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Crystal structures of 6-cyclopropyl-1,3-diphenylfulvene and 6-(2,3-dimethoxynaphthyl)-1,3-diphenylfulvene

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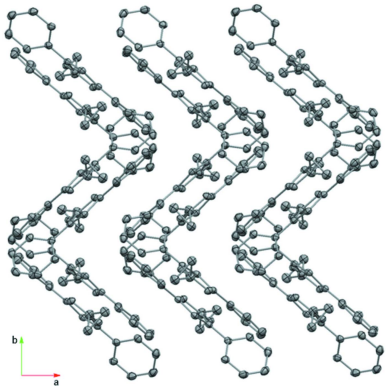
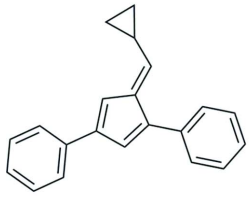
The title compounds, 6-cyclopropyl-1,3-diphenylfulvene, $C_{21}H_{18}$, [systematic name: 5-(cyclopropylmethylidene)-1,3-diphenylcyclopenta-1,3-diene], **1**, and 6-(2,3-dimethoxynaphthyl)-1,3-diphenylfulvene, $C_{30}H_{24}O_2$, {systematic name: 5-[(3,4-dimethoxynaphthalen-2-yl)methylidene]-1,3-diphenylcyclopenta-1,3-diene}, **2**, were prepared from 1,3-diphenylcyclopentadiene, pyrrolidine, and the corresponding aldehydes in an ethanolic solution. Each structure crystallizes with one molecule per asymmetric unit and exhibits the alternating short and long bond lengths typical of fulvenes. A network of C—H···C ring interactions as well as C—H···O interactions is observed, resulting in the compact packing found in each structure.

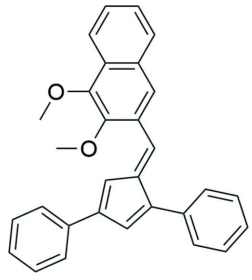
1. Chemical context

Pentafulvenes are a unique class of cross-conjugated organic molecules commonly synthesized using aldehyde and cyclopentadiene starting materials under a variety of conditions (Thiele, 1900; Stone & Little, 1984; Sieverding *et al.*, 2019). Substituted and highly colored pentafulvenes are of particular interest because of their unique optical and thermal properties and for their potential use in electronic applications (Peloquin *et al.*, 2012; Godman *et al.*, 2016; Shurdha *et al.*, 2014). In synthetic organometallic chemistry, the fulvene unit is known to coordinate to metals, forming organometallic complexes of varying hapticity (Peloquin *et al.*, 2018; Ma *et al.*, 2011, 2012; Beckhaus, 2018). More recently, 1,3,6-trisubstituted fulvenes have been used as starting materials in the synthesis of bridged cyclopentadiene ligands and *ansa-Ln* complexes (Adas & Balaich, 2018). As a continuation of our work in this area, we report herein the crystal structures of 6-cyclopropyl-1,3-diphenylfulvene, **1**, and 6-(2,3-dimethoxynaphthyl)-1,3-diphenylfulvene, **2**.





1



2

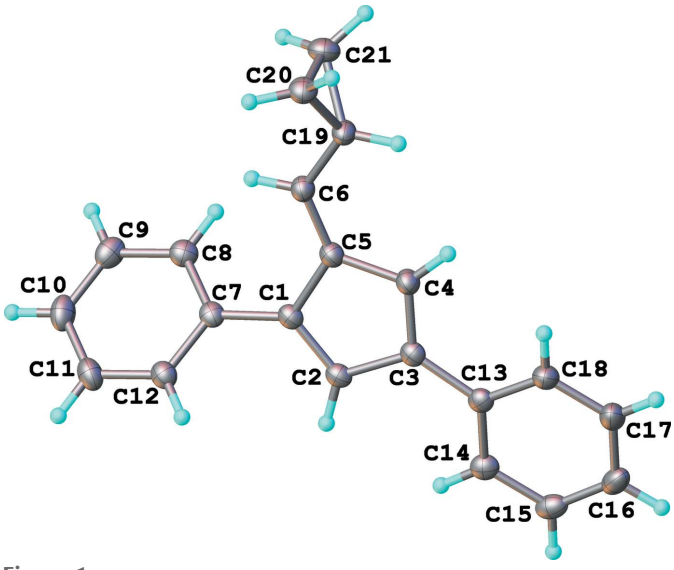


Figure 1
The molecular structure of **1**. Displacement ellipsoids are shown at the 50% probability level.

2. Structural commentary

Compounds **1** (Fig. 1) and **2** (Fig. 2) crystallize in the orthorhombic space groups *Pbca* and $P2_12_12_1$, respectively. Both fulvenes crystallize with one molecule per asymmetric unit (Z' = 1), exhibit the expected alternating short–long bond lengths within the fulvene core and display very similar bond lengths and angles (Table 1). Similar tilt angles of the phenyl substituents from the plane of the fulvene ring are also observed for **1** [1-Ph, 44.88 (4)°; 3-Ph 13.34 (4)°] and **2** [1-Ph, 30.82 (7)°; 3-Ph 17.19 (7)°]. Surprisingly, the rotation of the 6-substituent from the cyclopentadienyl core is greater for fulvene **1** [87.20 (6)°], than for the larger 2,3-dimethoxynaphthalene substituent in fulvene **2**, [55.63 (5)°].

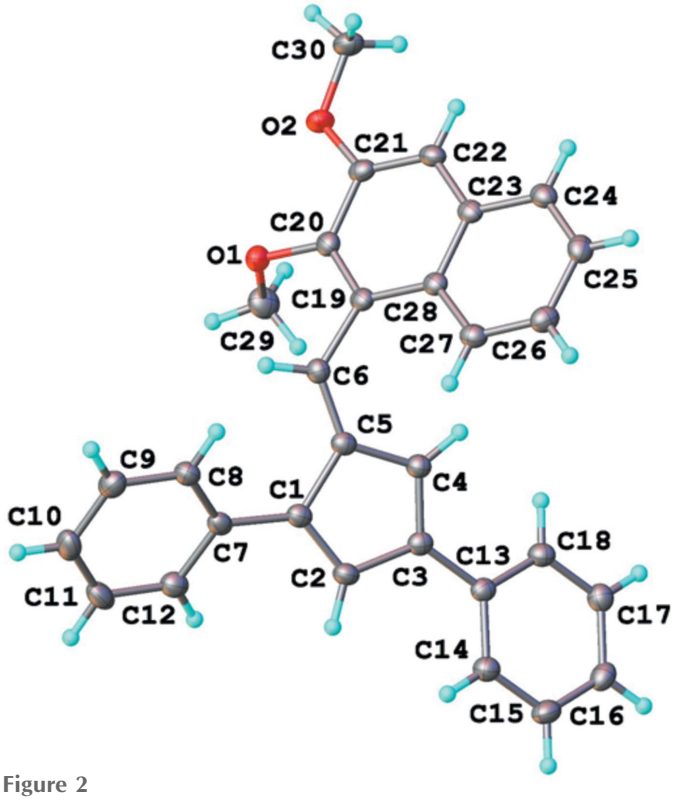


Figure 2
The molecular structure of **2**. Displacement ellipsoids are shown at the 50% probability level.

Table 1
Selected bond distances and angles (Å) for fulvenes **1** and **2**.

	1	**2**
C1—C2	1.3577 (13)	1.357 (2)
C1—C5	1.4774 (13)	1.489 (2)
C2—C3	1.4699 (13)	1.475 (2)
C3—C4	1.3621 (13)	1.354 (2)
C4—C5	1.4538 (13)	1.455 (2)
C5—C6	1.3520 (13)	1.351 (2)
C1—C7	1.4721 (13)	1.475 (2)
C3—13	1.4704 (13)	1.469 (2)
C6—C19	1.4570 (13)	1.476 (2)
C2—C1—C5	107.36 (8)	106.64 (15)
C1—C2—C3	109.71 (8)	110.03 (15)
C2—C3—C4	107.70 (8)	107.90 (15)
C3—C4—C5	109.12 (8)	109.07 (16)
C4—C5—C1	106.08 (8)	106.34 (14)
C4—C5—C6	126.91 (9)	126.27 (16)
C1—C5—C6	126.72 (9)	127.15 (16)

3. Supramolecular features

Fulvene **1** packs side by side along the *a*-axis direction with molecules oriented in such a way that the 6-cyclopropyl groups are sandwiched between the 1-Ph and 3-Ph rings of adjacent fulvene molecules. The closest contacts caused by this stacking sequence in the *a*-axis direction are between the 1-Ph ring atom H9 and the exocyclic C6 atom of an adjacent fulvene (C—H···C = 2.90 Å). Other C—H···C contacts (C12···H19 = 2.83, C2···H10 = 2.85, C14···H20*B* = 2.85, C11···H19 = 2.86 Å) lead to the formation of a network that results in sets of zigzag chains running perpendicular to the *a*-axis direction and that extend in the direction parallel to the *bc* plane (Fig. 3).

Fulvene **2** packs so that the 1-Ph groups are oriented towards the space between the 2,3-dimethoxynaphthyl groups and the 3-Ph rings of adjacent fulvene molecules along the *b*-axis direction. A view down the *a* axis (Fig. 4) reveals layers of interlaced 2,3-dimethoxynaphthyl groups (H, head) oriented H–H and separated from layers of interlaced 1,3-diphenylfulvene groups (T, tail) oriented T–T, with the layers running perpendicular to the *c*-axis direction and producing a layer sequence of H–H–T–T along the *c*-axis direction. In the H–H layers, short intermolecular contacts of the C—H···O type (O1···H22 = 2.50 and O2···H24 = 2.53 Å; Table 2) occur between adjacent 2,3-dimethoxynaphthyl groups running along the *b*-axis direction (Fig. 4). The methoxy groups apparently prevent the naphthyl rings from forming any π–π stacking interactions, with the angle between the mean planes of the 2,3-dimethoxynaphthyl groups oriented at 124.37 (5)° at least partially enforced by the C—H···O interactions.

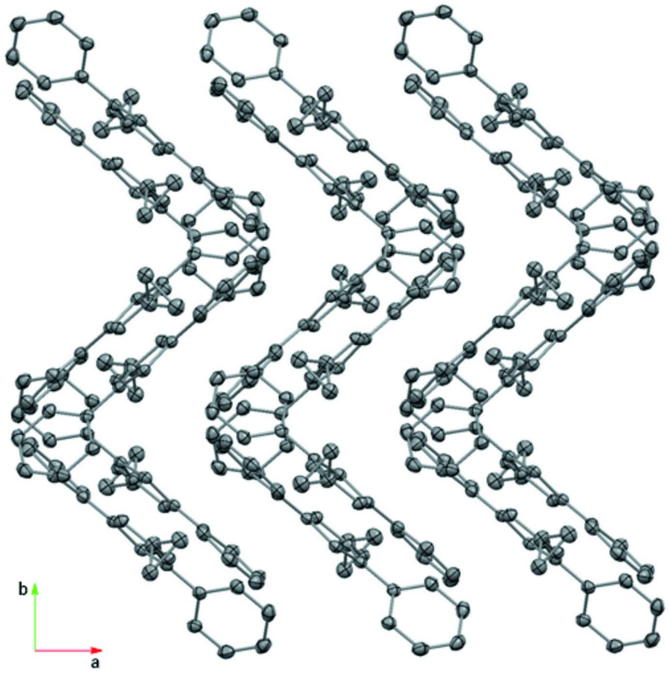


Figure 3
The packing of 6-cyclopropyl-1,3-diphenylfulvene, **1**, viewed along the *c*-axis direction. Hydrogen atoms are omitted for clarity.

4. Database survey

A survey of the December 2019 release of the Cambridge Structural Database, with updates through November 2019, was made using the program *Conquest* (Groom *et al.*, 2016). A search for 1,3-diphenyl-6-substituted fulvenes yielded 88 results. The bond lengths and angles in **1** and **2** are consistent with those in the previously reported literature.

Table 2
Hydrogen-bond geometry (Å, °) for **2**.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C22—H22···O1^{i}	0.95	2.50	3.429 (2)	164
C24—H24···O2^{i}	0.95	2.53	3.241 (2)	131

Symmetry code: (i) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$.

5. Synthesis and crystallization

Each compound was prepared according to the established literature procedure (Peloquin *et al.*, 2012; Godman *et al.*, 2016).

6-(Cyclopropyl)-1,3-diphenylfulvene, 1. Orange crystals suitable for single crystal X-ray diffraction were obtained from petroleum ether by slow evaporation.

6-(2,3-Dimethoxynapthyl)-1,3-diphenylfulvene, **2**. Red crystals suitable for single crystal X-ray diffraction were obtained from slow diffusion of petroleum ether into a DCM solution.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. H atoms were placed in calculated positions (0.95–1.00 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C-methyl).

Funding information

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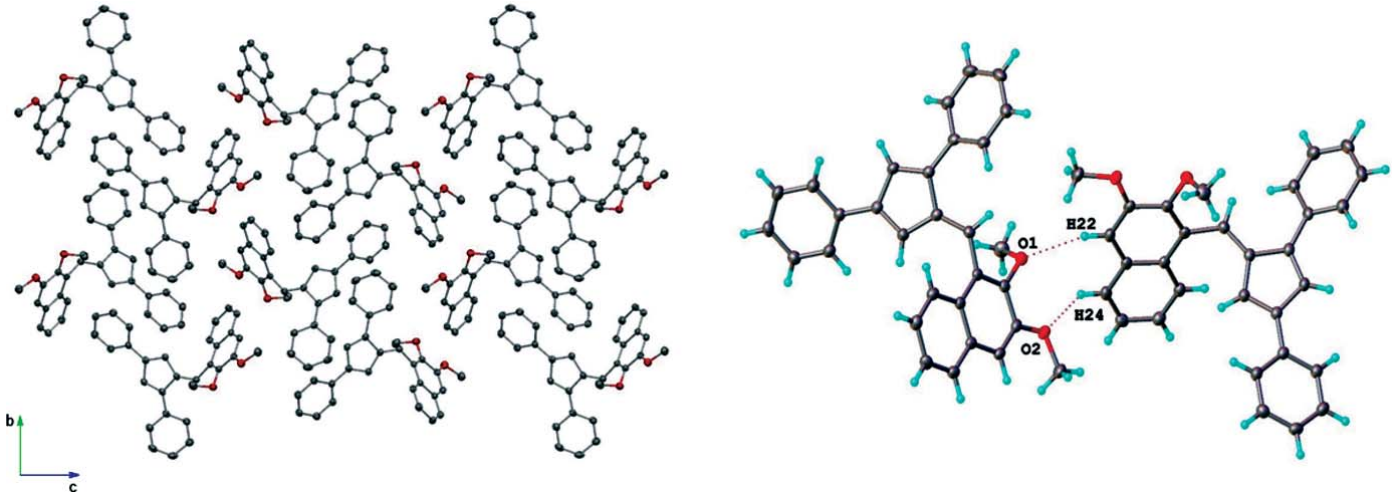

Figure 4
The packing of 6-(2,3-dimethoxynaphthyl)-1,3-diphenylfulvene, **2**, viewed along the *a*-axis direction (left) and C—H···O interactions (right). Hydrogen atoms are omitted for clarity (left).

Table 3
Experimental details.

	1	**2**
Crystal data		
Chemical formula	$C_{21}H_{18}$	$C_{30}H_{24}O_2$
M_r	270.35	416.49
Crystal system, space group	Orthorhombic, *Pbca*	Orthorhombic, $P2_12_12_1$
Temperature (K)	100	100
a, *b*, *c* (Å)	12.9844 (2), 11.9583 (1), 19.3729 (2)	7.3431 (1), 11.5468 (1), 25.7555 (3)
V (Å^3)	3008.06 (6)	2183.79 (4)
Z	8	4
Radiation type	Mo *Kα*	Mo *Kα*
μ (mm^{-1})	0.07	0.08
Crystal size (mm)	0.38 × 0.25 × 0.15	0.27 × 0.15 × 0.13
Data collection		
Diffractometer	XtaLAB Synergy, Single source at offset/far, HyPix3000	XtaLAB Synergy, Single source at offset/far, HyPix3000
Absorption correction	Empirical (using intensity measurements) (*CrysAlis PRO*; Rigaku OD, 2019)	Empirical (using intensity measurements) (*CrysAlis PRO*; Rigaku OD, 2019)
T_{min}, T_{max}	0.404, 1.000	0.735, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	74031, 3353, 2967	54562, 4798, 4437
R_{int}	0.030	0.036
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.648	0.647
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.036, 0.094, 1.08	0.034, 0.082, 1.04
No. of reflections	3353	4798
No. of parameters	190	291
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.20, −0.19	0.16, −0.19
Absolute structure	–	Flack *x* determined using 1774 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	–	−0.3 (4)

Computer programs: *CrysAlis PRO* (Rigaku OD, 2019), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

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supporting information

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Crystal structures of 6-cyclopropyl-1,3-diphenylfulvene and 6-(2,3-dimethoxy-naphthyl)-1,3-diphenylfulvene

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Computing details

For both structures, data collection: *CrysAlis PRO* (Rigaku OD, 2019); cell refinement: *CrysAlis PRO* (Rigaku OD, 2019); data reduction: *CrysAlis PRO* (Rigaku OD, 2019); program(s) used to solve structure: ShelXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

5-(Cyclopropylmethylidene)-1,3-diphenylcyclopenta-1,3-diene (1)

Crystal data

$C_{21}H_{18}$
M_r = 270.35
Orthorhombic, *Pbca*
a = 12.9844 (2) Å
b = 11.9583 (1) Å
c = 19.3729 (2) Å
V = 3008.06 (6) Å^3
Z = 8
F(000) = 1152
D_x = 1.194 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 50470 reflections
θ = 2.1–27.4°
μ = 0.07 mm^{-1}
T = 100 K
Block, orange
0.38 × 0.25 × 0.15 mm

Data collection

XtaLAB Synergy, Single source at offset/far, HyPix3000 diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: empirical (using intensity measurements) (CrysAlisPro; Rigaku OD, 2019)
T_{min} = 0.404, T_{max} = 1.000
74031 measured reflections
3353 independent reflections
2967 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 27.4°, θ_{min} = 2.1°
h = −16→16
k = −15→15
l = −24→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.094
S = 1.08
3353 reflections
190 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 0.7758P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ 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}
C1	0.44298 (7)	0.64490 (8)	0.32819 (5)	0.0198 (2)
C2	0.50655 (7)	0.60105 (8)	0.37681 (5)	0.0203 (2)
H2	0.5051	0.6191	0.4246	0.024*
C3	0.57790 (7)	0.52136 (8)	0.34441 (5)	0.0204 (2)
C4	0.55715 (7)	0.51958 (8)	0.27550 (5)	0.0227 (2)
H4	0.5918	0.4750	0.2422	0.027*
C5	0.47342 (7)	0.59679 (8)	0.26098 (5)	0.0216 (2)
C6	0.43760 (7)	0.62688 (8)	0.19814 (5)	0.0224 (2)
H6	0.3823	0.6788	0.1965	0.027*
C7	0.35860 (7)	0.72504 (8)	0.33954 (5)	0.0207 (2)
C8	0.26145 (8)	0.70904 (9)	0.30946 (5)	0.0249 (2)
H8	0.2502	0.6466	0.2801	0.030*
C9	0.18165 (8)	0.78343 (9)	0.32221 (5)	0.0293 (2)
H9	0.1163	0.7718	0.3013	0.035*
C10	0.19672 (8)	0.87480 (9)	0.36532 (6)	0.0316 (2)
H10	0.1422	0.9260	0.3737	0.038*
C11	0.29212 (8)	0.89076 (9)	0.39608 (6)	0.0298 (2)
H11	0.3026	0.9526	0.4260	0.036*
C12	0.37234 (8)	0.81683 (8)	0.38334 (5)	0.0242 (2)
H12	0.4373	0.8287	0.4046	0.029*
C13	0.65376 (7)	0.45139 (8)	0.38061 (5)	0.0214 (2)
C14	0.65376 (7)	0.44071 (8)	0.45261 (5)	0.0240 (2)
H14	0.6078	0.4848	0.4794	0.029*
C15	0.72009 (8)	0.36649 (8)	0.48540 (5)	0.0278 (2)
H15	0.7187	0.3597	0.5343	0.033*
C16	0.78805 (9)	0.30253 (9)	0.44717 (6)	0.0311 (2)
H16	0.8319	0.2503	0.4696	0.037*
C17	0.79200 (9)	0.31494 (9)	0.37585 (6)	0.0318 (2)
H17	0.8400	0.2727	0.3495	0.038*
C18	0.72593 (8)	0.38887 (8)	0.34322 (5)	0.0269 (2)
H18	0.7296	0.3974	0.2945	0.032*
C19	0.47766 (8)	0.58544 (8)	0.13271 (5)	0.0245 (2)
H19	0.5338	0.5284	0.1360	0.029*
C20	0.48463 (8)	0.66600 (9)	0.07225 (5)	0.0268 (2)
H20A	0.4598	0.7434	0.0795	0.032*
H20B	0.5447	0.6592	0.0411	0.032*

C21	0.40700 (9)	0.57559 (9)	0.07072 (5)	0.0298 (2)
H21A	0.4188	0.5125	0.0386	0.036*
H21B	0.3339	0.5968	0.0769	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0211 (4)	0.0180 (4)	0.0203 (4)	−0.0038 (3)	0.0031 (3)	−0.0006 (3)
C2	0.0216 (4)	0.0199 (4)	0.0194 (4)	−0.0045 (4)	0.0023 (3)	−0.0010 (3)
C3	0.0212 (4)	0.0181 (4)	0.0218 (5)	−0.0036 (4)	0.0017 (4)	0.0002 (3)
C4	0.0256 (5)	0.0218 (5)	0.0208 (5)	0.0028 (4)	0.0020 (4)	−0.0006 (4)
C5	0.0236 (5)	0.0202 (4)	0.0209 (5)	−0.0002 (4)	0.0021 (4)	−0.0007 (4)
C6	0.0236 (5)	0.0213 (5)	0.0223 (5)	0.0019 (4)	0.0016 (4)	0.0001 (4)
C7	0.0232 (5)	0.0203 (4)	0.0187 (4)	−0.0013 (4)	0.0045 (4)	0.0026 (3)
C8	0.0246 (5)	0.0271 (5)	0.0230 (5)	−0.0025 (4)	0.0035 (4)	0.0011 (4)
C9	0.0225 (5)	0.0363 (6)	0.0291 (5)	0.0013 (4)	0.0040 (4)	0.0068 (4)
C10	0.0300 (5)	0.0297 (5)	0.0350 (6)	0.0086 (4)	0.0107 (4)	0.0063 (4)
C11	0.0359 (6)	0.0217 (5)	0.0318 (5)	0.0025 (4)	0.0073 (4)	−0.0014 (4)
C12	0.0267 (5)	0.0211 (5)	0.0248 (5)	−0.0016 (4)	0.0026 (4)	0.0006 (4)
C13	0.0221 (4)	0.0184 (4)	0.0237 (5)	−0.0039 (4)	−0.0019 (4)	−0.0003 (4)
C14	0.0234 (5)	0.0243 (5)	0.0244 (5)	−0.0057 (4)	−0.0002 (4)	0.0007 (4)
C15	0.0312 (5)	0.0272 (5)	0.0252 (5)	−0.0087 (4)	−0.0065 (4)	0.0046 (4)
C16	0.0321 (5)	0.0248 (5)	0.0366 (6)	−0.0004 (4)	−0.0138 (5)	0.0019 (4)
C17	0.0314 (6)	0.0292 (5)	0.0348 (6)	0.0074 (4)	−0.0080 (4)	−0.0071 (4)
C18	0.0290 (5)	0.0277 (5)	0.0240 (5)	0.0028 (4)	−0.0036 (4)	−0.0037 (4)
C19	0.0285 (5)	0.0250 (5)	0.0199 (5)	0.0059 (4)	−0.0001 (4)	−0.0002 (4)
C20	0.0300 (5)	0.0298 (5)	0.0207 (5)	0.0005 (4)	0.0025 (4)	0.0014 (4)
C21	0.0327 (5)	0.0344 (6)	0.0223 (5)	−0.0031 (4)	−0.0024 (4)	−0.0032 (4)

Geometric parameters (Å, º)

C1—C2	1.3577 (13)	C11—C12	1.3882 (14)
C1—C5	1.4774 (13)	C12—H12	0.9500
C1—C7	1.4721 (13)	C13—C14	1.4008 (14)
C2—H2	0.9500	C13—C18	1.4006 (14)
C2—C3	1.4699 (13)	C14—H14	0.9500
C3—C4	1.3621 (13)	C14—C15	1.3903 (14)
C3—C13	1.4704 (13)	C15—H15	0.9500
C4—H4	0.9500	C15—C16	1.3829 (16)
C4—C5	1.4538 (13)	C16—H16	0.9500
C5—C6	1.3520 (13)	C16—C17	1.3905 (16)
C6—H6	0.9500	C17—H17	0.9500
C6—C19	1.4570 (13)	C17—C18	1.3847 (14)
C7—C8	1.4027 (14)	C18—H18	0.9500
C7—C12	1.3988 (13)	C19—H19	1.0000
C8—H8	0.9500	C19—C20	1.5193 (14)
C8—C9	1.3879 (14)	C19—C21	1.5159 (14)
C9—H9	0.9500	C20—H20A	0.9900

C9—C10	1.3891 (16)	C20—H20B	0.9900
C10—H10	0.9500	C20—C21	1.4784 (15)
C10—C11	1.3878 (16)	C21—H21A	0.9900
C11—H11	0.9500	C21—H21B	0.9900
C2—C1—C5	107.36 (8)	C14—C13—C3	121.79 (9)
C2—C1—C7	126.89 (9)	C18—C13—C3	120.35 (9)
C7—C1—C5	125.75 (8)	C18—C13—C14	117.80 (9)
C1—C2—H2	125.1	C13—C14—H14	119.6
C1—C2—C3	109.71 (8)	C15—C14—C13	120.88 (9)
C3—C2—H2	125.1	C15—C14—H14	119.6
C2—C3—C13	125.97 (8)	C14—C15—H15	119.9
C4—C3—C2	107.70 (8)	C16—C15—C14	120.24 (10)
C4—C3—C13	126.23 (9)	C16—C15—H15	119.9
C3—C4—H4	125.4	C15—C16—H16	120.1
C3—C4—C5	109.12 (8)	C15—C16—C17	119.78 (10)
C5—C4—H4	125.4	C17—C16—H16	120.1
C4—C5—C1	106.08 (8)	C16—C17—H17	120.0
C6—C5—C1	126.72 (9)	C18—C17—C16	119.93 (10)
C6—C5—C4	126.91 (9)	C18—C17—H17	120.0
C5—C6—H6	117.6	C13—C18—H18	119.4
C5—C6—C19	124.75 (9)	C17—C18—C13	121.28 (10)
C19—C6—H6	117.6	C17—C18—H18	119.4
C8—C7—C1	121.23 (9)	C6—C19—H19	115.9
C12—C7—C1	120.44 (9)	C6—C19—C20	118.44 (9)
C12—C7—C8	118.28 (9)	C6—C19—C21	119.97 (9)
C7—C8—H8	119.7	C20—C19—H19	115.9
C9—C8—C7	120.66 (10)	C21—C19—H19	115.9
C9—C8—H8	119.7	C21—C19—C20	58.30 (7)
C8—C9—H9	119.8	C19—C20—H20A	117.7
C8—C9—C10	120.40 (10)	C19—C20—H20B	117.7
C10—C9—H9	119.8	H20A—C20—H20B	114.8
C9—C10—H10	120.3	C21—C20—C19	60.74 (7)
C11—C10—C9	119.48 (10)	C21—C20—H20A	117.7
C11—C10—H10	120.3	C21—C20—H20B	117.7
C10—C11—H11	119.8	C19—C21—H21A	117.7
C10—C11—C12	120.38 (10)	C19—C21—H21B	117.7
C12—C11—H11	119.8	C20—C21—C19	60.96 (7)
C7—C12—H12	119.6	C20—C21—H21A	117.7
C11—C12—C7	120.79 (10)	C20—C21—H21B	117.7
C11—C12—H12	119.6	H21A—C21—H21B	114.8

5-[(3,4-Dimethoxynaphthalen-2-yl)methylidene]-1,3-diphenylcyclopenta-1,3-diene (2)

Crystal data

$C_{30}H_{24}O_2$	a = 7.3431 (1) Å
M_r = 416.49	b = 11.5468 (1) Å
Orthorhombic, $P2_12_12_1$	c = 25.7555 (3) Å

V = 2183.79 (4) Å^3
Z = 4
F(000) = 880
D_x = 1.267 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 35583 reflections
θ = 1.9–27.3°
μ = 0.08 mm^{-1}
T = 100 K
Block, red
0.27 × 0.15 × 0.13 mm

Data collection

XtaLAB Synergy, Single source at offset/far, HyPix3000 diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: empirical (using intensity measurements) (CrysAlisPro; Rigaku OD, 2019)
T_{min} = 0.735, T_{max} = 1.000
54562 measured reflections
4798 independent reflections
4437 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 27.4°, θ_{min} = 1.9°
h = −9→9
k = −14→14
l = −32→32

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.082
S = 1.04
4798 reflections
291 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0415P)^2 + 0.3848P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Absolute structure: Flack x determined using 1774 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −0.3 (4)

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	0.44518 (18)	0.62766 (11)	0.68257 (5)	0.0247 (3)
O2	0.61050 (17)	0.49358 (11)	0.75201 (5)	0.0259 (3)
C1	−0.0406 (2)	0.65397 (15)	0.55785 (7)	0.0198 (4)
C2	−0.0799 (2)	0.59770 (15)	0.51299 (7)	0.0211 (4)
H2	−0.1346	0.6322	0.4834	0.025*
C3	−0.0254 (2)	0.47506 (15)	0.51698 (7)	0.0197 (4)
C4	0.0428 (3)	0.45757 (16)	0.56522 (6)	0.0207 (4)
H4	0.0855	0.3857	0.5784	0.025*
C5	0.0400 (2)	0.56652 (15)	0.59360 (6)	0.0196 (4)
C6	0.1117 (2)	0.58571 (15)	0.64113 (7)	0.0204 (4)
H6	0.1098	0.6635	0.6531	0.024*

C7	−0.0696 (2)	0.77839 (15)	0.56788 (7)	0.0201 (4)
C8	−0.1120 (3)	0.82200 (15)	0.61722 (7)	0.0232 (4)
H8	−0.1192	0.7705	0.6459	0.028*
C9	−0.1436 (3)	0.93976 (16)	0.62481 (8)	0.0276 (4)
H9	−0.1710	0.9678	0.6586	0.033*
C10	−0.1352 (3)	1.01607 (17)	0.58346 (8)	0.0302 (4)
H10	−0.1570	1.0963	0.5887	0.036*
C11	−0.0945 (3)	0.97418 (17)	0.53435 (8)	0.0302 (4)
H11	−0.0895	1.0260	0.5058	0.036*
C12	−0.0612 (3)	0.85764 (16)	0.52664 (7)	0.0252 (4)
H12	−0.0321	0.8307	0.4928	0.030*
C13	−0.0383 (2)	0.38948 (15)	0.47494 (6)	0.0198 (4)
C14	−0.1483 (3)	0.40870 (16)	0.43146 (7)	0.0240 (4)
H14	−0.2167	0.4783	0.4289	0.029*
C15	−0.1585 (3)	0.32715 (17)	0.39190 (7)	0.0274 (4)
H15	−0.2351	0.3408	0.3628	0.033*
C16	−0.0574 (3)	0.22612 (16)	0.39474 (7)	0.0252 (4)
H16	−0.0638	0.1707	0.3675	0.030*
C17	0.0532 (3)	0.20609 (16)	0.43754 (7)	0.0243 (4)
H17	0.1233	0.1371	0.4395	0.029*
C18	0.0617 (3)	0.28622 (16)	0.47734 (7)	0.0229 (4)
H18	0.1363	0.2711	0.5067	0.028*
C19	0.1926 (2)	0.49925 (15)	0.67648 (7)	0.0194 (4)
C20	0.3601 (2)	0.52592 (15)	0.69714 (7)	0.0206 (4)
C21	0.4485 (3)	0.45362 (15)	0.73404 (6)	0.0211 (4)
C22	0.3672 (3)	0.35129 (15)	0.74809 (7)	0.0216 (4)
H22	0.4269	0.3012	0.7719	0.026*
C23	0.1953 (3)	0.31938 (15)	0.72749 (6)	0.0198 (4)
C24	0.1143 (3)	0.21256 (16)	0.74162 (7)	0.0233 (4)
H24	0.1763	0.1620	0.7647	0.028*
C25	−0.0526 (3)	0.18134 (16)	0.72228 (7)	0.0254 (4)
H25	−0.1043	0.1087	0.7314	0.030*
C26	−0.1476 (3)	0.25666 (16)	0.68892 (7)	0.0250 (4)
H26	−0.2644	0.2353	0.6762	0.030*
C27	−0.0726 (2)	0.36052 (16)	0.67463 (6)	0.0222 (4)
H27	−0.1393	0.4111	0.6526	0.027*
C28	0.1027 (2)	0.39369 (15)	0.69210 (6)	0.0187 (4)
C29	0.5376 (3)	0.61847 (19)	0.63345 (8)	0.0317 (4)
H29A	0.4567	0.5819	0.6080	0.048*
H29B	0.6476	0.5714	0.6376	0.048*
H29C	0.5714	0.6960	0.6213	0.048*
C30	0.6930 (3)	0.42942 (18)	0.79361 (8)	0.0297 (4)
H30A	0.7212	0.3508	0.7817	0.044*
H30B	0.6085	0.4256	0.8230	0.044*
H30C	0.8055	0.4681	0.8045	0.044*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0290 (7)	0.0200 (6)	0.0252 (6)	−0.0041 (6)	−0.0044 (6)	0.0013 (5)
O2	0.0258 (6)	0.0266 (7)	0.0251 (7)	−0.0008 (6)	−0.0095 (6)	0.0015 (5)
C1	0.0178 (8)	0.0220 (9)	0.0195 (8)	−0.0005 (7)	−0.0003 (7)	0.0025 (7)
C2	0.0220 (9)	0.0227 (9)	0.0186 (8)	0.0009 (8)	−0.0026 (7)	0.0029 (7)
C3	0.0175 (8)	0.0227 (9)	0.0189 (8)	−0.0013 (7)	0.0002 (7)	0.0006 (7)
C4	0.0225 (8)	0.0208 (9)	0.0187 (8)	0.0006 (7)	−0.0008 (7)	0.0008 (7)
C5	0.0182 (8)	0.0218 (9)	0.0188 (8)	−0.0006 (7)	0.0002 (7)	0.0022 (7)
C6	0.0214 (8)	0.0184 (8)	0.0212 (8)	0.0014 (7)	−0.0005 (7)	−0.0004 (7)
C7	0.0167 (8)	0.0206 (9)	0.0229 (9)	−0.0004 (7)	−0.0033 (7)	0.0032 (7)
C8	0.0244 (9)	0.0224 (9)	0.0226 (9)	0.0014 (8)	−0.0031 (7)	0.0023 (7)
C9	0.0281 (10)	0.0264 (10)	0.0283 (10)	0.0029 (8)	−0.0048 (8)	−0.0039 (8)
C10	0.0294 (10)	0.0193 (9)	0.0420 (11)	0.0030 (8)	−0.0075 (9)	0.0002 (8)
C11	0.0322 (10)	0.0247 (10)	0.0336 (10)	−0.0016 (9)	−0.0049 (9)	0.0108 (8)
C12	0.0250 (9)	0.0269 (10)	0.0239 (9)	−0.0012 (8)	−0.0017 (8)	0.0027 (7)
C13	0.0198 (8)	0.0220 (9)	0.0174 (8)	−0.0025 (7)	0.0010 (7)	0.0013 (7)
C14	0.0258 (9)	0.0248 (9)	0.0215 (9)	0.0000 (8)	−0.0017 (7)	0.0013 (7)
C15	0.0312 (10)	0.0309 (10)	0.0201 (9)	−0.0032 (8)	−0.0048 (8)	−0.0002 (8)
C16	0.0294 (10)	0.0253 (9)	0.0211 (9)	−0.0052 (8)	0.0031 (8)	−0.0043 (7)
C17	0.0251 (9)	0.0227 (9)	0.0252 (9)	0.0000 (8)	0.0026 (8)	−0.0002 (7)
C18	0.0249 (9)	0.0250 (9)	0.0189 (9)	0.0002 (8)	−0.0010 (7)	0.0015 (7)
C19	0.0240 (9)	0.0186 (9)	0.0155 (8)	0.0026 (7)	0.0000 (7)	−0.0027 (7)
C20	0.0251 (9)	0.0187 (9)	0.0179 (8)	0.0016 (8)	0.0005 (7)	−0.0025 (7)
C21	0.0228 (9)	0.0235 (9)	0.0170 (8)	0.0039 (7)	−0.0025 (7)	−0.0038 (7)
C22	0.0257 (9)	0.0227 (9)	0.0164 (8)	0.0047 (7)	−0.0025 (7)	0.0001 (7)
C23	0.0243 (9)	0.0207 (9)	0.0145 (8)	0.0031 (7)	0.0019 (7)	−0.0016 (7)
C24	0.0295 (10)	0.0232 (9)	0.0172 (8)	0.0031 (8)	0.0007 (7)	0.0007 (7)
C25	0.0317 (10)	0.0233 (9)	0.0210 (9)	−0.0039 (8)	0.0044 (8)	0.0011 (7)
C26	0.0236 (9)	0.0295 (10)	0.0219 (9)	−0.0030 (8)	0.0000 (8)	−0.0008 (7)
C27	0.0237 (9)	0.0248 (9)	0.0182 (8)	0.0027 (8)	0.0000 (7)	0.0005 (7)
C28	0.0219 (8)	0.0195 (8)	0.0148 (8)	0.0021 (7)	0.0016 (7)	−0.0019 (6)
C29	0.0296 (10)	0.0336 (11)	0.0320 (10)	−0.0052 (9)	0.0038 (9)	0.0054 (9)
C30	0.0308 (10)	0.0321 (11)	0.0261 (10)	0.0024 (9)	−0.0096 (9)	−0.0006 (8)

Geometric parameters (Å, º)

O1—C20	1.383 (2)	C14—C15	1.389 (3)
O1—C29	1.439 (2)	C15—H15	0.9500
O2—C21	1.357 (2)	C15—C16	1.385 (3)
O2—C30	1.436 (2)	C16—H16	0.9500
C1—C2	1.357 (2)	C16—C17	1.389 (3)
C1—C5	1.489 (2)	C17—H17	0.9500
C1—C7	1.475 (2)	C17—C18	1.382 (3)
C2—H2	0.9500	C18—H18	0.9500
C2—C3	1.475 (2)	C19—C20	1.375 (2)
C3—C4	1.354 (2)	C19—C28	1.443 (2)

C3—C13	1.469 (2)	C20—C21	1.422 (2)
C4—H4	0.9500	C21—C22	1.372 (3)
C4—C5	1.455 (2)	C22—H22	0.9500
C5—C6	1.351 (2)	C22—C23	1.418 (3)
C6—H6	0.9500	C23—C24	1.417 (3)
C6—C19	1.476 (2)	C23—C28	1.425 (2)
C7—C8	1.402 (2)	C24—H24	0.9500
C7—C12	1.403 (2)	C24—C25	1.371 (3)
C8—H8	0.9500	C25—H25	0.9500
C8—C9	1.393 (3)	C25—C26	1.408 (3)
C9—H9	0.9500	C26—H26	0.9500
C9—C10	1.384 (3)	C26—C27	1.370 (3)
C10—H10	0.9500	C27—H27	0.9500
C10—C11	1.387 (3)	C27—C28	1.417 (3)
C11—H11	0.9500	C29—H29A	0.9800
C11—C12	1.382 (3)	C29—H29B	0.9800
C12—H12	0.9500	C29—H29C	0.9800
C13—C14	1.398 (2)	C30—H30A	0.9800
C13—C18	1.402 (3)	C30—H30B	0.9800
C14—H14	0.9500	C30—H30C	0.9800

C20—O1—C29	112.90 (14)	C17—C16—H16	120.1
C21—O2—C30	116.64 (15)	C16—C17—H17	119.9
C2—C1—C5	106.64 (15)	C18—C17—C16	120.23 (18)
C2—C1—C7	125.79 (16)	C18—C17—H17	119.9
C7—C1—C5	127.55 (15)	C13—C18—H18	119.6
C1—C2—H2	125.0	C17—C18—C13	120.86 (17)
C1—C2—C3	110.03 (15)	C17—C18—H18	119.6
C3—C2—H2	125.0	C20—C19—C6	116.56 (16)
C4—C3—C2	107.90 (15)	C20—C19—C28	119.35 (16)
C4—C3—C13	126.85 (17)	C28—C19—C6	124.01 (16)
C13—C3—C2	125.22 (15)	O1—C20—C21	118.28 (16)
C3—C4—H4	125.5	C19—C20—O1	119.31 (16)
C3—C4—C5	109.07 (16)	C19—C20—C21	122.39 (17)
C5—C4—H4	125.5	O2—C21—C20	115.38 (16)
C4—C5—C1	106.34 (14)	O2—C21—C22	125.74 (16)
C6—C5—C1	127.15 (16)	C22—C21—C20	118.88 (17)
C6—C5—C4	126.27 (16)	C21—C22—H22	119.6
C5—C6—H6	116.4	C21—C22—C23	120.82 (16)
C5—C6—C19	127.22 (16)	C23—C22—H22	119.6
C19—C6—H6	116.4	C22—C23—C28	120.52 (16)
C8—C7—C1	122.72 (16)	C24—C23—C22	120.26 (17)
C8—C7—C12	117.49 (16)	C24—C23—C28	119.22 (17)
C12—C7—C1	119.75 (16)	C23—C24—H24	119.6
C7—C8—H8	119.5	C25—C24—C23	120.73 (18)
C9—C8—C7	120.99 (17)	C25—C24—H24	119.6
C9—C8—H8	119.5	C24—C25—H25	119.9
C8—C9—H9	119.8	C24—C25—C26	120.15 (18)

C10—C9—C8	120.40 (18)	C26—C25—H25	119.9
C10—C9—H9	119.8	C25—C26—H26	119.8
C9—C10—H10	120.3	C27—C26—C25	120.37 (18)
C9—C10—C11	119.30 (17)	C27—C26—H26	119.8
C11—C10—H10	120.3	C26—C27—H27	119.4
C10—C11—H11	119.7	C26—C27—C28	121.11 (17)
C12—C11—C10	120.59 (18)	C28—C27—H27	119.4
C12—C11—H11	119.7	C23—C28—C19	117.96 (16)
C7—C12—H12	119.4	C27—C28—C19	123.74 (16)
C11—C12—C7	121.22 (17)	C27—C28—C23	118.30 (16)
C11—C12—H12	119.4	O1—C29—H29A	109.5
C14—C13—C3	121.39 (16)	O1—C29—H29B	109.5
C14—C13—C18	118.22 (16)	O1—C29—H29C	109.5
C18—C13—C3	120.38 (16)	H29A—C29—H29B	109.5
C13—C14—H14	119.6	H29A—C29—H29C	109.5
C15—C14—C13	120.73 (17)	H29B—C29—H29C	109.5
C15—C14—H14	119.6	O2—C30—H30A	109.5
C14—C15—H15	119.9	O2—C30—H30B	109.5
C16—C15—C14	120.21 (18)	O2—C30—H30C	109.5
C16—C15—H15	119.9	H30A—C30—H30B	109.5
C15—C16—H16	120.1	H30A—C30—H30C	109.5
C15—C16—C17	119.73 (17)	H30B—C30—H30C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C22—H22···O1^{i}	0.95	2.50	3.429 (2)	164
C24—H24···O2^{i}	0.95	2.53	3.241 (2)	131

Symmetry code: (i) −*x*+1, *y*−1/2, −*z*+3/2.