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1,3-Bis(4-methylbenzoyl)-2,4-bis(2,4,5-trimethoxyphenyl)cyclobutane

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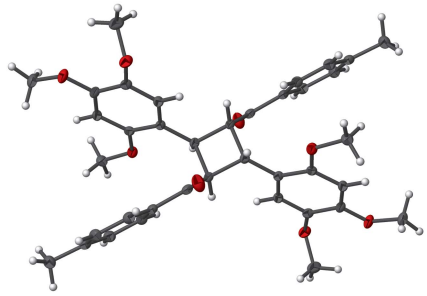
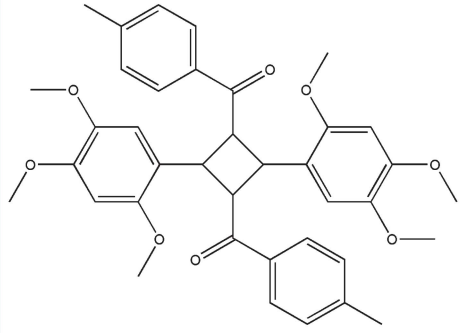
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{38}H_{40}O_8$, possess an inversion centre at the centroid of the four-membered ring. The dihedral angle between the methylbenzene and trimethoxybenzene rings is 46.19 (8)°. In the crystal, molecules are linked *via* weak C—H···π interactions, forming centrosymmetric supramolecular dimers.

3D view



Chemical scheme



Structure description

Recently, a new route to polysubstituted cyclobutanes *via* $K_2S_2O_8$-promoted [2 + 2]-cycloaddition was reported (Zhu *et al.*, 2016). We carried out a reaction of 2,4,5-trimethoxybenzaldehyde and 4-methyl acetophenone in the presence of in 95% ethyl alcohol under reflux conditions. After completion, the reaction unexpectedly yielded the title compound *via* the intermolecular [2 + 2]-cycloaddition of the expected (*E*)-1-(*p*-tolyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one.

The molecular structure of the title compound is shown in Fig. 1. The molecule is located about an inversion centre at the centroid of the four-membered ring. The dihedral angle between the aromatic rings is 46.19 (8)°. The methoxy groups at C4 and C6 lie close to the plane of theeir attached benzene ring, as indicated by the torsion angle values of −7.2 (2)° and 174.05 (14)° for C8—O3—C4—C5 and C7—O2—C6—C1 respectively whereas the methoxy group at C3 is twisted out of the plane of the benzene ring [C9—O4—C3—C2 = 119.22 (17)°] . In the crystal, the molecules are linked *via* weak C—H···π interactions (Table 1), forming centrosymmetric supramolecular dimers.

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

*Cg*2 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*C*···*Cg*2^{i}	0.96	2.75	3.5764 (19)	145

Symmetry code: (i) $-x, -y+1, -z+1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{38}H_{40}O_8$
M_r	624.70
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, b, c (Å)	8.1155 (4), 9.9316 (5), 11.0656 (6)
α, β, γ (°)	70.471 (1), 81.955 (1), 67.854 (1)
V (Å^3)	778.47 (7)
Z	1
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.76
Crystal size (mm)	0.28 × 0.25 × 0.22
Data collection	
Diffractometer	Bruker X8 Proteum
Absorption correction	Multi-scan (*SADABS*; Bruker, 2013)
T_{min}, T_{max}	0.817, 0.852
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8126, 2539, 2458
R_{int}	0.042
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.585
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.050, 0.154, 1.05
No. of reflections	2539
No. of parameters	212
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.34, −0.25

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2008) and *PLATON* (Spek, 2009).

Synthesis and crystallization

A mixture of 2,4,5-trimethoxybenzaldehyde (5 mmol), 4-methyl acetophenone (5 mmol) and sodium hydroxide (5 mmol) in 95% ethyl alcohol (25 ml) was refluxed on a water bath conditions for 1 h. The progress of the reaction was monitored by TLC. After the completion of the reaction, the mixture was poured into ice-cold water and kept in the refrigerator overnight. The solid that formed was filtered, and washed with cold hydrochloric acid (5%). Recrystallization from methanol solution yielded yellow slabs of the title compound. Yield 78%, m.p. 108–110 °C.

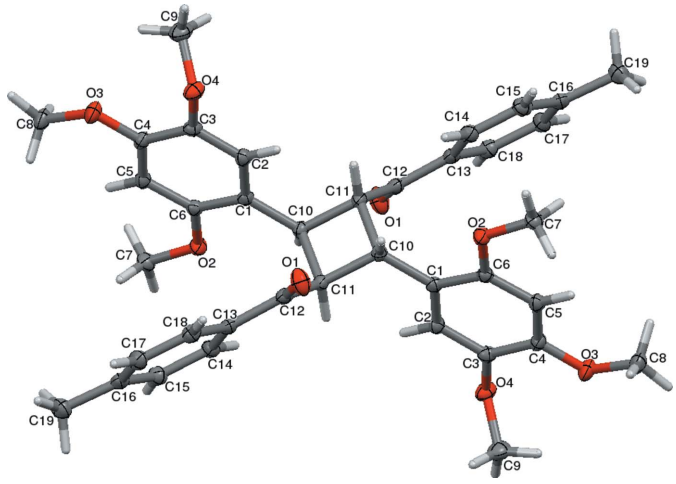


Figure 1
The molecular structure of the title compound with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2017). **2**, x170113 [https://doi.org/10.1107/S2414314617001134]

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Crystal data

$C_{38}H_{40}O_8$
M_r = 624.70
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 8.1155 (4) Å
b = 9.9316 (5) Å
c = 11.0656 (6) Å
α = 70.471 (1)°
β = 81.955 (1)°
γ = 67.854 (1)°
V = 778.47 (7) Å^3
Z = 1
$F(000)$ = 332
D_x = 1.332 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 2458 reflections
θ = 5.9–64.3°
μ = 0.76 mm^{-1}
T = 296 K
Rectangle, yellow
0.28 × 0.25 × 0.22 mm

Data collection

Bruker X8 Proteum diffractometer
Radiation source: Bruker MicroStar microfocus rotating anode
Helios multilayer optics monochromator
Detector resolution: 18.4 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2013)
T_{min} = 0.817, T_{max} = 0.852
8126 measured reflections
2539 independent reflections
2458 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 64.3°, θ_{min} = 5.9°
h = −8→9
k = −11→11
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.154
S = 1.05
2539 reflections
212 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1056P)^2 + 0.2971P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating -R-factor-obs 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.44259 (16)	0.19813 (15)	0.23209 (11)	0.0295 (4)
O2	0.01550 (14)	0.26130 (12)	0.51807 (11)	0.0198 (3)
O3	0.11060 (15)	0.46294 (13)	0.83588 (11)	0.0230 (3)
O4	0.47051 (15)	0.31512 (13)	0.81383 (11)	0.0223 (3)
C1	0.3046 (2)	0.20001 (17)	0.58511 (14)	0.0162 (4)
C2	0.4151 (2)	0.21892 (17)	0.65963 (15)	0.0175 (5)
C3	0.3506 (2)	0.30910 (17)	0.74005 (15)	0.0175 (5)
C4	0.1670 (2)	0.38172 (17)	0.75073 (15)	0.0179 (5)
C5	0.0533 (2)	0.36687 (17)	0.67677 (15)	0.0180 (5)
C6	0.1219 (2)	0.27751 (17)	0.59445 (14)	0.0162 (5)
C7	−0.1718 (2)	0.32646 (19)	0.53436 (16)	0.0220 (5)
C8	−0.0744 (2)	0.55118 (19)	0.83837 (17)	0.0248 (5)
C9	0.4770 (2)	0.4642 (2)	0.78940 (19)	0.0297 (6)
C10	0.3761 (2)	0.10060 (17)	0.49825 (15)	0.0164 (5)
C11	0.5654 (2)	0.07947 (17)	0.44097 (15)	0.0165 (5)
C12	0.5760 (2)	0.14150 (17)	0.29597 (15)	0.0184 (5)
C13	0.7536 (2)	0.13001 (17)	0.23417 (15)	0.0186 (5)
C14	0.9074 (2)	0.07966 (18)	0.30416 (15)	0.0199 (5)
C15	1.0701 (2)	0.06841 (18)	0.24225 (16)	0.0222 (5)
C16	1.0864 (2)	0.10438 (18)	0.10918 (16)	0.0215 (5)
C17	0.9319 (2)	0.15728 (18)	0.03947 (16)	0.0228 (5)
C18	0.7680 (2)	0.17084 (18)	0.10031 (16)	0.0208 (5)
C19	1.2655 (2)	0.0854 (2)	0.04421 (17)	0.0269 (5)
H2	0.53720	0.16870	0.65500	0.0210*
H5	−0.06890	0.41670	0.68220	0.0220*
H7A	−0.20530	0.28090	0.62070	0.0330*
H7B	−0.23080	0.30790	0.47550	0.0330*
H7C	−0.20570	0.43450	0.51790	0.0330*
H8A	−0.11030	0.62020	0.75420	0.0370*
H8B	−0.09580	0.60840	0.89740	0.0370*
H8C	−0.14150	0.48430	0.86520	0.0370*
H9A	0.50480	0.50400	0.70040	0.0450*
H9B	0.56710	0.45760	0.84120	0.0450*
H9C	0.36360	0.53080	0.81030	0.0450*
H10	0.29200	0.13580	0.42910	0.0200*

H11	0.62770	0.12040	0.48140	0.0200*
H14	0.90040	0.05350	0.39320	0.0240*
H15	1.17070	0.03610	0.29040	0.0270*
H17	0.93920	0.18390	−0.04950	0.0270*
H18	0.66660	0.20740	0.05180	0.0250*
H19A	1.30410	0.16480	0.04640	0.0400*
H19B	1.25710	0.09150	−0.04330	0.0400*
H19C	1.34970	−0.01230	0.08800	0.0400*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0193 (6)	0.0386 (8)	0.0237 (7)	−0.0057 (5)	−0.0007 (5)	−0.0061 (5)
O2	0.0152 (6)	0.0226 (6)	0.0242 (6)	−0.0059 (5)	−0.0006 (4)	−0.0114 (5)
O3	0.0209 (6)	0.0233 (6)	0.0253 (6)	−0.0018 (5)	−0.0009 (5)	−0.0149 (5)
O4	0.0221 (6)	0.0211 (6)	0.0260 (6)	−0.0057 (5)	−0.0059 (5)	−0.0101 (5)
C1	0.0171 (8)	0.0130 (7)	0.0174 (8)	−0.0050 (6)	0.0013 (6)	−0.0044 (6)
C2	0.0148 (8)	0.0156 (8)	0.0203 (8)	−0.0042 (6)	0.0006 (6)	−0.0050 (6)
C3	0.0183 (8)	0.0150 (8)	0.0187 (8)	−0.0058 (6)	−0.0024 (6)	−0.0039 (6)
C4	0.0212 (8)	0.0149 (8)	0.0166 (8)	−0.0048 (6)	0.0018 (6)	−0.0063 (6)
C5	0.0152 (8)	0.0154 (8)	0.0210 (8)	−0.0037 (6)	0.0022 (6)	−0.0054 (6)
C6	0.0168 (8)	0.0149 (8)	0.0171 (8)	−0.0070 (6)	0.0002 (6)	−0.0038 (6)
C7	0.0160 (8)	0.0233 (9)	0.0282 (9)	−0.0074 (7)	−0.0006 (6)	−0.0091 (7)
C8	0.0212 (9)	0.0236 (9)	0.0293 (9)	−0.0026 (7)	0.0020 (7)	−0.0148 (7)
C9	0.0263 (9)	0.0270 (10)	0.0428 (11)	−0.0105 (8)	−0.0044 (8)	−0.0166 (8)
C10	0.0147 (8)	0.0157 (8)	0.0186 (8)	−0.0041 (6)	−0.0002 (6)	−0.0065 (6)
C11	0.0149 (8)	0.0158 (8)	0.0212 (8)	−0.0053 (6)	0.0013 (6)	−0.0093 (6)
C12	0.0202 (9)	0.0132 (7)	0.0223 (8)	−0.0048 (6)	−0.0008 (7)	−0.0070 (6)
C13	0.0215 (9)	0.0135 (8)	0.0218 (8)	−0.0068 (6)	0.0031 (7)	−0.0073 (6)
C14	0.0239 (9)	0.0175 (8)	0.0175 (8)	−0.0079 (7)	0.0015 (6)	−0.0045 (6)
C15	0.0208 (8)	0.0194 (8)	0.0259 (9)	−0.0071 (7)	−0.0014 (7)	−0.0061 (7)
C16	0.0240 (9)	0.0145 (8)	0.0264 (9)	−0.0081 (6)	0.0039 (7)	−0.0072 (6)
C17	0.0289 (9)	0.0204 (8)	0.0180 (8)	−0.0096 (7)	0.0039 (7)	−0.0051 (6)
C18	0.0223 (9)	0.0183 (8)	0.0212 (8)	−0.0062 (7)	−0.0014 (6)	−0.0060 (6)
C19	0.0269 (9)	0.0259 (9)	0.0271 (9)	−0.0110 (7)	0.0058 (7)	−0.0076 (7)

Geometric parameters (Å, º)

O1—C12	1.216 (2)	C16—C19	1.502 (3)
O2—C6	1.373 (2)	C17—C18	1.384 (2)
O2—C7	1.422 (2)	C2—H2	0.9300
O3—C4	1.363 (2)	C5—H5	0.9300
O3—C8	1.427 (2)	C7—H7A	0.9600
O4—C3	1.384 (2)	C7—H7B	0.9600
O4—C9	1.434 (2)	C7—H7C	0.9600
C1—C2	1.399 (2)	C8—H8A	0.9600
C1—C6	1.397 (2)	C8—H8B	0.9600
C1—C10	1.511 (2)	C8—H8C	0.9600

C2—C3	1.385 (2)	C9—H9A	0.9600
C3—C4	1.397 (2)	C9—H9B	0.9600
C4—C5	1.392 (2)	C9—H9C	0.9600
C5—C6	1.398 (2)	C10—H10	0.9800
C10—C11	1.543 (2)	C11—H11	0.9800
C10—C11^{i}	1.588 (2)	C14—H14	0.9300
C11—C12	1.517 (2)	C15—H15	0.9300
C12—C13	1.487 (2)	C17—H17	0.9300
C13—C14	1.396 (2)	C18—H18	0.9300
C13—C18	1.399 (2)	C19—H19A	0.9600
C14—C15	1.383 (2)	C19—H19B	0.9600
C15—C16	1.394 (2)	C19—H19C	0.9600
C16—C17	1.395 (2)		

C6—O2—C7	117.55 (13)	C6—C5—H5	120.00
C4—O3—C8	117.44 (14)	O2—C7—H7A	109.00
C3—O4—C9	114.77 (13)	O2—C7—H7B	109.00
C2—C1—C6	116.90 (14)	O2—C7—H7C	109.00
C2—C1—C10	122.54 (15)	H7A—C7—H7B	109.00
C6—C1—C10	120.56 (15)	H7A—C7—H7C	109.00
C1—C2—C3	122.91 (16)	H7B—C7—H7C	109.00
O4—C3—C2	118.38 (15)	O3—C8—H8A	109.00
O4—C3—C4	122.34 (14)	O3—C8—H8B	109.00
C2—C3—C4	119.12 (16)	O3—C8—H8C	109.00
O3—C4—C3	116.61 (15)	H8A—C8—H8B	109.00
O3—C4—C5	123.95 (15)	H8A—C8—H8C	109.00
C3—C4—C5	119.45 (15)	H8B—C8—H8C	110.00
C4—C5—C6	120.32 (16)	O4—C9—H9A	109.00
O2—C6—C1	116.25 (14)	O4—C9—H9B	109.00
O2—C6—C5	122.50 (15)	O4—C9—H9C	109.00
C1—C6—C5	121.26 (15)	H9A—C9—H9B	109.00
C1—C10—C11	118.41 (14)	H9A—C9—H9C	110.00
C1—C10—C11^{i}	119.20 (13)	H9B—C9—H9C	109.00
C11—C10—C11^{i}	89.08 (12)	C1—C10—H10	110.00
C10—C11—C12	115.28 (14)	C11—C10—H10	110.00
C10—C11—C10^{i}	90.92 (12)	C11^{i}—C10—H10	110.00
C10^{i}—C11—C12	116.86 (13)	C10—C11—H11	111.00
O1—C12—C11	120.71 (15)	C12—C11—H11	111.00
O1—C12—C13	120.96 (14)	C10^{i}—C11—H11	111.00
C11—C12—C13	118.33 (14)	C13—C14—H14	120.00
C12—C13—C14	122.74 (14)	C15—C14—H14	120.00
C12—C13—C18	118.87 (15)	C14—C15—H15	119.00
C14—C13—C18	118.39 (16)	C16—C15—H15	119.00
C13—C14—C15	120.54 (15)	C16—C17—H17	119.00
C14—C15—C16	121.40 (16)	C18—C17—H17	119.00
C15—C16—C17	117.85 (16)	C13—C18—H18	120.00
C15—C16—C19	120.54 (16)	C17—C18—H18	120.00
C17—C16—C19	121.61 (15)	C16—C19—H19A	110.00

C16—C17—C18	121.23 (16)	C16—C19—H19B	109.00
C13—C18—C17	120.54 (16)	C16—C19—H19C	109.00
C1—C2—H2	119.00	H19A—C19—H19B	109.00
C3—C2—H2	119.00	H19A—C19—H19C	109.00
C4—C5—H5	120.00	H19B—C19—H19C	109.00
C7—O2—C6—C1	174.05 (14)	C1—C10—C11—C12	−116.41 (16)
C7—O2—C6—C5	−6.3 (2)	C1—C10—C11—C10^{i}	123.10 (14)
C8—O3—C4—C3	173.45 (14)	C11^{i}—C10—C11—C12	120.49 (14)
C8—O3—C4—C5	−7.2 (2)	C11^{i}—C10—C11—C10^{i}	0.00 (11)
C9—O4—C3—C2	119.22 (17)	C1—C10—C11^{i}—C10^{i}	−122.43 (16)
C9—O4—C3—C4	−65.4 (2)	C1—C10—C11^{i}—C12^{i}	−3.3 (2)
C6—C1—C2—C3	0.2 (2)	C11—C10—C11^{i}—C10^{i}	0.00 (10)
C10—C1—C2—C3	−179.61 (15)	C11—C10—C11^{i}—C12^{i}	119.13 (15)
C2—C1—C6—O2	178.24 (14)	C10—C11—C12—O1	−2.6 (2)
C2—C1—C6—C5	−1.4 (2)	C10—C11—C12—C13	177.95 (14)
C10—C1—C6—O2	−2.0 (2)	C10^{i}—C11—C12—O1	102.45 (19)
C10—C1—C6—C5	178.38 (15)	C10^{i}—C11—C12—C13	−77.0 (2)
C2—C1—C10—C11	−28.5 (2)	O1—C12—C13—C14	173.58 (17)
C2—C1—C10—C11^{i}	77.8 (2)	O1—C12—C13—C18	−6.6 (2)
C6—C1—C10—C11	151.72 (15)	C11—C12—C13—C14	−6.9 (2)
C6—C1—C10—C11^{i}	−101.93 (18)	C11—C12—C13—C18	172.89 (15)
C1—C2—C3—O4	177.31 (15)	C12—C13—C14—C15	178.84 (16)
C1—C2—C3—C4	1.8 (2)	C18—C13—C14—C15	−1.0 (3)
O4—C3—C4—O3	1.6 (2)	C12—C13—C18—C17	−178.03 (16)
O4—C3—C4—C5	−177.83 (15)	C14—C13—C18—C17	1.8 (3)
C2—C3—C4—O3	176.91 (14)	C13—C14—C15—C16	−1.0 (3)
C2—C3—C4—C5	−2.5 (2)	C14—C15—C16—C17	2.1 (3)
O3—C4—C5—C6	−178.04 (15)	C14—C15—C16—C19	−177.55 (17)
C3—C4—C5—C6	1.3 (2)	C15—C16—C17—C18	−1.3 (3)
C4—C5—C6—O2	−178.93 (14)	C19—C16—C17—C18	178.38 (17)
C4—C5—C6—C1	0.7 (2)	C16—C17—C18—C13	−0.7 (3)

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

Hydrogen-bond geometry (Å, º)

Cg2 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*C*···*Cg*2^{ii}	0.96	2.75	3.5764 (19)	145

Symmetry code: (ii) −*x*, −*y*+1, −*z*+1.