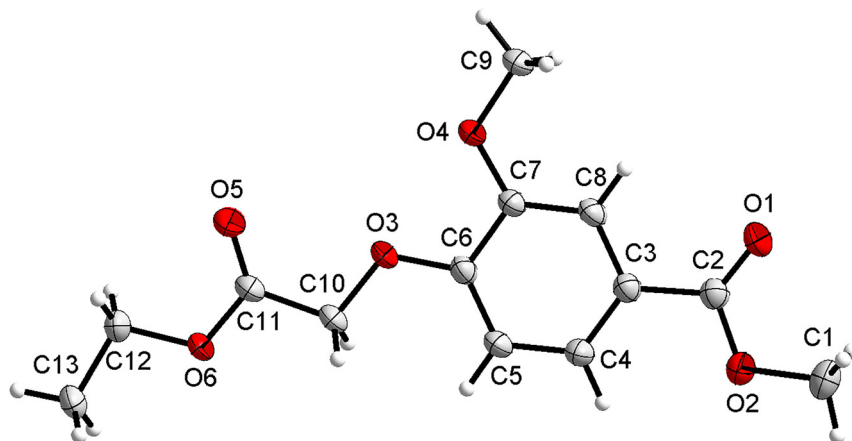


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Crystal structure of methyl 4-(2-ethoxy-2-oxoethoxy)-3-methoxybenzoate, C₁₃H₁₆O₆



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Abstract

C₁₃H₁₆O₆, monoclinic, *P*₂/c (no. 14), *a* = 12.180(3) Å, *b* = 13.813(4) Å, *c* = 7.842(2) Å, β = 93.917(4)°, *V* = 1316.2(6) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0442, *wR*_{ref}(*F*²) = 0.1375, *T* = 296(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

The mixture of methyl 4-hydroxy-3-methoxybenzoate (1.82 g, 0.01 mol), ethyl bromoacetate (2.03 g, 0.12 mol), K₂CO₃ (2.76 g, 0.02 mol) and acetone (10 mL) was reacted at 65 °C for 24 h.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.21 × 0.16 × 0.14 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.11 mm ^{−1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ _{max} , completeness:	25.5°, >99 %
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	9947, 2447, 0.044
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2σ(<i>I</i> _{obs}), 1662
<i>N</i> (<i>param</i>) _{refined} :	175
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

After the reaction completed (monitored by TLC), acetone was removed by steam distillation and extracted with ethyl acetate. The colorless product was filtered, and washed with water 3 times respectively. Yield 64.6 % (based on methyl 4-hydroxy-3-methoxybenzoate). Then crystals suitable for X-ray analysis were obtained.

2 Experimental details

All H atoms were included in calculated positions and refined as riding atoms, with C–H = 0.90–0.97 Å with *U*_{iso}(H) = 1.5 *U*_{eq}(C) for methyl H atoms and 1.2 *U*_{eq}(C) for all other H atoms.

3 Comment

Methyl vanillic acid is one of the components of *Hovenia dulcis Thunb.*, which can activate Wnt/β-catenin signaling pathway and induce osteogenic differentiation *in vitro*, and

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U _{iso} [*] /U _{eq}
C1	1.28321 (19)	0.3571 (2)	0.5413 (3)	0.0674 (7)
H1A	1.2629	0.3236	0.6417	0.101*
H1B	1.3261	0.4132	0.5743	0.101*
H1C	1.3258	0.3148	0.4744	0.101*
C2	1.12135 (17)	0.31513 (16)	0.3775 (3)	0.0477 (5)
C3	1.02163 (16)	0.35245 (14)	0.2804 (2)	0.0441 (5)
C4	1.00042 (17)	0.44978 (15)	0.2588 (3)	0.0495 (5)
H4	1.0496	0.4950	0.3079	0.059*
C5	0.90706 (17)	0.48103 (15)	0.1652 (3)	0.0485 (5)
H5	0.8937	0.5470	0.1514	0.058*
C6	0.83343 (16)	0.41457 (14)	0.0921 (2)	0.0421 (5)
C7	0.85325 (16)	0.31470 (14)	0.1161 (2)	0.0416 (5)
C8	0.94684 (16)	0.28495 (14)	0.2085 (2)	0.0440 (5)
H8	0.9606	0.2192	0.2234	0.053*
C9	0.78584 (19)	0.15443 (15)	0.0721 (3)	0.0552 (6)
H9A	0.8515	0.1315	0.0242	0.083*
H9B	0.7231	0.1212	0.0194	0.083*
H9C	0.7905	0.1423	0.1929	0.083*
C10	0.71362 (17)	0.53521 (15)	−0.0291 (3)	0.0487 (5)
H10A	0.7719	0.5674	−0.0855	0.058*
H10B	0.7063	0.5664	0.0804	0.058*
C11	0.60800 (18)	0.54243 (15)	−0.1364 (3)	0.0510 (5)
C12	0.47585 (17)	0.65324 (16)	−0.2522 (3)	0.0551 (6)
H12A	0.4154	0.6292	−0.1898	0.066*
H12B	0.4734	0.6214	−0.3627	0.066*
C13	0.46690 (18)	0.76000 (16)	−0.2746 (3)	0.0574 (6)
H13A	0.4709	0.7907	−0.1646	0.086*
H13B	0.3979	0.7755	−0.3349	0.086*
H13C	0.5261	0.7827	−0.3389	0.086*
O1	1.14375 (13)	0.23130 (12)	0.3974 (2)	0.0664 (5)
O2	1.18573 (12)	0.38656 (12)	0.4417 (2)	0.0617 (5)
O3	0.73963 (11)	0.43646 (10)	−0.00428 (18)	0.0507 (4)
O4	0.77455 (12)	0.25538 (10)	0.04221 (19)	0.0537 (4)
O5	0.55585 (19)	0.47689 (13)	−0.1954 (3)	0.1140 (9)
O6	0.58010 (11)	0.63393 (10)	−0.15752 (19)	0.0518 (4)

it is also the raw material and precursor for the synthesis of many drugs and chemical products [5–7]. Wang et al. have synthesized two 4-alkyl methyl vanillic acid derivatives [8, 9]. Wang et al. also have synthesized methyl 4-(benzyloxy)-3-methoxybenzoate [10]. However, its poor fat solubility and instability hinder its application. So it is necessary to modify their structures. We have synthesized a series of methyl vanillic acid derivatives and reported a crystal structure of methyl 4-acetoxy-3-methoxybenzoate [11].

In the molecule of the title structure bond lengths and angles are very similar to those given in the literature [11–13]. Non-hydrogen atoms in the title compound are approximately planar. The dihedral angles formed by the C3–C8 phenyl plane, the carboxylate group C2–O1–O2 plane and the

carboxylate group C11–O5–O6 plane are 1.4(1)°, 3.1(1)° and 2.3(1)°, respectively. The torsion angles of C6–C7–O4–C9, C7–C6–O3–C10, C6–O3–C10–C11 and C11–O6–C12–C13 are −175.6(2)°, 178.3(2)°, −179.6(2)° and −174.6(2)°, respectively.

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