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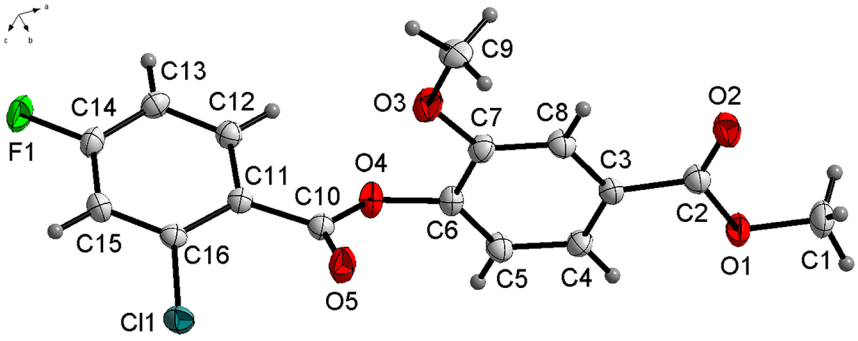
Crystal structure of 2-methoxy-4-(methoxycarbonyl)phenyl 2-chloro-4-fluorobenzoate, $C_{16}H_{12}ClFO_5$



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Abstract

$C_{16}H_{12}ClFO_5$, monclinic, $P2_1/c$ (no. 14), a = 14.7536(6) Å, b = 7.8281(3) Å, c = 14.0501(7) Å, β = 111.174(5)°, V = 1513.13(12) Å^3, Z = 4, $R_{gt}(F)$ = 0.0367, $wR_{ref}(F^2)$ = 0.0865, 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:	0.21 × 0.16 × 0.13 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.29 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}:	6080, 2822, 0.017
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2363
$N(param)_{refined}$:	211
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

1 Source of materials

2-Chloro-4-fluorobenzoyl chloride (1.86 g, 0.12 mol) was added dropwise under stirring into the mixture of methyl 4-hydroxy-3-methoxybenzoate (1.82 g, 0.01 mol), K_2CO_3 (2.76 g, 0.02 mol) and CH_2Cl_2 (20 mL) at 0 °C. The mixture was reacted for 3 h (monitored by TLC) then washed with water. CH_2Cl_2 was removed by steam distillation, colorless product was produced. Yield 90.6% (based on methyl 4-hydroxy-3-methoxybenzoate). The crystals were obtained by evaporating ethyl acetate slowly at room temperature.

2 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.

3 Comment

Vanillic acid derivatives possess anti-inflammatory, antibacterial activity and have attracted the attention of the pharmaceutical industry [5–7]. However, its poor fat

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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	0.76644 (15)	0.9570 (3)	0.08987 (18)	0.0672 (7)
H1A	0.8002	0.8606	0.0764	0.101*
H1B	0.8091	1.0186	0.1478	0.101*
H1C	0.7455	1.0308	0.0313	0.101*
C2	0.60911 (13)	0.8369 (2)	0.03095 (15)	0.0414 (4)
C3	0.53025 (12)	0.7670 (2)	0.06189 (13)	0.0364 (4)
C4	0.53905 (13)	0.7536 (3)	0.16331 (13)	0.0415 (4)
H4	0.5937	0.7970	0.2146	0.050*
C5	0.46630 (13)	0.6756 (3)	0.18803 (14)	0.0429 (5)
H5	0.4724	0.6645	0.2560	0.052*
C6	0.38527 (12)	0.6146 (2)	0.11191 (13)	0.0369 (4)
C7	0.37411 (12)	0.6290 (2)	0.00935 (14)	0.0401 (4)
C8	0.44730 (13)	0.7049 (2)	−0.01518 (14)	0.0408 (4)
H8	0.4413	0.7148	−0.0832	0.049*
C9	0.27435 (16)	0.5799 (4)	−0.16443 (16)	0.0722 (7)
H9A	0.2748	0.6991	−0.1803	0.108*
H9B	0.2124	0.5316	−0.2042	0.108*
H9C	0.3247	0.5222	−0.1801	0.108*
C10	0.22594 (12)	0.5868 (2)	0.11369 (13)	0.0366 (4)
C11	0.15806 (12)	0.4512 (2)	0.12021 (12)	0.0322 (4)
C12	0.18156 (12)	0.2795 (2)	0.11238 (13)	0.0368 (4)
H12	0.2412	0.2533	0.1075	0.044*
C13	0.11903 (13)	0.1485 (2)	0.11167 (13)	0.0403 (4)
H13	0.1352	0.0352	0.1057	0.048*
C14	0.03212 (13)	0.1910 (2)	0.12010 (14)	0.0411 (4)
C15	0.00527 (13)	0.3559 (2)	0.12967 (14)	0.0398 (4)
H15	−0.0540	0.3796	0.1360	0.048*
C16	0.06870 (12)	0.4859 (2)	0.12965 (12)	0.0334 (4)
Cl1	0.03207 (3)	0.69181 (6)	0.14373 (4)	0.04743 (17)
F1	−0.03085 (9)	0.06512 (15)	0.11894 (10)	0.0646 (4)
O1	0.68287 (9)	0.8983 (2)	0.11094 (10)	0.0555 (4)
O2	0.60874 (10)	0.8366 (2)	−0.05449 (11)	0.0566 (4)
O3	0.29090 (9)	0.5605 (2)	−0.05882 (10)	0.0591 (4)
O4	0.31685 (8)	0.52082 (16)	0.13770 (10)	0.0448 (3)
O5	0.20704 (10)	0.73283 (17)	0.08994 (12)	0.0577 (4)

solubility and instability hinder its application. So it is necessary to modify their structures. Wang et al. have synthesized two 4-alkyl methyl vanillic acid derivatives [8, 9] and also methyl 4-(benzyloxy)-3-methoxybenzoate [10]. We have synthesized methyl 4-acetoxy-3-methoxybenzoate [11]. As part of our ongoing studies on phenolic acid derivatives fungicides [12–16].

In the molecules of the title structure, bond lengths and angles are very similar to those given in the literature for methyl 4-acetoxy-3-methoxybenzoate [11–13]. In the title structure, the dihedral angle formed by the C3–C8 phenyl plane, the C11–C16 phenyl plane, the carboxylate group C2–O1–O2 plane and the carboxylate group C10–O4–O5 plane are 6.7°, 81.8° and 76.7°, respectively. The torsion angles of C3–C2–O1–C1, C6–C7–O3–C9, C7–C6–O4–C10 and C11–C10–O4–C6 are 175.4°, 177.0°, −70.7° and 164.3°, respectively.

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

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