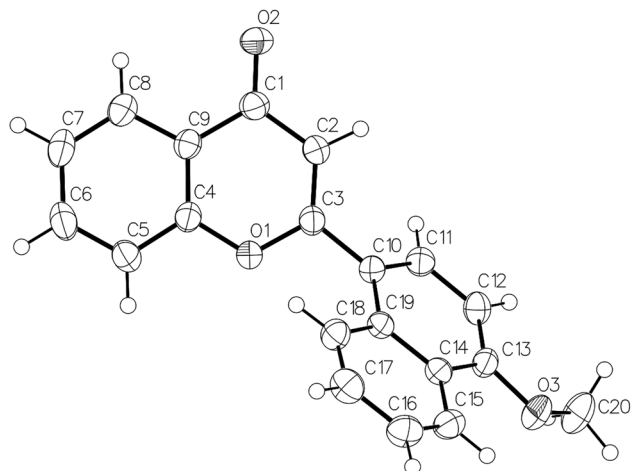


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The crystal structure of 2-(4-methoxynaphthalen-1-yl)-4*H*-chromen-4-one, C₂₀H₁₄O₃



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Abstract

C₂₀H₁₄O₃, triclinic, $P\bar{1}$ (no. 2), $a = 8.3085(10)$ Å, $b = 8.3918(8)$ Å, $c = 12.1359(14)$ Å, $\alpha = 91.691(5)^\circ$, $\beta = 107.708(5)^\circ$, $\gamma = 113.607(5)^\circ$, $V = 727.42(14)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0471$, $wR_{\text{ref}}(F^2) = 0.1235$, $T = 223(2)$ 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.

Source of material

The title compound of 2-(4-methoxynaphthalen-1-yl)-4*H*-chromen-4-one was prepared through a two-step reaction

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.22 × 0.18 × 0.11 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.09 mm ⁻¹
Diffractometer, scan mode:	PHOTON 100 CMOS, φ and ω
θ_{max} , completeness:	28.6°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	30,362, 3696, 0.046
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2605
$N(\text{param})_{\text{refined}}$:	209
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

of chalcone formation followed by intramolecular oxidative cyclization reaction. To a mixture of 1-(2-hydroxyphenyl) ethanone (272 mg, 2 mmol) and 4-methoxy-1-naphth aldehyde (362 mg, 2 mmol) in 40 mL of ethanol, 3 ml of aq. KOH (50%) was added. The reaction mixture was stirred at room temperature for 24 h. After the completion of reaction, the reaction mixture was poured into crushed ice water (70 ml) and acidified with 3 *N* HCl (pH = 3) to give precipitations. After cooling to room temperature, the resulting solid was filtered, washed with water and purified form ethanol to afford chalcone intermediate. To a solution of the intermediate chalcone compound (152 mg, 0.5 mmol) in 5 ml of DMSO, catalytic amount of oxidant iodine (I₂, 0.2 eq.) was added and mixture was refluxed for 4 h at 415 K. Then the mixture was cooled to room temperature. The reaction mixture was poured in crushed ice water (30 ml) and the resulting solid was separated by filtration and was washed with water. Recrystallization of the solid from ethanol solution gave crystals of title compound.

Experimental details

Data collections and reduction were carried out using the Bruker software APEX2 and SAINT including SADABS [1]. Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

Cancer cells are known to exhibit relatively higher ROS levels than normal cells, so an increase in ROS in cancer cells has the effect of killing some types of cancer cells. By

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.7412 (2)	1.1182 (2)	0.35417 (14)	0.0337 (3)
C2	0.6748 (2)	0.9777 (2)	0.25696 (14)	0.0332 (3)
H2	0.7629	0.9547	0.2341	0.040*
C3	0.4921 (2)	0.8789 (2)	0.19809 (13)	0.0306 (3)
O1	0.35604 (15)	0.90392 (15)	0.22624 (10)	0.0378 (3)
C4	0.4064 (2)	1.0325 (2)	0.31913 (13)	0.0311 (3)
C5	0.2595 (2)	1.0474 (2)	0.34379 (15)	0.0412 (4)
H5	0.1346	0.9717	0.2992	0.049*
C6	0.3008 (3)	1.1747 (3)	0.43452 (16)	0.0451 (4)
H6	0.2032	1.1868	0.4522	0.054*
C7	0.4852 (3)	1.2861 (2)	0.50060 (15)	0.0419 (4)
H7	0.5116	1.3732	0.5623	0.050*
C8	0.6299 (2)	1.2698 (2)	0.47627 (14)	0.0373 (4)
H8	0.7545	1.3450	0.5218	0.045*
C9	0.5918 (2)	1.1410 (2)	0.38363 (13)	0.0302 (3)
O2	0.90816 (17)	1.21270 (18)	0.40833 (11)	0.0505 (4)
C10	0.4128 (2)	0.7363 (2)	0.09618 (13)	0.0301 (3)
C11	0.4745 (2)	0.7693 (2)	0.00315 (14)	0.0338 (4)
H11	0.5706	0.8804	0.0078	0.041*
C12	0.3991 (2)	0.6429 (2)	−0.09910 (14)	0.0346 (4)
H12	0.4431	0.6703	−0.1621	0.042*
C13	0.2617 (2)	0.4801 (2)	−0.10625 (13)	0.0315 (3)
C14	0.1989 (2)	0.4344 (2)	−0.00934 (13)	0.0294 (3)
C15	0.0657 (2)	0.2631 (2)	−0.01243 (15)	0.0369 (4)
H15	0.0155	0.1770	−0.0800	0.044*
C16	0.0089 (2)	0.2210 (2)	0.08119 (16)	0.0421 (4)
H16	−0.0788	0.1059	0.0781	0.050*
C17	0.0800 (2)	0.3477 (2)	0.18187 (16)	0.0416 (4)
H17	0.0399	0.3177	0.2461	0.050*
C18	0.2077 (2)	0.5153 (2)	0.18702 (14)	0.0359 (4)
H18	0.2536	0.5997	0.2549	0.043*
C19	0.2726 (2)	0.5645 (2)	0.09243 (13)	0.0289 (3)
O3	0.17446 (17)	0.34911 (15)	−0.20199 (10)	0.0414 (3)
C20	0.2105 (3)	0.3948 (3)	−0.30710 (15)	0.0524 (5)
H20A	0.1807	0.4929	−0.3278	0.079*
H20B	0.1338	0.2941	−0.3702	0.079*
H20C	0.3416	0.4284	−0.2948	0.079*

using this phenomenon to increase ROS in cancer cells, cancer cells can be selectively killed [5, 6]. Piperlongumin derivatives containing α , β -unsaturated carbonyl groups in the scavenge glutathione (GSH) from cancer cells, may cause an increase in intracellular ROS [7–9]. The α , β -unsaturated carbonyl group play an important role in removing GSH through the Michael addition reaction with the thiol group of GSH. Flavones are compounds containing an α , β -unsaturated carbonyl group in a chromone ring. In order to develop a compound with more effective ROS

generating activity, the title flavone compound was synthesized based on our previous studies [10–12].

In the title compound, the C2=C3 double bond adopts a *Z* configuration (see the figure), which was defined by the dihedral angle of $-0.2(3)^\circ$ for C1–C2–C3–O1. The naphthylene ring ([C11–C19]; r.m.s. deviations 0.028 Å) is tilted at an angle of $53.2(2)^\circ$ with respect to the chromone ring ([C1–C3, O1, C4–C9]; r.m.s. deviations 0.007 Å). The methoxy group in naphthylene ring is slightly twisted from the ring [C12–C13–O3–C20 = $-9.6(2)^\circ$]. Bond lengths and angles are all in the expected ranges [13, 14].

In the crystal, pairs of weak C6–H6...O2 and C17–H17...O2 hydrogen bonds link the molecules into chains propagating along the *b*-axis direction.

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