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# 6-Chloro-4-(4-methylphenoxymethyl)-2*H*-chromen-2-one

**Ramakrishna Gowda,[a]* K.V. Arjuna Gowda,[b] Mahantesha Basanagouda[c] and Manohar V. Kulkarni[c]**

[a]Department of Physics, Govt. College for Women, Kolar 563 101, Karnataka, India, [b]Department of Physics, Govt. College for Women, Mandya 571 401, Karnataka, India, and [c]Department of Chemistry, Karnatak University, Dharwad 580 003, Karnataka, India
Correspondence e-mail: kvarjunagowda@gmail.com

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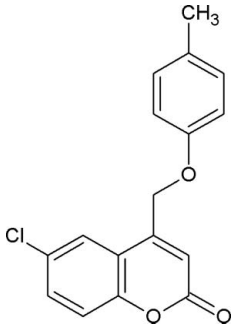
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.053; $wR$ factor = 0.170; data-to-parameter ratio = 20.0.

In the title compound, $C_{17}H_{13}ClO_3$, the coumarin and phenoxy moieties are essentially co-planar, making a dihedral angle of 1.99 (7)°. The phenoxy moiety is oriented antiperiplanar with respect to the coumarin ring as indicated by the C—C—O—C angle of −179.97 (16)°. In the crystal, the sheet-like packing is stabilized by intermolecular C—H···O and C—H···Cl hydrogen bonds.

## Related literature

For the structure of 7-methyl-4-tolyloxymethylcoumarin, see: Vasudevan *et al.* (1990).




## Experimental

### Crystal data

$C_{17}H_{13}ClO_3$ $M_r$ = 300.72
Monoclinic, $P2_1/c$
$a$ = 15.3068 (5) Å
$b$ = 6.9353 (2) Å
$c$ = 14.9566 (5) Å
$\beta$ = 116.923 (2)°
$V$ = 1415.66 (8) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.28 mm$^{-1}$
$T$ = 293 K
0.30 × 0.20 × 0.20 mm

### Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min}$ = 0.932, $T_{max}$ = 0.967
16899 measured reflections
3818 independent reflections
2448 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.038

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.170
$S$ = 1.08
3818 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.22 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C10—H10$B$···O2$^{i}$ | 0.97 | 2.47 | 3.303 (5) | 143 |
| C4—H4···O2$^{i}$ | 0.93 | 2.69 | 3.553 (4) | 154 |
| C1—H1···Cl1$^{ii}$ | 0.93 | 2.88 | 3.693 (4) | 146 |

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: VM2090).

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# supporting information

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## 6-Chloro-4-(4-methylphenoxymethyl)-2*H*-chromen-2-one

**Ramakrishna Gowda, K.V. Arjuna Gowda, Mahantesha Basanagouda and Manohar V. Kulkarni**

### S1. Comment

The first report on X-ray diffraction studies on 4-aryloxymethylcoumarins has revealed that in solid state the molecules exist as head-tail dimers as observed in the case of 7-methyl-4-tolyloxymethylcoumarin (Vasudevan *et al.*, 1990). In the light of these observations a chloro substituted 4-aryloxymethylcoumarin has been subjected to X-ray diffraction studies. A significant bond deviation is observed at C5—C7 (1.449 (2) Å) due to the bridging of $\alpha$-pyrone and benzene ring at C5 and the substituent present at C7. This is also reflected at C8—C9 and C7—C10 due to the presence of O2 at C9 and a phenoxy group at C10, respectively. Significant bond angle deviations are observed at C6—C5—C4 (117.91 (17)°) and C6—C5—C7 (117.61 (18)°). Another significant bond angle deviation is observed at C15—C14—C13 (117.46 (18)°) due to presence of the electron donating methyl group on C14. The molecules are oriented as parallel layers along the *c* axis as shown in Fig 2. The sheet-like packing is stabilized by intermolecular C—H···O and C—H···Cl hydrogen bonds (Table 1, Fig. 3).

### S2. Experimental

A mixture of 4-methyl-phenol (10 mmol) and anhydrous potassium carbonate (10 mmol) was stirred for 30 minutes in dry acetone (30 ml). To this, 6-chloro-4-bromomethylcoumarin (10 mmol) was added and the stirring was continued for 24 h. Then, the resulting reaction mixture was poured to crushed ice. The separated solid was filtered and washed with 1:1 HCl (30 ml) and with water. Then product 6-chloro-4-[(4-methyl)phenoxymethyl]coumarin was recrystallized from ethyl acetate.

### S3. Refinement

Hydrogen atoms were positioned geometrically with C—H = 0.93–0.97 A° and included in the refinment in a riding-model approximation with $U_{iso}$(H) = 1.2 $U_{eq}$(C) or 1.5 $U_{eq}$(C) for methyl C atoms.

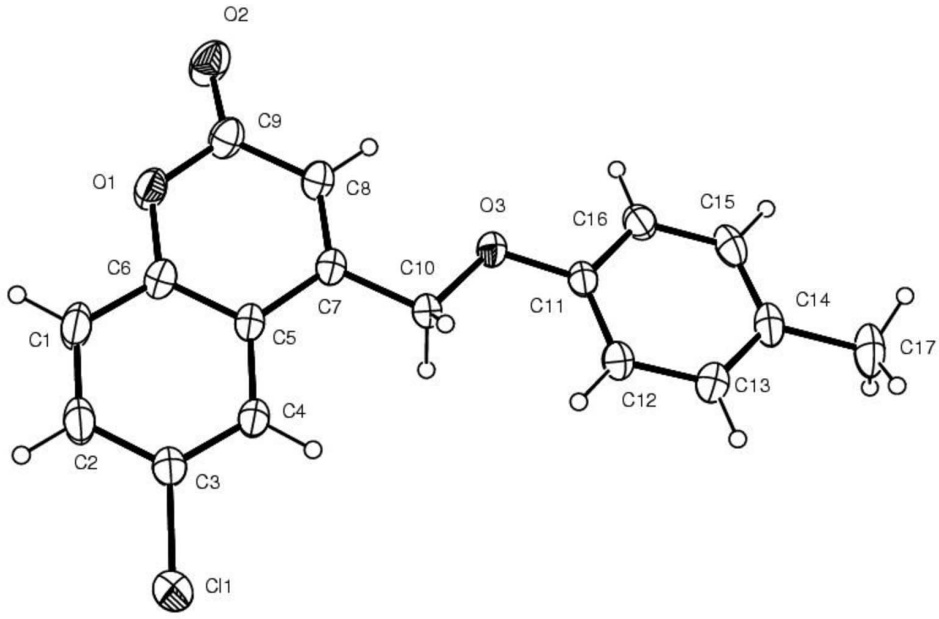


**Figure 1**

The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering.

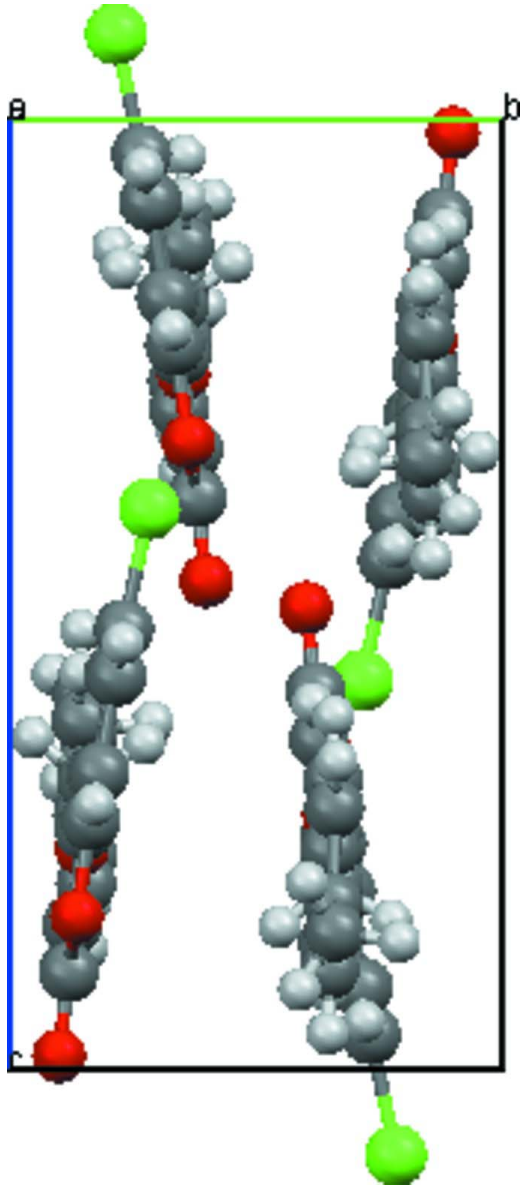


**Figure 2**

Packing diagram viewed down *a* axis and molecules oriented as parallel layers along *c* axis.

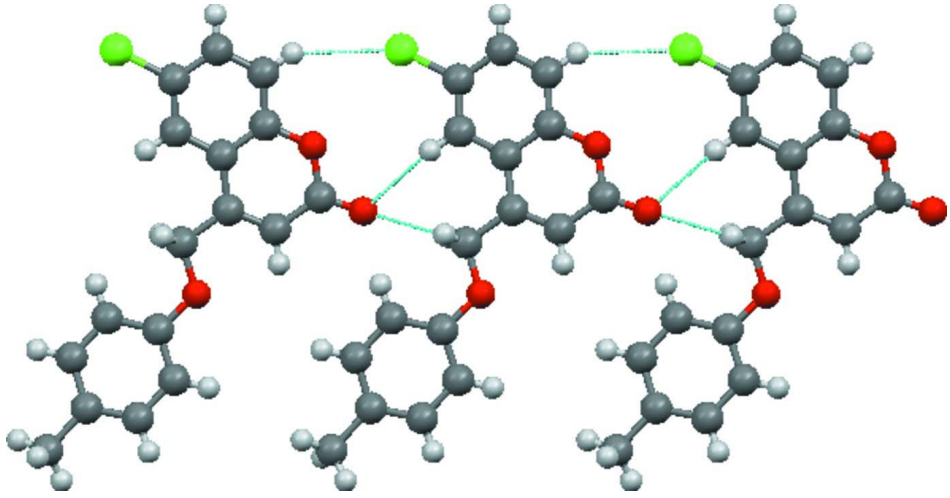

**Figure 3**

Packing diagram showing C—H···O and C—H···Cl hydrogen bonding.

### 6-Chloro-4-(4-methylphenoxymethyl)-2*H*-chromen-2-one

*Crystal data*

$C_{17}H_{13}ClO_3$
$M_r = 300.72$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 15.3068$ (5) Å
$b = 6.9353$ (2) Å
$c = 14.9566$ (5) Å
$\beta = 116.923$ (2)°
$V = 1415.66$ (8) Å$^3$
$Z = 4$
$F(000) = 624$
$D_x = 1.411$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4071 reflections
$\theta = 2.7$–$28.3°$
$\mu = 0.28$ mm$^{-1}$
$T = 293$ K
Block, colourless
0.30 × 0.20 × 0.20 mm

*Data collection*

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ and $\varphi$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min} = 0.932$, $T_{max} = 0.967$
16899 measured reflections
3818 independent reflections
2448 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 29.2°$, $\theta_{min} = 1.5°$
$h = -20 \rightarrow 20$
$k = -9 \rightarrow 6$
$l = -20 \rightarrow 20$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.170$
$S = 1.08$
3818 reflections
191 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
$w = 1/[\sigma^2(F_o^2) + (0.0833P)^2 + 0.2177P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å$^{-3}$
$\Delta\rho_{min} = -0.22$ e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.09025 (16) | 0.1827 (4) | 0.68708 (19) | 0.0627 (7) |
| H1 | 0.0528 | 0.1718 | 0.7215 | 0.075* |
| C2 | 0.04581 (17) | 0.2170 (4) | 0.5863 (2) | 0.0652 (7) |
| H2 | −0.0219 | 0.2299 | 0.5514 | 0.078* |
| C3 | 0.10297 (15) | 0.2323 (4) | 0.53657 (16) | 0.0509 (5) |
| C4 | 0.20280 (14) | 0.2106 (3) | 0.58555 (15) | 0.0430 (5) |
| H4 | 0.2395 | 0.2191 | 0.5503 | 0.052* |
| C5 | 0.24892 (13) | 0.1756 (3) | 0.68853 (14) | 0.0371 (4) |
| C6 | 0.19059 (15) | 0.1644 (3) | 0.73768 (16) | 0.0448 (5) |
| C7 | 0.35374 (13) | 0.1530 (3) | 0.74835 (14) | 0.0359 (4) |
| C8 | 0.39041 (14) | 0.1275 (3) | 0.84735 (15) | 0.0428 (5) |
| H8 | 0.4578 | 0.1139 | 0.8853 | 0.051* |
| C9 | 0.32906 (15) | 0.1204 (3) | 0.89761 (16) | 0.0482 (5) |
| C10 | 0.41673 (12) | 0.1566 (3) | 0.69576 (14) | 0.0384 (4) |
| H10A | 0.4010 | 0.0477 | 0.6503 | 0.046* |
| H10B | 0.4049 | 0.2741 | 0.6568 | 0.046* |
| C11 | 0.58438 (13) | 0.1494 (3) | 0.73188 (15) | 0.0380 (4) |
| C12 | 0.56245 (14) | 0.1461 (3) | 0.63200 (15) | 0.0437 (5) |
| H12 | 0.4976 | 0.1427 | 0.5829 | 0.052* |
| C13 | 0.63822 (15) | 0.1481 (3) | 0.60532 (17) | 0.0488 (5) |
| H13 | 0.6233 | 0.1448 | 0.5377 | 0.059* |
| C14 | 0.73516 (15) | 0.1549 (3) | 0.67619 (18) | 0.0505 (5) |
| C15 | 0.75450 (15) | 0.1587 (3) | 0.77520 (19) | 0.0525 (6) |
| H15 | 0.8193 | 0.1637 | 0.8243 | 0.063* |
| C16 | 0.68085 (14) | 0.1553 (3) | 0.80433 (16) | 0.0458 (5) |
| H16 | 0.6960 | 0.1571 | 0.8720 | 0.055* |
| C17 | 0.81634 (18) | 0.1592 (4) | 0.6449 (2) | 0.0747 (8) |
| H17A | 0.8295 | 0.2904 | 0.6344 | 0.112* |
| H17B | 0.7965 | 0.0876 | 0.5839 | 0.112* |
| H17C | 0.8745 | 0.1026 | 0.6967 | 0.112* |
| O1 | 0.23009 (10) | 0.1370 (2) | 0.83898 (11) | 0.0524 (4) |
| O2 | 0.35626 (12) | 0.1007 (3) | 0.98589 (11) | 0.0684 (5) |
| O3 | 0.51603 (9) | 0.1474 (2) | 0.76774 (10) | 0.0459 (4) |
| Cl1 | 0.04586 (4) | 0.28400 (13) | 0.40961 (5) | 0.0790 (3) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0443 (12) | 0.093 (2) | 0.0644 (14) | −0.0023 (12) | 0.0368 (11) | −0.0043 (13) |
| C2 | 0.0363 (10) | 0.099 (2) | 0.0655 (15) | 0.0039 (12) | 0.0278 (10) | −0.0017 (14) |
| C3 | 0.0389 (10) | 0.0683 (15) | 0.0476 (11) | 0.0036 (10) | 0.0213 (9) | 0.0021 (10) |
| C4 | 0.0393 (10) | 0.0470 (12) | 0.0499 (11) | 0.0003 (9) | 0.0266 (9) | −0.0001 (9) |
| C5 | 0.0359 (9) | 0.0345 (10) | 0.0472 (10) | −0.0020 (7) | 0.0243 (8) | −0.0036 (8) |
| C6 | 0.0444 (10) | 0.0494 (13) | 0.0495 (11) | −0.0037 (9) | 0.0291 (9) | −0.0048 (9) |
| C7 | 0.0382 (9) | 0.0289 (9) | 0.0473 (10) | −0.0025 (7) | 0.0252 (8) | −0.0040 (8) |
| C8 | 0.0407 (10) | 0.0450 (12) | 0.0470 (11) | −0.0061 (8) | 0.0236 (8) | −0.0072 (9) |
| C9 | 0.0512 (11) | 0.0538 (14) | 0.0463 (11) | −0.0096 (10) | 0.0278 (9) | −0.0128 (10) |
| C10 | 0.0314 (8) | 0.0445 (11) | 0.0413 (9) | 0.0016 (8) | 0.0183 (7) | 0.0024 (8) |
| C11 | 0.0337 (9) | 0.0344 (10) | 0.0497 (10) | 0.0020 (7) | 0.0223 (8) | 0.0023 (8) |
| C12 | 0.0342 (9) | 0.0480 (12) | 0.0501 (11) | −0.0015 (8) | 0.0201 (8) | −0.0021 (9) |
| C13 | 0.0473 (11) | 0.0518 (13) | 0.0556 (12) | −0.0010 (10) | 0.0308 (10) | −0.0020 (10) |
| C14 | 0.0416 (11) | 0.0450 (12) | 0.0747 (15) | 0.0031 (9) | 0.0351 (11) | −0.0021 (11) |
| C15 | 0.0299 (9) | 0.0511 (13) | 0.0724 (15) | 0.0033 (9) | 0.0195 (9) | 0.0012 (11) |
| C16 | 0.0387 (10) | 0.0475 (12) | 0.0492 (11) | 0.0051 (9) | 0.0181 (8) | 0.0031 (9) |
| C17 | 0.0522 (14) | 0.084 (2) | 0.108 (2) | 0.0047 (13) | 0.0538 (15) | −0.0040 (16) |
| O1 | 0.0474 (8) | 0.0715 (11) | 0.0489 (8) | −0.0054 (7) | 0.0310 (7) | −0.0060 (7) |
| O2 | 0.0664 (10) | 0.1026 (15) | 0.0437 (9) | −0.0135 (10) | 0.0315 (8) | −0.0150 (9) |
| O3 | 0.0328 (7) | 0.0636 (10) | 0.0443 (8) | 0.0027 (6) | 0.0201 (6) | 0.0057 (6) |
| Cl1 | 0.0481 (3) | 0.1308 (7) | 0.0550 (4) | 0.0162 (3) | 0.0206 (3) | 0.0183 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.365 (3) | C10—O3 | 1.411 (2) |
| C1—C6 | 1.377 (3) | C10—H10A | 0.9700 |
| C1—H1 | 0.9300 | C10—H10B | 0.9700 |
| C2—C3 | 1.385 (3) | C11—O3 | 1.375 (2) |
| C2—H2 | 0.9300 | C11—C12 | 1.375 (3) |
| C3—C4 | 1.371 (3) | C11—C16 | 1.381 (3) |
| C3—Cl1 | 1.731 (2) | C12—C13 | 1.387 (3) |
| C4—C5 | 1.394 (3) | C12—H12 | 0.9300 |
| C4—H4 | 0.9300 | C13—C14 | 1.380 (3) |
| C5—C6 | 1.392 (2) | C13—H13 | 0.9300 |
| C5—C7 | 1.449 (2) | C14—C15 | 1.373 (3) |
| C6—O1 | 1.367 (2) | C14—C17 | 1.513 (3) |
| C7—C8 | 1.336 (3) | C15—C16 | 1.381 (3) |
| C7—C10 | 1.495 (2) | C15—H15 | 0.9300 |
| C8—C9 | 1.445 (3) | C16—H16 | 0.9300 |
| C8—H8 | 0.9300 | C17—H17A | 0.9600 |
| C9—O2 | 1.199 (2) | C17—H17B | 0.9600 |
| C9—O1 | 1.369 (3) | C17—H17C | 0.9600 |
| | | | |
| C2—C1—C6 | 119.81 (19) | O3—C10—H10B | 109.9 |
| C2—C1—H1 | 120.1 | C7—C10—H10B | 109.9 |

| | | | |
|---|---|---|---|
| C6—C1—H1 | 120.1 | H10A—C10—H10B | 108.3 |
| C1—C2—C3 | 119.1 (2) | O3—C11—C12 | 124.69 (16) |
| C1—C2—H2 | 120.5 | O3—C11—C16 | 115.23 (17) |
| C3—C2—H2 | 120.5 | C12—C11—C16 | 120.08 (17) |
| C4—C3—C2 | 121.8 (2) | C11—C12—C13 | 119.20 (18) |
| C4—C3—Cl1 | 119.73 (16) | C11—C12—H12 | 120.4 |
| C2—C3—Cl1 | 118.47 (17) | C13—C12—H12 | 120.4 |
| C3—C4—C5 | 119.56 (18) | C14—C13—C12 | 121.9 (2) |
| C3—C4—H4 | 120.2 | C14—C13—H13 | 119.1 |
| C5—C4—H4 | 120.2 | C12—C13—H13 | 119.1 |
| C6—C5—C4 | 117.91 (17) | C15—C14—C13 | 117.46 (18) |
| C6—C5—C7 | 117.61 (18) | C15—C14—C17 | 121.8 (2) |
| C4—C5—C7 | 124.48 (16) | C13—C14—C17 | 120.7 (2) |
| O1—C6—C1 | 116.47 (17) | C14—C15—C16 | 122.1 (2) |
| O1—C6—C5 | 121.71 (18) | C14—C15—H15 | 118.9 |
| C1—C6—C5 | 121.8 (2) | C16—C15—H15 | 118.9 |
| C8—C7—C5 | 119.38 (16) | C15—C16—C11 | 119.2 (2) |
| C8—C7—C10 | 122.52 (17) | C15—C16—H16 | 120.4 |
| C5—C7—C10 | 118.09 (16) | C11—C16—H16 | 120.4 |
| C7—C8—C9 | 122.34 (18) | C14—C17—H17A | 109.5 |
| C7—C8—H8 | 118.8 | C14—C17—H17B | 109.5 |
| C9—C8—H8 | 118.8 | H17A—C17—H17B | 109.5 |
| O2—C9—O1 | 116.54 (18) | C14—C17—H17C | 109.5 |
| O2—C9—C8 | 126.4 (2) | H17A—C17—H17C | 109.5 |
| O1—C9—C8 | 117.07 (17) | H17B—C17—H17C | 109.5 |
| O3—C10—C7 | 109.05 (15) | C6—O1—C9 | 121.85 (15) |
| O3—C10—H10A | 109.9 | C11—O3—C10 | 116.67 (14) |
| C7—C10—H10A | 109.9 | | |

| | | | |
|---|---|---|---|
| C6—C1—C2—C3 | 0.1 (4) | C7—C8—C9—O1 | 1.3 (3) |
| C1—C2—C3—C4 | 1.2 (4) | C8—C7—C10—O3 | −5.3 (3) |
| C1—C2—C3—Cl1 | −177.8 (2) | C5—C7—C10—O3 | 175.47 (16) |
| C2—C3—C4—C5 | −1.2 (4) | O3—C11—C12—C13 | −179.84 (19) |
| Cl1—C3—C4—C5 | 177.74 (16) | C16—C11—C12—C13 | 0.3 (3) |
| C3—C4—C5—C6 | 0.0 (3) | C11—C12—C13—C14 | −0.5 (3) |
| C3—C4—C5—C7 | −179.1 (2) | C12—C13—C14—C15 | 0.3 (3) |
| C2—C1—C6—O1 | 177.8 (2) | C12—C13—C14—C17 | −179.3 (2) |
| C2—C1—C6—C5 | −1.4 (4) | C13—C14—C15—C16 | 0.2 (3) |
| C4—C5—C6—O1 | −177.77 (18) | C17—C14—C15—C16 | 179.8 (2) |
| C7—C5—C6—O1 | 1.4 (3) | C14—C15—C16—C11 | −0.5 (3) |
| C4—C5—C6—C1 | 1.3 (3) | O3—C11—C16—C15 | −179.67 (19) |
| C7—C5—C6—C1 | −179.5 (2) | C12—C11—C16—C15 | 0.2 (3) |
| C6—C5—C7—C8 | −1.7 (3) | C1—C6—O1—C9 | −178.8 (2) |
| C4—C5—C7—C8 | 177.35 (19) | C5—C6—O1—C9 | 0.4 (3) |
| C6—C5—C7—C10 | 177.49 (17) | O2—C9—O1—C6 | 178.5 (2) |
| C4—C5—C7—C10 | −3.4 (3) | C8—C9—O1—C6 | −1.7 (3) |
| C5—C7—C8—C9 | 0.4 (3) | C12—C11—O3—C10 | −4.7 (3) |
| C10—C7—C8—C9 | −178.76 (19) | C16—C11—O3—C10 | 175.20 (17) |

| | | | |
|---|---|---|---|
| C7—C8—C9—O2 | −178.9 (2) | C7—C10—O3—C11 | −179.97 (16) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C10—H10*B*···O2$^{i}$ | 0.97 | 2.47 | 3.303 (5) | 143 |
| C4—H4···O2$^{i}$ | 0.93 | 2.69 | 3.553 (4) | 154 |
| C1—H1···Cl1$^{ii}$ | 0.93 | 2.88 | 3.693 (4) | 146 |

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