# organic compounds

# Methyl 4-(benzyloxy)-3-methoxybenzoate

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In the title compound, $C_{16}H_{16}O_4$, the aromatic rings are almost normal to one another, making a dihedral angle of 85.81 (10)°. In the crystal, molecules are linked by C—H···O hydrogen bonds, forming chains propagating along the *b*-axis direction. There are also C—H···$\pi$ interactions present which link the chains, forming two-dimensional networks lying parallel to (102).

## Related literature

For details of the anticancer properties of the drug Cediranib {systematic name: 4-[(4-fluoro-2-methyl-1*H*-indol-5-yl)oxy]-6-methoxy-7-[3-(pyrrolidin-1-yl)propoxy]quinazoline}, for which the title compound is an important intermediate in the synthesis, see: Folkman (1996). For the synthetic procedure, see: Li & Zhang (2012). For bond-length data, see: Allen *et al.* (1987).
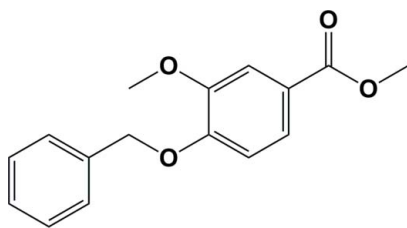
## Experimental

### Crystal data

| | |
|---|---|
| $C_{16}H_{16}O_4$ | $V$ = 1399.6 (3) Å$^3$ |
| $M_r$ = 272.29 | $Z$ = 4 |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation |
| $a$ = 5.2466 (7) Å | $\mu$ = 0.09 mm$^{-1}$ |
| $b$ = 17.973 (2) Å | $T$ = 293 K |
| $c$ = 14.8785 (18) Å | 0.12 × 0.12 × 0.10 mm |
| $\beta$ = 94.018 (3)° | |

### Data collection

| | |
|---|---|
| Enraf–Nonius CAD-4 diffractometer | 2454 independent reflections |
| Absorption correction: $\psi$ scan (North *et al.*, 1968) | 1666 reflections with $I > 2\sigma(I)$ |
| $T_{min}$ = 0.989, $T_{max}$ = 0.991 | $R_{int}$ = 0.029 |
| 7732 measured reflections | 3 standard reflections every 120 min intensity decay: 1% |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.045 | 182 parameters |
| $wR(F^2)$ = 0.138 | H-atom parameters constrained |
| $S$ = 0.91 | $\Delta\rho_{max}$ = 0.18 e Å$^{-3}$ |
| 2454 reflections | $\Delta\rho_{min}$ = −0.17 e Å$^{-3}$ |

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

*Cg* is the centroid of the C1–C6 ring

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C14—H14$B$···O3$^i$ | 0.96 | 2.53 | 3.379 (3) | 147 |
| C14—H14$A$···$Cg^{ii}$ | 0.96 | 2.75 | 3.519 (2) | 137 |

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x-1, -y-\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

## References

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# Methyl 4-(benzyloxy)-3-methoxybenzoate

## Kai Wang, ChaoFan Ju, Jian Xiao and Qiang Chen

### 1. Comment

The title compound is an important organic intermediate which has been used to synthesis the antineoplastic drug Cediranib. The drug has shown promising activity against diseases which include lung and breast cancer (Folkman, 1996).

The molecular structure of the title molecule is shown in Fig. 1. The bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. In the molecule the two aromatic rings (C1-C6) and (C8-C13) are almost normal to one another with a dihedral angle of 85.81 (10) °.

In the crystal, molecules are linked by C—H⋯O hydrogen bonds forming chains propagating along the b axis direction (Table 1 and Fig. 2). There are also C-H⋯$\pi$ interactions present (Table 1) linking the chains to form two-dimensional networks lying parallel to (102).

### 2. Experimental

The title compound was prepared according to the procedure reported by Li & Zhang (2012). A solution of 4-(benzyl-oxy)-3-methoxybenzoic acid (5 g, 19.36 mmol) was added slowly to a solution of methanol and concentrated sulfuric acid (2 ml). After being stirred for 12 h at reflux, saturated sodium bicarbonate solution was added to adjust the pH to 7. Dichloromethane was added, and the mixture was then filtered and the organic phase evaporated on a rotary evaporator and to obtain the title compound. Block-like colourless crystals were obtained by dissolving (0.5 g, 1.84 mmol) of the title compound in ethanol (25 ml) and evaporating the solvent slowly at room temperature for about 7 days.

### 3. Refinement

All the H atoms were positioned geometrically and constrained to ride on their parent atom: C—H = 0.93 - 0.96 Å with $U_{iso}(H) = 1.5U_{eq}(C\text{-methyl})$ and $= 1.2U_{eq}(C)$ for other H atoms.

**Computing details**

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software* (Enraf–Nonius, 1985); data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).
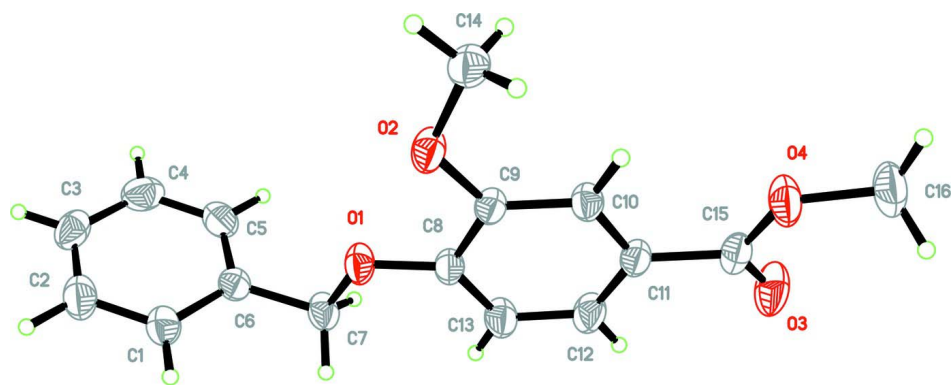
**Figure 1**

The molecular structure of the title molecule, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.
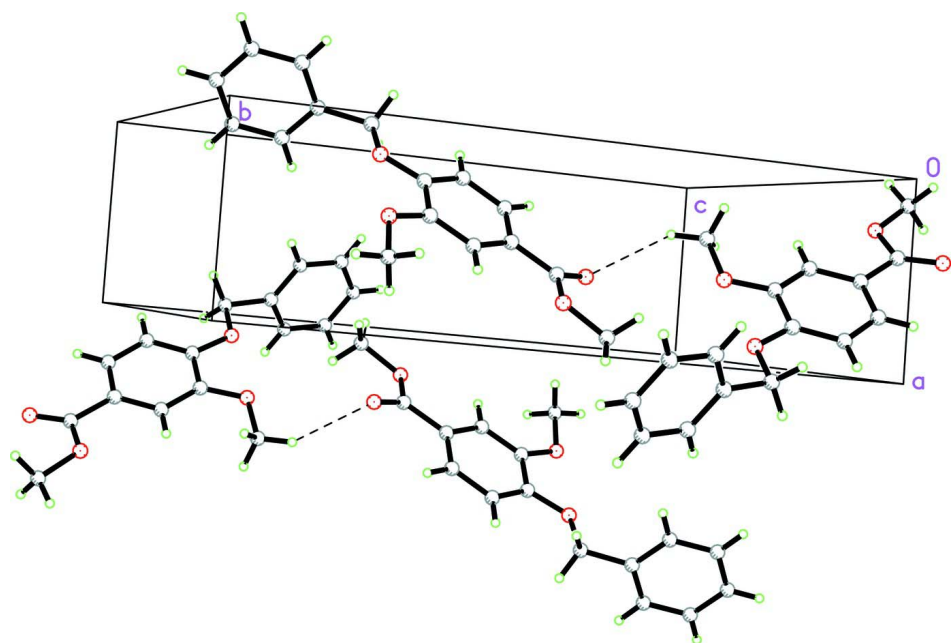


**Figure 2**

A view of the crystal packing of the title compound. The C—H···O hydrogen bonds are shown by dashed lines (see Table 1 for details; H atoms not involved in hydrogen bonding have been omitted for clarity).

**Methyl 4-(benzyloxy)-3-methoxybenzoate**

*Crystal data*

| | |
|---|---|
| $C_{16}H_{16}O_4$ | $V = 1399.6\ (3)$ Å$^3$ |
| $M_r = 272.29$ | $Z = 4$ |
| Monoclinic, $P2_1/c$ | $F(000) = 576$ |
| Hall symbol: -P 2ybc | $D_x = 1.292$ Mg m$^{-3}$ |
| $a = 5.2466\ (7)$ Å | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $b = 17.973\ (2)$ Å | Cell parameters from 1683 reflections |
| $c = 14.8785\ (18)$ Å | $\theta = 5.3–45.5°$ |
| $\beta = 94.018\ (3)°$ | $\mu = 0.09$ mm$^{-1}$ |

$T = 293$ K

Block, colourless

$0.12 \times 0.12 \times 0.10$ mm

*Data collection*

| | |
|---|---|
| Enraf–Nonius CAD-4 diffractometer | 2454 independent reflections |
| Radiation source: fine-focus sealed tube | 1666 reflections with $I > 2\sigma(I)$ |
| Graphite monochromator | $R_{int} = 0.029$ |
| $\omega/2\theta$ scans | $\theta_{max} = 25.0°$, $\theta_{min} = 2.3°$ |
| Absorption correction: $\psi$ scan (North *et al.*, 1968) | $h = -6 \rightarrow 6$ |
| | $k = -21 \rightarrow 14$ |
| | $l = -16 \rightarrow 17$ |
| $T_{min} = 0.989$, $T_{max} = 0.991$ | 3 standard reflections every 120 min |
| 7732 measured reflections | intensity decay: 1% |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.045$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.138$ | |
| $S = 0.91$ | H-atom parameters constrained |
| 2454 reflections | $w = 1/[\sigma^2(F_o^2) + (0.0874P)^2 + 0.101P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 182 parameters | |
| 0 restraints | $(\Delta/\sigma)_{max} = 0.005$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{max} = 0.18$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -0.17$ 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 | 0.8880 (3) | 0.30300 (7) | 0.59482 (9) | 0.0538 (4) |
| O2 | 0.5720 (3) | 0.29367 (7) | 0.71794 (9) | 0.0573 (4) |
| O3 | 0.3782 (4) | 0.61921 (8) | 0.64985 (12) | 0.0841 (5) |
| O4 | 0.2344 (3) | 0.55257 (8) | 0.76146 (10) | 0.0702 (5) |
| C1 | 1.3108 (4) | 0.18726 (11) | 0.53747 (14) | 0.0585 (6) |
| H1 | 1.4248 | 0.2124 | 0.5774 | 0.070* |
| C2 | 1.3529 (5) | 0.11390 (13) | 0.51813 (17) | 0.0714 (7) |
| H2 | 1.4940 | 0.0896 | 0.5457 | 0.086* |
| C3 | 1.1895 (5) | 0.07597 (12) | 0.45856 (17) | 0.0703 (7) |
| H3 | 1.2194 | 0.0263 | 0.4453 | 0.084* |
| C4 | 0.9838 (5) | 0.11185 (14) | 0.41926 (18) | 0.0789 (7) |
| H4 | 0.8709 | 0.0866 | 0.3790 | 0.095* |
| C5 | 0.9409 (4) | 0.18540 (14) | 0.43850 (16) | 0.0682 (6) |
| H5 | 0.8002 | 0.2095 | 0.4104 | 0.082* |

| C6 | 1.1025 (4) | 0.22385 (11) | 0.49856 (13) | 0.0475 (5) |
| C7 | 1.0484 (4) | 0.30341 (11) | 0.52037 (14) | 0.0552 (5) |
| H7A | 1.2064 | 0.3297 | 0.5366 | 0.066* |
| H7B | 0.9622 | 0.3279 | 0.4687 | 0.066* |
| C8 | 0.7756 (3) | 0.36807 (10) | 0.61691 (12) | 0.0456 (5) |
| C9 | 0.6001 (3) | 0.36283 (10) | 0.68347 (12) | 0.0430 (4) |
| C10 | 0.4714 (3) | 0.42556 (10) | 0.70828 (12) | 0.0463 (5) |
| H10 | 0.3552 | 0.4222 | 0.7525 | 0.056* |
| C11 | 0.5133 (4) | 0.49364 (10) | 0.66796 (12) | 0.0481 (5) |
| C12 | 0.6864 (4) | 0.49822 (11) | 0.60332 (15) | 0.0629 (6) |
| H12 | 0.7146 | 0.5438 | 0.5760 | 0.075* |
| C13 | 0.8196 (4) | 0.43625 (11) | 0.57813 (16) | 0.0631 (6) |
| H13 | 0.9389 | 0.4404 | 0.5350 | 0.076* |
| C14 | 0.3712 (4) | 0.28318 (11) | 0.77630 (14) | 0.0581 (6) |
| H14A | 0.3996 | 0.3146 | 0.8282 | 0.087* |
| H14B | 0.3679 | 0.2321 | 0.7951 | 0.087* |
| H14C | 0.2109 | 0.2958 | 0.7450 | 0.087* |
| C15 | 0.3720 (4) | 0.56143 (11) | 0.69069 (15) | 0.0561 (5) |
| C16 | 0.0836 (5) | 0.61549 (13) | 0.78568 (19) | 0.0836 (8) |
| H16A | 0.1948 | 0.6546 | 0.8085 | 0.100* |
| H16B | −0.0272 | 0.6009 | 0.8312 | 0.100* |
| H16C | −0.0171 | 0.6329 | 0.7335 | 0.100* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0645 (9) | 0.0414 (7) | 0.0585 (8) | 0.0127 (6) | 0.0255 (7) | 0.0029 (6) |
| O2 | 0.0783 (10) | 0.0372 (7) | 0.0597 (8) | 0.0080 (6) | 0.0284 (7) | 0.0058 (6) |
| O3 | 0.1240 (15) | 0.0404 (9) | 0.0900 (12) | 0.0199 (8) | 0.0231 (10) | 0.0051 (8) |
| O4 | 0.0835 (11) | 0.0497 (9) | 0.0804 (10) | 0.0211 (7) | 0.0277 (9) | −0.0038 (7) |
| C1 | 0.0590 (13) | 0.0555 (13) | 0.0617 (13) | 0.0077 (10) | 0.0084 (10) | −0.0034 (10) |
| C2 | 0.0729 (15) | 0.0581 (14) | 0.0848 (17) | 0.0183 (12) | 0.0168 (13) | 0.0063 (13) |
| C3 | 0.0827 (17) | 0.0462 (12) | 0.0868 (17) | −0.0010 (12) | 0.0399 (14) | −0.0076 (12) |
| C4 | 0.0712 (16) | 0.0758 (18) | 0.0911 (18) | −0.0194 (13) | 0.0155 (14) | −0.0256 (14) |
| C5 | 0.0511 (12) | 0.0743 (16) | 0.0796 (16) | 0.0061 (11) | 0.0058 (11) | −0.0059 (13) |
| C6 | 0.0469 (11) | 0.0491 (11) | 0.0487 (11) | 0.0023 (9) | 0.0188 (9) | 0.0002 (9) |
| C7 | 0.0582 (12) | 0.0511 (12) | 0.0591 (12) | 0.0094 (9) | 0.0242 (10) | 0.0056 (10) |
| C8 | 0.0485 (10) | 0.0385 (10) | 0.0504 (10) | 0.0063 (8) | 0.0082 (8) | −0.0017 (8) |
| C9 | 0.0503 (10) | 0.0344 (9) | 0.0448 (10) | 0.0026 (8) | 0.0062 (8) | 0.0007 (8) |
| C10 | 0.0510 (11) | 0.0433 (11) | 0.0456 (10) | 0.0046 (8) | 0.0097 (8) | −0.0038 (9) |
| C11 | 0.0548 (11) | 0.0358 (10) | 0.0538 (11) | 0.0039 (8) | 0.0054 (9) | −0.0034 (9) |
| C12 | 0.0768 (14) | 0.0374 (10) | 0.0771 (15) | 0.0008 (10) | 0.0244 (12) | 0.0060 (10) |
| C13 | 0.0676 (13) | 0.0468 (12) | 0.0786 (14) | 0.0061 (10) | 0.0323 (11) | 0.0080 (11) |
| C14 | 0.0702 (14) | 0.0482 (12) | 0.0581 (12) | −0.0017 (10) | 0.0208 (10) | 0.0073 (10) |
| C15 | 0.0660 (13) | 0.0408 (11) | 0.0613 (13) | 0.0066 (9) | 0.0027 (11) | −0.0086 (10) |
| C16 | 0.0882 (18) | 0.0625 (15) | 0.1027 (19) | 0.0262 (13) | 0.0258 (15) | −0.0171 (14) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C8 | 1.360 (2) | C7—H7A | 0.9700 |
| O1—C7 | 1.437 (2) | C7—H7B | 0.9700 |
| O2—C9 | 1.357 (2) | C8—C13 | 1.381 (3) |
| O2—C14 | 1.424 (2) | C8—C9 | 1.402 (3) |
| O3—C15 | 1.205 (2) | C9—C10 | 1.378 (2) |
| O4—C15 | 1.327 (3) | C10—C11 | 1.387 (3) |
| O4—C16 | 1.440 (2) | C10—H10 | 0.9300 |
| C1—C6 | 1.369 (3) | C11—C12 | 1.371 (3) |
| C1—C2 | 1.371 (3) | C11—C15 | 1.477 (3) |
| C1—H1 | 0.9300 | C12—C13 | 1.381 (3) |
| C2—C3 | 1.371 (3) | C12—H12 | 0.9300 |
| C2—H2 | 0.9300 | C13—H13 | 0.9300 |
| C3—C4 | 1.354 (3) | C14—H14A | 0.9600 |
| C3—H3 | 0.9300 | C14—H14B | 0.9600 |
| C4—C5 | 1.374 (3) | C14—H14C | 0.9600 |
| C4—H4 | 0.9300 | C16—H16A | 0.9601 |
| C5—C6 | 1.374 (3) | C16—H16B | 0.9601 |
| C5—H5 | 0.9300 | C16—H16C | 0.9600 |
| C6—C7 | 1.498 (3) | | |
| | | | |
| C8—O1—C7 | 117.96 (14) | O2—C9—C10 | 125.47 (16) |
| C9—O2—C14 | 117.16 (14) | O2—C9—C8 | 115.00 (15) |
| C15—O4—C16 | 116.30 (17) | C10—C9—C8 | 119.53 (16) |
| C6—C1—C2 | 120.6 (2) | C9—C10—C11 | 120.70 (17) |
| C6—C1—H1 | 119.7 | C9—C10—H10 | 119.7 |
| C2—C1—H1 | 119.7 | C11—C10—H10 | 119.7 |
| C1—C2—C3 | 120.8 (2) | C12—C11—C10 | 119.29 (17) |
| C1—C2—H2 | 119.6 | C12—C11—C15 | 118.61 (17) |
| C3—C2—H2 | 119.6 | C10—C11—C15 | 122.08 (18) |
| C4—C3—C2 | 119.1 (2) | C11—C12—C13 | 120.99 (18) |
| C4—C3—H3 | 120.5 | C11—C12—H12 | 119.5 |
| C2—C3—H3 | 120.5 | C13—C12—H12 | 119.5 |
| C3—C4—C5 | 120.3 (2) | C12—C13—C8 | 120.00 (19) |
| C3—C4—H4 | 119.8 | C12—C13—H13 | 120.0 |
| C5—C4—H4 | 119.8 | C8—C13—H13 | 120.0 |
| C4—C5—C6 | 121.1 (2) | O2—C14—H14A | 109.5 |
| C4—C5—H5 | 119.4 | O2—C14—H14B | 109.5 |
| C6—C5—H5 | 119.4 | H14A—C14—H14B | 109.5 |
| C1—C6—C5 | 118.12 (19) | O2—C14—H14C | 109.5 |
| C1—C6—C7 | 121.67 (18) | H14A—C14—H14C | 109.5 |
| C5—C6—C7 | 120.21 (18) | H14B—C14—H14C | 109.5 |
| O1—C7—C6 | 106.99 (15) | O3—C15—O4 | 122.60 (18) |
| O1—C7—H7A | 110.3 | O3—C15—C11 | 124.3 (2) |
| C6—C7—H7A | 110.3 | O4—C15—C11 | 113.09 (18) |
| O1—C7—H7B | 110.3 | O4—C16—H16A | 109.5 |
| C6—C7—H7B | 110.3 | O4—C16—H16B | 109.4 |
| H7A—C7—H7B | 108.6 | H16A—C16—H16B | 109.5 |
| O1—C8—C13 | 125.04 (17) | O4—C16—H16C | 109.5 |

| | | | |
|---|---|---|---|
| O1—C8—C9 | 115.48 (15) | H16A—C16—H16C | 109.5 |
| C13—C8—C9 | 119.47 (16) | H16B—C16—H16C | 109.5 |
| | | | |
| C6—C1—C2—C3 | 0.9 (3) | O1—C8—C9—C10 | −178.12 (16) |
| C1—C2—C3—C4 | −0.5 (3) | C13—C8—C9—C10 | 0.9 (3) |
| C2—C3—C4—C5 | 0.4 (4) | O2—C9—C10—C11 | −179.44 (17) |
| C3—C4—C5—C6 | −0.9 (4) | C8—C9—C10—C11 | 0.2 (3) |
| C2—C1—C6—C5 | −1.3 (3) | C9—C10—C11—C12 | −0.5 (3) |
| C2—C1—C6—C7 | 178.4 (2) | C9—C10—C11—C15 | 177.82 (18) |
| C4—C5—C6—C1 | 1.3 (3) | C10—C11—C12—C13 | −0.2 (3) |
| C4—C5—C6—C7 | −178.4 (2) | C15—C11—C12—C13 | −178.6 (2) |
| C8—O1—C7—C6 | −168.61 (16) | C11—C12—C13—C8 | 1.3 (4) |
| C1—C6—C7—O1 | −90.8 (2) | O1—C8—C13—C12 | 177.3 (2) |
| C5—C6—C7—O1 | 88.9 (2) | C9—C8—C13—C12 | −1.6 (3) |
| C7—O1—C8—C13 | −5.6 (3) | C16—O4—C15—O3 | 2.0 (3) |
| C7—O1—C8—C9 | 173.40 (16) | C16—O4—C15—C11 | −177.77 (18) |
| C14—O2—C9—C10 | 8.2 (3) | C12—C11—C15—O3 | 8.1 (3) |
| C14—O2—C9—C8 | −171.46 (16) | C10—C11—C15—O3 | −170.3 (2) |
| O1—C8—C9—O2 | 1.5 (2) | C12—C11—C15—O4 | −172.12 (18) |
| C13—C8—C9—O2 | −179.44 (18) | C10—C11—C15—O4 | 9.5 (3) |

*Hydrogen-bond geometry (Å, º)*

Cg is the centroid of the C1–C6 ring

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C14—H14*B*···O3[i] | 0.96 | 2.53 | 3.379 (3) | 147 |
| C14—H14*A*···*Cg*[ii] | 0.96 | 2.75 | 3.519 (2) | 137 |

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+3/2; (ii) *x*−1, −*y*−1/2, *z*−1/2.