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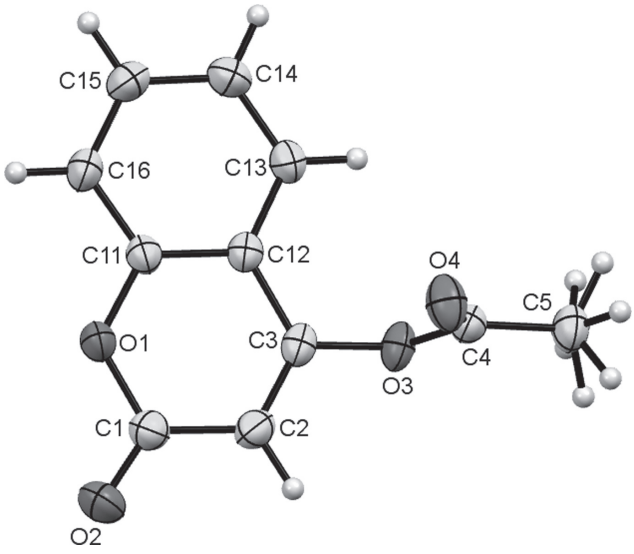
The crystal structure of 2-oxo-2*H*-chromen-4-yl acetate, $C_{11}H_8O_4$



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Abstract

$C_{11}H_8O_4$, monoclinic, $P2_1/c$ (no. 14), $a = 4.5947(2)$ Å, $b = 10.5414(3)$ Å, $c = 19.1611(7)$ Å, $\beta = 94.084(2)°$, $V = 925.70(6)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0376$, $wR_{ref}(F^2) = 0.1109$, $T = 200(2)$ 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 1: Data collection and handling.

Crystal:	Colourless rods
Size:	0.42 × 0.14 × 0.13 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.11 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	28.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	25347, 2302, 0.026
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1942
$N(param)_{refined}$:	136
Programs:	Bruker [1, 2], SHELX [3, 4], PLATON [5], Mercury [6]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.82657(18)	0.83516(8)	0.72062(4)	0.0334(2)
O2	1.1323(2)	0.73321(10)	0.79444(5)	0.0469(3)
O3	0.45735(18)	0.49621(7)	0.65059(5)	0.0338(2)
O4	0.7566(2)	0.47910(9)	0.56357(5)	0.0430(2)
C1	0.9379(3)	0.72398(11)	0.74893(6)	0.0334(3)
C2	0.8118(3)	0.60688(11)	0.72183(6)	0.0328(3)
H2	0.885109	0.528084	0.739571	0.039*
C3	0.5929(2)	0.60842(10)	0.67202(6)	0.0283(2)
C4	0.5611(2)	0.43678(11)	0.59372(6)	0.0298(2)
C5	0.3937(3)	0.31855(12)	0.57719(7)	0.0380(3)
H5A[a]	0.245240	0.307445	0.610963	0.057*
H5B[a]	0.298634	0.324481	0.529871	0.057*
H5C[a]	0.526797	0.245801	0.579852	0.057*
H5D[a]	0.468541	0.277707	0.536161	0.057*
H5E[a]	0.415147	0.260670	0.617253	0.057*
H5F[a]	0.186984	0.339350	0.567272	0.057*
C11	0.6040(2)	0.83662(10)	0.66864(6)	0.0282(2)
C12	0.4775(2)	0.72476(10)	0.64228(6)	0.0267(2)
C13	0.2503(3)	0.73219(11)	0.58980(6)	0.0323(3)
H13	0.160644	0.656976	0.571357	0.039*
C14	0.1574(3)	0.84886(13)	0.56503(7)	0.0402(3)
H14	0.003267	0.854209	0.529372	0.048*
C15	0.2886(3)	0.95932(12)	0.59211(7)	0.0413(3)
H15	0.222576	1.039385	0.574604	0.050*
C16	0.5129(3)	0.95426(11)	0.64388(7)	0.0352(3)
H16	0.602328	1.029711	0.662056	0.042*

[a]Occupancy: 0.5.

Source of material

All commercially available starting materials were used without further purification. Preparation of 2-oxo-chromen-4-yl acetate is similar to the synthesis reported by our group [7]. The white solid was recrystallized from methanol/diethyl ether to obtain colourless rod crystals.

Experimental details

H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U(H) set to 1.2 U_{eq}(C). The H atoms of the methyl group were allowed to rotate with a fixed angle around the C—C bond to best fit the experimental electron density of a 1/1 disorder model (see the figure), with U_{iso}(H) set to 1.5 U_{eq}(C).

Comment

Coumarins are known to be present in a wide range of mammals, microorganisms, as well as in plants, and have been evaluated as therapeutic agents. These compounds have been observed to have multiple biological and pharmaceutical activities [8–10]. Coumarins have been prepared by several methods which include von Pechmann, Perkin, Reformatsky, Knoevenagel and Wittig reactions [11]. In addition, coumarins serve as building blocks in the synthesis of novel biological active compounds. Recently, we reported a coumarin derivative 3-acetyl-bromo-4-hydroxycoumarin [7].

In the crystal structure of the title compound, the methyl group shows rotational disorder with two positions rotated from each other by 60 degrees. The compound exhibits weak intramolecular hydrogen bonding of the C—H···O type. The packing of crystal structure is dominated by weak intermolecular interactions. Additional π–π stacking interactions between rings (centroid distances: 3.323(2) and 3.328(2) Å) further stabilize the molecule.

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