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Crystal structure of 4-(3-fluorophenyl)-7-hydroxy-2H-chromen-2-one, C₁₅H₉FO₃

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.31 × 0.25 × 0.14 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.12 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	27.1°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	17738, 4955, 0.020
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4536
$N(\text{param})_{\text{refined}}$:	389
Programs:	Bruker [1], SHELX [2, 4], Mercury [3]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1A	−0.39027(6)	0.79129(10)	−0.16979(12)	0.0251(3)
H1A	−0.421440	0.803290	−0.130546	0.038*
O2A	−0.47714(5)	1.15134(9)	−0.38902(10)	0.0188(2)
O3A	−0.51755(5)	1.32314(9)	−0.47811(11)	0.0236(2)
C1A	−0.39497(7)	1.03994(13)	−0.47514(14)	0.0177(3)
C2A	−0.35111(7)	0.94112(14)	−0.45569(15)	0.0205(3)
H2A	−0.322205	0.929491	−0.514956	0.025*
C4A	−0.39225(7)	0.87612(13)	−0.26462(15)	0.0199(3)
C5	−0.34938(7)	0.86130(14)	−0.35263(16)	0.0216(3)
H5	−0.319071	0.795752	−0.340742	0.026*
C5A	−0.43453(7)	0.97482(13)	−0.27769(15)	0.0186(3)
H5A	−0.462515	0.987276	−0.216804	0.022*
C6A	−0.43477(7)	1.05466(13)	−0.38196(15)	0.0172(3)
C7A	−0.47999(7)	1.23974(14)	−0.48320(15)	0.0188(3)
C8A	−0.44051(7)	1.22464(14)	−0.58149(15)	0.0193(3)
H8A	−0.442187	1.284425	−0.648239	0.023*
C9A	−0.40096(7)	1.12764(13)	−0.58172(14)	0.0179(3)
C10A	−0.36638(7)	1.11111(13)	−0.69329(15)	0.0186(3)
C11A	−0.37429(8)	1.00541(14)	−0.76822(15)	0.0216(3)
H11A	−0.400156	0.942129	−0.744945	0.026*
C12A	−0.34440(8)	0.99258(14)	−0.87682(16)	0.0240(3)
H12A	−0.350459	0.920963	−0.927889	0.029*
C13A	−0.30585(8)	1.08387(14)	−0.91068(16)	0.0243(3)
H13A	−0.284547	1.075226	−0.983367	0.029*
C14A	−0.29925(7)	1.18762(14)	−0.83604(16)	0.0231(3)
C15A	−0.32857(7)	1.20389(13)	−0.72868(15)	0.0207(3)
H15A	−0.323156	1.276689	−0.679860	0.025*
F1A	−0.26349(5)	1.27808(9)	−0.87144(11)	0.0311(2)
O1B	−0.10961(6)	1.21146(10)	0.21746(12)	0.0265(3)

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Abstract

C₁₅H₉FO₃, monoclinic, $P2_1/c$ (no. 14), $a = 20.352(2)$ Å, $b = 11.2575(9)$ Å, $c = 10.1604(8)$ Å, $\beta = 104.434(1)^\circ$, $V = 2254.4(3)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0340$, $wR_{\text{ref}}(F^2) = 0.0961$, $T = 100(2)$ 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.

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Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} ^a / <i>U</i> _{eq}
H1B	−0.079557	1.197978	0.289151	0.040*
O2B	−0.02338(5)	0.85007(9)	0.08902(10)	0.0186(2)
O3B	0.01765(5)	0.67810(10)	0.04247(11)	0.0233(2)
C1B	−0.10437(7)	0.96150(13)	−0.08176(15)	0.0175(3)
C2B	−0.14739(7)	1.06171(14)	−0.10751(15)	0.0196(3)
H2B	−0.175966	1.073295	−0.195970	0.024*
C3B	−0.14875(7)	1.14291(14)	−0.00704(16)	0.0208(3)
H3B	−0.177792	1.210090	−0.026890	0.025*
C4B	−0.10734(7)	1.12687(13)	0.12488(15)	0.0196(3)
C5B	−0.06597(7)	1.02710(13)	0.15531(15)	0.0191(3)
H5B	−0.038780	1.014278	0.244769	0.023*
C6B	−0.06532(7)	0.94677(13)	0.05208(15)	0.0173(3)
C7B	−0.01990(7)	0.76116(14)	−0.00138(16)	0.0182(3)
C8B	−0.05896(7)	0.77546(14)	−0.13951(15)	0.0197(3)
H8B	−0.057017	0.715224	−0.203914	0.024*
C9B	−0.09851(7)	0.87233(13)	−0.18073(15)	0.0186(3)
F1B ^a	−0.23440(6)	0.71324(13)	−0.60135(14)	0.0406(4)
C11C ^a	−0.12789(16)	0.9926(2)	−0.3939(3)	0.0245(7)
H11C ^a	−0.102896	1.057148	−0.345730	0.029*
C12C ^a	−0.15898(15)	1.0030(3)	−0.5325(2)	0.0302(6)
H12C ^a	−0.154742	1.074961	−0.578417	0.036*
C13C ^a	−0.19593(13)	0.9098(3)	−0.6039(2)	0.0299(6)
H13C ^a	−0.217594	0.917417	−0.697822	0.036*
C14C ^a	−0.20045(11)	0.8057(3)	−0.5353(2)	0.0267(5)
C15C ^a	−0.17004(11)	0.7910(2)	−0.3992(3)	0.0233(7)
H15C ^a	−0.173547	0.717671	−0.355209	0.028*
F2B ^b	−0.1691(3)	1.0726(4)	−0.6130(5)	0.0278(15)
C11D ^b	−0.1365(4)	0.9827(4)	−0.4128(5)	0.032(4)
H11D ^b	−0.112641	1.053531	−0.379739	0.039*
C10B	−0.13397(7)	0.88589(14)	−0.32684(15)	0.0221(3)
C15D ^b	−0.1689(4)	0.7823(4)	−0.3752(5)	0.026(4)
H15D ^b	−0.167152	0.716126	−0.316481	0.031*
C14D ^b	−0.2063(5)	0.7756(7)	−0.5096(6)	0.032(3)
H14D ^b	−0.230205	0.704814	−0.542700	0.039*
C13D ^b	−0.2089(4)	0.8725(9)	−0.5956(3)	0.024(3)
H13D ^b	−0.234478	0.867860	−0.687440	0.029*
C12D ^b	−0.1740(5)	0.9760(8)	−0.5472(5)	0.027(3)

^aOccupancy: 0.816(3), ^bOccupancy: 0.184(3).

Source of material

The titled compound was prepared by mixing resorcinol (2.0 g) and methyl 3-fluorobenzoylacetate (3.5 g) and then added conc. H₂SO₄ (8 mL, 75%). The temperature of the stirred mixture was increased to 35 °C. After stirring for 5 h, the mixture was poured into crushed ice and neutralized with a NaOH solution. The mixture was filtered under vacuum and the residue was washed with water. The resulting product was purified by silica gel column chromatography with 70% ethylacetate in hexane as eluent and the product was obtained as a yellow solid (4.5 g, 97%) and then recrystallized from ethanol by slow evaporation to obtain light-yellow crystals. **m.p.**: 209–211 °C.

Experimental details

A Bruker Smart APEX2 diffractometer with Mo Kα radiation was used for crystal evaluation and data collection [1]. The structure was solved by the direct method using the *SHELXS* [2] program and refined. The visual crystal structure information was performed using *Mercury* [3] system software. All C–H_{aromatic} and O–H bond distances were restrained to 0.95 Å, 0.98 Å and 0.99 Å with *U*_{iso}(H_{aromatic}) = 1.2*U*_{eq} and *U*_{iso}(H_{hydroxyl}) = 1.5*U*_{eq} of parent atom, respectively. The *meta*-fluorophenyl unit appeared to be disordered over two positions and PART instructions were used to model the disorder. The major component had site occupancy factor of 0.816. A two-component, non-merohedral twin with refined volume ratio of 0.784(1):0.216(1) was determined. The twin law used was [−1 0 −1; 0 −1 0; 0 0 1].

Comment

Coumarins are natural compounds and the first coumarin was already isolated in 1820 and now more than 1300 coumarins have been identified from natural sources [5, 6]. The class of coumarin compounds is known to possess a variety of biological activities such as anti-inflammatory [7], anti-oxidant [8, 9], antibacterial [10], anticoagulant [8], anti-tumor [11], and anti-viral activities [12]. It is profound that the presence of a fluorine in an organic molecule may have effects on the biological activity of the compound [13, 14].

The crystal structure of the title compound consists of two symmetrically independent molecules and each molecule has a *meta*-fluorophenyl and coumarinyl unit, respectively. The dihedral angle between the two units was found to be 127.28(4)° and 129.7(1)°, respectively. All bond parameters are comparable to closely related compounds in literature [15–21]. Neighbouring molecules are joined together *via* classical intermolecular O–H···O hydrogen bonds between the hydroxyl and carbonyl groups. Linking the molecules in this manner results in the formation of a one dimensional structure, which propagates along the crystallographic *b* axis.

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