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# 3,3′-Dihydroxy-6,6′-bis(hydroxymethyl)-2,2′-(pentane-1,1-diyl)di-4*H*-pyran-4-one

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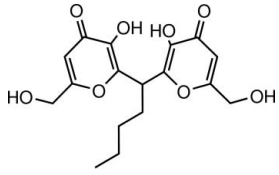
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.002 Å; disorder in main residue; $R$ factor = 0.043; $wR$ factor = 0.120; data-to-parameter ratio = 15.3.

In the title molecule, $C_{17}H_{20}O_8$, the two pyran rings form a dihedral angle of 61.2 (2)°. The two hydroxymethyl groups are each disordered over two sets of sites in a 0.764 (3):0.236 (3) ratio. In the crystal, O—H···O hydrogen bonds link the molecules into layers parallel to the *ac* plane.

## Related literature

For the biological properties of kojic acid, see: Kobayashi *et al.* (1995). For related structures, see: Nurchi *et al.* (2010); Kakkar & Singh (2011); Lokaj *et al.* (1991). For the preparation of the title compound, see: Barham & Nathan Reed (1938).



## Experimental

### Crystal data

$C_{17}H_{20}O_8$
$M_r$ = 352.33
Triclinic, $P\overline{1}$
$a$ = 6.4234 (3) Å
$b$ = 9.2394 (4) Å
$c$ = 15.8494 (7) Å
$\alpha$ = 79.993 (1)°
$\beta$ = 86.689 (2)°
$\gamma$ = 66.622 (1)°
$V$ = 850.22 (7) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 296 K
0.49 × 0.47 × 0.33 mm

### Data collection

Rigaku R-AXIS RAPID/ZJUG diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min}$ = 0.938, $T_{max}$ = 0.965
8442 measured reflections
3840 independent reflections
2907 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.017

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.120
$S$ = 1.00
3840 reflections
251 parameters
6 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.30 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3···O2$^{i}$ | 0.82 | 1.96 | 2.7275 (15) | 155 |
| O3—H3···O2 | 0.82 | 2.32 | 2.7440 (15) | 113 |
| O5—H5···O6$^{ii}$ | 0.82 | 2.00 | 2.7488 (16) | 151 |
| O5—H5···O6 | 0.82 | 2.32 | 2.7436 (16) | 113 |
| O4*A*—H4*A*···O2$^{iii}$ | 0.82 | 2.07 | 2.839 (2) | 157 |
| O8*A*—H8*A*···O6$^{iii}$ | 0.82 | 2.15 | 2.894 (2) | 152 |

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

Data collection: *PROCESS-AUTO* (Rigaku, 2006); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia,1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5255).

## References

Barham, H. N. & Nathan Reed, G. (1938). *J. Am. Chem. Soc.* **60**, 1541–1545.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Higashi, T. (1995). *ABSCOR*.Rigaku Corporation, Tokyo, Japan.
Kakkar, R. & Singh, C. (2011). *Int. J. Quantum Chem.* **111**, 4318–4329.
Kobayashi, Y., Kayahara, H., Tadasa, K., Nakamura, T. & Hiroshi, T. (1995). *Biosci. Biotech. Biochem.* **59**, 1745–1746.
Lokaj, J., Kožíšek, J., Koreň, B., Uher, M. & Vrábel, V. (1991). *Acta Cryst.* C**47**, 193–194.
Nurchi, V. M., Crisponi, G., Lachowicz, J. I., Murgia, S., Pivetta, T., Remelli, M., Rescigno, A., Niclos-Gutierrez, J., Gonzalez-Perez, J. M., Dominguez-Martin, A., Castineiras, A. & Szewczuk, K. (2010). *J. Inorg. Biochem.* **104**, 560–569.
Rigaku (2006). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku (2007). *CrystalStructure*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supplementary materials

*Acta Cryst.* (2012). E**68**, o1064 [doi:10.1107/S1600536812010276]

## 3,3′-Dihydroxy-6,6′-bis(hydroxymethyl)-2,2′-(pentane-1,1-diyl)di-4*H*-pyran-4-one

**Mu-Song Liu, Pan-Pan Hu and Tao Zhou**

### Comment

Kojic acid possesses appreciable inhibitory activity against tyrosinase, a key enzyme in the biosynthesis of melanin, due to its ability to chelate copper in the active site of this enzyme. Thus kojic acid inhibits the production of melanin pigment, consequently being used in cosmetic. Therefore, in an attempt to seek potent tyrosinase inhibitors, the derivatives of kojic acid have been widely investigated (Kobayashi *et al.*, 1995).There were little attentation to crystal structure of kojic and derivatives.The similar crystal structure of Kojic acid have been reported on 5-hydroxy-2-(hydroxymenthyl) -4*H*-pyran-one (Lokaj *et al.*, 1991; Kakkar *et al.*, 2011) and 6,6′-methylenebis (5-hydroxy-2-(hydroxymethyl)-4*H*-pyran-4-one) (Nurchi *et al.*, 2010). Herein, we report the crystal structure of the title compound (I).

In (I) (Fig. 1), two pyranone rings are planar forming the dihedral angle of 61.2 (2)°. Hydroxyl groups are almost coplanar with their linked pyranone rings forming the torsion angles O2—C3—C4—O3 of -0.10° and O6—C10—C9—O5 of 1.24°.

Intermolecular hydrogen bonds O3—H3···O2$^{i}$ and O5—H5···O6$^{ii}$ (Table 1) link molecules into zigzag chains along the *c* axis. Further, intermolecular hydrogen bonds O4A—H4···O2$^{iii}$ and O8A—H8A···O6$^{iii}$ (Table 1) link all of the components of the structure into layers parallel to *ac* plane.

### Experimental

To a solution of 5-hydroxy-(2-hydroxymethyl)-4*H*-pyran-4-one (kojic acid) (1.42 g, 10 mmol), sodium carbonate (1.06 g, 10 mmol) in water (10 ml) and methanol (10 ml) was added pentanal (10 mmol) at 343k with stirring. The stirring was continued for 3 h at that temperature. After removal of about half volume of the solvent, the soltion was neutrilized to pH=1 with concentrated hydrochloride. The crude product was obtained by filtration as an off-white solid (1.32, 75%), which was recrystallized from dichloromethane solution, giving colorless crystals of the title compound suitable for X-ray diffraction. 1H NMR (400 MHz DMSO) / d 0.84 (t, J = 6.8 Hz, 3H), 1.21 (m, 4H), 1.29 (2, m), 1.92 (m, 2H), 4.28 (s, 4H), 4.68 (t, J = 8.0 Hz, 1H), 5.62 (s, 2H), 6.28 (s, 2H), 9.03 (s, 2H).

### Refinement

H atoms were placed in calculated positions with O—H = 0.82 and C—H = 0.93–0.98 Å, and included in the refinement in riding model, with $U_{iso}$(H)= 1.2 - 1.5 $U_{eq}$(carrier atom). Atoms O4 and O8 of hydroxyl groups were treated as disordered over two positions - A and B, respectively - with the occupancies refined to 0.764 (3) and 0.236 (3), respectively.

### Computing details

Data collection: *PROCESS-AUTO* (Rigaku, 2006); cell refinement: *PROCESS-AUTO* (Rigaku, 2006); data reduction: *CrystalStructure* (Rigaku, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia,1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

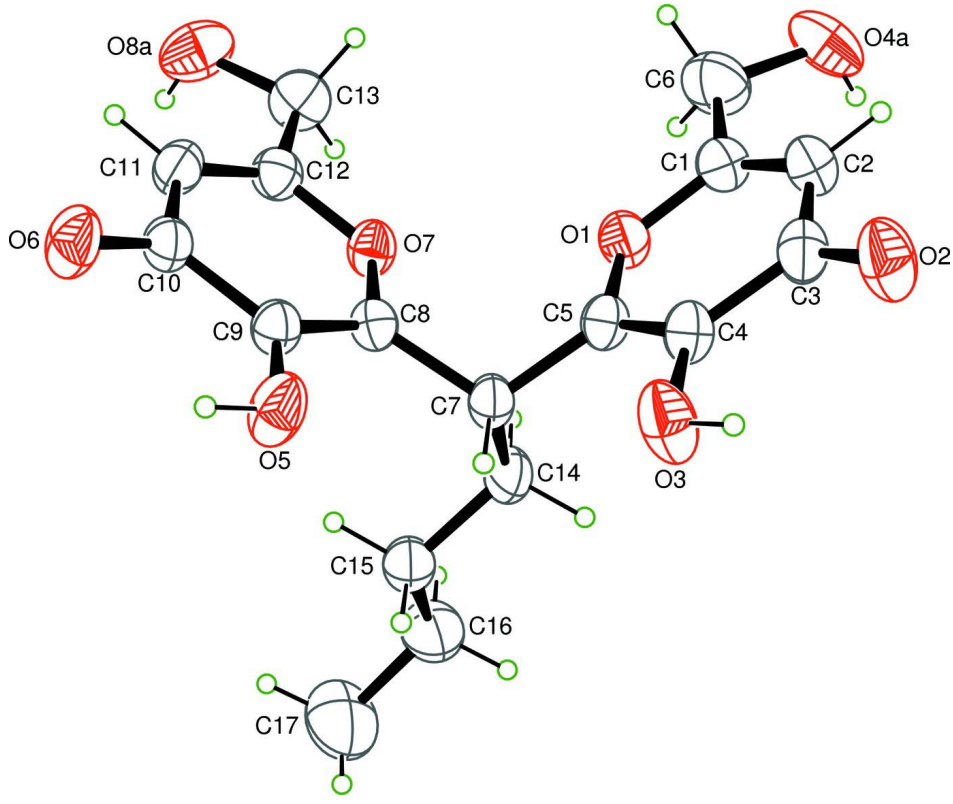


**Figure 1**

Molecular structure of (I) showing atom-labelling scheme and displacement ellipsoids at 50% probability level. Only major components of the disordered groups are shown.

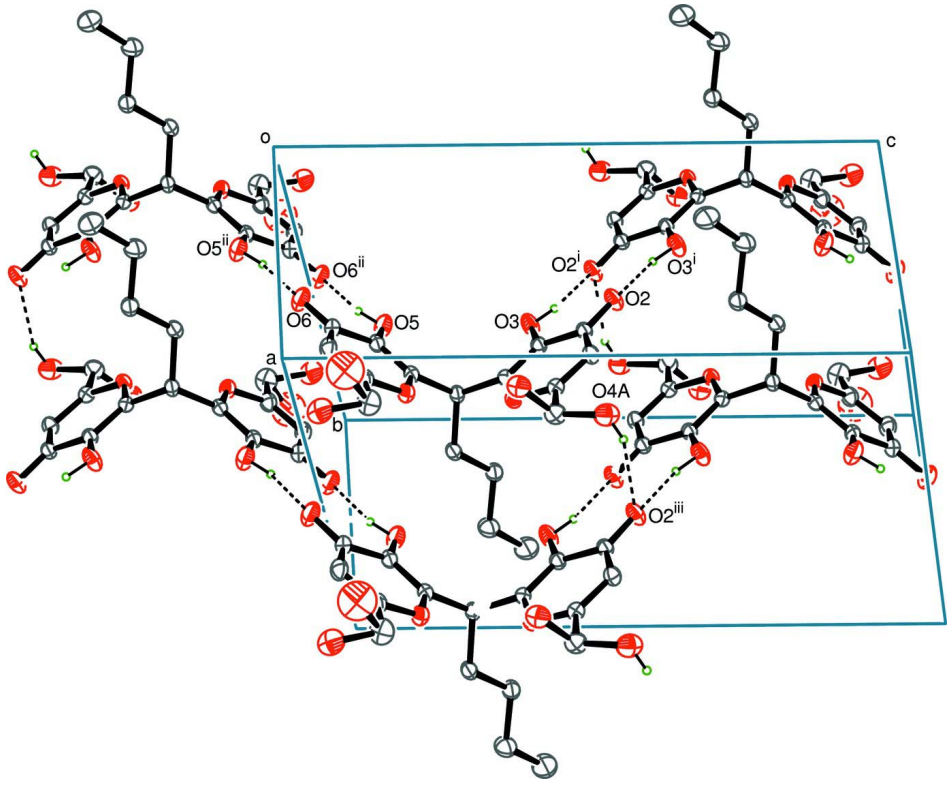


**Figure 2**

A portion of the crystal packing of (I) with hydrogen bonds shown by dashed lines [symmetry codes: (i) -*x*, 1 - *y*, -*z*; (ii) -*x*, 1 - *y*, 1 - *z*; (iii) 1 + *x*, *y*, *z*]. H atoms not involved in hydrogen bonding have been omitted for clarity.

### 3,3′-Dihydroxy-6,6′-bis(hydroxymethyl)-2,2′-(pentane-1,1-diyl)di- 4*H*-pyran-4-one

*Crystal data*

$C_{17}H_{20}O_8$
$M_r$ = 352.33
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a$ = 6.4234 (3) Å
$b$ = 9.2394 (4) Å
$c$ = 15.8494 (7) Å
$\alpha$ = 79.993 (1)°
$\beta$ = 86.689 (2)°
$\gamma$ = 66.622 (1)°
$V$ = 850.22 (7) Å$^3$

$Z$ = 2
$F$(000) = 372
$D_x$ = 1.376 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 6451 reflections
$\theta$ = 3.3–27.4°
$\mu$ = 0.11 mm$^{-1}$
$T$ = 296 K
Chunk, yellow
0.49 × 0.47 × 0.33 mm

*Data collection*

Rigaku R-AXIS RAPID/ZJUG diffractometer
Radiation source: rotating anode
Graphite monochromator
Detector resolution: 10.00 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min}$ = 0.938, $T_{max}$ = 0.965

8442 measured reflections
3840 independent reflections
2907 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.017
$\theta_{max}$ = 27.4°, $\theta_{min}$ = 3.3°
$h$ = −8→8
$k$ = −11→10
$l$ = −20→20

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.120$
$S = 1.00$
3840 reflections
251 parameters
6 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
$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 0.2871P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å$^{-3}$
$\Delta\rho_{min} = -0.30$ 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| O1 | 0.70599 (16) | 0.37814 (12) | 0.33797 (6) | 0.0343 (2) | |
| O2 | 0.24351 (19) | 0.37234 (15) | 0.52086 (7) | 0.0489 (3) | |
| O3 | 0.10419 (18) | 0.56659 (15) | 0.36549 (7) | 0.0499 (3) | |
| H3 | 0.0303 | 0.5617 | 0.4091 | 0.075* | |
| O5 | 0.10800 (19) | 0.56285 (16) | 0.11434 (8) | 0.0534 (3) | |
| H5 | 0.0366 | 0.5594 | 0.0740 | 0.080* | |
| O6 | 0.2475 (2) | 0.35803 (17) | −0.00299 (8) | 0.0562 (3) | |
| O7 | 0.70230 (17) | 0.35409 (12) | 0.16422 (6) | 0.0381 (3) | |
| O4A | 1.1115 (3) | 0.1209 (2) | 0.50117 (12) | 0.0591 (5) | 0.764 (3) |
| H4A | 1.1380 | 0.1883 | 0.5209 | 0.089* | 0.764 (3) |
| O8A | 1.1284 (3) | 0.0846 (2) | 0.04825 (13) | 0.0635 (6) | 0.764 (3) |
| H8A | 1.1580 | 0.1543 | 0.0169 | 0.095* | 0.764 (3) |
| O4B | 1.0634 (8) | 0.0558 (6) | 0.3693 (3) | 0.0474 (14) | 0.236 (3) |
| H4B | 1.0161 | −0.0084 | 0.3968 | 0.071* | 0.236 (3) |
| O8B | 1.061 (2) | −0.0030 (16) | 0.0999 (9) | 0.153 (5) | 0.236 (3) |
| H8B | 1.0323 | 0.0071 | 0.0490 | 0.230* | 0.236 (3) |
| C1 | 0.7734 (2) | 0.28416 (17) | 0.41491 (9) | 0.0348 (3) | |
| C2 | 0.6275 (3) | 0.27937 (18) | 0.47792 (9) | 0.0375 (3) | |
| H2 | 0.6811 | 0.2135 | 0.5301 | 0.045* | |
| C3 | 0.3903 (2) | 0.37414 (17) | 0.46608 (9) | 0.0347 (3) | |
| C4 | 0.3252 (2) | 0.47378 (17) | 0.38243 (9) | 0.0329 (3) | |
| C5 | 0.4816 (2) | 0.47156 (16) | 0.32159 (8) | 0.0302 (3) | |
| C6 | 1.0238 (3) | 0.1844 (2) | 0.41614 (11) | 0.0474 (4) | |
| H6A | 1.0525 | 0.0971 | 0.3848 | 0.057* | 0.764 (3) |
| H6B | 1.1022 | 0.2494 | 0.3873 | 0.057* | 0.764 (3) |
| H6C | 1.1034 | 0.2466 | 0.3877 | 0.057* | 0.236 (3) |

| | | | | | |
|---|---|---|---|---|---|
| H6D | 1.0788 | 0.1461 | 0.4755 | 0.057* | 0.236 (3) |
| C7 | 0.4372 (2) | 0.56545 (17) | 0.23201 (8) | 0.0331 (3) | |
| H7 | 0.2759 | 0.6362 | 0.2279 | 0.040* | |
| C8 | 0.4808 (2) | 0.45696 (17) | 0.16640 (8) | 0.0332 (3) | |
| C9 | 0.3266 (3) | 0.46005 (18) | 0.11099 (9) | 0.0363 (3) | |
| C10 | 0.3925 (3) | 0.35414 (19) | 0.04794 (9) | 0.0400 (3) | |
| C11 | 0.6259 (3) | 0.24898 (19) | 0.04981 (10) | 0.0437 (4) | |
| H11 | 0.6790 | 0.1780 | 0.0109 | 0.052* | |
| C12 | 0.7702 (3) | 0.25138 (18) | 0.10724 (10) | 0.0407 (4) | |
| C13 | 1.0161 (3) | 0.1441 (2) | 0.12089 (14) | 0.0593 (5) | |
| H13A | 1.0933 | 0.2027 | 0.1416 | 0.071* | 0.764 (3) |
| H13B | 1.0285 | 0.0544 | 0.1654 | 0.071* | 0.764 (3) |
| H13C | 1.1133 | 0.1895 | 0.0883 | 0.071* | 0.236 (3) |
| H13D | 1.0587 | 0.1215 | 0.1812 | 0.071* | 0.236 (3) |
| C14 | 0.5730 (3) | 0.67220 (18) | 0.21157 (9) | 0.0396 (3) | |
| H14A | 0.7336 | 0.6051 | 0.2178 | 0.047* | |
| H14B | 0.5444 | 0.7244 | 0.1523 | 0.047* | |
| C15 | 0.5152 (3) | 0.79877 (19) | 0.26837 (10) | 0.0421 (4) | |
| H15A | 0.5501 | 0.7469 | 0.3275 | 0.050* | |
| H15B | 0.3537 | 0.8638 | 0.2640 | 0.050* | |
| C16 | 0.6444 (4) | 0.9060 (2) | 0.24434 (13) | 0.0565 (5) | |
| H16A | 0.8057 | 0.8408 | 0.2504 | 0.068* | |
| H16B | 0.6136 | 0.9542 | 0.1845 | 0.068* | |
| C17 | 0.5856 (5) | 1.0365 (3) | 0.29752 (16) | 0.0846 (8) | |
| H17A | 0.4309 | 1.1097 | 0.2863 | 0.127* | |
| H17B | 0.6839 | 1.0927 | 0.2830 | 0.127* | |
| H17C | 0.6046 | 0.9906 | 0.3572 | 0.127* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0301 (5) | 0.0425 (6) | 0.0298 (5) | −0.0143 (4) | 0.0034 (4) | −0.0054 (4) |
| O2 | 0.0423 (6) | 0.0612 (7) | 0.0361 (6) | −0.0176 (6) | 0.0120 (5) | −0.0004 (5) |
| O3 | 0.0323 (6) | 0.0651 (8) | 0.0361 (6) | −0.0075 (5) | 0.0071 (4) | 0.0030 (5) |
| O5 | 0.0355 (6) | 0.0775 (9) | 0.0462 (7) | −0.0136 (6) | −0.0004 (5) | −0.0285 (6) |
| O6 | 0.0540 (7) | 0.0746 (9) | 0.0452 (7) | −0.0240 (6) | −0.0054 (5) | −0.0247 (6) |
| O7 | 0.0378 (6) | 0.0412 (6) | 0.0340 (5) | −0.0127 (5) | 0.0010 (4) | −0.0101 (4) |
| O4A | 0.0509 (10) | 0.0545 (10) | 0.0667 (11) | −0.0207 (8) | −0.0180 (8) | 0.0091 (8) |
| O8A | 0.0518 (10) | 0.0561 (11) | 0.0838 (14) | −0.0127 (9) | 0.0167 (9) | −0.0404 (10) |
| O4B | 0.038 (3) | 0.037 (3) | 0.062 (3) | −0.008 (2) | 0.009 (2) | −0.014 (2) |
| O8B | 0.153 (5) | 0.153 (5) | 0.154 (5) | −0.060 (2) | 0.0009 (10) | −0.0263 (13) |
| C1 | 0.0354 (7) | 0.0357 (7) | 0.0338 (7) | −0.0140 (6) | −0.0016 (6) | −0.0066 (6) |
| C2 | 0.0399 (8) | 0.0384 (8) | 0.0306 (7) | −0.0136 (6) | −0.0006 (6) | −0.0007 (6) |
| C3 | 0.0393 (8) | 0.0364 (7) | 0.0302 (7) | −0.0170 (6) | 0.0054 (6) | −0.0066 (6) |
| C4 | 0.0318 (7) | 0.0363 (7) | 0.0301 (7) | −0.0130 (6) | 0.0024 (5) | −0.0058 (6) |
| C5 | 0.0312 (7) | 0.0334 (7) | 0.0282 (6) | −0.0141 (6) | 0.0012 (5) | −0.0071 (5) |
| C6 | 0.0351 (8) | 0.0514 (9) | 0.0512 (9) | −0.0134 (7) | −0.0007 (7) | −0.0052 (8) |
| C7 | 0.0350 (7) | 0.0381 (7) | 0.0256 (6) | −0.0136 (6) | 0.0025 (5) | −0.0064 (5) |
| C8 | 0.0349 (7) | 0.0377 (7) | 0.0274 (6) | −0.0157 (6) | 0.0044 (5) | −0.0046 (6) |
| C9 | 0.0369 (8) | 0.0450 (8) | 0.0283 (7) | −0.0179 (7) | 0.0036 (6) | −0.0067 (6) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C10 | 0.0471 (9) | 0.0484 (9) | 0.0293 (7) | −0.0235 (7) | 0.0023 (6) | −0.0082 (6) |
| C11 | 0.0541 (10) | 0.0420 (8) | 0.0358 (8) | −0.0171 (7) | 0.0047 (7) | −0.0140 (7) |
| C12 | 0.0460 (9) | 0.0372 (8) | 0.0362 (8) | −0.0134 (7) | 0.0047 (6) | −0.0077 (6) |
| C13 | 0.0489 (10) | 0.0527 (10) | 0.0658 (12) | −0.0064 (8) | 0.0031 (9) | −0.0169 (9) |
| C14 | 0.0500 (9) | 0.0420 (8) | 0.0309 (7) | −0.0233 (7) | 0.0081 (6) | −0.0063 (6) |
| C15 | 0.0520 (9) | 0.0409 (8) | 0.0358 (8) | −0.0207 (7) | 0.0033 (7) | −0.0075 (6) |
| C16 | 0.0716 (12) | 0.0488 (10) | 0.0576 (11) | −0.0329 (9) | 0.0096 (9) | −0.0106 (8) |
| C17 | 0.129 (2) | 0.0774 (15) | 0.0786 (15) | −0.0695 (16) | 0.0189 (15) | −0.0289 (12) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.3502 (17) | C6—H6B | 0.9700 |
| O1—C5 | 1.3669 (16) | C6—H6C | 0.9553 |
| O2—C3 | 1.2474 (17) | C6—H6D | 0.9795 |
| O3—C4 | 1.3492 (17) | C7—C8 | 1.5069 (19) |
| O3—H3 | 0.8200 | C7—C14 | 1.542 (2) |
| O5—C9 | 1.3518 (18) | C7—H7 | 0.9800 |
| O5—H5 | 0.8200 | C8—C9 | 1.350 (2) |
| O6—C10 | 1.2531 (19) | C9—C10 | 1.448 (2) |
| O7—C12 | 1.3529 (18) | C10—C11 | 1.426 (2) |
| O7—C8 | 1.3641 (17) | C11—C12 | 1.345 (2) |
| O4A—C6 | 1.424 (2) | C11—H11 | 0.9300 |
| O4A—H4A | 0.8200 | C12—C13 | 1.499 (2) |
| O4A—H6D | 0.4491 | C13—H13A | 0.9700 |
| O8A—C13 | 1.399 (3) | C13—H13B | 0.9700 |
| O8A—H8A | 0.8200 | C13—H13C | 0.9635 |
| O8A—H13C | 1.2178 | C13—H13D | 0.9733 |
| O4B—C6 | 1.439 (5) | C14—C15 | 1.516 (2) |
| O4B—H4B | 0.8200 | C14—H14A | 0.9700 |
| O8B—C13 | 1.369 (13) | C14—H14B | 0.9700 |
| O8B—H8B | 0.8200 | C15—C16 | 1.517 (2) |
| C1—C2 | 1.337 (2) | C15—H15A | 0.9700 |
| C1—C6 | 1.503 (2) | C15—H15B | 0.9700 |
| C2—C3 | 1.428 (2) | C16—C17 | 1.504 (3) |
| C2—H2 | 0.9300 | C16—H16A | 0.9700 |
| C3—C4 | 1.4541 (19) | C16—H16B | 0.9700 |
| C4—C5 | 1.3484 (19) | C17—H17A | 0.9600 |
| C5—C7 | 1.5073 (19) | C17—H17B | 0.9600 |
| C6—H6A | 0.9700 | C17—H17C | 0.9600 |
| | | | |
| C1—O1—C5 | 120.29 (11) | O6—C10—C11 | 124.42 (15) |
| C4—O3—H3 | 109.5 | O6—C10—C9 | 120.11 (15) |
| C9—O5—H5 | 109.5 | C11—C10—C9 | 115.47 (13) |
| C12—O7—C8 | 120.36 (12) | C12—C11—C10 | 120.66 (14) |
| C6—O4A—H4A | 109.5 | C12—C11—H11 | 119.7 |
| H4A—O4A—H6D | 104.7 | C10—C11—H11 | 119.7 |
| C13—O8A—H8A | 109.5 | C11—C12—O7 | 121.98 (14) |
| H8A—O8A—H13C | 70.3 | C11—C12—C13 | 127.48 (15) |
| C6—O4B—H4B | 109.5 | O7—C12—C13 | 110.50 (14) |
| C13—O8B—H8B | 109.5 | O8B—C13—O8A | 52.8 (6) |

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| C2—C1—O1 | 122.32 (13) | O8B—C13—C12 | 111.3 (6) |
| C2—C1—C6 | 126.71 (14) | O8A—C13—C12 | 115.07 (17) |
| O1—C1—C6 | 110.89 (13) | O8B—C13—H13A | 140.2 |
| C1—C2—C3 | 120.82 (13) | O8A—C13—H13A | 108.5 |
| C1—C2—H2 | 119.6 | C12—C13—H13A | 108.5 |
| C3—C2—H2 | 119.6 | O8B—C13—H13B | 59.8 |
| O2—C3—C2 | 124.45 (13) | O8A—C13—H13B | 108.5 |
| O2—C3—C4 | 120.46 (13) | C12—C13—H13B | 108.5 |
| C2—C3—C4 | 115.07 (12) | H13A—C13—H13B | 107.5 |
| O3—C4—C5 | 120.03 (12) | O8B—C13—H13C | 108.9 |
| O3—C4—C3 | 119.06 (12) | O8A—C13—H13C | 58.6 |
| C5—C4—C3 | 120.89 (13) | C12—C13—H13C | 112.0 |
| C4—C5—O1 | 120.60 (12) | H13A—C13—H13C | 53.5 |
| C4—C5—C7 | 126.41 (13) | H13B—C13—H13C | 139.0 |
| O1—C5—C7 | 112.99 (11) | O8B—C13—H13D | 104.3 |
| O4A—C6—O4B | 109.7 (3) | O8A—C13—H13D | 133.4 |
| O4A—C6—C1 | 111.96 (15) | C12—C13—H13D | 111.1 |
| O4B—C6—C1 | 108.5 (2) | H13A—C13—H13D | 60.5 |
| O4A—C6—H6A | 109.2 | H13B—C13—H13D | 48.6 |
| C1—C6—H6A | 109.2 | H13C—C13—H13D | 108.8 |
| O4A—C6—H6B | 109.2 | C15—C14—C7 | 113.65 (12) |
| O4B—C6—H6B | 108.1 | C15—C14—H14A | 108.8 |
| C1—C6—H6B | 109.2 | C7—C14—H14A | 108.8 |
| H6A—C6—H6B | 107.9 | C15—C14—H14B | 108.8 |
| O4A—C6—H6C | 109.0 | C7—C14—H14B | 108.8 |
| O4B—C6—H6C | 107.4 | H14A—C14—H14B | 107.7 |
| C1—C6—H6C | 110.2 | C14—C15—C16 | 112.37 (14) |
| H6A—C6—H6C | 107.1 | C14—C15—H15A | 109.1 |
| O4B—C6—H6D | 112.6 | C16—C15—H15A | 109.1 |
| C1—C6—H6D | 109.6 | C14—C15—H15B | 109.1 |
| H6A—C6—H6D | 112.1 | C16—C15—H15B | 109.1 |
| H6B—C6—H6D | 108.8 | H15A—C15—H15B | 107.9 |
| H6C—C6—H6D | 108.6 | C17—C16—C15 | 113.87 (17) |
| C5—C7—C8 | 111.20 (12) | C17—C16—H16A | 108.8 |
| C5—C7—C14 | 112.71 (12) | C15—C16—H16A | 108.8 |
| C8—C7—C14 | 110.77 (11) | C17—C16—H16B | 108.8 |
| C5—C7—H7 | 107.3 | C15—C16—H16B | 108.8 |
| C8—C7—H7 | 107.3 | H16A—C16—H16B | 107.7 |
| C14—C7—H7 | 107.3 | C16—C17—H17A | 109.5 |
| C9—C8—O7 | 120.75 (13) | C16—C17—H17B | 109.5 |
| C9—C8—C7 | 126.05 (13) | H17A—C17—H17B | 109.5 |
| O7—C8—C7 | 113.13 (12) | C16—C17—H17C | 109.5 |
| C8—C9—O5 | 119.76 (13) | H17A—C17—H17C | 109.5 |
| C8—C9—C10 | 120.76 (14) | H17B—C17—H17C | 109.5 |
| O5—C9—C10 | 119.48 (13) | | |

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| C5—O1—C1—C2 | −0.8 (2) | C5—C7—C8—C9 | 118.54 (16) |
| C5—O1—C1—C6 | 176.06 (12) | C14—C7—C8—C9 | −115.30 (16) |
| O1—C1—C2—C3 | 0.4 (2) | C5—C7—C8—O7 | −64.36 (15) |

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| C6—C1—C2—C3 | −175.94 (14) | C14—C7—C8—O7 | 61.80 (15) |
| C1—C2—C3—O2 | 177.73 (16) | O7—C8—C9—O5 | 179.93 (13) |
| C1—C2—C3—C4 | −0.6 (2) | C7—C8—C9—O5 | −3.2 (2) |
| O2—C3—C4—O3 | 1.3 (2) | O7—C8—C9—C10 | 0.1 (2) |
| C2—C3—C4—O3 | 179.63 (13) | C7—C8—C9—C10 | 176.96 (13) |
| O2—C3—C4—C5 | −177.25 (14) | C8—C9—C10—O6 | 179.85 (15) |
| C2—C3—C4—C5 | 1.1 (2) | O5—C9—C10—O6 | 0.0 (2) |
| O3—C4—C5—O1 | 179.94 (13) | C8—C9—C10—C11 | 0.5 (2) |
| C3—C4—C5—O1 | −1.5 (2) | O5—C9—C10—C11 | −179.41 (14) |
| O3—C4—C5—C7 | 0.1 (2) | O6—C10—C11—C12 | −179.32 (16) |
| C3—C4—C5—C7 | 178.57 (13) | C9—C10—C11—C12 | 0.0 (2) |
| C1—O1—C5—C4 | 1.4 (2) | C10—C11—C12—O7 | −1.1 (2) |
| C1—O1—C5—C7 | −178.71 (12) | C10—C11—C12—C13 | 176.58 (17) |
| C2—C1—C6—O4A | −20.6 (2) | C8—O7—C12—C11 | 1.7 (2) |
| O1—C1—C6—O4A | 162.71 (14) | C8—O7—C12—C13 | −176.37 (14) |
| C2—C1—C6—O4B | 100.7 (3) | C11—C12—C13—O8B | −29.2 (7) |
| O1—C1—C6—O4B | −76.1 (3) | O7—C12—C13—O8B | 148.7 (7) |
| C4—C5—C7—C8 | −111.66 (16) | C11—C12—C13—O8A | 28.5 (3) |
| O1—C5—C7—C8 | 68.45 (15) | O7—C12—C13—O8A | −153.56 (17) |
| C4—C5—C7—C14 | 123.26 (16) | C5—C7—C14—C15 | −61.92 (17) |
| O1—C5—C7—C14 | −56.62 (16) | C8—C7—C14—C15 | 172.77 (13) |
| C12—O7—C8—C9 | −1.1 (2) | C7—C14—C15—C16 | −177.67 (14) |
| C12—O7—C8—C7 | −178.40 (12) | C14—C15—C16—C17 | 178.09 (18) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···O2$^{i}$ | 0.82 | 1.96 | 2.7275 (15) | 155 |
| O3—H3···O2 | 0.82 | 2.32 | 2.7440 (15) | 113 |
| O5—H5···O6$^{ii}$ | 0.82 | 2.00 | 2.7488 (16) | 151 |
| O5—H5···O6 | 0.82 | 2.32 | 2.7436 (16) | 113 |
| O4*A*—H4*A*···O2$^{iii}$ | 0.82 | 2.07 | 2.839 (2) | 157 |
| O8*A*—H8*A*···O6$^{iii}$ | 0.82 | 2.15 | 2.894 (2) | 152 |

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