
C7—C8—C9—O38	−178.6 (3)	C19—C24—C25—C26	179.6 (3)
C7—C8—C9—C10	1.1 (6)	C23—C24—C25—C26	0.2 (6)
C8—C9—C10—N11	−179.7 (3)	C19—C24—C25—C17	−0.1 (3)
O38—C9—C10—N11	0.0 (5)	C23—C24—C25—C17	−179.5 (3)

C8—C9—C10—C5	−3.5 (5)	C17—C25—C26—C27	1.0 (4)
O38—C9—C10—C5	176.2 (3)	C24—C25—C26—C27	−178.7 (3)
C6—C5—C10—N11	179.3 (2)	C25—C26—C27—N1	175.9 (2)
C4—C5—C10—N11	2.0 (3)	C25—C26—C27—C15	−1.8 (4)
C6—C5—C10—C9	2.5 (4)	C2—N1—C27—C26	−177.5 (3)
C4—C5—C10—C9	−174.8 (3)	C28—N1—C27—C26	15.8 (4)
C9—C10—N11—C12	176.1 (3)	C2—N1—C27—C15	0.4 (3)
C5—C10—N11—C12	−0.5 (3)	C28—N1—C27—C15	−166.3 (2)
C10—N11—C12—C13	178.1 (3)	C16—C15—C27—C26	1.6 (4)
C10—N11—C12—C4	−1.3 (3)	C14—C15—C27—C26	178.5 (2)
C3—C4—C12—C13	4.4 (4)	C16—C15—C27—N1	−176.4 (2)
C5—C4—C12—C13	−177.0 (2)	C14—C15—C27—N1	0.4 (3)
C3—C4—C12—N11	−176.2 (2)	C2—N1—C28—C29A	−45.1 (12)
C5—C4—C12—N11	2.4 (3)	C27—N1—C28—C29A	119.3 (12)
N11—C12—C13—C14	178.4 (2)	C2—N1—C28—C29	−88.8 (3)
C4—C12—C13—C14	−2.3 (4)	C27—N1—C28—C29	75.5 (3)
C12—C13—C14—C2	−2.0 (3)	N1—C28—C29—C30	71.9 (4)
C12—C13—C14—C15	176.7 (2)	N1—C28—C29A—C30	−30 (2)
C3—C2—C14—C13	4.5 (4)	C28—C29—C30—C31	−176.3 (3)
N1—C2—C14—C13	−179.7 (2)	C28—C29A—C30—C31	−121.9 (13)
C3—C2—C14—C15	−174.5 (2)	C29—C30—C31—C32	−179.9 (4)
N1—C2—C14—C15	1.3 (3)	C29A—C30—C31—C32	−139.7 (14)
C13—C14—C15—C16	−3.5 (5)	C30—C31—C32—C33	176.6 (4)
C2—C14—C15—C16	175.4 (3)	C31—C32—C33—C34	−177.4 (5)
C13—C14—C15—C27	−179.8 (2)	C32—C33—C34—C35A	−60.1 (12)
C2—C14—C15—C27	−1.0 (3)	C32—C33—C34—C35	172.8 (6)
C27—C15—C16—C17	−0.7 (4)	C7—C6—O36—C37	2.9 (5)
C14—C15—C16—C17	−176.6 (2)	C5—C6—O36—C37	−177.3 (2)
C15—C16—C17—N18	179.1 (2)	C8—C9—O38—C39	14.1 (5)
C15—C16—C17—C25	−0.1 (4)	C10—C9—O38—C39	−165.6 (3)
C16—C17—N18—C19	−179.7 (3)	C21—C20—O40—C41	−10.8 (5)
C25—C17—N18—C19	−0.5 (3)	C19—C20—O40—C41	170.2 (3)
C17—N18—C19—C24	0.4 (3)	C22—C23—O42—C43	1.7 (6)
C17—N18—C19—C20	−179.6 (3)	C24—C23—O42—C43	−177.2 (5)
N18—C19—C20—O40	0.0 (4)	C22—C23—O42—C43A	−28.2 (8)
C24—C19—C20—O40	−180.0 (3)	C24—C23—O42—C43A	152.9 (7)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···O1*W*	0.88	2.03	2.879 (5)	162
N11—H11···O1*W*^{i}	0.88	2.45	3.238 (6)	149
N18—H18···O36^{i}	1.09	2.03	3.068 (3)	157
O1*W*—H1*W*···N11	0.84	2.43	2.879 (5)	114
O1*W*—H1*W*···O38	0.84	2.41	2.900 (6)	118
O1*W*—H2*W*···O38^{i}	0.75	2.48	3.181 (6)	158

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