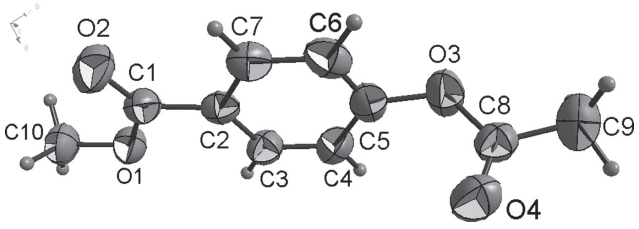
Fang Li-Min, Huang Jian-Ping, Dai Jian-Cheng, Nie Xu-Liang*, Liu Chang-Xiang, Kang Nian-Qian and Huang Lin*

Crystal structure of methyl 4-acetoxybenzoate, $C_{10}H_{10}O_4$



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Abstract

$C_{10}H_{10}O_4$, monoclinic, $C2/c$ (no. 15), $a = 25.400(4)$ Å, $b = 5.9738(10)$ Å, $c = 12.746(2)$ Å, $\beta = 94.318(2)°$, $V = 1928.5(6)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0434$, $wR_{ref}(F^2) = 0.1276$, $T = 296(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The mixture of methyl 4-hydroxybenzoate (15.2 g, 0.1 mol), acetic anhydride (20.4 g, 0.2 mol) and sulfuric acid (1 mL) was reacted at 80 °C for 1 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. Then the crystals suitable for crystal structure analysis were obtained. Yield 90% (based on methyl 4-hydroxybenzoate). m.p. 83–85 °C (m.p. 82–84 °C). Elemental Anal. Calcd. (%) for $C_{10}H_{10}O_4$(152.05): C, 61.85; H, 5.19. Found (%): C, 60.45; H, 5.33.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.24 × 0.18 × 0.18 mm
Wavelength:	Mo $K\alpha$ 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}:	4908, 1787, 0.023
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1496
$N(param)_{refined}$:	130
Programs:	Bruker [1], SHELX [2]

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.31186(6)	0.1456(3)	0.08294(12)	0.0497(4)
C2	0.33954(6)	0.3413(3)	0.13256(11)	0.0464(4)
C3	0.38768(6)	0.4182(3)	0.10100(13)	0.0536(4)
H3	0.4042	0.3426	0.0488	0.064*
C4	0.41086(7)	0.6065(3)	0.14710(14)	0.0596(5)
H4	0.4430	0.6583	0.1265	0.072*
C5	0.38564(7)	0.7167(3)	0.22430(13)	0.0564(5)
C6	0.33877(7)	0.6404(3)	0.25816(13)	0.0609(5)
H6	0.3228	0.7148	0.3114	0.073*
C7	0.31583(7)	0.4529(3)	0.21235(13)	0.0547(4)
H7	0.2841	0.4000	0.2349	0.066*
C8	0.44297(7)	0.9219(3)	0.34400(13)	0.0565(5)
C9	0.45889(10)	1.1540(4)	0.37491(18)	0.0850(7)
H9A	0.4384	1.2037	0.4308	0.128*
H9B	0.4528	1.2518	0.3155	0.128*
H9C	0.4957	1.1562	0.3984	0.128*
C10	0.31264(8)	−0.1320(3)	−0.04792(16)	0.0673(5)
H10A	0.3092	−0.2524	0.0010	0.101*
H10B	0.3337	−0.1804	−0.1031	0.101*
H10C	0.2783	−0.0885	−0.0776	0.101*
O1	0.33776(4)	0.05600(19)	0.00601(9)	0.0563(4)
O2	0.27017(5)	0.0745(2)	0.10763(12)	0.0754(5)
O3	0.40570(6)	0.9218(2)	0.26309(10)	0.0739(4)
O4	0.45881(6)	0.7520(3)	0.38437(12)	0.0835(5)

***Corresponding authors: Nie Xu-Liang,** College of Sciences, Jiangxi Agricultural University, Nanchang 330045, P.R. China, e-mail: niexuliang1981@163.com; and **Huang Lin,** College of Bioscience and Bioengineering, Jiangxi Agricultural University, Nanchang 330045, P.R. China, e-mail: huanglin213@126.com
Fang Li-Min and Dai Jian-Cheng: College of Bioscience and Bioengineering, Jiangxi Agricultural University, Nanchang 330045, P.R. China
Huang Jian-Ping: Institute of Functional Materials and Agricultural Applied Chemistry/Jiangxi Key Laboratory for Conservation and Utilization of Fungal Resources, Jiangxi Agricultural University, Nanchang 330045, P.R. China
Liu Chang-Xiang and Kang Nian-Qian: College of Sciences, Jiangxi Agricultural University, Nanchang 330045, P.R. China

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

Pyromethyl, methyl hydroxybenzoate, as a preservative has good inhibition of mold, yeast, bacteria in pH 1–8 [3]. *p*-Hydroxybenzoate is able to destroy the cell membrane of microorganisms, inhibit the electron transfer and the activity of the respiratory enzyme of microbial cells [4–6]. Because of its strong activities and its applications, pyromethyl was widely used as preservatives in the pharmaceutical industry and food industry, cosmetics. The synthesis and application of *p*-hydroxybenzoate and its derivatives have attracted much attention [7–9]. We are still focused on the synthesis and antibacterial activities of preservatives. In order to synthesis novel preservatives, we have designed and synthesized a series of flavonoids carboxylate glycosides.

There is one crystallographic independant molecule in the asymmtric unit. The molecule is in a general position, giving eight molecules in each unit cell. In the molecule of the title compound bond lengths and angles are very similar to those given in the literature for methyl *p*-hydroxybenzoate [10, 11]. In the title structure, the part of methyl *p*-hydroxybenzoate is approximately planar. The dihedral angle formed by the C2—C7 plane with the carboxlate group C1—O1—O2 plane is 3.2(1)°. The acetyl group is perpendicular to methyl *p*-hydroxybenzoate and the dihedral angle is 85.6(1)°.

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