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Crystal structure of methyl 4-acetoxy-3,5-dimethoxybenzoate, $C_{12}H_{14}O_6$

Abstract

$C_{12}H_{14}O_6$, orthorhombic, *Pbca* (no. 61), $a = 18.0207(13)$ Å, $b = 7.6885(5)$ Å, $c = 18.2326(13)$ Å, $V = 2526.2(3)$ Å³, $Z = 8$, $R_{gt}(F) = 0.0411$, $wR_{ref}(F^2) = 0.1214$, $T = 296(2)$ K.

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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 × 0.14 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} :	18,059, 2350, 0.029
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 1937
$N(param)_{refined}$:	168
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

Source of materials

The mixture of methyl 4-hydroxy-3,5-dimethoxybenzoate (2.12 g, 0.01 mol), acetic anhydride (2.04 g, 0.02 mol) was reacted at 80 °C for 1 h. After the reaction completed (monitored by TLC), colorless crystal appeared after cooled slowly. The product was filtered and washed with water three times respectively: yield 92% (based on methyl 4-hydroxybenzoate).

Elemental Anal. Calcd. (%) for $C_{12}H_{14}O_6$ (254.23): C, 56.69; H, 5.55. Found (%): C, 54.75; H, 5.74.

Experimental details

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

Phenolic acids are polyphenols that occur naturally in plants, which are found in a variety of plant-based foods [5].

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

Atom	x	y	z	U _{iso} [*] /U _{eq}
C1	0.09115 (10)	0.9821 (2)	0.26869 (9)	0.0494 (4)
H1	0.0594	1.0142	0.3066	0.059*
C2	0.06371 (9)	0.9536 (2)	0.19840 (9)	0.0457 (4)
C3	0.11230 (9)	0.9086 (2)	0.14246 (8)	0.0413 (4)
C4	0.18750 (9)	0.8889 (2)	0.15531 (8)	0.0434 (4)
C5	0.21473 (9)	0.9154 (2)	0.22570 (9)	0.0488 (4)
H5	0.2651	0.9019	0.2354	0.059*
C6	0.16642 (9)	0.9619 (2)	0.28132 (8)	0.0477 (4)
C7	0.19424 (11)	0.9916 (3)	0.35735 (9)	0.0573 (5)
C8	0.29867 (14)	0.9840 (4)	0.43527 (12)	0.0910 (8)
H8A	0.2992	1.1060	0.4466	0.136*
H8B	0.3486	0.9403	0.4352	0.136*
H8C	0.2700	0.9231	0.4715	0.136*
C9	−0.06253 (11)	0.9828 (3)	0.23395 (12)	0.0724 (6)
H9A	−0.0569	0.8897	0.2686	0.109*
H9B	−0.1112	0.9778	0.2126	0.109*
H9C	−0.0562	1.0922	0.2585	0.109*
C10	0.30770 (10)	0.8598 (3)	0.10271 (11)	0.0647 (6)
H10A	0.3197	0.9742	0.1202	0.097*
H10B	0.3302	0.8418	0.0556	0.097*
H10C	0.3260	0.7747	0.1367	0.097*
C11	0.05057 (8)	0.7398 (2)	0.05478 (9)	0.0433 (4)
C12	0.01851 (12)	0.7444 (3)	−0.02022 (10)	0.0648 (6)
H12A	0.0495	0.6789	−0.0529	0.097*
H12B	0.0157	0.8628	−0.0368	0.097*
H12C	−0.0303	0.6947	−0.0194	0.097*
O1	0.15657 (9)	1.0397 (3)	0.40764 (7)	0.0893 (6)
O2	0.26587 (7)	0.9579 (2)	0.36356 (7)	0.0760 (5)
O3	−0.00855 (7)	0.9662 (2)	0.17826 (7)	0.0637 (4)
O4	0.22926 (6)	0.8432 (2)	0.09629 (6)	0.0580 (4)
O5	0.08545 (6)	0.89220 (15)	0.07113 (6)	0.0451 (3)
O6	0.04805 (8)	0.62225 (18)	0.09706 (7)	0.0660 (4)

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]. The syringic acid, is a natural phenolic acid of benzoic acid category, which has antibacterial activities, sedative activities and local anesthetic effects [9–12]. Because of its strong activities and its wide applications, pyromethyl was widely used in perfume, medicine, pesticide chemistry and organic synthesis. The synthesis and application of syringic acid and its derivatives have attracted much attention [13–16]. We still focused on the synthesis and antibacterial activities of preservatives. Thus we have designed and synthesised a series of phenolic acids derivatives. Herein, we report the synthesis and crystal structure of an important intermediate.

There is one crystallographic independent 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 methyl 4-acetoxybenzoate [17]. The dihedral angle formed between the C1–C6 plane the carboxylate group O1–C7–O2 plane is 4.0°. The acetyl group O5–C16–O6 plane is perpendicular to methyl p-hydroxy-3,5-dimethoxybenzoate and the dihedral angle is 81.5°.

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