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Crystal, molecular structure and Hirshheld surface analysis of 5-hydroxy-3,6,7,8-tetramethoxyflavone

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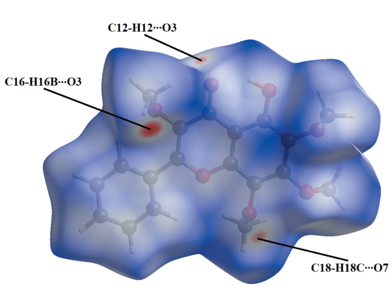
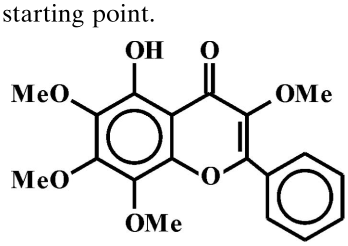
The title compound (systematic name: 5-hydroxy-3,6,7,8-tetramethoxy-2-phenyl-4*H*-chromen-4-one), $C_{19}H_{18}O_7$, is a flavone that was isolated from a butanol extract of the herb *Scutellaria nepetoides M. Pop*. The flavone molecule is almost planar, with a dihedral angle between the planes of the benzopyran-4-one group and the attached phenyl ring of 6.4 (4)°. The 5-hydroxy group forms a strong intramolecular hydrogen bond with the carbonyl group, resulting in a six-membered hydrogen-bonded ring. The crystal structure has triclinic ($P\bar{1}$) symmetry. In the crystal, the molecules are linked by C—H···O hydrogen bonds into a two dimensional network parallel to the *ab* plane. The Hirshfeld surface analysis indicates that the most important contributions to the crystal packing are from H···H (53.9%) and H···O/O···H (20.9%) interactions.



1. Chemical context

Flavonoids are the most numerous class of natural phenolic compounds, which are characterized by structural diversity, high and versatile activity and low toxicity. Plants of the genus *Scutellaria L.* are widespread in Europe, North America, East Asia and are extensively used in traditional Chinese medicine (Shang *et al.*, 2010). Flavonoids isolated from plants of the genus *Scutellaria L.* exhibit antitumor (Yu *et al.*, 2007), hepatoprotective (Jang *et al.*, 2003), antioxidant (Sauvage *et al.*, 2010), anti-inflammatory (Dai *et al.*, 2013), anticonvulsant (Park *et al.*, 2007), antimicrobial (Arituluk *et al.*, 2019) and antiviral activity (Leonova *et al.*, 2020). The creation of drugs based on flavonoids is based on the establishment of the 'chemical structure–pharmacological properties' relationship, and the determination of the structure of a new flavonoid may become a key starting point.



2. Structural commentary

The molecular structure of the title compound is presented in Fig. 1. The benzopyran moieties are practically planar, with

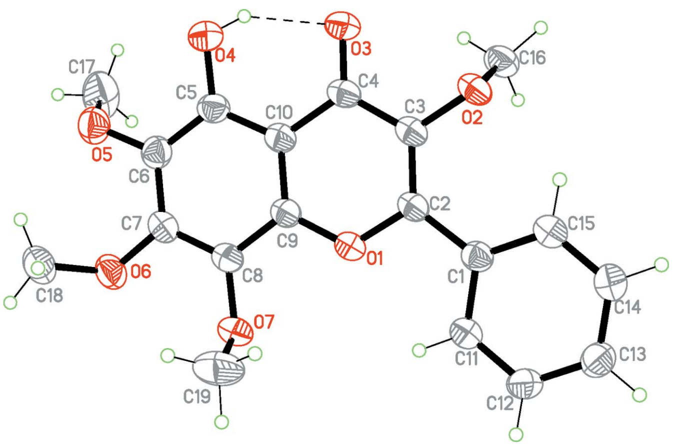


Figure 1
The molecular structure of the title compound with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

r.m.s. deviations of 0.01 Å. The molecular conformation is restricted by the relative positions of the benzopyran unit and the phenyl ring, the dihedral angle between them being 6.4 (4)°. Atoms C3, C6, C7 and C8 of the methoxy substituent have an out-of-plane conformation with the methoxy groups at atoms C3 and C6 pointing in the same direction [C16—O2—C3—C2 = 109.3 (2) and C17—O5—C6—C5 = 66. 7(4)°], while the methoxy groups at atoms C7 and C8 point in opposite direction [C18—O6—C7—C6 = −56.3 (3) and C19—O7—C8—C7 = −91.4 (3)°]. The conformation of the molecule is fixed because of the intramolecular O4—H4···O3 hydrogen bond [2.599 (2) Å, 147°], which closes a six-membered ring with graph-set notation *S*(6) (Etter, 1990).

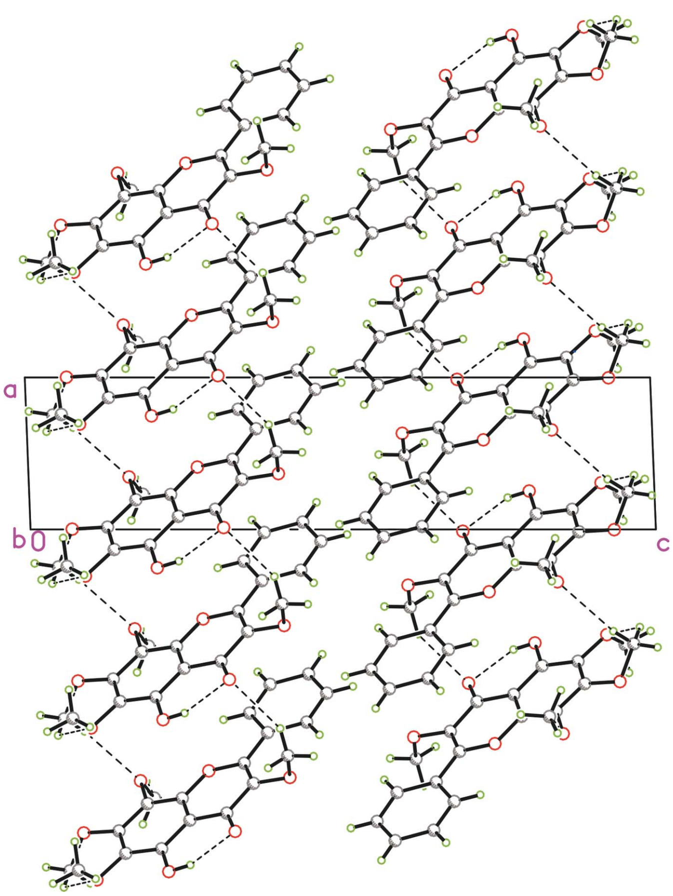


Figure 2
Crystal structure of the title compound in projection on the *ac* plane. Hydrogen bonds are shown as dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O3	0.82	1.87	2.599 (2)	147
C16—H16*A*···O3	0.96	2.51	3.079 (3)	118
C16—H16*B*···O3^{i}	0.96	2.39	3.258 (3)	150
C18—H18*B*···O5	0.96	2.28	2.897 (4)	121
C18—H18*C*···O7^{ii}	0.96	2.53	3.278 (4)	135
C17—H17*C*···O4	0.96	2.52	3.010 (4)	111

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

3. Supramolecular features

In the crystal, the molecules are linked by C—H···O hydrogen bonds into a two dimensional network parallel to the *ab* plane. A perspective view of the crystal packing in the unit cell is depicted in Fig. 2 and numerical details of the hydrogen bonds are presented in Table 1.

4. Hirshfeld surface analysis

In order to visualize the intermolecular interactions in the crystals of the title compound, a Hirshfeld surface analysis was carried out using *Crystal Explorer 17.5* (Turner *et al.*, 2017). The Hirshfeld surface mapped over d_{norm} (Fig. 3) shows the expected bright-red spots near atoms O3, O7, H16*B*, which are involved in the C—H···O hydrogen-bonding interactions. Fingerprint plots (Fig. 4) reveal that H···H and H···O/O···H interactions make the greatest contributions to the surface

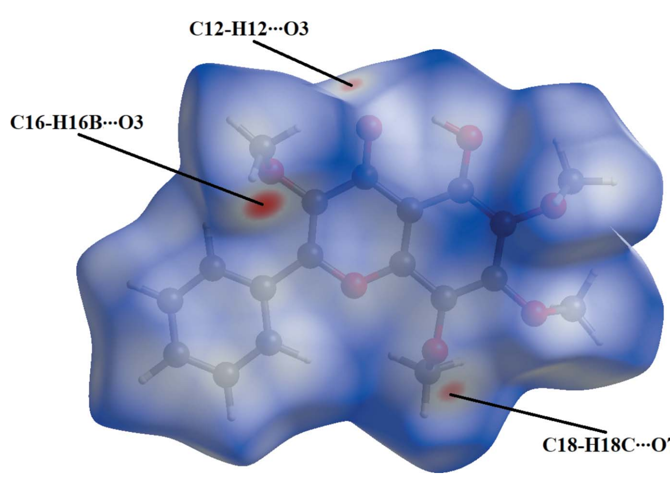


Figure 3
The Hirshfeld surface analysis indicates that the most important contributions to the crystal packing are from H···H (53.9%) and H···O/O···H (20.9%) interactions.

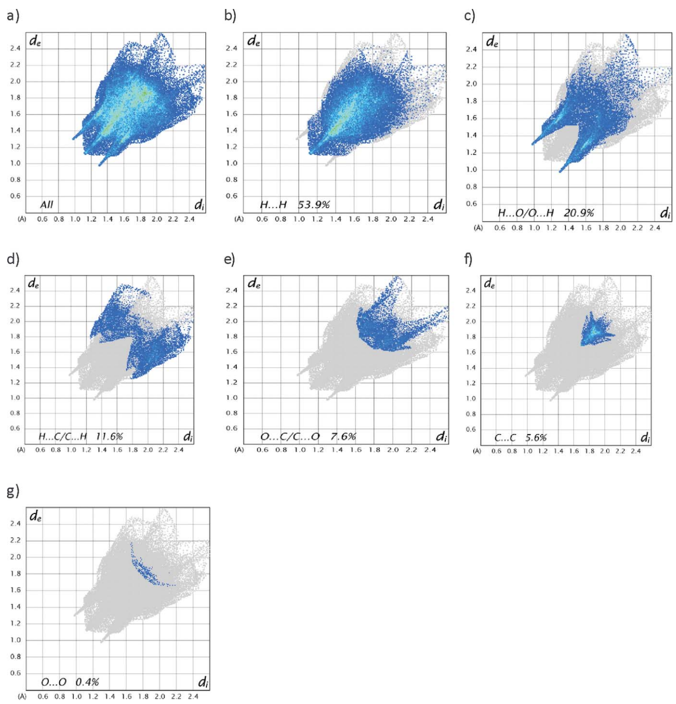


Figure 4
Full two-dimensional fingerprint plots for the title compound, showing all interactions (*a*), and delineated into (*b*) H···H, (*c*) H···O/O···H, (*d*) H···C/C···H, (*e*) O···C/C···O, (*f*) C···C and (*g*) O···O interactions. The d_i and d_e values are the closest internal and external distances (in Å) from a given point on the Hirshfeld surface.

contacts, while H···C/C···H, O···C/C···O, C···C and O···O contacts are less significant.

5. Database survey

A search of the Cambridge Structural Database (CSD Version 5.41, update of November 2019; Groom *et al.*, 2016) found 311 hits for the term 'flavones'. Among these, nine are tetramethoxyflavones: 3,4′,6,7 (DAVREN; Geng *et al.*, 2011), 6,2′3′,4′- (JEMGIN; Wallet *et al.*, 1990*a*) and 2′,4′,5,7- (KEPLEW; Wallet *et al.*, 1990*b*), 3,4′,6,7- (MENSII; Meng *et al.*, 2006), 3′,4′,5,7- (PIQPEK; Shoja, 1997), 3,4′5,7- (PUGKEI; Aree *et al.*, 2009), 3′,5,5′,6- (TMOFLV10; Ting *et al.*, 1972), 3,7,4′,5′- (YASCIF; Etti *et al.*, 2005). The compound FATZOR (Vijayalakshmi *et al.*, 1986) is also a 3,6,7,8 tetramethylflavone, but with two hydroxy substituents at the 5,4′-positions.

6. Synthesis and crystallization

Air-dried whole plants (1.1 kg) of *Scutellaria nepetoides M. Pop*. were extracted three times (each 3 h) with butanol (5 l) at 353 K. The butanol filtrates were collected and concentrated under reduced pressure to provide 10.2 g of butanol extract. The butanol extract (1 g) was subjected to silica gel (60–100 mesh) column (gradient of butanol:water = 0:1, 2:8, 1:1, 8:2, 1:0) as eluent, and five fractions were collected according to TLC analysis. All fractions were concentrated under reduced pressure. A crystallization procedure with different solvents at high temperature was used to obtain the pure compounds. Fraction 5 (0.23 g) was eluted with butanol (100%) at 353 K and with ethanol (95%) at 343 K. The obtained polycrystals were removed from the butanol solution by filtration. Yellow prismatic single crystals were prepared by slow evaporation of butanol solution at room temperature.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{19}H_{18}O_7$
M_r	358.33
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	293
a, *b*, *c* (Å)	5.0789 (4), 8.0801 (6), 20.8682 (19)
α, β, γ (°)	92.481 (7), 91.984 (7), 94.253 (6)
V (Å^3)	852.62 (12)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.90
Crystal size (mm)	0.03 × 0.02 × 0.01
Data collection	
Diffractometer	Agilent Xcalibur, Ruby
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.818, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6484, 3458, 2408
R_{int}	0.023
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.630
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.056, 0.174, 1.03
No. of reflections	3458
No. of parameters	240
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.22, −0.18

Computer programs: *CrysAlis PRO* (Agilent, 2014), *XP* (Siemens, 1994), *SHELXT2018/2* (Sheldrick, 2015*a*) and *SHELXL2018/3* (Sheldrick, 2015*b*).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The C-bound H atoms were positioned geometrically and were included in the refinement in the riding-model approximation, with C—H = 0.96 Å (CH_3), 0.93 Å (aryl H) and O—H = 0.82 Å and with U_{iso}(H) = 1.2U_{eq}(C) (aryl H) and 1.5U_{eq}(C-methyl, O).

Acknowledgements

We are especially grateful to Jamshid Ashurov DSc for help in discussing the results.

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Computing details

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO* (Agilent, 2014); data reduction: *CrysAlis PRO* (Agilent, 2014); program(s) used to solve structure: *SHELXT2018/2* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015b); molecular graphics: *XP* (Siemens, 1994).

5-Hydroxy-3,6,7,8-tetramethoxy-2-phenyl-4*H*-chromen-4-one

Crystal data

$C_{19}H_{18}O_7$
M_r = 358.33
Triclinic, $P\overline{1}$
a = 5.0789 (4) Å
b = 8.0801 (6) Å
c = 20.8682 (19) Å
α = 92.481 (7)°
β = 91.984 (7)°
γ = 94.253 (6)°
V = 852.62 (12) Å^3
Z = 2
F(000) = 376
D_x = 1.396 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 1498 reflections
θ = 5.5–75.0°
μ = 0.90 mm^{-1}
T = 293 K
Prism, yellow
0.03 × 0.02 × 0.01 mm

Data collection

Agilent Xcalibur, Ruby diffractometer
Radiation source: Enhance (Cu) X-ray Source
/ω scans
Absorption correction: multi-scan (CrysAlisPro; Agilent, 2014)
T_{min} = 0.818, T_{max} = 1.000
6484 measured reflections
3458 independent reflections
2408 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 76.1°, θ_{min} = 4.2°
h = −6→4
k = −10→9
l = −25→25
3 standard reflections every 100 reflections
intensity decay: 2.6%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.174
S = 1.03
3458 reflections
240 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0902P)^2 + 0.0534P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.4105 (3)	0.28804 (17)	0.27044 (7)	0.0575 (4)
O2	0.3672 (3)	0.59876 (19)	0.39973 (8)	0.0659 (4)
O7	0.3450 (3)	0.10219 (19)	0.16067 (8)	0.0685 (4)
O3	0.0122 (3)	0.69625 (19)	0.30889 (9)	0.0714 (5)
O4	−0.2527 (4)	0.6406 (2)	0.20033 (9)	0.0740 (5)
H4	−0.209283	0.687910	0.235051	0.111*
O6	0.0019 (3)	0.1855 (2)	0.06667 (8)	0.0725 (5)
O5	−0.3071 (4)	0.4564 (2)	0.08488 (9)	0.0792 (5)
C9	0.2302 (4)	0.3341 (2)	0.22626 (10)	0.0532 (5)
C2	0.4615 (4)	0.3764 (2)	0.32748 (10)	0.0531 (5)
C1	0.6627 (4)	0.3000 (2)	0.36663 (10)	0.0547 (5)
C3	0.3289 (4)	0.5145 (2)	0.34102 (10)	0.0559 (5)
C10	0.0862 (4)	0.4721 (2)	0.23720 (11)	0.0549 (5)
C8	0.1951 (4)	0.2349 (3)	0.17031 (11)	0.0570 (5)
C4	0.1327 (4)	0.5696 (3)	0.29666 (11)	0.0576 (5)
C5	−0.1022 (4)	0.5106 (3)	0.19027 (12)	0.0598 (5)
C7	0.0153 (4)	0.2777 (3)	0.12324 (11)	0.0591 (5)
C11	0.7909 (5)	0.1680 (3)	0.33913 (12)	0.0621 (5)
H11	0.747162	0.131003	0.297010	0.074*
C6	−0.1322 (4)	0.4180 (3)	0.13258 (12)	0.0620 (5)
C13	1.0487 (5)	0.1457 (3)	0.43582 (13)	0.0693 (6)
H13	1.178385	0.095483	0.458888	0.083*
C12	0.9801 (5)	0.0925 (3)	0.37353 (13)	0.0690 (6)
H12	1.062501	0.004788	0.354580	0.083*
C15	0.7318 (5)	0.3509 (3)	0.42988 (11)	0.0676 (6)
H15	0.648243	0.437172	0.449493	0.081*
C14	0.9221 (5)	0.2750 (3)	0.46366 (13)	0.0735 (7)
H14	0.966483	0.310908	0.505855	0.088*
C16	0.5102 (5)	0.7581 (3)	0.39759 (14)	0.0755 (7)
H16A	0.429300	0.821692	0.365563	0.113*
H16B	0.689782	0.743522	0.387050	0.113*
H16C	0.507312	0.815778	0.438727	0.113*
C18	−0.2471 (6)	0.1157 (4)	0.04230 (15)	0.0884 (9)
H18A	−0.223724	0.046870	0.004568	0.133*
H18B	−0.356737	0.203034	0.031583	0.133*
H18C	−0.329956	0.049683	0.074221	0.133*
C19	0.2318 (8)	−0.0502 (3)	0.1815 (2)	0.1081 (11)
H19A	0.058829	−0.073200	0.161721	0.162*
H19B	0.218626	−0.043294	0.227342	0.162*

H19C	0.341133	−0.137746	0.169734	0.162*
C17	−0.2436 (9)	0.6092 (4)	0.05619 (19)	0.1199 (14)
H17A	−0.331407	0.608230	0.014676	0.180*
H17B	−0.056050	0.624212	0.051646	0.180*
H17C	−0.300412	0.698631	0.082812	0.180*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0628 (8)	0.0517 (7)	0.0575 (8)	0.0094 (6)	−0.0003 (7)	−0.0097 (6)
O2	0.0782 (10)	0.0584 (8)	0.0601 (9)	0.0080 (7)	0.0036 (8)	−0.0145 (7)
O7	0.0779 (10)	0.0586 (9)	0.0692 (10)	0.0145 (7)	0.0065 (8)	−0.0124 (7)
O3	0.0744 (10)	0.0578 (9)	0.0823 (11)	0.0181 (7)	0.0011 (8)	−0.0153 (8)
O4	0.0755 (10)	0.0626 (9)	0.0847 (12)	0.0200 (8)	−0.0046 (9)	−0.0056 (8)
O6	0.0745 (10)	0.0820 (11)	0.0589 (9)	0.0029 (8)	0.0009 (8)	−0.0155 (8)
O5	0.0868 (11)	0.0719 (10)	0.0773 (12)	0.0038 (9)	−0.0178 (9)	0.0043 (9)
C9	0.0550 (10)	0.0496 (10)	0.0547 (11)	0.0038 (8)	0.0037 (9)	−0.0023 (8)
C2	0.0550 (10)	0.0492 (10)	0.0541 (11)	0.0013 (8)	0.0058 (9)	−0.0070 (8)
C1	0.0545 (10)	0.0497 (10)	0.0591 (12)	0.0011 (8)	0.0036 (9)	−0.0022 (9)
C3	0.0616 (11)	0.0486 (10)	0.0565 (12)	0.0016 (9)	0.0064 (9)	−0.0079 (8)
C10	0.0585 (11)	0.0474 (10)	0.0583 (12)	0.0025 (8)	0.0050 (9)	−0.0016 (9)
C8	0.0612 (11)	0.0510 (10)	0.0585 (12)	0.0047 (9)	0.0063 (9)	−0.0053 (9)
C4	0.0595 (11)	0.0472 (10)	0.0657 (13)	0.0040 (9)	0.0096 (10)	−0.0058 (9)
C5	0.0591 (11)	0.0503 (11)	0.0702 (14)	0.0062 (9)	0.0036 (10)	−0.0006 (10)
C7	0.0628 (12)	0.0570 (11)	0.0560 (12)	−0.0022 (9)	0.0039 (10)	−0.0046 (9)
C11	0.0675 (12)	0.0539 (11)	0.0639 (13)	0.0065 (10)	−0.0025 (10)	−0.0081 (9)
C6	0.0617 (12)	0.0585 (12)	0.0651 (13)	0.0012 (10)	−0.0036 (10)	0.0039 (10)
C13	0.0669 (13)	0.0645 (13)	0.0766 (16)	0.0085 (11)	−0.0065 (12)	0.0067 (11)
C12	0.0695 (13)	0.0565 (12)	0.0816 (16)	0.0148 (10)	−0.0023 (12)	−0.0039 (11)
C15	0.0737 (14)	0.0685 (13)	0.0608 (13)	0.0133 (11)	0.0015 (11)	−0.0085 (11)
C14	0.0782 (15)	0.0806 (16)	0.0610 (14)	0.0112 (13)	−0.0066 (12)	−0.0056 (12)
C16	0.0753 (15)	0.0613 (13)	0.0871 (17)	0.0041 (11)	−0.0025 (13)	−0.0212 (12)
C18	0.0800 (16)	0.0908 (19)	0.090 (2)	−0.0031 (14)	−0.0024 (14)	−0.0297 (15)
C19	0.129 (3)	0.0560 (15)	0.142 (3)	0.0176 (16)	0.020 (2)	0.0052 (17)
C17	0.164 (4)	0.085 (2)	0.107 (3)	−0.005 (2)	−0.047 (3)	0.0312 (19)

Geometric parameters (Å, º)

O1—C9	1.360 (3)	C5—C6	1.387 (3)
O1—C2	1.368 (2)	C7—C6	1.414 (3)
O2—C3	1.376 (2)	C11—C12	1.374 (3)
O2—C16	1.434 (3)	C11—H11	0.9300
O7—C8	1.373 (3)	C13—C12	1.376 (4)
O7—C19	1.413 (3)	C13—C14	1.384 (4)
O3—C4	1.252 (3)	C13—H13	0.9300
O4—C5	1.357 (3)	C12—H12	0.9300
O4—H4	0.8200	C15—C14	1.373 (4)
O6—C7	1.365 (3)	C15—H15	0.9300

O6—C18	1.415 (3)	C14—H14	0.9300
O5—C6	1.372 (3)	C16—H16A	0.9600
O5—C17	1.416 (4)	C16—H16B	0.9600
C9—C8	1.386 (3)	C16—H16C	0.9600
C9—C10	1.393 (3)	C18—H18A	0.9600
C2—C3	1.370 (3)	C18—H18B	0.9600
C2—C1	1.474 (3)	C18—H18C	0.9600
C1—C15	1.391 (3)	C19—H19A	0.9600
C1—C11	1.403 (3)	C19—H19B	0.9600
C3—C4	1.444 (3)	C19—H19C	0.9600
C10—C5	1.406 (3)	C17—H17A	0.9600
C10—C4	1.443 (3)	C17—H17B	0.9600
C8—C7	1.390 (3)	C17—H17C	0.9600

C9—O1—C2	121.52 (16)	C5—C6—C7	119.4 (2)
C3—O2—C16	114.26 (18)	C12—C13—C14	119.1 (2)
C8—O7—C19	114.8 (2)	C12—C13—H13	120.4
C5—O4—H4	109.5	C14—C13—H13	120.4
C7—O6—C18	119.0 (2)	C11—C12—C13	120.5 (2)
C6—O5—C17	115.0 (2)	C11—C12—H12	119.7
O1—C9—C8	116.32 (18)	C13—C12—H12	119.7
O1—C9—C10	121.69 (19)	C14—C15—C1	120.7 (2)
C8—C9—C10	122.0 (2)	C14—C15—H15	119.7
O1—C2—C3	119.82 (19)	C1—C15—H15	119.7
O1—C2—C1	110.68 (17)	C15—C14—C13	120.9 (2)
C3—C2—C1	129.50 (19)	C15—C14—H14	119.5
C15—C1—C11	117.9 (2)	C13—C14—H14	119.5
C15—C1—C2	123.54 (19)	O2—C16—H16A	109.5
C11—C1—C2	118.60 (19)	O2—C16—H16B	109.5
C2—C3—O2	120.3 (2)	H16A—C16—H16B	109.5
C2—C3—C4	121.67 (19)	O2—C16—H16C	109.5
O2—C3—C4	117.84 (18)	H16A—C16—H16C	109.5
C9—C10—C5	118.9 (2)	H16B—C16—H16C	109.5
C9—C10—C4	119.0 (2)	O6—C18—H18A	109.5
C5—C10—C4	122.2 (2)	O6—C18—H18B	109.5
O7—C8—C9	120.3 (2)	H18A—C18—H18B	109.5
O7—C8—C7	121.05 (19)	O6—C18—H18C	109.5
C9—C8—C7	118.6 (2)	H18A—C18—H18C	109.5
O3—C4—C10	121.9 (2)	H18B—C18—H18C	109.5
O3—C4—C3	121.7 (2)	O7—C19—H19A	109.5
C10—C4—C3	116.34 (18)	O7—C19—H19B	109.5
O4—C5—C6	119.3 (2)	H19A—C19—H19B	109.5
O4—C5—C10	120.4 (2)	O7—C19—H19C	109.5
C6—C5—C10	120.2 (2)	H19A—C19—H19C	109.5
O6—C7—C8	117.0 (2)	H19B—C19—H19C	109.5
O6—C7—C6	122.1 (2)	O5—C17—H17A	109.5
C8—C7—C6	120.8 (2)	O5—C17—H17B	109.5
C12—C11—C1	120.9 (2)	H17A—C17—H17B	109.5

C12—C11—H11	119.5	O5—C17—H17C	109.5
C1—C11—H11	119.5	H17A—C17—H17C	109.5
O5—C6—C5	121.6 (2)	H17B—C17—H17C	109.5
O5—C6—C7	119.0 (2)		

C2—O1—C9—C8	−179.93 (18)	C2—C3—C4—C10	−0.5 (3)
C2—O1—C9—C10	−0.8 (3)	O2—C3—C4—C10	−176.28 (18)
C9—O1—C2—C3	0.1 (3)	C9—C10—C5—O4	177.6 (2)
C9—O1—C2—C1	179.66 (17)	C4—C10—C5—O4	−1.6 (3)
O1—C2—C1—C15	−172.2 (2)	C9—C10—C5—C6	−3.7 (3)
C3—C2—C1—C15	7.3 (4)	C4—C10—C5—C6	177.1 (2)
O1—C2—C1—C11	7.3 (3)	C18—O6—C7—C8	128.1 (3)
C3—C2—C1—C11	−173.2 (2)	C18—O6—C7—C6	−56.3 (3)
O1—C2—C3—O2	176.21 (17)	O7—C8—C7—O6	−2.2 (3)
C1—C2—C3—O2	−3.2 (3)	C9—C8—C7—O6	174.70 (19)
O1—C2—C3—C4	0.6 (3)	O7—C8—C7—C6	−177.85 (19)
C1—C2—C3—C4	−178.9 (2)	C9—C8—C7—C6	−1.0 (3)
C16—O2—C3—C2	109.3 (2)	C15—C1—C11—C12	−0.5 (3)
C16—O2—C3—C4	−74.9 (2)	C2—C1—C11—C12	180.0 (2)
O1—C9—C10—C5	−178.38 (18)	C17—O5—C6—C5	66.7 (4)
C8—C9—C10—C5	0.7 (3)	C17—O5—C6—C7	−115.1 (3)
O1—C9—C10—C4	0.8 (3)	O4—C5—C6—O5	1.2 (4)
C8—C9—C10—C4	179.88 (19)	C10—C5—C6—O5	−177.50 (19)
C19—O7—C8—C9	91.8 (3)	O4—C5—C6—C7	−177.0 (2)
C19—O7—C8—C7	−91.4 (3)	C10—C5—C6—C7	4.4 (3)
O1—C9—C8—O7	−2.4 (3)	O6—C7—C6—O5	4.3 (3)
C10—C9—C8—O7	178.53 (19)	C8—C7—C6—O5	179.8 (2)
O1—C9—C8—C7	−179.25 (18)	O6—C7—C6—C5	−177.5 (2)
C10—C9—C8—C7	1.7 (3)	C8—C7—C6—C5	−2.0 (3)
C9—C10—C4—O3	179.0 (2)	C1—C11—C12—C13	−0.3 (4)
C5—C10—C4—O3	−1.8 (3)	C14—C13—C12—C11	0.8 (4)
C9—C10—C4—C3	−0.2 (3)	C11—C1—C15—C14	0.8 (4)
C5—C10—C4—C3	179.03 (19)	C2—C1—C15—C14	−179.7 (2)
C2—C3—C4—O3	−179.7 (2)	C1—C15—C14—C13	−0.3 (4)
O2—C3—C4—O3	4.6 (3)	C12—C13—C14—C15	−0.5 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O3	0.82	1.87	2.599 (2)	147
C16—H16*A*···O3	0.96	2.51	3.079 (3)	118
C16—H16*B*···O3^{i}	0.96	2.39	3.258 (3)	150
C18—H18*B*···O5	0.96	2.28	2.897 (4)	121
C18—H18*C*···O7^{ii}	0.96	2.53	3.278 (4)	135
C17—H17*C*···O4	0.96	2.52	3.010 (4)	111

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