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1,5-Bis(3,4-dimethoxyphenyl)penta-1,4-dien-3-one

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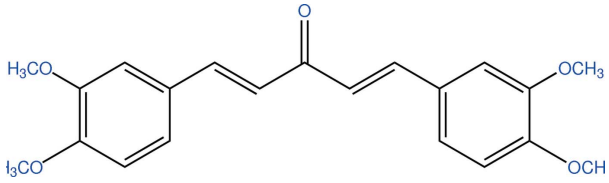
Key indicators: single-crystal X-ray study; T = 103 K; mean σ(C–C) = 0.001 Å; R factor = 0.041; wR factor = 0.114; data-to-parameter ratio = 21.3.

The title compound, $C_{21}H_{22}O_5$, a derivative of the antioxidant, anti-inflammatory compound curcumin, crystallizes with two molecules (*A* and *B*) in the asymmetric unit. The mean planes of the two 3,4-dimethoxyphenyl groups in *A* and *B* make dihedral angles of 57.9 (7) and 33.4 (3)°. An extensive network of intermolecular C—H···O hydrogen bonds stabilizes the crystal packing.

Related literature

For biological activity of the title compound and curcumin, see: Reksohadiprodjo *et al.* (2004). For hydrogen-bonding interactions and related structures, see: Girija *et al.* (2004) and Butcher *et al.* (2006). For related literature, see: Furniss *et al.* (1989).



Experimental

Crystal data

$C_{21}H_{22}O_5$
M_r = 354.39
Monoclinic, $P2_1/c$
a = 19.1646 (13) Å
b = 7.7488 (5) Å
c = 24.8495 (17) Å
β = 100.4140 (10)°
V = 3629.4 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 103 K
0.56 × 0.35 × 0.24 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan *DENZO/SCALEPACK* (Otwinowski & Minor, 1997)
T_{min} = 0.950, T_{max} = 0.988
40342 measured reflections
10146 independent reflections
8569 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.114
S = 1.03
10146 reflections
477 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6*A*—H6*AA*···O3*B*^{i}	0.95	2.54	3.2898 (13)	137
C8*A*—H8*AC*···O3*A*^{i}	0.98	2.58	3.5513 (15)	173
C20*A*—H20*C*···O1*A*^{i}	0.98	2.57	3.5085 (15)	161
C8*B*—H8*BA*···O4*A*^{ii}	0.98	2.41	3.2334 (13)	141
C8*B*—H8*BB*···O5*B*^{iii}	0.98	2.54	3.4056 (14)	147
C10*B*—H10*B*···O3*A*^{iv}	0.95	2.54	3.4325 (13)	157
C12*B*—H12*B*···O3*A*	0.95	2.47	3.3968 (13)	164

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Bruker, 2000).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2246).

References

Bruker (2000). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Butcher, R. J., Yathirajan, H. S., Sarojini, B. K., Narayana, B. & Vijaya Raj, K. K. (2006). *Acta Cryst.* E**62**, o1973–o1975.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Furniss, B. S., Hannaford, A. J., Smith, P. W. G. & Tatchell, A. R. (1989). *Vogel's Textbook of Practical Organic Chemistry*, 5th ed., p. 1033. UK: Longman.
Girija, C. R., Begum, N. S., Syed, A. A. & Thiruvenkatam, V. (2004). *Acta Cryst.* C**60**, o611–o613.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, Macromolecular Crystallography, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Reksohadiprodjo, M. S., Timmerman, H., Sardjiman, M. S. A., Martono, S., Sugiyanto, H. L. R., Nurlaila, H. A. R., Puspitasari, I., Nurrochmad, A., Purwantiningsih, O. & Yuwono, T. (2004). US Patent 6 777 447 B2.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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supplementary materials

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1,5-Bis(3,4-dimethoxyphenyl)penta-1,4-dien-3-one

R. J. Butcher, J. P. Jasinski, H. S. Yathirajan, S. Bindya, B. Narayana and B. K. Sarojini

Comment

The title compound, $C_{21}H_{22}O_5$, 1,5-bis(3,4-dimethoxyphenyl) penta-1,4-dien-3-one, (I), is a biologically active compound. Derivatives of dibenzylidene acetone, cyclopentanone and cyclohexanone exhibit potent anti-inflammatory, antibacterial and antioxidation activity. Closely related to the title compound is Curcumin, (1,7-bis(4-hydroxy-3-methoxy-phenyl)hepta-1,6-diene-3,5-dione), which is the main constituent of turmeric, the commonly used spice. It has been widely used as an anti-inflammatory, antibacterial, antioxidant, antihepatotoxic, hypocholesterolanemia, anti-cyclooxygenase, anti-cancer and radical scavenger agent. It is reported that curcumin is nontoxic at high doses and substitution on the aromatic rings with electron donating and withdrawing groups increases anti-inflammatory activity (Reksohadiprodjo *et al.*, 2004).

Hydrogen-bonding interactions in 1,7-bis(4-hydroxy-3-methoxyphenyl)heptane-3,5-dione have been reported (Girija *et al.*, 2004) as well as related crystal and molecular structural studies of 1,5-bis(4-chlorophenyl)penta-1,4-dien-3-one (Butcher *et al.*, 2006). In view of the importance of these dibenzylidene derivatives, the present paper reports the crystal structure of the title compound $C_{21}H_{22}O_5$.

Experimental

The title compound was synthesized with a yield of 75% according to the method reported in Author? (1989). The compound was purified by recrystallization from ethanol. The crystal was grown in acetone:toluene [50:50] solvent by the slow evaporation technique (m.p.:368–373 K). Analysis for $C_{21}H_{22}O_5$: Found (Calculated): C: 71.20 (71.17%); H: 6.18 (6.22%).

Refinement

The H atoms were included in calculated positions (C—H 0.95–0.98 Å) and refined in riding model approximation, with $U_{iso}(H) = 1.2–1.5U_{eq}(C)$.

Figures

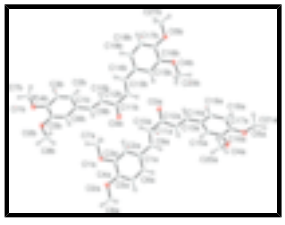

Fig. 1. Molecular structure of (I), showing atomic labeling and 50% probability displacement ellipsoids.

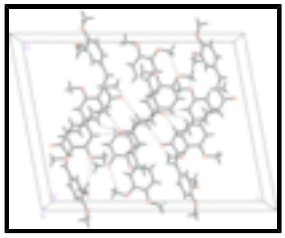

Fig. 2. Packing diagram of (I), viewed down the *b* axis. Dashed lines indicate C—H···O hydrogen bonds.

1,5-Bis(3,4-dimethoxyphenyl)penta-1,4-dien-3-one

Crystal data

$C_{21}H_{22}O_5$	F_{000} = 1504
M_r = 354.39	D_x = 1.297 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 19.1646 (13) Å	Cell parameters from 7695 reflections
b = 7.7488 (5) Å	θ = 2.8–29.6º
c = 24.8495 (17) Å	μ = 0.09 mm^{-1}
β = 100.4140 (10)º	T = 103 K
V = 3629.4 (4) Å^3	Prism, colourless
Z = 8	0.56 × 0.35 × 0.24 mm

Data collection

CCD area-detector diffractometer	10146 independent reflections
Radiation source: fine-focus sealed tube	8569 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.027
T = 103 K	θ_{max} = 29.7º
φ and ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan DENZO/SCALEPACK (Otwinowski & Minor, 1997)	h = −26→26
T_{min} = 0.950, T_{max} = 0.988	k = −10→10
40342 measured reflections	l = −33→34

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.041	H-atom parameters constrained
$wR(F^2)$ = 0.114	$w = 1/[\sigma^2(F_o^2) + (0.0612P)^2 + 0.9436P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max}$ = 0.002
10146 reflections	$\Delta\rho_{max}$ = 0.34 e Å^{-3}
477 parameters	$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Primary atom site location: structure-invariant direct methods

Extinction correction: none

Special details

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 $F^2 > 2\text{sigma}(F^2)$ 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}
O1A	0.70296 (4)	0.41530 (11)	0.25313 (3)	0.02419 (17)
O2A	0.70709 (4)	0.51375 (11)	0.35211 (3)	0.02445 (17)
O3A	0.35878 (4)	0.45882 (11)	0.07001 (3)	0.02442 (17)
O4A	0.07684 (4)	0.86400 (10)	0.20454 (3)	0.02421 (17)
O5A	−0.02272 (4)	0.63640 (10)	0.18184 (3)	0.02352 (16)
O1B	0.96885 (4)	0.17449 (11)	0.03878 (3)	0.02650 (17)
O2B	0.93567 (4)	0.07348 (11)	0.13096 (3)	0.02318 (16)
O3B	0.55536 (4)	0.26262 (11)	0.10438 (3)	0.02574 (17)
O4B	0.22391 (4)	0.28208 (11)	−0.09563 (3)	0.02277 (16)
O5B	0.23280 (4)	0.19771 (11)	−0.19409 (3)	0.02292 (16)
C1A	0.51544 (5)	0.55102 (12)	0.24296 (4)	0.01736 (18)
C2A	0.57740 (5)	0.48719 (13)	0.22675 (4)	0.01750 (18)
H2AA	0.5761	0.4518	0.1900	0.021*
C3A	0.63995 (5)	0.47606 (13)	0.26423 (4)	0.01795 (19)
C4A	0.64228 (5)	0.52836 (13)	0.31906 (4)	0.01887 (19)
C5A	0.58134 (6)	0.58896 (13)	0.33525 (4)	0.0203 (2)
H5AA	0.5824	0.6228	0.3721	0.024*
C6A	0.51839 (5)	0.60011 (13)	0.29714 (4)	0.01938 (19)
H6AA	0.4767	0.6420	0.3084	0.023*
C7A	0.70226 (6)	0.35031 (15)	0.19927 (4)	0.0241 (2)
H7AA	0.7499	0.3101	0.1962	0.036*
H7AB	0.6877	0.4422	0.1725	0.036*
H7AC	0.6687	0.2540	0.1921	0.036*
C8A	0.70952 (7)	0.54299 (16)	0.40909 (5)	0.0300 (3)
H8AA	0.7580	0.5249	0.4289	0.045*
H8AB	0.6774	0.4624	0.4228	0.045*
H8AC	0.6948	0.6618	0.4148	0.045*
C9A	0.44789 (5)	0.56426 (13)	0.20514 (4)	0.01823 (19)
H9AA	0.4086	0.6059	0.2198	0.022*
C10A	0.43560 (5)	0.52354 (13)	0.15173 (4)	0.01880 (19)
H10A	0.4753	0.4958	0.1354	0.023*

C11A	0.36532 (5)	0.51882 (13)	0.11681 (4)	0.01848 (19)
C12A	0.30154 (5)	0.57637 (13)	0.13791 (4)	0.01858 (19)
H12A	0.3056	0.6593	0.1665	0.022*
C13A	0.23810 (5)	0.50987 (13)	0.11618 (4)	0.01900 (19)
H13A	0.2375	0.4299	0.0871	0.023*
C14A	0.16988 (5)	0.54584 (13)	0.13193 (4)	0.01782 (19)
C15A	0.15812 (5)	0.69605 (13)	0.16107 (4)	0.01781 (19)
H15A	0.1948	0.7794	0.1698	0.021*
C16A	0.09345 (5)	0.72227 (13)	0.17689 (4)	0.01791 (19)
C17A	0.03876 (5)	0.59741 (13)	0.16468 (4)	0.01875 (19)
C18A	0.05038 (5)	0.44979 (14)	0.13599 (4)	0.0204 (2)
H18A	0.0143	0.3649	0.1279	0.024*
C19A	0.11532 (5)	0.42641 (14)	0.11907 (4)	0.0204 (2)
H19A	0.1224	0.3269	0.0984	0.025*
C20A	0.11770 (6)	1.01596 (15)	0.20083 (6)	0.0313 (3)
H20A	0.0951	1.1144	0.2156	0.047*
H20B	0.1204	1.0380	0.1624	0.047*
H20C	0.1656	0.9999	0.2219	0.047*
C21A	−0.08162 (6)	0.52357 (15)	0.16435 (5)	0.0269 (2)
H21A	−0.1246	0.5741	0.1739	0.040*
H21B	−0.0723	0.4115	0.1825	0.040*
H21C	−0.0882	0.5079	0.1246	0.040*
C1B	0.75664 (5)	0.19846 (13)	0.05722 (4)	0.01803 (19)
C2B	0.77562 (6)	0.25613 (14)	0.00882 (4)	0.0209 (2)
H2BA	0.7403	0.3014	−0.0194	0.025*
C3B	0.84584 (6)	0.24834 (14)	0.00126 (4)	0.0225 (2)
H3BA	0.8577	0.2864	−0.0323	0.027*
C4B	0.89870 (5)	0.18555 (14)	0.04228 (4)	0.0205 (2)
C5B	0.88064 (5)	0.12898 (13)	0.09200 (4)	0.01874 (19)
C6B	0.81052 (5)	0.13416 (13)	0.09856 (4)	0.01809 (19)
H6BA	0.7984	0.0934	0.1317	0.022*
C7B	0.98831 (7)	0.22779 (19)	−0.01151 (5)	0.0341 (3)
H7BA	1.0395	0.2129	−0.0093	0.051*
H7BB	0.9759	0.3496	−0.0182	0.051*
H7BC	0.9629	0.1575	−0.0416	0.051*
C8B	0.91785 (6)	0.02067 (15)	0.18211 (4)	0.0234 (2)
H8BA	0.9608	−0.0180	0.2068	0.035*
H8BB	0.8836	−0.0743	0.1758	0.035*
H8BC	0.8970	0.1182	0.1987	0.035*
C9B	0.68407 (5)	0.20273 (13)	0.06781 (4)	0.01842 (19)
H9BA	0.6772	0.1543	0.1016	0.022*
C10B	0.62599 (5)	0.26665 (14)	0.03565 (4)	0.01972 (19)
H10B	0.6289	0.3116	0.0006	0.024*
C11B	0.55731 (5)	0.26688 (13)	0.05515 (4)	0.01883 (19)
C12B	0.49040 (5)	0.27168 (14)	0.01548 (4)	0.0203 (2)
H12B	0.4485	0.3035	0.0285	0.024*
C13B	0.48442 (5)	0.23422 (13)	−0.03785 (4)	0.0198 (2)
H13B	0.5272	0.2112	−0.0507	0.024*
C14B	0.41874 (5)	0.22514 (13)	−0.07822 (4)	0.01829 (19)

C15B	0.35178 (5)	0.26085 (13)	−0.06460 (4)	0.01829 (19)
H15B	0.3484	0.2924	−0.0282	0.022*
C16B	0.29111 (5)	0.24990 (13)	−0.10418 (4)	0.01745 (19)
C17B	0.29605 (5)	0.20299 (13)	−0.15844 (4)	0.01816 (19)
C18B	0.36156 (6)	0.16712 (13)	−0.17167 (4)	0.0200 (2)
H18B	0.3650	0.1348	−0.2080	0.024*
C19B	0.42248 (5)	0.17860 (14)	−0.13161 (4)	0.0205 (2)
H19B	0.4674	0.1542	−0.1410	0.025*
C20B	0.21431 (6)	0.28988 (17)	−0.04009 (5)	0.0278 (2)
H20D	0.1639	0.3061	−0.0390	0.042*
H20E	0.2416	0.3868	−0.0217	0.042*
H20F	0.2309	0.1820	−0.0215	0.042*
C21B	0.23441 (6)	0.13995 (15)	−0.24840 (4)	0.0253 (2)
H21D	0.1859	0.1326	−0.2691	0.038*
H21E	0.2567	0.0259	−0.2470	0.038*
H21F	0.2617	0.2217	−0.2664	0.038*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.0163 (3)	0.0342 (4)	0.0214 (4)	0.0047 (3)	0.0016 (3)	−0.0053 (3)
O2A	0.0222 (4)	0.0294 (4)	0.0191 (4)	0.0030 (3)	−0.0033 (3)	−0.0023 (3)
O3A	0.0203 (4)	0.0366 (4)	0.0169 (3)	0.0041 (3)	0.0046 (3)	−0.0010 (3)
O4A	0.0183 (3)	0.0207 (4)	0.0351 (4)	−0.0012 (3)	0.0089 (3)	−0.0060 (3)
O5A	0.0137 (3)	0.0242 (4)	0.0338 (4)	−0.0017 (3)	0.0073 (3)	−0.0027 (3)
O1B	0.0181 (4)	0.0362 (4)	0.0271 (4)	−0.0012 (3)	0.0092 (3)	−0.0004 (3)
O2B	0.0148 (3)	0.0317 (4)	0.0227 (4)	0.0029 (3)	0.0026 (3)	0.0037 (3)
O3B	0.0219 (4)	0.0383 (4)	0.0172 (4)	0.0051 (3)	0.0040 (3)	0.0003 (3)
O4B	0.0158 (3)	0.0340 (4)	0.0187 (3)	0.0047 (3)	0.0037 (3)	−0.0016 (3)
O5B	0.0203 (4)	0.0321 (4)	0.0153 (3)	−0.0008 (3)	0.0004 (3)	−0.0028 (3)
C1A	0.0166 (4)	0.0167 (4)	0.0188 (4)	0.0001 (3)	0.0034 (3)	0.0011 (3)
C2A	0.0176 (4)	0.0192 (4)	0.0158 (4)	0.0001 (3)	0.0033 (3)	0.0003 (3)
C3A	0.0162 (4)	0.0181 (4)	0.0197 (5)	0.0012 (3)	0.0037 (4)	0.0006 (4)
C4A	0.0201 (5)	0.0173 (4)	0.0178 (4)	−0.0005 (4)	0.0000 (4)	0.0013 (3)
C5A	0.0247 (5)	0.0192 (4)	0.0170 (4)	−0.0006 (4)	0.0041 (4)	−0.0011 (4)
C6A	0.0203 (5)	0.0191 (4)	0.0199 (5)	0.0007 (4)	0.0067 (4)	0.0000 (4)
C7A	0.0212 (5)	0.0289 (5)	0.0229 (5)	0.0041 (4)	0.0054 (4)	−0.0039 (4)
C8A	0.0350 (6)	0.0329 (6)	0.0187 (5)	0.0019 (5)	−0.0041 (4)	−0.0009 (4)
C9A	0.0158 (4)	0.0179 (4)	0.0215 (5)	0.0017 (3)	0.0049 (4)	0.0013 (4)
C10A	0.0145 (4)	0.0220 (5)	0.0204 (5)	0.0029 (4)	0.0046 (4)	0.0011 (4)
C11A	0.0170 (4)	0.0207 (4)	0.0184 (4)	0.0022 (4)	0.0050 (4)	0.0032 (4)
C12A	0.0172 (4)	0.0218 (5)	0.0173 (4)	0.0036 (4)	0.0045 (4)	0.0013 (4)
C13A	0.0181 (4)	0.0230 (5)	0.0164 (4)	0.0027 (4)	0.0044 (3)	0.0009 (4)
C14A	0.0152 (4)	0.0227 (5)	0.0156 (4)	0.0019 (4)	0.0027 (3)	0.0027 (4)
C15A	0.0142 (4)	0.0202 (4)	0.0187 (4)	−0.0003 (3)	0.0021 (3)	0.0016 (4)
C16A	0.0158 (4)	0.0189 (4)	0.0188 (4)	0.0010 (3)	0.0027 (3)	0.0005 (3)
C17A	0.0134 (4)	0.0223 (5)	0.0209 (5)	0.0006 (3)	0.0038 (3)	0.0030 (4)
C18A	0.0167 (4)	0.0216 (5)	0.0222 (5)	−0.0025 (4)	0.0018 (4)	−0.0001 (4)

C19A	0.0190 (5)	0.0223 (5)	0.0195 (5)	0.0005 (4)	0.0024 (4)	−0.0021 (4)
C20A	0.0262 (5)	0.0203 (5)	0.0491 (7)	−0.0033 (4)	0.0111 (5)	−0.0044 (5)
C21A	0.0152 (4)	0.0283 (5)	0.0373 (6)	−0.0037 (4)	0.0050 (4)	−0.0003 (5)
C1B	0.0166 (4)	0.0194 (4)	0.0179 (4)	0.0002 (3)	0.0026 (3)	−0.0019 (3)
C2B	0.0197 (5)	0.0249 (5)	0.0176 (5)	−0.0002 (4)	0.0018 (4)	0.0001 (4)
C3B	0.0234 (5)	0.0272 (5)	0.0181 (5)	−0.0021 (4)	0.0065 (4)	−0.0006 (4)
C4B	0.0170 (4)	0.0227 (5)	0.0225 (5)	−0.0022 (4)	0.0058 (4)	−0.0042 (4)
C5B	0.0167 (4)	0.0192 (4)	0.0197 (5)	0.0008 (3)	0.0018 (4)	−0.0015 (4)
C6B	0.0175 (4)	0.0197 (4)	0.0171 (4)	0.0009 (3)	0.0032 (3)	−0.0006 (3)
C7B	0.0270 (6)	0.0455 (7)	0.0339 (7)	−0.0015 (5)	0.0167 (5)	0.0033 (5)
C8B	0.0185 (5)	0.0284 (5)	0.0230 (5)	0.0034 (4)	0.0031 (4)	0.0049 (4)
C9B	0.0182 (4)	0.0204 (4)	0.0169 (4)	0.0007 (4)	0.0039 (4)	−0.0006 (4)
C10B	0.0179 (4)	0.0239 (5)	0.0174 (4)	0.0002 (4)	0.0033 (4)	0.0013 (4)
C11B	0.0174 (4)	0.0207 (4)	0.0183 (5)	0.0025 (4)	0.0029 (4)	0.0002 (4)
C12B	0.0157 (4)	0.0249 (5)	0.0206 (5)	0.0030 (4)	0.0035 (4)	0.0003 (4)
C13B	0.0156 (4)	0.0229 (5)	0.0214 (5)	0.0016 (4)	0.0048 (4)	−0.0006 (4)
C14B	0.0170 (4)	0.0194 (4)	0.0186 (5)	0.0004 (3)	0.0034 (4)	−0.0007 (4)
C15B	0.0180 (4)	0.0214 (5)	0.0155 (4)	0.0018 (4)	0.0031 (4)	−0.0009 (3)
C16B	0.0166 (4)	0.0186 (4)	0.0175 (4)	0.0018 (3)	0.0043 (4)	0.0002 (3)
C17B	0.0191 (4)	0.0186 (4)	0.0161 (4)	−0.0013 (4)	0.0015 (4)	0.0005 (3)
C18B	0.0233 (5)	0.0213 (5)	0.0164 (4)	−0.0014 (4)	0.0059 (4)	−0.0017 (4)
C19B	0.0181 (4)	0.0239 (5)	0.0207 (5)	−0.0003 (4)	0.0067 (4)	−0.0021 (4)
C20B	0.0225 (5)	0.0411 (6)	0.0212 (5)	0.0049 (5)	0.0075 (4)	−0.0036 (5)
C21B	0.0302 (6)	0.0287 (5)	0.0156 (5)	−0.0008 (4)	0.0005 (4)	−0.0035 (4)

Geometric parameters (Å, °)

O1A—C3A	1.3698 (12)	C18A—C19A	1.3956 (15)
O1A—C7A	1.4277 (13)	C18A—H18A	0.9500
O2A—C4A	1.3642 (12)	C19A—H19A	0.9500
O2A—C8A	1.4265 (14)	C20A—H20A	0.9800
O3A—C11A	1.2375 (13)	C20A—H20B	0.9800
O4A—C16A	1.3633 (12)	C20A—H20C	0.9800
O4A—C20A	1.4262 (13)	C21A—H21A	0.9800
O5A—C17A	1.3574 (12)	C21A—H21B	0.9800
O5A—C21A	1.4319 (13)	C21A—H21C	0.9800
O1B—C4B	1.3654 (12)	C1B—C2B	1.3917 (15)
O1B—C7B	1.4280 (14)	C1B—C6B	1.4092 (13)
O2B—C5B	1.3657 (12)	C1B—C9B	1.4619 (14)
O2B—C8B	1.4342 (13)	C2B—C3B	1.3933 (15)
O3B—C11B	1.2310 (13)	C2B—H2BA	0.9500
O4B—C16B	1.3655 (12)	C3B—C4B	1.3890 (15)
O4B—C20B	1.4261 (13)	C3B—H3BA	0.9500
O5B—C17B	1.3667 (12)	C4B—C5B	1.4117 (15)
O5B—C21B	1.4273 (13)	C5B—C6B	1.3833 (14)
C1A—C6A	1.3903 (14)	C6B—H6BA	0.9500
C1A—C2A	1.4105 (14)	C7B—H7BA	0.9800
C1A—C9A	1.4594 (13)	C7B—H7BB	0.9800
C2A—C3A	1.3815 (13)	C7B—H7BC	0.9800

C2A—H2AA	0.9500	C8B—H8BA	0.9800
C3A—C4A	1.4142 (14)	C8B—H8BB	0.9800
C4A—C5A	1.3843 (15)	C8B—H8BC	0.9800
C5A—C6A	1.3952 (14)	C9B—C10B	1.3420 (14)
C5A—H5AA	0.9500	C9B—H9BA	0.9500
C6A—H6AA	0.9500	C10B—C11B	1.4824 (14)
C7A—H7AA	0.9800	C10B—H10B	0.9500
C7A—H7AB	0.9800	C11B—C12B	1.4699 (14)
C7A—H7AC	0.9800	C12B—C13B	1.3411 (15)
C8A—H8AA	0.9800	C12B—H12B	0.9500
C8A—H8AB	0.9800	C13B—C14B	1.4626 (14)
C8A—H8AC	0.9800	C13B—H13B	0.9500
C9A—C10A	1.3429 (14)	C14B—C19B	1.3893 (14)
C9A—H9AA	0.9500	C14B—C15B	1.4123 (14)
C10A—C11A	1.4649 (13)	C15B—C16B	1.3833 (13)
C10A—H10A	0.9500	C15B—H15B	0.9500
C11A—C12A	1.4832 (14)	C16B—C17B	1.4159 (14)
C12A—C13A	1.3409 (14)	C17B—C18B	1.3824 (14)
C12A—H12A	0.9500	C18B—C19B	1.3935 (14)
C13A—C14A	1.4578 (14)	C18B—H18B	0.9500
C13A—H13A	0.9500	C19B—H19B	0.9500
C14A—C19A	1.3897 (14)	C20B—H20D	0.9800
C14A—C15A	1.4102 (14)	C20B—H20E	0.9800
C15A—C16A	1.3813 (14)	C20B—H20F	0.9800
C15A—H15A	0.9500	C21B—H21D	0.9800
C16A—C17A	1.4184 (14)	C21B—H21E	0.9800
C17A—C18A	1.3870 (15)	C21B—H21F	0.9800
C3A—O1A—C7A	116.80 (8)	H21A—C21A—H21B	109.5
C4A—O2A—C8A	116.57 (9)	O5A—C21A—H21C	109.5
C16A—O4A—C20A	116.84 (9)	H21A—C21A—H21C	109.5
C17A—O5A—C21A	116.75 (9)	H21B—C21A—H21C	109.5
C4B—O1B—C7B	116.98 (9)	C2B—C1B—C6B	118.16 (9)
C5B—O2B—C8B	116.16 (8)	C2B—C1B—C9B	123.64 (9)
C16B—O4B—C20B	116.66 (8)	C6B—C1B—C9B	118.19 (9)
C17B—O5B—C21B	117.00 (8)	C1B—C2B—C3B	120.82 (10)
C6A—C1A—C2A	118.92 (9)	C1B—C2B—H2BA	119.6
C6A—C1A—C9A	118.71 (9)	C3B—C2B—H2BA	119.6
C2A—C1A—C9A	122.37 (9)	C4B—C3B—C2B	120.69 (10)
C3A—C2A—C1A	120.14 (9)	C4B—C3B—H3BA	119.7
C3A—C2A—H2AA	119.9	C2B—C3B—H3BA	119.7
C1A—C2A—H2AA	119.9	O1B—C4B—C3B	124.82 (10)
O1A—C3A—C2A	125.08 (9)	O1B—C4B—C5B	115.87 (9)
O1A—C3A—C4A	114.65 (8)	C3B—C4B—C5B	119.30 (9)
C2A—C3A—C4A	120.27 (9)	O2B—C5B—C6B	124.61 (9)
O2A—C4A—C5A	125.26 (9)	O2B—C5B—C4B	115.98 (9)
O2A—C4A—C3A	115.02 (9)	C6B—C5B—C4B	119.42 (9)
C5A—C4A—C3A	119.71 (9)	C5B—C6B—C1B	121.59 (10)
C4A—C5A—C6A	119.69 (9)	C5B—C6B—H6BA	119.2
C4A—C5A—H5AA	120.2	C1B—C6B—H6BA	119.2

C6A—C5A—H5AA	120.2	O1B—C7B—H7BA	109.5
C1A—C6A—C5A	121.27 (9)	O1B—C7B—H7BB	109.5
C1A—C6A—H6AA	119.4	H7BA—C7B—H7BB	109.5
C5A—C6A—H6AA	119.4	O1B—C7B—H7BC	109.5
O1A—C7A—H7AA	109.5	H7BA—C7B—H7BC	109.5
O1A—C7A—H7AB	109.5	H7BB—C7B—H7BC	109.5
H7AA—C7A—H7AB	109.5	O2B—C8B—H8BA	109.5
O1A—C7A—H7AC	109.5	O2B—C8B—H8BB	109.5
H7AA—C7A—H7AC	109.5	H8BA—C8B—H8BB	109.5
H7AB—C7A—H7AC	109.5	O2B—C8B—H8BC	109.5
O2A—C8A—H8AA	109.5	H8BA—C8B—H8BC	109.5
O2A—C8A—H8AB	109.5	H8BB—C8B—H8BC	109.5
H8AA—C8A—H8AB	109.5	C10B—C9B—C1B	127.94 (10)
O2A—C8A—H8AC	109.5	C10B—C9B—H9BA	116.0
H8AA—C8A—H8AC	109.5	C1B—C9B—H9BA	116.0
H8AB—C8A—H8AC	109.5	C9B—C10B—C11B	119.72 (9)
C10A—C9A—C1A	126.63 (9)	C9B—C10B—H10B	120.1
C10A—C9A—H9AA	116.7	C11B—C10B—H10B	120.1
C1A—C9A—H9AA	116.7	O3B—C11B—C12B	119.19 (9)
C9A—C10A—C11A	124.72 (9)	O3B—C11B—C10B	120.86 (9)
C9A—C10A—H10A	117.6	C12B—C11B—C10B	119.96 (9)
C11A—C10A—H10A	117.6	C13B—C12B—C11B	124.44 (9)
O3A—C11A—C10A	119.48 (9)	C13B—C12B—H12B	117.8
O3A—C11A—C12A	119.69 (9)	C11B—C12B—H12B	117.8
C10A—C11A—C12A	120.75 (9)	C12B—C13B—C14B	126.68 (10)
C13A—C12A—C11A	119.16 (9)	C12B—C13B—H13B	116.7
C13A—C12A—H12A	120.4	C14B—C13B—H13B	116.7
C11A—C12A—H12A	120.4	C19B—C14B—C15B	119.05 (9)
C12A—C13A—C14A	127.61 (10)	C19B—C14B—C13B	118.75 (9)
C12A—C13A—H13A	116.2	C15B—C14B—C13B	122.20 (9)
C14A—C13A—H13A	116.2	C16B—C15B—C14B	120.08 (9)
C19A—C14A—C15A	118.97 (9)	C16B—C15B—H15B	120.0
C19A—C14A—C13A	119.01 (9)	C14B—C15B—H15B	120.0
C15A—C14A—C13A	122.00 (9)	O4B—C16B—C15B	124.98 (9)
C16A—C15A—C14A	120.15 (9)	O4B—C16B—C17B	114.99 (8)
C16A—C15A—H15A	119.9	C15B—C16B—C17B	120.03 (9)
C14A—C15A—H15A	119.9	O5B—C17B—C18B	125.24 (9)
O4A—C16A—C15A	124.41 (9)	O5B—C17B—C16B	114.89 (9)
O4A—C16A—C17A	115.24 (9)	C18B—C17B—C16B	119.88 (9)
C15A—C16A—C17A	120.35 (9)	C17B—C18B—C19B	119.79 (9)
O5A—C17A—C18A	125.04 (9)	C17B—C18B—H18B	120.1
O5A—C17A—C16A	115.52 (9)	C19B—C18B—H18B	120.1
C18A—C17A—C16A	119.43 (9)	C14B—C19B—C18B	121.17 (9)
C17A—C18A—C19A	119.79 (9)	C14B—C19B—H19B	119.4
C17A—C18A—H18A	120.1	C18B—C19B—H19B	119.4
C19A—C18A—H18A	120.1	O4B—C20B—H20D	109.5
C14A—C19A—C18A	121.27 (10)	O4B—C20B—H20E	109.5
C14A—C19A—H19A	119.4	H20D—C20B—H20E	109.5
C18A—C19A—H19A	119.4	O4B—C20B—H20F	109.5

O4A—C20A—H20A	109.5	H20D—C20B—H20F	109.5
O4A—C20A—H20B	109.5	H20E—C20B—H20F	109.5
H20A—C20A—H20B	109.5	O5B—C21B—H21D	109.5
O4A—C20A—H20C	109.5	O5B—C21B—H21E	109.5
H20A—C20A—H20C	109.5	H21D—C21B—H21E	109.5
H20B—C20A—H20C	109.5	O5B—C21B—H21F	109.5
O5A—C21A—H21A	109.5	H21D—C21B—H21F	109.5
O5A—C21A—H21B	109.5	H21E—C21B—H21F	109.5
C6A—C1A—C2A—C3A	−0.78 (14)	C6B—C1B—C2B—C3B	0.80 (15)
C9A—C1A—C2A—C3A	−179.53 (9)	C9B—C1B—C2B—C3B	−179.93 (10)
C7A—O1A—C3A—C2A	−3.51 (15)	C1B—C2B—C3B—C4B	−1.13 (16)
C7A—O1A—C3A—C4A	176.01 (9)	C7B—O1B—C4B—C3B	−1.64 (16)
C1A—C2A—C3A—O1A	179.57 (9)	C7B—O1B—C4B—C5B	178.82 (10)
C1A—C2A—C3A—C4A	0.06 (15)	C2B—C3B—C4B—O1B	−179.40 (10)
C8A—O2A—C4A—C5A	8.28 (15)	C2B—C3B—C4B—C5B	0.12 (16)
C8A—O2A—C4A—C3A	−171.83 (9)	C8B—O2B—C5B—C6B	−1.15 (15)
O1A—C3A—C4A—O2A	1.32 (13)	C8B—O2B—C5B—C4B	178.23 (9)
C2A—C3A—C4A—O2A	−179.13 (9)	O1B—C4B—C5B—O2B	1.32 (14)
O1A—C3A—C4A—C5A	−178.78 (9)	C3B—C4B—C5B—O2B	−178.24 (9)
C2A—C3A—C4A—C5A	0.77 (15)	O1B—C4B—C5B—C6B	−179.26 (9)
O2A—C4A—C5A—C6A	179.01 (9)	C3B—C4B—C5B—C6B	1.18 (15)
C3A—C4A—C5A—C6A	−0.88 (15)	O2B—C5B—C6B—C1B	177.85 (9)
C2A—C1A—C6A—C5A	0.67 (15)	C4B—C5B—C6B—C1B	−1.51 (15)
C9A—C1A—C6A—C5A	179.47 (9)	C2B—C1B—C6B—C5B	0.52 (15)
C4A—C5A—C6A—C1A	0.16 (15)	C9B—C1B—C6B—C5B	−178.78 (9)
C6A—C1A—C9A—C10A	179.67 (10)	C2B—C1B—C9B—C10B	−3.14 (17)
C2A—C1A—C9A—C10A	−1.58 (16)	C6B—C1B—C9B—C10B	176.12 (10)
C1A—C9A—C10A—C11A	172.18 (9)	C1B—C9B—C10B—C11B	−176.83 (9)
C9A—C10A—C11A—O3A	−170.90 (10)	C9B—C10B—C11B—O3B	23.27 (15)
C9A—C10A—C11A—C12A	5.81 (16)	C9B—C10B—C11B—C12B	−156.53 (10)
O3A—C11A—C12A—C13A	23.71 (15)	O3B—C11B—C12B—C13B	−162.80 (11)
C10A—C11A—C12A—C13A	−153.00 (10)	C10B—C11B—C12B—C13B	17.01 (16)
C11A—C12A—C13A—C14A	178.36 (9)	C11B—C12B—C13B—C14B	175.38 (10)
C12A—C13A—C14A—C19A	−158.01 (11)	C12B—C13B—C14B—C19B	−177.88 (11)
C12A—C13A—C14A—C15A	20.32 (16)	C12B—C13B—C14B—C15B	1.64 (17)
C19A—C14A—C15A—C16A	0.62 (14)	C19B—C14B—C15B—C16B	−0.23 (15)
C13A—C14A—C15A—C16A	−177.70 (9)	C13B—C14B—C15B—C16B	−179.76 (9)
C20A—O4A—C16A—C15A	21.79 (15)	C20B—O4B—C16B—C15B	−14.48 (15)
C20A—O4A—C16A—C17A	−158.30 (10)	C20B—O4B—C16B—C17B	165.79 (9)
C14A—C15A—C16A—O4A	−179.19 (9)	C14B—C15B—C16B—O4B	−179.77 (9)
C14A—C15A—C16A—C17A	0.91 (15)	C14B—C15B—C16B—C17B	−0.05 (15)
C21A—O5A—C17A—C18A	−6.10 (15)	C21B—O5B—C17B—C18B	4.18 (15)
C21A—O5A—C17A—C16A	172.56 (9)	C21B—O5B—C17B—C16B	−175.94 (9)
O4A—C16A—C17A—O5A	0.40 (13)	O4B—C16B—C17B—O5B	0.25 (13)
C15A—C16A—C17A—O5A	−179.69 (9)	C15B—C16B—C17B—O5B	−179.49 (9)
O4A—C16A—C17A—C18A	179.14 (9)	O4B—C16B—C17B—C18B	−179.85 (9)
C15A—C16A—C17A—C18A	−0.94 (15)	C15B—C16B—C17B—C18B	0.41 (15)
O5A—C17A—C18A—C19A	178.06 (10)	O5B—C17B—C18B—C19B	179.41 (10)
C16A—C17A—C18A—C19A	−0.56 (15)	C16B—C17B—C18B—C19B	−0.48 (15)

C15A—C14A—C19A—C18A	−2.16 (15)	C15B—C14B—C19B—C18B	0.16 (16)
C13A—C14A—C19A—C18A	176.22 (9)	C13B—C14B—C19B—C18B	179.70 (9)
C17A—C18A—C19A—C14A	2.13 (16)	C17B—C18B—C19B—C14B	0.20 (16)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6A—H6AA···O3B^{i}	0.95	2.54	3.2898 (13)	137
C8A—H8AC···O3A^{i}	0.98	2.58	3.5513 (15)	173
C20A—H20C···O1A^{i}	0.98	2.57	3.5085 (15)	161
C8B—H8BA···O4A^{ii}	0.98	2.41	3.2334 (13)	141
C8B—H8BB···O5B^{iii}	0.98	2.54	3.4056 (14)	147
C10B—H10B···O3A^{iv}	0.95	2.54	3.4325 (13)	157
C12B—H12B···O3A	0.95	2.47	3.3968 (13)	164

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

Fig. 1

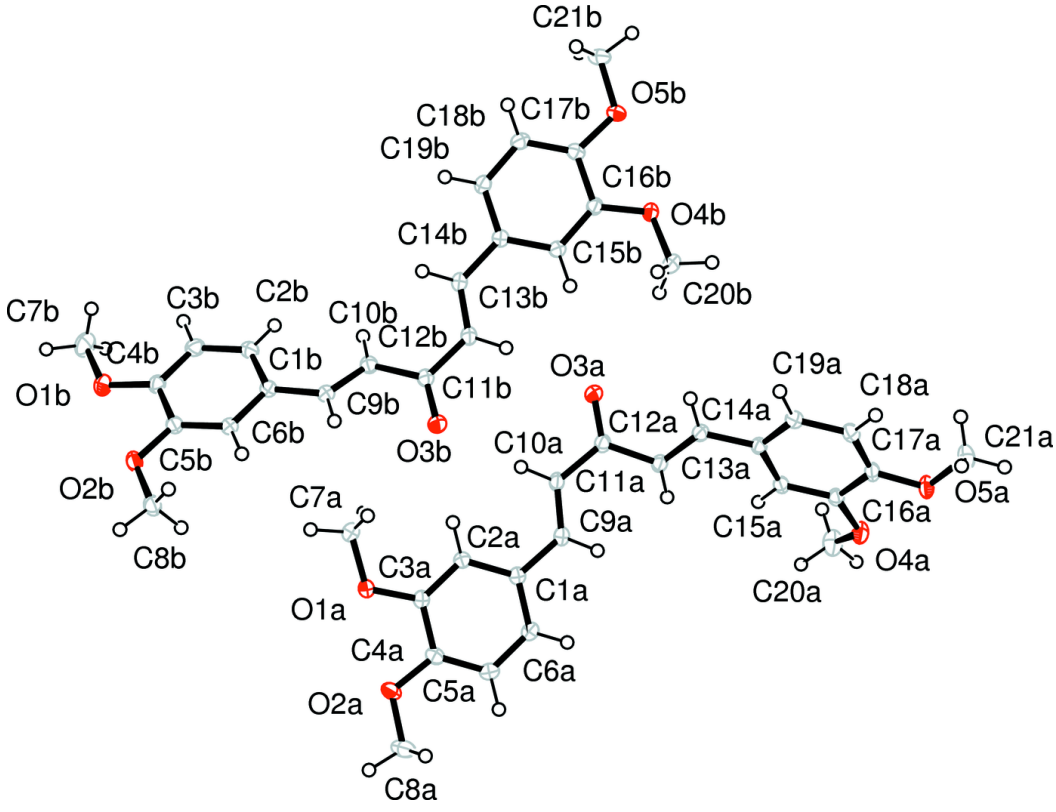
C21b
O5b
C18b
C17b
C19b
C16b
O4b
C14b
C15b
C20b
C13b
C7b
C3b
C2b
C10b
C12b
C4b
C1b
O1b
C9b
C11b
O3a
C19a
C18a
C12a
C14a
C6b
O3b
C10a
C17a
C21a
C5b
C11a
C13a
O2b
C7a
C9a
C16a
O5a
C15a
C2a
C8b
C3a
C1a
O4a
O1a
C20a
C4a
C6a
C5a
O2a
C8a

Fig. 2