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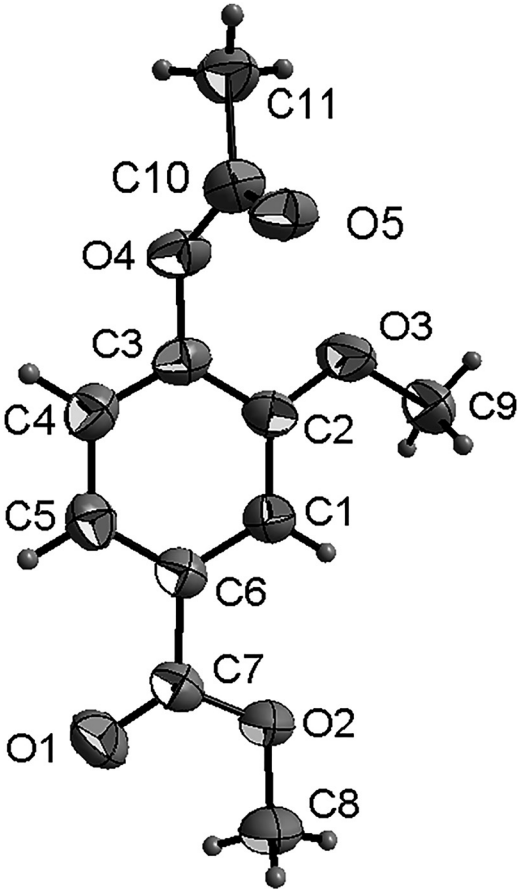
Crystal structure of methyl 4-acetoxy-3-methoxybenzoate, $C_{11}H_{12}O_5$



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Abstract

$C_{11}H_{12}O_5$, orthorhombic, $P2_12_12_1$ (no. 19), a = 5.5523(7) Å, b = 12.7610(17) Å, c = 15.374(2) Å, V = 1089.3(2) Å^3, Z = 4, $R_{gt}(F)$ = 0.0273, $wR_{ref}(F^2)$ = 0.0787, T = 296(2) K.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.18 × 0.14 × 0.12 mm
Wavelength:	Mo $K\alpha$ 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}:	8424, 2025, 0.020
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 1858
$N(param)_{refined}$:	149
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

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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.

Source of material

The mixture of methyl 4-hydroxy-3-methoxybenzoate (1.82 g, 0.01 mol), acetic anhydride (3.06 g, 0.03 mol) was reacted at 80 °C for 2 h. After the reaction was completed (monitored by thin layer chromatography (TLC)), colorless crystals were produced during the cooling to room temperature. The product was filtered, and washed with water three times respectively. Then crystals were obtained. Yield 83% (based on methyl 4-hydroxybenzoate). **Elemental Anal. Calcd.** (%) for $C_{11}H_{12}O_5$(224.21): C, 56.69; H, 5.55. Found (%): C, 58.93; H, 5.39.

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.

Comment

Polyphenols that occur naturally in plants, which are found in a variety of plant-based foods [5]. There are many

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5118 (4)	0.09658 (15)	0.15142 (12)	0.0473 (5)
H1	0.3788	0.0731	0.1826	0.057*
C2	0.6444 (4)	0.18063 (15)	0.18166 (12)	0.0473 (5)
C3	0.8390 (4)	0.21561 (15)	0.13224 (13)	0.0482 (5)
C4	0.9024 (4)	0.16798 (16)	0.05595 (13)	0.0537 (5)
H4	1.0325	0.1928	0.0239	0.064*
C5	0.7715 (4)	0.08243 (14)	0.02673 (13)	0.0510 (5)
H5	0.8146	0.0491	−0.0248	0.061*
C6	0.5776 (4)	0.04688 (14)	0.07416 (12)	0.0439 (4)
C7	0.4447 (4)	−0.04705 (14)	0.04274 (12)	0.0467 (5)
C8	0.1116 (5)	−0.15816 (18)	0.06569 (16)	0.0670 (6)
H8A	0.0115	−0.1394	0.0172	0.101*
H8B	0.0123	−0.1798	0.1135	0.101*
H8C	0.2164	−0.2146	0.0492	0.101*
C9	0.4122 (5)	0.19674 (19)	0.31070 (14)	0.0644 (6)
H9A	0.2631	0.2040	0.2796	0.097*
H9B	0.4062	0.2380	0.3629	0.097*
H9C	0.4366	0.1244	0.3255	0.097*
C10	0.8958 (4)	0.39492 (16)	0.16569 (13)	0.0524 (5)
C11	1.0699 (5)	0.46698 (19)	0.20890 (15)	0.0633 (6)
H11A	1.0559	0.5357	0.1840	0.095*
H11B	1.2308	0.4413	0.2005	0.095*
H11C	1.0352	0.4703	0.2700	0.095*
O1	0.5057 (3)	−0.09909 (12)	−0.01877 (11)	0.0721 (5)
O2	0.2537 (3)	−0.06905 (10)	0.09129 (9)	0.0580 (4)
O3	0.6046 (3)	0.23179 (12)	0.25771 (9)	0.0660 (5)
O4	0.9855 (3)	0.29545 (11)	0.16520 (9)	0.0567 (4)
O5	0.7066 (3)	0.41688 (12)	0.13460 (14)	0.0762 (5)

different phenolic acid derivatives found in nature, and they can be divided into two categories: benzoic acid derivatives, such as gallic acid, and cinnamic acid derivatives, including caffeic acid [6–8]. Vanillic acid, 4-Hydroxy-3-methoxybenzoic acid, is a natural phenolic acid derivatives of benzoic acid category, and is widely found in nature, such as vanilla beans, vanilla pods, peruvian balsam, benzoin and javanese oil. Vanillic acid has antibacterial activities, antioxidant activities, anti-inflammatory, anti-virus and antithrombotic effect [9–12]. Because of its strong activities and its wide applications, the synthesis and application of vanillic acid and its derivatives have attracted much attention [10–15]. We still focused on the synthesis and antibacterial activities of preservatives. In order to synthesis novel preservatives, we have designed and synthesized a series of phenolic acids derivatives [16–18]. Herein we report the synthesis and single crystals of the title compound as an important intermediate.

There is one title molcelule in the asymmetric unit. In the molecule of the title compound bond lengths and angles within methyl 4-acetoxy-3-methoxybenzoate are very similar to those given in the literature [17, 18]. In the title structure, the part of methyl 4-hydroxy-3-methoxybenzoate is approximately planar. The dihedral angle formed by the C1–C6 plane with the carboxylate group O1–C7–O2 plane is 6.5°. The acetyl group O4–C10–O5 plane is perpendicular to methyl *p*-hydroxy-3,5-dimethoxybenzoate and the dihedral angle is 73.5°. The torsion angles of C1–C2–O3–C9, C1–C6–C7–O2, C6–C7–O2–C8, C2–C3–O4–C10 and C3–O4–C10–C11 are 0.0(3)°, 5.6(3)°, 179.4(2)°, −73.8(2)° and 175.9(2)°, respectively.

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

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