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9-(3,4-Dimethoxyphenyl)-3,4,5,6,7,9-hexahydroanthene-1,8(2H)-dione

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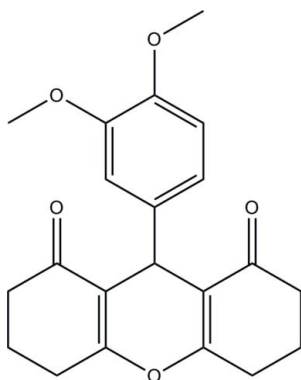
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.048; wR factor = 0.130; data-to-parameter ratio = 21.6.

In the title compound, $\text{C}_{21}\text{H}_{22}\text{O}_5$, the mean planes of the pyran and dimethoxyphenyl rings are nearly perpendicular to one another, with the dihedral angle between them being $88.21(8)^\circ$. The pyran ring adopts a boat conformation whereas the two fused cyclohexane rings adopt envelope conformations. In the crystal, molecules are linked into a three-dimensional network by intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For condensation reactions between carbonyl compounds with active methylene compounds, see: Chalais *et al.* (1985); Prajapati & Sanduh (1993); Texier-Boullet & Foucaud (1982); Jone (1967). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986). For ring conformations, see: Cremer & Pople (1975).



‡ Thomson Reuters ResearcherID: A-5523-2009.

§ Thomson Reuters ResearcherID: A-3561-2009.

Experimental

Crystal data

$\text{C}_{21}\text{H}_{22}\text{O}_5$
 $M_r = 354.39$
 Monoclinic, $P2_1/c$
 $a = 8.7733(3)$ Å
 $b = 15.2246(5)$ Å
 $c = 14.6646(4)$ Å
 $\beta = 116.891(2)^\circ$

$V = 1746.95(10)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 100$ K
 $0.58 \times 0.30 \times 0.18$ mm

Data collection

Bruker SMART APEXII CCD
 area-detector diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2009)
 $T_{\min} = 0.947$, $T_{\max} = 0.983$

19254 measured reflections
 5118 independent reflections
 4066 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.130$
 $S = 1.05$
 5118 reflections

237 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.35$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.28$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C3}-\text{H3A}\cdots\text{O4}^i$	0.99	2.41	3.3647 (18)	161
$\text{C5}-\text{H5A}\cdots\text{O5}^{ii}$	0.99	2.56	3.2366 (17)	126
$\text{C9}-\text{H9A}\cdots\text{O2}^{iii}$	0.99	2.55	3.3210 (17)	135
$\text{C10}-\text{H10B}\cdots\text{O5}^{iii}$	0.99	2.50	3.4822 (19)	173
$\text{C21}-\text{H21C}\cdots\text{O2}^{iv}$	0.98	2.57	3.1069 (18)	114

Symmetry codes: (i) $x - 1, y, z$; (ii) $-x, y - \frac{1}{2}, -z + \frac{1}{2}$; (iii) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$; (iv) $x, -y + \frac{3}{2}, z - \frac{1}{2}$.

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); 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: BQ2301).

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supplementary materials

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9-(3,4-Dimethoxyphenyl)-3,4,5,6,7,9-hexahydroxanthene-1,8(2H)-dione

S. H. Mehdi, R. Hashim, R. M. Ghalib, C. S. Yeap and H.-K. Fun

Comment

The most important synthetic method for the preparation of substituted alkenes is the condensation reaction between carbonyl compounds with active methylene compounds. Xonotlite, cadmium iodide aluminium oxide and other Lewis acids and bases have been previously used for such type of reactions (Chalais *et al.*, 1985; Prajapati & Sanduh, 1993; Texier-Boullet & Foucaud, 1982; Jone, 1967). In this paper we are reporting the synthesis of title compound (Fig. 1) by simple heating of 1,3-cyclohexanedione with veratraldehyde in acetic acid without the use of any catalyst. The structure is supported by spectral analysis like IR, ^1H NMR, ^{13}C NMR and finally confirmed by *x*-ray crystallography.

In the title compound, the mean plane of pyran ring and the dimethoxyphenyl ring are nearly perpendicular to each other with the dihedral angle between them being $88.21(8)^\circ$. The dimethoxyphenyl ring is planar with the torsion angle of $\text{C}20\text{--O}4\text{--C}18\text{--C}19 = -5.34(18)^\circ$ and $\text{C}21\text{--O}5\text{--C}17\text{--C}16 = -5.67(18)^\circ$. The two cyclohexane rings adopt envelope conformations [puckering amplitude $Q = 0.4284(16) \text{ \AA}$, $\theta = 124.3(2)^\circ$, $\phi = 359.0(3)^\circ$; $Q = 0.4899(15) \text{ \AA}$, $\theta = 57.91(18)^\circ$, $\phi = 132.8(2)^\circ$], whereas the pyran ring adopt a boat conformation [$Q = 0.2125(13) \text{ \AA}$, $\theta = 77.9(4)^\circ$, $\phi = 187.0(4)^\circ$] (Cremer & Pople, 1975). In the crystal structure, the molecules are linked into a three-dimensional network (Fig. 2) by intermolecular $\text{C}3\text{--H}3\text{A}\cdots\text{O}4$, $\text{C}5\text{--H}5\text{A}\cdots\text{O}5$, $\text{C}9\text{--H}9\text{A}\cdots\text{O}2$, $\text{C}10\text{--H}10\text{B}\cdots\text{O}5$ and $\text{C}21\text{--H}21\text{C}\cdots\text{O}2$ hydrogen bonds (Table 1).

Experimental

A mixture of 1,3-cyclohexanedione (1.12 g m, 10 mmol) and veratraldehyde (1.66 g m, 10 mmol) was heated in 25 ml of glacial acetic acid for three hours. Completion of the reaction was monitored by TLC. The reaction mixture was dried on rotary evaporator under reduced pressure. The crude mixture thus obtained was successively treated with chloroform and ethanol. The ethanol fraction on crystallization furnished cream colored crystals of title compound (yield 90%, m. pt. 216°C). IR (KBr) ν_{max} : 2966, 2933, 2868, 2833, 1664, 1620, 1513, 1441, 1358, 1261, 1240, 1171, 1132, 1025, 956, 906, 859, 807, 726 cm^{-1} . ^1H NMR (300 MHz, DMSO- d_6): δ 1.75–1.88 (m, 2H), 1.94–2.06 (m, 2H), 2.20–2.38 (m, 42H), 2.48–2.68 (m, 4H), 4.21 (s, 1H), 6.78–6.96 (3H, m). ^{13}C NMR (75 MHz, DMSO- d_6): δ 21.6, 26.9, 30.2, 36.6, 56.8, 112.6, 114.4, 115.8, 123.2, 136.4, 146.2, 148.4, 198.2. IR spectrum was taken on Shimadzu IR-408 Perkin Elmer 1800 (FTIR). ^1H NMR was recorded on Bruker Avance 300 MHz with TMS as an internal standard and 75 MHz for ^{13}C NMR. Spectrum was recorded in DMSO- d_6 . The melting point was taken on Thermo Fisher digital melting point apparatus of IA9000 series and is uncorrected.

Refinement

All hydrogen atoms were positioned geometrically [$\text{C--H} = 0.95\text{--}1.00 \text{ \AA}$] and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5 U_{\text{eq}}(\text{C})$.

Figures

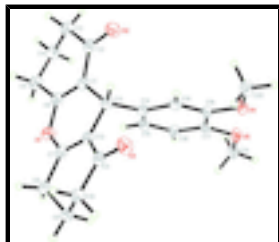


Fig. 1. The molecular structure of the title compound, with atom labels and 50% probability ellipsoids for non-H atoms.

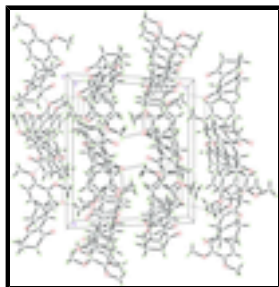


Fig. 2. The crystal packing of title compound, viewed down *a* axis, showing molecules linked into a three-dimensional network. Hydrogen bonds are shown as dashed lines.

9-(3,4-Dimethoxyphenyl)-3,4,5,6,7,9-hexahydroxanthene-1,8(2H)-dione

Crystal data

$C_{21}H_{22}O_5$

$M_r = 354.39$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.7733$ (3) Å

$b = 15.2246$ (5) Å

$c = 14.6646$ (4) Å

$\beta = 116.891$ (2)°

$V = 1746.95$ (10) Å³

$Z = 4$

$F(000) = 752$

$D_x = 1.347$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 6186 reflections

$\theta = 2.6$ – 30.0 °

$\mu = 0.10$ mm⁻¹

$T = 100$ K

Block, yellow

$0.58 \times 0.30 \times 0.18$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

φ and ω scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2009)

$T_{\min} = 0.947$, $T_{\max} = 0.983$

19254 measured reflections

5118 independent reflections

4066 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.042$

$\theta_{\max} = 30.1$ °, $\theta_{\min} = 2.1$ °

$h = -12 \rightarrow 9$

$k = -21 \rightarrow 18$

$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.048$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.130$	H-atom parameters constrained
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.0608P)^2 + 0.471P]$
5118 reflections	where $P = (F_o^2 + 2F_c^2)/3$
237 parameters	$(\Delta/\sigma)_{\max} < 0.001$
0 restraints	$\Delta\rho_{\max} = 0.35 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	-0.23678 (10)	0.45054 (6)	0.11930 (7)	0.0253 (2)
O2	-0.11008 (12)	0.63020 (7)	0.40709 (7)	0.0307 (2)
O3	0.34649 (11)	0.44914 (7)	0.36290 (7)	0.0293 (2)
O4	0.46323 (11)	0.77498 (6)	0.38751 (7)	0.0252 (2)
O5	0.31222 (11)	0.84133 (6)	0.20530 (7)	0.0254 (2)
C1	-0.16143 (15)	0.54404 (8)	0.26377 (9)	0.0212 (2)
C2	-0.21561 (16)	0.59572 (9)	0.32848 (10)	0.0240 (3)
C3	-0.40468 (17)	0.60323 (10)	0.29722 (11)	0.0296 (3)
H3A	-0.4278	0.6619	0.3173	0.035*
H3B	-0.4359	0.5588	0.3352	0.035*
C4	-0.51736 (17)	0.59051 (10)	0.18363 (11)	0.0317 (3)
H4A	-0.5069	0.6424	0.1461	0.038*
H4B	-0.6381	0.5859	0.1706	0.038*
C5	-0.46837 (16)	0.50797 (10)	0.14364 (10)	0.0269 (3)
H5A	-0.5066	0.4553	0.1672	0.032*
H5B	-0.5268	0.5083	0.0680	0.032*

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C6	-0.27955 (15)	0.50362 (9)	0.18052 (9)	0.0220 (2)
C7	-0.06793 (15)	0.42479 (9)	0.15659 (9)	0.0219 (2)
C8	-0.04677 (16)	0.35533 (9)	0.09158 (10)	0.0259 (3)
H8A	-0.1171	0.3700	0.0185	0.031*
H8B	-0.0871	0.2984	0.1051	0.031*
C9	0.14076 (16)	0.34741 (9)	0.11409 (10)	0.0260 (3)
H9A	0.1567	0.2942	0.0805	0.031*
H9B	0.1739	0.3991	0.0862	0.031*
C10	0.25342 (16)	0.34178 (9)	0.22886 (10)	0.0263 (3)
H10A	0.2253	0.2876	0.2555	0.032*
H10B	0.3746	0.3381	0.2427	0.032*
C11	0.22973 (15)	0.42044 (8)	0.28392 (10)	0.0225 (2)
C12	0.05790 (14)	0.45987 (8)	0.24136 (9)	0.0205 (2)
C13	0.02759 (14)	0.53739 (8)	0.29541 (9)	0.0199 (2)
H13A	0.0892	0.5268	0.3708	0.024*
C14	0.09637 (14)	0.62177 (8)	0.27121 (9)	0.0200 (2)
C15	0.01165 (15)	0.66138 (9)	0.17626 (10)	0.0229 (3)
H15A	-0.0931	0.6372	0.1269	0.028*
C16	0.07768 (15)	0.73634 (8)	0.15190 (9)	0.0225 (3)
H16A	0.0175	0.7631	0.0867	0.027*
C17	0.23114 (15)	0.77157 (8)	0.22307 (9)	0.0213 (2)
C18	0.31465 (14)	0.73401 (8)	0.32119 (9)	0.0206 (2)
C19	0.24887 (14)	0.65911 (8)	0.34429 (9)	0.0210 (2)
H19A	0.3075	0.6330	0.4100	0.025*
C20	0.54591 (18)	0.74117 (11)	0.48942 (10)	0.0346 (3)
H20A	0.6472	0.7767	0.5304	0.052*
H20B	0.5804	0.6802	0.4879	0.052*
H20C	0.4668	0.7435	0.5198	0.052*
C21	0.23844 (18)	0.87415 (10)	0.10281 (10)	0.0292 (3)
H21A	0.2185	0.8253	0.0552	0.044*
H21B	0.3167	0.9165	0.0959	0.044*
H21C	0.1296	0.9031	0.0871	0.044*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0120 (4)	0.0367 (5)	0.0227 (4)	0.0006 (4)	0.0037 (3)	-0.0034 (4)
O2	0.0227 (5)	0.0359 (5)	0.0304 (5)	0.0013 (4)	0.0094 (4)	-0.0061 (4)
O3	0.0163 (4)	0.0326 (5)	0.0286 (5)	0.0025 (4)	0.0011 (4)	-0.0044 (4)
O4	0.0158 (4)	0.0308 (5)	0.0219 (4)	-0.0054 (4)	0.0021 (3)	-0.0042 (4)
O5	0.0195 (4)	0.0279 (5)	0.0243 (4)	-0.0053 (4)	0.0060 (4)	0.0001 (4)
C1	0.0141 (5)	0.0245 (6)	0.0237 (6)	0.0015 (4)	0.0072 (4)	0.0024 (5)
C2	0.0185 (5)	0.0238 (6)	0.0290 (6)	0.0017 (5)	0.0102 (5)	0.0021 (5)
C3	0.0197 (6)	0.0320 (7)	0.0388 (7)	0.0028 (5)	0.0148 (6)	-0.0020 (6)
C4	0.0181 (6)	0.0386 (8)	0.0367 (7)	0.0058 (5)	0.0108 (5)	0.0064 (6)
C5	0.0129 (5)	0.0376 (7)	0.0253 (6)	0.0017 (5)	0.0044 (5)	0.0022 (5)
C6	0.0139 (5)	0.0278 (6)	0.0230 (6)	0.0015 (5)	0.0071 (4)	0.0023 (5)
C7	0.0129 (5)	0.0268 (6)	0.0240 (6)	-0.0001 (4)	0.0065 (4)	0.0013 (5)

C8	0.0187 (6)	0.0313 (7)	0.0256 (6)	-0.0031 (5)	0.0082 (5)	-0.0054 (5)
C9	0.0188 (6)	0.0306 (7)	0.0284 (6)	-0.0010 (5)	0.0106 (5)	-0.0045 (5)
C10	0.0181 (6)	0.0266 (6)	0.0308 (7)	0.0027 (5)	0.0081 (5)	-0.0016 (5)
C11	0.0151 (5)	0.0238 (6)	0.0254 (6)	-0.0002 (4)	0.0065 (5)	0.0014 (5)
C12	0.0138 (5)	0.0229 (6)	0.0222 (5)	0.0005 (4)	0.0059 (4)	0.0007 (4)
C13	0.0121 (5)	0.0243 (6)	0.0199 (5)	0.0004 (4)	0.0043 (4)	0.0000 (4)
C14	0.0125 (5)	0.0241 (6)	0.0217 (5)	0.0013 (4)	0.0063 (4)	-0.0020 (4)
C15	0.0126 (5)	0.0263 (6)	0.0233 (6)	-0.0005 (4)	0.0023 (4)	-0.0013 (5)
C16	0.0159 (5)	0.0261 (6)	0.0204 (5)	0.0017 (5)	0.0039 (4)	0.0007 (5)
C17	0.0161 (5)	0.0227 (6)	0.0240 (6)	-0.0001 (4)	0.0081 (5)	-0.0030 (5)
C18	0.0112 (5)	0.0256 (6)	0.0214 (5)	-0.0005 (4)	0.0044 (4)	-0.0055 (5)
C19	0.0136 (5)	0.0270 (6)	0.0191 (5)	0.0018 (4)	0.0045 (4)	-0.0023 (4)
C20	0.0244 (7)	0.0513 (9)	0.0197 (6)	-0.0129 (6)	0.0027 (5)	-0.0044 (6)
C21	0.0289 (7)	0.0315 (7)	0.0244 (6)	-0.0070 (6)	0.0095 (5)	-0.0010 (5)

Geometric parameters (Å, °)

O1—C6	1.3800 (15)	C9—C10	1.5205 (19)
O1—C7	1.3836 (14)	C9—H9A	0.9900
O2—C2	1.2231 (16)	C9—H9B	0.9900
O3—C11	1.2283 (15)	C10—C11	1.5103 (18)
O4—C18	1.3738 (14)	C10—H10A	0.9900
O4—C20	1.4297 (16)	C10—H10B	0.9900
O5—C17	1.3674 (15)	C11—C12	1.4736 (16)
O5—C21	1.4309 (16)	C12—C13	1.5108 (17)
C1—C6	1.3411 (17)	C13—C14	1.5277 (17)
C1—C2	1.4676 (17)	C13—H13A	1.0000
C1—C13	1.5099 (16)	C14—C15	1.3852 (17)
C2—C3	1.5122 (17)	C14—C19	1.4017 (16)
C3—C4	1.517 (2)	C15—C16	1.3975 (18)
C3—H3A	0.9900	C15—H15A	0.9500
C3—H3B	0.9900	C16—C17	1.3851 (16)
C4—C5	1.528 (2)	C16—H16A	0.9500
C4—H4A	0.9900	C17—C18	1.4074 (17)
C4—H4B	0.9900	C18—C19	1.3874 (18)
C5—C6	1.4935 (17)	C19—H19A	0.9500
C5—H5A	0.9900	C20—H20A	0.9800
C5—H5B	0.9900	C20—H20B	0.9800
C7—C12	1.3446 (17)	C20—H20C	0.9800
C7—C8	1.4906 (18)	C21—H21A	0.9800
C8—C9	1.5294 (17)	C21—H21B	0.9800
C8—H8A	0.9900	C21—H21C	0.9800
C8—H8B	0.9900		
C6—O1—C7	117.80 (9)	C9—C10—H10A	109.3
C18—O4—C20	116.55 (10)	C11—C10—H10B	109.3
C17—O5—C21	116.37 (10)	C9—C10—H10B	109.3
C6—C1—C2	119.46 (11)	H10A—C10—H10B	107.9
C6—C1—C13	122.55 (11)	O3—C11—C12	120.76 (12)
C2—C1—C13	117.98 (11)	O3—C11—C10	121.91 (11)

supplementary materials

O2—C2—C1	120.73 (11)	C12—C11—C10	117.29 (11)
O2—C2—C3	120.81 (12)	C7—C12—C11	118.98 (11)
C1—C2—C3	118.43 (11)	C7—C12—C13	121.98 (10)
C2—C3—C4	113.83 (11)	C11—C12—C13	119.03 (10)
C2—C3—H3A	108.8	C1—C13—C12	108.70 (10)
C4—C3—H3A	108.8	C1—C13—C14	111.50 (10)
C2—C3—H3B	108.8	C12—C13—C14	110.69 (9)
C4—C3—H3B	108.8	C1—C13—H13A	108.6
H3A—C3—H3B	107.7	C12—C13—H13A	108.6
C3—C4—C5	111.70 (11)	C14—C13—H13A	108.6
C3—C4—H4A	109.3	C15—C14—C19	118.98 (11)
C5—C4—H4A	109.3	C15—C14—C13	120.72 (10)
C3—C4—H4B	109.3	C19—C14—C13	120.28 (11)
C5—C4—H4B	109.3	C14—C15—C16	121.10 (11)
H4A—C4—H4B	107.9	C14—C15—H15A	119.4
C6—C5—C4	110.85 (11)	C16—C15—H15A	119.4
C6—C5—H5A	109.5	C17—C16—C15	119.84 (11)
C4—C5—H5A	109.5	C17—C16—H16A	120.1
C6—C5—H5B	109.5	C15—C16—H16A	120.1
C4—C5—H5B	109.5	O5—C17—C16	124.70 (11)
H5A—C5—H5B	108.1	O5—C17—C18	115.81 (10)
C1—C6—O1	122.37 (10)	C16—C17—C18	119.48 (11)
C1—C6—C5	125.67 (12)	O4—C18—C19	124.51 (11)
O1—C6—C5	111.95 (11)	O4—C18—C17	115.33 (11)
C12—C7—O1	122.51 (11)	C19—C18—C17	120.13 (11)
C12—C7—C8	125.84 (11)	C18—C19—C14	120.35 (11)
O1—C7—C8	111.66 (10)	C18—C19—H19A	119.8
C7—C8—C9	110.54 (10)	C14—C19—H19A	119.8
C7—C8—H8A	109.5	O4—C20—H20A	109.5
C9—C8—H8A	109.5	O4—C20—H20B	109.5
C7—C8—H8B	109.5	H20A—C20—H20B	109.5
C9—C8—H8B	109.5	O4—C20—H20C	109.5
H8A—C8—H8B	108.1	H20A—C20—H20C	109.5
C10—C9—C8	109.91 (11)	H20B—C20—H20C	109.5
C10—C9—H9A	109.7	O5—C21—H21A	109.5
C8—C9—H9A	109.7	O5—C21—H21B	109.5
C10—C9—H9B	109.7	H21A—C21—H21B	109.5
C8—C9—H9B	109.7	O5—C21—H21C	109.5
H9A—C9—H9B	108.2	H21A—C21—H21C	109.5
C11—C10—C9	111.68 (11)	H21B—C21—H21C	109.5
C11—C10—H10A	109.3		
C6—C1—C2—O2	176.94 (12)	C10—C11—C12—C13	178.63 (11)
C13—C1—C2—O2	-1.98 (19)	C6—C1—C13—C12	-17.44 (16)
C6—C1—C2—C3	-1.40 (18)	C2—C1—C13—C12	161.44 (11)
C13—C1—C2—C3	179.68 (11)	C6—C1—C13—C14	104.85 (14)
O2—C2—C3—C4	156.87 (13)	C2—C1—C13—C14	-76.26 (14)
C1—C2—C3—C4	-24.78 (18)	C7—C12—C13—C1	19.86 (16)
C2—C3—C4—C5	49.18 (16)	C11—C12—C13—C1	-158.95 (11)
C3—C4—C5—C6	-47.29 (15)	C7—C12—C13—C14	-102.92 (13)

C2—C1—C6—O1	-176.73 (11)	C11—C12—C13—C14	78.27 (13)
C13—C1—C6—O1	2.14 (19)	C1—C13—C14—C15	-47.46 (15)
C2—C1—C6—C5	2.0 (2)	C12—C13—C14—C15	73.68 (13)
C13—C1—C6—C5	-179.18 (12)	C1—C13—C14—C19	134.08 (11)
C7—O1—C6—C1	13.08 (18)	C12—C13—C14—C19	-104.78 (12)
C7—O1—C6—C5	-165.77 (11)	C19—C14—C15—C16	1.38 (18)
C4—C5—C6—C1	23.06 (18)	C13—C14—C15—C16	-177.10 (11)
C4—C5—C6—O1	-158.14 (11)	C14—C15—C16—C17	0.62 (19)
C6—O1—C7—C12	-10.48 (18)	C21—O5—C17—C16	-5.67 (18)
C6—O1—C7—C8	169.57 (11)	C21—O5—C17—C18	173.51 (11)
C12—C7—C8—C9	-16.76 (18)	C15—C16—C17—O5	175.93 (11)
O1—C7—C8—C9	163.19 (11)	C15—C16—C17—C18	-3.22 (18)
C7—C8—C9—C10	48.72 (15)	C20—O4—C18—C19	-5.34 (18)
C8—C9—C10—C11	-57.64 (15)	C20—O4—C18—C17	176.71 (11)
C9—C10—C11—O3	-148.95 (12)	O5—C17—C18—O4	2.67 (16)
C9—C10—C11—C12	33.42 (16)	C16—C17—C18—O4	-178.10 (11)
O1—C7—C12—C11	171.57 (11)	O5—C17—C18—C19	-175.38 (10)
C8—C7—C12—C11	-8.48 (19)	C16—C17—C18—C19	3.84 (18)
O1—C7—C12—C13	-7.24 (19)	O4—C18—C19—C14	-179.71 (11)
C8—C7—C12—C13	172.71 (12)	C17—C18—C19—C14	-1.85 (18)
O3—C11—C12—C7	-177.87 (12)	C15—C14—C19—C18	-0.75 (17)
C10—C11—C12—C7	-0.21 (17)	C13—C14—C19—C18	177.73 (10)
O3—C11—C12—C13	0.98 (18)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C3—H3A \cdots O4 ⁱ	0.99	2.41	3.3647 (18)	161
C5—H5A \cdots O5 ⁱⁱ	0.99	2.56	3.2366 (17)	126
C9—H9A \cdots O2 ⁱⁱ	0.99	2.55	3.3210 (17)	135
C10—H10B \cdots O5 ⁱⁱⁱ	0.99	2.50	3.4822 (19)	173
C21—H21C \cdots O2 ^{iv}	0.98	2.57	3.1069 (18)	114

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

Fig. 1

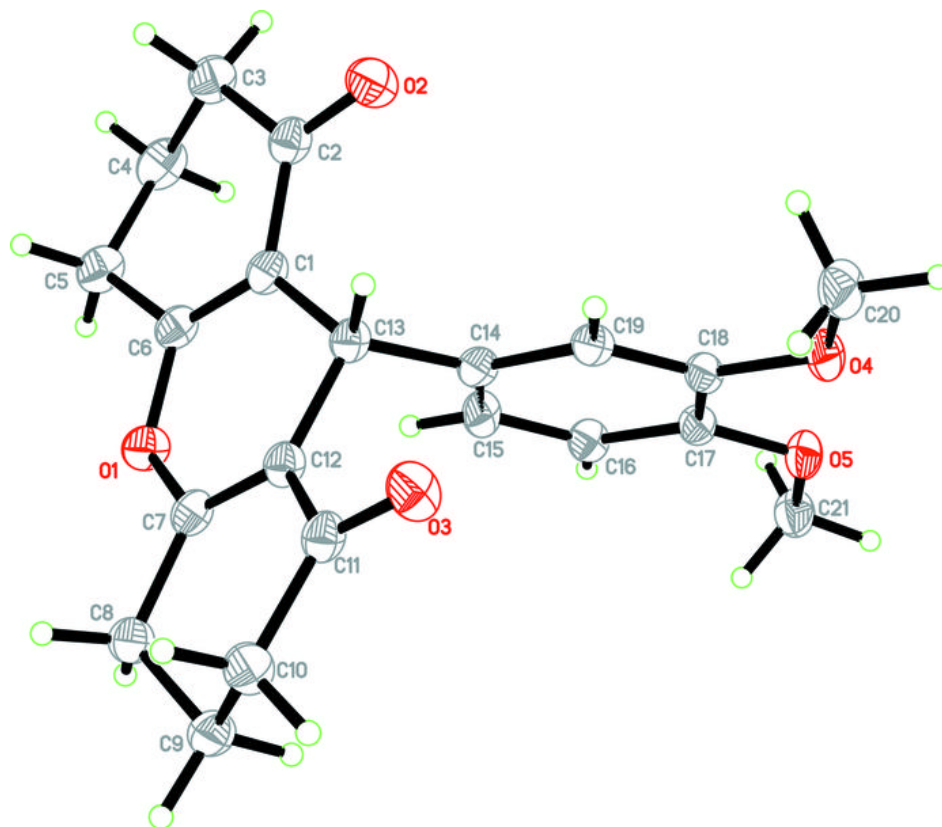


Fig. 2

