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Crystal structure of methyl (*E*)-3-(4-acetoxyphenyl)acrylate, C₁₂H₁₂O₄

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.19 × 0.17 × 0.15 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.10 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8500, 2121, 0.024
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 1743
$N(\text{param})_{\text{refined}}$:	148
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

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Abstract

C₁₂H₁₂O₄, monoclinic, $P2_1/c$ (no. 14), $a = 16.071(6)$ Å, $b = 9.424(4)$ Å, $c = 7.640(3)$ Å, $\beta = 101.861(5)^\circ$, $V = 1132.4(8)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0366$, $wR_{\text{ref}}(F^2) = 0.1043$, $T = 296(2)$ K.

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The molecular structure is shown in 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 (*E*)-3-(4-hydroxyphenyl)acrylate (1.78 g, 0.01 mol), acetic anhydride (3.06 g, 0.03 mol) was reacted at

80 °C for 2 h. After the reaction completed (monitored by TLC), colorless crystal was produced after cooled slowly. The product was filtered, and washed with water 3 times respectively. yield 88% (based on methyl (*E*)-3-(4-hydroxyphenyl)acrylate). **Elemental Anal. Calcd.** (%) for C₁₄H₁₂O₄ (220.07): C, 65.45; H, 5.49. Found (%): C, 63.71; H, 5.64. The crystals were obtained after one week of slow volatilisation at room temperature.

Experimental details

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

Comment

Phenolic acids are polyphenols that occur naturally in plants, which are found in a variety of plant-based foods [5]. There are many different phenolic acids 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]. *p*-Coumaric acid, (*E*)-3-(4-hydroxyphenyl)acrylic acid, is a natural phenolic acid of cinnamic acid core structure [9]. *p*-Coumaric acid is mainly found in fruits, vegetables, grains, and fungi, and is also abundant in Chinese herbal medicines [10–13]. The pharmacological effects of *p*-coumaric acid has anti-oxidant, anti-inflammatory, antitumor effects, antiplatelet

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.77969 (10)	0.70368 (15)	0.41943 (18)	0.0597 (4)
H1	0.8056	0.7831	0.4795	0.072*
C2	0.69447 (10)	0.68050 (16)	0.41328 (19)	0.0641 (4)
H2	0.6632	0.7431	0.4683	0.077*
C3	0.65670 (9)	0.56314 (16)	0.32436 (18)	0.0586 (4)
C4	0.70259 (9)	0.46899 (16)	0.24450 (19)	0.0611 (4)
H4	0.6762	0.3895	0.1857	0.073*
C5	0.78802 (9)	0.49278 (15)	0.25186 (18)	0.0564 (4)
H5	0.8191	0.4286	0.1984	0.068*
C6	0.82809 (8)	0.61174 (14)	0.33844 (16)	0.0507 (3)
C7	0.91809 (9)	0.64496 (14)	0.34732 (17)	0.0544 (3)
H7	0.9386	0.7260	0.4115	0.065*
C8	0.97347 (9)	0.57423 (14)	0.27570 (19)	0.0570 (4)
H8	0.9566	0.4927	0.2094	0.068*
C9	1.06182 (9)	0.62359 (14)	0.30012 (18)	0.0543 (3)
C10	1.19626 (9)	0.57792 (18)	0.2373 (2)	0.0695 (4)
H10A	1.1987	0.6607	0.1653	0.104*
H10B	1.2272	0.5020	0.1961	0.104*
H10C	1.2212	0.5989	0.3598	0.104*
C11	0.51052 (10)	0.60018 (18)	0.2080 (2)	0.0676 (4)
C12	0.42499 (11)	0.5670 (2)	0.2429 (3)	0.0896 (6)
H12A	0.3822	0.6099	0.1522	0.134*
H12B	0.4206	0.6036	0.3580	0.134*
H12C	0.4169	0.4660	0.2412	0.134*
O1	1.10930 (6)	0.53626 (10)	0.22332 (14)	0.0655 (3)
O2	1.09026 (7)	0.72883 (11)	0.38053 (15)	0.0719 (3)
O3	0.57155 (6)	0.53325 (13)	0.32673 (14)	0.0738 (3)
O4	0.52631 (8)	0.67367 (16)	0.09311 (18)	0.0974 (5)

aggregation, and cardiovascular protection, while the anti-oxidant activities is the important basis of other pharmacological effects [14–16]. In addition, *p*-coumaric acid has a certain inhibitory effect on bacteria, and also can inhibit melanin formation and delay skin aging. Because of its strong activities and its wide applications, *p*-coumaric acid and its derivatives was widely used in cosmetics, medicine, pesticide chemistry and organic synthesis. the synthesis and application of *p*-coumaric acid and its derivatives have attracted much attention [14–18]. We are still focused on the synthesis and anti-bacterial activities of preservatives. In order to the synthesis novel preservatives from natural products with bacteriostatic activity, we have designed and synthesised a series of phenolic acids derivatives [19–21]. Herein we report the synthesis and single crystals of the title compound as an important intermediate.

There is one molecule in the asymmetric unit (shown in the figure). In the molecule of the title compound bond lengths and angles are very similar to those given in the

literature [21]. In the title structure, the parts of methyl *p*-coumaric acid were approximately planar. The dihedral angles of the C1–C6 plane, the carbon-carbon double bond group C6–C7–C8 and the carboxylate group O1–C9–O2 plane were 2.15(11)°, 0.38(5)° and 1.91(14)°, respectively. The acetyl group O3–C11–O4 plane is perpendicular to methyl *p*-coumaric acid and the dihedral angle is 84.3(4)°. The torsion angles of C5–C6–C7–C8, C6–C7–C8–C9, C7–C8–C9–O1 and C8–C9–O1–C10 are –177.97(15)°, –179.58(13)°, 177.91(13)° and 178.73(12)°, respectively.

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