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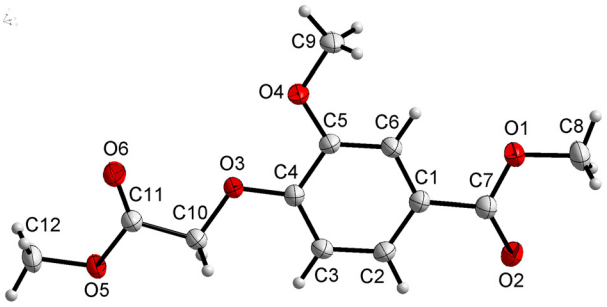
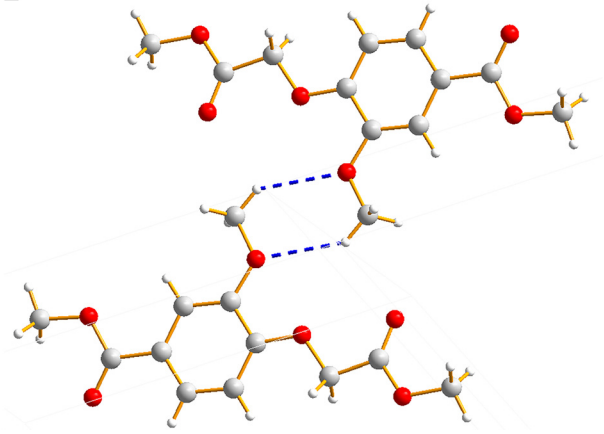
Crystal structure of methyl 3-methoxy-4-(2-methoxy-2-oxoethoxy)benzoate, $C_{12}H_{14}O_6$





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Abstract

$C_{12}H_{14}O_6$, triclinic, $P\bar{1}$ (no. 2), a = 5.3644(9) Å, b = 11.1880(18) Å, c = 11.4159(18) Å, α = 66.378(2)°, β = 79.970(2)°, γ = 78.138(2)°, V = 611.11(17) Å^3, Z = 2, $R_{gt}(F)$ = 0.0430, w$R_{ref}(F^2)$ = 0.1260, T = 296(2) K.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.20 × 0.15 × 0.13 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.11 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.5°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4757, 2270, 0.014
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 $\sigma(I_{obs})$, 1680
$N(param)_{refined}$:	167
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

Dissolve methyl 4-hydroxy-3-methoxybenzoate (1.82 g, 0.01 mol) in acetone (10 mL), add potassium carbonate (2.76 g, 0.02 mol), stir at 65 °C for half an hour, and then add methyl bromoacetate (1.86 g, 0.12 mol) to react for 24 h. After the reaction was detected by thin layer chromatography (TLC), the acetone solution was removed by steam distillation and extracted with ethyl acetate. Removal of ethyl acetate produced a colorless product. The product was filtered and washed with water (10 mL) three times yield 66.2 % (based on methyl 4-hydroxy-3-methoxybenzoate). The product was dissolved in ethyl acetate, filtered, and the filtrate was allowed to stand at room temperature. After two weeks, the crystals 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

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5644 (3)	0.51808 (16)	0.75214 (16)	0.0469 (4)
C2	0.4123 (4)	0.54780 (17)	0.65606 (17)	0.0539 (5)
H2	0.3924	0.6325	0.5931	0.065*
C3	0.2887 (4)	0.45275 (17)	0.65227 (17)	0.0536 (5)
H3	0.1855	0.4741	0.5870	0.064*
C4	0.3171 (3)	0.32663 (16)	0.74427 (16)	0.0460 (4)
C5	0.4738 (3)	0.29491 (16)	0.84266 (15)	0.0480 (4)
C6	0.5939 (3)	0.39079 (17)	0.84598 (16)	0.0503 (4)
H6	0.6958	0.3705	0.9115	0.060*
C7	0.6929 (4)	0.62365 (18)	0.75180 (18)	0.0532 (5)
C8	0.9481 (5)	0.6833 (2)	0.8628 (2)	0.0751 (6)
H8A	1.0668	0.7113	0.7872	0.113*
H8B	1.0397	0.6439	0.9376	0.113*
H8C	0.8285	0.7582	0.8678	0.113*
C9	0.6755 (4)	0.12498 (19)	1.0195 (2)	0.0718 (6)
H9A	0.8429	0.1390	0.9755	0.108*
H9B	0.6760	0.0328	1.0706	0.108*
H9C	0.6297	0.1743	1.0742	0.108*
C10	0.0558 (4)	0.25203 (18)	0.64833 (17)	0.0517 (4)
H10A	0.1598	0.2791	0.5662	0.062*
H10B	−0.0847	0.3227	0.6466	0.062*
C11	−0.0463 (4)	0.12817 (18)	0.67164 (17)	0.0530 (5)
C12	−0.3263 (5)	0.0471 (2)	0.5932 (2)	0.0799 (7)
H12A	−0.1940	−0.0248	0.5920	0.120*
H12B	−0.4209	0.0752	0.5203	0.120*
H12C	−0.4397	0.0187	0.6708	0.120*
O1	0.8114 (3)	0.58785 (13)	0.85621 (13)	0.0667 (4)
O2	0.6943 (3)	0.72961 (14)	0.66621 (15)	0.0873 (6)
O3	0.2054 (2)	0.22571 (11)	0.74901 (11)	0.0555 (4)
O4	0.4932 (3)	0.16816 (12)	0.92735 (13)	0.0696 (4)
O5	−0.2121 (3)	0.15567 (12)	0.58803 (13)	0.0676 (4)
O6	0.0125 (3)	0.02197 (14)	0.74954 (16)	0.0916 (6)

pathway and induce osteogenic differentiation *in vitro*. It is also the raw material and precursor for the synthesis of many drugs and chemical products, and possesses anti-inflammatory, antibacterial activity [5–7]. However, its poor fat solubility and instability limit its application, so it is necessary to modify its structure. Zhang et al. reported the synthesis and crystal structures of two derivatives of 4-alkyl-substituted methyl vanillic acid [8, 9]. Wang et al. synthesized a derivative of 4-benzyl-substituted methyl vanillic acid [10]. Xiong et al. also synthesized 2-methoxy-4-(methoxy-carbonyl)phenyl-2-chloro-4-fluorobenzoate [11]. Nie et al. synthesised a series of acyl-substituted methyl phenolate derivatives [12–14]. Recently, Nie et al. reported two 2-ethoxy-2-oxoethoxy substituted methyl vanillic acid and methyl coumaric acid derivatives [15, 16].

In the molecules of the title structure bond lengths and angles are very similar to those given in the literature [15–17]; upper part of the figure. In the title structure, the non hydrogen atoms in the title compound are approximately planar. The dihedral angle formed by the C1–C6 phenyl plane, the carboxlate group C7–O1–O2 plane and the carboxlate group C11–O5–O6 plane are 7.6° and 6.2°. The torsion angles of C4–C5–O4–C9, C5–C4–O3–C10 and C4–O3–C10–C11 are 171.9°, –177.2° and 179.5°, respectively. Intermolecular C–H···O hydrogen bonds connect two adjacent compounds into a dimer lower part of the figure. Then it forms a three-dimensional structure by π-π stacking.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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