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Crystal structure of *N'*-((1*E*,2*E*)-4-(7-methoxy-2-oxo-2*H*-chromen-8-yl)-2-methylbut-2-en-1-ylidene)-4-methylbenzenesulfonohydrazide,**C₂₂H₂₂O₅N₂S** **Table 1:** Data collection and handling.

Crystal:	Block, clear light colorless
Size:	0.27 × 0.25 × 0.23 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.19 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω -scans
$\theta_{\max}$, completeness:	28.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	49042, 5048, 0.038
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4120
$N(\text{param})_{\text{refined}}$:	274
Programs:	CrysAlis ^{PRO} [1], OLEX2 [2], SHELX [3, 4]

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Abstract

C₂₂H₂₂O₅N₂S, monoclinic, $P2_1/n$ (no. 14), $a = 8.1080(2)$ Å, $b = 14.4763(4)$ Å, $c = 18.2819(5)$ Å, $\beta = 95.270(3)^\circ$, $V = 2136.75(10)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0488$, $wR_{\text{ref}}(F^2) = 0.1220$, $T = 291$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

In a representative experiment 7-methoxy-8-(3-methylbut-2-en-1-yl)-2*H*-chromen-2-one (1.0 mmol) reacted with SeO₂ (1.1 mmol) in dioxane (10 mL) at 80 °C to afford the aldehyde, and then reacted with 4-methylphenylsulfonyl hydrazide (1.0 mmol), and two drops of HOAc in EtOH (10 mL) at room temperature. Suitable colorless block crystals of this

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.14406(6)	0.03124(3)	0.70599(2)	0.04709(14)
O1	0.91781(15)	0.31548(9)	0.56626(7)	0.0482(3)
O2	1.07076(18)	0.19479(11)	0.54633(8)	0.0679(4)
O3	0.61834(18)	0.57433(9)	0.62844(7)	0.0569(3)
O4	0.21414(19)	0.00325(11)	0.77665(7)	0.0648(4)
O5	−0.02880(17)	0.02218(11)	0.68566(8)	0.0635(4)
N1	0.35034(18)	0.16385(10)	0.71085(8)	0.0458(3)
N2	0.18078(18)	0.14265(11)	0.69929(8)	0.0483(4)
H2	0.1361	0.1599	0.6555	0.058*
C1	0.84101(19)	0.39695(12)	0.54385(9)	0.0413(4)
C2	0.7664(2)	0.44602(12)	0.59680(9)	0.0410(4)
C3	0.6918(2)	0.52955(12)	0.57493(9)	0.0441(4)
C4	0.6931(2)	0.56175(13)	0.50301(10)	0.0488(4)
H4	0.6445	0.6182	0.4897	0.059*
C5	0.7657(2)	0.51032(13)	0.45221(10)	0.0492(4)
H5	0.7648	0.5320	0.4043	0.059*
C6	0.8412(2)	0.42589(13)	0.47088(9)	0.0449(4)
C7	0.9119(2)	0.36615(15)	0.42026(10)	0.0533(5)
H7	0.9085	0.3827	0.3710	0.064*
C8	0.9832(2)	0.28626(16)	0.44286(11)	0.0571(5)
H8	1.0247	0.2469	0.4087	0.069*
C9	0.9963(2)	0.26071(14)	0.51897(11)	0.0525(5)
C10	0.5425(3)	0.66115(15)	0.61270(13)	0.0630(5)
H10A	0.4604	0.6550	0.5717	0.094*
H10B	0.6250	0.7050	0.6010	0.094*
H10C	0.4908	0.6823	0.6548	0.094*
C11	0.7547(2)	0.40887(13)	0.67360(9)	0.0459(4)
H11A	0.8440	0.3654	0.6860	0.055*
H11B	0.7653	0.4592	0.7088	0.055*

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C12	0.5912(2)	0.36169(12)	0.67713(9)	0.0408(4)
H12	0.4983	0.3973	0.6630	0.049*
C13	0.5613(2)	0.27558(12)	0.69775(9)	0.0422(4)
C14	0.6913(3)	0.20661(17)	0.72406(16)	0.0816(8)
H14A	0.6913	0.1567	0.6895	0.122*
H14B	0.6679	0.1830	0.7711	0.122*
H14C	0.7979	0.2360	0.7284	0.122*
C15	0.3899(2)	0.24489(12)	0.69237(9)	0.0420(4)
H15	0.3069	0.2857	0.6749	0.050*
C16	0.2544(2)	−0.02485(11)	0.64047(9)	0.0413(4)
C17	0.4185(3)	−0.04798(14)	0.65727(11)	0.0559(5)
H17	0.4701	−0.0365	0.7039	0.067*
C18	0.5055(3)	−0.08842(15)	0.60421(13)	0.0642(6)
H18	0.6160	−0.1041	0.6156	0.077*
C19	0.4315(3)	−0.10593(13)	0.53475(11)	0.0566(5)
C20	0.2683(3)	−0.08159(15)	0.51911(11)	0.0585(5)
H20	0.2169	−0.0926	0.4723	0.070*
C21	0.1789(2)	−0.04124(14)	0.57120(10)	0.0519(5)
H21	0.0685	−0.0252	0.5596	0.062*
C22	0.5264(4)	−0.14976(17)	0.47656(14)	0.0820(8)
H22A	0.6371	−0.1635	0.4969	0.123*
H22B	0.5303	−0.1079	0.4360	0.123*
H22C	0.4722	−0.2058	0.4597	0.123*

compound were then obtained after recrystallization 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

Recent studies show that introduction of hydrazone fragments into natural products could display the potent biological activity [5, 6]. In order to discover more compounds with diverse bioactivities, we prepared a coumarin-based hydrazone derivative by introduction of a hydrazone moiety.

The asymmetric unit of the title compound consists of one formula unit. Geometric parameters of the title structure are in the usual ranges. The compound contains one methoxy

group, one lactone and two methyl-groups, one C=C double bond and one C=N double bond. The length of C=C double bond (atoms C11–C12) was 1.384(3) Å, longer than the length of C–N double bond (atoms N1–C13) with 1.270(2) Å. In addition, the configuration of the double bonds are in type *E*, similar to related hydrazone structures, which were recently reported [7, 8].

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