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5,7-Dihydroxy-6,4′-dimethoxyflavone

Ming-Yue Mou,[a] Ke Pi,[a] Qi-Long Zhang,[b] Yun-Qian Zhang[b] and Qian-Jun Zhang[c]*

[a]Research and Development Center of Fine Chemicals of Guizhou University, Guiyang 550025, People's Republic of China, [b]Key Laboratory of Macrocyclic and Supramolecular Chemistry of Guizhou Province, Guizhou University, Guiyang 550025, People's Republic of China, and [c]Science College of Guizhou University, Guiyang 550025, People's Republic of China
Correspondence e-mail: qianjunzhang@126.com

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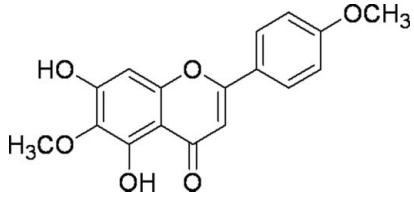
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.110; data-to-parameter ratio = 11.8.

In the title compound, $C_{17}H_{14}O_6$, the benzopyran ring system is essentially planar and forms a dihedral angle of 6.84 (4)° with the other benzene ring. In the crystal structure, centrosymmetrically related molecules are linked into dimers by O—H···O hydrogen bonds. The crystal packing is controlled by C—H···π and π–π stacking interactions involving the benzopyran and benzene rings, with centroid–centroid distances between 3.645 (2) and 3.986 (2) Å.

Related literature

For related literature, see: Guo *et al.* (2006); Wang & Cheng (2007); Wu *et al.* (2007).



Experimental

Crystal data

$C_{17}H_{14}O_6$
M_r = 314.28
Triclinic, $P\overline{1}$
a = 6.9115 (11) Å
b = 7.2583 (12) Å
c = 14.649 (2) Å
α = 82.739 (6)°
β = 88.424 (6)°
γ = 76.907 (6)°
V = 710.0 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 (2) K
0.18 × 0.12 × 0.09 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.980, T_{max} = 0.990
7597 measured reflections
2469 independent reflections
2095 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.110
S = 1.08
2469 reflections
209 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O2	0.82	1.85	2.5774 (15)	148
O5—H5···O4	0.82	2.28	2.7431 (15)	116
O5—H5···O4^{i}	0.82	2.29	2.8562 (15)	127
C1—H1B···$Cg3^{ii}$	0.96	2.93	3.799 (2)	150

Symmetry code: (i) $-x, -y+1, -z$; (ii) $-x, -y+2, -z+3$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (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: RZ2178).

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supporting information

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5,7-Dihydroxy-6,4′-dimethoxyflavone

Ming-Yue Mou, Ke Pi, Qi-Long Zhang, Yun-Qian Zhang and Qian-Jun Zhang

S1. Comment

Flavone compounds exhibit different physiological functions and activities (Wu *et al.*, 2007), such as antibacterial and antioxidative activities, and are useful in diminshing inflammation, relieving cough and dispelling phlegm. In these compounds, different structures having different conformations exhibit a wide range macroscopic physiological activities (Guo *et al.*, 2006; Wang *et al.*, 2007). The title compound, 6,4′-dimethoxy-5,7-dihydroxyflavone, which is a natural product extracted from Teucrium pilosum found in the Guizhou Province of China, effects on phenol red excretion volume of mouse trachea and on the ammonia-induced cough in mice.

In the title compound (Fig. 1), the benzopyran ring is essentially planar (maximum displacement 0.0258 (14) Å for atom C10) and forms a dihedral angle of 6.84 (4)° with the benzene ring C2—C7. The molecular conformation is stabilized by two O—H···O intramolecular hydrogen bonds (Table 1). Moreover, centrosymmetrically related molecules are linked into dimers by O—H···O hydrogen bonds (Table 1). In the crystal structure, $\pi\cdots\pi$ stacking interactions occur between adjacent rings, with centroid-centroid separations of 3.645 (2), 3.656 (2) and 3.986 (2) Å for $Cg1\cdots Cg2^{i}$, $Cg1\cdots Cg2^{ii}$ and $Cg2\cdots Cg3^{i}$ respectively (*Cg*1, *Cg*2 and *Cg*3 are the centroids of the O6/C8—C12, C2—C7 and C11—C16 rings; symmetry codes: (i) -*x*, 1 - *y*, 1 - *z*; (ii) -*x*, 2 - *y*, 1 - *z*). The structure is further stabilized by a C—H···π interaction (C1—H1B···$Cg(3)^{ii}$ = 150.3°; H1B···$Cg(3)^{ii}$ = 2.93 Å; C1···$Cg(3)^{ii}$ = 3.799 (2) Å).

S2. Experimental

30 kg of dried whole plant Teucrium pilosum was powdered and extracted with ethanol (120 *L*) three times at room temperature and the residue was separated after removing the solvent under vacuum. The residue was suspended in water and extracted with ethyl acetate and n-butanol respectively. The ethyl acetate fraction (4.5 kg) was subjected repeatedly to column chromatography on silica gel using petroleum with a gradient of ethyl acetate (0–100% EtOAc) to yield the title compound (916.3 mg). Single crystals suitable for X-ray diffraction analysis were obtained from an ether-$CHCl_3$ mixture (1:10 *v*/*v*) by slow evaporation of the solvent at room temperature.

S3. Refinement

All H atoms were placed in calculated positions with C—H = 0.93–0.96 Å, O—H = 0.82 Å, and refined using the riding model approximation, with U_{iso}(H) = 1.2 U_{eq}(C, O) or 1.5 U_{eq}(C) for methyl H atoms.

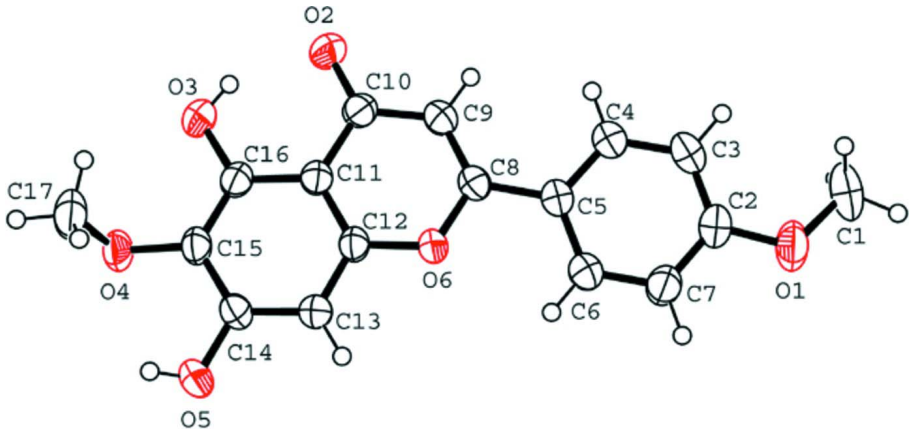

Figure 1

The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

5,7-Dihydroxy-6,4′-dimethoxyflavone

Crystal data

$C_{17}H_{14}O_6$
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Triclinic, $P\overline{1}$
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$\beta = 88.424$ (6)°
$\gamma = 76.907$ (6)°
$V = 710.0$ (2) Å^3
$Z = 2$
$F(000) = 328$
$D_x = 1.470$ Mg m^{-3}
Melting point: 224-226° C K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7597 reflections
$\theta = 1.4$–25.0°
$\mu = 0.11$ mm^{-1}
$T = 293$ K
Prism, colourless
0.18 × 0.12 × 0.09 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scan
Absorption correction: multi-scan *SADABS* (Bruker, 2005)
$T_{min} = 0.980$, $T_{max} = 0.990$
7597 measured reflections
2469 independent reflections
2095 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.4°$
$h = -8\rightarrow8$
$k = -8\rightarrow8$
$l = -17\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.110$
$S = 1.08$
2469 reflections
209 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
$w = 1/[\sigma^2(F_o^2) + (0.0576P)^2 + 0.1294P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

Extinction correction: *SHELXL*, $Fc^{*}=kFc[1+0.001xFc^{2}\lambda^{3}/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.036 (5)

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}
C1	−0.3004 (3)	0.9240 (3)	0.86019 (11)	0.0675 (5)
H1A	−0.3983	0.9715	0.9043	0.101*
H1B	−0.1984	0.9951	0.8563	0.101*
H1C	−0.2428	0.7918	0.8791	0.101*
C2	−0.2756 (2)	0.8843 (2)	0.70068 (10)	0.0444 (4)
C3	−0.0702 (2)	0.8314 (2)	0.70196 (10)	0.0476 (4)
H3	−0.0005	0.8308	0.7555	0.057*
C4	0.0309 (2)	0.7793 (2)	0.62275 (10)	0.0441 (4)
H4	0.1689	0.7443	0.6237	0.053*
C5	−0.0691 (2)	0.77829 (18)	0.54212 (9)	0.0361 (3)
C6	−0.2764 (2)	0.8273 (2)	0.54371 (10)	0.0443 (4)
H6	−0.3473	0.8244	0.4911	0.053*
C7	−0.3773 (2)	0.8797 (2)	0.62189 (11)	0.0494 (4)
H7	−0.5154	0.9123	0.6215	0.059*
C8	0.0387 (2)	0.72640 (18)	0.45786 (9)	0.0353 (3)
C9	0.2359 (2)	0.6569 (2)	0.44969 (10)	0.0416 (4)
H9	0.3163	0.6392	0.5014	0.050*
C10	0.3248 (2)	0.6095 (2)	0.36394 (10)	0.0398 (3)
C11	0.18831 (19)	0.63688 (18)	0.28746 (9)	0.0351 (3)
C12	−0.0140 (2)	0.71097 (19)	0.30008 (9)	0.0349 (3)
C13	−0.1511 (2)	0.7425 (2)	0.22975 (10)	0.0412 (4)
H13	−0.2849	0.7941	0.2395	0.049*
C14	−0.0826 (2)	0.6948 (2)	0.14458 (10)	0.0400 (4)
C15	0.1185 (2)	0.6180 (2)	0.12867 (9)	0.0392 (4)
C16	0.2539 (2)	0.5884 (2)	0.19985 (10)	0.0381 (3)
C17	0.2732 (3)	0.7014 (3)	−0.01218 (11)	0.0595 (5)
H17A	0.3096	0.6582	−0.0709	0.089*
H17B	0.3904	0.7090	0.0195	0.089*
H17C	0.1849	0.8250	−0.0211	0.089*
O1	−0.39076 (18)	0.94398 (18)	0.77344 (8)	0.0630 (4)
O2	0.50778 (15)	0.54639 (17)	0.35414 (7)	0.0547 (3)
O3	0.44741 (15)	0.51494 (17)	0.18454 (7)	0.0531 (3)
H3A	0.5105	0.5051	0.2322	0.080*

O4	0.17552 (15)	0.56933 (15)	0.04163 (7)	0.0475 (3)
O5	−0.21639 (15)	0.72526 (17)	0.07437 (7)	0.0550 (3)
H5	−0.1587	0.6919	0.0275	0.083*
O6	−0.08666 (13)	0.75600 (14)	0.38456 (6)	0.0384 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.1075 (16)	0.0602 (11)	0.0344 (9)	−0.0146 (10)	0.0078 (9)	−0.0132 (8)
C2	0.0545 (9)	0.0402 (8)	0.0368 (8)	−0.0072 (7)	0.0084 (7)	−0.0060 (6)
C3	0.0604 (10)	0.0488 (9)	0.0346 (8)	−0.0115 (7)	−0.0065 (7)	−0.0093 (6)
C4	0.0451 (8)	0.0491 (9)	0.0393 (8)	−0.0105 (7)	−0.0012 (6)	−0.0104 (6)
C5	0.0439 (8)	0.0322 (7)	0.0325 (7)	−0.0094 (6)	0.0009 (6)	−0.0040 (5)
C6	0.0447 (8)	0.0520 (9)	0.0354 (8)	−0.0092 (7)	−0.0015 (6)	−0.0057 (6)
C7	0.0448 (8)	0.0585 (10)	0.0414 (9)	−0.0043 (7)	0.0053 (7)	−0.0068 (7)
C8	0.0414 (8)	0.0336 (7)	0.0322 (7)	−0.0109 (6)	−0.0015 (6)	−0.0039 (5)
C9	0.0410 (8)	0.0495 (9)	0.0332 (8)	−0.0080 (6)	−0.0044 (6)	−0.0045 (6)
C10	0.0360 (8)	0.0430 (8)	0.0396 (8)	−0.0085 (6)	0.0014 (6)	−0.0036 (6)
C11	0.0374 (8)	0.0352 (7)	0.0335 (8)	−0.0099 (6)	0.0025 (6)	−0.0054 (6)
C12	0.0388 (7)	0.0364 (7)	0.0309 (7)	−0.0098 (6)	0.0041 (6)	−0.0082 (5)
C13	0.0349 (7)	0.0514 (9)	0.0379 (8)	−0.0072 (6)	0.0000 (6)	−0.0122 (6)
C14	0.0437 (8)	0.0465 (8)	0.0324 (8)	−0.0127 (6)	−0.0017 (6)	−0.0097 (6)
C15	0.0457 (8)	0.0430 (8)	0.0321 (8)	−0.0131 (6)	0.0064 (6)	−0.0119 (6)
C16	0.0374 (7)	0.0394 (8)	0.0385 (8)	−0.0095 (6)	0.0064 (6)	−0.0086 (6)
C17	0.0735 (11)	0.0701 (11)	0.0389 (9)	−0.0230 (9)	0.0171 (8)	−0.0130 (8)
O1	0.0702 (8)	0.0756 (8)	0.0383 (6)	−0.0025 (6)	0.0113 (5)	−0.0158 (5)
O2	0.0339 (6)	0.0805 (8)	0.0451 (7)	−0.0032 (5)	0.0010 (5)	−0.0082 (5)
O3	0.0380 (6)	0.0738 (8)	0.0454 (7)	−0.0040 (5)	0.0078 (5)	−0.0170 (5)
O4	0.0556 (7)	0.0591 (7)	0.0341 (6)	−0.0197 (5)	0.0098 (5)	−0.0196 (5)
O5	0.0479 (6)	0.0816 (8)	0.0360 (6)	−0.0076 (5)	−0.0052 (5)	−0.0203 (5)
O6	0.0362 (5)	0.0496 (6)	0.0294 (5)	−0.0064 (4)	0.0009 (4)	−0.0107 (4)

Geometric parameters (Å, º)

C1—O1	1.408 (2)	C10—O2	1.2545 (17)
C1—H1A	0.9600	C10—C11	1.4498 (19)
C1—H1B	0.9600	C11—C12	1.3959 (19)
C1—H1C	0.9600	C11—C16	1.409 (2)
C2—O1	1.3678 (18)	C12—O6	1.3727 (16)
C2—C7	1.376 (2)	C12—C13	1.3812 (19)
C2—C3	1.384 (2)	C13—C14	1.379 (2)
C3—C4	1.387 (2)	C13—H13	0.9300
C3—H3	0.9300	C14—O5	1.3651 (17)
C4—C5	1.387 (2)	C14—C15	1.400 (2)
C4—H4	0.9300	C15—C16	1.384 (2)
C5—C6	1.396 (2)	C15—O4	1.3890 (16)
C5—C8	1.470 (2)	C16—O3	1.3471 (17)
C6—C7	1.374 (2)	C17—O4	1.4383 (19)

C6—H6	0.9300	C17—H17A	0.9600
C7—H7	0.9300	C17—H17B	0.9600
C8—C9	1.349 (2)	C17—H17C	0.9600
C8—O6	1.3632 (16)	O3—H3A	0.8200
C9—C10	1.430 (2)	O5—H5	0.8200
C9—H9	0.9300		

O1—C1—H1A	109.5	O2—C10—C11	121.21 (13)
O1—C1—H1B	109.5	C9—C10—C11	115.46 (12)
H1A—C1—H1B	109.5	C12—C11—C16	118.45 (12)
O1—C1—H1C	109.5	C12—C11—C10	119.87 (12)
H1A—C1—H1C	109.5	C16—C11—C10	121.68 (13)
H1B—C1—H1C	109.5	O6—C12—C13	116.48 (12)
O1—C2—C7	115.54 (14)	O6—C12—C11	120.89 (12)
O1—C2—C3	124.67 (14)	C13—C12—C11	122.63 (13)
C7—C2—C3	119.78 (14)	C14—C13—C12	117.67 (13)
C2—C3—C4	119.42 (14)	C14—C13—H13	121.2
C2—C3—H3	120.3	C12—C13—H13	121.2
C4—C3—H3	120.3	O5—C14—C13	118.33 (13)
C5—C4—C3	121.51 (14)	O5—C14—C15	119.79 (13)
C5—C4—H4	119.2	C13—C14—C15	121.87 (13)
C3—C4—H4	119.2	C16—C15—O4	121.77 (13)
C4—C5—C6	117.71 (13)	C16—C15—C14	119.65 (13)
C4—C5—C8	121.32 (13)	O4—C15—C14	118.56 (12)
C6—C5—C8	120.97 (12)	O3—C16—C15	119.67 (13)
C7—C6—C5	121.01 (14)	O3—C16—C11	120.61 (13)
C7—C6—H6	119.5	C15—C16—C11	119.71 (13)
C5—C6—H6	119.5	O4—C17—H17A	109.5
C6—C7—C2	120.53 (15)	O4—C17—H17B	109.5
C6—C7—H7	119.7	H17A—C17—H17B	109.5
C2—C7—H7	119.7	O4—C17—H17C	109.5
C9—C8—O6	121.73 (13)	H17A—C17—H17C	109.5
C9—C8—C5	126.79 (13)	H17B—C17—H17C	109.5
O6—C8—C5	111.48 (11)	C2—O1—C1	118.62 (14)
C8—C9—C10	121.96 (13)	C16—O3—H3A	109.5
C8—C9—H9	119.0	C15—O4—C17	114.00 (11)
C10—C9—H9	119.0	C14—O5—H5	109.5
O2—C10—C9	123.33 (13)	C8—O6—C12	120.07 (11)

O1—C2—C3—C4	−177.85 (14)	C10—C11—C12—C13	179.21 (13)
C7—C2—C3—C4	1.7 (2)	O6—C12—C13—C14	−178.69 (12)
C2—C3—C4—C5	−0.2 (2)	C11—C12—C13—C14	1.1 (2)
C3—C4—C5—C6	−1.5 (2)	C12—C13—C14—O5	−179.95 (12)
C3—C4—C5—C8	178.85 (13)	C12—C13—C14—C15	−0.4 (2)
C4—C5—C6—C7	1.7 (2)	O5—C14—C15—C16	179.62 (13)
C8—C5—C6—C7	−178.62 (13)	C13—C14—C15—C16	0.1 (2)
C5—C6—C7—C2	−0.2 (2)	O5—C14—C15—O4	−1.9 (2)
O1—C2—C7—C6	178.11 (14)	C13—C14—C15—O4	178.50 (13)

C3—C2—C7—C6	−1.5 (2)	O4—C15—C16—O3	1.8 (2)
C4—C5—C8—C9	7.4 (2)	C14—C15—C16—O3	−179.86 (13)
C6—C5—C8—C9	−172.29 (14)	O4—C15—C16—C11	−178.83 (12)
C4—C5—C8—O6	−172.95 (12)	C14—C15—C16—C11	−0.4 (2)
C6—C5—C8—O6	7.41 (19)	C12—C11—C16—O3	−179.45 (12)
O6—C8—C9—C10	−0.2 (2)	C10—C11—C16—O3	−0.2 (2)
C5—C8—C9—C10	179.42 (13)	C12—C11—C16—C15	1.1 (2)
C8—C9—C10—O2	179.30 (14)	C10—C11—C16—C15	−179.60 (12)
C8—C9—C10—C11	−1.5 (2)	C7—C2—O1—C1	170.14 (15)
O2—C10—C11—C12	−178.72 (13)	C3—C2—O1—C1	−10.3 (2)
C9—C10—C11—C12	2.0 (2)	C16—C15—O4—C17	−77.04 (18)
O2—C10—C11—C16	2.0 (2)	C14—C15—O4—C17	104.56 (16)
C9—C10—C11—C16	−177.22 (12)	C9—C8—O6—C12	1.4 (2)
C16—C11—C12—O6	178.30 (12)	C5—C8—O6—C12	−178.27 (11)
C10—C11—C12—O6	−1.0 (2)	C13—C12—O6—C8	179.03 (12)
C16—C11—C12—C13	−1.5 (2)	C11—C12—O6—C8	−0.80 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O2	0.82	1.85	2.5774 (15)	148
O5—H5···O4	0.82	2.28	2.7431 (15)	116
O5—H5···O4^{i}	0.82	2.29	2.8562 (15)	127
C1—H1*B*···*Cg*3^{ii}	0.96	2.93	3.799 (2)	150

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