Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 2,9,16,19,22,25-Hexaoxatetracyclo-[24.4.0.2$^{4,7}$.0$^{10,15}$]dotriaconta-1(26),4,6,10(15),11,13,27,29,31-nonaene

**Jai Young Lee,[a] Ji-Eun Lee,[b] Wonbo Sim[a]* and Ki-Min Park[c]***

[a]Department of Chemistry, Konyang University, Nonsan 320-711, Republic of Korea, [b]Central Instrument Facility, Gyeongsang National University, Jinju 660-701, Republic of Korea, and [c]Research Institute of Natural Science, Gyeongsang National University, Jinju 660-701, Republic of Korea
Correspondence e-mail: wsim@konyang.ac.kr, kmpark@gnu.ac.kr

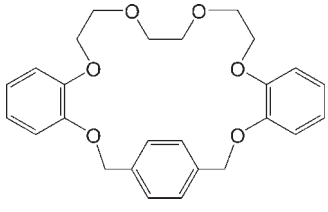
Received 25 August 2009; accepted 2 September 2009

Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.041; $wR$ factor = 0.116; data-to-parameter ratio = 13.5.

The title 22-crown-6 unit, $C_{26}H_{28}O_6$, comprising of three benzo groups and triethylene glycol, was prepared by the reaction of $\alpha,\alpha'$-dibromo-*p*-xylene with 1,8-bis(2-hydroxyphenoxy)-3,6-dioxaoctane in the presence of $Cs_2CO_3$ with tetrahydrofuran (THF) and recrystallized from dichloromethane–hexane (1:20 *v*/*v*) at room temperature. In the molecular structure, two O atoms of the central ethylene glycol in the triethylene glycol unit exhibit *exo* conformations due to intramolecular C—H···O interactions. A number of C—H···O and C—H···$\pi$ intermolecular interactions contribute to the stabilization of the crystal packing.

## Related literature

For the preparation of related compounds, see: Sim *et al.* (2001); Weber & Vögtle (1976). For a related structure, see: Sim *et al.* (2001). For background to crown ether-based macrocyclic compounds and their inclusion behaviour, see: Gokel & Korzeniowski (1982); Izatt & Christensen (1981); Lindoy (1989); Pedersen (1967); Vögtle & Weber (1985); Weber *et al.* (1989); Wolf *et al.* (1987).



## Experimental

### Crystal data

$C_{26}H_{28}O_6$
$M_r$ = 436.48
Monoclinic, $P2_1/c$
$a$ = 12.348 (3) Å
$b$ = 18.908 (4) Å
$c$ = 9.824 (2) Å
$\beta$ = 105.70 (3)°
$V$ = 2208.0 (8) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 293 K
0.25 × 0.20 × 0.10 mm

### Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
4142 measured reflections
3896 independent reflections
2654 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.057

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.116
$S$ = 1.02
3896 reflections
289 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.19 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C9—H9$A$···O2 | 0.97 | 2.62 | 3.114 (2) | 112 |
| C10—H10$A$···O5 | 0.97 | 2.70 | 3.117 (2) | 106 |
| C2—H2···O1$^{i}$ | 0.93 | 2.76 | 3.471 (2) | 134 |
| C7—H7$B$···O5$^{ii}$ | 0.97 | 2.72 | 3.607 (2) | 153 |
| C11—H11$B$···O1$^{ii}$ | 0.97 | 2.82 | 3.501 (3) | 128 |
| C11—H11$B$···O2$^{ii}$ | 0.97 | 2.90 | 3.798 (3) | 154 |
| C12—H12$B$···O4$^{iii}$ | 0.97 | 2.42 | 3.294 (3) | 149 |
| C25—H25···O3$^{ii}$ | 0.93 | 2.71 | 3.433 (2) | 135 |
| C12—H12$A$···$Cg1^{ii}$ | 0.97 | 2.76 | 3.47 | 138 |
| C21—H21···$Cg2^{i}$ | 0.93 | 2.97 | 3.47 | 115 |
| C26—H26$A$···$Cg3^{i}$ | 0.97 | 3.06 | 3.83 | 138 |

Symmetry codes: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $-x, -y, -z+2$; (iii) $-x, -y, -z+1$. $Cg1$, $Cg2$ and $Cg3$ are the centroids of the C1–C6, C13–C18 and C20–C25 rings, respectively.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT-Plus* (Bruker, 2000); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXTL*.

This work was supported by the Konyang University Research Fund in 2009 and also by a Korea Research Foundation Grant funded by the Korean Government (MOEHRD) (KRF-2007–359-C00019).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2101).

## References

Brandenburg, K. (1998). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2000). *SMART* and *SAINT-Plus*. Bruker AXS Inc., Madison, Wisconsin, USA.
Gokel, G. W. & Korzeniowski, S. H. (1982). In *Macrocyclic Polyether Syntheses*. Berlin/Heidelberg/New York: Springer-Verlag.
Izatt, R. M. & Christensen, J. J. (1981). In *Progress in Macrocyclic Chemistry*, Vol. 2. New York: John Wiley and Sons.

Lindoy, L. F. (1989). In *The Chemistry of Macrocyclic Ligand Complexes*. Cambridge University Press.
Pedersen, C. J. (1967). *J. Am. Chem. Soc.* **89**, 2495–2496.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sim, W., Lee, J. Y., Kim, J. S., Kim, M.-J., Kim, J.-G. & Suh, I.-H. (2001). *Acta Cryst.* E**57**, o416–o418.
Vögtle, F. & Weber, E. (1985). In *Host Guest Complex Chemistry Macrocycles*. Berlin/Heidelberg/New York: Springer-Verlag.
Weber, E., Toner, J. L., Goldberg, S., Vögtle, F., Laidler, D. A., Stoddart, J. F., Bartsch, R. A. & Liotta, C. L. (1989). In *Crown Ethers and Analogs*. New York: John Wiley and Sons.
Weber, E. & Vögtle, F. (1976). *Chem. Ber.* **109**, 1803–1831.
Wolf, R. E. Jr, Hartman, J. R., Storey, J. M. E., Foxman, B. M. & Cooper, S. R. (1987). *J. Am. Chem. Soc.* **109**, 4328–4335.

# supporting information

*Acta Cryst.* (2009). E**65**, o2369–o2370 [doi:10.1107/S1600536809035399]

## 2,9,16,19,22,25-Hexaoxatetracyclo-[24.4.0.2$^{4,7}$.0$^{10,15}$]dotriaconta-1(26),4,6,10(15),11,13,27,29,31-nonaene

**Jai Young Lee, Ji-Eun Lee, Wonbo Sim and Ki-Min Park**

### S1. Comment

An extraordinary variety of crown ether-based macrocyclic compounds have been synthesized and reported since crown ether was first discovered (Pedersen, 1967) because their topological interesting as well as their inclusion behaviour (Gokel & Korzeniowski, 1982; Izatt & Christensen, 1981; Lindoy 1989; Vögtle & Weber, 1985; Weber *et al.* 1989). We have also synthesized and reported the preparation and the solid-state structure of new crown ether (I) bearing three aromatic subunits (Sim *et al.*, 2001). As a part of our continuing interest in the development of new crown compounds, the preparation and crystal structure of new crown ether-based macrocyclic compound (II) containing three benzo units of which ring size is larger than that of the previous reported compound (I) are presented here.

The title compound (II), 2,9,16,19,22,25-hexaoxatetracyclo[24.4.2$^{4,7}$.0.0$^{10,15}$]-dotriaconta-1(26),4,6, 10 (15),11,13,27,29,31-nonaene was prepared by the reaction of $\alpha,\alpha'$-dibromo-*p*-xylene with 1,8-bis(2-hydroxyphenoxy)-3,6-dioxaoctane in the presence of $Cs_2CO_3$ with tetrahydrofuran (THF) and recrystallized from dichloromethane/hexane (1:20) at room temperature to give colorless single crystals suitable for X-ray analysis.

In the molecular structure of (II), the torsion angels of C—C—O—C connecting A-to-B rings and A-to-C rings aromatic are 167.0 (2)° and 163.4 (2)°, respectively, which indicate that the A ring is situated *trans* to both the B and C rings, with dihedral angles of 57.04 (8)° between A and B and 44.41 (8)° between A and C. The dihedral angle between B and C rings is 14.2 (1)°. The all O—C—C—O and C—C—O—C torsion angles except two C—C—O—C in the triethylene glycol group exhibit *gauche* conformation. Two exceptional C—C—O—C (C10—C9—O3—C8 and C9—C10—O4—C11) torsion angles are *trans* conformation with the values of -162.5 (2)° and -156.1 (2)°, respectively.

Interestingly, two oxygen atoms of the central ethylene glycol in the triethylene glycol unit exhibit the *exo*-orientations which are very different from those found in the ethylene glycol backbone of (I) (Sim *et al.*, 2001) or common crown ether-based compounds. In general, oxygen atoms of ethylene glycol group in crown ether-based compounds favor *endo*-orientation (Wolf *et al.*, 1987). The *exo*-orientations of two oxygen atoms (O3 and O4) in (II) may be due to the intramolecular C—H···O interactions; 2.62 Å for C9—H9A···O2 and 2.70 Å for C10—H10A···O6 (Fig. 1 & Table 1).

The crystal packing is stabilized by not only intermolecular C—H···O hydrogen bonds with C—H···O separation in the range of 2.71–2.90 Å but intermolecular C—H···$\pi$ interactions with C—H···*Cg* separations in the range of 2.76–3.06 Å (Fig. 2 & Table 1; *Cg* is the centroid of aromatic ring).

### S2. Experimental

To a refluxing suspension of caesium carbonate (15.2 mmol) in THF under nitrogen was added dropwise a solution of $\alpha,\alpha'$-dibromo-*p*-xylene (3.75 mmol) and 1,8-bis(2-hydroxyphenoxy)-3,6-dioxaoctane (3.79 mmol) in THF over a period of 3 h. The mixture was then refluxed for an additional 24 h. 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 di-

chloromethane. 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, and recrystallized from dichloromethane/*n*-hexane (1:20, *v*/*v*) to give as a crystalline solid in 70% yield (m.p. 424 K). IR (KBr pellet): 2926, 1600, 1504, 1235, 1126, 996 and 735 $cm^{-1}$. $^1H$ NMR ($CDCl_3$): $\delta$ 7.54 (d, 4H, Ar-*H*), 7.06–6.91 (m, 8H, Ar-*H*), 5.06 (s, 4H, Ar$CH_2$O), 4.12 (t, 4H, ArO$CH_2CH_2OCH_2$), 3.88 (t, 4H, $ArOCH_2CH_2OCH_2$) and 3.70 (t, 4H, $ArOCH_2CH_2OCH_2$).

### S3. Refinement

(type here to add refinement details)

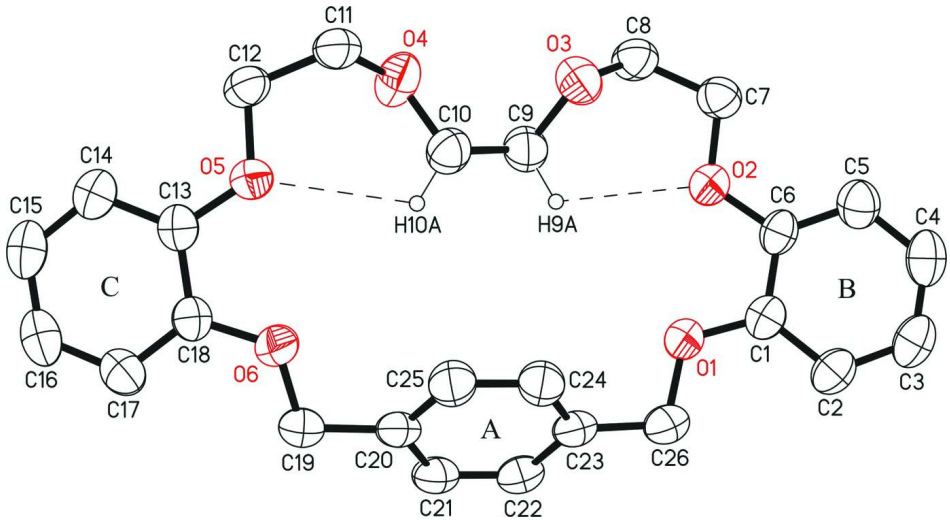



**Figure 1**

*ORTEP* drawing of (II) with the atom-numbering scheme and intramolecular C—H···O interactions (dotted lines). Displacement ellipsoids are drawn at the 50% probability level. All H atoms except two H atoms related to intramolecular C—H···O hydrogen bonds have been omitted for clarity.

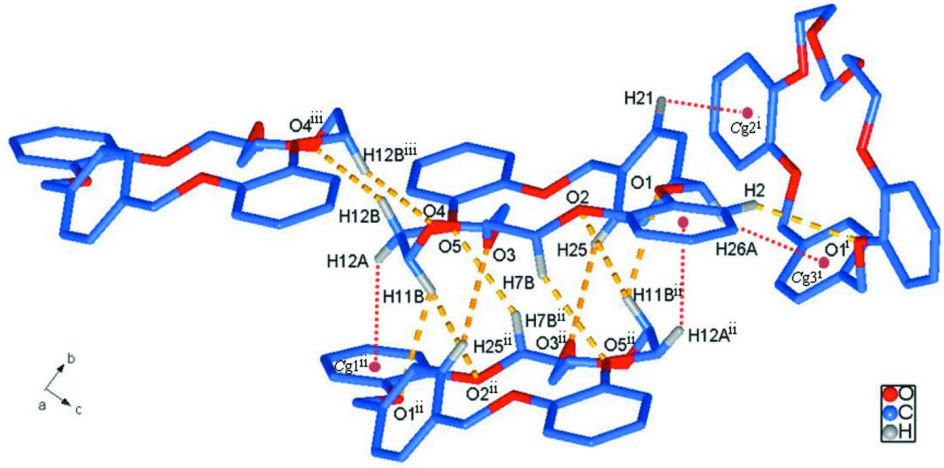



**Figure 2**

Intermolecular C—H···$\pi$ (red dotted lines) and C—H···O (yellow dotted lines) interactions in the title compound. All H atoms except those related to intermolecular interactions have been omitted for clarity. *Cg*1, *Cg*2 and *Cg*3 denote the centroids of rings consisting of C1/C2/C3/C4/C5/C6, C13/C14/C15/C16/C17/C18 and C20/C21/C22/C23/C24/C25, respectively. [Symmetry codes: (i) *x*, -*y* + 1/2, *z* + 1/2; (ii) -*x*, -*y*, -*z* + 2; (iii) -*x*, -*y*, -*z* + 1]

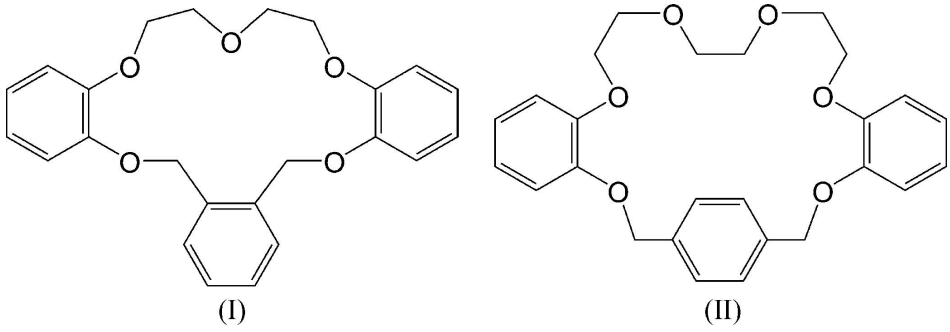



**Figure 3**

The structures of (I) and (II).

**2,9,16,19,22,25-Hexaoxatetracyclo[24.4.0.2$^{4,7}$.0$^{10,15}$]dotriaconta- 1(26),4,6,10 (15),11,13,27,29,31-nonaene**

*Crystal data*

$C_{26}H_{28}O_6$
$M_r$ = 436.48
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a$ = 12.348 (3) Å
$b$ = 18.908 (4) Å
$c$ = 9.824 (2) Å
$\beta$ = 105.70 (3)°
$V$ = 2208.0 (8) Å$^3$
$Z$ = 4
$F$(000) = 928
$D_x$ = 1.313 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 3896 reflections
$\theta$ = 1.7–25.0°
$\mu$ = 0.09 mm$^{-1}$
$T$ = 293 K
Plate, colorless
0.25 × 0.20 × 0.10 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
4142 measured reflections
3896 independent reflections
2654 reflections with $I > 2\sigma(I)$
$R_{int} = 0.057$
$\theta_{max} = 25.1°$, $\theta_{min} = 1.7°$
$h = -14 \rightarrow 14$
$k = 0 \rightarrow 22$
$l = -11 \rightarrow 0$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.116$
$S = 1.02$
3896 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
$w = 1/[\sigma^2(F_o^2) + (0.0672P)^2 + 0.0279P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.14$ e Å$^{-3}$
$\Delta\rho_{min} = -0.19$ 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | −0.14749 (11) | 0.20218 (7) | 1.10961 (13) | 0.0498 (3) |
| O2 | −0.28741 (11) | 0.11040 (7) | 0.97137 (13) | 0.0520 (4) |
| O3 | −0.17603 (11) | 0.00949 (7) | 0.83350 (15) | 0.0568 (4) |
| O4 | −0.00678 (11) | 0.00265 (9) | 0.68633 (14) | 0.0624 (4) |
| O5 | 0.23169 (10) | 0.03178 (7) | 0.73303 (12) | 0.0474 (3) |
| O6 | 0.29941 (11) | 0.14286 (7) | 0.87761 (14) | 0.0552 (4) |
| C1 | −0.23684 (15) | 0.18893 (10) | 1.16215 (19) | 0.0446 (4) |
| C2 | −0.25554 (17) | 0.21956 (11) | 1.2812 (2) | 0.0538 (5) |
| H2 | −0.2057 | 0.2534 | 1.3313 | 0.065* |
| C3 | −0.34797 (19) | 0.20021 (12) | 1.3264 (2) | 0.0626 (6) |
| H3 | −0.3593 | 0.2204 | 1.4077 | 0.075* |
| C4 | −0.42279 (19) | 0.15159 (12) | 1.2525 (2) | 0.0639 (6) |
| H4 | −0.4854 | 0.1393 | 1.2828 | 0.077* |
| C5 | −0.40553 (17) | 0.12059 (11) | 1.1322 (2) | 0.0571 (5) |
| H5 | −0.4568 | 0.0876 | 1.0820 | 0.069* |
| C6 | −0.31315 (15) | 0.13823 (10) | 1.08660 (18) | 0.0449 (4) |
| C7 | −0.33649 (16) | 0.04425 (10) | 0.9193 (2) | 0.0509 (5) |

| | | | | |
|---|---|---|---|---|
| H7A | −0.4178 | 0.0485 | 0.8889 | 0.061* |
| H7B | −0.3169 | 0.0088 | 0.9933 | 0.061* |
| C8 | −0.29280 (16) | 0.02303 (12) | 0.7974 (2) | 0.0557 (5) |
| H8A | −0.3324 | −0.0192 | 0.7546 | 0.067* |
| H8B | −0.3098 | 0.0603 | 0.7271 | 0.067* |
| C9 | −0.11025 (18) | 0.06755 (10) | 0.8132 (2) | 0.0567 (5) |
| H9A | −0.1033 | 0.1014 | 0.8893 | 0.068* |
| H9B | −0.1464 | 0.0910 | 0.7247 | 0.068* |
| C10 | 0.00358 (16) | 0.04204 (11) | 0.8106 (2) | 0.0532 (5) |
| H10A | 0.0533 | 0.0820 | 0.8129 | 0.064* |
| H10B | 0.0354 | 0.0127 | 0.8927 | 0.064* |
| C11 | 0.07916 (16) | −0.04707 (10) | 0.6910 (2) | 0.0551 (5) |
| H11A | 0.0465 | −0.0882 | 0.6360 | 0.066* |
| H11B | 0.1099 | −0.0623 | 0.7881 | 0.066* |
| C12 | 0.17270 (15) | −0.02012 (10) | 0.6366 (2) | 0.0480 (5) |
| H12A | 0.2229 | −0.0585 | 0.6292 | 0.058* |
| H12B | 0.1430 | 0.0006 | 0.5436 | 0.058* |
| C13 | 0.31566 (15) | 0.06729 (10) | 0.69757 (18) | 0.0426 (4) |
| C14 | 0.36356 (17) | 0.04880 (11) | 0.5914 (2) | 0.0541 (5) |
| H14 | 0.3391 | 0.0084 | 0.5380 | 0.065* |
| C15 | 0.44755 (18) | 0.08973 (12) | 0.5636 (2) | 0.0617 (6) |
| H15 | 0.4794 | 0.0768 | 0.4916 | 0.074* |
| C16 | 0.48412 (16) | 0.14911 (12) | 0.6415 (2) | 0.0588 (6) |
| H16 | 0.5405 | 0.1767 | 0.6223 | 0.071* |
| C17 | 0.43756 (16) | 0.16805 (11) | 0.7484 (2) | 0.0530 (5) |
| H17 | 0.4633 | 0.2083 | 0.8018 | 0.064* |
| C18 | 0.35325 (14) | 0.12816 (10) | 0.77722 (19) | 0.0427 (4) |
| C19 | 0.30700 (16) | 0.21254 (10) | 0.9329 (2) | 0.0521 (5) |
| H19A | 0.2975 | 0.2468 | 0.8571 | 0.063* |
| H19B | 0.3801 | 0.2199 | 0.9994 | 0.063* |
| C20 | 0.21604 (15) | 0.22107 (9) | 1.00562 (19) | 0.0457 (4) |
| C21 | 0.16951 (16) | 0.28661 (10) | 1.0145 (2) | 0.0501 (5) |
| H21 | 0.1968 | 0.3260 | 0.9777 | 0.060* |
| C22 | 0.08275 (17) | 0.29458 (10) | 1.0775 (2) | 0.0503 (5) |
| H22 | 0.0532 | 0.3394 | 1.0833 | 0.060* |
| C23 | 0.03947 (16) | 0.23753 (10) | 1.1316 (2) | 0.0492 (5) |
| C24 | 0.08909 (18) | 0.17237 (10) | 1.1269 (2) | 0.0565 (5) |
| H24 | 0.0634 | 0.1333 | 1.1664 | 0.068* |
| C25 | 0.17552 (17) | 0.16431 (10) | 1.0652 (2) | 0.0548 (5) |
| H25 | 0.2073 | 0.1198 | 1.0635 | 0.066* |
| C26 | −0.05795 (17) | 0.24454 (11) | 1.1932 (2) | 0.0574 (5) |
| H26A | −0.0373 | 0.2284 | 1.2905 | 0.069* |
| H26B | −0.0813 | 0.2936 | 1.1914 | 0.069* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0493 (7) | 0.0561 (8) | 0.0464 (7) | −0.0046 (6) | 0.0169 (6) | −0.0083 (6) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O2 | 0.0533 (8) | 0.0572 (8) | 0.0480 (7) | −0.0055 (6) | 0.0178 (6) | −0.0082 (6) |
| O3 | 0.0508 (8) | 0.0470 (8) | 0.0751 (9) | 0.0046 (6) | 0.0214 (7) | 0.0069 (7) |
| O4 | 0.0447 (7) | 0.0944 (11) | 0.0434 (7) | 0.0134 (8) | 0.0038 (6) | −0.0129 (7) |
| O5 | 0.0492 (7) | 0.0495 (7) | 0.0453 (7) | −0.0086 (6) | 0.0156 (6) | −0.0089 (6) |
| O6 | 0.0656 (9) | 0.0480 (8) | 0.0600 (8) | −0.0131 (7) | 0.0305 (7) | −0.0144 (6) |
| C1 | 0.0464 (10) | 0.0450 (11) | 0.0425 (10) | 0.0107 (8) | 0.0121 (9) | 0.0034 (8) |
| C2 | 0.0593 (12) | 0.0511 (12) | 0.0526 (12) | 0.0106 (10) | 0.0179 (10) | −0.0054 (9) |
| C3 | 0.0652 (13) | 0.0716 (14) | 0.0568 (13) | 0.0154 (12) | 0.0268 (11) | −0.0045 (11) |
| C4 | 0.0554 (12) | 0.0785 (16) | 0.0661 (14) | 0.0117 (12) | 0.0305 (11) | 0.0083 (12) |
| C5 | 0.0474 (11) | 0.0663 (14) | 0.0587 (13) | 0.0043 (10) | 0.0163 (10) | 0.0020 (11) |
| C6 | 0.0449 (10) | 0.0507 (11) | 0.0406 (10) | 0.0109 (9) | 0.0139 (8) | 0.0031 (9) |
| C7 | 0.0446 (10) | 0.0529 (12) | 0.0528 (11) | −0.0026 (9) | 0.0092 (9) | −0.0038 (9) |
| C8 | 0.0479 (11) | 0.0608 (12) | 0.0550 (12) | −0.0029 (10) | 0.0080 (9) | −0.0118 (10) |
| C9 | 0.0617 (13) | 0.0452 (11) | 0.0686 (13) | 0.0005 (10) | 0.0269 (11) | 0.0013 (10) |
| C10 | 0.0517 (11) | 0.0608 (13) | 0.0468 (11) | −0.0001 (10) | 0.0127 (9) | −0.0047 (10) |
| C11 | 0.0494 (11) | 0.0522 (12) | 0.0626 (12) | −0.0069 (10) | 0.0129 (10) | −0.0170 (10) |
| C12 | 0.0466 (11) | 0.0488 (11) | 0.0455 (10) | 0.0017 (9) | 0.0070 (9) | −0.0105 (9) |
| C13 | 0.0396 (10) | 0.0469 (11) | 0.0412 (10) | 0.0054 (8) | 0.0107 (8) | 0.0063 (8) |
| C14 | 0.0577 (12) | 0.0583 (12) | 0.0500 (11) | 0.0051 (10) | 0.0209 (10) | −0.0012 (10) |
| C15 | 0.0609 (13) | 0.0730 (15) | 0.0609 (13) | 0.0107 (12) | 0.0328 (11) | 0.0073 (12) |
| C16 | 0.0469 (11) | 0.0640 (14) | 0.0714 (14) | 0.0053 (10) | 0.0261 (11) | 0.0164 (12) |
| C17 | 0.0442 (10) | 0.0521 (11) | 0.0617 (13) | −0.0023 (9) | 0.0130 (10) | 0.0027 (10) |
| C18 | 0.0406 (10) | 0.0445 (10) | 0.0440 (10) | 0.0028 (8) | 0.0133 (8) | 0.0031 (8) |
| C19 | 0.0529 (12) | 0.0452 (11) | 0.0591 (12) | −0.0097 (9) | 0.0168 (10) | −0.0106 (9) |
| C20 | 0.0462 (10) | 0.0429 (10) | 0.0452 (10) | −0.0079 (8) | 0.0073 (9) | −0.0096 (8) |
| C21 | 0.0584 (12) | 0.0397 (10) | 0.0499 (11) | −0.0091 (9) | 0.0106 (9) | −0.0050 (9) |
| C22 | 0.0595 (12) | 0.0394 (10) | 0.0496 (11) | −0.0002 (9) | 0.0106 (10) | −0.0082 (9) |
| C23 | 0.0516 (11) | 0.0464 (11) | 0.0488 (11) | −0.0063 (9) | 0.0121 (9) | −0.0148 (9) |
| C24 | 0.0676 (13) | 0.0405 (11) | 0.0681 (14) | −0.0068 (10) | 0.0296 (11) | −0.0043 (10) |
| C25 | 0.0629 (13) | 0.0379 (11) | 0.0679 (13) | 0.0010 (9) | 0.0250 (11) | −0.0031 (9) |
| C26 | 0.0605 (13) | 0.0522 (12) | 0.0618 (13) | −0.0069 (10) | 0.0205 (11) | −0.0191 (10) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.362 (2) | C11—C12 | 1.488 (3) |
| O1—C26 | 1.431 (2) | C11—H11A | 0.9700 |
| O2—C6 | 1.362 (2) | C11—H11B | 0.9700 |
| O2—C7 | 1.423 (2) | C12—H12A | 0.9700 |
| O3—C9 | 1.412 (2) | C12—H12B | 0.9700 |
| O3—C8 | 1.412 (2) | C13—C14 | 1.376 (3) |
| O4—C10 | 1.406 (2) | C13—C18 | 1.398 (3) |
| O4—C11 | 1.409 (2) | C14—C15 | 1.379 (3) |
| O5—C13 | 1.357 (2) | C14—H14 | 0.9300 |
| O5—C12 | 1.420 (2) | C15—C16 | 1.365 (3) |
| O6—C18 | 1.359 (2) | C15—H15 | 0.9300 |
| O6—C19 | 1.418 (2) | C16—C17 | 1.374 (3) |
| C1—C2 | 1.380 (3) | C16—H16 | 0.9300 |
| C1—C6 | 1.407 (3) | C17—C18 | 1.376 (3) |

| | | | |
|---|---|---|---|
| C2—C3 | 1.381 (3) | C17—H17 | 0.9300 |
| C2—H2 | 0.9300 | C19—C20 | 1.494 (3) |
| C3—C4 | 1.365 (3) | C19—H19A | 0.9700 |
| C3—H3 | 0.9300 | C19—H19B | 0.9700 |
| C4—C5 | 1.386 (3) | C20—C21 | 1.379 (3) |
| C4—H4 | 0.9300 | C20—C25 | 1.380 (3) |
| C5—C6 | 1.375 (3) | C21—C22 | 1.383 (3) |
| C5—H5 | 0.9300 | C21—H21 | 0.9300 |
| C7—C8 | 1.495 (3) | C22—C23 | 1.373 (3) |
| C7—H7A | 0.9700 | C22—H22 | 0.9300 |
| C7—H7B | 0.9700 | C23—C24 | 1.382 (3) |
| C8—H8A | 0.9700 | C23—C26 | 1.491 (3) |
| C8—H8B | 0.9700 | C24—C25 | 1.371 (3) |
| C9—C10 | 1.493 (3) | C24—H24 | 0.9300 |
| C9—H9A | 0.9700 | C25—H25 | 0.9300 |
| C9—H9B | 0.9700 | C26—H26A | 0.9700 |
| C10—H10A | 0.9700 | C26—H26B | 0.9700 |
| C10—H10B | 0.9700 | | |

| | | | |
|---|---|---|---|
| C1—O1—C26 | 117.71 (14) | O5—C12—C11 | 107.74 (14) |
| C6—O2—C7 | 117.76 (14) | O5—C12—H12A | 110.2 |
| C9—O3—C8 | 114.27 (16) | C11—C12—H12A | 110.2 |
| C10—O4—C11 | 115.74 (15) | O5—C12—H12B | 110.2 |
| C13—O5—C12 | 117.39 (13) | C11—C12—H12B | 110.2 |
| C18—O6—C19 | 118.25 (14) | H12A—C12—H12B | 108.5 |
| O1—C1—C2 | 125.65 (18) | O5—C13—C14 | 125.71 (17) |
| O1—C1—C6 | 114.90 (15) | O5—C13—C18 | 115.11 (15) |
| C2—C1—C6 | 119.44 (18) | C14—C13—C18 | 119.17 (17) |
| C1—C2—C3 | 120.2 (2) | C13—C14—C15 | 120.5 (2) |
| C1—C2—H2 | 119.9 | C13—C14—H14 | 119.7 |
| C3—C2—H2 | 119.9 | C15—C14—H14 | 119.7 |
| C4—C3—C2 | 120.5 (2) | C16—C15—C14 | 120.18 (19) |
| C4—C3—H3 | 119.8 | C16—C15—H15 | 119.9 |
| C2—C3—H3 | 119.8 | C14—C15—H15 | 119.9 |
| C3—C4—C5 | 120.0 (2) | C15—C16—C17 | 119.98 (19) |
| C3—C4—H4 | 120.0 | C15—C16—H16 | 120.0 |
| C5—C4—H4 | 120.0 | C17—C16—H16 | 120.0 |
| C6—C5—C4 | 120.5 (2) | C16—C17—C18 | 120.7 (2) |
| C6—C5—H5 | 119.7 | C16—C17—H17 | 119.6 |
| C4—C5—H5 | 119.7 | C18—C17—H17 | 119.6 |
| O2—C6—C5 | 125.33 (18) | O6—C18—C17 | 125.69 (17) |
| O2—C6—C1 | 115.35 (16) | O6—C18—C13 | 114.89 (15) |
| C5—C6—C1 | 119.32 (17) | C17—C18—C13 | 119.41 (17) |
| O2—C7—C8 | 108.22 (16) | O6—C19—C20 | 107.65 (15) |
| O2—C7—H7A | 110.1 | O6—C19—H19A | 110.2 |
| C8—C7—H7A | 110.1 | C20—C19—H19A | 110.2 |
| O2—C7—H7B | 110.1 | O6—C19—H19B | 110.2 |
| C8—C7—H7B | 110.1 | C20—C19—H19B | 110.2 |

| | | | |
|---|---|---|---|
| H7A—C7—H7B | 108.4 | H19A—C19—H19B | 108.5 |
| O3—C8—C7 | 114.40 (16) | C21—C20—C25 | 117.85 (18) |
| O3—C8—H8A | 108.7 | C21—C20—C19 | 120.50 (17) |
| C7—C8—H8A | 108.7 | C25—C20—C19 | 121.65 (17) |
| O3—C8—H8B | 108.7 | C20—C21—C22 | 120.86 (18) |
| C7—C8—H8B | 108.7 | C20—C21—H21 | 119.6 |
| H8A—C8—H8B | 107.6 | C22—C21—H21 | 119.6 |
| O3—C9—C10 | 109.36 (16) | C23—C22—C21 | 121.17 (18) |
| O3—C9—H9A | 109.8 | C23—C22—H22 | 119.4 |
| C10—C9—H9A | 109.8 | C21—C22—H22 | 119.4 |
| O3—C9—H9B | 109.8 | C22—C23—C24 | 117.69 (18) |
| C10—C9—H9B | 109.8 | C22—C23—C26 | 121.89 (18) |
| H9A—C9—H9B | 108.2 | C24—C23—C26 | 120.41 (18) |
| O4—C10—C9 | 108.74 (17) | C25—C24—C23 | 121.26 (19) |
| O4—C10—H10A | 109.9 | C25—C24—H24 | 119.4 |
| C9—C10—H10A | 109.9 | C23—C24—H24 | 119.4 |
| O4—C10—H10B | 109.9 | C24—C25—C20 | 121.07 (19) |
| C9—C10—H10B | 109.9 | C24—C25—H25 | 119.5 |
| H10A—C10—H10B | 108.3 | C20—C25—H25 | 119.5 |
| O4—C11—C12 | 114.25 (17) | O1—C26—C23 | 107.49 (15) |
| O4—C11—H11A | 108.7 | O1—C26—H26A | 110.2 |
| C12—C11—H11A | 108.7 | C23—C26—H26A | 110.2 |
| O4—C11—H11B | 108.7 | O1—C26—H26B | 110.2 |
| C12—C11—H11B | 108.7 | C23—C26—H26B | 110.2 |
| H11A—C11—H11B | 107.6 | H26A—C26—H26B | 108.5 |

| | | | |
|---|---|---|---|
| C26—O1—C1—C2 | 8.2 (3) | C13—C14—C15—C16 | 0.1 (3) |
| C26—O1—C1—C6 | −170.39 (16) | C14—C15—C16—C17 | 0.3 (3) |
| O1—C1—C2—C3 | −178.12 (17) | C15—C16—C17—C18 | −0.7 (3) |
| C6—C1—C2—C3 | 0.5 (3) | C19—O6—C18—C17 | 17.7 (3) |
| C1—C2—C3—C4 | −1.2 (3) | C19—O6—C18—C13 | −161.32 (17) |
| C2—C3—C4—C5 | 0.8 (3) | C16—C17—C18—O6 | −178.25 (18) |
| C3—C4—C5—C6 | 0.2 (3) | C16—C17—C18—C13 | 0.7 (3) |
| C7—O2—C6—C5 | −20.6 (3) | O5—C13—C18—O6 | −0.4 (2) |
| C7—O2—C6—C1 | 159.34 (16) | C14—C13—C18—O6 | 178.72 (16) |
| C4—C5—C6—O2 | 179.09 (18) | O5—C13—C18—C17 | −179.49 (16) |
| C4—C5—C6—C1 | −0.9 (3) | C14—C13—C18—C17 | −0.3 (3) |
| O1—C1—C6—O2 | −0.7 (2) | C18—O6—C19—C20 | 163.38 (15) |
| C2—C1—C6—O2 | −179.42 (16) | O6—C19—C20—C21 | −151.26 (17) |
| O1—C1—C6—C5 | 179.28 (16) | O6—C19—C20—C25 | 28.5 (2) |
| C2—C1—C6—C5 | 0.5 (3) | C25—C20—C21—C22 | −1.9 (3) |
| C6—O2—C7—C8 | −177.71 (15) | C19—C20—C21—C22 | 177.84 (17) |
| C9—O3—C8—C7 | −95.6 (2) | C20—C21—C22—C23 | −0.7 (3) |
| O2—C7—C8—O3 | 64.9 (2) | C21—C22—C23—C24 | 3.0 (3) |
| C8—O3—C9—C10 | −162.52 (16) | C21—C22—C23—C26 | −176.78 (18) |
| C11—O4—C10—C9 | −156.07 (16) | C22—C23—C24—C25 | −2.6 (3) |
| O3—C9—C10—O4 | 69.2 (2) | C26—C23—C24—C25 | 177.11 (19) |
| C10—O4—C11—C12 | −93.9 (2) | C23—C24—C25—C20 | 0.0 (3) |

| | | | |
|---|---|---|---|
| C13—O5—C12—C11 | −175.06 (15) | C21—C20—C25—C24 | 2.2 (3) |
| O4—C11—C12—O5 | 68.5 (2) | C19—C20—C25—C24 | −177.50 (19) |
| C12—O5—C13—C14 | −13.4 (3) | C1—O1—C26—C23 | 166.99 (15) |
| C12—O5—C13—C18 | 165.73 (15) | C22—C23—C26—O1 | 118.0 (2) |
| O5—C13—C14—C15 | 179.01 (17) | C24—C23—C26—O1 | −61.7 (2) |
| C18—C13—C14—C15 | −0.1 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C9—H9*A*···O2 | 0.97 | 2.62 | 3.114 (2) | 112 |
| C10—H10*A*···O5 | 0.97 | 2.70 | 3.117 (2) | 106 |
| C2—H2···O1$^{i}$ | 0.93 | 2.76 | 3.471 (2) | 134 |
| C7—H7*B*···O5$^{ii}$ | 0.97 | 2.72 | 3.607 (2) | 153 |
| C11—H11*B*···O1$^{ii}$ | 0.97 | 2.82 | 3.501 (3) | 128 |
| C11—H11*B*···O2$^{ii}$ | 0.97 | 2.90 | 3.798 (3) | 154 |
| C12—H12*B*···O4$^{iii}$ | 0.97 | 2.42 | 3.294 (3) | 149 |
| C25—H25···O3$^{ii}$ | 0.93 | 2.71 | 3.433 (2) | 135 |
| C12—H12*A*···*Cg*1$^{ii}$ | 0.97 | 2.76 | 3.47 | 138 |
| C21—H21···*Cg*2$^{i}$ | 0.93 | 2.97 | 3.47 | 115 |
| C26—H26*A*···*Cg*3$^{i}$ | 0.97 | 3.06 | 3.83 | 138 |

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