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Pseudosymmetric (*E*)-methyl 2-oxo-4-phenylbut-3-enoate

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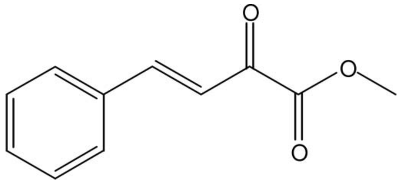
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.005 Å; R factor = 0.047; wR factor = 0.130; data-to-parameter ratio = 7.6.

The structure of the title compound, $C_{11}H_{10}O_3$, shows pseudosymmetry. Since the molecule is nearly planar and all non-H atoms lie close to an approximate mirror plane perpendicular to the crystallographic *b* axis, the structure could almost (85%) be described in space group $P2_1/m$. However, the correct space group is $P2_1$.

Related literature

For related literature, see: Dujardin *et al.* (2001); Jørgensen (2003); Jensen *et al.* (2001); Spek (2003).



Experimental

Crystal data

$C_{11}H_{10}O_3$
M_r = 190.19
Monoclinic, $P2_1$
a = 7.1114 (10) Å
b = 6.5381 (6) Å
c = 10.5106 (15) Å
β = 99.527 (11)°
V = 481.95 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 173 (2) K
0.34 × 0.31 × 0.22 mm

Data collection

Stoe IPDS II two-circle diffractometer
Absorption correction: none
4453 measured reflections
977 independent reflections
937 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.130
S = 1.19
977 reflections
128 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{\rm max}$ = 0.26 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.35 e Å^{-3}

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2168).

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supplementary materials

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Pseudosymmetric (*E*)-methyl 2-oxo-4-phenylbut-3-enoate

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Comment

Unsaturated α-ketoesters are of great synthetic interest and are widely used as substrates for enantioselective 1,4-additions. For example, Jensen *et al.* (2001) could develop an highly enantioselective Friedel-Crafts type addition of different electron rich arenes to (*E*)-methyl 2-oxo-4-phenylbut-3-enoate (Jensen *et al.*, 2001; Jørgensen, 2003). The title compound, (I), was synthesized by an Lewis-Acid catalysed aldol condensation of benzaldehyde and methylpyruvate.

Geometric parameters of (I) are in the normal ranges. The C—C double bond is *trans* configured and the two carbonyl groups are also *trans* to each other (Fig. 1). The molecule is almost planar, the r.m.s. deviation for all non-H atoms being 0.208 Å. The structure of (I) shows pseudo-symmetry. The packing diagram (Fig. 2) reveals that the non-H atoms almost lie on a mirror plane perpendicular to the crystallographic *b* axis. A check for missed symmetry with *PLATON* (Spek, 2003) suggests space group $P2_1/m$ with the higher symmetry being fulfilled by 85% of the atoms. However, the correct space group is $P2_1$.

Experimental

The title compound was synthesized by an Lewis-Acid catalysed aldol condensation of benzaldehyde and methylpyruvate (Dujardin *et al.*, 2001).

Refinement

In the absence of anomalous scatterers Friedel pairs had been merged prior to refinement. The absolute structure was arbitrarily assigned. H atoms were found in a difference map, but they were refined using a riding model with C—H = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ or C—H = 0.98 Å and $U_{iso}(H) = 1.5U_{eq}(C_{methyl})$. The methyl group was allowed to rotate but not to tip.

Figures

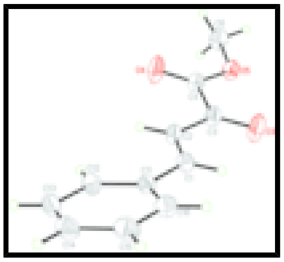

Fig. 1. Perspective view of (I) with the atom numbering. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level.

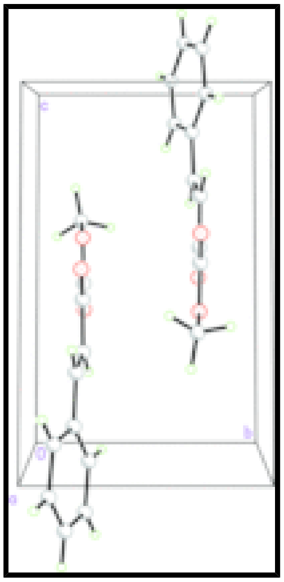

Fig. 2. Packing diagram of (I), viewed onto the *bc* plane.

(*E*)-Methyl 2-oxo-4-phenylbut-3-enoate

Crystal data

$C_{11}H_{10}O_3$

$M_r = 190.19$

Monoclinic, $P2_1$

Hall symbol: P 2yb

$a = 7.1114$ (10) Å

$b = 6.5381$ (6) Å

$c = 10.5106$ (15) Å

$\beta = 99.527$ (11)°

$V = 481.95$ (11) Å^3

$Z = 2$

$F_{000} = 200$

$D_x = 1.311$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 5679 reflections

$\theta = 3.8$–25.2°

$\mu = 0.10$ mm^{-1}

$T = 173$ (2) K

Plate, colourless

0.34 × 0.31 × 0.22 mm

Data collection

Stoe IPDS II two-circle-diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 173(2)$ K

ω scans

Absorption correction: none

4453 measured reflections

977 independent reflections

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

$R_{\rm int} = 0.035$

$\theta_{\rm max} = 25.6$°

$\theta_{\rm min} = 3.7$°

$h = -8 \rightarrow 8$

$k = -7 \rightarrow 7$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.047$	$w = 1/[\sigma^2(F_o^2) + (0.043P)^2 + 0.4561P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.130$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.19$	$\Delta\rho_{max} = 0.26$ e Å^{-3}
977 reflections	$\Delta\rho_{min} = -0.35$ e Å^{-3}
128 parameters	Extinction correction: none
1 restraint	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

Special details

Experimental. ;

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 > 2sigma(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*	$U_{iso}*/U_{eq}$
C1	0.6767 (5)	0.2097 (7)	0.2504 (3)	0.0233 (8)
H1	0.7866	0.1968	0.3149	0.028*
C2	0.5092 (5)	0.2322 (8)	0.2914 (3)	0.0242 (8)
H2	0.3959	0.2509	0.2305	0.029*
C3	0.4997 (5)	0.2281 (8)	0.4302 (3)	0.0257 (8)
O3	0.6377 (3)	0.2281 (8)	0.5165 (2)	0.0438 (9)
C4	0.2961 (5)	0.2250 (8)	0.4643 (3)	0.0244 (8)
O4	0.1529 (3)	0.2226 (8)	0.3846 (2)	0.0442 (9)
O5	0.3006 (3)	0.2202 (6)	0.5911 (2)	0.0286 (7)
C6	0.1147 (5)	0.2062 (10)	0.6332 (3)	0.0339 (10)
H6A	0.0452	0.3348	0.6139	0.051*
H6B	0.1333	0.1806	0.7263	0.051*
H6C	0.0414	0.0936	0.5876	0.051*
C11	0.7074 (5)	0.2029 (7)	0.1152 (3)	0.0229 (8)
C12	0.5656 (5)	0.2600 (6)	0.0122 (3)	0.0261 (9)

H12	0.4461	0.3088	0.0287	0.031*
C13	0.5998 (5)	0.2451 (8)	−0.1155 (3)	0.0303 (9)
H13	0.5044	0.2851	−0.1853	0.036*
C14	0.7741 (6)	0.1715 (6)	−0.1390 (4)	0.0309 (10)
H14	0.7969	0.1603	−0.2253	0.037*
C15	0.9150 (6)	0.1141 (8)	−0.0380 (4)	0.0323 (10)
H15	1.0338	0.0643	−0.0551	0.039*
C16	0.8823 (5)	0.1295 (7)	0.0884 (4)	0.0276 (9)
H16	0.9793	0.0899	0.1574	0.033*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0243 (15)	0.0251 (19)	0.0206 (15)	0.003 (2)	0.0041 (12)	−0.0008 (18)
C2	0.0222 (15)	0.031 (2)	0.0187 (15)	0.002 (2)	0.0009 (12)	−0.001 (2)
C3	0.0219 (15)	0.034 (2)	0.0210 (15)	0.001 (2)	0.0029 (12)	−0.002 (2)
O3	0.0215 (12)	0.087 (3)	0.0219 (12)	−0.003 (2)	−0.0003 (9)	−0.001 (2)
C4	0.0227 (15)	0.029 (2)	0.0222 (15)	0.002 (2)	0.0051 (12)	−0.003 (2)
O4	0.0223 (12)	0.081 (2)	0.0268 (13)	0.003 (2)	−0.0029 (10)	0.000 (2)
O5	0.0228 (11)	0.0454 (17)	0.0186 (11)	−0.0030 (18)	0.0068 (9)	−0.0006 (17)
C6	0.0232 (17)	0.053 (3)	0.0283 (18)	0.001 (2)	0.0117 (14)	0.002 (2)
C11	0.0257 (16)	0.023 (2)	0.0195 (16)	−0.0025 (19)	0.0037 (13)	0.0008 (18)
C12	0.0275 (17)	0.026 (2)	0.0246 (17)	0.0041 (18)	0.0037 (14)	0.0006 (17)
C13	0.0368 (19)	0.032 (2)	0.0209 (16)	0.000 (2)	0.0013 (14)	0.004 (2)
C14	0.040 (2)	0.031 (3)	0.0235 (17)	−0.0031 (19)	0.0114 (16)	−0.0015 (17)
C15	0.034 (2)	0.035 (2)	0.030 (2)	−0.001 (2)	0.0132 (17)	0.0004 (19)
C16	0.0253 (18)	0.030 (2)	0.0283 (19)	0.0013 (18)	0.0078 (15)	−0.0005 (18)

Geometric parameters (Å, °)

C1—C2	1.340 (5)	C6—H6C	0.9800
C1—C11	1.473 (4)	C11—C12	1.403 (5)
C1—H1	0.9500	C11—C16	1.405 (5)
C2—C3	1.472 (4)	C12—C13	1.407 (5)
C2—H2	0.9500	C12—H12	0.9500
C3—O3	1.221 (4)	C13—C14	1.389 (6)
C3—C4	1.548 (4)	C13—H13	0.9500
C4—O4	1.207 (4)	C14—C15	1.386 (6)
C4—O5	1.328 (4)	C14—H14	0.9500
O5—C6	1.464 (4)	C15—C16	1.390 (5)
C6—H6A	0.9800	C15—H15	0.9500
C6—H6B	0.9800	C16—H16	0.9500
C2—C1—C11	126.5 (3)	C12—C11—C16	118.9 (3)
C2—C1—H1	116.7	C12—C11—C1	122.3 (3)
C11—C1—H1	116.7	C16—C11—C1	118.8 (3)
C1—C2—C3	120.4 (3)	C11—C12—C13	120.2 (3)
C1—C2—H2	119.8	C11—C12—H12	119.9
C3—C2—H2	119.8	C13—C12—H12	119.9

O3—C3—C2	125.0 (3)	C14—C13—C12	119.6 (3)
O3—C3—C4	119.7 (3)	C14—C13—H13	120.2
C2—C3—C4	115.3 (3)	C12—C13—H13	120.2
O4—C4—O5	125.0 (3)	C15—C14—C13	120.7 (3)
O4—C4—C3	123.6 (3)	C15—C14—H14	119.6
O5—C4—C3	111.3 (3)	C13—C14—H14	119.6
C4—O5—C6	115.6 (3)	C14—C15—C16	119.9 (4)
O5—C6—H6A	109.5	C14—C15—H15	120.0
O5—C6—H6B	109.5	C16—C15—H15	120.0
H6A—C6—H6B	109.5	C15—C16—C11	120.7 (4)
O5—C6—H6C	109.5	C15—C16—H16	119.6
H6A—C6—H6C	109.5	C11—C16—H16	119.6
H6B—C6—H6C	109.5		

C11—C1—C2—C3	177.6 (4)	C2—C1—C11—C16	−164.6 (5)
C1—C2—C3—O3	8.6 (9)	C16—C11—C12—C13	−0.5 (6)
C1—C2—C3—C4	−171.7 (4)	C1—C11—C12—C13	−178.1 (4)
O3—C3—C4—O4	−179.2 (7)	C11—C12—C13—C14	0.7 (7)
C2—C3—C4—O4	1.2 (7)	C12—C13—C14—C15	−0.5 (7)
O3—C3—C4—O5	−0.7 (7)	C13—C14—C15—C16	0.2 (7)
C2—C3—C4—O5	179.6 (5)	C14—C15—C16—C11	0.0 (7)
O4—C4—O5—C6	1.5 (8)	C12—C11—C16—C15	0.2 (6)
C3—C4—O5—C6	−176.9 (4)	C1—C11—C16—C15	177.9 (4)
C2—C1—C11—C12	12.9 (8)		

Fig. 1

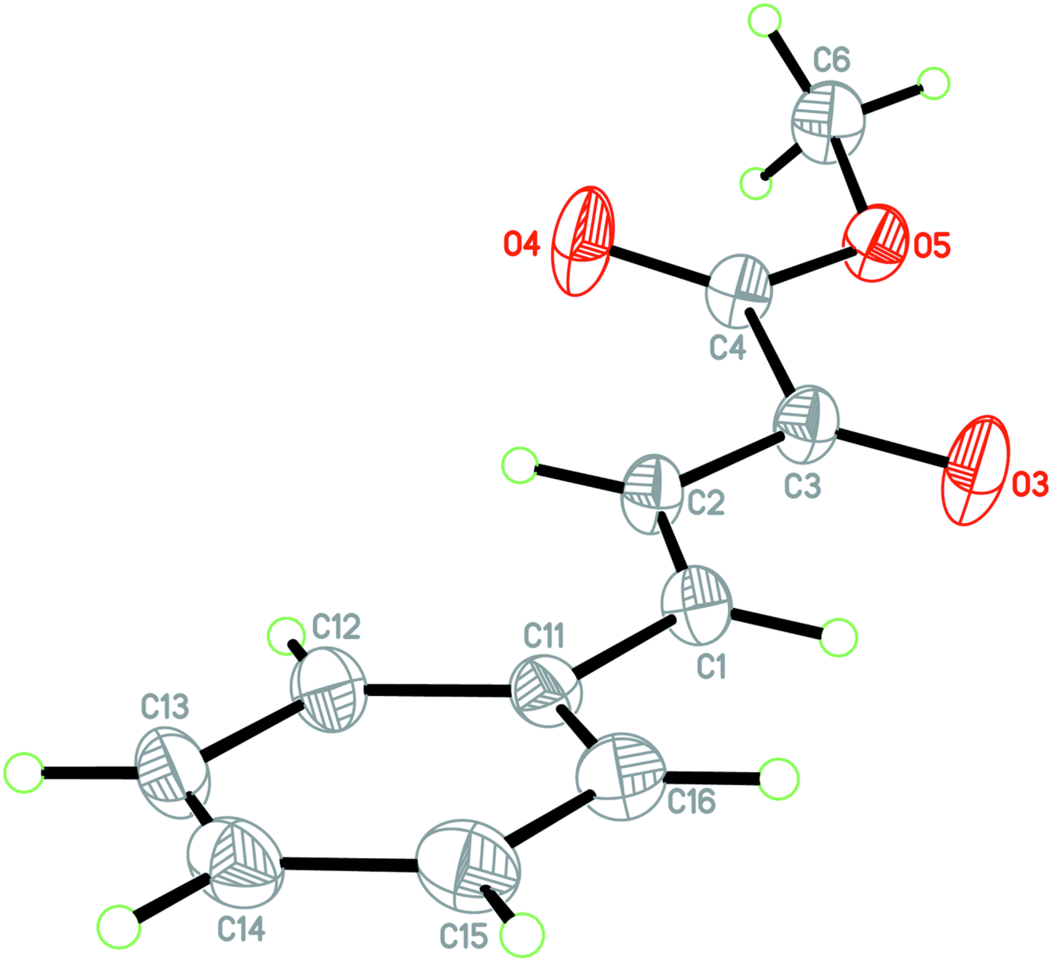

Fig. 2