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Methyl 2,6-dimethoxybenzoate

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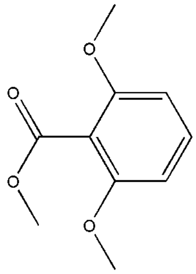
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.076; data-to-parameter ratio = 15.6.

The title compound, $C_{10}H_{12}O_4$, is an important intermediate for the synthesis of biologically active heterocyclic compounds. The planar ester group is oriented with respect to the benzene ring at a dihedral angle of 81.46 (3)°.

Related literature

For general background, see: Qadeer *et al.* (2007); Zheng *et al.* (2003); Al-Talib *et al.* (1990); Yousif *et al.* (1986); Ahmad *et al.* (2001); Al-Soud *et al.* (2004); El-Emam *et al.* (2004); Weinstock *et al.* (1991); Bruno *et al.* (2004).



Experimental

Crystal data

$C_{10}H_{12}O_4$
M_r = 196.20
Orthorhombic, $Pbca$
a = 7.2306 (9) Å
b = 14.1058 (17) Å
c = 19.403 (2) Å
V = 1978.9 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 (2) K
0.40 × 0.30 × 0.30 mm

Data collection

Bruker APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.960, T_{max} = 0.970
10579 measured reflections
2033 independent reflections
1271 reflections with $I > 2\sigma(I)$
R_{int} = 0.064

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.076
S = 0.85
2033 reflections
130 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2257).

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supplementary materials

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Comment

The title compound, (I), is a lachrymator and a drug intermediate. It is widely employed in synthetic organic chemistry for example; 2,6-dimehthoxybenzohydr- azide (Qadeer *et al.*, 2007), 2,5-disubstituted-1,3,4-oxadiazoles (Zheng *et al.*, 2003; Al-Talib *et al.*, 1990) and 5-substituted-2-mercapto-1,3,4-oxadiazoles (Yousif *et al.*, 1986; Ahmad *et al.*, 2001; Al-Soud *et al.*, 2004; El-Emam *et al.*, 2004). In addition, methyl 4-(bromomethyl)benzoate has been used in the synthesis of 1-(carboxybenzyl)imidazole-5-acrylic acids, which are potent and selective angiotensin II receptor antagonists (Weinstock *et al.*, 1991). In view of the versatility of these compounds, we have synthesized the title compound, (I), and reported its crystal structure.

The title molecule (Fig. 1) shows no unusual features when compared with the Mogul norms (Bruno *et al.*, 2004). The dihedral angle between the planar ester group and benzene ring is 81.46 (3)°.

As can be seen from the packing diagram (Fig. 2), the molecules of (I) are stacked along the *a* axis and elongated along the *b* axis. Dipole-dipole and van der Waals interactions are effective in the molecular packing.

Experimental

For the preparation of (I), a mixture of methyl-2,6-dimethoxybenzoate (1.96 g, 10 mmol) and hydrazine hydrate (80%, 15 ml) in absolute ethanol (50 ml) was refluxed for 5 h at 413–423 K. The excess solvent was removed by distillation. The solid residue was filtered off, washed with water and recrystallized from ethanol (30%) to give the title compound (yield: 91%). Colorless single crystals of (I) were obtained by slow evaporation of an ethanol solution at room temperature.

Refinement

H atoms were positioned geometrically, with C—H = 0.95 and 0.98 Å for aromatic and methyl H atoms, respectively, and constrained to ride on their parent atoms, with $U_{\rm iso}({\rm H}) = xU_{\rm eq}({\rm C},N)$, where $x = 1.5$ for methyl H, and $x = 1.2$ for aromatic H atoms.

Figures

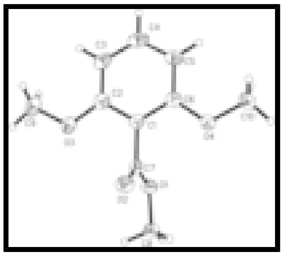


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

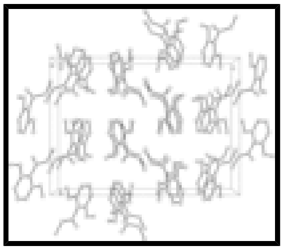


Fig. 2. A packing diagram for (I).

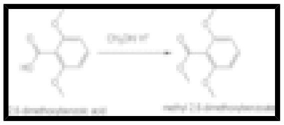


Fig. 3. The synthesis route for the formation of the title compound.

methyl 2,6-dimethoxybenzoate

Crystal data

$C_{10}H_{12}O_4$

M_r = 196.20

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

a = 7.2306 (9) Å

b = 14.1058 (17) Å

c = 19.403 (2) Å

V = 1978.9 (4) $Å^3$

Z = 8

F_{000} = 832

D_x = 1.317 Mg m^{-3}

Melting point: 450(1) K

Mo *K*α radiation
λ = 0.71073 Å

Cell parameters from 1609 reflections

θ = 2.9–23.6°

μ = 0.10 mm^{-1}

T = 100 (2) K

Block, colourless

0.40 × 0.30 × 0.30 mm

Data collection

Bruker APEXII
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 100(2) K

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

T_{min} = 0.960, T_{max} = 0.970

10579 measured reflections

2033 independent reflections

1271 reflections with $I > 2\sigma(I)$

R_{int} = 0.064

θ_{max} = 26.4°

θ_{min} = 2.1°

h = −8→9

k = −17→15

l = −24→22

Standard reflections: none

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.037

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$wR(F^2) = 0.076$	$w = 1/[\sigma^2(F_o^2) + (0.0303P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.85$	$(\Delta/\sigma)_{max} < 0.001$
2033 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
130 parameters	$\Delta\rho_{min} = -0.17$ 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}
O1	0.32641 (14)	0.23587 (7)	0.03432 (5)	0.0274 (3)
O2	0.52183 (14)	0.26194 (7)	0.12196 (5)	0.0305 (3)
O3	0.57947 (15)	0.04433 (7)	0.08234 (5)	0.0343 (3)
O4	0.07719 (14)	0.21697 (7)	0.16359 (5)	0.0272 (3)
C1	0.3235 (2)	0.12555 (11)	0.12428 (7)	0.0230 (4)
C2	0.4173 (2)	0.03933 (11)	0.11826 (7)	0.0276 (4)
C3	0.3440 (2)	−0.04231 (11)	0.14695 (8)	0.0323 (4)
H3	0.4061	−0.1013	0.1422	0.039*
C4	0.1787 (2)	−0.03647 (12)	0.18270 (8)	0.0344 (4)
H4	0.1288	−0.0924	0.2026	0.041*
C5	0.0838 (2)	0.04769 (11)	0.19046 (7)	0.0303 (4)
H5	−0.0287	0.0500	0.2157	0.036*
C6	0.1568 (2)	0.12911 (11)	0.16044 (7)	0.0245 (4)
C7	0.4041 (2)	0.21447 (10)	0.09467 (7)	0.0226 (4)
C8	0.3873 (2)	0.32382 (11)	0.00241 (8)	0.0297 (4)
H8A	0.3664	0.3767	0.0343	0.045*
H8B	0.3171	0.3346	−0.0401	0.045*
H8C	0.5194	0.3195	−0.0085	0.045*
C9	0.6844 (2)	−0.04143 (12)	0.07533 (9)	0.0462 (5)
H9A	0.7218	−0.0639	0.1210	0.069*
H9B	0.7948	−0.0291	0.0474	0.069*
H9C	0.6084	−0.0899	0.0528	0.069*
C10	−0.0943 (2)	0.22482 (12)	0.20085 (8)	0.0341 (4)
H10A	−0.1840	0.1796	0.1821	0.051*
H10B	−0.1429	0.2894	0.1962	0.051*

H10C	−0.0728	0.2109	0.2497	0.051*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0275 (6)	0.0282 (7)	0.0267 (5)	−0.0047 (5)	−0.0031 (5)	0.0051 (5)
O2	0.0282 (6)	0.0268 (7)	0.0364 (6)	−0.0056 (5)	−0.0072 (5)	0.0012 (5)
O3	0.0338 (7)	0.0246 (7)	0.0446 (7)	0.0085 (5)	0.0067 (6)	0.0003 (5)
O4	0.0242 (6)	0.0274 (7)	0.0301 (6)	0.0005 (5)	0.0052 (5)	0.0022 (5)
C1	0.0247 (9)	0.0221 (9)	0.0221 (8)	−0.0016 (7)	−0.0039 (7)	−0.0006 (6)
C2	0.0288 (10)	0.0266 (10)	0.0273 (8)	−0.0016 (8)	−0.0045 (8)	−0.0004 (7)
C3	0.0395 (11)	0.0221 (10)	0.0352 (9)	0.0000 (8)	−0.0078 (8)	−0.0005 (7)
C4	0.0421 (11)	0.0263 (10)	0.0350 (9)	−0.0121 (8)	−0.0094 (8)	0.0057 (8)
C5	0.0300 (10)	0.0320 (10)	0.0289 (8)	−0.0085 (8)	−0.0013 (7)	0.0037 (7)
C6	0.0257 (9)	0.0242 (9)	0.0237 (8)	−0.0030 (7)	−0.0047 (7)	−0.0006 (7)
C7	0.0198 (8)	0.0221 (9)	0.0260 (8)	0.0044 (7)	0.0012 (7)	−0.0039 (7)
C8	0.0292 (10)	0.0279 (10)	0.0320 (9)	0.0000 (8)	0.0035 (7)	0.0091 (7)
C9	0.0456 (12)	0.0309 (11)	0.0622 (12)	0.0143 (9)	0.0069 (10)	−0.0013 (9)
C10	0.0269 (9)	0.0396 (11)	0.0358 (9)	0.0027 (8)	0.0091 (7)	0.0041 (8)

Geometric parameters (Å, °)

O1—C7	1.3335 (16)	C4—C5	1.379 (2)
O1—C8	1.4548 (17)	C4—H4	0.9500
O2—C7	1.2055 (17)	C5—C6	1.392 (2)
O3—C2	1.3658 (18)	C5—H5	0.9500
O3—C9	1.4345 (17)	C8—H8A	0.9800
O4—C6	1.3680 (17)	C8—H8B	0.9800
O4—C10	1.4397 (17)	C8—H8C	0.9800
C1—C6	1.3951 (19)	C9—H9A	0.9800
C1—C2	1.398 (2)	C9—H9B	0.9800
C1—C7	1.498 (2)	C9—H9C	0.9800
C2—C3	1.384 (2)	C10—H10A	0.9800
C3—C4	1.385 (2)	C10—H10B	0.9800
C3—H3	0.9500	C10—H10C	0.9800

C7—O1—C8	116.06 (12)	O2—C7—O1	123.88 (14)
C2—O3—C9	117.32 (12)	O2—C7—C1	124.86 (13)
C6—O4—C10	117.04 (12)	O1—C7—C1	111.25 (13)
C6—C1—C2	119.51 (14)	O1—C8—H8A	109.5
C6—C1—C7	119.94 (13)	O1—C8—H8B	109.5
C2—C1—C7	120.50 (14)	H8A—C8—H8B	109.5
O3—C2—C3	125.22 (15)	O1—C8—H8C	109.5
O3—C2—C1	114.49 (14)	H8A—C8—H8C	109.5
C3—C2—C1	120.29 (15)	H8B—C8—H8C	109.5
C4—C3—C2	118.84 (16)	O3—C9—H9A	109.5
C4—C3—H3	120.6	O3—C9—H9B	109.5
C2—C3—H3	120.6	H9A—C9—H9B	109.5
C5—C4—C3	122.38 (16)	O3—C9—H9C	109.5

C5—C4—H4	118.8	H9A—C9—H9C	109.5
C3—C4—H4	118.8	H9B—C9—H9C	109.5
C4—C5—C6	118.39 (16)	O4—C10—H10A	109.5
C4—C5—H5	120.8	O4—C10—H10B	109.5
C6—C5—H5	120.8	H10A—C10—H10B	109.5
O4—C6—C5	124.68 (14)	O4—C10—H10C	109.5
O4—C6—C1	114.74 (13)	H10A—C10—H10C	109.5
C5—C6—C1	120.57 (15)	H10B—C10—H10C	109.5

C9—O3—C2—C3	−1.7 (2)	C4—C5—C6—O4	179.56 (13)
C9—O3—C2—C1	178.89 (13)	C4—C5—C6—C1	−0.9 (2)
C6—C1—C2—O3	−179.47 (12)	C2—C1—C6—O4	179.59 (13)
C7—C1—C2—O3	−1.9 (2)	C7—C1—C6—O4	2.00 (19)
C6—C1—C2—C3	1.1 (2)	C2—C1—C6—C5	0.0 (2)
C7—C1—C2—C3	178.67 (13)	C7—C1—C6—C5	−177.57 (13)
O3—C2—C3—C4	179.34 (13)	C8—O1—C7—O2	−2.0 (2)
C1—C2—C3—C4	−1.3 (2)	C8—O1—C7—C1	176.88 (12)
C2—C3—C4—C5	0.4 (2)	C6—C1—C7—O2	96.75 (18)
C3—C4—C5—C6	0.7 (2)	C2—C1—C7—O2	−80.82 (19)
C10—O4—C6—C5	0.1 (2)	C6—C1—C7—O1	−82.08 (16)
C10—O4—C6—C1	−179.47 (12)	C2—C1—C7—O1	100.35 (16)

Fig. 1

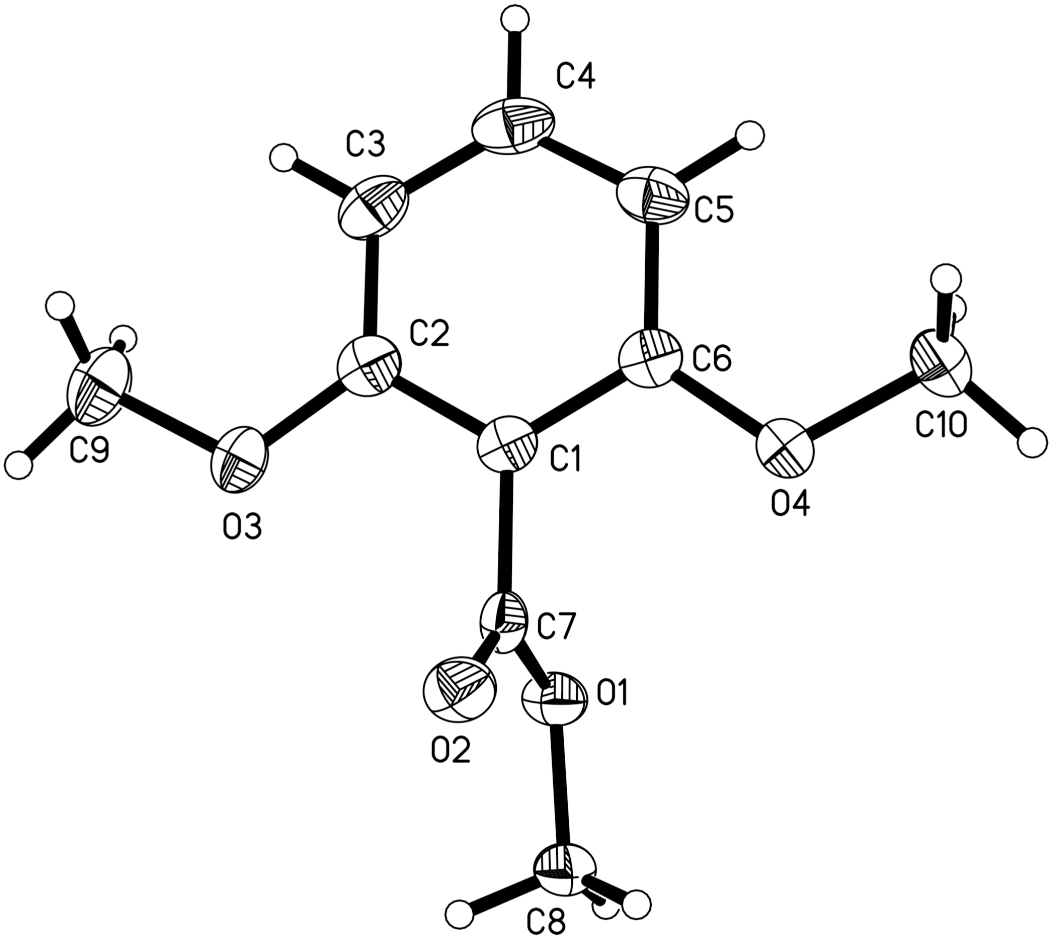


Fig. 2

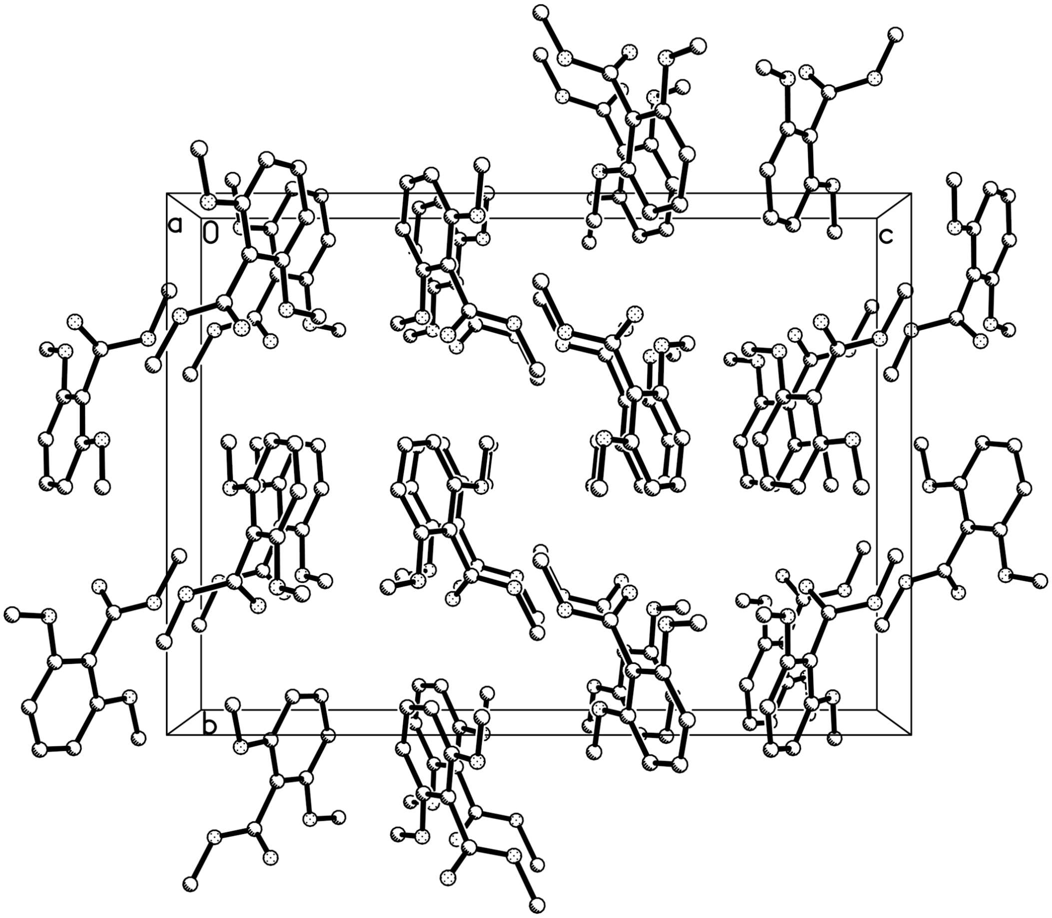
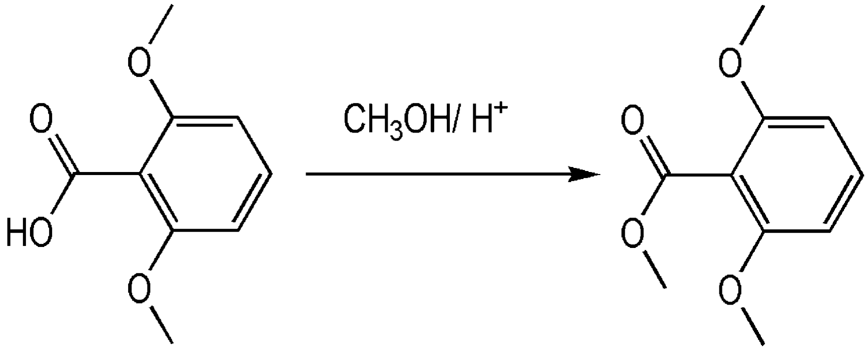


Fig. 3



2,6-dimethoxybenzoic acid

methyl 2,6-dimethoxybenzoate