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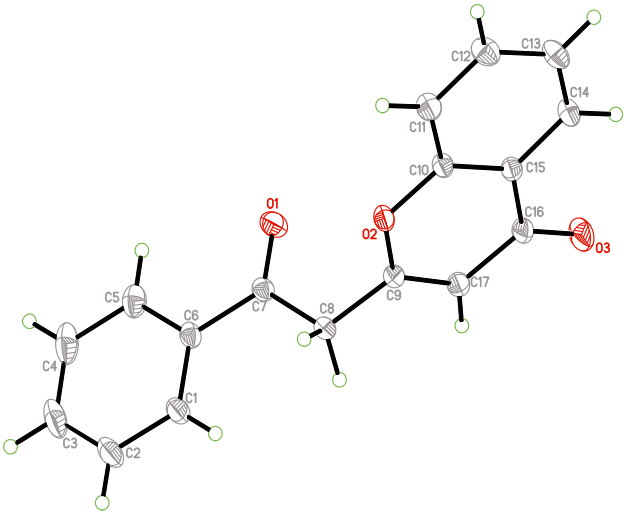
Synthesis and crystal structure of 2-(2-oxo-2-phenylethyl)-4*H*-chromen-4-one, $C_{17}H_{12}O_3$



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Abstract

$C_{17}H_{12}O_3$, monoclinic, $P2_1/c$ (no. 14), a = 9.2911(15) Å, b = 17.525(3) Å, c = 8.5586(15) Å, β = 109.358(7)° V = 1314.8(4) Å^3, Z = 4, $R_{gt}(F)$ = 0.0550, w$R_{ref}(F^2)$ = 0.1789, 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	0.20 × 0.10 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II
θ_{max}, completeness:	35.3°, 98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	20,222, 5866, 0.047
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 4208
$N(param)_{refined}$:	181
Programs:	Bruker [1], SHELX [2, 3]

Source of material

The round-bottomed flask is charged with 2-hydroxyacetophenone, -4*H*-chromen-4-one, carbon disulfide, potassium carbonate and bromoethane and heated to 35 °C with efficient stirring. The reaction was monitored by thin layer chromatography (TLC, 254 nm). After the reaction was completed, the resulting mixture was cooled to room temperature, then poured into ice water and extracted with ethyl acetate, the combined organic layers were washed with anhydrous Na_2SO_4, and the solvent was removed under reduced pressure. The crude product was purified by column chromatography (petroleum ether/ethyl acetate 5:1) to give the title compound as a light yellow solid.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms, with constrained isotropic displacement parameters.

Comment

Chromone derivatives, as a kind of natural products, exist widely in plants and have diverse structural skeletons and biological activities. Some chromones are also important resources for the preparation of fluorescent probes. When a specific metal ion is added, the ring-opening process of the fluorescein part can be observed when the probe recombines with the metal ion, which leads to the appearance of color and fluorescence [4]. It can be used for fluorescence detection of Zn^{2+} [5], Cu^{2+} [6, 7], Mg^{2+} [7–9], Al^{3+} [10], and the preparation of discoloration dyes [7, 11].

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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}
O1	0.48607 (9)	0.32069 (5)	0.65961 (10)	0.03866 (19)
C1	0.56547 (14)	0.43019 (6)	0.33778 (16)	0.0379 (2)
H1A	0.4886	0.4119	0.2453	0.045*
O2	0.14057 (8)	0.28875 (4)	0.45857 (9)	0.02961 (16)
C2	0.66908 (17)	0.48384 (7)	0.3190 (2)	0.0488 (3)
H2A	0.6614	0.5015	0.2140	0.059*
O3	0.18237 (12)	0.05678 (4)	0.45217 (15)	0.0514 (3)
C3	0.78340 (16)	0.51084 (7)	0.4567 (2)	0.0546 (4)
H3A	0.8531	0.5463	0.4438	0.066*
C4	0.79493 (15)	0.48560 (7)	0.6131 (2)	0.0539 (4)
H4A	0.8719	0.5043	0.7051	0.065*
C5	0.69132 (13)	0.43218 (7)	0.63354 (17)	0.0404 (2)
H5A	0.6986	0.4155	0.7390	0.048*
C6	0.57688 (11)	0.40388 (5)	0.49533 (13)	0.02925 (19)
C7	0.47216 (10)	0.34448 (5)	0.52173 (12)	0.02690 (18)
C8	0.35020 (11)	0.31260 (6)	0.36904 (12)	0.02945 (19)
H8A	0.2872	0.3542	0.3087	0.035*
H8B	0.3993	0.2893	0.2971	0.035*
C9	0.25091 (11)	0.25487 (5)	0.41135 (12)	0.02708 (17)
C10	0.03868 (10)	0.24332 (5)	0.50129 (12)	0.02673 (17)
C11	−0.07174 (12)	0.28160 (6)	0.54927 (14)	0.0339 (2)
H11A	−0.0752	0.3346	0.5501	0.041*
C12	−0.17562 (13)	0.23866 (7)	0.59540 (16)	0.0387 (2)
H12A	−0.2491	0.2631	0.6294	0.046*
C13	−0.17188 (13)	0.15882 (7)	0.59168 (16)	0.0389 (2)
H13A	−0.2434	0.1307	0.6219	0.047*
C14	−0.06292 (12)	0.12189 (6)	0.54358 (14)	0.0331 (2)
H14A	−0.0611	0.0689	0.5411	0.040*
C15	0.04583 (10)	0.16409 (5)	0.49806 (11)	0.02678 (17)
C16	0.16658 (12)	0.12662 (5)	0.45059 (13)	0.0313 (2)
C17	0.26758 (12)	0.17865 (5)	0.40546 (13)	0.03060 (19)
H17A	0.3455	0.1590	0.3717	0.037*

There is a molecule in the asymmetric unit (see the figure). This molecule consists of a phenyl ring, chromone skeleton and the acetyl group. The structural analysis of the crystal shows that the benzo moiety (C10/C11/C12/C13/C14/C15) and the oxygen containing heterocyclic moiety (C10/C15/C16/C17/C9/O2) are almost in the same plane; the dihedral angle between them is 0.8°. The dihedral angle between the phenyl ring (C1/C2/C3/C4/C5/C6) and the chromone skeleton is 83°. The bond length and bond angle of the compound are in the normal range [12–14]. No strong hydrogen bond was observed in the compound.

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

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