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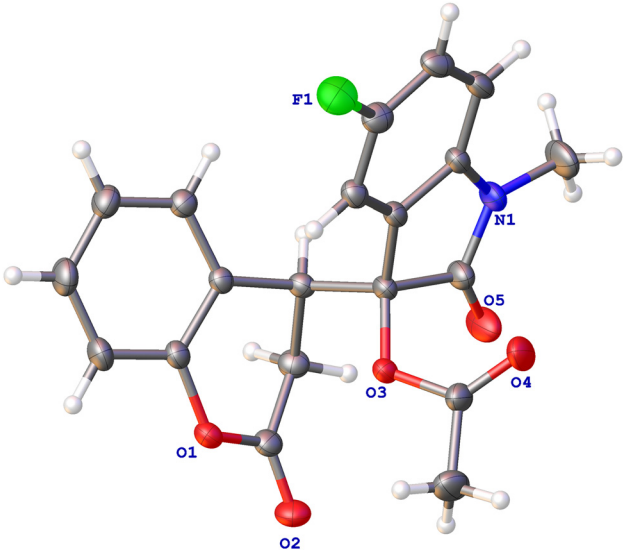
Synthesis and crystal structure of 5-fluoro-1-methyl-2-oxo-3-(2-oxochroman-4-yl)indolin-3-yl acetate, $C_{20}H_{16}FNO_5$



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Abstract

$C_{20}H_{16}FNO_5$, monoclinic, $P2_1/c$ (no. 14), a = 9.5235(2) Å, b = 22.1323(2) Å, c = 8.95249(19) Å, β = 101.445(3)°, V = 1779.66(7) Å^3, Z = 4, $R_{gt}(F)$ = 0.0410, $wR_{ref}(F^2)$ = 0.1194, T = 279.5 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 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.15 × 0.12 × 0.10 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ:	0.90 mm^{-1}
Diffractometer, scan mode:	XtaLAB synergy, ω
θ_{max}, completeness:	73.3°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9435, 3390, 0.017
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2988
$N(param)_{refined}$:	247
Programs:	Olex2 [1], SHELX [2, 3]

1 Source of material

To a suspension of the coumarin-3-carboxylic acid (0.1 mmol), 5-fluoro-1-methyl-2-oxoindolin-3-yl acetate (0.12 mmol) and triethylamine (0.02 mmol) was added in acetone (1 mL) at room temperature and stirred for 22 h. After the reaction is completed, the solvent was removed in vacuo and purification of the crude product was achieved by flash column chromatography (petroleumether/ethylacetate = 3:1) to afford the product.

2 Experimental details

The carbon-bound hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

3 Comment

Coumarin and its derivatives are important bioactive compounds [4, 5]. They are widely used in the field of pharmacology, such as anti-inflammatory [6, 7], anti-cancer [8, 9], anticoagulation [9] etc. Therefore, the synthesis and modification of coumarins have always been a hot topic in the medical field. The title compound contains one coumarin, a fluorine atom, and one oxindole group. The bond lengths and angles derived from the title structure are within normal ranges. Consistent with those previously reported in the similar structure [10, 11]. The two ketogroups

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
F1	0.25318 (14)	0.48943 (5)	0.89868 (15)	0.0880 (4)
O1	−0.27482 (11)	0.63248 (5)	0.27223 (12)	0.0593 (3)
O2	−0.22559 (15)	0.68950 (7)	0.09750 (13)	0.0789 (4)
O3	0.05967 (10)	0.62115 (5)	0.35314 (11)	0.0540 (3)
O4	0.29259 (14)	0.60648 (10)	0.35627 (18)	0.1078 (6)
O5	0.20614 (17)	0.74271 (8)	0.39178 (17)	0.0987 (5)
N1	0.33814 (14)	0.70258 (8)	0.63438 (16)	0.0677 (4)
C1	−0.13455 (14)	0.64173 (6)	0.55391 (16)	0.0447 (3)
C2	−0.12723 (16)	0.62705 (7)	0.70751 (18)	0.0532 (3)
H2	−0.048804	0.641647	0.792679	0.064*
C3	−0.2344 (2)	0.59121 (9)	0.7355 (2)	0.0671 (4)
H3	−0.228079	0.581911	0.838855	0.080*
C4	−0.3509 (2)	0.56919 (10)	0.6101 (2)	0.0787 (5)
H4	−0.422661	0.544795	0.629214	0.094*
C5	−0.36171 (18)	0.58312 (9)	0.4568 (2)	0.0708 (5)
H5	−0.440150	0.568251	0.372124	0.085*
C6	−0.25443 (15)	0.61947 (7)	0.43067 (17)	0.0507 (3)
C7	−0.02786 (15)	0.68457 (6)	0.51757 (15)	0.0467 (3)
H7	0.005548	0.713569	0.605029	0.056*
C8	−0.11602 (18)	0.71976 (7)	0.36770 (18)	0.0568 (4)
H8A	−0.182553	0.747754	0.394129	0.068*
H8B	−0.046911	0.743343	0.332696	0.068*
C9	−0.20519 (16)	0.68054 (8)	0.23455 (17)	0.0560 (4)
C10	0.11280 (14)	0.65436 (7)	0.49930 (15)	0.0491 (3)
C11	0.20154 (14)	0.61798 (7)	0.64077 (16)	0.0488 (3)
C12	0.17424 (16)	0.56312 (7)	0.69815 (17)	0.0534 (4)
H12	0.089884	0.540517	0.645806	0.064*
C13	0.27882 (19)	0.54323 (8)	0.83790 (19)	0.0625 (4)
C14	0.40464 (19)	0.57516 (9)	0.9182 (2)	0.0689 (5)
H14	0.470419	0.560255	1.012756	0.083*
C15	0.43351 (17)	0.62969 (9)	0.85817 (19)	0.0655 (4)
H15	0.519281	0.651603	0.909760	0.079*
C16	0.33097 (15)	0.65039 (8)	0.71950 (17)	0.0553 (4)
C17	0.4610 (2)	0.74536 (13)	0.6792 (3)	0.1042 (8)
H17A	0.476557	0.759677	0.784737	0.156*
H17B	0.437836	0.778867	0.607021	0.156*
H17C	0.549739	0.725783	0.675519	0.156*
C18	0.22356 (18)	0.70540 (9)	0.49561 (19)	0.0669 (5)
C19	0.16236 (18)	0.60044 (10)	0.2908 (2)	0.0708 (5)
C20	0.0899 (3)	0.57123 (14)	0.1352 (2)	0.1001 (8)
H20A	0.018878	0.541985	0.144624	0.150*
H20B	0.163977	0.551472	0.101484	0.150*
H20C	0.039955	0.601308	0.058761	0.150*

and a fluorine atom in the structure show the following distances 1.1933(18) Å (C_9–O_2), 1.213(2) Å (C_{18}–O_5), 1.364(2) Å (C_{13}–F_1). There are three carbon atoms, in which the only N atoms participate: 1.398(2) Å (N_1–C_{16}), 1.454(2) Å (N_1–C_{17}), and 1.355(2) Å (N_1–C_{18}).

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