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2,8,15,18,21,24,31,37,44,47,50,53-Dodecaoxaheptacyclo[52.4.0.0^{4,35}.0^{6,33}.0^{9,14}.0^{25,30}.0^{38,43}]octapentaconta-1(54),4,6(33),9(14),10,12,25(30),26,28,34,38(43),39,41,55,57-pentadecaene dichloromethane disolvate

Ji Ye Yun, Sung Wan Ahn, Dong Hwan Kim, Wonbo Sim* and Jai Young Lee*

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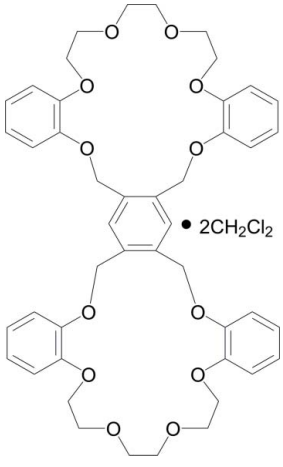
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.005 Å; R factor = 0.051; wR factor = 0.136; data-to-parameter ratio = 14.6.

In the title compound, $C_{46}H_{50}O_{12}\cdot 2CH_2Cl_2$, each dual 20-crown-6 unit crystallizes with two dichloromethane solvent molecules. The crown unit molecule lies about an inversion centre located at the central benzene ring. The two crown ring groups adopt an *anti* conformation, stabilized by weak intramolecular C—H···O interactions. In the crystal, the crown unit molecules and the solvent molecules are linked by C—H···O interactions into a three-dimensional network.

Related literature

For the preparation and crystal structures of related compounds, see: Lee *et al.* (2009); Beack *et al.* (2012). For background to dual crown ethers and their inclusion behavior, see: Lee *et al.* (1992, 1997).



Experimental

Crystal data

$C_{46}H_{50}O_{12}\cdot 2CH_2Cl_2$
M_r = 964.71
Orthorhombic, *Pccn*
a = 23.0916 (11) Å
b = 23.9520 (11) Å
c = 8.6426 (4) Å
V = 4780.1 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 200 K
0.25 × 0.24 × 0.18 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.927, T_{max} = 0.947
26165 measured reflections
4211 independent reflections
2588 reflections with $I > 2\sigma(I)$
R_{int} = 0.077

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.136
S = 1.02
4211 reflections
289 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O6^{i}	0.95	2.47	2.828 (3)	103
C3—H3B···O4^{ii}	0.99	2.59	3.457 (4)	146
C22—H22A···O1^{ii}	0.99	2.32	3.252 (3)	156
C22—H22B···O1	0.99	2.53	2.909 (3)	103
C24—H24A···O6	0.99	2.51	3.455 (4)	160
C24—H24B···O3	0.99	2.36	3.331 (5)	168

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT-Plus* (Bruker, 2008); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supporting information for this paper is available from the IUCr electronic archives (Reference: WM5018).

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supporting information

Acta Cryst. (2014). E**70**, o558 [doi:10.1107/S1600536814007971]

2,8,15,18,21,24,31,37,44,47,50,53-Dodecaoxaheptacyclo-[52.4.0.0^{4,35}.0^{6,33}.0^{9,14}.0^{25,30}.0^{38,43}]octapentaconta-1(54),4,6(33),9(14),10,12,25(30),26,28,34,38(43),39,41,55,57-pentadecaene dichloromethane disolvate

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S1. Chemical context

In a previous article we reported the synthesis and complexation behavior of common-nuclear bis-crown ethers (Lee *et al.*, 1992, 1997). Within this context we also reported the precursor of the common-nuclear biscrown ether, bearing three aromatic subunits (Lee *et al.*, 2009; Beack *et al.*, 2012). The reaction of 1,2,4,5-tetrakis(bromomethyl)benzene and bis-phenol in the presence of sodium hydride afforded the title compound, $C_{46}H_{50}O_{12}$, that crystallizes with two molecules of dichloromethane.

S2. Structural commentary

In the title molecule (Fig. 1), in the *A*-to-*B* ring and *A*-to-*C* ring connectivities, the torsion angles C2–C3–O1–C4 and C23–C22–O6–C21 are -72.7 (3) ° and 99.0 (3)°, respectively. This indicates that the *A* ring is situated *gauche* to the *B* ring, also situated *gauche* to the *C* ring, with dihedral angles of 81.64 (14)° between *A* and *B* and 81.16 (14)° between *A* and *C*. The dihedral angle between the *B* and *C* ring is 1.92 (15)°. All C–C–O–C torsion angles in the triethylene glycol group are related to *trans* conformations. Weak intramolecular C—H···O hydrogen bonds (C22—H22B···O1; C1—H1A···O6) stabilize the conformation of the crown unit molecule (Fig. 1, Table 1).

S3. Supramolecular features

In the crystal, the crown unit molecules and the solvent molecules are linked by C—H···O interactions (Figs. 1, 2; Table 1). The C atom of the dichloromethane molecule (C24) is shifted by 2.383 Å outwards to the crown ring cavity (least squares plane defined by atoms O1–O6), and forms two C—H···O hydrogen bonds with two O atoms (O3 and O6) in the crown ring (Fig. 1, Table 1).

S4. Synthesis and crystallization

To a refluxing suspension of sodium hydride (10.6 mmol) in THF under nitrogen was added dropwise a solution of 1,2,4,5-tetrakis(bromomethyl)benzene (2.20 mmol) and 1,8-bis(2-hydroxyphenoxy)-3,6-dioxaoctane (4.40 mmol) in THF over a period of 3 h. The mixture was then refluxed for additional 3 days. After cooling to room temperature, 10%$_{wt}$ aqueous hydrochloric acid was added. The solvent was removed under reduced pressure and the residual mixture was extracted with dichloromethane. The organic layer was washed with water, dried over anhydrous magnesium sulfate, and evaporated *in vacuo*. The crude product was chromatographed on a silica-gel column using a mixed solvent of ethyl acetate and *n*-hexane (1:1) as eluent. Recrystallization from dichloromethane/*n*-hexane (1:20, v/v) resulted in 53% yield (m.p. 423 K). IR (KBr pellet): 2927, 1599, 1505, 1459, 1255, 1122, 1003, and 738 cm^{-1}. ^{1}H NMR ($CDCl_3$): d 7.69 (s, 2 H, OCH_2***Ar***), 6.92~6.80 (m, 16 H, O***Ar***O), 5.29 (s, 8 H, OC***H***$_2$Ar), 4.12 (t, 8 H, ArOC***H***$_2$CH$_2$), 3.81 (t, 8 H, ArOCH$_2$C***H***$_2$) and

3.66 (t, 8 H, $ArOCH_2CH_2OCH_2$).

S5. Refinement details

All H-atoms were positioned geometrically and refined using a riding model with d(C—H)=0.95 Å, U_{iso}=1.2U_{eq}(C) for aromatic and 0.99 Å, U_{iso}=1.2U_{eq}(C) for CH_2 atoms. Reflection (110) was affected by the beamstop and was omitted from the refinement.

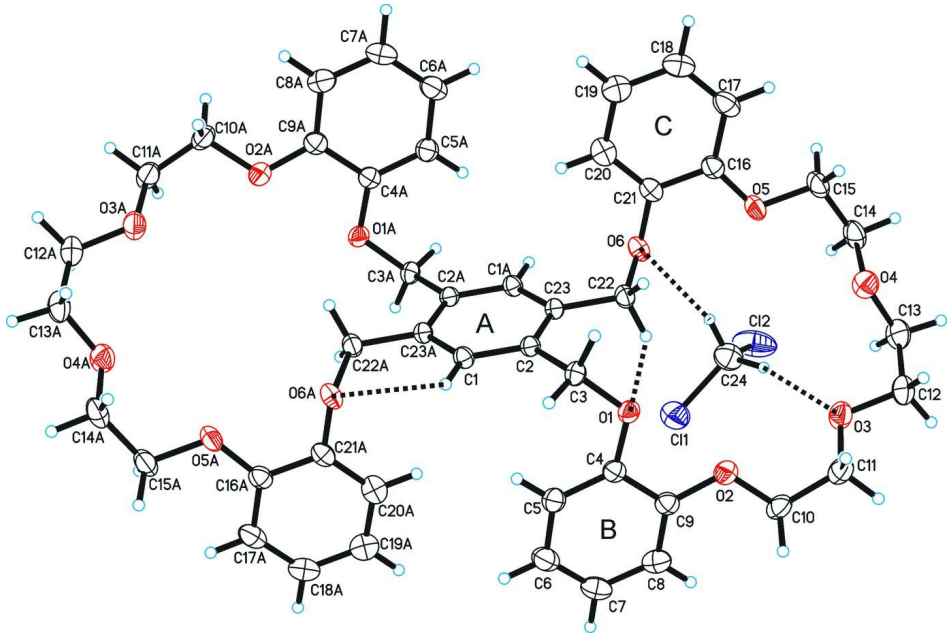

Figure 1

The molecular entities of the title compound with the atom numbering scheme and C—H···O interactions (dotted lines). Displacement ellipsoids are drawn at the 30% probability level. H atoms are presented as small spheres of arbitrary radius. [Symmetry code A): -*x* + 1, -*y* + 1, -*z* + 1].

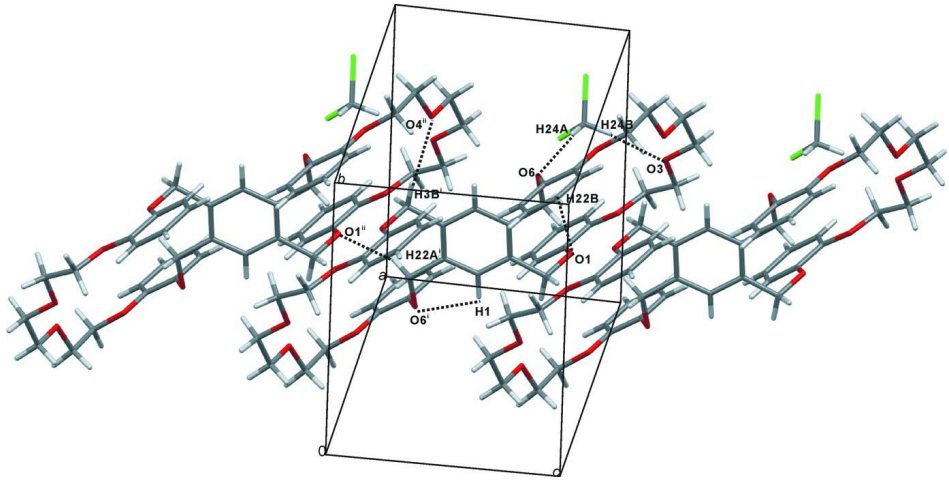

Figure 2

Crystal packing of the title compound with intra- and intermolecular C—H···O hydrogen bonds shown as dashed lines. [Symmetry codes: (i) -*x* + 1, -*y* + 1, -*z* + 1; (ii) -*x* + 1, -*y* + 1, -*z*.]

2,8,15,18,21,24,31,37,44,47,50,53-Dodecaoxaheptacyclo[52.4.0.0^{4,35}.0^{6,33}.0^{9,14}.0^{25,30}.0^{38,43}]octapentaconta-1(54),4,6(33),9(14),10,12,25(30),26,28,34,38(43),39,41,55,57-pentadecaene dichloromethane disolvate

Crystal data

$C_{46}H_{50}O_{12}\cdot 2CH_2Cl_2$
M_r = 964.71
Orthorhombic, *Pccn*
Hall symbol: -P 2ab 2ac
a = 23.0916 (11) Å
b = 23.9520 (11) Å
c = 8.6426 (4) Å
V = 4780.1 (4) Å^3
Z = 4
F(000) = 2024
D_x = 1.340 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 4261 reflections
θ = 2.5–22.2°
μ = 0.31 mm^{-1}
T = 200 K
Block, colorless
0.25 × 0.24 × 0.18 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.927, T_{max} = 0.947
26165 measured reflections
4211 independent reflections
2588 reflections with $I > 2\sigma(I)$
R_{int} = 0.077
θ_{max} = 25.0°, θ_{min} = 2.5°
h = −27→22
k = −28→27
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.136
S = 1.02
4211 reflections
289 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
$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 2.3252P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.54545 (8)	0.54550 (8)	0.1042 (2)	0.0372 (5)
O2	0.63386 (9)	0.52751 (8)	−0.0718 (2)	0.0451 (5)

O3	0.65434 (10)	0.42923 (8)	−0.2639 (2)	0.0527 (6)
O4	0.58731 (10)	0.32856 (8)	−0.2708 (2)	0.0542 (6)
O5	0.51826 (10)	0.31134 (8)	−0.0013 (2)	0.0492 (6)
O6	0.52007 (9)	0.37487 (7)	0.2487 (2)	0.0405 (5)
C1	0.48858 (12)	0.55573 (11)	0.4876 (3)	0.0315 (7)
H1A	0.4806	0.5945	0.4790	0.038*
C2	0.49824 (11)	0.52536 (11)	0.3538 (3)	0.0300 (6)
C3	0.49439 (12)	0.55343 (12)	0.1976 (3)	0.0353 (7)
H3A	0.4604	0.5385	0.1413	0.042*
H3B	0.4881	0.5939	0.2131	0.042*
C4	0.59462 (13)	0.57372 (11)	0.1441 (3)	0.0353 (7)
C5	0.59937 (14)	0.61083 (12)	0.2657 (3)	0.0434 (8)
H5A	0.5667	0.6184	0.3290	0.052*
C6	0.65181 (16)	0.63729 (13)	0.2960 (4)	0.0532 (9)
H6A	0.6548	0.6628	0.3799	0.064*
C7	0.69873 (15)	0.62667 (13)	0.2057 (4)	0.0567 (9)
H7A	0.7345	0.6446	0.2275	0.068*
C8	0.69505 (14)	0.58999 (13)	0.0822 (4)	0.0515 (9)
H8A	0.7281	0.5830	0.0198	0.062*
C9	0.64311 (13)	0.56349 (11)	0.0498 (3)	0.0398 (7)
C10	0.68141 (14)	0.52010 (13)	−0.1761 (4)	0.0494 (8)
H10A	0.7139	0.5015	−0.1219	0.059*
H10B	0.6951	0.5569	−0.2133	0.059*
C11	0.66245 (15)	0.48529 (13)	−0.3103 (4)	0.0530 (9)
H11A	0.6257	0.5003	−0.3525	0.064*
H11B	0.6921	0.4870	−0.3929	0.064*
C12	0.65282 (15)	0.39212 (13)	−0.3923 (4)	0.0527 (9)
H12A	0.6898	0.3944	−0.4502	0.063*
H12B	0.6210	0.4028	−0.4631	0.063*
C13	0.64360 (15)	0.33379 (14)	−0.3356 (4)	0.0572 (9)
H13A	0.6480	0.3072	−0.4226	0.069*
H13B	0.6731	0.3246	−0.2563	0.069*
C14	0.57832 (16)	0.27550 (12)	−0.2012 (4)	0.0555 (9)
H14A	0.6080	0.2690	−0.1205	0.067*
H14B	0.5820	0.2458	−0.2802	0.067*
C15	0.51951 (16)	0.27369 (13)	−0.1309 (4)	0.0539 (9)
H15A	0.4901	0.2849	−0.2083	0.065*
H15B	0.5106	0.2353	−0.0961	0.065*
C16	0.46768 (14)	0.31477 (12)	0.0792 (3)	0.0408 (7)
C17	0.41664 (16)	0.28668 (12)	0.0421 (4)	0.0525 (9)
H17A	0.4157	0.2623	−0.0446	0.063*
C18	0.36749 (16)	0.29396 (13)	0.1303 (4)	0.0557 (9)
H18A	0.3328	0.2751	0.1032	0.067*
C19	0.36849 (15)	0.32842 (14)	0.2573 (4)	0.0554 (9)
H19A	0.3347	0.3331	0.3185	0.066*
C20	0.41895 (14)	0.35637 (13)	0.2958 (4)	0.0494 (8)
H20A	0.4195	0.3805	0.3831	0.059*
C21	0.46838 (13)	0.34958 (11)	0.2089 (3)	0.0381 (7)

C22	0.52138 (13)	0.43413 (11)	0.2221 (3)	0.0384 (7)
H22A	0.4919	0.4437	0.1431	0.046*
H22B	0.5598	0.4444	0.1801	0.046*
C23	0.50998 (12)	0.46813 (11)	0.3662 (3)	0.0309 (7)
C24	0.65709 (16)	0.37292 (13)	0.0881 (4)	0.0657 (10)
H24A	0.6180	0.3635	0.1271	0.079*
H24B	0.6532	0.3846	−0.0213	0.079*
Cl1	0.68538 (5)	0.42883 (4)	0.19717 (12)	0.0760 (3)
Cl2	0.70129 (5)	0.31375 (4)	0.09828 (17)	0.1023 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0369 (12)	0.0466 (12)	0.0280 (10)	−0.0047 (9)	0.0004 (9)	0.0016 (9)
O2	0.0423 (13)	0.0467 (12)	0.0462 (12)	−0.0017 (10)	0.0089 (10)	−0.0019 (10)
O3	0.0675 (16)	0.0459 (13)	0.0448 (13)	0.0044 (11)	0.0116 (11)	−0.0022 (10)
O4	0.0609 (16)	0.0430 (13)	0.0586 (14)	0.0097 (11)	0.0076 (12)	−0.0021 (11)
O5	0.0646 (16)	0.0397 (12)	0.0433 (13)	−0.0064 (11)	0.0002 (11)	−0.0150 (10)
O6	0.0478 (13)	0.0330 (11)	0.0407 (12)	0.0052 (9)	−0.0076 (10)	−0.0096 (9)
C1	0.0347 (17)	0.0285 (14)	0.0314 (16)	0.0019 (12)	−0.0023 (13)	−0.0001 (12)
C2	0.0309 (16)	0.0350 (15)	0.0242 (15)	−0.0015 (12)	−0.0018 (12)	0.0008 (12)
C3	0.0363 (18)	0.0404 (16)	0.0293 (15)	0.0018 (13)	0.0005 (13)	0.0006 (13)
C4	0.0412 (18)	0.0330 (15)	0.0318 (16)	−0.0015 (13)	−0.0045 (14)	0.0070 (13)
C5	0.049 (2)	0.0461 (18)	0.0356 (17)	−0.0046 (15)	−0.0006 (15)	0.0023 (14)
C6	0.062 (2)	0.0460 (19)	0.052 (2)	−0.0118 (17)	−0.0069 (18)	−0.0019 (16)
C7	0.046 (2)	0.053 (2)	0.070 (2)	−0.0133 (17)	−0.0107 (19)	−0.0001 (19)
C8	0.041 (2)	0.0488 (19)	0.064 (2)	−0.0030 (16)	0.0037 (17)	0.0056 (17)
C9	0.046 (2)	0.0339 (16)	0.0397 (17)	0.0005 (14)	−0.0002 (15)	0.0047 (14)
C10	0.046 (2)	0.0490 (19)	0.054 (2)	0.0010 (16)	0.0166 (16)	0.0026 (17)
C11	0.056 (2)	0.049 (2)	0.053 (2)	0.0049 (17)	0.0181 (17)	0.0034 (17)
C12	0.054 (2)	0.060 (2)	0.0443 (19)	0.0097 (17)	0.0051 (16)	−0.0080 (17)
C13	0.058 (2)	0.054 (2)	0.060 (2)	0.0160 (17)	0.0065 (19)	−0.0147 (18)
C14	0.077 (3)	0.0380 (18)	0.051 (2)	0.0061 (17)	−0.0004 (19)	−0.0140 (16)
C15	0.077 (3)	0.0376 (17)	0.0471 (19)	−0.0057 (17)	−0.0002 (19)	−0.0130 (15)
C16	0.051 (2)	0.0325 (16)	0.0385 (17)	−0.0021 (15)	−0.0043 (16)	0.0021 (14)
C17	0.071 (3)	0.0365 (18)	0.050 (2)	−0.0106 (17)	−0.0112 (19)	−0.0001 (15)
C18	0.052 (2)	0.047 (2)	0.068 (2)	−0.0098 (17)	−0.014 (2)	0.0106 (19)
C19	0.049 (2)	0.058 (2)	0.059 (2)	0.0033 (17)	−0.0062 (18)	0.0075 (19)
C20	0.052 (2)	0.0482 (19)	0.047 (2)	0.0087 (17)	−0.0097 (17)	−0.0010 (16)
C21	0.046 (2)	0.0294 (15)	0.0389 (17)	0.0039 (14)	−0.0078 (15)	0.0012 (14)
C22	0.0486 (19)	0.0345 (16)	0.0322 (16)	−0.0012 (14)	0.0008 (14)	−0.0061 (13)
C23	0.0330 (17)	0.0345 (15)	0.0253 (15)	−0.0006 (12)	−0.0013 (12)	−0.0034 (12)
C24	0.065 (3)	0.061 (2)	0.071 (2)	−0.0013 (19)	−0.010 (2)	0.0017 (19)
Cl1	0.0777 (7)	0.0795 (7)	0.0707 (7)	−0.0151 (5)	−0.0071 (5)	−0.0053 (5)
Cl2	0.0663 (7)	0.0586 (6)	0.1820 (13)	−0.0014 (5)	−0.0233 (8)	0.0133 (7)

Geometric parameters (Å, º)

O1—C4	1.366 (3)	C10—H10B	0.9900
O1—C3	1.441 (3)	C11—H11A	0.9900
O2—C9	1.376 (3)	C11—H11B	0.9900
O2—C10	1.432 (3)	C12—C13	1.496 (4)
O3—C11	1.414 (3)	C12—H12A	0.9900
O3—C12	1.423 (3)	C12—H12B	0.9900
O4—C13	1.421 (4)	C13—H13A	0.9900
O4—C14	1.421 (4)	C13—H13B	0.9900
O5—C16	1.362 (4)	C14—C15	1.488 (5)
O5—C15	1.438 (3)	C14—H14A	0.9900
O6—C21	1.382 (3)	C14—H14B	0.9900
O6—C22	1.438 (3)	C15—H15A	0.9900
C1—C2	1.384 (4)	C15—H15B	0.9900
C1—C23^{i}	1.387 (4)	C16—C17	1.394 (4)
C1—H1A	0.9500	C16—C21	1.397 (4)
C2—C23	1.402 (3)	C17—C18	1.378 (5)
C2—C3	1.510 (4)	C17—H17A	0.9500
C3—H3A	0.9900	C18—C19	1.373 (5)
C3—H3B	0.9900	C18—H18A	0.9500
C4—C5	1.381 (4)	C19—C20	1.384 (4)
C4—C9	1.407 (4)	C19—H19A	0.9500
C5—C6	1.392 (4)	C20—C21	1.376 (4)
C5—H5A	0.9500	C20—H20A	0.9500
C6—C7	1.360 (5)	C22—C23	1.511 (4)
C6—H6A	0.9500	C22—H22A	0.9900
C7—C8	1.385 (4)	C22—H22B	0.9900
C7—H7A	0.9500	C23—C1^{i}	1.387 (4)
C8—C9	1.386 (4)	C24—Cl2	1.749 (4)
C8—H8A	0.9500	C24—Cl1	1.763 (3)
C10—C11	1.494 (4)	C24—H24A	0.9900
C10—H10A	0.9900	C24—H24B	0.9900
C4—O1—C3	118.3 (2)	H12A—C12—H12B	108.3
C9—O2—C10	116.1 (2)	O4—C13—C12	110.0 (3)
C11—O3—C12	112.0 (2)	O4—C13—H13A	109.7
C13—O4—C14	112.3 (2)	C12—C13—H13A	109.7
C16—O5—C15	116.9 (2)	O4—C13—H13B	109.7
C21—O6—C22	114.3 (2)	C12—C13—H13B	109.7
C2—C1—C23^{i}	122.7 (2)	H13A—C13—H13B	108.2
C2—C1—H1A	118.6	O4—C14—C15	109.4 (3)
C23^{i}—C1—H1A	118.6	O4—C14—H14A	109.8
C1—C2—C23	118.7 (2)	C15—C14—H14A	109.8
C1—C2—C3	120.2 (2)	O4—C14—H14B	109.8
C23—C2—C3	121.0 (2)	C15—C14—H14B	109.8
O1—C3—C2	113.2 (2)	H14A—C14—H14B	108.2
O1—C3—H3A	108.9	O5—C15—C14	108.5 (3)

C2—C3—H3A	108.9	O5—C15—H15A	110.0
O1—C3—H3B	108.9	C14—C15—H15A	110.0
C2—C3—H3B	108.9	O5—C15—H15B	110.0
H3A—C3—H3B	107.8	C14—C15—H15B	110.0
O1—C4—C5	125.2 (3)	H15A—C15—H15B	108.4
O1—C4—C9	115.4 (2)	O5—C16—C17	125.4 (3)
C5—C4—C9	119.3 (3)	O5—C16—C21	115.8 (3)
C4—C5—C6	120.4 (3)	C17—C16—C21	118.8 (3)
C4—C5—H5A	119.8	C18—C17—C16	120.5 (3)
C6—C5—H5A	119.8	C18—C17—H17A	119.7
C7—C6—C5	120.0 (3)	C16—C17—H17A	119.7
C7—C6—H6A	120.0	C19—C18—C17	120.3 (3)
C5—C6—H6A	120.0	C19—C18—H18A	119.9
C6—C7—C8	120.8 (3)	C17—C18—H18A	119.9
C6—C7—H7A	119.6	C18—C19—C20	119.8 (3)
C8—C7—H7A	119.6	C18—C19—H19A	120.1
C7—C8—C9	120.0 (3)	C20—C19—H19A	120.1
C7—C8—H8A	120.0	C21—C20—C19	120.6 (3)
C9—C8—H8A	120.0	C21—C20—H20A	119.7
O2—C9—C8	125.2 (3)	C19—C20—H20A	119.7
O2—C9—C4	115.4 (3)	C20—C21—O6	121.9 (3)
C8—C9—C4	119.5 (3)	C20—C21—C16	119.9 (3)
O2—C10—C11	109.5 (3)	O6—C21—C16	118.1 (3)
O2—C10—H10A	109.8	O6—C22—C23	113.4 (2)
C11—C10—H10A	109.8	O6—C22—H22A	108.9
O2—C10—H10B	109.8	C23—C22—H22A	108.9
C11—C10—H10B	109.8	O6—C22—H22B	108.9
H10A—C10—H10B	108.2	C23—C22—H22B	108.9
O3—C11—C10	110.4 (3)	H22A—C22—H22B	107.7
O3—C11—H11A	109.6	C1^{i}—C23—C2	118.5 (2)
C10—C11—H11A	109.6	C1^{i}—C23—C22	121.6 (2)
O3—C11—H11B	109.6	C2—C23—C22	119.8 (2)
C10—C11—H11B	109.6	Cl2—C24—Cl1	111.9 (2)
H11A—C11—H11B	108.1	Cl2—C24—H24A	109.2
O3—C12—C13	109.3 (3)	Cl1—C24—H24A	109.2
O3—C12—H12A	109.8	Cl2—C24—H24B	109.2
C13—C12—H12A	109.8	Cl1—C24—H24B	109.2
O3—C12—H12B	109.8	H24A—C24—H24B	107.9
C13—C12—H12B	109.8		

C23^{i}—C1—C2—C23	0.0 (4)	C16—O5—C15—C14	−179.3 (3)
C23^{i}—C1—C2—C3	178.2 (3)	O4—C14—C15—O5	−68.1 (3)
C4—O1—C3—C2	−72.7 (3)	C15—O5—C16—C17	−2.9 (4)
C1—C2—C3—O1	126.4 (3)	C15—O5—C16—C21	177.1 (2)
C23—C2—C3—O1	−55.4 (3)	O5—C16—C17—C18	−178.9 (3)
C3—O1—C4—C5	−1.8 (4)	C21—C16—C17—C18	1.2 (4)
C3—O1—C4—C9	179.1 (2)	C16—C17—C18—C19	−1.0 (5)
O1—C4—C5—C6	179.9 (3)	C17—C18—C19—C20	0.6 (5)

C9—C4—C5—C6	−0.9 (4)	C18—C19—C20—C21	−0.6 (5)
C4—C5—C6—C7	0.0 (5)	C19—C20—C21—O6	−176.9 (3)
C5—C6—C7—C8	0.5 (5)	C19—C20—C21—C16	0.9 (4)
C6—C7—C8—C9	−0.2 (5)	C22—O6—C21—C20	−72.7 (3)
C10—O2—C9—C8	−3.7 (4)	C22—O6—C21—C16	109.4 (3)
C10—O2—C9—C4	175.2 (2)	O5—C16—C21—C20	178.9 (3)
C7—C8—C9—O2	178.1 (3)	C17—C16—C21—C20	−1.1 (4)
C7—C8—C9—C4	−0.7 (4)	O5—C16—C21—O6	−3.2 (4)
O1—C4—C9—O2	1.5 (3)	C17—C16—C21—O6	176.7 (2)
C5—C4—C9—O2	−177.7 (2)	C21—O6—C22—C23	98.8 (3)
O1—C4—C9—C8	−179.5 (2)	C1—C2—C23—C1^{i}	0.0 (4)
C5—C4—C9—C8	1.2 (4)	C3—C2—C23—C1^{i}	−178.2 (3)
C9—O2—C10—C11	−173.6 (2)	C1—C2—C23—C22	−179.3 (3)
C12—O3—C11—C10	−163.0 (3)	C3—C2—C23—C22	2.5 (4)
O2—C10—C11—O3	−71.6 (3)	O6—C22—C23—C1^{i}	13.6 (4)
C11—O3—C12—C13	−179.5 (3)	O6—C22—C23—C2	−167.1 (2)
C14—O4—C13—C12	−174.2 (3)	C2—C3—O1—C4	−72.7 (3)
O3—C12—C13—O4	67.2 (3)	C23—C22—O6—C21	98.8 (3)
C13—O4—C14—C15	177.9 (3)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O6^{i}	0.95	2.47	2.828 (3)	103
C3—H3*B*···O4^{ii}	0.99	2.59	3.457 (4)	146
C22—H22*A*···O1^{ii}	0.99	2.32	3.252 (3)	156
C22—H22*B*···O1	0.99	2.53	2.909 (3)	103
C24—H24*A*···O6	0.99	2.51	3.455 (4)	160
C24—H24*B*···O3	0.99	2.36	3.331 (5)	168

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