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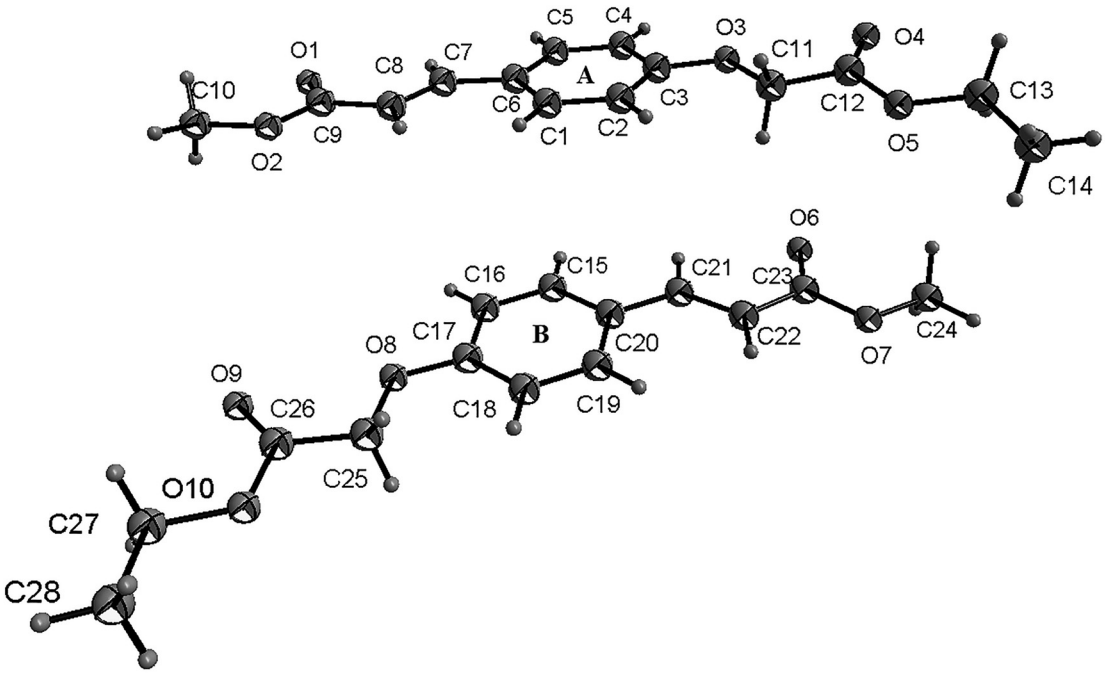
Crystal structure of methyl (*E*)-3-(4-(2-ethoxy-2-oxoethoxy)phenyl) acrylate, $C_{14}H_{16}O_5$



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Abstract

$C_{14}H_{16}O_5$, triclinic, $P\bar{1}$ (no. 2), a = 8.5293(8) Å, b = 11.5626(11) Å, c = 14.2196(13) Å, α = 88.888(10)°, β = 74.988(10)°, γ = 87.556(10)°, V = 1353.2(2) Å^3, Z = 4, $R_{gt}(F)$ = 0.0430, $wR_{ref}(F^2)$ = 0.1202, 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.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.20 × 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
θ_{max}, completeness:	25.5°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10,485, 5000, 0.021
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3803
$N(param)_{refined}$:	348
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

Source of material

The mixtrue of methyl (*E*)-3-(4-hydroxyphenyl)acrylate (1.78 g, 0.01 mol), ethyl 2-bromoacetate (2.00 g, 0.012 mol), K_2CO_3 (2.76 g, 0.02 mol) and DMF (10 mL) was reacted at 80 °C for 2 h. After the reaction completed (monitored by TLC), the mixture was poured into 50 mL ice water and a

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5279 (2)	−0.03036 (15)	0.83500 (13)	0.0583 (5)
H1	0.5195	−0.0209	0.9009	0.070*
C2	0.4461 (2)	0.04737 (15)	0.78830 (13)	0.0591 (5)
H2	0.3840	0.1086	0.8223	0.071*
C3	0.4571 (2)	0.03348 (14)	0.69001 (12)	0.0488 (4)
C4	0.5508 (2)	−0.05755 (14)	0.63956 (12)	0.0524 (4)
H4	0.5586	−0.0670	0.5737	0.063*
C5	0.6323 (2)	−0.13374 (14)	0.68744 (12)	0.0523 (4)
H5	0.6954	−0.1943	0.6530	0.063*
C6	0.6226 (2)	−0.12251 (14)	0.78623 (12)	0.0498 (4)
C7	0.7077 (2)	−0.20735 (15)	0.83439 (13)	0.0545 (4)
H7	0.7680	−0.2651	0.7946	0.065*
C8	0.7106 (2)	−0.21326 (16)	0.92638 (14)	0.0587 (5)
H8	0.6561	−0.1561	0.9690	0.070*
C9	0.7981 (2)	−0.30808 (15)	0.96279 (13)	0.0544 (4)
C10	0.8606 (3)	−0.39440 (18)	1.10074 (14)	0.0696 (5)
H10A	0.9752	−0.3823	1.0833	0.104*
H10B	0.8408	−0.4679	1.0767	0.104*
H10C	0.8202	−0.3933	1.1703	0.104*
C11	0.2865 (2)	0.19980 (14)	0.68563 (13)	0.0568 (4)
H11A	0.3556	0.2497	0.7099	0.068*
H11B	0.2027	0.1727	0.7406	0.068*
C12	0.2107 (2)	0.26569 (14)	0.61603 (12)	0.0512 (4)
C13	0.0553 (3)	0.43844 (16)	0.60416 (15)	0.0674 (5)
H13A	−0.0353	0.3993	0.5914	0.081*
H13B	0.1279	0.4600	0.5423	0.081*
C14	−0.0044 (3)	0.54338 (17)	0.66396 (16)	0.0737 (6)
H14A	0.0864	0.5834	0.6736	0.111*
H14B	−0.0726	0.5208	0.7260	0.111*
H14C	−0.0657	0.5936	0.6309	0.111*
C15	0.8044 (2)	0.18059 (16)	0.86747 (13)	0.0598 (5)
H15	0.8318	0.1430	0.8079	0.072*
C16	0.8569 (2)	0.13283 (15)	0.94355 (13)	0.0583 (5)
H16	0.9198	0.0642	0.9351	0.070*
C17	0.8157 (2)	0.18734 (14)	1.03318 (12)	0.0507 (4)
C18	0.7214 (2)	0.28931 (15)	1.04514 (13)	0.0574 (5)
H18	0.6922	0.3258	1.1052	0.069*
C19	0.6711 (2)	0.33635 (16)	0.96771 (13)	0.0587 (5)
H19	0.6086	0.4051	0.9763	0.070*
C20	0.7111 (2)	0.28407 (15)	0.87698 (12)	0.0529 (4)
C21	0.6633 (2)	0.33272 (16)	0.79310 (13)	0.0590 (5)
H21	0.6886	0.2865	0.7380	0.071*
C22	0.5891 (2)	0.43337 (16)	0.78423 (13)	0.0601 (5)
H22	0.5563	0.4818	0.8378	0.072*
C23	0.5575 (2)	0.46999 (16)	0.69200 (14)	0.0585 (5)
C24	0.4518 (3)	0.61708 (17)	0.60725 (15)	0.0704 (5)
H24A	0.5530	0.6258	0.5592	0.106*
H24B	0.3943	0.6908	0.6187	0.106*
H24C	0.3874	0.5635	0.5843	0.106*
C25	0.8304 (2)	0.18562 (15)	1.19728 (12)	0.0534 (4)
H25A	0.7132	0.1962	1.2191	0.064*
H25B	0.8775	0.2610	1.1938	0.064*
C26	0.8909 (2)	0.10903 (15)	1.26773 (12)	0.0516 (4)
C27	0.8913 (3)	0.09761 (17)	1.43497 (13)	0.0647 (5)
H27A	1.0080	0.0890	1.4249	0.078*
H27B	0.8469	0.0212	1.4408	0.078*
C28	0.8182 (3)	0.16530 (18)	1.52402 (15)	0.0772 (6)
H28A	0.8626	0.2408	1.5172	0.116*
H28B	0.8420	0.1263	1.5792	0.116*
H28C	0.7027	0.1727	1.5334	0.116*
O1	0.8814 (2)	−0.38141 (13)	0.91315 (11)	0.0909 (5)
O2	0.77875 (17)	−0.30365 (12)	1.05856 (9)	0.0685 (4)
O3	0.37976 (15)	0.10434 (10)	0.63651 (8)	0.0579 (3)
O4	0.21016 (19)	0.23713 (12)	0.53647 (10)	0.0748 (4)
O5	0.14039 (17)	0.36314 (11)	0.65826 (9)	0.0675 (4)
O6	0.5950 (3)	0.41540 (14)	0.61845 (11)	0.1085 (7)
O7	0.48292 (18)	0.57381 (11)	0.69658 (9)	0.0696 (4)
O8	0.87424 (16)	0.13399 (10)	1.10454 (8)	0.0593 (3)
O9	0.95911 (18)	0.01679 (11)	1.25174 (9)	0.0709 (4)
O10	0.85264 (17)	0.16062 (10)	1.35406 (9)	0.0624 (3)

large amount of white product was precipitated. The product was filtered and washed with water three times respectively. The yield was 86% (based on methyl (*E*)-3-(4-hydroxyphenyl)acrylate). **Elemental Anal.** Calcd. (%) for $C_{14}H_{16}O_5$(264.27): C, 63.63; H, 6.10. Found (%): C, 61.53; H, 6.27. 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 = 0.93 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

The *p*-coumaric acid, (*E*)-3-(4-hydroxyphenyl) acrylic acid, is a natural phenolic acid of cinnamic acid core structure [5]. *p*-Coumaric acid is mainly found in fruits, vegetables, grains, and fungi, and is also abundant in Chinese herbal medicines [6–9]. The pharmacological effects of *p*-coumaric acid has anti-oxidant, anti-inflammatory, antitumor effects, antiplatelet aggregation, and cardiovascular protection, while the anti-oxidant activities is the important basis of other pharmacological effects [10–12]. The synthesis and application of *p*-coumaric acid and its derivatives have attracted much attention [10–16]. We are committed to the detection and regulation of cosmetics. In order to establish a rapid and effective method for the determination of coumaric acid derivatives, a series of *p*-coumaric acid derivatives were synthesized.

There are two crystallographic independent molecules in the asymmetric unit (shown in the figure). In the molecules of the title structure bond lengths and angles are very similar to those given in the literature for *p*-coumaric acid derivatives [17, 18]. In the title structure, the parts of methyl *p*-coumaric acid of molecule A and B were approximately planar. The dihedral angles of molecule A formed by the C1–C6 plane, the carboxylate group O1–C9–O2 plane and the carboxylate group O4–C12–O5 plane were 5.0°, 10.1° and 14.6, respectively, while the dihedral angles of molecule B formed by the C15–C20 plane, the carboxylate group O6–C23–O7 plane and the carboxylate group O9–C26–O10 plane were 6.5°, 6.1° and 1.1°, respectively.

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