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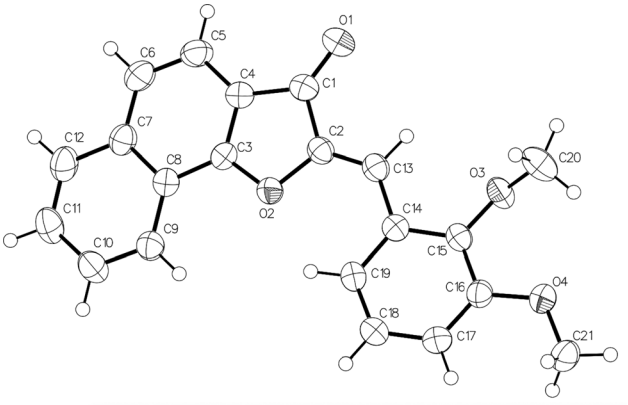
The crystal structure of (*Z*)-2-(2,3-dimethoxybenzylidene)naphtho[1,2-*b*]furan-3(2*H*)-one, $C_{21}H_{16}O_4$



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Abstract

$C_{21}H_{16}O_4$, monoclinic, $P2_1/n$ (no. 14), a = 13.531(6) Å, b = 8.390(3) Å, c = 14.288(5) Å, β = 95.322(15)°, V = 1615.1(11) Å^3, Z = 4, $R_{gt}(F)$ = 0.0588, $wR_{ref}(F^2)$ = 0.1795, T = 243(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:	Yellow block
Size:	0.35 × 0.19 × 0.12 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Photon III M14, φ and ω
θ_{max}, completeness:	28.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	22,301, 3996, 0.104
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2950
$N(param)_{refined}$:	228
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

Source of material

The title compound was obtained through a two-step reaction with the chalcone intermediate. To a mixture of 1′-hydroxy-2′-acetonaphthone (186 mg, 1 mmol) and 2,3-dimethoxybenzaldehyde (166 mg, 1 mmol) in 25 mL of ethanol, 2 mL of aq. KOH (40%) was added. The reaction mixture was stirred at room temperature for 30 h. After the completion of reaction, the reaction mixture was poured into ice water (40 mL) and was acidified with 3 N HCl (pH = 3) to give precipitations. The resulting solid was filtered, washed with water and purified form ethanol to afford pure chalcone intermediate. Chalcone compound (133 mg, 0.4 mmol) was dissolved in 3.5 mL of pyridine and then mercury (II) acetate (159 mg, 0.5 mmol) was added to a reaction solution. The reaction mixture was heated at 383 K for 2 h. The reaction mixture was cooled to room temperature. It gave a precipitate which was filtered under vacuum. To obtain single crystal of the title compound, the crude solid was recrystallized in ethanol.

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

Aurone is a 5-membered ring analogue of flavonone with a six-membered ring. While flavonone has an *endo*-cyclic

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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}
C1	0.44741 (14)	0.1623 (2)	0.46621 (12)	0.0357 (4)
C2	0.50026 (13)	0.2727 (2)	0.53500 (12)	0.0344 (4)
C3	0.34777 (12)	0.24218 (19)	0.57697 (12)	0.0329 (4)
C4	0.34783 (13)	0.1507 (2)	0.49765 (12)	0.0350 (4)
C5	0.26188 (15)	0.0662 (2)	0.46344 (14)	0.0445 (5)
H5	0.2612	0.0039	0.4087	0.053*
C6	0.18013 (15)	0.0771 (2)	0.51164 (15)	0.0477 (5)
H6	0.1222	0.0221	0.4891	0.057*
C7	0.17957 (14)	0.1695 (2)	0.59558 (14)	0.0398 (4)
C8	0.26597 (12)	0.25499 (19)	0.63075 (12)	0.0348 (4)
C9	0.26689 (14)	0.3431 (2)	0.71501 (14)	0.0407 (4)
H9	0.3244	0.3986	0.7382	0.049*
C10	0.18381 (16)	0.3475 (2)	0.76279 (16)	0.0482 (5)
H10	0.1846	0.4054	0.8192	0.058*
C11	0.09771 (16)	0.2661 (3)	0.72817 (17)	0.0514 (5)
H11	0.0408	0.2708	0.7612	0.062*
C12	0.09554 (15)	0.1800 (3)	0.64707 (17)	0.0488 (5)
H12	0.0369	0.1265	0.6250	0.059*
C13	0.59487 (13)	0.3185 (2)	0.53678 (13)	0.0357 (4)
H13	0.6303	0.2741	0.4895	0.043*
C14	0.65183 (13)	0.42726 (19)	0.60096 (12)	0.0332 (4)
C15	0.75494 (13)	0.4328 (2)	0.59891 (12)	0.0346 (4)
C16	0.81202 (13)	0.5389 (2)	0.65694 (12)	0.0364 (4)
C17	0.76600 (14)	0.6373 (2)	0.71726 (14)	0.0419 (4)
H17	0.8039	0.7099	0.7557	0.050*
C18	0.66408 (14)	0.6294 (2)	0.72118 (13)	0.0419 (4)
H18	0.6336	0.6955	0.7631	0.050*
C19	0.60726 (13)	0.5258 (2)	0.66439 (13)	0.0373 (4)
H19	0.5383	0.5209	0.6681	0.045*
C20	0.85538 (19)	0.3787 (3)	0.47303 (16)	0.0585 (6)
H20A	0.8207	0.4667	0.4404	0.088*
H20B	0.8642	0.2932	0.4289	0.088*
H20C	0.9198	0.4148	0.5005	0.088*
C21	0.97169 (15)	0.6384 (3)	0.70991 (15)	0.0494 (5)
H21A	0.9539	0.7478	0.6942	0.074*
H21B	1.0411	0.6213	0.7011	0.074*
H21C	0.9608	0.6179	0.7750	0.074*
O1	0.48285 (10)	0.09806 (16)	0.40039 (9)	0.0452 (4)
O2	0.43599 (9)	0.31936 (14)	0.60111 (9)	0.0366 (3)
O3	0.79900 (10)	0.32251 (15)	0.54507 (10)	0.0434 (3)
O4	0.91200 (10)	0.53332 (18)	0.65050 (10)	0.0480 (4)

α, β-unsaturated carbonyl group in the chromenone structure, aurone has a benzofuranone structure and an *exo*-cyclic α, β-unsaturated carbonyl group. The α, β-unsaturated carbonyl group reacts with the thiol group of glutathione (GSH) as a Michael acceptor to reduce the intracellular GSH concentration [5–7]. In normal cells, GSH reduces ROS, making cell function beneficial [8]. Since cancer cells have a higher concentration of ROS than normal cells [9], when GSH that captures ROS decreases, the rapid increase in ROS leads to death of cancer cells [10]. The development of anticancer drugs using this principle is actively underway [11]. As an extension of research on compounds with more effective ROS generating activity [12–14], the title aurone compound was synthesized.

In the title compound shown in the figure, the C2=C13 double bond adopts a *Z*-configuration, which was defined by the dihedral angle of –2.82(3)° for O2–C2–C13–C14. One methoxy group at the ortho position (C-16) of the benzene ring is nearly coplanar with the ring (C17–C16–O4–C21 [0.7(3)°]), while the other methoxy group at the meta position (C-15) is highly twisted in the ring (C16–C15–O3–C20 [–64.1(2)°]). The naphthlene ring ([C3–C12]; r.m.s. deviations 0.016 Å) is tilted at an angle of 16.80(2)° with respect to the benzene ring ([C14–C19]; r.m.s. deviations 0.008 Å). In the crystal, pairs of weak C11–11···O1 and C21–H21···O1 hydrogen bonds link the molecules into chains propagating along the *ac*-plane.

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

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