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2-(4-Chlorophenyl)-2-oxoethyl 4-hydroxybenzoate

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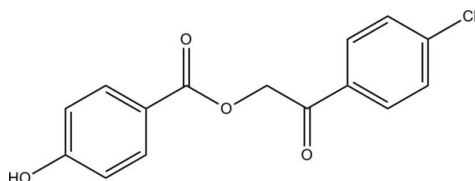
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.044; wR factor = 0.106; data-to-parameter ratio = 19.2.

The title compound, $\text{C}_{15}\text{H}_{11}\text{ClO}_4$, consists of a chlorobenzene ring and a phenol ring which are linked together by a 1,4-dioxo-2-oxabutane-1,4-diyl group. The dihedral angle between the chlorobenzene and phenol rings is $65.70(11)^\circ$. In the crystal, intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into chains along [010].

Related literature

For background to phenacyl benzoate, see: Sheehan & Umezawa (1973); Gandhi *et al.* (1995); Huang *et al.* (1996); Ruzicka *et al.* (2002); Litera *et al.* (2006). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{11}\text{ClO}_4$
 $M_r = 290.69$
Monoclinic, $P2_1$
 $a = 5.5307(10)$ Å

$b = 8.1324(15)$ Å
 $c = 14.857(2)$ Å
 $\beta = 95.120(4)^\circ$
 $V = 665.57(19)$ Å³

$Z = 2$
Mo $K\alpha$ radiation
 $\mu = 0.30$ mm⁻¹

$T = 296$ K
 $0.56 \times 0.23 \times 0.07$ mm

Data collection

Bruker APEXII DUO CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2009)
 $T_{\min} = 0.852$, $T_{\max} = 0.981$

6399 measured reflections
3571 independent reflections
2457 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.106$
 $S = 1.02$
3571 reflections
186 parameters
1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.20$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.21$ e Å⁻³
Absolute structure: Flack (1983), 1511 Friedel pairs
Flack parameter: $-0.20(8)$

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{O4}-\text{H1O4}\cdots\text{O3}^i$ | 0.78 (4) | 2.01 (4) | 2.783 (3) | 168 (3) |

Symmetry code: (i) $x, y - 1, z$.

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; 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 and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2305).

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2-(4-Chlorophenyl)-2-oxoethyl 4-hydroxybenzoate

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S1. Comment

Phenacyl benzoate is a derivative of an acid formed by reaction between an acid and phenacyl bromide. These compounds find applications in the field of synthetic chemistry (Huang *et al.*, 1996; Gandhi *et al.*, 1995) such as in the synthesis of oxazoles, imidazoles, and benzoxazepines. They are also useful for photo-removable protecting groups for carboxylic acids in organic synthesis and biochemistry (Ruzicka *et al.*, 2002; Litera *et al.*, 2006; Sheehan & Umezaw, 1973). Keeping this in view, the title compound was synthesized to study its crystal structure.

The title compound, (Fig. 1), consists of a chlorobenzene (C11/C1–C6) ring and a phenol(O4/C10–C15) ring which are linked together by a 2-oxopropyl acetate (C7–C9/O1–O3) group. The dihedral angle formed between the chlorobenzene and a phenol ring is 65.70 (11)°. The bond lengths (Allen *et al.*, 1987) and angles are within normal ranges.

In the crystal packing (Fig. 2), intermolecular O4—H1O4···O3 hydrogen bonds (Table 1) link the molecules in one-dimensional chains along [010].

S2. Experimental

The mixture of 4-hydroxybenzoic acid (1.0 g, 0.0072 mol), potassium carbonate (1.10 g, 0.0079 mol) and 2-bromo-1-(4-chlorophenyl)ethanone (1.68 g, 0.0072 mol) in dimethylformamide (10 ml) was stirred at room temperature for 2 h. On cooling, colorless needle-shaped 2-(4-chlorophenyl)-2-oxoethyl 4-hydroxybenzoate begin to separate out. It was collected by filtration and recrystallized from ethanol. Yield: 1.95 g, 92.8%, M.p: 453–454 K.

S3. Refinement

The hydrogen atoms bound to C atoms were positioned geometrically [C–H = 0.9300–0.9700 Å] with $U_{\text{iso}}(\text{H}) = 1.2$. The hydrogen atoms attached to the O atom was located from the difference map and refined freely, [O–H = 0.79 (3) Å].

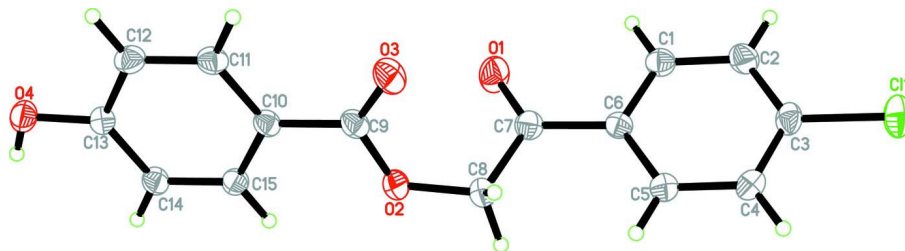


Figure 1

The molecular structure of the title compound, showing 30% probability displacement ellipsoids.

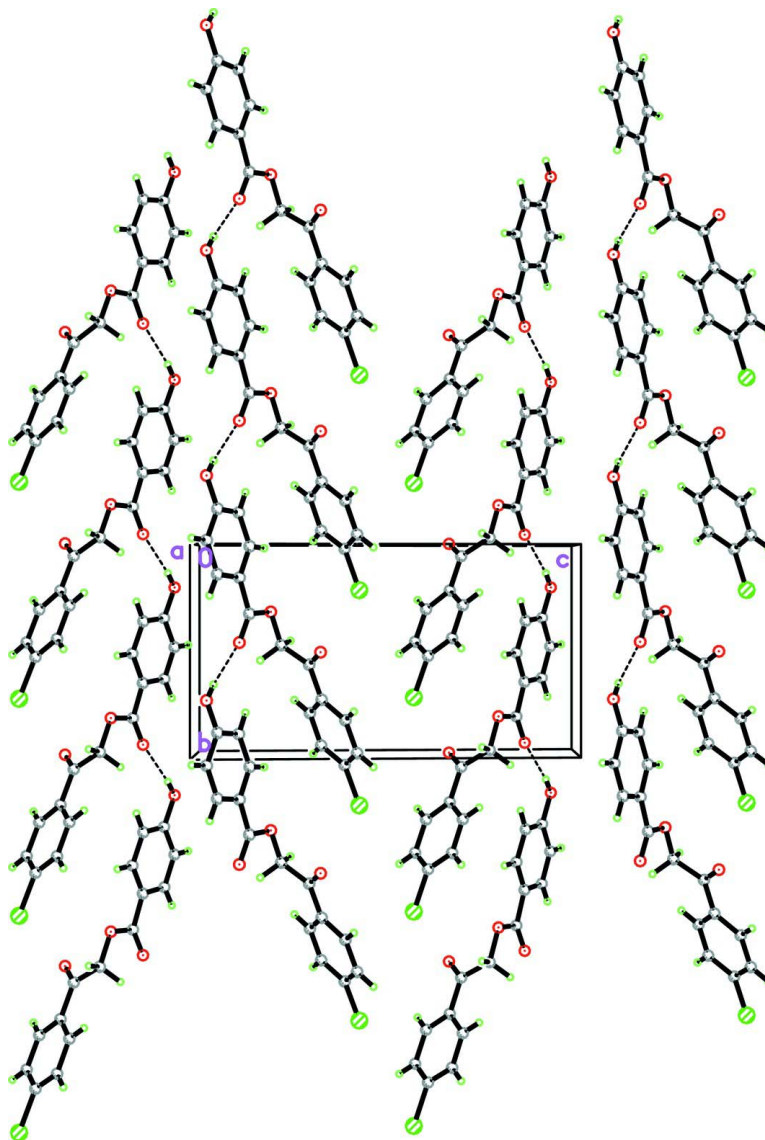


Figure 2

The crystal packing of the title compound, viewed along the a axis, showing a one-dimensional chain along $[010]$.

2-(4-Chlorophenyl)-2-oxoethyl 4-hydroxybenzoate

Crystal data

$C_{15}H_{11}ClO_4$

$M_r = 290.69$

Monoclinic, $P2_1$

Hall symbol: $P\ 2_1yb$

$a = 5.5307$ (10) Å

$b = 8.1324$ (15) Å

$c = 14.857$ (2) Å

$\beta = 95.120$ (4)°

$V = 665.57$ (19) Å³

$Z = 2$

$F(000) = 300$

$D_x = 1.450$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1832 reflections

$\theta = 3.7$ – 24.1 °

$\mu = 0.30$ mm⁻¹

$T = 296$ K

Plate, colourless

$0.56 \times 0.23 \times 0.07$ mm

Data collection

Bruker APEXII DUO CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)

$T_{\min} = 0.852$, $T_{\max} = 0.981$

6399 measured reflections

3571 independent reflections

2457 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 30.0^\circ$, $\theta_{\min} = 2.8^\circ$

$h = -5 \rightarrow 7$

$k = -11 \rightarrow 11$

$l = -20 \rightarrow 20$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.044$

$wR(F^2) = 0.106$

$S = 1.02$

3571 reflections

186 parameters

1 restraint

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0346P)^2 + 0.0709P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.20 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.21 \text{ e } \text{\AA}^{-3}$

Absolute structure: Flack (1983), 1511 Friedel
pairs

Absolute structure parameter: -0.20 (8)

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| C11 | 0.97678 (16) | 1.71550 (10) | 0.56971 (5) | 0.0916 (3) |
| O1 | 0.4790 (3) | 0.9949 (3) | 0.66410 (13) | 0.0797 (5) |
| O2 | 0.7288 (3) | 0.81404 (19) | 0.79114 (12) | 0.0616 (4) |
| O3 | 0.4559 (4) | 0.95006 (19) | 0.86448 (13) | 0.0700 (5) |
| O4 | 0.2024 (3) | 0.2039 (2) | 0.93940 (13) | 0.0633 (4) |
| C1 | 0.6002 (4) | 1.3072 (3) | 0.60310 (14) | 0.0559 (5) |
| H1A | 0.4508 | 1.2636 | 0.5815 | 0.067* |
| C2 | 0.6676 (5) | 1.4603 (4) | 0.57413 (16) | 0.0634 (6) |
| H2A | 0.5650 | 1.5196 | 0.5331 | 0.076* |
| C3 | 0.8875 (5) | 1.5239 (3) | 0.60661 (15) | 0.0579 (6) |
| C4 | 1.0422 (4) | 1.4387 (3) | 0.66746 (16) | 0.0587 (6) |
| H4A | 1.1905 | 1.4837 | 0.6893 | 0.070* |
| C5 | 0.9738 (4) | 1.2856 (3) | 0.69547 (15) | 0.0548 (5) |
| H5A | 1.0778 | 1.2268 | 0.7362 | 0.066* |
| C6 | 0.7520 (4) | 1.2176 (3) | 0.66383 (12) | 0.0466 (4) |

| | | | | |
|------|------------|------------|--------------|-------------|
| C7 | 0.6694 (4) | 1.0548 (3) | 0.69417 (15) | 0.0544 (5) |
| C8 | 0.8296 (4) | 0.9684 (3) | 0.76653 (18) | 0.0609 (6) |
| H8A | 0.9879 | 0.9496 | 0.7451 | 0.073* |
| H8B | 0.8517 | 1.0382 | 0.8195 | 0.073* |
| C9 | 0.5374 (4) | 0.8211 (3) | 0.84121 (15) | 0.0521 (5) |
| C10 | 0.4532 (4) | 0.6572 (3) | 0.86395 (13) | 0.0458 (5) |
| C11 | 0.2512 (4) | 0.6426 (3) | 0.91413 (14) | 0.0535 (5) |
| H11A | 0.1713 | 0.7367 | 0.9312 | 0.064* |
| C12 | 0.1709 (4) | 0.4916 (3) | 0.93820 (14) | 0.0565 (6) |
| H12A | 0.0353 | 0.4834 | 0.9707 | 0.068* |
| C13 | 0.2901 (4) | 0.3505 (3) | 0.91444 (14) | 0.0467 (5) |
| C14 | 0.4930 (4) | 0.3639 (3) | 0.86646 (15) | 0.0520 (5) |
| H14A | 0.5763 | 0.2698 | 0.8516 | 0.062* |
| C15 | 0.5711 (4) | 0.5153 (3) | 0.84087 (15) | 0.0495 (5) |
| H15A | 0.7052 | 0.5229 | 0.8075 | 0.059* |
| H1O4 | 0.289 (6) | 0.135 (5) | 0.924 (2) | 0.083 (11)* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C11 | 0.1111 (6) | 0.0700 (4) | 0.0958 (5) | −0.0168 (4) | 0.0211 (4) | 0.0258 (4) |
| O1 | 0.0787 (12) | 0.0704 (12) | 0.0853 (11) | −0.0250 (11) | −0.0197 (9) | 0.0065 (11) |
| O2 | 0.0618 (9) | 0.0398 (8) | 0.0840 (10) | 0.0020 (8) | 0.0114 (8) | 0.0072 (9) |
| O3 | 0.0857 (12) | 0.0393 (9) | 0.0855 (12) | 0.0051 (9) | 0.0107 (10) | −0.0103 (9) |
| O4 | 0.0671 (10) | 0.0527 (10) | 0.0728 (10) | −0.0026 (10) | 0.0207 (8) | 0.0070 (9) |
| C1 | 0.0540 (12) | 0.0605 (15) | 0.0527 (11) | 0.0018 (12) | 0.0012 (9) | 0.0002 (12) |
| C2 | 0.0657 (14) | 0.0673 (16) | 0.0562 (12) | 0.0059 (13) | 0.0010 (11) | 0.0138 (12) |
| C3 | 0.0674 (14) | 0.0545 (13) | 0.0546 (11) | −0.0056 (12) | 0.0208 (10) | 0.0081 (11) |
| C4 | 0.0508 (12) | 0.0629 (15) | 0.0633 (13) | −0.0090 (12) | 0.0096 (10) | −0.0003 (12) |
| C5 | 0.0509 (11) | 0.0545 (13) | 0.0587 (12) | −0.0006 (11) | 0.0032 (9) | 0.0043 (11) |
| C6 | 0.0480 (10) | 0.0479 (11) | 0.0444 (9) | 0.0032 (10) | 0.0066 (8) | −0.0032 (10) |
| C7 | 0.0560 (12) | 0.0485 (12) | 0.0582 (12) | −0.0018 (11) | 0.0018 (10) | −0.0061 (11) |
| C8 | 0.0563 (12) | 0.0468 (13) | 0.0788 (15) | −0.0019 (11) | 0.0014 (11) | 0.0101 (12) |
| C9 | 0.0566 (12) | 0.0415 (12) | 0.0567 (12) | 0.0036 (11) | −0.0031 (10) | −0.0033 (11) |
| C10 | 0.0485 (10) | 0.0385 (10) | 0.0499 (11) | 0.0024 (9) | 0.0009 (8) | −0.0027 (9) |
| C11 | 0.0536 (12) | 0.0481 (12) | 0.0595 (12) | 0.0093 (10) | 0.0085 (10) | −0.0084 (11) |
| C12 | 0.0519 (12) | 0.0622 (15) | 0.0568 (12) | 0.0049 (12) | 0.0128 (9) | −0.0031 (12) |
| C13 | 0.0510 (11) | 0.0441 (11) | 0.0451 (10) | −0.0010 (10) | 0.0052 (9) | 0.0012 (9) |
| C14 | 0.0580 (12) | 0.0401 (12) | 0.0594 (12) | 0.0063 (10) | 0.0128 (10) | −0.0010 (10) |
| C15 | 0.0489 (11) | 0.0435 (11) | 0.0569 (11) | 0.0025 (11) | 0.0101 (9) | −0.0013 (11) |

Geometric parameters (Å, °)

| | | | |
|--------|-----------|--------|-----------|
| C11—C3 | 1.738 (3) | C5—H5A | 0.9300 |
| O1—C7 | 1.209 (3) | C6—C7 | 1.483 (3) |
| O2—C9 | 1.349 (3) | C7—C8 | 1.505 (3) |
| O2—C8 | 1.434 (3) | C8—H8A | 0.9700 |
| O3—C9 | 1.204 (3) | C8—H8B | 0.9700 |

| | | | |
|--------------|--------------|-----------------|--------------|
| O4—C13 | 1.351 (3) | C9—C10 | 1.461 (3) |
| O4—H1O4 | 0.79 (3) | C10—C15 | 1.384 (3) |
| C1—C2 | 1.380 (4) | C10—C11 | 1.402 (3) |
| C1—C6 | 1.383 (3) | C11—C12 | 1.364 (4) |
| C1—H1A | 0.9300 | C11—H11A | 0.9300 |
| C2—C3 | 1.369 (4) | C12—C13 | 1.385 (3) |
| C2—H2A | 0.9300 | C12—H12A | 0.9300 |
| C3—C4 | 1.375 (4) | C13—C14 | 1.386 (3) |
| C4—C5 | 1.376 (3) | C14—C15 | 1.370 (3) |
| C4—H4A | 0.9300 | C14—H14A | 0.9300 |
| C5—C6 | 1.389 (3) | C15—H15A | 0.9300 |
| C9—O2—C8 | 116.48 (19) | C7—C8—H8A | 109.2 |
| C13—O4—H1O4 | 108 (2) | O2—C8—H8B | 109.2 |
| C2—C1—C6 | 120.9 (2) | C7—C8—H8B | 109.2 |
| C2—C1—H1A | 119.5 | H8A—C8—H8B | 107.9 |
| C6—C1—H1A | 119.5 | O3—C9—O2 | 121.8 (2) |
| C3—C2—C1 | 119.2 (2) | O3—C9—C10 | 126.4 (2) |
| C3—C2—H2A | 120.4 | O2—C9—C10 | 111.78 (19) |
| C1—C2—H2A | 120.4 | C15—C10—C11 | 118.4 (2) |
| C2—C3—C4 | 121.5 (2) | C15—C10—C9 | 122.44 (19) |
| C2—C3—C11 | 119.7 (2) | C11—C10—C9 | 119.07 (19) |
| C4—C3—C11 | 118.8 (2) | C12—C11—C10 | 120.6 (2) |
| C3—C4—C5 | 118.9 (2) | C12—C11—H11A | 119.7 |
| C3—C4—H4A | 120.5 | C10—C11—H11A | 119.7 |
| C5—C4—H4A | 120.5 | C11—C12—C13 | 120.35 (19) |
| C4—C5—C6 | 121.1 (2) | C11—C12—H12A | 119.8 |
| C4—C5—H5A | 119.5 | C13—C12—H12A | 119.8 |
| C6—C5—H5A | 119.5 | O4—C13—C12 | 118.0 (2) |
| C1—C6—C5 | 118.5 (2) | O4—C13—C14 | 122.5 (2) |
| C1—C6—C7 | 118.9 (2) | C12—C13—C14 | 119.5 (2) |
| C5—C6—C7 | 122.6 (2) | C15—C14—C13 | 120.2 (2) |
| O1—C7—C6 | 122.0 (2) | C15—C14—H14A | 119.9 |
| O1—C7—C8 | 120.9 (2) | C13—C14—H14A | 119.9 |
| C6—C7—C8 | 117.15 (19) | C14—C15—C10 | 120.92 (19) |
| O2—C8—C7 | 111.9 (2) | C14—C15—H15A | 119.5 |
| O2—C8—H8A | 109.2 | C10—C15—H15A | 119.5 |
| C6—C1—C2—C3 | -0.3 (3) | C8—O2—C9—O3 | -0.4 (3) |
| C1—C2—C3—C4 | -0.1 (4) | C8—O2—C9—C10 | -178.72 (19) |
| C1—C2—C3—C11 | 179.18 (18) | O3—C9—C10—C15 | -174.2 (2) |
| C2—C3—C4—C5 | 0.5 (3) | O2—C9—C10—C15 | 4.1 (3) |
| C11—C3—C4—C5 | -178.78 (17) | O3—C9—C10—C11 | 3.2 (3) |
| C3—C4—C5—C6 | -0.5 (3) | O2—C9—C10—C11 | -178.51 (18) |
| C2—C1—C6—C5 | 0.3 (3) | C15—C10—C11—C12 | -1.1 (3) |
| C2—C1—C6—C7 | 178.9 (2) | C9—C10—C11—C12 | -178.6 (2) |
| C4—C5—C6—C1 | 0.1 (3) | C10—C11—C12—C13 | 0.9 (3) |
| C4—C5—C6—C7 | -178.4 (2) | C11—C12—C13—O4 | -179.6 (2) |

| | | | |
|-------------|-------------|-----------------|-----------|
| C1—C6—C7—O1 | 3.7 (3) | C11—C12—C13—C14 | 0.4 (3) |
| C5—C6—C7—O1 | -177.8 (2) | O4—C13—C14—C15 | 178.4 (2) |
| C1—C6—C7—C8 | -174.6 (2) | C12—C13—C14—C15 | -1.6 (3) |
| C5—C6—C7—C8 | 4.0 (3) | C13—C14—C15—C10 | 1.4 (3) |
| C9—O2—C8—C7 | -73.7 (3) | C11—C10—C15—C14 | -0.1 (3) |
| O1—C7—C8—O2 | 0.8 (3) | C9—C10—C15—C14 | 177.3 (2) |
| C6—C7—C8—O2 | 179.02 (18) | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|---------------------------|-------------|---------------|-----------------------|-------------------------|
| O4—H1O4...O3 ⁱ | 0.78 (4) | 2.01 (4) | 2.783 (3) | 168 (3) |

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