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# 5,6,7,5′-Tetramethoxy-3′,4′-methylenedioxyflavone monohydrate

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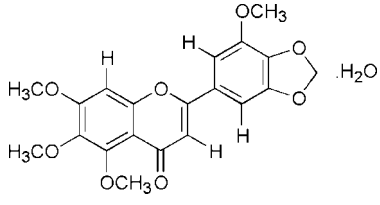
Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.040; $wR$ factor = 0.117; data-to-parameter ratio = 11.8.

The title compound [systematic name: 5,6,7-trimethoxy-2-(7-methoxy-1,3-dihydro-2-benzofuran-5-yl)-4*H*-chromen-4-one monohydrate], $C_{20}H_{18}O_8\cdot H_2O$, was isolated from the popular Chinese medicinal plant *Entada phaseoloides*. In the crystal, inversion-related molecules are joined by pairs of weak C—H···O hydrogen bonds. The dimers are further interconnected by a bridging water molecule *via* weak C—H···$O_{water}$ and pairs of $(O-H)_{water}\cdots O$ hydrogen bonds into a linear tape running parallel to the *b* axis.

## Related literature

For the isolation of 5,6,7,5′-tetramethoxy-3′,4′-methylenedioxyflavone, see: Chen *et al.* (1984); Vyas *et al.* (1986*a*); Souza *et al.* (1995); Tomazela *et al.* (2000). For the NMR spectroscopic studies, see Vyas *et al.* (1986*b*). For the biological activity of flavonoids, see: Genoux *et al.* (2011); Bodewes *et al.* (2011); Jacob *et al.* (2011); Veitch & Grayer (2011).




## Experimental

### Crystal data

$C_{20}H_{18}O_8\cdot H_2O$
$M_r$ = 404.36
Triclinic, $P\overline{1}$
$a$ = 9.3014 (17) Å
$b$ = 9.3146 (17) Å
$c$ = 11.009 (2) Å
$\alpha$ = 105.413 (3)°
$\beta$ = 91.798 (3)°
$\gamma$ = 100.985 (3)°
$V$ = 899.3 (3) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.12 mm$^{-1}$
$T$ = 173 K
0.41 × 0.35 × 0.32 mm

### Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min}$ = 0.772, $T_{max}$ = 1.000
6560 measured reflections
3181 independent reflections
2608 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.018

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.117
$S$ = 1.07
3181 reflections
270 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.49 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.32 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1$W$—H11···O5 | 0.83 (2) | 2.03 (2) | 2.823 (2) | 160 (3) |
| O1$W$—H12···O4 | 0.83 (2) | 2.33 (3) | 2.987 (2) | 137 (3) |
| C3—H3$A$···O5$^{i}$ | 0.95 | 2.41 | 3.255 (2) | 147 |
| C8—H8$A$···O1$W^{ii}$ | 0.95 | 2.42 | 3.372 (2) | 175 |

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: BG2446).

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# supplementary materials

*Acta Cryst.* (2012). E**68**, o1390 [doi:10.1107/S1600536812015139]

## 5,6,7,5′-Tetramethoxy-3′,4′-methylenedioxyflavone monohydrate

**Hou-Jin Li, Da-Lang Zhou, Ting-Juan Xu, Chi-Keung Lam and Wen-Jian Lan**

### Comment

Flavonoids are an important secondary metabolites produced in some medicinal or dietary plants. They are reported to show diverse biological activities, including antioxidant, anti-inflammatory, anti-cancer activities, as well as slowing down the progression of cardiovascular and neurodegenerative diseases (Genoux *et al.*, 2011; Bodewes *et al.*, 2011; Jacob *et al.*, 2011; Veitch *et al.*, 2011).

*Entada phaseoloides* (*L.*) Merr., a popular Chinese medicinal plant, is distributed widely in the South China and the trunk has been used clinically for a long time to treat rheumatism. The title compound, 5,6,7,5′-tetramethoxy-3′,4′-methylenedioxyflavone was isolated from the ethanol extract of *Entada phaseoloides*. In the crystals, the methoxy oxygen atom and the adjacent carbonyl oxygen atom are bridged by a water molecule *via* O1w—H11···O5 and O1w—H12···O4 hydrogen bonds. Neighboring molecules related by an inversion centre are joined together *via* a pair of weak C3—H···O5═C4 hydrogen bonds. The molecules are further cross linked by a weak C8′-H···O5═C and C8—H8A···O1w intermolecular interactions.

### Experimental

The trunk of *Entada phaseoloides* (*L.*) Merr. was successively extracted three times with ethanol. The extract was concentrated by low-temperature rotary evaporation and chromatographed on a silica gel column with a petroleum ether–EtOAc–MeOH gradient as the eluent to afford 20 fractions (Fr. 1–Fr. 20). *Fr.* 8–10 were further purified by RP-HPLC eluted with $H_2O$–MeCN (60:40, *v*/*v*) to yield 5,6,7,5′-tetramethoxy-3′,4′-methylenedioxyflavone. Crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution of the compound in EtOAc.

### Refinement

The H atoms on carbon atoms were placed geometrically and treated as riding on their parent atoms with C—H = 0.98 Å (methyl) or 0.95 Å (aromatic) with $U_{iso}$(H) = 1.2$U_{eq}$(C). The H atoms of the water molecule were found in a difference Fourier map and refined with an O—H distance restraint of 0.82 (1)Å and free $U_{iso}$(H). An antibumping condition between symmetry-related H's was applied in order to preserve a meaningful geometry.

### Computing details

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

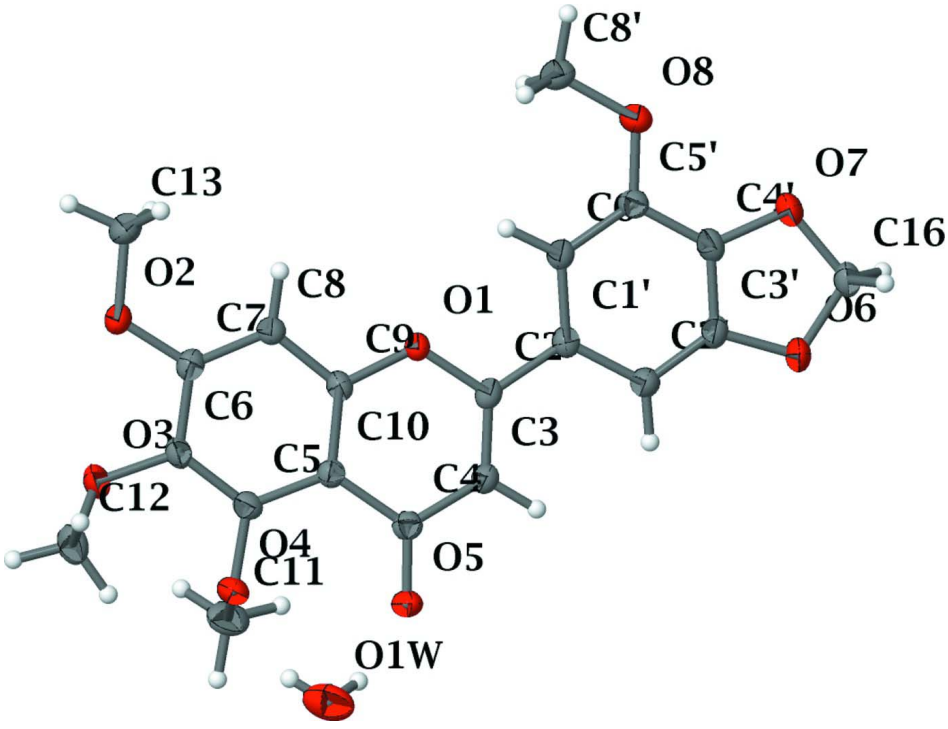


**Figure 1**

View of the title compound showing displacement ellipsoids at the 50% probability level.

**5,6,7-trimethoxy-2-(7-methoxy-1,3-dihydro-2-benzofuran-5-yl)- 4*H*-chromen-4-one monohydrate**

*Crystal data*

$C_{20}H_{18}O_8 \cdot H_2O$
$M_r = 404.36$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.3014$ (17) Å
$b = 9.3146$ (17) Å
$c = 11.009$ (2) Å
$\alpha = 105.413$ (3)°
$\beta = 91.798$ (3)°
$\gamma = 100.985$ (3)°
$V = 899.3$ (3) Å$^3$
$Z = 2$
$F(000) = 424$
$D_x = 1.493$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4354 reflections
$\theta = 2.2$–27.0°
$\mu = 0.12$ mm$^{-1}$
$T = 173$ K
Block, colourless
0.41 × 0.35 × 0.32 mm

*Data collection*

Bruker SMART 1000 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scan
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min} = 0.772$, $T_{max} = 1.000$
6560 measured reflections
3181 independent reflections
2608 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 25.3°$, $\theta_{min} = 2.2°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -13 \rightarrow 13$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.117$
$S = 1.07$
3181 reflections
270 parameters
4 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0641P)^2 + 0.3125P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.49$ e Å$^{-3}$
$\Delta\rho_{min} = -0.32$ 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 $F^2 > \sigma(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.53779 (12) | 0.48854 (12) | 0.20513 (10) | 0.0240 (3) |
| O1W | 0.4889 (2) | −0.09163 (18) | 0.36332 (16) | 0.0525 (4) |
| H11 | 0.487 (3) | −0.071 (4) | 0.295 (2) | 0.093 (11)* |
| H12 | 0.422 (2) | −0.055 (5) | 0.3978 (15) | 0.23 (3)* |
| O2 | 0.22755 (13) | 0.59084 (13) | 0.53871 (11) | 0.0311 (3) |
| O3 | 0.14023 (13) | 0.30245 (14) | 0.51890 (12) | 0.0341 (3) |
| O4 | 0.25387 (13) | 0.08319 (13) | 0.34924 (11) | 0.0286 (3) |
| O5 | 0.44095 (14) | 0.03101 (13) | 0.16068 (11) | 0.0317 (3) |
| O6 | 0.93016 (15) | 0.40373 (14) | −0.22376 (12) | 0.0388 (3) |
| O7 | 0.97888 (14) | 0.66696 (14) | −0.17464 (12) | 0.0363 (3) |
| O8 | 0.85089 (14) | 0.85819 (13) | 0.01787 (12) | 0.0356 (3) |
| C2 | 0.59340 (17) | 0.39067 (18) | 0.11252 (14) | 0.0218 (3) |
| C3 | 0.56048 (18) | 0.23941 (18) | 0.09729 (15) | 0.0241 (4) |
| H3A | 0.6018 | 0.1753 | 0.0316 | 0.029* |
| C4 | 0.46578 (18) | 0.17052 (18) | 0.17582 (15) | 0.0237 (4) |
| C5 | 0.30091 (18) | 0.23401 (18) | 0.35496 (15) | 0.0234 (4) |
| C6 | 0.24590 (17) | 0.34069 (19) | 0.44266 (15) | 0.0244 (4) |
| C7 | 0.29046 (17) | 0.49663 (19) | 0.45016 (15) | 0.0243 (4) |
| C8 | 0.38842 (17) | 0.54320 (18) | 0.36968 (15) | 0.0229 (4) |
| H8A | 0.4182 | 0.6479 | 0.3738 | 0.027* |
| C9 | 0.44252 (17) | 0.43264 (18) | 0.28223 (14) | 0.0214 (3) |
| C10 | 0.40336 (17) | 0.27721 (18) | 0.27200 (15) | 0.0215 (3) |
| C11 | 0.1301 (2) | 0.0098 (2) | 0.2581 (2) | 0.0422 (5) |
| H11A | 0.1008 | −0.0971 | 0.2579 | 0.063* |
| H11B | 0.1571 | 0.0153 | 0.1739 | 0.063* |

| | | | | |
|---|---|---|---|---|
| H11C | 0.0479 | 0.0610 | 0.2804 | 0.063* |
| C12 | 0.1733 (2) | 0.2124 (2) | 0.59780 (18) | 0.0384 (5) |
| H12A | 0.0901 | 0.1921 | 0.6476 | 0.058* |
| H12B | 0.2611 | 0.2671 | 0.6549 | 0.058* |
| H12C | 0.1916 | 0.1158 | 0.5453 | 0.058* |
| C13 | 0.2578 (2) | 0.7486 (2) | 0.54402 (18) | 0.0372 (4) |
| H13A | 0.2063 | 0.8038 | 0.6116 | 0.056* |
| H13B | 0.2240 | 0.7610 | 0.4629 | 0.056* |
| H13C | 0.3638 | 0.7893 | 0.5612 | 0.056* |
| C16 | 1.01507 (19) | 0.5318 (2) | −0.25493 (16) | 0.0295 (4) |
| H16A | 0.9920 | 0.5253 | −0.3449 | 0.035* |
| H16B | 1.1213 | 0.5340 | −0.2413 | 0.035* |
| C1′ | 0.69092 (17) | 0.46871 (18) | 0.03700 (15) | 0.0222 (3) |
| C2′ | 0.75922 (19) | 0.38195 (19) | −0.06075 (16) | 0.0270 (4) |
| H2′A | 0.7408 | 0.2740 | −0.0814 | 0.032* |
| C3′ | 0.85314 (18) | 0.46137 (19) | −0.12415 (15) | 0.0261 (4) |
| C4′ | 0.88199 (18) | 0.61765 (19) | −0.09635 (15) | 0.0257 (4) |
| C5′ | 0.81543 (18) | 0.70520 (18) | −0.00182 (15) | 0.0247 (4) |
| C6′ | 0.71763 (17) | 0.62730 (18) | 0.06452 (15) | 0.0231 (4) |
| H6′A | 0.6686 | 0.6832 | 0.1294 | 0.028* |
| C8′ | 0.7778 (2) | 0.95003 (19) | 0.11035 (17) | 0.0326 (4) |
| H8′A | 0.8132 | 1.0575 | 0.1148 | 0.049* |
| H8′B | 0.7982 | 0.9339 | 0.1931 | 0.049* |
| H8′C | 0.6716 | 0.9218 | 0.0868 | 0.049* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0278 (6) | 0.0202 (6) | 0.0242 (6) | 0.0049 (5) | 0.0109 (5) | 0.0057 (5) |
| O1W | 0.0677 (11) | 0.0415 (8) | 0.0526 (10) | 0.0061 (8) | −0.0019 (8) | 0.0246 (7) |
| O2 | 0.0364 (7) | 0.0241 (6) | 0.0331 (7) | 0.0078 (5) | 0.0176 (5) | 0.0059 (5) |
| O3 | 0.0319 (7) | 0.0366 (7) | 0.0437 (7) | 0.0131 (5) | 0.0209 (6) | 0.0218 (6) |
| O4 | 0.0325 (6) | 0.0218 (6) | 0.0335 (7) | 0.0043 (5) | 0.0092 (5) | 0.0113 (5) |
| O5 | 0.0449 (7) | 0.0193 (6) | 0.0331 (7) | 0.0082 (5) | 0.0145 (6) | 0.0087 (5) |
| O6 | 0.0485 (8) | 0.0321 (7) | 0.0413 (7) | 0.0131 (6) | 0.0296 (6) | 0.0134 (6) |
| O7 | 0.0420 (7) | 0.0320 (7) | 0.0404 (7) | 0.0088 (6) | 0.0233 (6) | 0.0162 (6) |
| O8 | 0.0439 (8) | 0.0223 (6) | 0.0413 (7) | 0.0049 (5) | 0.0173 (6) | 0.0097 (5) |
| C2 | 0.0217 (8) | 0.0242 (8) | 0.0195 (8) | 0.0070 (6) | 0.0031 (6) | 0.0041 (6) |
| C3 | 0.0261 (8) | 0.0234 (8) | 0.0235 (8) | 0.0075 (7) | 0.0064 (7) | 0.0057 (7) |
| C4 | 0.0254 (8) | 0.0226 (8) | 0.0236 (8) | 0.0055 (7) | 0.0018 (7) | 0.0067 (7) |
| C5 | 0.0227 (8) | 0.0223 (8) | 0.0266 (8) | 0.0039 (6) | 0.0024 (7) | 0.0098 (7) |
| C6 | 0.0217 (8) | 0.0279 (9) | 0.0265 (8) | 0.0058 (7) | 0.0074 (7) | 0.0114 (7) |
| C7 | 0.0228 (8) | 0.0264 (9) | 0.0234 (8) | 0.0071 (7) | 0.0038 (7) | 0.0047 (7) |
| C8 | 0.0237 (8) | 0.0203 (8) | 0.0238 (8) | 0.0034 (6) | 0.0036 (6) | 0.0053 (6) |
| C9 | 0.0206 (8) | 0.0237 (8) | 0.0206 (8) | 0.0031 (6) | 0.0041 (6) | 0.0081 (6) |
| C10 | 0.0213 (8) | 0.0228 (8) | 0.0214 (8) | 0.0056 (6) | 0.0027 (6) | 0.0069 (6) |
| C11 | 0.0330 (10) | 0.0297 (10) | 0.0580 (13) | −0.0048 (8) | 0.0017 (9) | 0.0104 (9) |
| C12 | 0.0452 (11) | 0.0461 (11) | 0.0305 (9) | 0.0121 (9) | 0.0147 (8) | 0.0186 (9) |
| C13 | 0.0461 (11) | 0.0255 (9) | 0.0400 (10) | 0.0096 (8) | 0.0178 (9) | 0.0057 (8) |
| C16 | 0.0284 (9) | 0.0347 (10) | 0.0293 (9) | 0.0097 (7) | 0.0120 (7) | 0.0125 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C1′ | 0.0206 (8) | 0.0251 (8) | 0.0213 (8) | 0.0045 (6) | 0.0027 (6) | 0.0072 (7) |
| C2′ | 0.0310 (9) | 0.0239 (8) | 0.0284 (9) | 0.0079 (7) | 0.0092 (7) | 0.0086 (7) |
| C3′ | 0.0273 (9) | 0.0299 (9) | 0.0239 (8) | 0.0108 (7) | 0.0083 (7) | 0.0083 (7) |
| C4′ | 0.0240 (8) | 0.0314 (9) | 0.0250 (8) | 0.0058 (7) | 0.0052 (7) | 0.0134 (7) |
| C5′ | 0.0267 (8) | 0.0231 (8) | 0.0253 (8) | 0.0047 (7) | 0.0024 (7) | 0.0085 (7) |
| C6′ | 0.0232 (8) | 0.0258 (8) | 0.0213 (8) | 0.0076 (7) | 0.0042 (6) | 0.0063 (7) |
| C8′ | 0.0386 (10) | 0.0241 (9) | 0.0339 (9) | 0.0072 (7) | 0.0044 (8) | 0.0056 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C2 | 1.3627 (18) | C8—C9 | 1.393 (2) |
| O1—C9 | 1.3723 (19) | C8—H8A | 0.9500 |
| O1W—H11 | 0.829 (17) | C9—C10 | 1.397 (2) |
| O1W—H12 | 0.826 (19) | C11—H11A | 0.9800 |
| O2—C7 | 1.3564 (19) | C11—H11B | 0.9800 |
| O2—C13 | 1.427 (2) | C11—H11C | 0.9800 |
| O3—C6 | 1.369 (2) | C12—H12A | 0.9800 |
| O3—C12 | 1.422 (2) | C12—H12B | 0.9800 |
| O4—C5 | 1.3732 (19) | C12—H12C | 0.9800 |
| O4—C11 | 1.436 (2) | C13—H13A | 0.9800 |
| O5—C4 | 1.240 (2) | C13—H13B | 0.9800 |
| O6—C3′ | 1.374 (2) | C13—H13C | 0.9800 |
| O6—C16 | 1.428 (2) | C16—H16A | 0.9900 |
| O7—C4′ | 1.367 (2) | C16—H16B | 0.9900 |
| O7—C16 | 1.438 (2) | C1′—C6′ | 1.398 (2) |
| O8—C5′ | 1.356 (2) | C1′—C2′ | 1.411 (2) |
| O8—C8′ | 1.426 (2) | C2′—C3′ | 1.365 (2) |
| C2—C3 | 1.347 (2) | C2′—H2′A | 0.9500 |
| C2—C1′ | 1.470 (2) | C3′—C4′ | 1.376 (2) |
| C3—C4 | 1.436 (2) | C4′—C5′ | 1.383 (2) |
| C3—H3A | 0.9500 | C5′—C6′ | 1.400 (2) |
| C4—C10 | 1.465 (2) | C6′—H6′A | 0.9500 |
| C5—C6 | 1.377 (2) | C8′—H8′A | 0.9800 |
| C5—C10 | 1.417 (2) | C8′—H8′B | 0.9800 |
| C6—C7 | 1.412 (2) | C8′—H8′C | 0.9800 |
| C7—C8 | 1.381 (2) | | |

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| C2—O1—C9 | 119.79 (12) | O3—C12—H12B | 109.5 |
| H11—O1W—H12 | 103 (3) | H12A—C12—H12B | 109.5 |
| C7—O2—C13 | 117.60 (13) | O3—C12—H12C | 109.5 |
| C6—O3—C12 | 116.78 (13) | H12A—C12—H12C | 109.5 |
| C5—O4—C11 | 112.69 (13) | H12B—C12—H12C | 109.5 |
| C3′—O6—C16 | 106.39 (13) | O2—C13—H13A | 109.5 |
| C4′—O7—C16 | 105.55 (13) | O2—C13—H13B | 109.5 |
| C5′—O8—C8′ | 117.93 (13) | H13A—C13—H13B | 109.5 |
| C3—C2—O1 | 121.29 (14) | O2—C13—H13C | 109.5 |
| C3—C2—C1′ | 125.84 (14) | H13A—C13—H13C | 109.5 |
| O1—C2—C1′ | 112.86 (13) | H13B—C13—H13C | 109.5 |
| C2—C3—C4 | 123.01 (15) | O6—C16—O7 | 107.82 (13) |
| C2—C3—H3A | 118.5 | O6—C16—H16A | 110.1 |

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| C4—C3—H3A | 118.5 | O7—C16—H16A | 110.1 |
| O5—C4—C3 | 121.05 (15) | O6—C16—H16B | 110.1 |
| O5—C4—C10 | 124.15 (15) | O7—C16—H16B | 110.1 |
| C3—C4—C10 | 114.80 (14) | H16A—C16—H16B | 108.5 |
| O4—C5—C6 | 118.34 (14) | C6′—C1′—C2′ | 120.90 (15) |
| O4—C5—C10 | 120.27 (14) | C6′—C1′—C2 | 119.88 (14) |
| C6—C5—C10 | 121.39 (14) | C2′—C1′—C2 | 119.20 (14) |
| O3—C6—C5 | 122.89 (14) | C3′—C2′—C1′ | 116.46 (15) |
| O3—C6—C7 | 117.07 (14) | C3′—C2′—H2′A | 121.8 |
| C5—C6—C7 | 119.88 (15) | C1′—C2′—H2′A | 121.8 |
| O2—C7—C8 | 124.83 (15) | C2′—C3′—O6 | 127.60 (15) |
| O2—C7—C6 | 114.65 (14) | C2′—C3′—C4′ | 123.06 (15) |
| C8—C7—C6 | 120.50 (15) | O6—C3′—C4′ | 109.34 (14) |
| C7—C8—C9 | 118.29 (15) | O7—C4′—C3′ | 110.80 (14) |
| C7—C8—H8A | 120.9 | O7—C4′—C5′ | 127.57 (15) |
| C9—C8—H8A | 120.9 | C3′—C4′—C5′ | 121.61 (15) |
| O1—C9—C8 | 114.47 (14) | O8—C5′—C4′ | 117.04 (15) |
| O1—C9—C10 | 122.08 (14) | O8—C5′—C6′ | 126.13 (15) |
| C8—C9—C10 | 123.45 (14) | C4′—C5′—C6′ | 116.82 (15) |
| C9—C10—C5 | 116.48 (14) | C1′—C6′—C5′ | 121.13 (15) |
| C9—C10—C4 | 118.96 (14) | C1′—C6′—H6′A | 119.4 |
| C5—C10—C4 | 124.54 (14) | C5′—C6′—H6′A | 119.4 |
| O4—C11—H11A | 109.5 | O8—C8′—H8′A | 109.5 |
| O4—C11—H11B | 109.5 | O8—C8′—H8′B | 109.5 |
| H11A—C11—H11B | 109.5 | H8′A—C8′—H8′B | 109.5 |
| O4—C11—H11C | 109.5 | O8—C8′—H8′C | 109.5 |
| H11A—C11—H11C | 109.5 | H8′A—C8′—H8′C | 109.5 |
| H11B—C11—H11C | 109.5 | H8′B—C8′—H8′C | 109.5 |
| O3—C12—H12A | 109.5 | | |

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| C9—O1—C2—C3 | −2.1 (2) | C6—C5—C10—C4 | −179.51 (14) |
| C9—O1—C2—C1′ | 178.78 (12) | O5—C4—C10—C9 | 178.31 (15) |
| O1—C2—C3—C4 | 0.1 (2) | C3—C4—C10—C9 | −1.8 (2) |
| C1′—C2—C3—C4 | 179.04 (14) | O5—C4—C10—C5 | −3.6 (3) |
| C2—C3—C4—O5 | −178.26 (15) | C3—C4—C10—C5 | 176.28 (14) |
| C2—C3—C4—C10 | 1.8 (2) | C3′—O6—C16—O7 | −3.22 (18) |
| C11—O4—C5—C6 | 93.10 (18) | C4′—O7—C16—O6 | 2.76 (18) |
| C11—O4—C5—C10 | −86.81 (18) | C3—C2—C1′—C6′ | −178.88 (15) |
| C12—O3—C6—C5 | 57.9 (2) | O1—C2—C1′—C6′ | 0.2 (2) |
| C12—O3—C6—C7 | −126.79 (17) | C3—C2—C1′—C2′ | −0.4 (2) |
| O4—C5—C6—O3 | −4.0 (2) | O1—C2—C1′—C2′ | 178.63 (13) |
| C10—C5—C6—O3 | 175.91 (14) | C6′—C1′—C2′—C3′ | 1.0 (2) |
| O4—C5—C6—C7 | −179.23 (14) | C2—C1′—C2′—C3′ | −177.43 (14) |
| C10—C5—C6—C7 | 0.7 (2) | C1′—C2′—C3′—O6 | −178.60 (16) |
| C13—O2—C7—C8 | 4.0 (2) | C1′—C2′—C3′—C4′ | 0.0 (2) |
| C13—O2—C7—C6 | −174.70 (15) | C16—O6—C3′—C2′ | −178.74 (17) |
| O3—C6—C7—O2 | 3.6 (2) | C16—O6—C3′—C4′ | 2.46 (18) |
| C5—C6—C7—O2 | 179.09 (14) | C16—O7—C4′—C3′ | −1.28 (19) |
| O3—C6—C7—C8 | −175.18 (14) | C16—O7—C4′—C5′ | 179.85 (16) |

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| C5—C6—C7—C8 | 0.3 (2) | C2′—C3′—C4′—O7 | −179.62 (15) |
| O2—C7—C8—C9 | −179.16 (14) | O6—C3′—C4′—O7 | −0.7 (2) |
| C6—C7—C8—C9 | −0.5 (2) | C2′—C3′—C4′—C5′ | −0.7 (3) |
| C2—O1—C9—C8 | −177.44 (13) | O6—C3′—C4′—C5′ | 178.20 (15) |
| C2—O1—C9—C10 | 2.1 (2) | C8′—O8—C5′—C4′ | 176.80 (14) |
| C7—C8—C9—O1 | 179.29 (13) | C8′—O8—C5′—C6′ | −3.2 (2) |
| C7—C8—C9—C10 | −0.3 (2) | O7—C4′—C5′—O8 | −1.0 (3) |
| O1—C9—C10—C5 | −178.32 (13) | C3′—C4′—C5′—O8 | −179.78 (15) |
| C8—C9—C10—C5 | 1.2 (2) | O7—C4′—C5′—C6′ | 178.95 (15) |
| O1—C9—C10—C4 | −0.1 (2) | C3′—C4′—C5′—C6′ | 0.2 (2) |
| C8—C9—C10—C4 | 179.43 (14) | C2′—C1′—C6′—C5′ | −1.5 (2) |
| O4—C5—C10—C9 | 178.51 (13) | C2—C1′—C6′—C5′ | 176.92 (14) |
| C6—C5—C10—C9 | −1.4 (2) | O8—C5′—C6′—C1′ | −179.14 (15) |
| O4—C5—C10—C4 | 0.4 (2) | C4′—C5′—C6′—C1′ | 0.9 (2) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1*W*—H11···O5 | 0.83 (2) | 2.03 (2) | 2.823 (2) | 160 (3) |
| O1*W*—H12···O4 | 0.83 (2) | 2.33 (3) | 2.987 (2) | 137 (3) |
| C3—H3*A*···O5$^{i}$ | 0.95 | 2.41 | 3.255 (2) | 147 |
| C8—H8*A*···O1*W*$^{ii}$ | 0.95 | 2.42 | 3.372 (2) | 175 |

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