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Synthesis and crystal structure of 1-(2-hydroxyphenyl)-3-(5-methylthiophen-2-yl)prop-2-en-1-one

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ABSTRACT

In the structure of the title compound, $C_{14}H_{12}O_2S$, the molecule is almost planar as evidenced by the very low values of torsion angles. The hydroxy group substituted at C16 position is in anti-periplanar conformation with respect to the phenyl ring. The crystal structure exhibits weak intramolecular hydrogen bonds of the type O–H···O.

KEYWORDS

Chalcone;
Flovonoids;
Methylthiophen;
Crystal structure;
Trigonal planargeometry.

INTRODUCTION

Chalcone (1,3-diarylprop-2-en-1-one) constitutes basic skeleton of an important class of oxygenated heterocyclic compounds of plant origin i.e., flavonoids. Various chalcones of natural and synthetic origin have been reported to exhibit a wide spectrum of biological activities such as anti-cancer[1,2], anti-malarial[3], anti-bacterial[4], hypoglycemic[5], anti-oxidant[6], anti-leishmanial[7], anti-inflammatory[8], activity. The biological importance of chalcones prompted us to attempt to synthesize a series of chalcones with various substitutions made at 1 and 3 positions of prop-2-en-1-one. This paper reports the synthesis and the crystal structure of 1-(2-hydroxyphenyl)-3- (5-methylthiophen-2-yl)prop-2-en-1-one.

EXPERIMENTAL

The title compound 1-(2-hydroxyphenyl)-3-(5-methylthiophen-2-yl)prop-2-en-1-one was synthesized by dissolving 5 m mole of 2-hydroxyacetophenone in 15 ml of methanol taken in a conical flask, to which 5 ml of aqueous solution of sodium hydroxide was added with stirring, followed by slow addition of 5 m mole of 5-methyl-2-thiophenaldehyde and continued stirring for

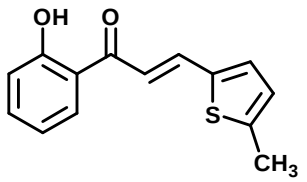

Figure 1 : Schematic diagram of the title molecule

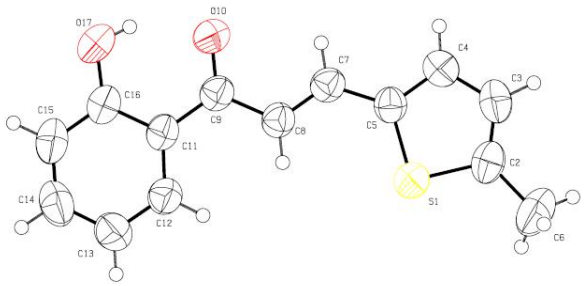

Figure 2 : ORTEP of the molecule with thermal ellipsoids drawn at 50% probability

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TABLE 1 : Crystal data and structure refinement table

Empirical formula	$C_{14}H_{12}O_2S$
Formula weight	244.31
Temperature	293K
Wavelength	0.71073° A
Crystal system	Monoclinic
Space group	$P2_1/c$
Cell dimensions	a=8.253(5)°A
	b=13.153(1)° A
	c=14.157(1)°A
Volume	1249.9(2)°A^3
Z	4
Density(calculated)	1.298Mg/m^3
Absorption coefficient	0.245 mm^{-1}
Fooo	512
Crystal size	0.290×0.250×0.220 mm
Theta range for data collection	2.35° to 25.02°
Index ranges	-8≤ h≤8
	-14≤ k≤15
	-16≤ l≤16
Reflections collected	3572
Independent reflections	2072
Absorption correction	None
Refinement method	Full-matrix least- squares on F^2
Data/restraints/parameters	2072/0/156
Goodness-of-fit on F^2	1.071
Final R indices	R1=0.048, wR2=0.1621
R indices (all data)	R1=0.0718, wR2=0.1824
Extinction coefficient	0.017(9)
Largest diff. peak and hole	0.250 and –0.290 e.°A^{-3}

TABLE 2 : Atomic coordinates and equivalent thermal parameters of the non-hydrogen atoms

Atom	x	y	z	U_{eq}
S1	0.31013(10)	0.98072(5)	0.26141(5)	0.0730(3)
O10	0.2413(3)	1.11453(14)	-0.12065(15)	0.0767(5)
C8	0.2082(3)	1.09331(17)	0.03171(19)	0.0589(6)
C7	0.3077(3)	1.00603(17)	0.0657(2)	0.0595(6)
C9	0.1739(3)	1.14947(17)	-0.06778(19)	0.0591(6)
C11	0.0654(3)	1.24592(17)	-0.10452(18)	0.0576(5)
C5	0.3541(3)	0.94368(17)	0.16224(18)	0.0579(6)
C16	0.0261(4)	1.29571(19)	-0.2047(2)	0.0660(6)
O17	0.0862(3)	1.25773(15)	-0.26744(16)	0.0884(6)
C4	0.4356(4)	0.84835(19)	0.1887(2)	0.0682(6)
C12	-0.0015(4)	1.29316(18)	-0.0447(2)	0.0668(6)
C3	0.4610(4)	0.8067(2)	0.2871(2)	0.0760(7)
C15	-0.0784(4)	1.3869(2)	-0.2409(2)	0.0797(8)
C2	0.4018(4)	0.8682(2)	0.3373(2)	0.0720(7)
C13	-0.1030(4)	1.3839(2)	-0.0811(3)	0.0821(8)
C14	-0.1421(4)	1.4299(2)	-0.1802(3)	0.0864(8)
C6	0.4044(5)	0.8495(3)	0.4430(3)	0.1061(11)

TABLE 3 : Bond lengths (A°)

Atoms	Length	Atoms	Length
S1-C5	1.712(2)	C5-C4	1.368(3)
S1-C2	1.723(3)	C16-O17	1.340(3)
O10-C9	1.251(3)	C16-C15	1.390(4)
C8-C7	1.328(3)	C4-C3	1.395(4)
C8-C9	1.465(3)	C12-C13	1.374(4)
C7-C5	1.442(3)	C3-C2	1.342(4)
C9-C11	1.463(3)	C15-C14	1.363(4)
C11-C12	1.397(3)	C2-C6	1.506(4)
C11-C16	1.418(3)	C13-C14	1.383(4)

24 hours.

The progress of reaction was monitored by TLC using n-hexane and ethyl acetate (9:1) as solvent system. After completion of reaction, the mixture was poured into ice cold water and acidified with hydrochloric acid. The title compound separated as precipitate which was filtered, dried and recrystallised from methanol. The product was confirmed by spectroscopic characterisation. Figure 1 represents schematic diagram of the molecule.

Crystal structure determination

A single crystal of suitable size was chosen for X-ray diffraction studies. The data were collected at room temperature on a DIPLabo Image Plate system with graphite monochromated radiation MoK_α. Each exposure of the image plate was set to a period of 400s. Thirty-six frames of data were collected in the oscillation mode with an oscillation range of 5° and processed using Denzo[15]. The reflection were merged with Scalepack. All the frames could be indexed using a primitive monoclinic lattice. The structure was solved by direct methods using SHELXS-97[9]. Least-squares refinement using SHELXL-97[9] with isotropic displacement parameters for all the non-hydrogen atoms converged the residual to R_1 = 0.1621. Subsequent refinements were carried out with anisotropic thermal parameters for the non-hydrogen atoms. After eight cycles

TABLE 4: Bond angles (A°)

Atoms	Angle	Atoms	Angle
C5-S1-C2	92.34(12)	O17-C16-C15	117.9(2)
C7-C8-C9	121.1(2)	O17-C16-C11	122.2(2)
C8-C7-C5	127.0(2)	C15-C16-C11	119.9(2)
O10-C9-C11	119.9(2)	C5-C4-C3	113.4(2)
O10-C9-C8	119.1(2)	C13-C12-C11	121.9(2)
C11-C9-C8	121.0(2)	C2-C3-C4	113.7(2)
C12-C11-C16	117.4(2)	C14-C15-C16	120.6(2)
C12-C11-C9	123.1(2)	C3-C2-C6	129.0(3)
C16-C11-C9	119.5(2)	C3-C2-S1	110.56(19)
C4-C5-C7	126.7(2)	C6-C2-S1	120.5(2)
C4-C5-S1	109.98(18)	C12-C13-C14	119.5(3)
C7-C5-S1	123.34(18)	C15-C14-C13	120.7(3)

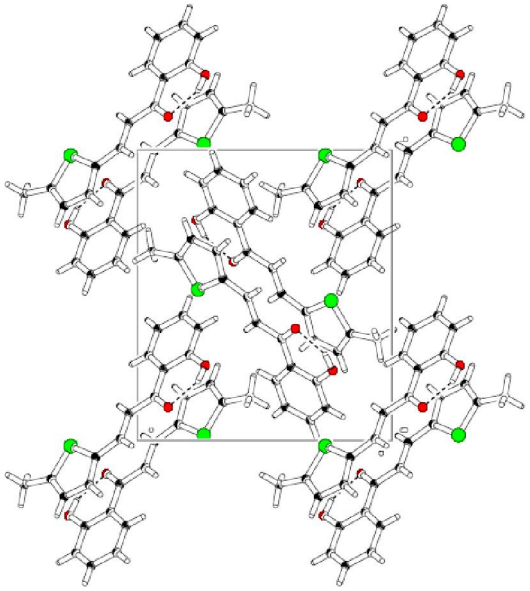

Figure 3 : Packing of molecules when viewed down the a axis

TABLE 5 : Torsion angles (A°)

Atoms	Angle
C5 S1 C2 C3	-0.3(3)
C5 S1 C2 C6	-179.4(3)
C2 S1 C5 C4	0.1(2)
C2 S1 C5 C7	179.5(2)
S1 C2 C3 C4	0.4(4)
C6 C2 C3 C4	179.4(3)
C2 C3 C4 C5	-0.4(4)
C3 C4 C5 S1	0.1(3)
C3 C4 C5 C7	-179.3(3)
S1 C5 C7 C8	-7.7(4)
C4 C5 C7 C8	171.7(3)
C5 C7 C8 C9	179.2(2)
C7 C8 C9 O10	-0.6(4)
C7 C8 C9 C11	-179.6(2)
O10 C9 C11 C12	-175.3(3)
O10 C9 C11 C16	4.2(4)
C8 C9 C11 C12	3.8(4)
C8 C9 C11 C16	-176.8(2)
C9 C11 C12 C13	179.9(3)
C16 C11 C12 C13	0.5(4)
C9 C11 C16 O17	-0.4(4)
C9 C11 C16 C15	179.4(3)
C12 C11 C16 O17	179.0(3)
C12 C11 C16 C15	-1.1(4)
C11 C12 C13 C14	0.5(5)
C12 C13 C14 C15	-1.0(5)
C13 C14 C15 C16	0.4(5)
C14 C15 C16 O17	-179.4(3)
C14 C15 C16 C11	0.7(5)

of refinement the residuals converged to 0.048. The hydrogen atoms were fixed at chemically acceptable positions and were allowed to ride on their parent atoms.

The crystallographic data have been deposited in Cambridge Crystallographic Data Center, under reference CCDC No.768945.

RESULTS AND DISCUSSION

The details of crystal data and refinement are given in TABLE 1. The final atomic coordinates and equivalent thermal parameters for all the non-hydrogenatoms are given in TABLE 2. The bond lengths and angles of all the non-hydrogen atoms are given TABLE 3 and 4, respectively. Figure 2 represent the ORTEP[10] diagram of the molecule with thermal ellipsoids drawn at 50% probability.

The molecular structure 1-(2-hydroxyphenyl)-3-(5-methylthiophen-2-yl)prop-2-en-1- one, consists of a phenyl ring and a thiophen ring attached to the either ends of the propanone chain. The dihedral angles between the mean plane of the prop-2-en-1-one unit and those of the thiophen and phenyl rings are 1.56(12)° and 6.66(11)° respectively. The bond lengths C2–C6, C8–C9, C9–O10, C16–O17 and bond angles C8–C9–O10, C11–C9–O10, C11–C16–O17 are in good agreement with the values for a compound reported

earlier[11]. The angles of C8–C9–O10, C11–C9–O10 and C8–C9–C11 are 119.1(2)°, 119.9(2)° and 121.0(2)° respectively which indicate that the position of C9 atom is in nearly trigonal planar geometry. The packing of the molecules is characterized by intramolecular O–H···O hydrogen bonding between the hydroxy donor group and the C=O acceptor group. The packing of the molecules down a axis is shown in the figure 3.

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