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Methyl 3-(2,5-dimethoxy-3,4,6-trimethylphenyl)-propanoate

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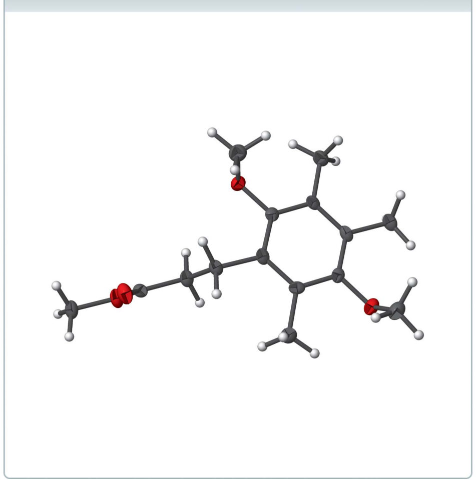
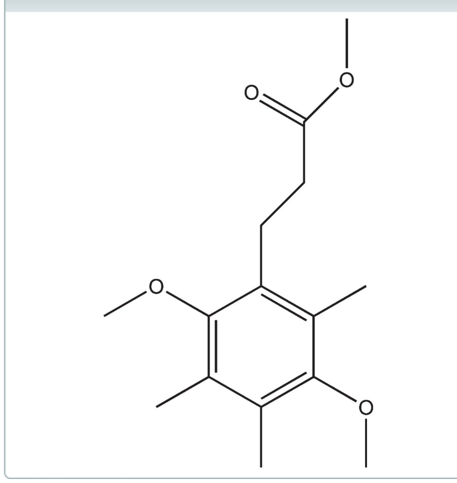
In the title compound, $C_{15}H_{22}O_4$, the fully substituted benzene ring carries a methyl propanoate, two methoxy and three methyl substituents. Both methoxy substituents are almost orthogonal to the benzene ring plane. The methyl propanoate group is almost planar, r.m.s. deviation 0.0138 Å, and is inclined to the benzene ring plane by 80.26 (14)°. In the crystal, C—H···O hydrogen bonds form head-to-tail chains of molecules along the *b*-axis direction that are supported by very weak C—H···π(ring) contacts.

3D view



Chemical scheme



Structure description

The title compound, Fig. 1, is an intermediate in the synthesis of dimethoxybenzene-appended acrylate and methacrylate monomers (Goswami *et al.*, 2017). The fully substituted benzene ring carries a methyl propanoate, two methoxy and three methyl substituents. The methoxy substituents are *para* to each other and lie almost at right angles to the benzene ring in a *cis* conformation; the C2/O2/C21 and C5/O5/C51 planes are inclined to the benzene ring plane by 83.7 (3) and 84.8 (3)°, respectively, and overall present a C21–O2···O5–C51 torsion angle of approximately 11.57°. The C1/C7/C8/C9/O9/O91/C91 methyl propanoate group is almost planar, r.m.s. deviation 0.0138 Å; the dihedral angle between this plane and that of the benzene ring is 80.26 (14)°.

In the crystal, C41—H41*B*···O91 and C41—H41*C*···O9 hydrogen bonds, supported by very weak C8—H8*A*···*Cg* contacts form chains of molecules arranged in a head-to-tail fashion along the *b* axis, Fig. 2 and Table 1. No significant additional contacts are found between adjacent chains that stack the molecules along the *b*-axis direction, Fig. 3.

Structures of compounds with 2,5-dimethoxy-3,4,6-trimethyl-substituted benzene rings are rare with only two entries (Wickramasinhage *et al.*, 2016; Wiedenfeld *et al.*, 2003) found in the CSD (Version 5.37, November 2015 with three updates; Groom *et al.* 2016).

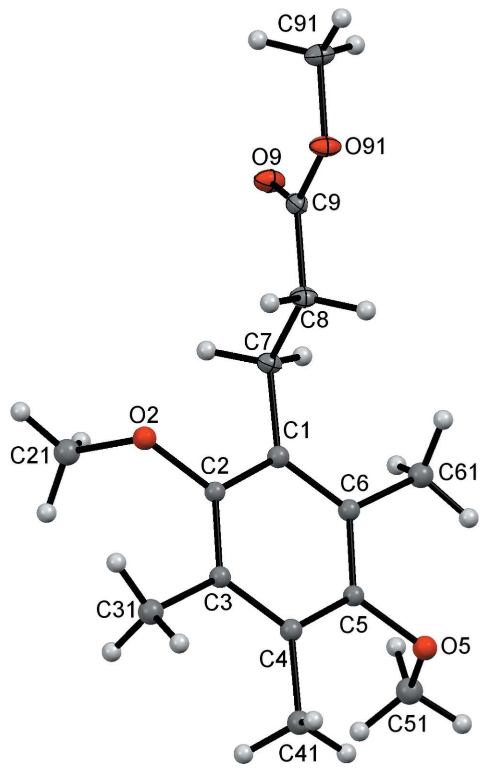


Figure 1
The structure of the title compound showing the atom numbering, with displacement ellipsoids drawn at the 50% probability level.

In addition, we have recently reported the structure of a third related compound, 3-(2,5-dimethoxy-3,4,6-trimethylphenyl)-propyl methacrylate (Goswami *et al.*, 2017).

Synthesis and crystallization

Synthesis is by methylation of 6-hydroxy-5,7,8-trimethyl-chroman-2-one (Goswami *et al.*, 2011) as reported previously

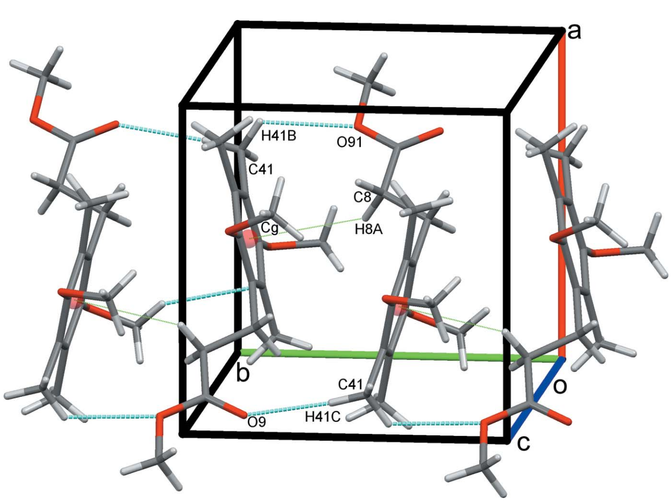


Figure 2
Chains of molecules along the *b* axis with hydrogen bonds drawn as dashed lines. Ring centroids are shown as red spheres and C—H$\cdots\pi$(ring) contacts are drawn as green dotted lines.

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

Cg is the centroid of the C1–C6 benzene ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C41—H41$B\cdots$O91^{i}	0.98	2.59	3.205 (6)	121
C41—H41$C\cdots$O9^{ii}	0.98	2.41	3.392 (5)	175
C8—H8$A\cdots Cg^{ii}$	0.99	3.15	3.763 (6)	121

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{22}O_4$
M_r	266.32
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	92
a, b, c (Å)	8.7453 (13), 8.6888 (10), 9.2511 (11)
β (°)	95.936 (8)
V (Å^3)	699.19 (16)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.42 × 0.08 × 0.07
Data collection	
Diffractometer	Bruker APEXII CCD area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2013)
T_{min}, T_{max}	0.660, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5680, 1929, 1781
R_{int}	0.042
θ_{max} (°)	23.0
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.549
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.098, 1.09
No. of reflections	1929
No. of parameters	178
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.14, −0.20
Absolute structure	Flack x determined using 713 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ Parsons *et al.* (2013)
Absolute structure parameter	0.4 (10)

Computer programs: *APEX2* and (Bruker, 2013), *SHELXS2013* (Sheldrick, 2008), *SHELXL2014/7* (Sheldrick, 2015), *TITAN* (Hunter & Simpson, 1999), *Mercury* (Macrae *et al.*, 2008), *enCIFer* (Allen *et al.*, 2004), *PLATON* (Spek, 2009) and *publCIF* (Westrip 2010).

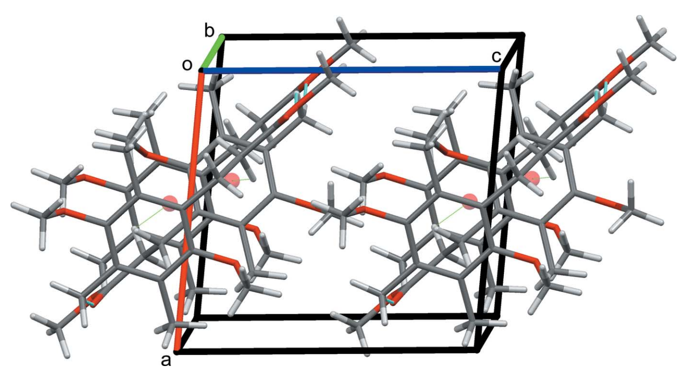


Figure 3
Overall packing of the title compound viewed along the *b*-axis direction.

(Goswami *et al.*, 2017). Crystals for this study were obtained by slow crystallization of the pure liquid at room temperature.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. With no heavy atoms in the molecule, the absolute structure could not be determined reliably.

Acknowledgements

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Funding information

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full crystallographic data

IUCrData (2017). **2**, x170500 [https://doi.org/10.1107/S2414314617005004]

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Methyl 3-(2,5-dimethoxy-3,4,6-trimethylphenyl)propanoate

Crystal data

$C_{15}H_{22}O_4$
M_r = 266.32
Monoclinic, $P2_1$
a = 8.7453 (13) Å
b = 8.6888 (10) Å
c = 9.2511 (11) Å
β = 95.936 (8)°
V = 699.19 (16) Å^3
Z = 2
F(000) = 288
D_x = 1.265 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1870 reflections
θ = 2.3–22.9°
μ = 0.09 mm^{-1}
T = 92 K
Needle, colourless
0.42 × 0.08 × 0.07 mm

Data collection

Bruker APEXII CCD area detector diffractometer
Radiation source: fine-focus sealed tube
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2013)
T_{min} = 0.660, T_{max} = 0.745
5680 measured reflections
1929 independent reflections
1781 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 23.0°, θ_{min} = 3.3°
$h = -9\rightarrow9$
$k = -9\rightarrow9$
$l = -9\rightarrow10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.098
S = 1.09
1929 reflections
178 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.026P)^2 + 0.2556P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Absolute structure: Flack x determined using 713 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ Parsons *et al.* (2013)
Absolute structure parameter: 0.4 (10)

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	0.5363 (5)	0.2874 (4)	1.0124 (4)	0.0200 (10)
C2	0.3827 (5)	0.3202 (5)	0.9690 (4)	0.0182 (9)
O2	0.3348 (3)	0.3264 (3)	0.8198 (3)	0.0230 (7)
C21	0.2666 (6)	0.1845 (5)	0.7645 (5)	0.0293 (12)
H21A	0.3403	0.1003	0.7840	0.044*
H21B	0.2389	0.1936	0.6595	0.044*
H21C	0.1741	0.1631	0.8125	0.044*
C3	0.2758 (5)	0.3498 (4)	1.0677 (4)	0.0187 (10)
C31	0.1112 (5)	0.3877 (6)	1.0153 (5)	0.0264 (10)
H31A	0.1033	0.4137	0.9117	0.040*
H31B	0.0773	0.4755	1.0702	0.040*
H31C	0.0459	0.2985	1.0298	0.040*
C4	0.3252 (5)	0.3437 (4)	1.2163 (4)	0.0197 (10)
C41	0.2135 (5)	0.3755 (5)	1.3269 (4)	0.0236 (10)
H41A	0.2616	0.3490	1.4242	0.035*
H41B	0.1207	0.3132	1.3043	0.035*
H41C	0.1859	0.4848	1.3241	0.035*
C5	0.4781 (5)	0.3086 (5)	1.2598 (4)	0.0187 (10)
O5	0.5276 (4)	0.3041 (3)	1.4082 (3)	0.0249 (8)
C51	0.5258 (6)	0.1493 (5)	1.4649 (5)	0.0298 (12)
H51A	0.4198	0.1112	1.4571	0.045*
H51B	0.5684	0.1493	1.5672	0.045*
H51C	0.5880	0.0824	1.4090	0.045*
C6	0.5844 (5)	0.2821 (4)	1.1611 (4)	0.0199 (10)
C61	0.7503 (5)	0.2498 (6)	1.2148 (5)	0.0268 (11)
H6A	0.7664	0.2667	1.3200	0.040*
H61B	0.8171	0.3190	1.1661	0.040*
H61C	0.7749	0.1428	1.1930	0.040*
C7	0.6461 (5)	0.2592 (5)	0.8990 (5)	0.0213 (10)
H7A	0.5929	0.1994	0.8178	0.026*
H7B	0.7341	0.1972	0.9425	0.026*
C8	0.7060 (5)	0.4088 (5)	0.8402 (5)	0.0222 (10)
H8A	0.6181	0.4699	0.7951	0.027*
H8B	0.7573	0.4695	0.9219	0.027*
C9	0.8177 (5)	0.3819 (5)	0.7297 (4)	0.0209 (10)
O9	0.8613 (4)	0.2584 (3)	0.6916 (3)	0.0284 (8)
O91	0.8665 (4)	0.5155 (3)	0.6762 (3)	0.0253 (8)
C91	0.9723 (6)	0.4994 (5)	0.5678 (5)	0.0262 (11)
H91A	1.0648	0.4454	0.6099	0.039*
H91B	1.0007	0.6016	0.5345	0.039*
H91C	0.9235	0.4403	0.4854	0.039*

Atomic displacement parameters (Å[2])

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.023 (3)	0.015 (2)	0.023 (2)	−0.0006 (19)	0.007 (2)	0.0047 (19)
C2	0.020 (2)	0.0145 (19)	0.020 (2)	−0.002 (2)	0.0022 (18)	0.0028 (19)
O2	0.0250 (18)	0.0244 (16)	0.0191 (16)	−0.0028 (14)	0.0005 (13)	0.0022 (13)
C21	0.031 (3)	0.029 (3)	0.028 (3)	−0.002 (2)	0.001 (2)	−0.001 (2)
C3	0.017 (2)	0.014 (2)	0.025 (2)	−0.0024 (18)	0.0051 (19)	0.0043 (17)
C31	0.022 (3)	0.027 (2)	0.030 (3)	0.002 (2)	0.005 (2)	0.002 (2)
C4	0.020 (2)	0.014 (2)	0.026 (2)	0.0007 (18)	0.009 (2)	0.0005 (18)
C41	0.023 (3)	0.021 (2)	0.029 (2)	0.004 (2)	0.010 (2)	−0.002 (2)
C5	0.024 (3)	0.014 (2)	0.018 (2)	−0.002 (2)	0.0034 (19)	0.0021 (19)
O5	0.032 (2)	0.0235 (16)	0.0185 (16)	0.0000 (15)	0.0019 (14)	−0.0024 (14)
C51	0.037 (3)	0.030 (3)	0.022 (3)	−0.001 (2)	0.000 (2)	0.002 (2)
C6	0.019 (3)	0.017 (2)	0.023 (2)	−0.0027 (19)	−0.002 (2)	−0.0019 (18)
C61	0.026 (3)	0.030 (2)	0.024 (2)	−0.001 (2)	0.001 (2)	0.000 (2)
C7	0.021 (3)	0.020 (2)	0.024 (2)	−0.0018 (19)	0.006 (2)	0.0014 (18)
C8	0.026 (3)	0.018 (2)	0.024 (2)	0.000 (2)	0.008 (2)	0.0035 (19)
C9	0.018 (2)	0.022 (2)	0.022 (2)	−0.003 (2)	0.0018 (19)	0.001 (2)
O9	0.033 (2)	0.0203 (19)	0.0343 (19)	−0.0004 (15)	0.0155 (16)	−0.0031 (14)
O91	0.030 (2)	0.0189 (17)	0.0286 (18)	0.0002 (14)	0.0135 (15)	0.0040 (14)
C91	0.030 (3)	0.027 (2)	0.024 (2)	0.002 (2)	0.011 (2)	0.002 (2)

Geometric parameters (Å, º)

C1—C2	1.391 (6)	O5—C51	1.444 (5)
C1—C6	1.397 (6)	C51—H51A	0.9800
C1—C7	1.513 (6)	C51—H51B	0.9800
C2—C3	1.397 (6)	C51—H51C	0.9800
C2—O2	1.402 (5)	C6—C61	1.511 (6)
O2—C21	1.440 (5)	C61—H6A	0.9800
C21—H21A	0.9800	C61—H61B	0.9800
C21—H21B	0.9800	C61—H61C	0.9800
C21—H21C	0.9800	C7—C8	1.522 (6)
C3—C4	1.399 (6)	C7—H7A	0.9900
C3—C31	1.507 (6)	C7—H7B	0.9900
C31—H31A	0.9800	C8—C9	1.503 (6)
C31—H31B	0.9800	C8—H8A	0.9900
C31—H31C	0.9800	C8—H8B	0.9900
C4—C5	1.390 (6)	C9—O9	1.203 (5)
C4—C41	1.511 (6)	C9—O91	1.348 (5)
C41—H41A	0.9800	O91—C91	1.440 (5)
C41—H41B	0.9800	C91—H91A	0.9800
C41—H41C	0.9800	C91—H91B	0.9800
C5—C6	1.388 (6)	C91—H91C	0.9800
C5—O5	1.397 (5)		
C2—C1—C6	118.3 (4)	O5—C51—H51B	109.5

C2—C1—C7	119.8 (4)	H51A—C51—H51B	109.5
C6—C1—C7	121.9 (4)	O5—C51—H51C	109.5
C1—C2—C3	122.7 (4)	H51A—C51—H51C	109.5
C1—C2—O2	118.2 (4)	H51B—C51—H51C	109.5
C3—C2—O2	119.0 (4)	C5—C6—C1	119.3 (4)
C2—O2—C21	112.8 (3)	C5—C6—C61	120.0 (4)
O2—C21—H21A	109.5	C1—C6—C61	120.7 (4)
O2—C21—H21B	109.5	C6—C61—H6A	109.5
H21A—C21—H21B	109.5	C6—C61—H61B	109.5
O2—C21—H21C	109.5	H6A—C61—H61B	109.5
H21A—C21—H21C	109.5	C6—C61—H61C	109.5
H21B—C21—H21C	109.5	H6A—C61—H61C	109.5
C2—C3—C4	118.4 (4)	H61B—C61—H61C	109.5
C2—C3—C31	120.8 (4)	C1—C7—C8	112.1 (3)
C4—C3—C31	120.8 (4)	C1—C7—H7A	109.2
C3—C31—H31A	109.5	C8—C7—H7A	109.2
C3—C31—H31B	109.5	C1—C7—H7B	109.2
H31A—C31—H31B	109.5	C8—C7—H7B	109.2
C3—C31—H31C	109.5	H7A—C7—H7B	107.9
H31A—C31—H31C	109.5	C9—C8—C7	112.5 (4)
H31B—C31—H31C	109.5	C9—C8—H8A	109.1
C5—C4—C3	118.9 (4)	C7—C8—H8A	109.1
C5—C4—C41	120.9 (4)	C9—C8—H8B	109.1
C3—C4—C41	120.2 (4)	C7—C8—H8B	109.1
C4—C41—H41A	109.5	H8A—C8—H8B	107.8
C4—C41—H41B	109.5	O9—C9—O91	122.6 (4)
H41A—C41—H41B	109.5	O9—C9—C8	125.8 (4)
C4—C41—H41C	109.5	O91—C9—C8	111.6 (4)
H41A—C41—H41C	109.5	C9—O91—C91	115.0 (3)
H41B—C41—H41C	109.5	O91—C91—H91A	109.5
C6—C5—C4	122.3 (4)	O91—C91—H91B	109.5
C6—C5—O5	118.8 (4)	H91A—C91—H91B	109.5
C4—C5—O5	118.8 (4)	O91—C91—H91C	109.5
C5—O5—C51	111.6 (3)	H91A—C91—H91C	109.5
O5—C51—H51A	109.5	H91B—C91—H91C	109.5

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C1–C6 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C41—H41*B*···O91^{i}	0.98	2.59	3.205 (6)	121
C41—H41*C*···O9^{ii}	0.98	2.41	3.392 (5)	175
C8—H8*A*···*Cg*^{ii}	0.99	3.15	3.763 (6)	121

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