J. Chosun Natural Sci.
Vol. 7, No. 2 (2014) pp. 92 – 102
http://dx.doi.org/10.13160/ricns.2014.7.2.92

Crystal and Molecular Structure of Methyl 12-(3-bromophenyl)-9-[(4-methylbenzene)sulfonyl]-22-oxo-13,21-dioxa-9-azapentacyclo [12.8.0.0^{2,11}.0^{3,8}.0^{15,20}]docosa-1(14),3,5,7,15(20),16,18-heptaene-11-carboxylate

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Abstract

The crystal structure of the title compounds with both coumarin and sulfonamide moieties were examined. These two groups have very special for their pharmaceutical and medicinal properties have been determined from single crystal X-ray diffraction data. In the title compound crystallizes in the monoclinic space group C2/c with unit cell dimension a = 28.633(3) Å, b= 9.3215(7) Å and c= 24.590(2) Å [alpha & gamma=90° beta= 115.976(3)°]. In the structure The S1 atom shows a distorted tetrahedral geometry, with O1—S1—O2 [119.74 (2)°] and N1-S1-C5 [105.57(1)°] angles deviating from ideal tetrahedral values are attributed to the Thrope-Ingold effect. The sum of bond angles around N1 (316.2(1)°) indicates that N1 is in sp2 hybridization. The Pyridine ring adopts boat conformation and pyran rings adopt a sofa conformation. The carboxylate group of atoms were disordered over two positions with site occupancy factors 0.598 (9):0.402 (9). Crystal structure and packing is stabilized by C-H…O intra and inter molecular hydrogen bond interactions.

Key words: Pyrone, Pyran, Coumarin, Sulfonamide, Single Crystal Structure, X-ray Diffraction

1. Introduction

In our previous study, crystal structure of one of the coumarin derivatives have been reported[1]. Here we have analyzed a compound with both coumarin and sulfonamide moieties were examined. These two groups have very special for their pharmaceutical and medicinal properties. Hence, a practical method for the preparation of such compounds is of great interest in synthetic organic chemistry. Coumarin derivatives alone are known to be an interesting class of natural or synthetic compounds, whose biological activity varies according to the substitutes on the benzopyran ring and their antibacterial, antifungal, antitumor, anti-HIV, and anti-inflammatory. These derivatives show strong activity against cancer cell lines[2] and exhibit monoamine oxidase inhibitory activity[3]. Antiulcer activity of some naturally occurring pyranocoumarins has been reported[4]. It also shows specific inhibitory activity against Hepatitis B virus, anti-filarial[5], cytotoxic activites[6] and Mycobacterium tuberculosis[7]. One of the natural source coumarin derivatives named, Chalepin, inhibits the enzyme glyceraldehyde-3-phosphate dehydrogenase of Trypanosoma cruzi and its complex structure has deposited in Protein Data Bank (1K3T)[8] and also sulfonamide based derivatives has wide range biological activity and many of the sulfo based drugs are currently available in the market. In view of this biological importance, X-ray diffraction studies have been undertaken for one coumarin-sulonamide derivatives and here we report the crystal structure of the title compound.

2. Material and Methods

With the collaboration of Organic Chemistry Department at University of Madras, we obtained the title compound and crystallized by simple solvent slow

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(Received : May 27, 2014, Revised : June 13, 2014, Accepted : June 25, 2014)

evaporation method. Three rounds of crystallization trials, diffraction crystals.

The diffraction quality crystals after screening its size and stability, X-ray diffraction data collection was done at University of Madras – Technology Business Incubator facility. The data was reduced with appropriate corrections at the facility and the error free data was taken for structure determination.

Using WinGx suite, structure determination was done using SHELXS97 with Direct Methods protocols. After manual inspections and corrections, Isotropic refinement followed by anisotropic refinements was carried out. With the satisfied model (agreeable R factor, Goodness of Fit and other) hydrogen atoms were geometrically fixed and after the final refinement the R factor is 5.0%.

3. Experimental

3.1. Synthesis of the Title Compound

A well grounded mixture of methyl (2*E*)-3-(3-bromophenyl)-2-{[*N*-(2-formylphenyl)(4-methylbenzene) sulfonamido]methyl}prop-2-enoate (0.53 g, 1 mmol) and 4-hydroxy-2*H*-chromen-2-one, (0.15 g, 1 mmol) was placed in a round bottom flask and melted at 180°C for 1 h. After completion of the reaction as indicated by TLC, the crude product was washed with 5 mL of ethylacetate and hexane mixture (1:49 ratio) which successfully provided the pure product as colorless solid in 94% yield. Since the compound has not yield the diffraction quality crystals initially, the compound has been recrystallized with ethyl acetate by slow evaporation method to get better quality single crystals.

3.2. X-Ray Crystallography

For the crystal structure determination, the single crystal of the compound $C_{34}H_{26}BrNO_7S$ was used for data collection on a Bruker Kappa APEXII CCD diffractometer[9]. The MoK″ radiation of wavelength, (# = 0.71073 Å) and multi-scan technique for absorption correction were used for data collection. The lattice parameters were determined by the least-squares methods on the basis of all reflections with F2>2$ (F2). The structures were solved by direct methods using SHELXS-97 and refined by a full-matrix least-squares procedure using the program SHELXL-97[10,11]. H atoms were positioned geometrically and refined using

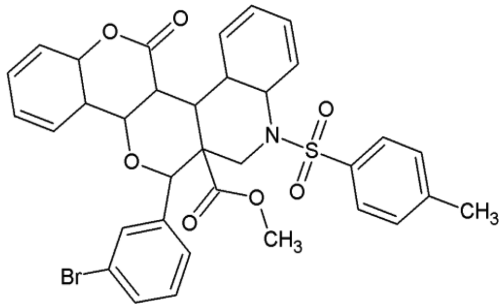

Fig. 1. Schematic diagram of the molecule.

a riding model, fixing the aromatic C-H distances at 0.93 Å [Uiso(H) = 1.2 Ueq (C)]. The softwares used for Molecular graphics are ORTEP-3 for Windows[12] and PLATON[13]. The software used to prepare material for publication is WinGX publication routines [14]. Experimental data are listed in Table 1. CCDC reference number: 902987. Fig. 1 shows schematic diagram of the molecule and molecular structure of the title compound along with the atom numbering scheme is depicted in Fig. 2 and a packing diagram is shown in Fig. 3. Table 1 shows the crystal data and crystal refinement. Table 2 gives the atomic coordinates, Table 3 describes the bond lengths and angles; Table 4 shows anisotropic displacement parameters, Table 5 shows the torsion angles and Table 6 shows hydrogen-bond geometry.

4. Results and Discussion

Title compound crystallizes in the monoclinic centrosymmetric space group C2/c with Z = 8. The structure of the compound consists of coumarine, pyran and sulfonamide fragments whose rings are connected to one another.

The molecular structure of the title compound is shown in Fig. 2.The S1 atom shows a distorted tetrahedral geometry, with O1—S1—O2 [119.74 (2)°] and N1-S1-C5 [105.57 (1)°] angles deviating from ideal tetrahedral values are attributed to the Thrope-Ingold effect[15]. The sum of bond angles around N1 (316.2 (1)°) indicates that N1 is in sp2 hybridization. The Pyridine ring (N1-C8-C13-C14-C15-C16) adopts C15 envelop conformation with C15 displaced by 0.330 (3) Å, from the least-square planes formed by the remaining ring atoms and it is also further conformed by puckering parameters [q2 = 0.462 (4) Å, q3 = 0.210(4) Å,

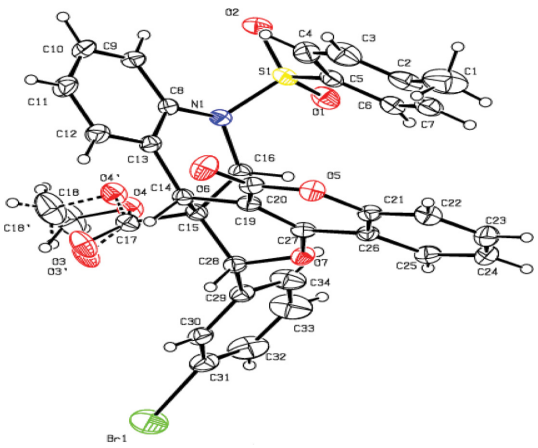

Fig. 2. Displacement ellipsoids are drawn at the 30% probability level.

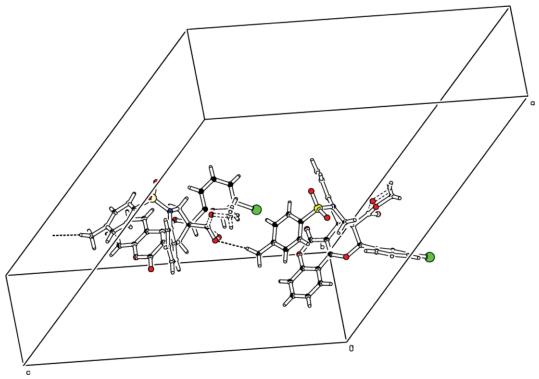

Fig. 3. Crystal packing of the title compound. H atoms have been omitted for clarity.

φ2 = 65.46 (4)°] and pyran ring (C14-C15-C28-O7-C27-C19) adopts a sofa conformation [q2 = 0.449 (4) Å, q3 = -0.339 (3) Å, φ2 = 127.07(4) °] defined by the puckering parameters [16]. The dihedral angle between the two mean-planes being 79.03 (1)°. The planar atoms of the pyran ring and coumarin ring (C19/C20/O5/C21/C26/C27/C25/C24/C23/C22/C21) are almost co-planar with dihedral angle between the meanplanes being 11.65 (1)°. Moreover, methylphenyl attached pyridine rings and bromophenyl attached pyran rings were tilted with the dihedral angle of 69.47 (1)° and 69.09 (1)° respectively. The carboxylate group of atoms (O3, O4 and C18) were disordered over two positions with site occupancy factors 0.598 (9):0.402 (9). Crystal packing is stabilized by C1-H1c…O7 inter molecular hydrogen bond interactions (Fig. 3 & Table 6).

Table 1. Crystal Data and Structure Refinement

Empirical formula	C_{34} $H_{26}BrNO_7S$
Formula weight	672.53
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, C2/c
Unit cell dimensions	a = 28.633(3) Å
	b = 9.321(7) Å β = 115.976 (2)°
	c = 24.590(2) Å
Volume	5900.1(9)Å^3
Z, Calculated density	8, 1.514 Mg/m^3
Absorption coefficient	1.517 mm^{-1}
F(000)	2752
Crystal size	0.25×0.20×0.20 mm
θ range for data collection	1.58 to 24.45°
Limiting indices	-33<=h<=31, -10<=k<=10, -28<=l<=28
Reflections collected/unique	23505/4888 [Rint = 0.0471]
Completeness to θ = 25.00	Completeness to θ = 24.45, 100%
Refinement method	Full-matrix least-squares on F2
Data/restraints/parameters	4888/0/588
Goodness-of-fit on F2	1.06
Final R indices [I>2σ(I)]	R1 = 0.0502, wR2 = 0.1432
R indices (all data)	R1 = 0.0849, wR2 = 0.1746
Largest diff. peak and hole	-0.951 and 1.020 e.Å^{-3}

Table 2. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*Y*	*Z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.5792 (3)	0.3360 (9)	0.2709 (3)	0.096 (2)	
H1A	0.5651	0.2406	0.2652	0.144*	
H1B	0.5517	0.4032	0.2501	0.144*	
H1C	0.5956	0.3583	0.3133	0.144*	
C2	0.6186 (2)	0.3449 (7)	0.2458 (2)	0.0601 (15)	
C3	0.6437 (2)	0.4736 (7)	0.2479 (2)	0.0648 (15)	
H3	0.6356	0.5539	0.2646	0.078*	
C4	0.6802 (2)	0.4856 (5)	0.2260 (2)	0.0509 (12)	
H4	0.6967	0.5727	0.2277	0.061*	
C5	0.69200 (17)	0.3653 (5)	0.20134 (18)	0.0382 (10)	
C6	0.66775 (18)	0.2359 (5)	0.19885 (19)	0.0444 (11)	
H6	0.6760	0.1553	0.1825	0.053*	
C7	0.6309 (2)	0.2275 (6)	0.2210 (2)	0.0584 (14)	
H7	0.6142	0.1407	0.2190	0.070*	
C8	0.69287 (16)	0.6064 (4)	0.10438 (17)	0.0324 (9)	
C9	0.72789 (18)	0.7125 (5)	0.13861 (19)	0.0429 (11)	
H9	0.7623	0.6881	0.1632	0.051*	
C10	0.7121 (2)	0.8521 (5)	0.1363 (2)	0.0481 (12)	
H10	0.7352	0.9208	0.1610	0.058*	
C11	0.6619 (2)	0.8908 (5)	0.0974 (2)	0.0494 (12)	
H11	0.6513	0.9858	0.0951	0.059*	
C12	0.62770 (18)	0.7878 (5)	0.0619 (2)	0.0437 (11)	
H12	0.5943	0.8147	0.0346	0.052*	
C13	0.64199 (15)	0.6453 (4)	0.06624 (18)	0.0318 (9)	
C14	0.60387 (15)	0.5356 (4)	0.02487 (17)	0.0308 (9)	
H14	0.5791	0.5868	-0.0110	0.037*	
C15	0.63382 (15)	0.4294 (4)	0.00378 (17)	0.0308 (9)	
C16	0.67873 (15)	0.3594 (4)	0.05763 (18)	0.0339 (9)	
H16A	0.6648	0.2844	0.0737	0.041*	
H16B	0.7019	0.3146	0.0434	0.041*	
C17	0.65009 (17)	0.5075 (5)	-0.03940 (19)	0.0373 (10)	
C19	0.57275 (15)	0.4519 (4)	0.05062 (17)	0.0313 (9)	
C20	0.54609 (15)	0.5283 (5)	0.08027 (18)	0.0350 (10)	
C21	0.51558 (15)	0.3023 (4)	0.10023 (18)	0.0341 (10)	
C22	0.48836 (17)	0.2355 (5)	0.1275 (2)	0.0437 (11)	
H22	0.4725	0.2887	0.1467	0.052*	
C23	0.48527 (19)	0.0882 (5)	0.1254 (2)	0.0497 (12)	
H23	0.4674	0.0410	0.1439	0.060*	
C24	0.50843 (18)	0.0097 (5)	0.0962 (2)	0.0473 (12)	
H24	0.5066	-0.0899	0.0958	0.057*	
C25	0.53402 (17)	0.0773 (5)	0.06793 (19)	0.0422 (11)	
H25	0.5491	0.0239	0.0478	0.051*	
C26	0.53757 (15)	0.2274 (4)	0.06927 (18)	0.0317 (9)	
C27	0.56452 (15)	0.3087 (4)	0.04155 (17)	0.0316 (9)	

Table 2. Continued

	x	*Y*	*Z*	U_{iso}*/U_{eq}	Occ. (<1)
C28	0.59387 (16)	0.3138 (4)	-0.03390 (18)	0.0341 (10)	
H28	0.5625	0.3638	-0.0622	0.041*	
C29	0.61013 (16)	0.2131 (5)	-0.07025 (19)	0.0365 (10)	
C30	0.58986 (18)	0.2336 (5)	-0.1322 (2)	0.0442 (11)	
H30	0.5659	0.3065	-0.1506	0.053*	
C31	0.6053 (2)	0.1455 (6)	-0.1666 (2)	0.0541 (14)	
C32	0.6400 (2)	0.0379 (7)	-0.1411 (3)	0.0718 (17)	
H32	0.6507	-0.0190	-0.1645	0.086*	
C33	0.6588 (2)	0.0143 (7)	-0.0803 (3)	0.0802 (19)	
H33	0.6819	-0.0608	-0.0625	0.096*	
C34	0.6440 (2)	0.1012 (6)	-0.0448 (2)	0.0595 (14)	
H34	0.6571	0.0837	-0.0035	0.071*	
N1	0.70913 (12)	0.4601 (4)	0.10717 (14)	0.0335 (8)	
O1	0.75069 (12)	0.2383 (3)	0.16027 (14)	0.0508 (8)	
O2	0.77916 (11)	0.4691 (4)	0.21298 (14)	0.0536 (9)	
O5	0.51878 (11)	0.4509 (3)	0.10437 (13)	0.0398 (7)	
O6	0.54389 (13)	0.6570 (3)	0.08378 (17)	0.0540 (9)	
O7	0.57995 (11)	0.2298 (3)	0.00611 (12)	0.0360 (7)	
Br1	0.57550 (3)	0.17201 (9)	-0.25142 (3)	0.0974 (3)	
S1	0.73869 (4)	0.37847 (12)	0.17349 (5)	0.0384 (3)	
C18	0.7154 (14)	0.514 (3)	-0.0748 (14)	0.083 (8)	0.60 (2)
H18A	0.6868	0.5469	-0.1110	0.124*	0.60 (2)
H18B	0.7333	0.4387	-0.0848	0.124*	0.60 (2)
H18C	0.7389	0.5916	-0.0561	0.124*	0.60 (2)
O3	0.6215 (11)	0.592 (3)	-0.0750 (6)	0.053 (4)	0.60 (2)
O4	0.6961 (3)	0.4582 (16)	-0.0332 (7)	0.048 (3)	0.60 (2)
C18'	0.714 (2)	0.575 (4)	-0.069 (2)	0.083 (8)	0.40 (2)
H18D	0.6965	0.5250	-0.1063	0.124*	0.40 (2)
H18E	0.7510	0.5693	-0.0555	0.124*	0.40 (2)
H18F	0.7037	0.6739	-0.0740	0.124*	0.40 (2)
O4'	0.7011 (3)	0.510 (2)	-0.0234 (11)	0.048 (3)	0.40 (2)
O3'	0.6228 (17)	0.564 (4)	-0.0863 (8)	0.053 (4)	0.40 (2)

Table 3. Bond lengths [Å] and angles [°]

C1—C2	1.507 (8)	C19—C20	1.452 (6)
C1—H1A	0.9600	C20—O6	1.207 (5)
C1—H1B	0.9600	C20—O5	1.374 (5)
C1—H1C	0.9600	C21—C26	1.372 (6)
C2—C7	1.372 (8)	C21—C22	1.378 (6)
C2—C3	1.388 (8)	C21—O5	1.389 (5)
C3—C4	1.375 (7)	C22—C23	1.375 (7)
C3—H3	0.9300	C22—H22	0.9300
C4—C5	1.385 (6)	C23—C24	1.382 (7)
C4—H4	0.9300	C23—H23	0.9300
C5—C6	1.380 (6)	C24—C25	1.365 (6)

Table 3. Continued

C5—S1	1.753 (5)	C24—H24	0.9300
C6—C7	1.386 (7)	C25—C26	1.403 (6)
C6—H6	0.9300	C25—H25	0.9300
C7—H7	0.9300	C26—C27	1.447 (6)
C8—C13	1.392 (6)	C27—O7	1.353 (5)
C8—C9	1.397 (6)	C28—O7	1.443 (5)
C8—N1	1.433 (5)	C28—C29	1.504 (6)
C9—C10	1.371 (7)	C28—H28	0.9800
C9—H9	0.9300	C29—C34	1.374 (7)
C10—C11	1.380 (7)	C29—C30	1.386 (6)
C10—H10	0.9300	C30—C31	1.383 (7)
C11—C12	1.376 (6)	C30—H30	0.9300
C11—H11	0.9300	C31—C32	1.357 (8)
C12—C13	1.380 (6)	C31—Br1	1.892 (5)
C12—H12	0.9300	C32—C33	1.366 (8)
C13—C14	1.517 (6)	C32—H32	0.9300
C14—C19	1.516 (6)	C33—C34	1.387 (7)
C14—C15	1.541 (5)	C33—H33	0.9300
C14—H14	0.9800	C34—H34	0.9300
C15—C17	1.519 (6)	N1—S1	1.657 (3)
C15—C16	1.530 (5)	O1—S1	1.424 (3)
C15—C28	1.549 (6)	O2—S1	1.419 (3)
C16—N1	1.481 (5)	C18—O4	1.453 (8)
C16—H16A	0.9700	C18—H18A	0.9600
C16—H16B	0.9700	C18—H18B	0.9600
C17—O3'	1.196 (9)	C18—H18C	0.9600
C17—O3	1.197 (7)	C18'—O4'	1.453 (10)
C17—O4'	1.336 (8)	C18'—H18D	0.9600
C17—O4	1.340 (7)	C18'—H18E	0.9600
C19—C27	1.357 (6)	C18'—H18F	0.9600
	Bond Angle		
C2—C1—H1A	109.5	C27—C19—C20	118.2 (4)
C2—C1—H1B	109.5	C27—C19—C14	121.9 (3)
H1A—C1—H1B	109.5	C20—C19—C14	119.5 (4)
C2—C1—H1C	109.5	O6—C20—O5	115.7 (4)
H1A—C1—H1C	109.5	O6—C20—C19	125.4 (4)
H1B—C1—H1C	109.5	O5—C20—C19	118.9 (4)
C7—C2—C3	118.5 (5)	C26—C21—C22	122.3 (4)
C7—C2—C1	121.5 (6)	C26—C21—O5	121.1 (4)
C3—C2—C1	120.0 (6)	C22—C21—O5	116.6 (4)
C4—C3—C2	121.7 (5)	C23—C22—C21	118.3 (4)
C4—C3—H3	119.2	C23—C22—H22	120.8
C2—C3—H3	119.2	C21—C22—H22	120.8
C3—C4—C5	118.6 (5)	C22—C23—C24	120.6 (4)
C3—C4—H4	120.7	C22—C23—H23	119.7
C5—C4—H4	120.7	C24—C23—H23	119.7
C6—C5—C4	120.8 (4)	C25—C24—C23	120.5 (4)
C6—C5—S1	119.9 (3)	C25—C24—H24	119.7
C4—C5—S1	119.2 (4)	C23—C24—H24	119.7
C5—C6—C7	119.1 (5)	C24—C25—C26	119.9 (4)

Table 3. Continued

C5—C6—H6	120.4	C24—C25—H25	120.1
C7—C6—H6	120.4	C26—C25—H25	120.1
C2—C7—C6	121.2 (5)	C21—C26—C25	118.3 (4)
C2—C7—H7	119.4	C21—C26—C27	117.7 (4)
C6—C7—H7	119.4	C25—C26—C27	124.0 (4)
C13—C8—C9	119.0 (4)	O7—C27—C19	123.9 (4)
C13—C8—N1	120.2 (3)	O7—C27—C26	114.3 (3)
C9—C8—N1	120.8 (4)	C19—C27—C26	121.8 (4)
C10—C9—C8	120.6 (4)	O7—C28—C29	108.4 (3)
C10—C9—H9	119.7	O7—C28—C15	108.8 (3)
C8—C9—H9	119.7	C29—C28—C15	116.7 (3)
C9—C10—C11	120.1 (4)	O7—C28—H28	107.5
C9—C10—H10	120.0	C29—C28—H28	107.5
C11—C10—H10	120.0	C15—C28—H28	107.5
C12—C11—C10	119.5 (4)	C34—C29—C30	118.5 (4)
C12—C11—H11	120.2	C34—C29—C28	122.9 (4)
C10—C11—H11	120.2	C30—C29—C28	118.5 (4)
C11—C12—C13	121.2 (4)	C31—C30—C29	119.9 (5)
C11—C12—H12	119.4	C31—C30—H30	120.1
C13—C12—H12	119.4	C29—C30—H30	120.1
C12—C13—C8	119.4 (4)	C32—C31—C30	121.4 (5)
C12—C13—C14	119.7 (4)	C32—C31—Br1	119.5 (4)
C8—C13—C14	120.6 (4)	C30—C31—Br1	119.1 (4)
C19—C14—C13	116.7 (3)	C31—C32—C33	119.0 (5)
C19—C14—C15	109.0 (3)	C31—C32—H32	120.5
C13—C14—C15	108.4 (3)	C33—C32—H32	120.5
C19—C14—H14	107.5	C32—C33—C34	120.8 (6)
C13—C14—H14	107.5	C32—C33—H33	119.6
C15—C14—H14	107.5	C34—C33—H33	119.6
C17—C15—C16	114.2 (3)	C29—C34—C33	120.4 (5)
C17—C15—C14	108.3 (3)	C29—C34—H34	119.8
C16—C15—C14	111.3 (3)	C33—C34—H34	119.8
C17—C15—C28	106.7 (3)	C8—N1—C16	120.1 (3)
C16—C15—C28	110.0 (3)	C8—N1—S1	120.0 (3)
C14—C15—C28	105.9 (3)	C16—N1—S1	112.4 (3)
N1—C16—C15	114.2 (3)	C20—O5—C21	121.5 (3)
N1—C16—H16A	108.7	C27—O7—C28	114.2 (3)
C15—C16—H16A	108.7	O2—S1—O1	119.7 (2)
N1—C16—H16B	108.7	O2—S1—N1	108.18 (18)
C15—C16—H16B	108.7	O1—S1—N1	105.48 (18)
H16A—C16—H16B	107.6	O2—S1—C5	108.0 (2)
O3'—C17—O3	19 (2)	O1—S1—C5	109.1 (2)
O3'—C17—O4'	116 (3)	N1—S1—C5	105.57 (18)
O3—C17—O4'	121 (2)	C17—O4—C18	117.1 (15)
O3'—C17—O4	117 (2)	O4'—C18'—H18D	109.5
O3—C17—O4	129.2 (16)	O4'—C18'—H18E	109.5
O4'—C17—O4	23.0 (9)	H18D—C18'—H18E	109.5
O3'—C17—C15	128 (3)	O4'—C18'—H18F	109.5
O3—C17—C15	120.5 (15)	H18D—C18'—H18F	109.5
O4'—C17—C15	115.7 (12)	H18E—C18'—H18F	109.5
O4—C17—C15	110.1 (7)	C17—O4'—C18'	113 (3)

Table 4. Anisotropic displacement parameters

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.074 (4)	0.165 (8)	0.062 (4)	-0.013 (4)	0.040 (3)	-0.017 (4)
C2	0.049 (3)	0.097 (5)	0.032 (3)	0.003 (3)	0.015 (2)	0.002 (3)
C3	0.076 (4)	0.075 (4)	0.049 (3)	0.020 (3)	0.033 (3)	-0.005 (3)
C4	0.065 (3)	0.045 (3)	0.046 (3)	0.004 (2)	0.027 (3)	-0.001 (2)
C5	0.042 (2)	0.039 (3)	0.027 (2)	0.005 (2)	0.0097 (19)	0.0042 (18)
C6	0.050 (3)	0.048 (3)	0.031 (2)	0.000 (2)	0.013 (2)	0.004 (2)
C7	0.056 (3)	0.078 (4)	0.034 (3)	-0.015 (3)	0.014 (2)	0.005 (3)
C8	0.036 (2)	0.033 (2)	0.028 (2)	-0.0051 (19)	0.0144 (18)	0.0012 (17)
C9	0.039 (3)	0.047 (3)	0.035 (2)	-0.013 (2)	0.010 (2)	-0.001 (2)
C10	0.058 (3)	0.037 (3)	0.046 (3)	-0.018 (2)	0.020 (2)	-0.008 (2)
C11	0.065 (3)	0.030 (2)	0.055 (3)	-0.005 (2)	0.029 (3)	-0.008 (2)
C12	0.045 (3)	0.036 (3)	0.045 (3)	0.000 (2)	0.015 (2)	0.000 (2)
C13	0.032 (2)	0.034 (2)	0.033 (2)	-0.0029 (18)	0.0170 (18)	-0.0011 (17)
C14	0.031 (2)	0.029 (2)	0.030 (2)	0.0035 (17)	0.0107 (18)	0.0012 (17)
C15	0.033 (2)	0.033 (2)	0.028 (2)	0.0021 (18)	0.0145 (18)	-0.0004 (17)
C16	0.035 (2)	0.033 (2)	0.031 (2)	0.0023 (19)	0.0120 (18)	-0.0020 (18)
C17	0.039 (2)	0.040 (2)	0.032 (2)	0.002 (2)	0.015 (2)	0.000 (2)
C19	0.028 (2)	0.035 (2)	0.029 (2)	0.0029 (18)	0.0103 (17)	-0.0027 (17)
C20	0.031 (2)	0.033 (3)	0.037 (2)	0.0045 (19)	0.0111 (19)	-0.0037 (18)
C21	0.030 (2)	0.035 (2)	0.034 (2)	-0.0004 (18)	0.0105 (19)	-0.0027 (18)
C22	0.040 (3)	0.052 (3)	0.044 (3)	0.004 (2)	0.023 (2)	-0.001 (2)
C23	0.052 (3)	0.051 (3)	0.049 (3)	-0.010 (2)	0.025 (2)	0.001 (2)
C24	0.055 (3)	0.038 (3)	0.048 (3)	-0.012 (2)	0.021 (2)	-0.003 (2)
C25	0.042 (3)	0.042 (3)	0.040 (2)	-0.001 (2)	0.016 (2)	-0.006 (2)
C26	0.026 (2)	0.032 (2)	0.031 (2)	-0.0012 (18)	0.0074 (18)	-0.0009 (18)
C27	0.027 (2)	0.037 (2)	0.029 (2)	0.0019 (18)	0.0113 (17)	-0.0058 (18)
C28	0.036 (2)	0.032 (2)	0.034 (2)	0.0000 (18)	0.0156 (19)	-0.0036 (18)
C29	0.039 (2)	0.036 (2)	0.038 (2)	-0.003 (2)	0.020 (2)	-0.0084 (19)
C30	0.053 (3)	0.042 (3)	0.039 (3)	-0.013 (2)	0.020 (2)	-0.009 (2)
C31	0.070 (3)	0.063 (3)	0.042 (3)	-0.026 (3)	0.036 (3)	-0.021 (2)
C32	0.069 (4)	0.086 (5)	0.074 (4)	-0.005 (4)	0.044 (3)	-0.038 (4)
C33	0.079 (4)	0.081 (4)	0.074 (4)	0.033 (3)	0.027 (3)	-0.021 (3)
C34	0.060 (3)	0.066 (3)	0.047 (3)	0.015 (3)	0.019 (3)	-0.014 (3)
N1	0.0327 (19)	0.0328 (19)	0.0293 (18)	0.0009 (15)	0.0086 (15)	0.0021 (15)
O1	0.052 (2)	0.049 (2)	0.0486 (19)	0.0216 (16)	0.0191 (16)	0.0094 (15)
O2	0.0397 (18)	0.065 (2)	0.0374 (17)	-0.0043 (16)	-0.0005 (14)	0.0034 (16)
O5	0.0403 (17)	0.0393 (18)	0.0477 (17)	0.0054 (14)	0.0266 (15)	-0.0022 (14)
O6	0.061 (2)	0.036 (2)	0.078 (2)	0.0038 (16)	0.042 (2)	-0.0075 (16)
O7	0.0439 (17)	0.0325 (16)	0.0389 (16)	-0.0051 (13)	0.0248 (14)	-0.0084 (13)
Br1	0.1485 (8)	0.1041 (6)	0.0498 (4)	-0.0266 (5)	0.0530 (4)	-0.0251 (3)
S1	0.0359 (6)	0.0432 (7)	0.0299 (5)	0.0062 (5)	0.0086 (5)	0.0053 (5)
C18	0.063 (5)	0.12 (2)	0.086 (8)	0.014 (16)	0.054 (5)	0.040 (16)
O3	0.066 (3)	0.064 (10)	0.047 (5)	0.027 (5)	0.041 (5)	0.019 (7)
O4	0.041 (2)	0.060 (9)	0.048 (5)	0.001 (3)	0.026 (3)	0.008 (6)
C18'	0.063 (5)	0.12 (2)	0.086 (8)	0.014 (16)	0.054 (5)	0.040 (16)
O4'	0.041 (2)	0.060 (9)	0.048 (5)	0.001 (3)	0.026 (3)	0.008 (6)
O3'	0.066 (3)	0.064 (10)	0.047 (5)	0.027 (5)	0.041 (5)	0.019 (7)

Table 5. Torsion angles

C7—C2—C3—C4	-0.1 (8)	C24—C25—C26—C27	179.0 (4)
C1—C2—C3—C4	179.5 (5)	C20—C19—C27—O7	168.8 (4)
C2—C3—C4—C5	-0.1 (8)	C14—C19—C27—O7	-4.5 (6)
C3—C4—C5—C6	-0.1 (7)	C20—C19—C27—C26	-10.7 (6)
C3—C4—C5—S1	-179.7 (4)	C14—C19—C27—C26	176.1 (4)
C4—C5—C6—C7	0.5 (6)	C21—C26—C27—O7	-172.8 (3)
S1—C5—C6—C7	-179.9 (3)	C25—C26—C27—O7	9.3 (6)
C3—C2—C7—C6	0.5 (7)	C21—C26—C27—C19	6.7 (6)
C1—C2—C7—C6	-179.1 (5)	C25—C26—C27—C19	-171.3 (4)
C5—C6—C7—C2	-0.7 (7)	C17—C15—C28—O7	176.0 (3)
C13—C8—C9—C10	2.1 (6)	C16—C15—C28—O7	51.6 (4)
N1—C8—C9—C10	-179.3 (4)	C14—C15—C28—O7	-68.8 (4)
C8—C9—C10—C11	-3.7 (7)	C17—C15—C28—C29	53.0 (5)
C9—C10—C11—C12	1.4 (7)	C16—C15—C28—C29	-71.5 (4)
C10—C11—C12—C13	2.5 (7)	C14—C15—C28—C29	168.2 (3)
C11—C12—C13—C8	-4.0 (7)	O7—C28—C29—C34	-47.0 (6)
C11—C12—C13—C14	-177.8 (4)	C15—C28—C29—C34	76.2 (6)
C9—C8—C13—C12	1.7 (6)	O7—C28—C29—C30	132.7 (4)
N1—C8—C13—C12	-176.9 (4)	C15—C28—C29—C30	-104.0 (4)
C9—C8—C13—C14	175.4 (4)	C34—C29—C30—C31	-2.3 (7)
N1—C8—C13—C14	-3.1 (6)	C28—C29—C30—C31	178.0 (4)
C12—C13—C14—C19	-97.1 (5)	C29—C30—C31—C32	0.3 (7)
C8—C13—C14—C19	89.2 (5)	C29—C30—C31—Br1	178.1 (3)
C12—C13—C14—C15	139.4 (4)	C30—C31—C32—C33	1.7 (9)
C8—C13—C14—C15	-34.3 (5)	Br1—C31—C32—C33	-176.1 (5)
C19—C14—C15—C17	161.8 (3)	C31—C32—C33—C34	-1.7 (10)
C13—C14—C15—C17	-70.3 (4)	C30—C29—C34—C33	2.2 (8)
C19—C14—C15—C16	-71.9 (4)	C28—C29—C34—C33	-178.1 (5)
C13—C14—C15—C16	56.1 (4)	C32—C33—C34—C29	-0.3 (10)
C19—C14—C15—C28	47.6 (4)	C13—C8—N1—C16	19.2 (5)
C13—C14—C15—C28	175.6 (3)	C9—C8—N1—C16	-159.3 (4)
C17—C15—C16—N1	79.8 (4)	C13—C8—N1—S1	-128.3 (3)
C14—C15—C16—N1	-43.2 (5)	C9—C8—N1—S1	53.1 (5)
C28—C15—C16—N1	-160.2 (3)	C15—C16—N1—C8	5.3 (5)
C16—C15—C17—O3'	175 (2)	C15—C16—N1—S1	155.1 (3)
C14—C15—C17—O3'	-60 (2)	O6—C20—O5—C21	176.2 (4)
C28—C15—C17—O3'	54 (2)	C19—C20—O5—C21	-0.9 (5)
C16—C15—C17—O3	-163.3 (16)	C26—C21—O5—C20	-3.2 (6)
C14—C15—C17—O3	-38.6 (16)	C22—C21—O5—C20	178.3 (4)
C28—C15—C17—O3	75.0 (16)	C19—C27—O7—C28	-15.3 (5)
C16—C15—C17—O4'	-2.8 (10)	C26—C27—O7—C28	164.1 (3)
C14—C15—C17—O4'	121.8 (10)	C29—C28—O7—C27	180.0 (3)
C28—C15—C17—O4'	-124.6 (10)	C15—C28—O7—C27	52.1 (4)
C16—C15—C17—O4	21.4 (9)	C8—N1—S1—O2	-49.1 (4)
C14—C15—C17—O4	146.0 (8)	C16—N1—S1—O2	161.0 (3)
C28—C15—C17—O4	-100.4 (8)	C8—N1—S1—O1	-178.3 (3)

Table 5. Continued

C13—C14—C19—C27	-137.4 (4)	C16—N1—S1—O1	31.8 (3)
C15—C14—C19—C27	-14.3 (5)	C8—N1—S1—C5	66.3 (3)
C13—C14—C19—C20	49.4 (5)	C16—N1—S1—C5	-83.6 (3)
C15—C14—C19—C20	172.6 (3)	C6—C5—S1—O2	-141.1 (3)
C27—C19—C20—O6	-169.1 (4)	C4—C5—S1—O2	38.5 (4)
C14—C19—C20—O6	4.3 (6)	C6—C5—S1—O1	-9.5 (4)
C27—C19—C20—O5	7.7 (5)	C4—C5—S1—O1	170.1 (3)
C14—C19—C20—O5	-178.8 (3)	C6—C5—S1—N1	103.4 (3)
C26—C21—C22—C23	2.8 (6)	C4—C5—S1—N1	-77.0 (4)
O5—C21—C22—C23	-178.6 (4)	O3'—C17—O4—C18	18 (3)
C21—C22—C23—C24	-0.7 (7)	O3—C17—O4—C18	0 (2)
C22—C23—C24—C25	-1.1 (7)	O4'—C17—O4—C18	-76 (5)
C23—C24—C25—C26	1.0 (7)	C15—C17—O4—C18	175.1 (15)
C22—C21—C26—C25	-3.0 (6)	O3'—C17—O4'—C18'	-4 (3)
O5—C21—C26—C25	178.6 (4)	O3—C17—O4'—C18'	-25 (3)
C22—C21—C26—C27	178.9 (4)	O4—C17—O4'—C18'	94 (5)
O5—C21—C26—C27	0.5 (6)	C15—C17—O4'—C18'	175 (2)
C24—C25—C26—C21	1.0 (6)		

Table 6. Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H(Å)	H···*A*(Å)	*D*···*A*(Å)	*D*—H···*A*(º)
C1-H1*C*···O3$^{(i)}$	0.96	2.56	3.501 (16)	169
C6-H6···O1	0.93	2.55	2.918 (7)	104
C9-H9···O2	0.93	2.32	2.880 (6)	118

Equivalent position
(i) x, 2-y,1/2+z

4. Conclusion

The title compound is crystallized from ethyl acetate solution by slow evaporation technique. The structure is determined using Direct Methods Protocol and refined using Least-squares Fit methods. The final R factor is 4.10%. In general the coumarine derivatives are well characterized interms of medicinal and biological applications. The title structure may be important from a medicinal point of view as well as their widespread biological significance. The structure may be useful for further investigation on the mechanism, potential activity, optimal reaction condition etc which will be further characterized as a future prospective of our project. As 3D structure is determined now, with the biological importance of such derivatives, the usefulness of the present derivative can be established using Bioinformatics tools.

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