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Synthesis and crystal structure of 2-(2-oxo-2-(thiophen-2-yl)ethyl)-4*H*-chromen-4-one, $C_{15}H_{10}O_3S$

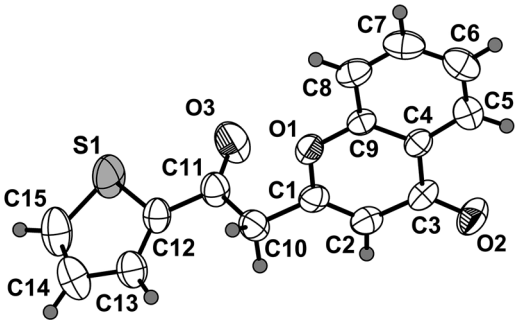

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.30 × 0.20 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.25 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.6°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	22478, 5933, 0.082
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3359
$N(param)_{refined}$:	343
Programs:	Bruker [1], SHELX [2, 3]

https://doi.org/10.1515/ncrs-2022-0371
Received July 22, 2022; accepted August 26, 2022;
published online September 12, 2022

Abstract

$C_{15}H_{10}O_3S$, monoclinic, $P2_1/c$ (no. 14), a = 12.391(5) Å, b = 21.034(8) Å, c = 10.777(4) Å, β = 113.589(7)°, V = 2574.1(16) Å^3, Z = 8, $R_{gt}(F)$ = 0.0704, $wR_{ref}(F^2)$ = 0.1988, T = 296(2) K.

CCDC no.: 2190717

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

In a 50 ml flask, a mixture of *o*-hydroxyacetophenone, carbon disulfide, potassium carbonate and bromoethane were stirred in DMSO at 40 °C. The reaction was monitored by silica gel thin layer chromatography (TLC, 254 nm). After completion of the reaction, the mixture was poured into ice water, stirred for 20 min and the yellow intermediate 2-(ethylthio)-4*H*-chromen-4-one was obtained by filtration. A solution of this intermediate, 2-acetylthiophene and t–BuOK in DMSO (10 ml) was stirred at room temperature for 2–3 h and then separated by chromatographic column (PE:EA = 5:1) to obtain the title product. A share of 20 mg of the obtained white title compound was dissolved in 2 ml of methanol to obtain colorless crystals after two days by slow evaporation.

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, has been proved to be related to biological activity [4–7]. Most research on chromone so far mainly focuses on 2-mono, 3-mono and substituted compounds to show different effects on biological activity.

The asymmetric unit contains two crystallographically independent molecules, and one of these molecules is shown in the figure. In the molecule, the benzene ring (C4/

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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}
S1	−0.02165 (9)	0.58715 (6)	0.34984 (12)	0.0787 (4)
O1	0.38585 (18)	0.45925 (10)	0.7059 (2)	0.0451 (5)
O2	0.7018 (2)	0.55179 (12)	0.8538 (3)	0.0627 (7)
O3	0.1955 (2)	0.56075 (17)	0.5979 (3)	0.0834 (10)
C1	0.4113 (3)	0.50569 (15)	0.6340 (3)	0.0428 (7)
C2	0.5125 (3)	0.53832 (16)	0.6811 (3)	0.0456 (8)
H2A	0.5231	0.5709	0.6287	0.055*
C3	0.6054 (3)	0.52437 (15)	0.8108 (3)	0.0445 (8)
C4	0.5795 (3)	0.47294 (14)	0.8868 (3)	0.0388 (7)
C5	0.6593 (3)	0.45248 (17)	1.0130 (3)	0.0512 (8)
H5A	0.7325	0.4721	1.0527	0.061*
C6	0.6314 (4)	0.40386 (18)	1.0794 (4)	0.0615 (10)
H6A	0.6856	0.3905	1.1636	0.074*
C7	0.5221 (4)	0.37463 (18)	1.0210 (4)	0.0619 (10)
H7A	0.5039	0.3416	1.0666	0.074*
C8	0.4408 (3)	0.39345 (16)	0.8975 (4)	0.0508 (8)
H8A	0.3677	0.3736	0.8588	0.061*
C9	0.4702 (3)	0.44313 (14)	0.8312 (3)	0.0383 (7)
C10	0.3155 (3)	0.51322 (18)	0.4964 (3)	0.0520 (9)
H10A	0.2954	0.4716	0.4547	0.062*
H10B	0.3453	0.5385	0.4415	0.062*
C11	0.2042 (3)	0.54443 (17)	0.4945 (4)	0.0513 (8)
C12	0.1096 (3)	0.55526 (16)	0.3611 (3)	0.0481 (8)
C13	0.1066 (3)	0.54333 (17)	0.2350 (4)	0.0556 (9)
H13A	0.1694	0.5262	0.2197	0.067*
C14	−0.0023 (4)	0.5599 (2)	0.1304 (4)	0.0715 (12)
H14A	−0.0196	0.5548	0.0387	0.086*
C15	−0.0779 (4)	0.5840 (2)	0.1791 (5)	0.0773 (13)
H15A	−0.1535	0.5976	0.1243	0.093*
S2	0.02856 (8)	0.27082 (5)	0.32347 (12)	0.0654 (3)
O4	0.47417 (19)	0.28071 (11)	0.2899 (2)	0.0490 (6)
O5	0.5275 (3)	0.12748 (12)	0.0881 (3)	0.0671 (7)
O6	0.2282 (2)	0.21088 (13)	0.2878 (4)	0.0851 (10)
C16	0.3850 (3)	0.25461 (16)	0.1814 (3)	0.0448 (7)
C17	0.4002 (3)	0.20556 (16)	0.1126 (4)	0.0495 (8)
H17A	0.3360	0.1909	0.0378	0.059*
C18	0.5116 (3)	0.17412 (16)	0.1489 (4)	0.0494 (8)
C19	0.6080 (3)	0.20308 (16)	0.2661 (3)	0.0463 (8)
C20	0.7238 (3)	0.18005 (19)	0.3170 (4)	0.0640 (11)
H20A	0.7423	0.1453	0.2758	0.077*
C21	0.8102 (4)	0.2081 (2)	0.4268 (5)	0.0743 (12)
H21A	0.8865	0.1921	0.4608	0.089*
C22	0.7832 (4)	0.2602 (2)	0.4867 (5)	0.0729 (12)
H22A	0.8425	0.2793	0.5604	0.088*
C23	0.6720 (3)	0.2845 (2)	0.4406 (4)	0.0610 (10)
H23A	0.6548	0.3196	0.4817	0.073*
C24	0.5851 (3)	0.25505 (16)	0.3302 (3)	0.0462 (8)
C25	0.2731 (3)	0.28920 (17)	0.1552 (4)	0.0521 (9)
H25A	0.2221	0.2858	0.0599	0.063*
H25B	0.2904	0.3339	0.1761	0.063*
C26	0.2088 (3)	0.26337 (16)	0.2388 (4)	0.0500 (8)
C27	0.1198 (3)	0.30401 (16)	0.2549 (3)	0.0458 (8)
C28	0.0966 (3)	0.36809 (17)	0.2296 (4)	0.0548 (9)
H28A	0.1376	0.3948	0.1950	0.066*
C29	0.0033 (4)	0.3876 (2)	0.2626 (5)	0.0726 (12)
H29A	−0.0256	0.4290	0.2505	0.087*
C30	−0.0404 (3)	0.3405 (2)	0.3138 (4)	0.0690 (11)
H30B	−0.1023	0.3461	0.3408	0.083*

C5/C6/C7/C8/C9) and the pyran ring (C1/C2/C3/C4/C9/O1) are approximately in the same plane and their dihedral angle is only 1.3°, while the thiophene ring (C12/C13/C14/C15/S1) and the 4H-chromen-4-one ring (C1/C2/C3/C4/C5/C6/C7/C8/C9/O1) present a dihedral angle of about 68.6°. This structure may increase its likelihood of interactions with bioactive molecules. The bond lengths and angles of the title compound are similar to those given in the references [8–11].

Acknowledgments: This work was supported by Nanjing Senega Medical Technology Co. Ltd., also, we appreciate Wang huaqin in Nanjing University and Wu wenyuan in Nanjing Tech University for some data analysis.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was supported by Nanjing Senega Medical Technology Co. Ltd.

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

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