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4,8-Dihydroxy-2,3-dimethoxy-1-(3-methylbut-2-enyl)-9*H*-xanthen-9-one

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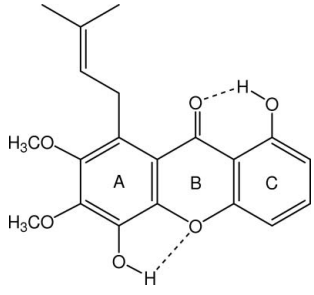
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.049; wR factor = 0.116; data-to-parameter ratio = 16.1.

The title xanthone compound, $C_{20}H_{20}O_6$, was isolated from the roots of *Cratoxylum formosum* ssp. *pruniflorum*. The xanthone ring system is essentially planar. The 3-methylbut-2-enyl substituent plane is not coplanar with the attached benzene ring, the dihedral angle being 62.59 (12) Å. The two methoxy groups are twisted away from the mean plane of the attached benzene ring. The two hydroxy groups are coplanar with the attached benzene rings and contribute to O—H···O intramolecular hydrogen bonds which generate S(5) and S(6) ring motifs. In the crystal structure, intermolecular O—H···O hydrogen bonds and weak C—H···O intermolecular interactions connect the molecules into infinite one-dimensional chains along the [201] direction. The crystal is further stabilized by C—H···π interactions.

Related literature

For related literature on hydrogen-bond motifs, see: Bernstein *et al.* (1995). For values of bond lengths, see: Allen *et al.* (1987). For related structures, see, for example: Boonnak *et al.* (2005); Boonnak, Chantrapromma & Fun (2006); Boonnak, Karalai *et al.* (2006); Chantrapromma *et al.* (2005, 2006); Fun *et al.* (2006). For related literature on the bioactivities of xanthones, see, for example: Aderson (1986); Boonnak, Karalai *et al.* (2006); Kitanov *et al.* (1988).



Experimental

Crystal data

$C_{20}H_{20}O_6$
M_r = 356.36
Monoclinic, $P2_1/c$
a = 4.3883 (2) Å
b = 32.2730 (13) Å
c = 11.8006 (5) Å
β = 92.733 (3)°
V = 1669.34 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 100.0 (1) K
0.30 × 0.16 × 0.11 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.969, T_{max} = 0.989
33106 measured reflections
3837 independent reflections
2645 reflections with $I > 2\sigma(I)$
R_{int} = 0.081

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.116
S = 1.05
3837 reflections
239 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

*Cg*1 is the centroid of the ring C6–C11.

D—H···A	D—H	H···A	D···A	D—H···A
O3—H1O3···O2	0.82	1.80	2.5343 (18)	148
O4—H1O4···O1	0.82	2.24	2.6782 (18)	114
O4—H1O4···O3^{i}	0.82	1.98	2.7531 (18)	158
C4—H4*A*···O2^{i}	0.93	2.51	3.375 (2)	154
C14—H14*B*···O4	0.96	2.58	3.105 (2)	115
C15—H15*C*···O5	0.96	2.48	3.024 (2)	116
C16—H16*A*···O2	0.97	2.34	2.828 (2)	111
C16—H16*B*···O6	0.97	2.35	2.806 (2)	108
C16—H16*A*···*Cg*1^{ii}	0.96	2.86	3.3671 (19)	114

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2251).

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supplementary materials

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4,8-Dihydroxy-2,3-dimethoxy-1-(3-methylbut-2-enyl)-9*H*-xanthen-9-one

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Comment

Secondary metabolites in the plants of *Cratoxylum* genus are mainly xanthones and antraquinones. The plants in this family were used in folk medicine such as, for treatment of diuretic, stomachic, and tonic effects (Kitanov *et al.*, 1988) as well as for diarrhea and flatulence (Aderson, 1986). We have reported previously a number of crystal structures of xanthones and antraquinones isolated from *Cratoxylum formosum* ssp. *pruniflorum* (Boonnak *et al.*, 2005; Boonnak, Chantrapromma & Fun, 2006; Boonnak, Karalai *et al.*, 2006; Chantrapromma *et al.*, 2005, 2006; Fun *et al.*, 2006). The title xanthone was isolated from the methylene chloride extract of the roots of *Cratoxylum formosum* ssp. *pruniflorum* which were collected from Nhongkai province in the northeasthern part of Thailand. The single-crystal *X*-ray structural study of the title xanthone was undertaken in order to establish the structure and conformation of the various groups for further study of Structural Activity Relationships (SAR).

In the molecular structure of the title xanthone (Fig. 1), the xanthone skeleton (rings *A*, *B* and *C*) is essentially planar, the maximum deviation from planarity is 0.018 (2) Å for atom C8. The two hydroxy groups are coplanar with the attached benzene rings and contribute to O—H···O intramolecular hydrogen bonds, namely O3—H1O3···O2 and O4—H1O4···O1 hydrogen bonds which generate the S(6) and S(5) ring motifs, respectively (Bernstein *et al.*, 1995) and help to stabilize the planarity of the structure. There are also weak intramolecular C—H···O interactions; C14—H14B···O4, C15—H15C···O5 and C16—H16B···O2 generate S(6) and C16—H16A···O6 generates S(5) ring motifs (Table 1). The two methoxy groups are twisted away from the mean plane of the attached benzene ring with torsion angles C15–O6–C9–C8 = 75.3 (2)° and C14–O5–C8–C7 = −76.4 (2)°. The orientation of the 3-methylbut-2-enyl (C16–C20) side chain with respect to the benzene ring *A* is indicated by the torsion angle of C9–C10–C16–C17 = 101.62 (19)°, indicating a (+)-anticlinal conformation (Fig. 1) Bond distances and angles in the title xanthone are in normal ranges (Allen *et al.*, 1987) and are comparable to other closely related compounds (Boonnak *et al.*, 2005; Boonnak, Chantrapromma & Fun, 2006; Boonnak, Karalai *et al.*, 2006; Chantrapromma *et al.*, 2005, 2006; Fun *et al.*, 2006).

In the crystal packing (Fig. 2), the molecules are linked together by an intermolecular O4—H1O4···O3^{i} hydrogen bond and a weak C4—H4A···O2^{i} interaction [symmetry code: (i) $1+x$, $1/2-y$, $1/2+z$; Table 1] into infinite one dimension chains along the [2 0 1] direction. The crystal packing is further stabilized by a C—H···π interaction between the 3-methylbut-2-enyl side chain and the centroid of C6–C11 benzene ring (*Cg*1); C16—H16A···*Cg*1^{ii} [symmetry code: (ii) $-1+x$, y, z; Table 1].

Experimental

Air-dried roots of *Cratoxylum formosum* ssp. *pruniflorum* (5.30 kg) were ground and extracted with CH_2Cl_2 (2 × 20 *L* for 2 × 5 days) at room temperature. The residue obtained after evaporation of the solvent was subjected to quick column chromatography (QCC) on silica gel using hexane as first eluent and then increasing the polarity with EtOAc and acetone, respectively, to afford 8 fractions (F1—F8). Fraction F4 was further separated by column chromatography with a gradient

of acetone–hexane to give the title xanthone. Yellow needle-shaped single crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from a $CHCl_3/CH_3OH$ (9:1 *v*/*v*) solvent after several days (m.p. 445–447 K).

Refinement

All H atoms were placed in calculated positions with O—H distance of 0.82 Å and C—H distances in the range 0.93–0.97 Å. The U_{iso}(H) values were constrained to be $1.5U_{eq}$ of the carrier atom for hydroxyl and methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups.

Figures

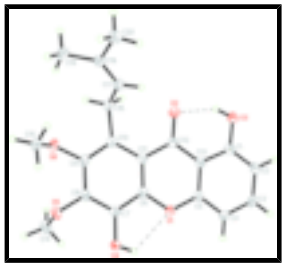

Fig. 1. The asymmetric unit of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering scheme. Hydrogen bonds were drawn as dash lines.

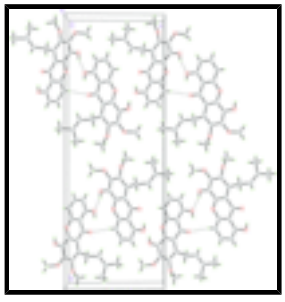

Fig. 2. The crystal packing of the title compound view along the *a* axis. Hydrogen bonds were drawn as dash lines.

4,8-Dihydroxy-2,3-dimethoxy-1-(3-methylbut-2-enyl)-9*H*-xanthen-9-one

Crystal data

$C_{20}H_{20}O_6$	F_{000} = 752
M_r = 356.36	D_x = 1.418 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 445-447 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 4.3883 (2) Å	Cell parameters from 3837 reflections
b = 32.2730 (13) Å	θ = 1.3–27.5º
c = 11.8006 (5) Å	μ = 0.11 mm^{-1}
β = 92.733 (3)º	*T* = 100.0 (1) K
V = 1669.34 (12) Å^3	Block, yellow
Z = 4	0.30 × 0.16 × 0.11 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	3837 independent reflections
Radiation source: fine-focus sealed tube	2645 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.081$
Detector resolution: 8.33 pixels mm^{-1}	$\theta_{max} = 27.5^{o}$
$T = 100.0(1)$ K	$\theta_{min} = 1.3^{o}$
ω scans	$h = -5 \rightarrow 5$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$k = -40 \rightarrow 41$
$T_{min} = 0.969$, $T_{max} = 0.989$	$l = -15 \rightarrow 15$
33106 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.049$	H-atom parameters constrained
$wR(F^2) = 0.116$	$w = 1/[\sigma^2(F_o^2) + (0.0438P)^2 + 0.5487P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} = <0.001$
3837 reflections	$\Delta\rho_{max} = 0.25$ e $Å^{-3}$
239 parameters	$\Delta\rho_{min} = -0.23$ e $Å^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. The low-temparture data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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 $F^2 > 2$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 ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.9952 (3)	0.28976 (4)	0.51586 (10)	0.0198 (3)
O2	0.3551 (3)	0.29827 (4)	0.25288 (11)	0.0266 (3)
O3	0.4148 (3)	0.22140 (4)	0.21607 (11)	0.0278 (3)

H1O3	0.3501	0.2451	0.2069	0.042*
O4	1.1908 (3)	0.35415 (4)	0.64269 (10)	0.0209 (3)
H1O4	1.2252	0.3293	0.6508	0.031*
O5	0.9565 (3)	0.43168 (4)	0.60360 (10)	0.0218 (3)
O6	0.5256 (3)	0.44539 (4)	0.43486 (11)	0.0207 (3)
C1	0.6196 (4)	0.22059 (6)	0.30595 (16)	0.0202 (4)
C2	0.7516 (4)	0.18357 (6)	0.33817 (16)	0.0232 (4)
H2A	0.7008	0.1594	0.2989	0.028*
C3	0.9616 (4)	0.18267 (6)	0.42990 (17)	0.0249 (4)
H3A	1.0498	0.1575	0.4517	0.030*
C4	1.0432 (4)	0.21804 (6)	0.48971 (17)	0.0230 (4)
H4A	1.1848	0.2170	0.5508	0.028*
C5	0.9081 (4)	0.25509 (5)	0.45601 (15)	0.0181 (4)
C6	0.8676 (4)	0.32749 (5)	0.48741 (15)	0.0174 (4)
C7	0.9768 (4)	0.35973 (5)	0.55695 (15)	0.0178 (4)
C8	0.8589 (4)	0.39895 (5)	0.53707 (15)	0.0172 (4)
C9	0.6441 (4)	0.40591 (5)	0.44743 (15)	0.0176 (4)
C10	0.5352 (4)	0.37432 (5)	0.37673 (15)	0.0171 (4)
C11	0.6496 (4)	0.33368 (5)	0.39838 (15)	0.0171 (4)
C12	0.5527 (4)	0.29696 (6)	0.33270 (15)	0.0188 (4)
C13	0.6946 (4)	0.25778 (5)	0.36497 (15)	0.0185 (4)
C14	0.8287 (5)	0.43194 (6)	0.71306 (16)	0.0281 (5)
H14A	0.8797	0.4575	0.7511	0.042*
H14B	0.9097	0.4091	0.7572	0.042*
H14C	0.6109	0.4294	0.7043	0.042*
C15	0.7311 (4)	0.47470 (6)	0.38786 (17)	0.0228 (4)
H15A	0.6584	0.5023	0.4002	0.034*
H15B	0.7418	0.4698	0.3079	0.034*
H15C	0.9303	0.4715	0.4240	0.034*
C16	0.3001 (4)	0.38489 (6)	0.28207 (15)	0.0199 (4)
H16A	0.1430	0.3637	0.2796	0.024*
H16B	0.2040	0.4110	0.3001	0.024*
C17	0.4283 (4)	0.38831 (6)	0.16660 (15)	0.0202 (4)
H17A	0.5317	0.3652	0.1409	0.024*
C18	0.4096 (4)	0.42083 (6)	0.09720 (15)	0.0209 (4)
C19	0.2711 (5)	0.46191 (6)	0.12536 (17)	0.0296 (5)
H19A	0.2072	0.4614	0.2020	0.044*
H19B	0.0979	0.4671	0.0746	0.044*
H19C	0.4196	0.4834	0.1175	0.044*
C20	0.5276 (5)	0.41855 (6)	−0.02071 (16)	0.0302 (5)
H20A	0.6190	0.3919	−0.0318	0.045*
H20B	0.6772	0.4398	−0.0297	0.045*
H20C	0.3616	0.4224	−0.0756	0.045*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0244 (7)	0.0134 (6)	0.0212 (7)	0.0013 (5)	−0.0045 (5)	−0.0008 (5)

O2	0.0326 (8)	0.0202 (7)	0.0258 (7)	−0.0002 (6)	−0.0111 (6)	0.0006 (6)
O3	0.0376 (8)	0.0168 (7)	0.0277 (8)	−0.0012 (6)	−0.0115 (6)	−0.0010 (6)
O4	0.0266 (7)	0.0161 (6)	0.0195 (7)	−0.0003 (5)	−0.0050 (6)	−0.0002 (5)
O5	0.0315 (7)	0.0161 (7)	0.0181 (7)	−0.0037 (5)	0.0023 (6)	−0.0020 (5)
O6	0.0205 (7)	0.0145 (6)	0.0273 (7)	0.0007 (5)	0.0054 (6)	0.0026 (5)
C1	0.0220 (10)	0.0202 (10)	0.0183 (9)	−0.0032 (8)	0.0010 (8)	0.0001 (8)
C2	0.0266 (10)	0.0181 (10)	0.0247 (10)	−0.0019 (8)	−0.0002 (8)	−0.0044 (8)
C3	0.0276 (10)	0.0162 (10)	0.0309 (11)	0.0048 (8)	0.0000 (9)	0.0010 (8)
C4	0.0255 (10)	0.0189 (10)	0.0238 (10)	0.0012 (8)	−0.0065 (8)	−0.0009 (8)
C5	0.0208 (9)	0.0152 (9)	0.0183 (9)	−0.0020 (7)	0.0023 (7)	−0.0031 (7)
C6	0.0184 (9)	0.0149 (9)	0.0193 (9)	−0.0008 (7)	0.0041 (7)	0.0021 (7)
C7	0.0183 (9)	0.0201 (9)	0.0150 (9)	−0.0016 (7)	0.0008 (7)	0.0017 (7)
C8	0.0205 (9)	0.0149 (9)	0.0166 (9)	−0.0040 (7)	0.0042 (7)	−0.0011 (7)
C9	0.0175 (9)	0.0142 (9)	0.0216 (9)	0.0012 (7)	0.0062 (8)	0.0023 (7)
C10	0.0158 (9)	0.0188 (9)	0.0172 (9)	0.0001 (7)	0.0051 (7)	0.0025 (7)
C11	0.0174 (9)	0.0168 (9)	0.0174 (9)	−0.0013 (7)	0.0036 (7)	0.0006 (7)
C12	0.0194 (9)	0.0183 (9)	0.0187 (9)	−0.0022 (7)	0.0015 (8)	0.0014 (7)
C13	0.0204 (9)	0.0157 (9)	0.0194 (9)	−0.0017 (7)	0.0029 (8)	0.0009 (7)
C14	0.0401 (12)	0.0250 (11)	0.0196 (10)	−0.0017 (9)	0.0070 (9)	−0.0053 (8)
C15	0.0267 (10)	0.0173 (10)	0.0247 (10)	−0.0012 (8)	0.0039 (8)	0.0034 (8)
C16	0.0196 (9)	0.0184 (9)	0.0217 (10)	0.0018 (7)	0.0016 (8)	0.0019 (8)
C17	0.0211 (9)	0.0168 (9)	0.0226 (10)	0.0011 (8)	−0.0010 (8)	−0.0029 (8)
C18	0.0225 (10)	0.0200 (10)	0.0198 (10)	−0.0012 (8)	−0.0011 (8)	−0.0004 (8)
C19	0.0379 (12)	0.0230 (11)	0.0280 (11)	0.0061 (9)	0.0026 (9)	0.0059 (9)
C20	0.0402 (12)	0.0287 (11)	0.0217 (10)	−0.0022 (9)	0.0014 (9)	0.0027 (9)

Geometric parameters (Å, °)

O1—C5	1.368 (2)	C9—C10	1.388 (2)
O1—C6	1.375 (2)	C10—C11	1.423 (2)
O2—C12	1.250 (2)	C10—C16	1.523 (2)
O3—C1	1.357 (2)	C11—C12	1.468 (2)
O3—H1O3	0.8200	C12—C13	1.452 (2)
O4—C7	1.359 (2)	C14—H14A	0.9600
O4—H1O4	0.8200	C14—H14B	0.9600
O5—C8	1.372 (2)	C14—H14C	0.9600
O5—C14	1.433 (2)	C15—H15A	0.9600
O6—C9	1.382 (2)	C15—H15B	0.9600
O6—C15	1.436 (2)	C15—H15C	0.9600
C1—C2	1.374 (3)	C16—C17	1.503 (3)
C1—C13	1.418 (2)	C16—H16A	0.9700
C2—C3	1.388 (3)	C16—H16B	0.9700
C2—H2A	0.9300	C17—C18	1.331 (2)
C3—C4	1.380 (3)	C17—H17A	0.9300
C3—H3A	0.9300	C18—C19	1.502 (3)
C4—C5	1.384 (2)	C18—C20	1.509 (3)
C4—H4A	0.9300	C19—H19A	0.9600
C5—C13	1.394 (2)	C19—H19B	0.9600
C6—C7	1.396 (2)	C19—H19C	0.9600

C6—C11	1.401 (2)	C20—H20A	0.9600
C7—C8	1.383 (2)	C20—H20B	0.9600
C8—C9	1.401 (2)	C20—H20C	0.9600
C5—O1—C6	119.85 (13)	C13—C12—C11	117.05 (15)
C1—O3—H1O3	109.5	C5—C13—C1	117.22 (16)
C7—O4—H1O4	109.5	C5—C13—C12	121.35 (16)
C8—O5—C14	113.28 (14)	C1—C13—C12	121.42 (16)
C9—O6—C15	114.12 (13)	O5—C14—H14A	109.5
O3—C1—C2	119.20 (16)	O5—C14—H14B	109.5
O3—C1—C13	119.80 (16)	H14A—C14—H14B	109.5
C2—C1—C13	121.00 (17)	O5—C14—H14C	109.5
C1—C2—C3	119.32 (17)	H14A—C14—H14C	109.5
C1—C2—H2A	120.3	H14B—C14—H14C	109.5
C3—C2—H2A	120.3	O6—C15—H15A	109.5
C4—C3—C2	121.86 (17)	O6—C15—H15B	109.5
C4—C3—H3A	119.1	H15A—C15—H15B	109.5
C2—C3—H3A	119.1	O6—C15—H15C	109.5
C3—C4—C5	118.04 (17)	H15A—C15—H15C	109.5
C3—C4—H4A	121.0	H15B—C15—H15C	109.5
C5—C4—H4A	121.0	C17—C16—C10	114.31 (15)
O1—C5—C4	116.92 (15)	C17—C16—H16A	108.7
O1—C5—C13	120.53 (16)	C10—C16—H16A	108.7
C4—C5—C13	122.55 (16)	C17—C16—H16B	108.7
O1—C6—C7	113.09 (15)	C10—C16—H16B	108.7
O1—C6—C11	124.40 (15)	H16A—C16—H16B	107.6
C7—C6—C11	122.51 (16)	C18—C17—C16	126.92 (17)
O4—C7—C8	119.15 (15)	C18—C17—H17A	116.5
O4—C7—C6	122.98 (16)	C16—C17—H17A	116.5
C8—C7—C6	117.87 (16)	C17—C18—C19	124.98 (18)
O5—C8—C7	120.21 (15)	C17—C18—C20	120.98 (17)
O5—C8—C9	119.28 (15)	C19—C18—C20	114.03 (17)
C7—C8—C9	120.50 (16)	C18—C19—H19A	109.5
O6—C9—C10	119.73 (15)	C18—C19—H19B	109.5
O6—C9—C8	117.72 (15)	H19A—C19—H19B	109.5
C10—C9—C8	122.43 (16)	C18—C19—H19C	109.5
C9—C10—C11	117.43 (16)	H19A—C19—H19C	109.5
C9—C10—C16	118.58 (16)	H19B—C19—H19C	109.5
C11—C10—C16	123.99 (16)	C18—C20—H20A	109.5
C6—C11—C10	119.24 (16)	C18—C20—H20B	109.5
C6—C11—C12	116.80 (15)	H20A—C20—H20B	109.5
C10—C11—C12	123.96 (16)	C18—C20—H20C	109.5
O2—C12—C13	120.03 (16)	H20A—C20—H20C	109.5
O2—C12—C11	122.91 (16)	H20B—C20—H20C	109.5
O3—C1—C2—C3	179.85 (17)	O1—C6—C11—C10	−179.68 (16)
C13—C1—C2—C3	0.0 (3)	C7—C6—C11—C10	0.5 (3)
C1—C2—C3—C4	−0.3 (3)	O1—C6—C11—C12	0.4 (3)
C2—C3—C4—C5	0.2 (3)	C7—C6—C11—C12	−179.40 (17)
C6—O1—C5—C4	−179.40 (17)	C9—C10—C11—C6	−1.1 (2)

C6—O1—C5—C13	1.1 (2)	C16—C10—C11—C6	179.55 (16)
C3—C4—C5—O1	−179.31 (17)	C9—C10—C11—C12	178.79 (17)
C3—C4—C5—C13	0.1 (3)	C16—C10—C11—C12	−0.6 (3)
C5—O1—C6—C7	179.28 (16)	C6—C11—C12—O2	178.42 (17)
C5—O1—C6—C11	−0.6 (3)	C10—C11—C12—O2	−1.5 (3)
O1—C6—C7—O4	0.9 (3)	C6—C11—C12—C13	−0.8 (2)
C11—C6—C7—O4	−179.21 (16)	C10—C11—C12—C13	179.28 (17)
O1—C6—C7—C8	−178.79 (16)	O1—C5—C13—C1	178.99 (16)
C11—C6—C7—C8	1.1 (3)	C4—C5—C13—C1	−0.4 (3)
C14—O5—C8—C7	−76.4 (2)	O1—C5—C13—C12	−1.6 (3)
C14—O5—C8—C9	105.05 (19)	C4—C5—C13—C12	178.95 (18)
O4—C7—C8—O5	−0.3 (3)	O3—C1—C13—C5	−179.50 (16)
C6—C7—C8—O5	179.45 (16)	C2—C1—C13—C5	0.4 (3)
O4—C7—C8—C9	178.28 (16)	O3—C1—C13—C12	1.1 (3)
C6—C7—C8—C9	−2.0 (3)	C2—C1—C13—C12	−179.01 (18)
C15—O6—C9—C10	−108.65 (18)	O2—C12—C13—C5	−177.83 (17)
C15—O6—C9—C8	75.3 (2)	C11—C12—C13—C5	1.4 (3)
O5—C8—C9—O6	−4.1 (2)	O2—C12—C13—C1	1.5 (3)
C7—C8—C9—O6	177.37 (16)	C11—C12—C13—C1	−179.19 (17)
O5—C8—C9—C10	180.00 (16)	C9—C10—C16—C17	101.62 (19)
C7—C8—C9—C10	1.4 (3)	C11—C10—C16—C17	−79.0 (2)
O6—C9—C10—C11	−175.71 (15)	C10—C16—C17—C18	−123.3 (2)
C8—C9—C10—C11	0.2 (3)	C16—C17—C18—C19	4.2 (3)
O6—C9—C10—C16	3.7 (2)	C16—C17—C18—C20	−174.94 (17)
C8—C9—C10—C16	179.57 (16)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H1O3···O2	0.82	1.80	2.5343 (18)	148
O4—H1O4···O1	0.82	2.24	2.6782 (18)	114
O4—H1O4···O3^{i}	0.82	1.98	2.7531 (18)	158
C4—H4A···O2^{i}	0.93	2.51	3.375 (2)	154
C14—H14B···O4	0.96	2.58	3.105 (2)	115
C15—H15C···O5	0.96	2.48	3.024 (2)	116
C16—H16A···O2	0.97	2.34	2.828 (2)	111
C16—H16B···O6	0.97	2.35	2.806 (2)	108
C16—H16A···Cg1^{ii}	0.96	2.86	3.3671 (19)	114

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

Fig. 1

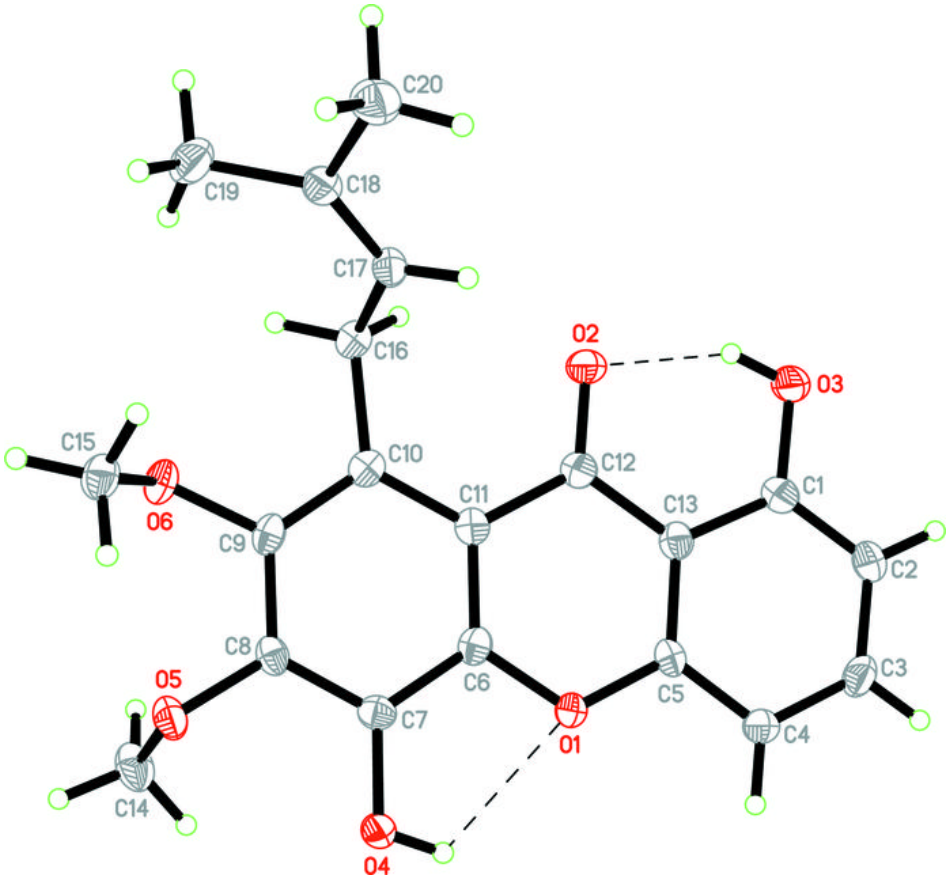
C20
C19
C18
C17
C16
O2
O3
C15
C10
C12
C1
O6
C11
C13
C9
C2
C8
O5
C6
C3
C7
C5
O1
C4
C14
O4

Fig. 2