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Synthesis and crystal structure of 4-(3-acetyl-5-(thiophen-2-yl)-2,3-dihydro-1,3,4-oxadiazol-2-yl)-7-(diethylamino)-2H-chromen-2-one, C₂₁H₂₁N₃O₄S

Table 1: Data collection and handling.

Crystal:	Size 0.35 × 0.34 × 0.30 mm
Wavelength:	Co K α radiation (1.54184 Å)
μ :	1.64 mm ⁻¹
Diffractometer, scan mode:	Xcalibur, ω -scans
$\theta_{\max}$, completeness:	71°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6696, 3974, 0.020
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3247
$N(\text{param})_{\text{refined}}$:	284
Programs:	CrysAlis ^{PRO} [1], OLEX2 [2], SHELX [3, 4]

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Abstract

C₂₁H₂₁N₃O₄S, triclinic, $P\bar{1}$ (no. 2), $a = 9.4256(9)$ Å, $b = 10.1906(9)$ Å, $c = 11.6539(9)$ Å, $\alpha = 101.896(7)^\circ$, $\beta = 104.770(8)^\circ$, $\gamma = 93.271(8)^\circ$, $V = 1052.01(16)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0469$, $wR_{\text{ref}}(F^2) = 0.1354$, $T = 293(2)$ 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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Source of material

In a representative experiment 7-(diethylamino)-4-methyl-2H-chromen-2-one (1.0 mmol) reacted with SeO₂ (1.1 mmol) in dioxane (10 mL) at 80 °C to give the aldehyde. This intermediate aldehyde was reacted with 2-thiophenecarboxylic acid hydrazide (1.0 mmol) in acetic anhydride (5 mL) under reflux. Suitable colorless block crystals of this compound were recrystallised from methanol.

Experimental details

The structure was solved with the Olex2 program [2] as an interface together with the SHELXT and SHELXL programs [3, 4]. Hydrogen atoms were treated using a riding model.

Discussion

Coumarins are heterocyclic compounds with benzopyranone, which diversified bioactivities such as antimicrobial activity [5], anti-inflammatory [6], and anti-tumor [7]. Recently, we prepared several coumarin-based derivatives by introduction of multiple substituents [8–11]. Here we introduced a substituted 1,3,4-oxadiazol moiety to find a compound with potential biological activity.

As shown in the figure, there is one crystallographically independent molecule in the asymmetric unit, and all bond lengths are in normal ranges [8–12]. The compound contains one coumarin group, one oxadiazole ring and one thiophene

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

Atom	x	y	z	<i>U</i> _{iso} ^a / <i>U</i> _{eq}
S1 ^a	0.3209(2)	0.86736(10)	0.54845(15)	0.0699(4)
S2 ^b	0.5752(10)	0.9964(8)	0.7440(9)	0.0658(12)
O1	0.49362(13)	1.25150(11)	0.67409(11)	0.0477(3)
O2	0.27241(19)	1.48231(15)	0.45825(14)	0.0722(4)
O3	0.35405(12)	1.64767(12)	0.93678(11)	0.0465(3)
O4	0.11499(14)	1.58523(16)	0.86756(14)	0.0648(4)
N1	0.28204(18)	1.16358(15)	0.52977(14)	0.0516(4)
N2	0.30037(17)	1.30349(14)	0.53937(14)	0.0483(4)
N3	0.86110(17)	1.81957(18)	1.09744(16)	0.0586(4)
C1	0.43234(18)	1.36717(16)	0.63566(15)	0.0413(4)
H1	0.5005	1.4136	0.6021	0.050*
C2	0.40280(17)	1.46273(15)	0.74320(14)	0.0368(3)
C3	0.26431(18)	1.47354(18)	0.75289(16)	0.0453(4)
H3	0.1858	1.4189	0.6940	0.054*
C4	0.23475(18)	1.56782(19)	0.85243(17)	0.0463(4)
C5	0.49654(16)	1.63830(16)	0.92865(14)	0.0375(3)
C6	0.52568(16)	1.54452(15)	0.83358(14)	0.0354(3)
C7	0.67543(17)	1.54263(16)	0.83497(15)	0.0407(4)
H7	0.7004	1.4807	0.7747	0.049*
C8	0.78497(17)	1.62892(18)	0.92206(16)	0.0443(4)
H8	0.8827	1.6230	0.9209	0.053*
C9	0.75276(18)	1.72769(18)	1.01458(15)	0.0432(4)
C10	0.60394(18)	1.72762(17)	1.01599(15)	0.0436(4)
H10	0.5783	1.7885	1.0765	0.052*
C11	1.0159(2)	1.8213(2)	1.0982(2)	0.0730(7)
H11A	1.0628	1.9137	1.1263	0.088*
H11B	1.0221	1.7881	1.0157	0.088*
C12	1.0963(3)	1.7386(4)	1.1769(4)	0.1264(15)
H12A	1.0960	1.7746	1.2597	0.190*
H12B	1.1963	1.7397	1.1715	0.190*
H12C	1.0489	1.6474	1.1507	0.190*
C13	0.8266(2)	1.9275(2)	1.18590(19)	0.0634(5)
H13A	0.9145	1.9597	1.2532	0.076*
H13B	0.7517	1.8909	1.2181	0.076*
C14	0.7726(3)	2.0446(3)	1.1348(3)	0.0891(8)
H14A	0.8484	2.0850	1.1072	0.134*
H14B	0.7488	2.1102	1.1970	0.134*
H14C	0.6860	2.0137	1.0676	0.134*
C15	0.2308(2)	1.3645(2)	0.45148(18)	0.0563(5)
C16	0.1037(3)	1.2825(3)	0.3536(2)	0.0835(8)
H16A	0.1360	1.2017	0.3145	0.125*
H16B	0.0665	1.3342	0.2945	0.125*
H16C	0.0269	1.2589	0.3887	0.125*
C17	0.39545(19)	1.14207(17)	0.60778(16)	0.0441(4)
C18	0.4296(2)	1.01254(18)	0.63274(18)	0.0509(4)
C19 ^a	0.5419(11)	0.9900(11)	0.7199(10)	0.076(3)
H19 ^a	0.6118	1.0574	0.7745	0.092*
C20	0.5414(3)	0.8439(3)	0.7188(3)	0.0794(7)
H20 ^a	0.6095	0.8064	0.7720	0.095*
H20A ^b	0.6025	0.7967	0.7678	0.095*
C21	0.4278(4)	0.7755(2)	0.6294(3)	0.0810(7)
H21 ^a	0.4092	0.6820	0.6130	0.097*
H21A ^b	0.3974	0.6833	0.6054	0.097*
C22 ^b	0.363(2)	0.8880(9)	0.5784(17)	0.068(3)
H22 ^b	0.2797	0.8722	0.5119	0.082*

Occupancies: ^a = 0.765(3), ^b = 0.235(3).

ring. The short C—N bond lengths of 1.277(2) Å (N1—C17) suggests a localized double bond, and the C—N bond lengths of 1.457(2) Å (N2—C1) suggests a localized single bond.

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