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3,6,8-Trihydroxy-3,4,5,7-tetramethyl-3,4-dihydroisocoumarin

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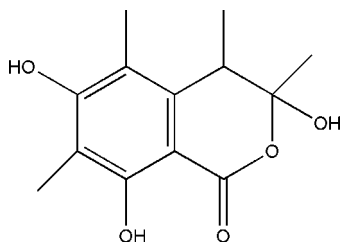
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 Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.025; wR factor = 0.069; data-to-parameter ratio = 12.9.

In the title compound, $\text{C}_{13}\text{H}_{16}\text{O}_5$, one of the three hydroxy groups is involved in intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds. The other two hydroxy groups contribute to the three-dimensional hydrogen-bonding network, which consolidates the crystal packing.

Related literature

 For related structures, see: Wang *et al.* (2003); Krohn *et al.* (1997).


Experimental

Crystal data

 $\text{C}_{13}\text{H}_{16}\text{O}_5$
 $M_r = 252.26$

 Orthorhombic, $P2_12_12_1$
 $a = 7.4731$ (3) Å

 $b = 9.9742$ (7) Å

 $c = 16.4066$ (12) Å

 $V = 1222.92$ (13) Å³
 $Z = 4$

 Cu $K\alpha$ radiation

 $\mu = 0.88$ mm⁻¹
 $T = 150$ K

 $0.40 \times 0.20 \times 0.20$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with Onyx (Nova) detector

 Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2006)

 $T_{\min} = 0.719$, $T_{\max} = 0.843$

11941 measured reflections

2199 independent reflections

 2165 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.069$
 $S = 1.07$

2199 reflections

171 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.22$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.14$ e Å⁻³

Absolute structure: Flack (1983), 883 Friedel pairs

Flack parameter: 0.13 (15)

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{O4}-\text{H4}\cdots\text{O5}$ | 0.84 | 1.87 | 2.6040 (13) | 145 |
| $\text{O2}-\text{H2}\cdots\text{O5}^i$ | 0.84 | 1.95 | 2.7851 (13) | 172 |
| $\text{O3}-\text{H3}\cdots\text{O2}^{ii}$ | 0.84 | 2.15 | 2.8942 (14) | 148 |

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2006); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5104).

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supporting information

Acta Cryst. (2011). E67, o1675 [doi:10.1107/S1600536811021751]

3,6,8-Trihydroxy-3,4,5,7-tetramethyl-3,4-dihydroisocoumarin

Yi-Wen Tao and Yun Wang

S1. Comment

The title compound, $C_{13}H_{16}O_5$ (I), also known as sclerotinin A, was isolated from culture filtrate of the endophytic fungus *phomopsis sp* from mangrove trees from the coast of the South China Sea. Herewith we present the structure of sclerotinin A.

The crystal structure of the title compound is shown in Fig. 1. The title compound crystallizes in orthorhombic cell setting $P2(1)2(1)2(1)$ space group, containing four formula units in the unit cell. As can be found,

In (I), all non-H atoms, except C1, C2, C4 and O2, are coplanar with the mean deviation of 0.039 (2) Å. The C2/C3/C5/C6/C7/O1 heterocycle exhibits an envelope configuration with C2 out of the plane formed by the other five atoms 0.308 (2) Å. All the bond lengths and angles are comparable with those found in similar compounds reported previously (Wang *et al.*, 2003; Krohn *et al.*, 1997).

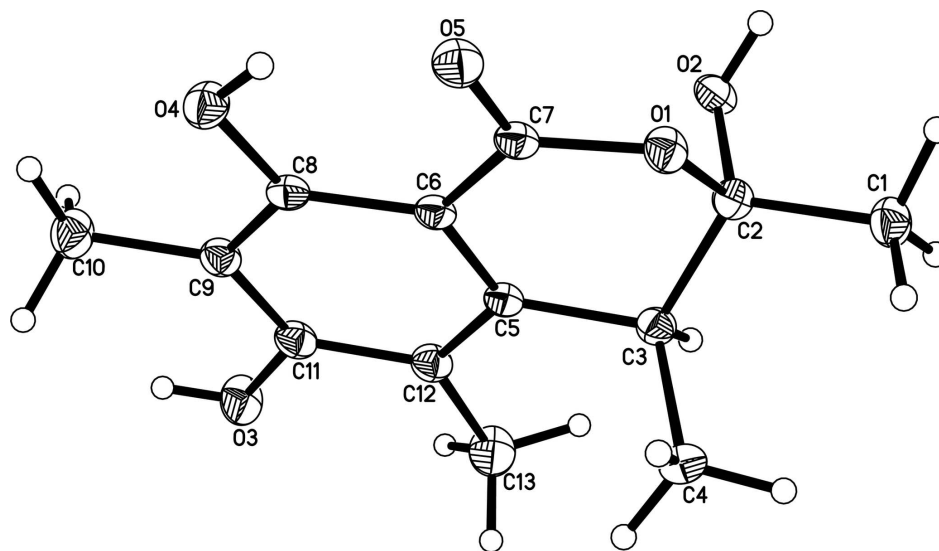
In the crystal structure, intermolecular O—H \cdots O hydrogen bonds (Table 1) generate three-dimensional network, which consolidate the crystal packing.

S2. Experimental

A strain of the fungus *phomopsis sp* was isolated from the mangrove tree, Zhanjiang, and was stored at the Department of Applied Chemistry, Zhongshan University, Guangzhou, China. Starter cultures (from Professor Shining Zhou) were maintained on cornmeal seawater agar. Plugs of agar supporting mycelial growth were cut and transferred aseptically to a 500 ml Erlenmeyer flask containing 300 ml of liquid medium (glucose 1%, peptone 0.2%, yeast extract 0.1%, NaCl 0.25%, pH 7.0). The flask was incubated at 303 K on a rotary shaker for 3 days. The mycelium was aseptically incubated at 303 K for 5 weeks. The cultures (130 L) were filtered through cheesecloth. The filtrate was concentrated to 3 L below 328 K and extracted several times by shaking with twofold volume of the ethyl acetate. The combined extracts were concentrated and chromatographed on silica gel using a gradient elution from petroleum to ethyl acetate, obtaining compound sclerotinin A from the 30% ethyl acetate/petroleum ether fraction. Crystals of the title compound suitable for single-crystal X-ray diffraction analysis were obtained by recrystallization from methanol.

S3. Refinement

All the H atoms were geometrically positioned (C—H 0.98 Å, O—H 0.84 Å), and were refined as riding, with $U_{iso}(H) = 1.2-1.5 U_{eq}(C, O)$.

**Figure 1**

The molecular structure of (I) with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

3,6,8-Trihydroxy-3,4,5,7-tetramethyl-3,4-dihydroisocoumarin

Crystal data

$C_{13}H_{16}O_5$

$M_r = 252.26$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 7.4731$ (3) Å

$b = 9.9742$ (7) Å

$c = 16.4066$ (12) Å

$V = 1222.92$ (13) Å³

$Z = 4$

$F(000) = 536$

$D_x = 1.370$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å

Cell parameters from 1678 reflections

$\theta = 5.2$ – 68.2°

$\mu = 0.88$ mm⁻¹

$T = 150$ K

Block, colourless

$0.40 \times 0.20 \times 0.20$ mm

Data collection

Oxford Diffraction Xcalibur

diffractometer with Onyx (Nova) detector

Radiation source: Nova (Cu) X-ray Source

Mirror monochromator

Detector resolution: 8.2417 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

(*CrysAlis PRO*; Oxford Diffraction, 2006)

$T_{\min} = 0.719$, $T_{\max} = 0.843$

11941 measured reflections

2199 independent reflections

2165 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.029$

$\theta_{\max} = 68.2^\circ$, $\theta_{\min} = 5.2^\circ$

$h = -7 \rightarrow 8$

$k = -11 \rightarrow 11$

$l = -19 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.025$

$wR(F^2) = 0.069$

$S = 1.07$

2199 reflections

171 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

$$w = 1/[\sigma^2(F_o^2) + (0.0427P)^2 + 0.1914P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

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

$$\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.14 \text{ e } \text{\AA}^{-3}$$

Extinction correction: *SHELXL*,

$$F_c^* = kF_c [1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$$

Extinction coefficient: 0.0039 (7)

Absolute structure: Flack (1983), 883 Friedel pairs

Absolute structure parameter: 0.13 (15)

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> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|-------------|----------------------------------|
| O1 | 0.50038 (12) | 0.41865 (9) | 0.52036 (5) | 0.0280 (2) |
| O2 | 0.26458 (13) | 0.35748 (9) | 0.43613 (5) | 0.0295 (2) |
| H2 | 0.2619 | 0.2802 | 0.4564 | 0.044* |
| O5 | 0.77901 (13) | 0.38899 (10) | 0.48436 (6) | 0.0319 (2) |
| C8 | 0.74508 (17) | 0.54758 (12) | 0.34132 (7) | 0.0242 (3) |
| O3 | 0.53133 (15) | 0.79291 (11) | 0.20856 (6) | 0.0397 (3) |
| H3 | 0.6232 | 0.7996 | 0.1793 | 0.060* |
| O4 | 0.90385 (13) | 0.48275 (9) | 0.34758 (6) | 0.0319 (2) |
| H4 | 0.9037 | 0.4346 | 0.3896 | 0.048* |
| C9 | 0.72242 (18) | 0.63290 (12) | 0.27480 (7) | 0.0273 (3) |
| C6 | 0.60748 (17) | 0.53081 (12) | 0.39893 (7) | 0.0228 (3) |
| C12 | 0.42314 (18) | 0.69223 (13) | 0.32709 (8) | 0.0280 (3) |
| C7 | 0.63436 (17) | 0.44348 (12) | 0.46828 (7) | 0.0244 (3) |
| C11 | 0.56276 (19) | 0.70428 (13) | 0.26991 (8) | 0.0284 (3) |
| C5 | 0.44607 (16) | 0.60298 (12) | 0.39092 (7) | 0.0231 (3) |
| C3 | 0.30879 (17) | 0.58956 (12) | 0.45805 (7) | 0.0253 (3) |
| H3A | 0.1876 | 0.6020 | 0.4333 | 0.030* |
| C4 | 0.3370 (2) | 0.70133 (14) | 0.52100 (9) | 0.0355 (3) |
| H4C | 0.4485 | 0.6850 | 0.5509 | 0.053* |
| H4B | 0.2364 | 0.7024 | 0.5593 | 0.053* |
| H4A | 0.3443 | 0.7880 | 0.4930 | 0.053* |
| C10 | 0.8647 (2) | 0.64783 (16) | 0.21033 (8) | 0.0379 (3) |
| H10A | 0.8158 | 0.6215 | 0.1573 | 0.057* |
| H10B | 0.9668 | 0.5903 | 0.2238 | 0.057* |
| H10C | 0.9041 | 0.7415 | 0.2080 | 0.057* |
| C2 | 0.31545 (17) | 0.44997 (13) | 0.49601 (7) | 0.0257 (3) |
| C1 | 0.20825 (19) | 0.43331 (14) | 0.57328 (8) | 0.0333 (3) |
| H1C | 0.2544 | 0.4943 | 0.6151 | 0.050* |
| H1B | 0.2184 | 0.3406 | 0.5925 | 0.050* |

| | | | | |
|------|------------|--------------|--------------|------------|
| H1A | 0.0823 | 0.4542 | 0.5624 | 0.050* |
| C13 | 0.2588 (2) | 0.77862 (17) | 0.31733 (10) | 0.0434 (4) |
| H13B | 0.1546 | 0.7315 | 0.3396 | 0.065* |
| H13A | 0.2390 | 0.7972 | 0.2594 | 0.065* |
| H13C | 0.2759 | 0.8633 | 0.3466 | 0.065* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|------------|------------|------------|-------------|-------------|-------------|
| O1 | 0.0301 (5) | 0.0298 (5) | 0.0240 (4) | 0.0021 (4) | -0.0010 (4) | 0.0057 (3) |
| O2 | 0.0414 (5) | 0.0225 (4) | 0.0245 (4) | -0.0063 (4) | -0.0003 (4) | 0.0013 (3) |
| O5 | 0.0311 (5) | 0.0304 (5) | 0.0341 (5) | 0.0052 (4) | -0.0028 (4) | 0.0079 (4) |
| C8 | 0.0268 (6) | 0.0197 (5) | 0.0262 (6) | 0.0001 (5) | -0.0028 (5) | -0.0033 (5) |
| O3 | 0.0429 (6) | 0.0429 (5) | 0.0332 (5) | 0.0073 (5) | 0.0039 (4) | 0.0184 (4) |
| O4 | 0.0303 (5) | 0.0333 (5) | 0.0322 (5) | 0.0061 (4) | 0.0016 (4) | 0.0053 (4) |
| C9 | 0.0333 (7) | 0.0245 (6) | 0.0240 (6) | -0.0027 (5) | 0.0005 (5) | -0.0006 (5) |
| C6 | 0.0287 (6) | 0.0180 (5) | 0.0218 (6) | -0.0013 (5) | -0.0018 (4) | -0.0017 (4) |
| C12 | 0.0319 (7) | 0.0245 (6) | 0.0278 (6) | 0.0014 (5) | -0.0017 (5) | 0.0031 (5) |
| C7 | 0.0299 (6) | 0.0195 (5) | 0.0239 (6) | -0.0007 (5) | -0.0021 (5) | -0.0017 (5) |
| C11 | 0.0364 (7) | 0.0235 (6) | 0.0253 (6) | -0.0007 (5) | -0.0022 (5) | 0.0042 (5) |
| C5 | 0.0259 (6) | 0.0193 (5) | 0.0240 (5) | -0.0013 (5) | -0.0020 (5) | -0.0015 (5) |
| C3 | 0.0271 (6) | 0.0229 (6) | 0.0259 (6) | 0.0018 (5) | -0.0001 (5) | 0.0017 (5) |
| C4 | 0.0423 (7) | 0.0277 (6) | 0.0364 (7) | 0.0009 (6) | 0.0059 (6) | -0.0067 (5) |
| C10 | 0.0398 (8) | 0.0414 (7) | 0.0326 (7) | 0.0026 (6) | 0.0068 (6) | 0.0077 (6) |
| C2 | 0.0281 (6) | 0.0263 (6) | 0.0226 (6) | -0.0004 (5) | -0.0016 (5) | -0.0002 (5) |
| C1 | 0.0375 (7) | 0.0366 (7) | 0.0259 (6) | -0.0019 (6) | 0.0041 (5) | 0.0035 (5) |
| C13 | 0.0406 (8) | 0.0431 (8) | 0.0464 (8) | 0.0119 (7) | 0.0008 (7) | 0.0166 (6) |

Geometric parameters (Å, °)

| | | | |
|---------|-------------|----------|-------------|
| O1—C7 | 1.3395 (15) | C5—C3 | 1.5111 (17) |
| O1—C2 | 1.4721 (16) | C3—C2 | 1.5260 (17) |
| O2—C2 | 1.4003 (15) | C3—C4 | 1.5343 (18) |
| O2—H2 | 0.8400 | C3—H3A | 1.0000 |
| O5—C7 | 1.2384 (16) | C4—H4C | 0.9800 |
| C8—O4 | 1.3552 (16) | C4—H4B | 0.9800 |
| C8—C9 | 1.3942 (17) | C4—H4A | 0.9800 |
| C8—C6 | 1.4067 (18) | C10—H10A | 0.9800 |
| O3—C11 | 1.3601 (15) | C10—H10B | 0.9800 |
| O3—H3 | 0.8400 | C10—H10C | 0.9800 |
| O4—H4 | 0.8400 | C2—C1 | 1.5088 (17) |
| C9—C11 | 1.3917 (19) | C1—H1C | 0.9800 |
| C9—C10 | 1.5073 (18) | C1—H1B | 0.9800 |
| C6—C5 | 1.4108 (18) | C1—H1A | 0.9800 |
| C6—C7 | 1.4469 (16) | C13—H13B | 0.9800 |
| C12—C5 | 1.3851 (17) | C13—H13A | 0.9800 |
| C12—C11 | 1.4082 (19) | C13—H13C | 0.9800 |
| C12—C13 | 1.509 (2) | | |

| | | | |
|--------------|--------------|---------------|--------------|
| C7—O1—C2 | 119.30 (9) | C3—C4—H4C | 109.5 |
| C2—O2—H2 | 109.5 | C3—C4—H4B | 109.5 |
| O4—C8—C9 | 117.19 (11) | H4C—C4—H4B | 109.5 |
| O4—C8—C6 | 122.16 (11) | C3—C4—H4A | 109.5 |
| C9—C8—C6 | 120.65 (11) | H4C—C4—H4A | 109.5 |
| C11—O3—H3 | 109.5 | H4B—C4—H4A | 109.5 |
| C8—O4—H4 | 109.5 | C9—C10—H10A | 109.5 |
| C11—C9—C8 | 117.48 (12) | C9—C10—H10B | 109.5 |
| C11—C9—C10 | 120.93 (11) | H10A—C10—H10B | 109.5 |
| C8—C9—C10 | 121.59 (12) | C9—C10—H10C | 109.5 |
| C8—C6—C5 | 120.11 (11) | H10A—C10—H10C | 109.5 |
| C8—C6—C7 | 119.90 (11) | H10B—C10—H10C | 109.5 |
| C5—C6—C7 | 119.94 (11) | O2—C2—O1 | 107.79 (10) |
| C5—C12—C11 | 117.83 (12) | O2—C2—C1 | 111.88 (10) |
| C5—C12—C13 | 123.22 (12) | O1—C2—C1 | 104.30 (10) |
| C11—C12—C13 | 118.93 (12) | O2—C2—C3 | 107.81 (9) |
| O5—C7—O1 | 115.81 (10) | O1—C2—C3 | 109.57 (10) |
| O5—C7—C6 | 123.57 (11) | C1—C2—C3 | 115.22 (11) |
| O1—C7—C6 | 120.61 (11) | C2—C1—H1C | 109.5 |
| O3—C11—C9 | 121.54 (12) | C2—C1—H1B | 109.5 |
| O3—C11—C12 | 114.87 (12) | H1C—C1—H1B | 109.5 |
| C9—C11—C12 | 123.58 (11) | C2—C1—H1A | 109.5 |
| C12—C5—C6 | 120.27 (11) | H1C—C1—H1A | 109.5 |
| C12—C5—C3 | 121.60 (11) | H1B—C1—H1A | 109.5 |
| C6—C5—C3 | 117.86 (10) | C12—C13—H13B | 109.5 |
| C5—C3—C2 | 110.86 (10) | C12—C13—H13A | 109.5 |
| C5—C3—C4 | 109.44 (10) | H13B—C13—H13A | 109.5 |
| C2—C3—C4 | 112.57 (11) | C12—C13—H13C | 109.5 |
| C5—C3—H3A | 107.9 | H13B—C13—H13C | 109.5 |
| C2—C3—H3A | 107.9 | H13A—C13—H13C | 109.5 |
| C4—C3—H3A | 107.9 | | |
| O4—C8—C9—C11 | -177.25 (11) | C11—C12—C5—C6 | 2.13 (18) |
| C6—C8—C9—C11 | 2.70 (17) | C13—C12—C5—C6 | -176.37 (13) |
| O4—C8—C9—C10 | 2.78 (17) | C11—C12—C5—C3 | 176.12 (11) |
| C6—C8—C9—C10 | -177.27 (11) | C13—C12—C5—C3 | -2.4 (2) |
| O4—C8—C6—C5 | 178.45 (10) | C8—C6—C5—C12 | -1.01 (17) |
| C9—C8—C6—C5 | -1.50 (17) | C7—C6—C5—C12 | 176.51 (11) |
| O4—C8—C6—C7 | 0.92 (17) | C8—C6—C5—C3 | -175.22 (10) |
| C9—C8—C6—C7 | -179.03 (11) | C7—C6—C5—C3 | 2.31 (16) |
| C2—O1—C7—O5 | -164.73 (11) | C12—C5—C3—C2 | 153.52 (11) |
| C2—O1—C7—C6 | 16.22 (16) | C6—C5—C3—C2 | -32.36 (14) |
| C8—C6—C7—O5 | 6.00 (18) | C12—C5—C3—C4 | -81.73 (14) |
| C5—C6—C7—O5 | -171.53 (11) | C6—C5—C3—C4 | 92.39 (13) |
| C8—C6—C7—O1 | -175.02 (10) | C7—O1—C2—O2 | 70.86 (13) |
| C5—C6—C7—O1 | 7.45 (17) | C7—O1—C2—C1 | -170.08 (10) |
| C8—C9—C11—O3 | 177.94 (11) | C7—O1—C2—C3 | -46.21 (14) |

| | | | |
|----------------|-------------|-------------|-------------|
| C10—C9—C11—O3 | -2.09 (19) | C5—C3—C2—O2 | -64.88 (12) |
| C8—C9—C11—C12 | -1.55 (19) | C4—C3—C2—O2 | 172.15 (11) |
| C10—C9—C11—C12 | 178.42 (12) | C5—C3—C2—O1 | 52.17 (12) |
| C5—C12—C11—O3 | 179.62 (11) | C4—C3—C2—O1 | -70.79 (13) |
| C13—C12—C11—O3 | -1.81 (19) | C5—C3—C2—C1 | 169.38 (11) |
| C5—C12—C11—C9 | -0.9 (2) | C4—C3—C2—C1 | 46.42 (15) |
| C13—C12—C11—C9 | 177.71 (13) | | |

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—H4...O5 | 0.84 | 1.87 | 2.6040 (13) | 145 |
| O2—H2...O5 ⁱ | 0.84 | 1.95 | 2.7851 (13) | 172 |
| O3—H3...O2 ⁱⁱ | 0.84 | 2.15 | 2.8942 (14) | 148 |

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