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7,7′,8,8′-Tetramethoxy-4,4′-dimethyl-3,5′-bichromene-2,2′-dione

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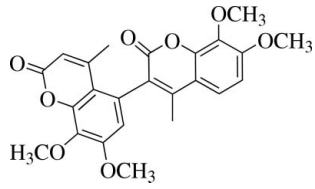
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.046; wR factor = 0.125; data-to-parameter ratio = 23.7.

In the title molecule, $C_{24}H_{22}O_8$, the mean planes of the two coumarin units are inclined to each other at a dihedral angle of 79.93 (3)°. The attached methoxy groups form torsion angles of 7.65 (19) and 78.36 (14)° with respect to one coumarin unit, and angles of 9.01 (16) and 99.08 (11)° with respect to the other coumarin unit. In the crystal structure, weak intermolecular C—H···O hydrogen bonds connect pairs of molecules to form dimers, generating $R_2^2(16)$ and $R_2^2(18)$ rings; the dimers are linked by further weak intermolecular C—H···O hydrogen bonds, forming extended chains. Additional stabilization is provided by weak C—H···π interactions.

Related literature

For the biological activity of coumarins, see: El-Agrody *et al.* (2001); El-Farargy (1991); Emmanuel-Giota *et al.* (2001); Ghate *et al.* (2005); Laakso *et al.* (1994); Nofal *et al.* (2000); Pratibha *et al.* (1999); Shaker (1996); Yang *et al.* (2005). For the pharmaceutical properties of coumarin derivatives, see: Kennedy *et al.* (1997). For related literature on natural and synthetic coumarins, see: Carlton *et al.* (1996); Zhou *et al.* (2000). For standard bond-length data, see: Allen *et al.* (1987). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{24}H_{22}O_8$
M_r = 438.42
Monoclinic, $P2_1/c$
a = 9.4724 (1) Å
b = 23.4766 (3) Å
c = 9.3525 (1) Å
β = 96.254 (1)°
V = 2067.43 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 100 K
0.50 × 0.27 × 0.14 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.949, T_{max} = 0.985
58385 measured reflections
7006 independent reflections
6023 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.125
S = 1.07
7006 reflections
295 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C21—H21A···O6^{i}	0.96	2.55	3.2921 (15)	134
C22—H22A···O6^{ii}	0.96	2.52	3.4385 (14)	161
C22—H22B···O8^{i}	0.96	2.56	3.4401 (15)	152
C6—H6A···$Cg1^{iii}$	0.93	2.92	3.6706 (12)	138
C19—H19A···$Cg2^{iv}$	0.96	2.60	3.5446 (14)	170

Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $-x, -y+2, -z+2$; (iii) $x+1, y, z$; (iv) $x+1, -y+\frac{1}{2}, z-\frac{3}{2}$. $Cg1$ is the centroid of the O7/C14–C18 ring and $Cg2$ is the centroid of the C10–C18 ring.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2822).

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supplementary materials

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7,7',8,8'-Tetramethoxy-4,4'-dimethyl-3,5'-bichromene-2,2'-dione

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Comment

Coumarins are a large group of naturally occurring oxygen heterocycles representing 2*H*-1-benzopyran-2-one derivatives. Many natural coumarins are reputed for their wide range of biological activites such as antibacterial (El-Agrody *et al.*, 2001; Pratibha *et al.*, 1999), antifungal (Shaker, 1996; El-Farargy, 1991), antioxidant (Yang *et al.*, 2005), analgesic (Ghate *et al.*, 2005), anti-inflammatory (Emmanuel-Giota *et al.*, 2001) and antitumor (Nofal *et al.*, 2000). Bi and tri-coumarins are a comparatively new group of compounds which are widespread in nature and their biological properties are also well known (Laakso *et al.*, 1994). One of the characteristic pharmacological properties of coumarin derivatives is anticoagulant action (Kennedy *et al.*, 1997). A large number of natural and semisynthetic coumarin and bicoumarin derivatives have been reported to demonstrate chemopreventive (Carlton *et al.*, 1996) and anti-HIV (Zhou *et al.*, 2000) activities. Keeping in view of these biological importance of coumarins and their dimers, we have synthesized the title compound (I) and report herein its crystal structure.

The molecular structure of the title compound is shown in Fig .1. In crystal structure of (I) molecules are linked by weak intermolecular C-H···O hydrogen bonds to form $R_2^2(16)$ and $R_2^2(18)$ rings (Bernstein *et al.*, (1995). The two coumarin units are essentially planar with the maximum deviation from planarity of 0.0665 (11)Å for atom C9 in the ring (O3/C1–C9) and 0.0419 (12)Å for atom C16 in the ring (O7/C10–C18). The two coumarin units forming a dihedral angle of 79.93 (3)° (O3/C1–C9:O7/C10–C18), indicating that they are inclined to each other. Two of the methoxy units attached to the each coumarin units are twisted from the plane of coumarin unit as indicated by the torsion angles of C19–O1—C4–C5=-7.65 (19)°; C20–O2–C3–C2=78.36 (14)°; C22–O5–C12–C11=9.01 (16)° and C23–O6–C13–C14=99.08 (11)°, respectively. The bond lengths Allen *et al.* (1987) and bond angles are normal.

The crystal packing is illustrated in Fig. 2. In addition C—H···π interactions help stabilize the crystal structure.

Experimental

A mixture of 7,8-dimethoxy-4-methyl coumarin (2.20 g, 10 mmol) and manganese(III) acetate (0.774 g, 1 mmol) was stirred at room temperature, then 70% perchloric acid (0.8 g, 6 mmol) was added. The reaction mixture was heated under reflux at 114°C with stirring in the atmosphere of nitrogen for 3 h. The reaction mixture was cooled and diluted with 50 ml of benzene. The benzene solution was washed with water and aq. $NaHCO_3$, dried over anhydrous Na_2SO_4 and left to evaporate. The residue showed two major compounds which were separated by column chromatography followed by preparative thin layer chromatography (Benzene: EtOAc, 9:1) into the title compound (I) (260 mg, 12%).

Refinement

H atoms were positioned geometrically [C–H = 0.93–0.96 Å] and refined using a riding model with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(methyl C). A rotating–group model was used for the methyl groups.

Figures

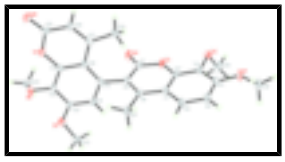

Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom numbering scheme.

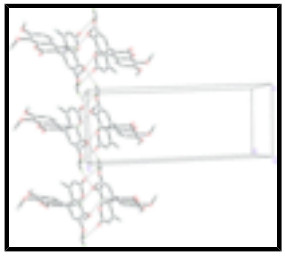

Fig. 2. Part of the crystal structure of (I). Dashed lines indicate the hydrogen bonds.

7,7',8,8'-Tetramethoxy-4,4'-dimethyl-3,5'-bichromene-2,2'-dione

Crystal data

$C_{24}H_{22}O_8$	F_{000} = 920
M_r = 438.42	D_x = 1.409 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 9875 reflections
a = 9.4724 (1) Å	θ = 2.7–33.0º
b = 23.4766 (3) Å	μ = 0.11 mm^{-1}
c = 9.3525 (1) Å	T = 100 K
β = 96.254 (1)º	Plate, colourless
V = 2067.43 (4) Å^3	0.50 × 0.27 × 0.14 mm
Z = 4	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	7006 independent reflections
Radiation source: fine-focus sealed tube	6023 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.030
T = 100 K	θ_{max} = 31.7º
φ and ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Bruker, 2005)	h = −13→13
T_{min} = 0.949, T_{max} = 0.985	k = −34→34
58385 measured reflections	l = −13→13

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.046$	H-atom parameters constrained
$wR(F^2) = 0.125$	$w = 1/[\sigma^2(F_o^2) + (0.0593P)^2 + 0.8142P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
7006 reflections	$\Delta\rho_{max} = 0.47$ e Å^{-3}
295 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cyrosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.69654 (9)	0.68242 (4)	0.29953 (11)	0.0282 (2)
O2	0.43643 (8)	0.65723 (3)	0.37372 (10)	0.02296 (17)
O3	0.26852 (8)	0.73969 (3)	0.46388 (9)	0.01987 (16)
O4	0.05685 (9)	0.76180 (3)	0.52477 (10)	0.02502 (18)
O5	0.03012 (9)	0.95276 (4)	0.88144 (9)	0.02254 (17)
O6	−0.18774 (8)	0.99197 (3)	0.69969 (9)	0.02027 (16)
O7	−0.22134 (8)	0.96109 (3)	0.42722 (8)	0.01782 (15)
O8	−0.35251 (9)	0.97343 (4)	0.21952 (10)	0.02664 (18)
C1	0.17182 (11)	0.77961 (4)	0.50060 (12)	0.01762 (19)
C2	0.40061 (11)	0.75552 (4)	0.43041 (12)	0.01703 (19)
C3	0.48594 (11)	0.71220 (4)	0.38541 (13)	0.0191 (2)
C4	0.62110 (12)	0.72617 (5)	0.34739 (13)	0.0217 (2)
C5	0.66991 (12)	0.78230 (5)	0.36156 (16)	0.0274 (3)
H5A	0.7607	0.7914	0.3398	0.033*
C6	0.58343 (12)	0.82435 (5)	0.40787 (15)	0.0247 (2)
H6A	0.6174	0.8615	0.4172	0.030*
C7	0.44578 (11)	0.81239 (4)	0.44116 (12)	0.01799 (19)
C8	0.34781 (11)	0.85546 (4)	0.48140 (12)	0.01690 (18)
C9	0.21437 (11)	0.83939 (4)	0.50522 (11)	0.01574 (18)
C10	0.10535 (11)	0.88049 (4)	0.54652 (11)	0.01548 (18)

C11	0.12224 (11)	0.89822 (4)	0.68924 (11)	0.01727 (19)
H11A	0.1996	0.8852	0.7502	0.021*
C12	0.02494 (11)	0.93527 (4)	0.74272 (11)	0.01696 (18)
C13	−0.09034 (11)	0.95541 (4)	0.65136 (11)	0.01643 (18)
C14	−0.10474 (10)	0.93843 (4)	0.50800 (11)	0.01503 (18)
C15	−0.24804 (11)	0.95111 (5)	0.28163 (12)	0.0192 (2)
C16	−0.15038 (12)	0.91360 (5)	0.21998 (12)	0.01956 (19)
H16A	−0.1646	0.9067	0.1215	0.023*
C17	−0.03892 (11)	0.88778 (4)	0.29753 (11)	0.01678 (18)
C18	−0.00961 (10)	0.90076 (4)	0.45039 (11)	0.01475 (17)
C19	0.82791 (13)	0.69680 (6)	0.24429 (17)	0.0318 (3)
H19A	0.8643	0.6639	0.1997	0.048*
H19B	0.8956	0.7094	0.3218	0.048*
H19C	0.8115	0.7267	0.1745	0.048*
C20	0.49521 (14)	0.62163 (6)	0.48972 (18)	0.0333 (3)
H20A	0.4725	0.5826	0.4674	0.050*
H20B	0.4560	0.6322	0.5763	0.050*
H20C	0.5965	0.6262	0.5031	0.050*
C21	0.39362 (12)	0.91653 (5)	0.49091 (15)	0.0257 (2)
H21A	0.3114	0.9406	0.4878	0.039*
H21B	0.4459	0.9254	0.4115	0.039*
H21C	0.4528	0.9227	0.5796	0.039*
C22	0.15479 (13)	0.93845 (5)	0.97486 (12)	0.0238 (2)
H22A	0.1452	0.9520	1.0701	0.036*
H22B	0.2362	0.9559	0.9401	0.036*
H22C	0.1667	0.8978	0.9770	0.036*
C23	−0.29354 (12)	0.96294 (5)	0.77235 (14)	0.0259 (2)
H23A	−0.3698	0.9887	0.7853	0.039*
H23B	−0.2515	0.9498	0.8645	0.039*
H23C	−0.3297	0.9310	0.7156	0.039*
C24	0.04626 (13)	0.84603 (5)	0.22071 (12)	0.0231 (2)
H24A	0.0104	0.8448	0.1207	0.035*
H24B	0.0389	0.8089	0.2622	0.035*
H24C	0.1440	0.8577	0.2301	0.035*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0195 (4)	0.0190 (4)	0.0486 (6)	0.0025 (3)	0.0146 (4)	−0.0069 (4)
O2	0.0185 (4)	0.0128 (3)	0.0376 (5)	−0.0003 (3)	0.0030 (3)	−0.0021 (3)
O3	0.0148 (3)	0.0137 (3)	0.0323 (4)	0.0002 (3)	0.0084 (3)	0.0006 (3)
O4	0.0187 (4)	0.0191 (4)	0.0394 (5)	−0.0007 (3)	0.0131 (3)	0.0040 (3)
O5	0.0217 (4)	0.0293 (4)	0.0165 (4)	0.0057 (3)	0.0015 (3)	−0.0044 (3)
O6	0.0202 (4)	0.0194 (3)	0.0224 (4)	0.0060 (3)	0.0074 (3)	−0.0010 (3)
O7	0.0160 (3)	0.0176 (3)	0.0196 (4)	0.0032 (3)	0.0009 (3)	0.0009 (3)
O8	0.0217 (4)	0.0313 (4)	0.0257 (4)	0.0055 (3)	−0.0034 (3)	0.0020 (3)
C1	0.0160 (4)	0.0151 (4)	0.0225 (5)	0.0022 (3)	0.0059 (4)	0.0022 (3)
C2	0.0130 (4)	0.0153 (4)	0.0234 (5)	0.0004 (3)	0.0045 (4)	0.0005 (3)

C3	0.0156 (4)	0.0136 (4)	0.0283 (5)	0.0003 (3)	0.0039 (4)	−0.0018 (4)
C4	0.0172 (5)	0.0167 (4)	0.0326 (6)	0.0026 (3)	0.0078 (4)	−0.0031 (4)
C5	0.0171 (5)	0.0187 (5)	0.0485 (7)	−0.0010 (4)	0.0136 (5)	−0.0040 (5)
C6	0.0175 (5)	0.0156 (4)	0.0428 (7)	−0.0013 (4)	0.0106 (5)	−0.0032 (4)
C7	0.0147 (4)	0.0138 (4)	0.0263 (5)	0.0005 (3)	0.0057 (4)	−0.0005 (3)
C8	0.0151 (4)	0.0137 (4)	0.0220 (5)	0.0010 (3)	0.0030 (4)	−0.0002 (3)
C9	0.0154 (4)	0.0139 (4)	0.0184 (4)	0.0017 (3)	0.0041 (3)	0.0004 (3)
C10	0.0148 (4)	0.0137 (4)	0.0185 (4)	0.0010 (3)	0.0044 (3)	0.0007 (3)
C11	0.0156 (4)	0.0187 (4)	0.0176 (4)	0.0030 (3)	0.0022 (3)	0.0003 (3)
C12	0.0172 (4)	0.0179 (4)	0.0161 (4)	0.0009 (3)	0.0031 (3)	−0.0010 (3)
C13	0.0165 (4)	0.0153 (4)	0.0181 (4)	0.0029 (3)	0.0047 (3)	−0.0006 (3)
C14	0.0132 (4)	0.0143 (4)	0.0176 (4)	0.0009 (3)	0.0019 (3)	0.0012 (3)
C15	0.0184 (5)	0.0194 (4)	0.0194 (5)	−0.0014 (3)	0.0007 (4)	0.0014 (4)
C16	0.0205 (5)	0.0215 (5)	0.0167 (4)	−0.0011 (4)	0.0020 (4)	−0.0004 (4)
C17	0.0181 (4)	0.0156 (4)	0.0173 (4)	−0.0018 (3)	0.0049 (3)	−0.0005 (3)
C18	0.0152 (4)	0.0132 (4)	0.0165 (4)	0.0004 (3)	0.0043 (3)	0.0004 (3)
C19	0.0205 (5)	0.0279 (6)	0.0496 (8)	0.0011 (4)	0.0157 (5)	−0.0096 (5)
C20	0.0235 (6)	0.0236 (5)	0.0527 (8)	0.0009 (4)	0.0034 (5)	0.0119 (5)
C21	0.0184 (5)	0.0143 (4)	0.0451 (7)	−0.0001 (4)	0.0063 (5)	−0.0029 (4)
C22	0.0240 (5)	0.0293 (5)	0.0176 (5)	0.0024 (4)	−0.0006 (4)	−0.0004 (4)
C23	0.0184 (5)	0.0284 (5)	0.0322 (6)	0.0002 (4)	0.0078 (4)	−0.0034 (5)
C24	0.0285 (6)	0.0238 (5)	0.0178 (5)	0.0052 (4)	0.0057 (4)	−0.0028 (4)

Geometric parameters (Å, °)

O1—C4	1.3548 (13)	C11—C12	1.3988 (14)
O1—C19	1.4386 (15)	C11—H11A	0.9300
O2—C3	1.3733 (12)	C12—C13	1.3939 (14)
O2—C20	1.4332 (16)	C13—C14	1.3911 (14)
O3—C2	1.3736 (12)	C14—C18	1.4107 (13)
O3—C1	1.3798 (12)	C15—C16	1.4421 (15)
O4—C1	1.2106 (13)	C16—C17	1.3569 (15)
O5—C12	1.3565 (13)	C16—H16A	0.9300
O5—C22	1.4305 (14)	C17—C18	1.4586 (14)
O6—C13	1.3723 (12)	C17—C24	1.5015 (15)
O6—C23	1.4419 (14)	C19—H19A	0.9600
O7—C14	1.3755 (12)	C19—H19B	0.9600
O7—C15	1.3777 (13)	C19—H19C	0.9600
O8—C15	1.2108 (13)	C20—H20A	0.9600
C1—C9	1.4594 (14)	C20—H20B	0.9600
C2—C3	1.3924 (14)	C20—H20C	0.9600
C2—C7	1.4023 (14)	C21—H21A	0.9600
C3—C4	1.4043 (15)	C21—H21B	0.9600
C4—C5	1.3980 (15)	C21—H21C	0.9600
C5—C6	1.3822 (15)	C22—H22A	0.9600
C5—H5A	0.9300	C22—H22B	0.9600
C6—C7	1.4016 (15)	C22—H22C	0.9600
C6—H6A	0.9300	C23—H23A	0.9600
C7—C8	1.4496 (14)	C23—H23B	0.9600

C8—C9	1.3605 (14)	C23—H23C	0.9600
C8—C21	1.4977 (14)	C24—H24A	0.9600
C9—C10	1.4945 (14)	C24—H24B	0.9600
C10—C11	1.3906 (14)	C24—H24C	0.9600
C10—C18	1.4168 (14)		
C4—O1—C19	116.67 (9)	O8—C15—O7	116.93 (10)
C3—O2—C20	112.74 (10)	O8—C15—C16	126.78 (10)
C2—O3—C1	121.26 (8)	O7—C15—C16	116.26 (9)
C12—O5—C22	117.03 (9)	C17—C16—C15	123.69 (10)
C13—O6—C23	112.73 (8)	C17—C16—H16A	118.2
C14—O7—C15	121.80 (8)	C15—C16—H16A	118.2
O4—C1—O3	116.55 (9)	C16—C17—C18	118.98 (9)
O4—C1—C9	125.29 (9)	C16—C17—C24	117.60 (10)
O3—C1—C9	118.15 (9)	C18—C17—C24	123.41 (9)
O3—C2—C3	116.39 (9)	C14—C18—C10	116.55 (9)
O3—C2—C7	121.30 (9)	C14—C18—C17	116.36 (9)
C3—C2—C7	122.30 (9)	C10—C18—C17	127.08 (9)
O2—C3—C2	120.39 (9)	O1—C19—H19A	109.5
O2—C3—C4	120.81 (9)	O1—C19—H19B	109.5
C2—C3—C4	118.76 (9)	H19A—C19—H19B	109.5
O1—C4—C5	124.40 (10)	O1—C19—H19C	109.5
O1—C4—C3	115.77 (10)	H19A—C19—H19C	109.5
C5—C4—C3	119.82 (10)	H19B—C19—H19C	109.5
C6—C5—C4	120.12 (10)	O2—C20—H20A	109.5
C6—C5—H5A	119.9	O2—C20—H20B	109.5
C4—C5—H5A	119.9	H20A—C20—H20B	109.5
C5—C6—C7	121.60 (10)	O2—C20—H20C	109.5
C5—C6—H6A	119.2	H20A—C20—H20C	109.5
C7—C6—H6A	119.2	H20B—C20—H20C	109.5
C6—C7—C2	117.31 (9)	C8—C21—H21A	109.5
C6—C7—C8	123.73 (9)	C8—C21—H21B	109.5
C2—C7—C8	118.93 (9)	H21A—C21—H21B	109.5
C9—C8—C7	118.78 (9)	C8—C21—H21C	109.5
C9—C8—C21	121.61 (9)	H21A—C21—H21C	109.5
C7—C8—C21	119.58 (9)	H21B—C21—H21C	109.5
C8—C9—C1	121.36 (9)	O5—C22—H22A	109.5
C8—C9—C10	122.93 (9)	O5—C22—H22B	109.5
C1—C9—C10	115.60 (9)	H22A—C22—H22B	109.5
C11—C10—C18	120.57 (9)	O5—C22—H22C	109.5
C11—C10—C9	115.52 (9)	H22A—C22—H22C	109.5
C18—C10—C9	123.91 (9)	H22B—C22—H22C	109.5
C10—C11—C12	121.19 (9)	O6—C23—H23A	109.5
C10—C11—H11A	119.4	O6—C23—H23B	109.5
C12—C11—H11A	119.4	H23A—C23—H23B	109.5
O5—C12—C13	115.33 (9)	O6—C23—H23C	109.5
O5—C12—C11	125.00 (9)	H23A—C23—H23C	109.5
C13—C12—C11	119.63 (9)	H23B—C23—H23C	109.5
O6—C13—C14	119.86 (9)	C17—C24—H24A	109.5
O6—C13—C12	121.34 (9)	C17—C24—H24B	109.5

C14—C13—C12	118.79 (9)	H24A—C24—H24B	109.5
O7—C14—C13	114.02 (8)	C17—C24—H24C	109.5
O7—C14—C18	122.74 (9)	H24A—C24—H24C	109.5
C13—C14—C18	123.23 (9)	H24B—C24—H24C	109.5
C2—O3—C1—O4	178.16 (10)	C8—C9—C10—C18	103.55 (13)
C2—O3—C1—C9	−0.94 (15)	C1—C9—C10—C18	−80.20 (12)
C1—O3—C2—C3	−175.74 (10)	C18—C10—C11—C12	1.57 (15)
C1—O3—C2—C7	4.15 (16)	C9—C10—C11—C12	−178.01 (9)
C20—O2—C3—C2	−103.96 (12)	C22—O5—C12—C13	−173.06 (9)
C20—O2—C3—C4	78.36 (14)	C22—O5—C12—C11	9.01 (16)
O3—C2—C3—O2	1.16 (16)	C10—C11—C12—O5	177.12 (10)
C7—C2—C3—O2	−178.72 (10)	C10—C11—C12—C13	−0.73 (16)
O3—C2—C3—C4	178.89 (10)	C23—O6—C13—C14	99.08 (11)
C7—C2—C3—C4	−1.00 (17)	C23—O6—C13—C12	−81.83 (12)
C19—O1—C4—C5	−7.65 (19)	O5—C12—C13—O6	2.19 (15)
C19—O1—C4—C3	172.97 (11)	C11—C12—C13—O6	−179.76 (9)
O2—C3—C4—O1	0.06 (17)	O5—C12—C13—C14	−178.71 (9)
C2—C3—C4—O1	−177.65 (10)	C11—C12—C13—C14	−0.66 (15)
O2—C3—C4—C5	−179.34 (11)	C15—O7—C14—C13	176.70 (9)
C2—C3—C4—C5	2.94 (18)	C15—O7—C14—C18	−4.07 (14)
O1—C4—C5—C6	178.29 (13)	O6—C13—C14—O7	−0.41 (14)
C3—C4—C5—C6	−2.4 (2)	C12—C13—C14—O7	−179.52 (9)
C4—C5—C6—C7	−0.2 (2)	O6—C13—C14—C18	−179.63 (9)
C5—C6—C7—C2	2.14 (19)	C12—C13—C14—C18	1.25 (15)
C5—C6—C7—C8	−175.98 (12)	C14—O7—C15—O8	−179.20 (9)
O3—C2—C7—C6	178.61 (11)	C14—O7—C15—C16	2.60 (14)
C3—C2—C7—C6	−1.51 (17)	O8—C15—C16—C17	−176.66 (11)
O3—C2—C7—C8	−3.18 (16)	O7—C15—C16—C17	1.34 (16)
C3—C2—C7—C8	176.71 (10)	C15—C16—C17—C18	−3.75 (16)
C6—C7—C8—C9	177.07 (11)	C15—C16—C17—C24	174.94 (10)
C2—C7—C8—C9	−1.03 (16)	O7—C14—C18—C10	−179.60 (9)
C6—C7—C8—C21	−0.87 (18)	C13—C14—C18—C10	−0.43 (14)
C2—C7—C8—C21	−178.97 (11)	O7—C14—C18—C17	1.53 (14)
C7—C8—C9—C1	4.21 (16)	C13—C14—C18—C17	−179.31 (9)
C21—C8—C9—C1	−177.89 (11)	C11—C10—C18—C14	−0.98 (14)
C7—C8—C9—C10	−179.74 (10)	C9—C10—C18—C14	178.57 (9)
C21—C8—C9—C10	−1.85 (17)	C11—C10—C18—C17	177.76 (10)
O4—C1—C9—C8	177.67 (11)	C9—C10—C18—C17	−2.69 (16)
O3—C1—C9—C8	−3.33 (15)	C16—C17—C18—C14	2.26 (14)
O4—C1—C9—C10	1.35 (16)	C24—C17—C18—C14	−176.35 (9)
O3—C1—C9—C10	−179.65 (9)	C16—C17—C18—C10	−176.47 (10)
C8—C9—C10—C11	−76.88 (13)	C24—C17—C18—C10	4.91 (16)
C1—C9—C10—C11	99.37 (11)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C21—H21A···O6^{i}	0.96	2.55	3.2921 (15)	134

C22—H22A···O6^{ii}	0.96	2.52	3.4385 (14)	161
C22—H22B···O8^{i}	0.96	2.56	3.4401 (15)	152
C6—H6A···Cg1^{iii}	0.93	2.92	3.6706 (12)	138
C19—H19A···Cg2^{iv}	0.96	2.60	3.5446 (14)	170

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

Fig. 1

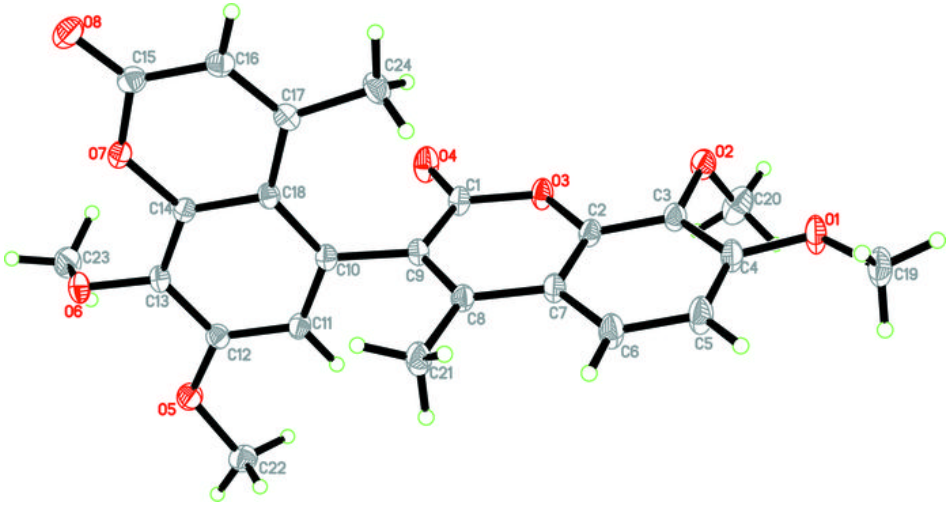
O8
C15
C16
C24
C17
O7
O4
C1
O3
C2
C3
O2
C20
C14
C18
O1
C23
C10
C9
C4
C19
O6
C13
C8
C7
C11
C6
C5
C12
C21
O5
C22

Fig. 2