# 8-(Biphenyl-4-yl)-8-hydroxypentacyclo-[5.4.0.0$^{2,6}$.0$^{3,10}$.0$^{5,9}$]undecan-11-one ethylene ketal

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The title compound, $C_{25}H_{24}O_3$, synthesized as a potential chiral catalyst, exhibits a range of C—C bond lengths in the pentacycloundecane cage between 1.5144 (18) and 1.5856 (16) Å. The two benzene rings are not planar with respect to each other, but rather are twisted at a torsion angle of 34.67 (17)°. The molecule has an intramolecular O—H···O interaction and participates in two C—H···O intermolecular interactions to form a one-dimensional chain.

## Related literature

For related literature, see: Flippen-Anderson *et al.* (1991); Linden *et al.* (2005); Kruger *et al.* (2005, 2006); Boyle *et al.* (2007).
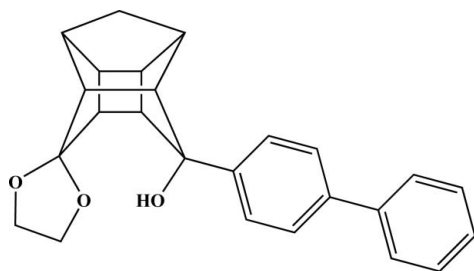


## Experimental

### Crystal data

| | |
|---|---|
| $C_{25}H_{24}O_3$ | $c$ = 10.3650 (2) Å |
| $M_r$ = 372.44 | $\beta$ = 90.5760 (10)° |
| Monoclinic, $P2_1/c$ | $V$ = 1804.70 (6) Å$^3$ |
| $a$ = 10.2527 (2) Å | $Z$ = 4 |
| $b$ = 16.9832 (3) Å | Mo $K\alpha$ radiation |

| | |
|---|---|
| $\mu$ = 0.09 mm$^{-1}$ | 0.47 × 0.45 × 0.37 mm |
| $T$ = 173 (2) K | |

### Data collection

| | |
|---|---|
| Bruker APEXII CCD area-detector diffractometer | 3904 independent reflections |
| Absorption correction: none | 3358 reflections with $I > 2\sigma(I)$ |
| 27563 measured reflections | $R_{int}$ = 0.037 |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.040 | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2)$ = 0.109 | |
| $S$ = 1.04 | $\Delta\rho_{max}$ = 0.31 e Å$^{-3}$ |
| 3904 reflections | $\Delta\rho_{min}$ = −0.24 e Å$^{-3}$ |
| 257 parameters | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3H···O2 | 0.877 (18) | 1.769 (19) | 2.6153 (12) | 161.6 (18) |
| C12—H12B···O1$^i$ | 0.99 | 2.54 | 3.2455 (18) | 128 |
| C24—H24···O3$^{ii}$ | 0.95 | 2.60 | 3.4955 (16) | 158 |

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 1999); program(s) used to refine structure: *SHELXTL*; molecular graphics: *Mercury* (Macrae *et al.*, 2006) and *WinGX* (Farrugia, 1999); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG2055).

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# supporting information

## 8-(Biphenyl-4-yl)-8-hydroxypentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecan-11-one ethylene ketal

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### S1. Comment

The molecule was synthesized as part of an ongoing study into the synthesis of chiral cage ligands for applications in asymmetric catalysis. The title molecule, which exists as a racemic mixture, has the potential to be a very unique ligand once it is resolved into an enantiopure compound.

A number of publications have focused on the molecular geometries of PCU cage derivatives as well as the bond lengths which deviate from the normal value of 1.54 Å (Flippen-Anderson *et al.*, 1991; Linden *et al.*, 2005; Kruger *et al.*, 2005, 2006, Boyle *et al.*, 2007). Certain bonds in the cage skeleton are longer (*e.g.* C9—C10, 1.5922 Å) while others are significantly shorter (*e.g.* C1—C11, 1.5106 Å). The molecule **(I)** consists of a large hydrophobic hydrocarbon skeleton as well as a hydrophilic ketal group and hydroxyl moiety. The two aromatic rings attached to C8 are not planar with respect to each other, but rather twisted at a torsion angle of 34.67 (17)° as expected due to steric factors. Fig. 1 shows the molecular structure and the numbering scheme employed.

The molecule exhibits intramolecular hydrogen bonding (Fig. 2) between the hydroxyl group and the ketal group (O3—H3H⋯O2). There is no intermolecular hydrogen bonding present in the structure, however a complex network of weak Van der Waals interactions between neighbouring molecules (Fig. 3) results in a layered packing effect with alternating hydrophilic and hydrophobic layers made up of the hydrophobic cage molecules and aromatic moeties, and the hydrophilic hydroxyl and ketal groups, respectively (Fig. 4).

### S2. Experimental

A solution of 4-bromobiphenyl in dry THF (3 mol eq) was cooled to -78°C using a dry-ice-acetone bath. Butyllithium solution (15% in hexane, 1.2 mole equivalents relative to bromobiphenyl) was added and the solution stirred for 10 minutes. A solution of pentacyclo-[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]-undecane-8,11-dione-mono-ethylene ketal (1 mol eq-up to 1 g scale) in dry THF was added and the solution stirred at -78°C for 1 h then at room temperature overnight. The reaction was quenched by adding water dropwise. The solvent was removed *in vacuo*. The product was isolated using column chromatography (EtOAc/Hexane, 10:90). The oily product crystallized on standing at room temperature overnight.

### S3. Refinement

Non-hydrogen atoms were first refined isotropically followed by anisotropic refinement by full matrix least-squares calculations based on $F^2$ using *SHELXTL*. Hydrogen atoms were first located in the difference map then positioned geometrically and allowed to ride on their respective parent atoms.
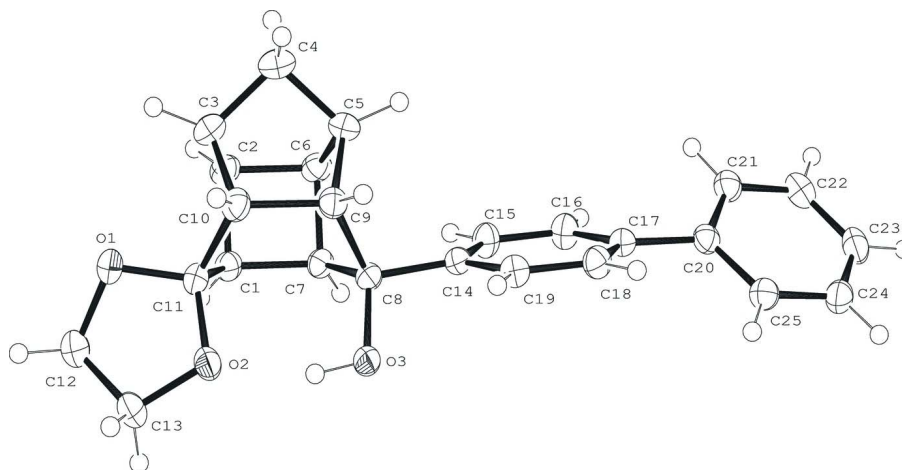
**Figure 1**

The asymmetric unit showing ellipsoids at the 50% probability level and the numbering scheme employed.
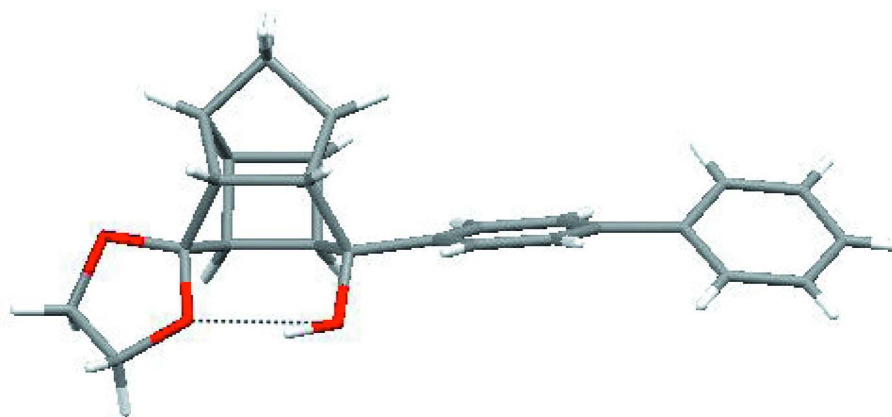


**Figure 2**

Diagram of the intermolecular hydrogen bonding.
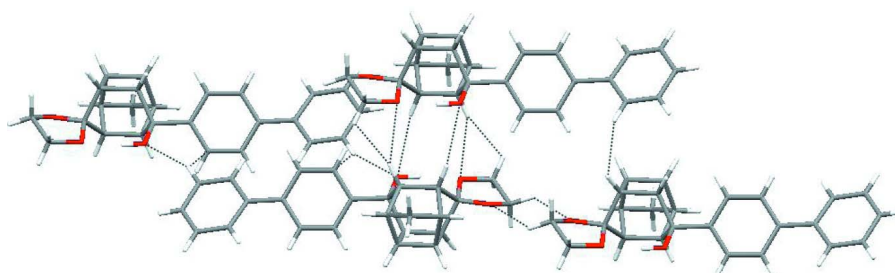


**Figure 3**

Diagram of the short intermolecular contacts.
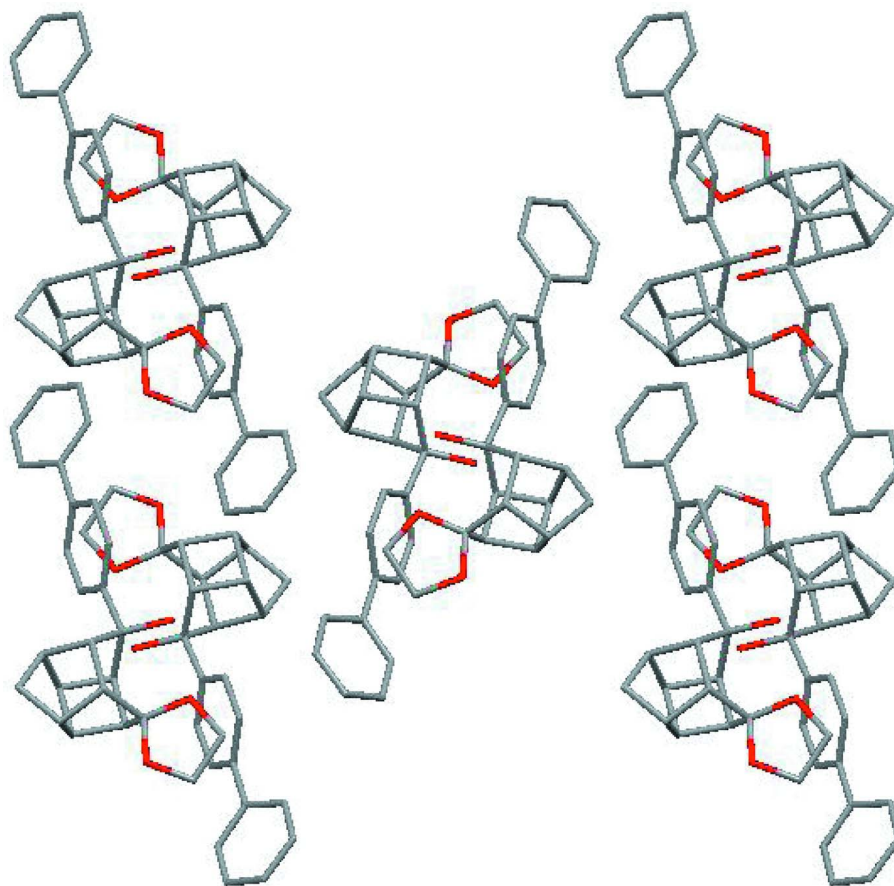
**Figure 4**

Depiction of the molecular packing. Hydrogen atoms have been ommited for clarity

**8-(Biphenyl-4-yl)-8-hydroxypentacyclo[5.4.0.0$^{2,6}$.0$^{3,10}$.0$^{5,9}$]undecan-11-one ethylene ketal**

*Crystal data*

| | |
|---|---|
| $C_{25}H_{24}O_3$ | $F(000) = 792$ |
| $M_r = 372.44$ | $D_x = 1.371$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2ybc | Cell parameters from 6796 reflections |
| $a = 10.2527$ (2) Å | $\theta = 2.3–28.5°$ |
| $b = 16.9832$ (3) Å | $\mu = 0.09$ mm$^{-1}$ |
| $c = 10.3650$ (2) Å | $T = 173$ K |
| $\beta = 90.576$ (1)° | Block, colourless |
| $V = 1804.70$ (6) Å$^3$ | $0.47 \times 0.45 \times 0.37$ mm |
| $Z = 4$ | |

*Data collection*

| | |
|---|---|
| Bruker APEXII CCD area-detector diffractometer | 3358 reflections with $I > 2\sigma(I)$ |
| | $R_{int} = 0.037$ |
| Radiation source: fine-focus sealed tube | $\theta_{max} = 27.0°$, $\theta_{min} = 2.0°$ |
| Graphite monochromator | $h = -13 \rightarrow 13$ |
| $\varphi$ and $\omega$ scans | $k = -21 \rightarrow 21$ |
| 27563 measured reflections | $l = -13 \rightarrow 13$ |
| 3904 independent reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier |
| Least-squares matrix: full | map |
| $R[F^2 > 2\sigma(F^2)] = 0.040$ | Hydrogen site location: inferred from |
| $wR(F^2) = 0.109$ | neighbouring sites |
| $S = 1.04$ | H atoms treated by a mixture of independent |
| 3904 reflections | and constrained refinement |
| 257 parameters | $w = 1/[\sigma^2(F_o^2) + (0.0579P)^2 + 0.5851P]$ |
| 0 restraints | where $P = (F_o^2 + 2F_c^2)/3$ |
| Primary atom site location: structure-invariant | $(\Delta/\sigma)_{max} < 0.001$ |
| direct methods | $\Delta\rho_{max} = 0.31$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -0.24$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| H3H | 0.2134 (18) | −0.0511 (11) | 0.4718 (18) | 0.045 (5)* |
| C1 | 0.27294 (12) | 0.04517 (7) | 0.25422 (12) | 0.0243 (3) |
| H1 | 0.3241 | 0.0168 | 0.1870 | 0.029* |
| C2 | 0.25040 (13) | 0.13435 (8) | 0.22877 (13) | 0.0286 (3) |
| H2 | 0.2840 | 0.1566 | 0.1462 | 0.034* |
| C3 | 0.10706 (13) | 0.15304 (8) | 0.26439 (13) | 0.0316 (3) |
| H3 | 0.0462 | 0.1613 | 0.1896 | 0.038* |
| C4 | 0.12017 (15) | 0.22221 (8) | 0.35620 (15) | 0.0365 (3) |
| H4B | 0.1570 | 0.2695 | 0.3144 | 0.044* |
| H4A | 0.0371 | 0.2355 | 0.3989 | 0.044* |
| C5 | 0.21666 (13) | 0.18202 (7) | 0.44640 (13) | 0.0288 (3) |
| H5 | 0.2460 | 0.2142 | 0.5221 | 0.035* |
| C6 | 0.32659 (12) | 0.15486 (7) | 0.35544 (12) | 0.0267 (3) |
| H6 | 0.4050 | 0.1895 | 0.3480 | 0.032* |
| C7 | 0.35006 (11) | 0.06574 (7) | 0.38116 (11) | 0.0224 (2) |
| H7 | 0.4436 | 0.0491 | 0.3825 | 0.027* |
| C8 | 0.27605 (11) | 0.05050 (7) | 0.50638 (11) | 0.0208 (2) |
| C9 | 0.15361 (11) | 0.10184 (7) | 0.48020 (12) | 0.0242 (3) |
| H9 | 0.0956 | 0.1053 | 0.5570 | 0.029* |
| C10 | 0.07520 (12) | 0.08160 (8) | 0.35180 (12) | 0.0268 (3) |
| H10 | −0.0204 | 0.0747 | 0.3661 | 0.032* |
| C11 | 0.13428 (12) | 0.01565 (7) | 0.27329 (12) | 0.0252 (3) |
| C12 | 0.08855 (16) | −0.06659 (9) | 0.10563 (14) | 0.0370 (3) |
| H12A | 0.1730 | −0.0701 | 0.0605 | 0.044* |

| | | | |
|---|---|---|---|
| H12B | 0.0177 | −0.0832 | 0.0460 | 0.044* |
| C13 | 0.08947 (14) | −0.11551 (8) | 0.22633 (13) | 0.0321 (3) |
| H13A | 0.0026 | −0.1390 | 0.2413 | 0.038* |
| H13B | 0.1547 | −0.1583 | 0.2207 | 0.038* |
| C14 | 0.34966 (11) | 0.08247 (7) | 0.62415 (11) | 0.0215 (2) |
| C15 | 0.47065 (12) | 0.11841 (8) | 0.61578 (12) | 0.0274 (3) |
| H15 | 0.5085 | 0.1264 | 0.5335 | 0.033* |
| C16 | 0.53703 (12) | 0.14274 (8) | 0.72545 (12) | 0.0284 (3) |
| H16 | 0.6196 | 0.1675 | 0.7168 | 0.034* |
| C17 | 0.48648 (12) | 0.13201 (7) | 0.84792 (11) | 0.0234 (3) |
| C18 | 0.36431 (12) | 0.09680 (7) | 0.85637 (12) | 0.0254 (3) |
| H18 | 0.3264 | 0.0891 | 0.9387 | 0.031* |
| C19 | 0.29713 (12) | 0.07281 (7) | 0.74651 (12) | 0.0245 (3) |
| H19 | 0.2136 | 0.0493 | 0.7549 | 0.029* |
| C20 | 0.56860 (11) | 0.15326 (7) | 0.96176 (12) | 0.0237 (3) |
| C21 | 0.65359 (12) | 0.21726 (8) | 0.95569 (12) | 0.0276 (3) |
| H21 | 0.6496 | 0.2517 | 0.8835 | 0.033* |
| C22 | 0.74373 (13) | 0.23134 (8) | 1.05342 (13) | 0.0316 (3) |
| H22 | 0.8027 | 0.2742 | 1.0467 | 0.038* |
| C23 | 0.74797 (13) | 0.18301 (8) | 1.16083 (13) | 0.0319 (3) |
| H23 | 0.8111 | 0.1919 | 1.2269 | 0.038* |
| C24 | 0.66019 (13) | 0.12197 (8) | 1.17160 (13) | 0.0315 (3) |
| H24 | 0.6603 | 0.0902 | 1.2470 | 0.038* |
| C25 | 0.57148 (13) | 0.10685 (8) | 1.07246 (12) | 0.0280 (3) |
| H25 | 0.5120 | 0.0643 | 1.0803 | 0.034* |
| O1 | 0.06660 (10) | 0.01105 (6) | 0.15323 (9) | 0.0329 (2) |
| O2 | 0.12307 (9) | −0.06169 (5) | 0.32656 (8) | 0.0302 (2) |
| O3 | 0.25380 (9) | −0.02990 (5) | 0.53817 (8) | 0.0260 (2) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0280 (6) | 0.0256 (6) | 0.0193 (6) | 0.0020 (5) | 0.0011 (5) | 0.0009 (5) |
| C2 | 0.0352 (7) | 0.0264 (6) | 0.0242 (6) | 0.0020 (5) | 0.0004 (5) | 0.0051 (5) |
| C3 | 0.0322 (7) | 0.0296 (7) | 0.0328 (7) | 0.0073 (5) | −0.0071 (5) | 0.0029 (5) |
| C4 | 0.0398 (7) | 0.0280 (7) | 0.0417 (8) | 0.0105 (6) | −0.0041 (6) | 0.0013 (6) |
| C5 | 0.0344 (7) | 0.0218 (6) | 0.0302 (7) | 0.0045 (5) | −0.0013 (5) | −0.0027 (5) |
| C6 | 0.0276 (6) | 0.0238 (6) | 0.0288 (7) | −0.0019 (5) | −0.0007 (5) | 0.0048 (5) |
| C7 | 0.0221 (5) | 0.0246 (6) | 0.0205 (6) | 0.0005 (4) | 0.0006 (4) | 0.0010 (4) |
| C8 | 0.0219 (5) | 0.0207 (5) | 0.0198 (6) | −0.0003 (4) | −0.0001 (4) | 0.0011 (4) |
| C9 | 0.0225 (6) | 0.0268 (6) | 0.0232 (6) | 0.0036 (5) | −0.0002 (5) | −0.0027 (5) |
| C10 | 0.0227 (6) | 0.0309 (6) | 0.0266 (6) | 0.0032 (5) | −0.0036 (5) | −0.0017 (5) |
| C11 | 0.0292 (6) | 0.0264 (6) | 0.0200 (6) | 0.0001 (5) | −0.0053 (5) | 0.0012 (5) |
| C12 | 0.0484 (8) | 0.0357 (7) | 0.0268 (7) | 0.0029 (6) | −0.0085 (6) | −0.0061 (6) |
| C13 | 0.0376 (7) | 0.0304 (7) | 0.0281 (7) | −0.0008 (5) | −0.0038 (5) | −0.0082 (5) |
| C14 | 0.0232 (5) | 0.0208 (5) | 0.0207 (6) | 0.0020 (4) | −0.0011 (4) | 0.0002 (4) |
| C15 | 0.0274 (6) | 0.0339 (7) | 0.0209 (6) | −0.0046 (5) | 0.0017 (5) | 0.0014 (5) |
| C16 | 0.0255 (6) | 0.0347 (7) | 0.0248 (6) | −0.0071 (5) | −0.0006 (5) | 0.0010 (5) |

| C17 | 0.0262 (6) | 0.0219 (6) | 0.0221 (6) | 0.0020 (4) | −0.0015 (5) | −0.0004 (4) |
| C18 | 0.0272 (6) | 0.0288 (6) | 0.0204 (6) | 0.0007 (5) | 0.0037 (5) | −0.0001 (5) |
| C19 | 0.0216 (5) | 0.0270 (6) | 0.0250 (6) | −0.0011 (4) | 0.0013 (5) | −0.0003 (5) |
| C20 | 0.0240 (6) | 0.0252 (6) | 0.0220 (6) | 0.0030 (4) | 0.0003 (5) | −0.0030 (5) |
| C21 | 0.0315 (6) | 0.0279 (6) | 0.0234 (6) | −0.0002 (5) | 0.0013 (5) | −0.0018 (5) |
| C22 | 0.0300 (6) | 0.0331 (7) | 0.0317 (7) | −0.0038 (5) | 0.0017 (5) | −0.0093 (5) |
| C23 | 0.0294 (6) | 0.0396 (7) | 0.0266 (7) | 0.0066 (5) | −0.0054 (5) | −0.0118 (5) |
| C24 | 0.0380 (7) | 0.0339 (7) | 0.0224 (6) | 0.0077 (6) | −0.0024 (5) | −0.0012 (5) |
| C25 | 0.0311 (6) | 0.0277 (6) | 0.0252 (6) | 0.0002 (5) | 0.0006 (5) | −0.0002 (5) |
| O1 | 0.0400 (5) | 0.0333 (5) | 0.0252 (5) | 0.0037 (4) | −0.0122 (4) | −0.0026 (4) |
| O2 | 0.0414 (5) | 0.0256 (5) | 0.0233 (5) | −0.0076 (4) | −0.0066 (4) | −0.0006 (4) |
| O3 | 0.0343 (5) | 0.0222 (4) | 0.0214 (4) | −0.0040 (3) | −0.0041 (4) | 0.0024 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C11 | 1.5222 (17) | C12—C13 | 1.502 (2) |
| C1—C2 | 1.5542 (17) | C12—H12A | 0.9900 |
| C1—C7 | 1.5677 (16) | C12—H12B | 0.9900 |
| C1—H1 | 1.0000 | C13—O2 | 1.4234 (15) |
| C2—C3 | 1.5517 (18) | C13—H13A | 0.9900 |
| C2—C6 | 1.5603 (17) | C13—H13B | 0.9900 |
| C2—H2 | 1.0000 | C14—C15 | 1.3860 (17) |
| C3—C4 | 1.5169 (19) | C14—C19 | 1.3925 (17) |
| C3—C10 | 1.5510 (18) | C15—C16 | 1.3823 (17) |
| C3—H3 | 1.0000 | C15—H15 | 0.9500 |
| C4—C5 | 1.5166 (18) | C16—C17 | 1.3879 (18) |
| C4—H4B | 0.9900 | C16—H16 | 0.9500 |
| C4—H4A | 0.9900 | C17—C18 | 1.3917 (17) |
| C5—C6 | 1.5470 (18) | C17—C20 | 1.4871 (16) |
| C5—C9 | 1.5490 (18) | C18—C19 | 1.3861 (17) |
| C5—H5 | 1.0000 | C18—H18 | 0.9500 |
| C6—C7 | 1.5551 (16) | C19—H19 | 0.9500 |
| C6—H6 | 1.0000 | C20—C25 | 1.3921 (18) |
| C7—C8 | 1.5321 (16) | C20—C21 | 1.3949 (18) |
| C7—H7 | 1.0000 | C21—C22 | 1.3851 (18) |
| C8—O3 | 1.4236 (14) | C21—H21 | 0.9500 |
| C8—C14 | 1.5284 (15) | C22—C23 | 1.383 (2) |
| C8—C9 | 1.5502 (16) | C22—H22 | 0.9500 |
| C9—C10 | 1.5856 (16) | C23—C24 | 1.378 (2) |
| C9—H9 | 1.0000 | C23—H23 | 0.9500 |
| C10—C11 | 1.5144 (17) | C24—C25 | 1.3893 (18) |
| C10—H10 | 1.0000 | C24—H24 | 0.9500 |
| C11—O1 | 1.4209 (14) | C25—H25 | 0.9500 |
| C11—O2 | 1.4299 (15) | O3—H3H | 0.876 (19) |
| C12—O1 | 1.4265 (17) | | |
| | | | |
| C11—C1—C2 | 101.86 (10) | C9—C10—H10 | 113.0 |
| C11—C1—C7 | 115.36 (10) | O1—C11—O2 | 104.31 (9) |

| | | | |
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| C2—C1—C7 | 89.89 (9) | O1—C11—C10 | 108.47 (10) |
| C11—C1—H1 | 115.4 | O2—C11—C10 | 115.96 (10) |
| C2—C1—H1 | 115.4 | O1—C11—C1 | 110.66 (10) |
| C7—C1—H1 | 115.4 | O2—C11—C1 | 115.56 (10) |
| C3—C2—C1 | 107.37 (10) | C10—C11—C1 | 101.85 (10) |
| C3—C2—C6 | 102.79 (10) | O1—C12—C13 | 102.88 (11) |
| C1—C2—C6 | 90.14 (9) | O1—C12—H12A | 111.2 |
| C3—C2—H2 | 117.5 | C13—C12—H12A | 111.2 |
| C1—C2—H2 | 117.5 | O1—C12—H12B | 111.2 |
| C6—C2—H2 | 117.5 | C13—C12—H12B | 111.2 |
| C4—C3—C10 | 104.90 (11) | H12A—C12—H12B | 109.1 |
| C4—C3—C2 | 103.27 (11) | O2—C13—C12 | 104.61 (11) |
| C10—C3—C2 | 100.63 (10) | O2—C13—H13A | 110.8 |
| C4—C3—H3 | 115.4 | C12—C13—H13A | 110.8 |
| C10—C3—H3 | 115.4 | O2—C13—H13B | 110.8 |
| C2—C3—H3 | 115.4 | C12—C13—H13B | 110.8 |
| C5—C4—C3 | 95.24 (10) | H13A—C13—H13B | 108.9 |
| C5—C4—H4B | 112.7 | C15—C14—C19 | 117.61 (11) |
| C3—C4—H4B | 112.7 | C15—C14—C8 | 122.78 (11) |
| C5—C4—H4A | 112.7 | C19—C14—C8 | 119.55 (10) |
| C3—C4—H4A | 112.7 | C16—C15—C14 | 120.91 (12) |
| H4B—C4—H4A | 110.2 | C16—C15—H15 | 119.5 |
| C4—C5—C6 | 103.49 (11) | C14—C15—H15 | 119.5 |
| C4—C5—C9 | 105.31 (11) | C15—C16—C17 | 121.85 (12) |
| C6—C5—C9 | 100.60 (9) | C15—C16—H16 | 119.1 |
| C4—C5—H5 | 115.2 | C17—C16—H16 | 119.1 |
| C6—C5—H5 | 115.2 | C16—C17—C18 | 117.30 (11) |
| C9—C5—H5 | 115.2 | C16—C17—C20 | 118.70 (11) |
| C5—C6—C7 | 107.35 (10) | C18—C17—C20 | 123.85 (11) |
| C5—C6—C2 | 102.58 (10) | C19—C18—C17 | 120.98 (11) |
| C7—C6—C2 | 90.13 (9) | C19—C18—H18 | 119.5 |
| C5—C6—H6 | 117.6 | C17—C18—H18 | 119.5 |
| C7—C6—H6 | 117.6 | C18—C19—C14 | 121.34 (11) |
| C2—C6—H6 | 117.6 | C18—C19—H19 | 119.3 |
| C8—C7—C6 | 103.45 (9) | C14—C19—H19 | 119.3 |
| C8—C7—C1 | 115.05 (9) | C25—C20—C21 | 118.05 (11) |
| C6—C7—C1 | 89.83 (9) | C25—C20—C17 | 121.59 (11) |
| C8—C7—H7 | 115.1 | C21—C20—C17 | 120.14 (11) |
| C6—C7—H7 | 115.1 | C22—C21—C20 | 120.93 (12) |
| C1—C7—H7 | 115.1 | C22—C21—H21 | 119.5 |
| O3—C8—C14 | 103.59 (9) | C20—C21—H21 | 119.5 |
| O3—C8—C7 | 116.11 (10) | C23—C22—C21 | 120.09 (12) |
| C14—C8—C7 | 111.83 (9) | C23—C22—H22 | 120.0 |
| O3—C8—C9 | 116.65 (9) | C21—C22—H22 | 120.0 |
| C14—C8—C9 | 109.42 (9) | C24—C23—C22 | 119.75 (12) |
| C7—C8—C9 | 99.43 (9) | C24—C23—H23 | 120.1 |
| C5—C9—C8 | 101.24 (9) | C22—C23—H23 | 120.1 |
| C5—C9—C10 | 102.12 (10) | C23—C24—C25 | 120.16 (12) |

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| C8—C9—C10 | 115.33 (10) | C23—C24—H24 | 119.9 |
| C5—C9—H9 | 112.4 | C25—C24—H24 | 119.9 |
| C8—C9—H9 | 112.4 | C24—C25—C20 | 120.88 (12) |
| C10—C9—H9 | 112.4 | C24—C25—H25 | 119.6 |
| C11—C10—C3 | 100.19 (10) | C20—C25—H25 | 119.6 |
| C11—C10—C9 | 114.16 (10) | C11—O1—C12 | 106.04 (9) |
| C3—C10—C9 | 102.28 (10) | C13—O2—C11 | 109.14 (9) |
| C11—C10—H10 | 113.0 | C8—O3—H3H | 106.7 (12) |
| C3—C10—H10 | 113.0 | | |
| | | | |
| C11—C1—C2—C3 | 12.21 (12) | C8—C9—C10—C3 | −108.23 (11) |
| C7—C1—C2—C3 | −103.71 (10) | C3—C10—C11—O1 | −63.21 (12) |
| C11—C1—C2—C6 | 115.67 (10) | C9—C10—C11—O1 | −171.72 (10) |
| C7—C1—C2—C6 | −0.25 (9) | C3—C10—C11—O2 | 179.88 (10) |
| C1—C2—C3—C4 | 127.66 (11) | C9—C10—C11—O2 | 71.37 (14) |
| C6—C2—C3—C4 | 33.44 (12) | C3—C10—C11—C1 | 53.55 (11) |
| C1—C2—C3—C10 | 19.42 (12) | C9—C10—C11—C1 | −54.96 (13) |
| C6—C2—C3—C10 | −74.81 (11) | C2—C1—C11—O1 | 74.93 (12) |
| C10—C3—C4—C5 | 51.75 (12) | C7—C1—C11—O1 | 170.52 (10) |
| C2—C3—C4—C5 | −53.26 (12) | C2—C1—C11—O2 | −166.82 (10) |
| C3—C4—C5—C6 | 53.65 (12) | C7—C1—C11—O2 | −71.23 (13) |
| C3—C4—C5—C9 | −51.53 (13) | C2—C1—C11—C10 | −40.23 (11) |
| C4—C5—C6—C7 | −128.13 (11) | C7—C1—C11—C10 | 55.37 (12) |
| C9—C5—C6—C7 | −19.40 (12) | O1—C12—C13—O2 | −23.62 (14) |
| C4—C5—C6—C2 | −33.99 (12) | O3—C8—C14—C15 | 123.86 (12) |
| C9—C5—C6—C2 | 74.74 (11) | C7—C8—C14—C15 | −1.91 (16) |
| C3—C2—C6—C5 | 0.29 (12) | C9—C8—C14—C15 | −111.07 (13) |
| C1—C2—C6—C5 | −107.56 (10) | O3—C8—C14—C19 | −53.41 (13) |
| C3—C2—C6—C7 | 108.11 (10) | C7—C8—C14—C19 | −179.18 (10) |
| C1—C2—C6—C7 | 0.25 (9) | C9—C8—C14—C19 | 71.66 (13) |
| C5—C6—C7—C8 | −12.78 (12) | C19—C14—C15—C16 | 0.82 (19) |
| C2—C6—C7—C8 | −116.01 (9) | C8—C14—C15—C16 | −176.51 (12) |
| C5—C6—C7—C1 | 102.98 (10) | C14—C15—C16—C17 | 0.4 (2) |
| C2—C6—C7—C1 | −0.25 (9) | C15—C16—C17—C18 | −1.12 (19) |
| C11—C1—C7—C8 | 1.95 (15) | C15—C16—C17—C20 | 174.66 (12) |
| C2—C1—C7—C8 | 105.05 (11) | C16—C17—C18—C19 | 0.68 (18) |
| C11—C1—C7—C6 | −102.85 (11) | C20—C17—C18—C19 | −174.86 (11) |
| C2—C1—C7—C6 | 0.25 (9) | C17—C18—C19—C14 | 0.51 (19) |
| C6—C7—C8—O3 | 165.61 (9) | C15—C14—C19—C18 | −1.25 (18) |
| C1—C7—C8—O3 | 69.38 (13) | C8—C14—C19—C18 | 176.16 (11) |
| C6—C7—C8—C14 | −75.83 (11) | C16—C17—C20—C25 | −139.90 (13) |
| C1—C7—C8—C14 | −172.06 (9) | C18—C17—C20—C25 | 35.57 (18) |
| C6—C7—C8—C9 | 39.61 (11) | C16—C17—C20—C21 | 34.67 (17) |
| C1—C7—C8—C9 | −56.62 (12) | C18—C17—C20—C21 | −149.86 (12) |
| C4—C5—C9—C8 | 151.54 (10) | C25—C20—C21—C22 | 3.91 (18) |
| C6—C5—C9—C8 | 44.25 (11) | C17—C20—C21—C22 | −170.85 (11) |
| C4—C5—C9—C10 | 32.28 (12) | C20—C21—C22—C23 | −1.9 (2) |
| C6—C5—C9—C10 | −75.02 (11) | C21—C22—C23—C24 | −1.5 (2) |

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| O3—C8—C9—C5 | −178.53 (10) | C22—C23—C24—C25 | 2.8 (2) |
| C14—C8—C9—C5 | 64.36 (11) | C23—C24—C25—C20 | −0.7 (2) |
| C7—C8—C9—C5 | −52.91 (11) | C21—C20—C25—C24 | −2.62 (18) |
| O3—C8—C9—C10 | −69.21 (14) | C17—C20—C25—C24 | 172.06 (12) |
| C14—C8—C9—C10 | 173.68 (10) | O2—C11—O1—C12 | −34.19 (13) |
| C7—C8—C9—C10 | 56.41 (12) | C10—C11—O1—C12 | −158.35 (11) |
| C4—C3—C10—C11 | −150.89 (10) | C1—C11—O1—C12 | 90.71 (12) |
| C2—C3—C10—C11 | −43.93 (11) | C13—C12—O1—C11 | 35.81 (14) |
| C4—C3—C10—C9 | −33.20 (12) | C12—C13—O2—C11 | 3.28 (14) |
| C2—C3—C10—C9 | 73.76 (11) | O1—C11—O2—C13 | 18.51 (13) |
| C5—C9—C10—C11 | 107.79 (12) | C10—C11—O2—C13 | 137.71 (11) |
| C8—C9—C10—C11 | −1.01 (15) | C1—C11—O2—C13 | −103.21 (12) |
| C5—C9—C10—C3 | 0.57 (11) | | |

*Hydrogen-bond geometry (Å, º)*

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3$H$···O2 | 0.877 (18) | 1.769 (19) | 2.6153 (12) | 161.6 (18) |
| C12—H12$B$···O1[i] | 0.99 | 2.54 | 3.2455 (18) | 128 |
| C24—H24···O3[ii] | 0.95 | 2.60 | 3.4955 (16) | 158 |

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