IUCrData

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5,31-Diaza-2,8,15,18,21,28,34,41,44,47-decaoxaheptacyclo[46.4.0.0^{4,32}.0^{6,30}.0^{9,14}.0^{22,27}.0^{35,40}]dopentaconta-1(48),4,6(30),9(14),10,12,22(27),23,25,31,35(40),36,38,49,51-pentadecaene monohydrate

Jong Sik Kim,[a] Jai Young Lee,[b]* So Yeon Lee,[b] Tae Yeon Lee[b] and Wonbo Sim[b]

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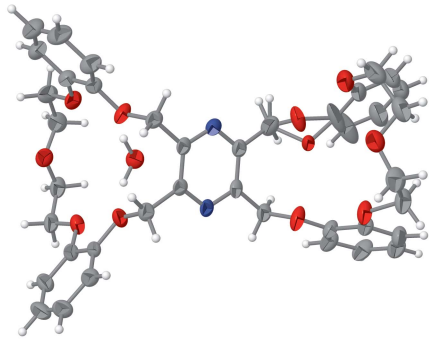
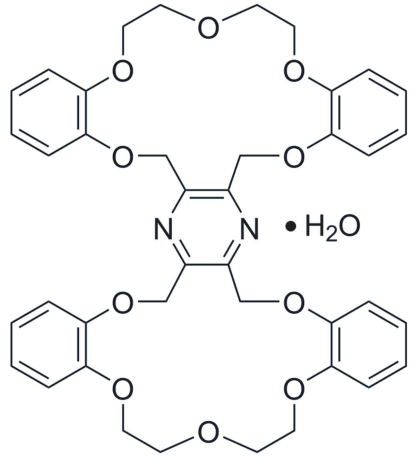
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{40}H_{40}N_2O_{10}\cdot H_2O$, each dual 17-crown-5 unit crystallizes with one solvent water molecule. The crown units are connected by a pyrazine ring. One oxo group in a crown moiety is disordered over two sites, with a refined occupancy ratio close to 1:1. The water molecule is linked to the crown unit by bifurcated O—H···O hydrogen bonds. In the crystal, molecules are linked by weak C—H···N and C—H···O interactions for the packing.

3D view



Chemical scheme



Structure description

In previous articles, we reported the synthesis and complexation behaviour of common-nuclear bis-crown ethers (Lee *et al.*, 1992, 1997). Within this context, we also reported the precursor of the common-nuclear bis-crown ether, bearing five aromatic subunits (Yun *et al.*, 2014).

The reaction of 1,2,4,5-tetrakis(bromomethyl)pyrazine (Assoumatine & Stoeckli-Evans, 2014) and bisphenol in the presence of sodium hydride afforded the title compound, that crystallizes with one water molecule (Fig. 1). For the four CH_2O connections between the central pyrazine ring (*A*) to the four benzene rings (*B*, *C*, *D* and *E*) of the crown units (Fig. 1), the torsion angles C12—C11—O4—C10, C13—C14—O5—C15, C23—C24—O7—C25 and C22—C21—O6—C40 are 158.5 (3), −171.5 (3), −160.6 (3) and 147.5 (8)°, respectively. Atom O6 is disordered over two positions. In the crystal, molecules are linked by weak C—H···N and C—H···O interactions for the packing (Table 1 and Fig. 2). The water molecule is placed close to the centre of a crown

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1WA···O3	0.98	2.20	3.069 (4)	147
O1W—H1WA···O4	0.98	2.20	3.013 (4)	140
O1W—H1WB···O1	0.97	2.23	3.087 (4)	146
O1W—H1WB···O5	0.97	2.27	3.090 (4)	142
C14—H14A···N1^{i}	0.99	2.61	3.540 (5)	157
C4—H4A···O9^{ii}	0.99	2.60	3.361 (5)	134

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

moiety, and forms bifurcated O—H···O hydrogen bonds with the host (Table 1 and Fig. 1).

Synthesis and crystallization

To a refluxing suspension of sodium hydride (4.40 mmol) in THF under N_2 was added dropwise a solution of 1,2,4,5-tetrakis(bromomethyl)pyrazine (2.20 mmol) and 1,8-bis(2-hydroxyphenoxy)-3,6-dioxaoctane (2.20 mmol) in THF, over a period of 1 h. The mixture was then refluxed for an additional 2 d. After cooling to room temperature, 10% aqueous hydrochloric acid was added. The solvent was removed under reduced pressure and the residual mixture was extracted with CH_2Cl_2. The organic layer was washed with water, dried over anhydrous $MgSO_4$, and evaporated *in vacuo*. The crude product was chromatographed on a silica-gel column using a mixture of ethyl acetate and *n*-hexane (1:2 *v*/*v*) as eluent, and recrystallized from CH_2Cl_2/*n*-hexane (1:20 *v*/*v*), to give a crystalline solid in 14% yield (m.p. 411 K). IR (KBr pellet, cm^{-1}): 2927, 1594, 1503, 1255, 1128, 1504, 774. ^{1}H-NMR ($CDCl_3$, 400 MHz): δ 7.06-6.82 (*m*, 16 H, Ar-***H***), 5.52 (*s*, 8H, pyz-C***H***$_2$O), 4.20–4.18 (*t*, 8H, ArOC***H***$_2$CH$_2$O), 3.96–3.93 (*t*, 8H, ArOCH$_2$C***H***$_2$O).

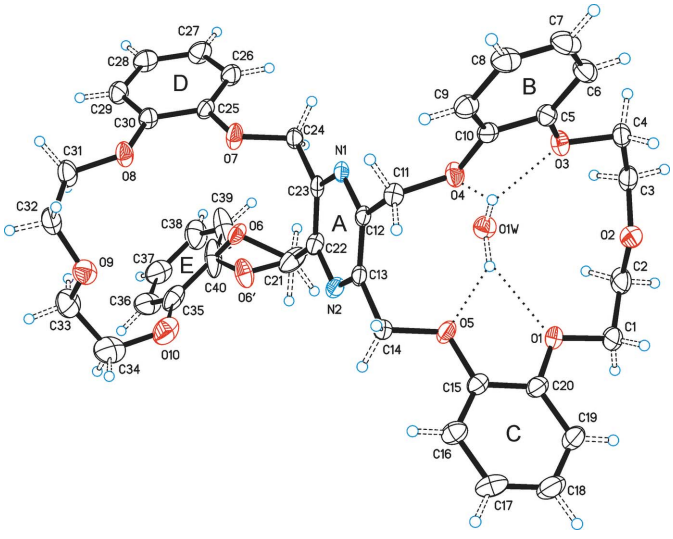


Figure 1
The molecular entities of the title compound, showing the atom-numbering scheme and O—H···O interactions (dotted lines). Displacement ellipsoids are drawn at the 30% probability level. H atoms are presented as small spheres of arbitrary radius.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{40}H_{42}N_2O_{11}$
M_r	726.75
Crystal system, space group	Orthorhombic, $Pna2_1$
Temperature (K)	173
a, b, c (Å)	14.3387 (16), 25.331 (3), 9.8536 (12)
V (Å^3)	3578.9 (7)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.40 × 0.30 × 0.20
Data collection	
Diffractometer	Bruker APEXII CCD
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	22423, 7980, 4373
R_{int}	0.061
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.667
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.047, 0.106, 1.00
No. of reflections	7980
No. of parameters	490
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.19

Computer programs: *SMART* and *SAINT-Plus* (Bruker, 2000), *SHELXTL* (Bruker, 2000) and *SHELXL2016* (Sheldrick, 2015).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. One oxo site is disordered over two positions, O6 and O6′, for which occupancies converged to 0.51 (2) and 0.49 (2), respectively.

Acknowledgements

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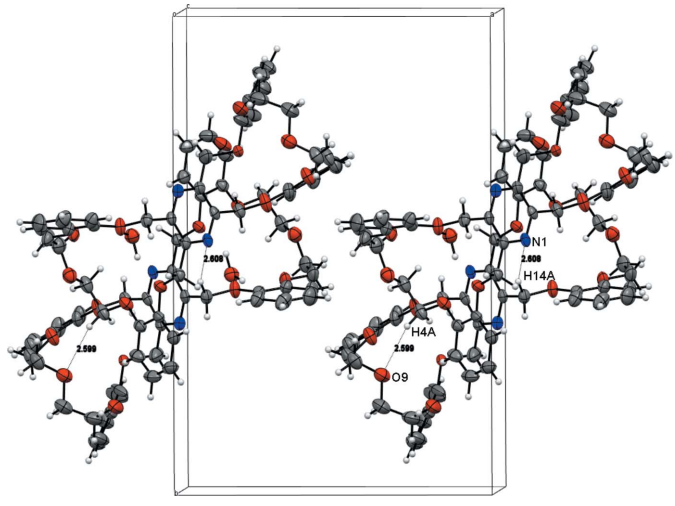

Figure 2
Crystal packing of the title compound, with intermolecular C—H···O and C—H···N hydrogen bonds shown as dashed lines. See Table 1 for hydrogen-bond details.

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full crystallographic data

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Crystal data

$C_{40}H_{42}N_2O_{11}$
M_r = 726.75
Orthorhombic, $Pna2_1$
a = 14.3387 (16) Å
b = 25.331 (3) Å
c = 9.8536 (12) Å
V = 3578.9 (7) Å^3
Z = 4
F(000) = 1536
D_x = 1.349 Mg m^{-3}
Melting point: 411 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 22423 reflections
θ = 1.6–28.3°
μ = 0.10 mm^{-1}
T = 173 K
Plate, colourless
0.40 × 0.30 × 0.20 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
22423 measured reflections
7980 independent reflections
4373 reflections with $I > 2\sigma(I)$
R_{int} = 0.061
θ_{max} = 28.3°, θ_{min} = 1.6°
h = −15→19
k = −33→33
l = −13→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.106
S = 1.00
7980 reflections
490 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0389P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.67821 (17)	0.45499 (11)	0.1761 (3)	0.0463 (8)	
O2	0.65641 (18)	0.53222 (11)	0.3859 (3)	0.0482 (8)	
O3	0.83422 (18)	0.58208 (11)	0.3799 (3)	0.0433 (7)	
O4	0.94981 (17)	0.54900 (10)	0.1994 (3)	0.0411 (7)	
O5	0.83100 (18)	0.44063 (12)	0.0478 (3)	0.0424 (8)	
O6	1.1920 (6)	0.3103 (3)	0.2965 (13)	0.043 (3)	0.51 (2)
O6′	1.1472 (11)	0.2964 (4)	0.2303 (12)	0.055 (5)	0.49 (2)
O7	1.27572 (18)	0.40252 (12)	0.3371 (3)	0.0478 (8)	
O8	1.41372 (17)	0.35393 (11)	0.2184 (3)	0.0458 (8)	
O9	1.3660 (2)	0.28879 (12)	−0.0169 (3)	0.0539 (8)	
O10	1.2092 (2)	0.23170 (13)	0.0849 (5)	0.0726 (12)	
N1	1.0973 (2)	0.46794 (12)	0.2427 (3)	0.0326 (8)	
N2	1.0092 (2)	0.37868 (12)	0.1419 (4)	0.0384 (9)	
C1	0.5946 (3)	0.46321 (19)	0.2534 (5)	0.0562 (14)	
H1A	0.557511	0.430266	0.257265	0.067*	
H1B	0.555963	0.491095	0.210969	0.067*	
C2	0.6233 (3)	0.47954 (19)	0.3932 (5)	0.0544 (13)	
H2A	0.569517	0.477379	0.455874	0.065*	
H2B	0.673105	0.455933	0.427271	0.065*	
C3	0.7038 (3)	0.55023 (18)	0.5037 (5)	0.0478 (11)	
H3A	0.746715	0.522578	0.537445	0.057*	
H3B	0.658379	0.558539	0.576322	0.057*	
C4	0.7573 (3)	0.59856 (17)	0.4659 (5)	0.0457 (12)	
H4A	0.716631	0.623637	0.416587	0.055*	
H4B	0.781337	0.616269	0.548381	0.055*	
C5	0.8806 (3)	0.62096 (16)	0.3113 (4)	0.0371 (10)	
C6	0.8710 (3)	0.67444 (17)	0.3334 (5)	0.0481 (12)	
H6	0.829458	0.686966	0.401278	0.058*	
C7	0.9220 (3)	0.70976 (17)	0.2563 (5)	0.0556 (13)	
H7	0.914702	0.746560	0.271549	0.067*	
C8	0.9828 (3)	0.69276 (17)	0.1584 (5)	0.0509 (12)	
H8	1.017221	0.717675	0.106581	0.061*	
C9	0.9942 (3)	0.63894 (16)	0.1350 (5)	0.0433 (11)	
H9	1.036197	0.626874	0.067211	0.052*	
C10	0.9437 (3)	0.60330 (15)	0.2115 (5)	0.0360 (10)	
C11	1.0130 (3)	0.52743 (14)	0.1006 (4)	0.0382 (10)	
H11A	1.071247	0.548407	0.097329	0.046*	
H11B	0.984180	0.527675	0.009302	0.046*	
C12	1.0334 (2)	0.47156 (14)	0.1448 (4)	0.0309 (9)	
C13	0.9898 (2)	0.42695 (15)	0.0926 (4)	0.0323 (10)	
C14	0.9176 (3)	0.42821 (16)	−0.0179 (4)	0.0370 (10)	
H14A	0.933293	0.455428	−0.086288	0.044*	
H14B	0.913658	0.393503	−0.063760	0.044*	
C15	0.7517 (3)	0.43693 (15)	−0.0308 (4)	0.0368 (10)	
C16	0.7501 (3)	0.42631 (16)	−0.1674 (5)	0.0463 (11)	

H16	0.806848	0.421272	−0.215367	0.056*	
C17	0.6655 (3)	0.42295 (19)	−0.2354 (5)	0.0587 (13)	
H17	0.664308	0.415440	−0.329848	0.070*	
C18	0.5844 (4)	0.43042 (19)	−0.1665 (6)	0.0641 (15)	
H18	0.526776	0.428547	−0.213797	0.077*	
C19	0.5847 (3)	0.44073 (18)	−0.0285 (6)	0.0572 (13)	
H19	0.527499	0.445193	0.018900	0.069*	
C20	0.6685 (3)	0.44449 (17)	0.0399 (5)	0.0419 (11)	
C21	1.0900 (3)	0.32022 (18)	0.2941 (7)	0.0648 (15)	
H21A	1.064089	0.316806	0.386817	0.078*	0.51 (2)
H21B	1.058819	0.293957	0.235105	0.078*	0.51 (2)
H21C	1.110879	0.322678	0.389643	0.078*	0.49 (2)
H21D	1.030653	0.300237	0.292784	0.078*	0.49 (2)
C22	1.0725 (3)	0.37501 (16)	0.2408 (5)	0.0387 (11)	
C23	1.1178 (2)	0.41978 (16)	0.2908 (4)	0.0327 (10)	
C24	1.1909 (2)	0.41773 (17)	0.3988 (4)	0.0365 (10)	
H24A	1.197724	0.452804	0.441999	0.044*	
H24B	1.173119	0.391873	0.469586	0.044*	
C25	1.3440 (3)	0.38267 (17)	0.4209 (4)	0.0386 (10)	
C26	1.3420 (3)	0.3873 (2)	0.5604 (5)	0.0492 (12)	
H26	1.292766	0.405898	0.603560	0.059*	
C27	1.4128 (3)	0.3645 (2)	0.6375 (5)	0.0569 (13)	
H27	1.410396	0.366393	0.733723	0.068*	
C28	1.4854 (3)	0.33944 (19)	0.5760 (5)	0.0543 (13)	
H28	1.533773	0.324387	0.629398	0.065*	
C29	1.4891 (3)	0.33585 (18)	0.4360 (5)	0.0480 (12)	
H29	1.540614	0.319093	0.393279	0.058*	
C30	1.4175 (3)	0.35674 (16)	0.3578 (4)	0.0383 (11)	
C31	1.4781 (3)	0.31804 (19)	0.1556 (5)	0.0557 (13)	
H31A	1.474371	0.282965	0.199606	0.067*	
H31B	1.542694	0.331404	0.165083	0.067*	
C32	1.4526 (3)	0.31375 (19)	0.0086 (5)	0.0556 (13)	
H32A	1.450759	0.349689	−0.030785	0.067*	
H32B	1.502170	0.293837	−0.038945	0.067*	
C33	1.3690 (3)	0.23297 (18)	−0.0042 (6)	0.0691 (15)	
H33A	1.395208	0.223015	0.085131	0.083*	
H33B	1.409112	0.217735	−0.076017	0.083*	
C34	1.2714 (4)	0.2123 (2)	−0.0176 (7)	0.0822 (19)	
H34A	1.246728	0.222163	−0.107882	0.099*	
H34B	1.273002	0.173227	−0.012777	0.099*	
C35	1.2295 (3)	0.21946 (18)	0.2174 (7)	0.0614 (16)	
C36	1.2665 (3)	0.17221 (19)	0.2627 (7)	0.0750 (18)	
H36	1.280979	0.145382	0.198746	0.090*	
C37	1.2827 (4)	0.1632 (2)	0.3979 (8)	0.0769 (19)	
H37	1.309802	0.130748	0.425715	0.092*	
C38	1.2603 (4)	0.2002 (2)	0.4927 (7)	0.0774 (17)	
H38	1.271628	0.194084	0.586394	0.093*	
C39	1.2207 (5)	0.2470 (2)	0.4491 (8)	0.103 (3)	

H39	1.202760	0.272723	0.514290	0.124*
C40	1.2068 (4)	0.25718 (19)	0.3137 (8)	0.082 (2)
O1W	0.85464 (19)	0.46186 (12)	0.3548 (3)	0.0468 (8)
H1WA	0.871426	0.498942	0.341851	0.11 (2)*
H1WB	0.817926	0.452542	0.275051	0.12 (2)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0281 (15)	0.0553 (19)	0.055 (2)	0.0030 (13)	0.0052 (14)	−0.0124 (16)
O2	0.0441 (16)	0.053 (2)	0.048 (2)	0.0041 (15)	−0.0055 (15)	−0.0055 (16)
O3	0.0417 (16)	0.0402 (17)	0.0481 (19)	0.0134 (14)	0.0037 (15)	−0.0008 (15)
O4	0.0374 (15)	0.0317 (16)	0.0543 (19)	0.0071 (13)	0.0088 (14)	0.0010 (14)
O5	0.0238 (15)	0.066 (2)	0.0376 (18)	0.0038 (14)	−0.0028 (13)	−0.0012 (14)
O6	0.023 (4)	0.028 (4)	0.079 (7)	0.003 (3)	−0.005 (4)	0.001 (4)
O6′	0.063 (9)	0.044 (5)	0.059 (7)	0.028 (5)	0.012 (6)	0.007 (5)
O7	0.0310 (16)	0.079 (2)	0.0337 (18)	0.0192 (15)	0.0001 (14)	0.0030 (16)
O8	0.0383 (17)	0.064 (2)	0.0354 (19)	0.0209 (14)	0.0000 (14)	−0.0004 (16)
O9	0.057 (2)	0.0455 (19)	0.059 (2)	0.0102 (15)	−0.0036 (18)	−0.0010 (17)
O10	0.052 (2)	0.044 (2)	0.121 (4)	0.0099 (17)	−0.014 (2)	0.005 (2)
N1	0.0230 (16)	0.040 (2)	0.035 (2)	0.0068 (14)	0.0035 (15)	−0.0046 (17)
N2	0.0263 (19)	0.040 (2)	0.049 (2)	−0.0003 (15)	0.0000 (17)	−0.0010 (19)
C1	0.033 (2)	0.061 (3)	0.074 (4)	−0.007 (2)	0.015 (3)	−0.019 (3)
C2	0.042 (3)	0.059 (3)	0.062 (4)	−0.001 (2)	0.020 (3)	−0.003 (3)
C3	0.039 (2)	0.062 (3)	0.042 (3)	0.014 (2)	0.003 (2)	−0.003 (3)
C4	0.038 (2)	0.055 (3)	0.044 (3)	0.016 (2)	−0.003 (2)	−0.011 (2)
C5	0.036 (2)	0.036 (3)	0.039 (3)	0.0062 (19)	−0.006 (2)	0.000 (2)
C6	0.055 (3)	0.037 (3)	0.052 (3)	0.009 (2)	−0.008 (2)	−0.009 (2)
C7	0.073 (3)	0.031 (3)	0.063 (4)	0.002 (2)	−0.017 (3)	−0.004 (3)
C8	0.059 (3)	0.035 (3)	0.059 (3)	−0.005 (2)	−0.012 (3)	0.007 (2)
C9	0.040 (3)	0.043 (3)	0.047 (3)	0.000 (2)	−0.007 (2)	0.003 (2)
C10	0.033 (2)	0.028 (2)	0.046 (3)	0.0051 (18)	−0.012 (2)	0.000 (2)
C11	0.033 (2)	0.040 (2)	0.042 (3)	0.0034 (19)	0.002 (2)	0.001 (2)
C12	0.022 (2)	0.037 (2)	0.034 (2)	0.0016 (17)	0.0061 (18)	−0.001 (2)
C13	0.022 (2)	0.040 (2)	0.035 (2)	0.0045 (18)	0.0060 (18)	−0.002 (2)
C14	0.030 (2)	0.046 (3)	0.036 (2)	0.0004 (18)	0.0013 (19)	−0.007 (2)
C15	0.033 (2)	0.034 (2)	0.044 (3)	−0.0024 (19)	−0.004 (2)	0.005 (2)
C16	0.043 (3)	0.056 (3)	0.040 (3)	−0.009 (2)	−0.005 (2)	0.008 (2)
C17	0.059 (3)	0.066 (3)	0.050 (3)	−0.016 (3)	−0.021 (3)	0.008 (3)
C18	0.048 (3)	0.070 (4)	0.074 (4)	−0.003 (3)	−0.025 (3)	−0.005 (3)
C19	0.033 (3)	0.058 (3)	0.080 (4)	0.000 (2)	−0.009 (3)	−0.011 (3)
C20	0.034 (3)	0.040 (3)	0.052 (3)	−0.003 (2)	−0.006 (2)	0.000 (2)
C21	0.043 (3)	0.051 (3)	0.101 (5)	−0.005 (2)	−0.020 (3)	0.021 (3)
C22	0.027 (2)	0.037 (3)	0.052 (3)	0.0030 (18)	0.003 (2)	0.005 (2)
C23	0.024 (2)	0.041 (3)	0.033 (2)	0.0106 (18)	0.0074 (18)	0.000 (2)
C24	0.031 (2)	0.044 (3)	0.035 (2)	0.0088 (18)	0.001 (2)	−0.003 (2)
C25	0.025 (2)	0.052 (3)	0.039 (3)	0.004 (2)	−0.004 (2)	−0.003 (2)
C26	0.036 (3)	0.072 (3)	0.039 (3)	0.002 (2)	−0.004 (2)	−0.007 (2)

C27	0.047 (3)	0.088 (4)	0.036 (3)	−0.002 (3)	−0.011 (2)	−0.001 (3)
C28	0.051 (3)	0.064 (3)	0.048 (3)	0.004 (3)	−0.018 (2)	0.006 (3)
C29	0.038 (3)	0.053 (3)	0.052 (3)	0.013 (2)	−0.005 (2)	−0.002 (3)
C30	0.035 (2)	0.045 (3)	0.034 (3)	0.005 (2)	−0.003 (2)	0.003 (2)
C31	0.047 (3)	0.071 (3)	0.049 (3)	0.024 (3)	−0.001 (2)	−0.007 (3)
C32	0.053 (3)	0.065 (3)	0.049 (3)	0.015 (3)	0.010 (3)	−0.006 (3)
C33	0.069 (4)	0.048 (3)	0.091 (4)	0.020 (3)	0.002 (3)	−0.002 (3)
C34	0.092 (5)	0.040 (3)	0.114 (6)	0.013 (3)	−0.024 (4)	−0.007 (3)
C35	0.034 (3)	0.034 (3)	0.117 (5)	0.003 (2)	−0.006 (3)	0.008 (3)
C36	0.070 (4)	0.042 (3)	0.113 (5)	0.018 (3)	−0.031 (4)	−0.021 (3)
C37	0.062 (4)	0.044 (3)	0.124 (6)	0.015 (3)	−0.030 (4)	−0.006 (4)
C38	0.071 (4)	0.053 (4)	0.108 (5)	0.016 (3)	0.015 (4)	0.006 (4)
C39	0.120 (5)	0.053 (4)	0.136 (7)	0.027 (3)	0.083 (5)	0.019 (4)
C40	0.073 (4)	0.035 (3)	0.139 (6)	0.025 (3)	0.062 (4)	0.029 (4)
O1W	0.0474 (17)	0.049 (2)	0.044 (2)	0.0017 (14)	−0.0030 (16)	0.0028 (16)

Geometric parameters (Å, º)

O1—C20	1.376 (5)	C14—H14B	0.9900
O1—C1	1.436 (5)	C15—C16	1.373 (6)
O2—C2	1.418 (5)	C15—C20	1.394 (6)
O2—C3	1.421 (5)	C16—C17	1.388 (6)
O3—C5	1.367 (5)	C16—H16	0.9500
O3—C4	1.452 (5)	C17—C18	1.359 (7)
O4—C10	1.383 (4)	C17—H17	0.9500
O4—C11	1.439 (4)	C18—C19	1.385 (7)
O5—C15	1.379 (4)	C18—H18	0.9500
O5—C14	1.436 (4)	C19—C20	1.381 (6)
O6—C40	1.372 (7)	C19—H19	0.9500
O6—C21	1.484 (10)	C21—C22	1.505 (6)
O6′—C21	1.196 (8)	C21—H21A	0.9900
O6′—C40	1.547 (12)	C21—H21B	0.9900
O7—C25	1.376 (4)	C21—H21C	0.9900
O7—C24	1.413 (4)	C21—H21D	0.9900
O8—C30	1.376 (5)	C22—C23	1.397 (5)
O8—C31	1.436 (5)	C23—C24	1.494 (5)
O9—C32	1.415 (5)	C24—H24A	0.9900
O9—C33	1.420 (5)	C24—H24B	0.9900
O10—C35	1.373 (6)	C25—C26	1.380 (5)
O10—C34	1.435 (7)	C25—C30	1.388 (5)
N1—C12	1.334 (5)	C26—C27	1.393 (6)
N1—C23	1.341 (5)	C26—H26	0.9500
N2—C22	1.336 (5)	C27—C28	1.361 (6)
N2—C13	1.345 (5)	C27—H27	0.9500
C1—C2	1.496 (6)	C28—C29	1.384 (6)
C1—H1A	0.9900	C28—H28	0.9500
C1—H1B	0.9900	C29—C30	1.388 (5)
C2—H2A	0.9900	C29—H29	0.9500

C2—H2B	0.9900	C31—C32	1.499 (7)
C3—C4	1.492 (6)	C31—H31A	0.9900
C3—H3A	0.9900	C31—H31B	0.9900
C3—H3B	0.9900	C32—H32A	0.9900
C4—H4A	0.9900	C32—H32B	0.9900
C4—H4B	0.9900	C33—C34	1.500 (7)
C5—C6	1.379 (5)	C33—H33A	0.9900
C5—C10	1.409 (6)	C33—H33B	0.9900
C6—C7	1.383 (6)	C34—H34A	0.9900
C6—H6	0.9500	C34—H34B	0.9900
C7—C8	1.370 (6)	C35—C36	1.383 (7)
C7—H7	0.9500	C35—C40	1.386 (8)
C8—C9	1.392 (5)	C36—C37	1.371 (8)
C8—H8	0.9500	C36—H36	0.9500
C9—C10	1.381 (5)	C37—C38	1.363 (8)
C9—H9	0.9500	C37—H37	0.9500
C11—C12	1.509 (5)	C38—C39	1.382 (8)
C11—H11A	0.9900	C38—H38	0.9500
C11—H11B	0.9900	C39—C40	1.373 (8)
C12—C13	1.390 (5)	C39—H39	0.9500
C13—C14	1.503 (5)	O1W—H1WA	0.9779
C14—H14A	0.9900	O1W—H1WB	0.9744

C20—O1—C1	117.5 (3)	O1—C20—C15	115.3 (4)
C2—O2—C3	114.9 (3)	C19—C20—C15	119.4 (4)
C5—O3—C4	116.8 (3)	O6′—C21—C22	113.3 (6)
C10—O4—C11	118.4 (3)	O6—C21—C22	109.0 (4)
C15—O5—C14	116.4 (3)	O6—C21—H21A	109.9
C40—O6—C21	108.7 (6)	C22—C21—H21A	109.9
C21—O6′—C40	115.0 (8)	O6—C21—H21B	109.9
C25—O7—C24	117.0 (3)	C22—C21—H21B	109.9
C30—O8—C31	116.0 (3)	H21Aa—C21—H21B	108.3
C32—O9—C33	113.8 (4)	O6′—C21—H21C	108.9
C35—O10—C34	117.4 (4)	C22—C21—H21C	108.9
C12—N1—C23	117.9 (3)	O6′—C21—H21D	108.9
C22—N2—C13	117.9 (3)	C22—C21—H21D	108.9
O1—C1—C2	107.3 (3)	H21Cb—C21—H21D	107.7
O1—C1—H1A	110.2	N2—C22—C23	121.1 (4)
C2—C1—H1A	110.2	N2—C22—C21	115.6 (4)
O1—C1—H1B	110.2	C23—C22—C21	123.3 (4)
C2—C1—H1B	110.2	N1—C23—C22	120.8 (4)
H1A—C1—H1B	108.5	N1—C23—C24	115.9 (4)
O2—C2—C1	107.8 (4)	C22—C23—C24	123.3 (4)
O2—C2—H2A	110.1	O7—C24—C23	107.9 (3)
C1—C2—H2A	110.1	O7—C24—H24A	110.1
O2—C2—H2B	110.1	C23—C24—H24A	110.1
C1—C2—H2B	110.1	O7—C24—H24B	110.1
H2A—C2—H2B	108.5	C23—C24—H24B	110.1

O2—C3—C4	107.8 (4)	H24A—C24—H24B	108.4
O2—C3—H3A	110.1	O7—C25—C26	123.5 (4)
C4—C3—H3A	110.1	O7—C25—C30	116.4 (4)
O2—C3—H3B	110.1	C26—C25—C30	120.1 (4)
C4—C3—H3B	110.1	C25—C26—C27	119.6 (4)
H3A—C3—H3B	108.5	C25—C26—H26	120.2
O3—C4—C3	107.5 (3)	C27—C26—H26	120.2
O3—C4—H4A	110.2	C28—C27—C26	120.5 (4)
C3—C4—H4A	110.2	C28—C27—H27	119.8
O3—C4—H4B	110.2	C26—C27—H27	119.8
C3—C4—H4B	110.2	C27—C28—C29	120.2 (4)
H4A—C4—H4B	108.5	C27—C28—H28	119.9
O3—C5—C6	125.5 (4)	C29—C28—H28	119.9
O3—C5—C10	115.4 (3)	C28—C29—C30	120.0 (4)
C6—C5—C10	119.1 (4)	C28—C29—H29	120.0
C5—C6—C7	119.7 (4)	C30—C29—H29	120.0
C5—C6—H6	120.1	O8—C30—C25	116.2 (3)
C7—C6—H6	120.1	O8—C30—C29	124.2 (4)
C8—C7—C6	121.4 (4)	C25—C30—C29	119.6 (4)
C8—C7—H7	119.3	O8—C31—C32	107.8 (3)
C6—C7—H7	119.3	O8—C31—H31A	110.2
C7—C8—C9	119.9 (4)	C32—C31—H31A	110.2
C7—C8—H8	120.0	O8—C31—H31B	110.2
C9—C8—H8	120.0	C32—C31—H31B	110.2
C10—C9—C8	119.2 (4)	H31A—C31—H31B	108.5
C10—C9—H9	120.4	O9—C32—C31	114.7 (4)
C8—C9—H9	120.4	O9—C32—H32A	108.6
C9—C10—O4	124.7 (4)	C31—C32—H32A	108.6
C9—C10—C5	120.6 (4)	O9—C32—H32B	108.6
O4—C10—C5	114.6 (4)	C31—C32—H32B	108.6
O4—C11—C12	106.4 (3)	H32A—C32—H32B	107.6
O4—C11—H11A	110.4	O9—C33—C34	108.2 (4)
C12—C11—H11A	110.4	O9—C33—H33A	110.1
O4—C11—H11B	110.4	C34—C33—H33A	110.1
C12—C11—H11B	110.4	O9—C33—H33B	110.1
H11A—C11—H11B	108.6	C34—C33—H33B	110.1
N1—C12—C13	121.4 (3)	H33A—C33—H33B	108.4
N1—C12—C11	114.0 (3)	O10—C34—C33	113.5 (5)
C13—C12—C11	124.6 (4)	O10—C34—H34A	108.9
N2—C13—C12	120.8 (4)	C33—C34—H34A	108.9
N2—C13—C14	115.1 (3)	O10—C34—H34B	108.9
C12—C13—C14	124.1 (4)	C33—C34—H34B	108.9
O5—C14—C13	105.9 (3)	H34A—C34—H34B	107.7
O5—C14—H14A	110.6	O10—C35—C36	125.7 (5)
C13—C14—H14A	110.6	O10—C35—C40	116.5 (5)
O5—C14—H14B	110.6	C36—C35—C40	117.8 (6)
C13—C14—H14B	110.6	C37—C36—C35	121.5 (6)
H14A—C14—H14B	108.7	C37—C36—H36	119.2

C16—C15—O5	125.3 (4)	C35—C36—H36	119.2
C16—C15—C20	120.1 (4)	C38—C37—C36	120.8 (5)
O5—C15—C20	114.5 (4)	C38—C37—H37	119.6
C15—C16—C17	120.0 (4)	C36—C37—H37	119.6
C15—C16—H16	120.0	C37—C38—C39	118.3 (7)
C17—C16—H16	120.0	C37—C38—H38	120.9
C18—C17—C16	119.8 (5)	C39—C38—H38	120.9
C18—C17—H17	120.1	C40—C39—C38	121.5 (6)
C16—C17—H17	120.1	C40—C39—H39	119.3
C17—C18—C19	121.0 (5)	C38—C39—H39	119.3
C17—C18—H18	119.5	O6—C40—C39	109.1 (8)
C19—C18—H18	119.5	O6—C40—C35	128.9 (8)
C20—C19—C18	119.6 (5)	C39—C40—C35	120.1 (5)
C20—C19—H19	120.2	C39—C40—O6′	135.8 (6)
C18—C19—H19	120.2	C35—C40—O6′	102.1 (7)
O1—C20—C19	125.3 (4)	H1WA—O1W—H1WB	105.1

C20—O1—C1—C2	−174.0 (4)	O6′—C21—C22—N2	86.3 (12)
C3—O2—C2—C1	−167.9 (3)	O6—C21—C22—N2	130.4 (7)
O1—C1—C2—O2	73.3 (4)	O6′—C21—C22—C23	−94.4 (12)
C2—O2—C3—C4	163.5 (3)	O6—C21—C22—C23	−50.3 (9)
C5—O3—C4—C3	166.9 (4)	C12—N1—C23—C22	−0.9 (5)
O2—C3—C4—O3	−70.8 (4)	C12—N1—C23—C24	179.0 (3)
C4—O3—C5—C6	11.3 (6)	N2—C22—C23—N1	1.5 (6)
C4—O3—C5—C10	−169.5 (3)	C21—C22—C23—N1	−177.8 (4)
O3—C5—C6—C7	−179.8 (4)	N2—C22—C23—C24	−178.4 (3)
C10—C5—C6—C7	1.0 (6)	C21—C22—C23—C24	2.3 (6)
C5—C6—C7—C8	−0.5 (7)	C25—O7—C24—C23	−160.6 (3)
C6—C7—C8—C9	0.0 (7)	N1—C23—C24—O7	−100.1 (4)
C7—C8—C9—C10	−0.1 (6)	C22—C23—C24—O7	79.8 (5)
C8—C9—C10—O4	−179.0 (4)	C24—O7—C25—C26	−14.3 (6)
C8—C9—C10—C5	0.6 (6)	C24—O7—C25—C30	165.5 (4)
C11—O4—C10—C9	0.0 (5)	O7—C25—C26—C27	178.0 (4)
C11—O4—C10—C5	−179.6 (3)	C30—C25—C26—C27	−1.8 (7)
O3—C5—C10—C9	179.6 (3)	C25—C26—C27—C28	2.5 (7)
C6—C5—C10—C9	−1.1 (6)	C26—C27—C28—C29	−0.8 (8)
O3—C5—C10—O4	−0.8 (5)	C27—C28—C29—C30	−1.5 (7)
C6—C5—C10—O4	178.5 (4)	C31—O8—C30—C25	−167.3 (4)
C10—O4—C11—C12	158.5 (3)	C31—O8—C30—C29	13.0 (6)
C23—N1—C12—C13	−0.7 (5)	O7—C25—C30—O8	0.1 (6)
C23—N1—C12—C11	178.2 (3)	C26—C25—C30—O8	179.9 (4)
O4—C11—C12—N1	−80.4 (4)	O7—C25—C30—C29	179.7 (4)
O4—C11—C12—C13	98.4 (4)	C26—C25—C30—C29	−0.4 (7)
C22—N2—C13—C12	−1.0 (5)	C28—C29—C30—O8	−178.3 (4)
C22—N2—C13—C14	−179.6 (3)	C28—C29—C30—C25	2.1 (7)
N1—C12—C13—N2	1.6 (5)	C30—O8—C31—C32	170.5 (4)
C11—C12—C13—N2	−177.1 (3)	C33—O9—C32—C31	−76.9 (5)
N1—C12—C13—C14	−179.8 (3)	O8—C31—C32—O9	−67.4 (5)

C11—C12—C13—C14	1.5 (6)	C32—O9—C33—C34	173.5 (4)
C15—O5—C14—C13	−171.5 (3)	C35—O10—C34—C33	−61.8 (6)
N2—C13—C14—O5	96.5 (4)	O9—C33—C34—O10	−60.9 (6)
C12—C13—C14—O5	−82.1 (4)	C34—O10—C35—C36	−36.9 (7)
C14—O5—C15—C16	−5.1 (6)	C34—O10—C35—C40	146.2 (4)
C14—O5—C15—C20	174.3 (4)	O10—C35—C36—C37	−178.6 (5)
O5—C15—C16—C17	179.2 (4)	C40—C35—C36—C37	−1.7 (8)
C20—C15—C16—C17	−0.2 (6)	C35—C36—C37—C38	1.8 (9)
C15—C16—C17—C18	0.3 (7)	C36—C37—C38—C39	0.2 (9)
C16—C17—C18—C19	−0.8 (7)	C37—C38—C39—C40	−2.2 (9)
C17—C18—C19—C20	1.3 (8)	C21—O6—C40—C39	−95.5 (10)
C1—O1—C20—C19	−0.6 (6)	C21—O6—C40—C35	100.7 (8)
C1—O1—C20—C15	179.9 (3)	C38—C39—C40—O6	−163.2 (6)
C18—C19—C20—O1	179.4 (4)	C38—C39—C40—C35	2.2 (10)
C18—C19—C20—C15	−1.1 (7)	C38—C39—C40—O6′	162.8 (11)
C16—C15—C20—O1	−179.9 (4)	O10—C35—C40—O6	−20.8 (10)
O5—C15—C20—O1	0.7 (5)	C36—C35—C40—O6	162.0 (8)
C16—C15—C20—C19	0.6 (6)	O10—C35—C40—C39	176.9 (5)
O5—C15—C20—C19	−178.9 (4)	C36—C35—C40—C39	−0.3 (8)
C40—O6′—C21—C22	147.5 (8)	O10—C35—C40—O6′	10.6 (8)
C40—O6—C21—C22	−165.9 (7)	C36—C35—C40—O6′	−166.6 (7)
C13—N2—C22—C23	−0.6 (5)	C21—O6′—C40—C39	−11 (2)
C13—N2—C22—C21	178.8 (4)	C21—O6′—C40—C35	151.8 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*WA*···O3	0.98	2.20	3.069 (4)	147
O1*W*—H1*WA*···O4	0.98	2.20	3.013 (4)	140
O1*W*—H1*WB*···O1	0.97	2.23	3.087 (4)	146
O1*W*—H1*WB*···O5	0.97	2.27	3.090 (4)	142
C14—H14*A*···N1^{i}	0.99	2.61	3.540 (5)	157
C4—H4*A*···O9^{ii}	0.99	2.60	3.361 (5)	134

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