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Crystal Structure and Hirshfeld Surfaces of (*E*)-1-(2-Hydroxyphenyl)-3-(5-methylthiophen-2-yl)prop-2-en-1-one

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The title compound, (*E*)-1-(2-hydroxyphenyl)-3-(5-methylthiophen-2-yl)prop-2-en-1-one, is crystallized into a monoclinic crystal class with cell parameters a = 8.2530(5)Å, b = 13.1530(14)Å, c = 14.1570(11)Å, β = 125.579(6)° and space group of $P2_1/c$. The structure is solved by a direct method, and it is refined to R = 0.048. The structure exhibits short C–H···O interactions. Hirshfeld surface computation studies shows that H···H, C···H and O···H are major contributions.

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Chalcone is an aromatic ketone that forms the central core for many important aromatic compounds that are biologically active. These are also known as flavonoids which are found in vegetables and fruits. Chalcone exhibits attractive therapeutic activities, such as antibacterial, antifungal, antioxidant, antineoplastic, antiinflammatory and antiviral.[1] The thiophene ring attached to the chalcone, is a five-membered heterocyclic ring with sulfur as a heteroatom having the molecular formula C_4H_4S; its derivatives exist in petroleum or coal.[2] Thiophene is widely known owing to its important electronic plausibility and biological activities. Further, its derivatives are widely used in organic light-emitting diodes (OLEDs), organic field-effect transistors and in solar cells.[3] That of biological activities, such as anti-breast cancer, antimicrobial, anticancer, anti-inflammatory, anti-hypertensive further raloxifine, is a drug used for the prevention and treatment of osteoporosis in postmenopausal women. This based on the benzothiophene system.[4,5] Recently tiaprofenic acid and tenidap are drugs containing thiophene rings, used as non-steroidal anti-inflammatory drugs (NSAIDs) for pain killing and for inflammatory disorders.[6] In addition to this, the thiophene nucleus is treated being as important in the synthesis of heterocyclic compounds with the pharmalogical activities antihypertensive, diabetes mellitus, cholestorol inhibitors.[7] Considering these to be important biological activities we hereby report on the crystal structure, Hirshfeld surfaces and computational studies of (*E*)-1-(2-hydroxyphenyl)-3-(5-methylthiophen-2-yl)prop-2-en-1-one.

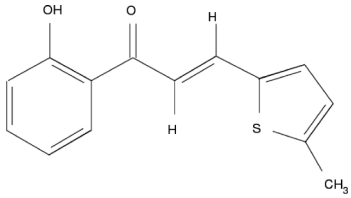

Fig. 1 Scheme of the compound (*E*)-1-(2-hydroxyphenyl)-3-(5-methylthiophen-2-yl)prop-2-en-1-one.

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2′-Hydroxyacetophenone of 0.005 mols was added to 15 ml of methanol taken in a conical flask; to this, 5 ml of aqueous NaOH was added and underwent stirring at room temperature; later, 0.005 mols of 5-methyl-2-thiophene-carboxaldehyde was slowly added while continuing stirring for 48 h. The mixture poured into ice cold water, mixed properly and acidified with dilute HCl. The title compound separates as a precipitate, which was collected by filtration and recrystallized from methanol (Fig. 1).

The X-ray intensity data for the title compound, $C_{14}H_{12}O_2S$, was collected at a temperature of 293 K on a Bruker X8 APEX II diffractometer using graphite-monochromated Mo $K\alpha$ radiation (λ = 0.71073 Å). A complete data set was processed using SAINT. The structure was solved by direct method SHELXS and refined by the full-matrix least-squares method on F^2 using SHELXL programs, respectively. Geometrical

Table 1 Crystal data and structure refinement details for the title compound

CCDC Number: 1577562	
Chemical formula: $C_{14}H_{12}O_2S$	
Molecular weight = 244.31	
T = 293 K	
Space group: $P2_1/c$	
Crystal system: monoclinic	
a = 8.2530(5)Å	V = 1249.87(18)Å^3
b = 13.1530(14)Å	Z = 4
c = 14.1570(11)Å	Radiation type : Mo $K\alpha$
β = 125.579(6)°	Wavelength = 0.71073 Å
Absorption coefficient = 0.25 mm^{-1}	
Crystal size = 0.30 × 0.27 × 0.25 mm	
Diffractometer: APEX (Bruker, 1999)	
Absorption correction: psi-scan; T_{min}, T_{max} = 0.944, 0.953	
No. of reflections measured = 2072	
No of independent reflections = 2072	
No. of reflections observed = 1748	
R_{int} = 0.021 (sin $\theta/\lambda)_{max}$ = 0.595 Å^{-1}	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S: 0.048, 0.162, 1.07	
Data/Restraints/Parameters: 2072/0/156	
H-atom treatment: H-atom parameters constrained	
$(\Delta\rho)_{max}$ = 0.25 eÅ^{-3}	$(\Delta\rho)_{min}$ = –0.29 eÅ^{-3}

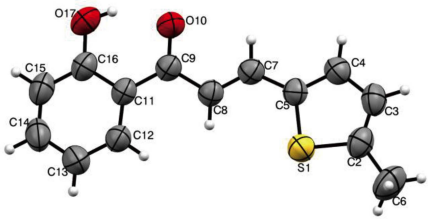

Fig. 2 ORTEP drawing of the ligand, showing at 50% probability displacement ellipsoids.

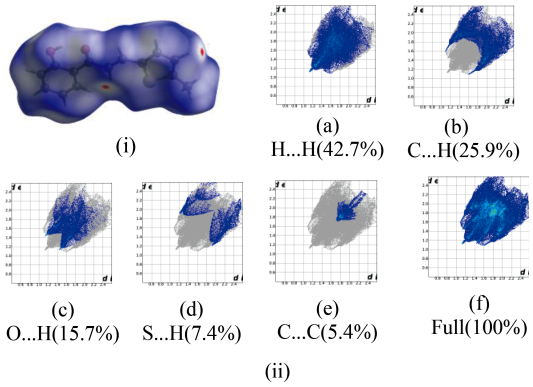

Fig. 3 (i) 3D Hirshfeld surface and (ii) 2D finger print plots.

calculations were carried out using PLATON software, and ORTEP diagrams were generated using the software MERCURY.

The crystal data and structure refinement details are summarized in Table 1. The titled compound was crystallized into a monoclinic system with cell parameters a = 8.2530(5)Å, b = 13.1530(14)Å, c = 14.1570(11)Å, β = 125.579(6)°, volume 1249.87(18)Å^3 and Z = 4 with space group $P2_1/c$ which consists of thiophene and a phenyl rings bridged by a propenone chain with the chemical formula $C_{14}H_{12}O_2S$. An Oak Ridge Thermal Ellipsoid Plot (ORTEP) of the molecule drawn at 50% probability level is shown in Fig. 2. The dihedral angle of the mean plane of the thiophene ring (S1-C2-C3-C4-C5) with a phenyl ring (C11-C12-C13-C14-C15-C16) is 6.68(15)°, while a propenone chain (O10-C7-C8-C9) with a phenyl ring (C11-C12-C13-C14-C15-C16) is 3.9(2)°. This indicates that molecule adopts a nearly planar structure further, the position of C9 atom forms a slightly distorted trigonal planar geometry, explained by the bond angles for the atoms (O10-C9-C11) = 119.9(2)°, (O10-C9-C8) = 119.1(2)° and (C8-C9-C11) = 121.0(2)°, which is due to a steric hindrance of the oxygen atom.

The conformation of the methyl group at C2 is oriented *anti-periplanar* to the thiophene ring, described by a torsion value of –179.40° for the atoms (C5-S1-C2-C6). No ring puckering analysis has been observed. Further, the thiophene ring is affected by pi-conjugation, explained saying that the value of (S1=C2) = 1.723(3)Å is larger, compared to (S1=C5) = 1.712(2)Å[8]. The molecule exhibits intermolecular interactions (Fig. S1) between C8···H3 with a bond length of 2.830 Å. The intramolecular interactions O17-H17···O10, C7-H7···O10, C8-H8···S1 their bond lengths and angles are listed in Table S1, is in agreement with previously reported compounds.[9] The selected bond lengths and angles are depicted in Table S2.

The Hirshfeld surface analysis is an valid tool to display the intermolecular interactions, and also the packing mode in the crystal structure. Hirshfeld surface computations were carried out using the software CrystalExplorer.[10,11] The area and the volume of the Hirshfeld surfaces are 291.33 Å^2 and 305.96 Å^3, respectively; d_{norm} is mapped over the color scale of –0.0606 to 1.2500 Å; also, 3D surfcaes are made transparent to visualize the arrangement of all atoms in the molecule. Red spots at the C3 and C8 positions over the d_{norm} surafce of the molecule represent strong hydrogen bond interactions, as shown in Fig. 3(i). 2D fingerprnt plots reveal that the percentage of intermolecular contacts in the packing of the molecule are calculated, as illustarted in Figs. 3(a) - 3(f). The H···H intermolecular contacts are shown as pairs of spikes of almost the same length merged in the region 1.15 Å<(d_e+d_i)<1.17 Å. The C···H interaction were seen as two sharp, wide and slightly curved blue colored spikes of the same length, appearing in the region 1.15 Å<(d_e+d_i)<1.20 Å. The O···H close intermolecular contacts appeared as two distict spikes of dark-blue color in the region 1.17 Å<(d_e+d_i)<1.42 Å. The S···H intermolecular contacts are visible as wings of blue color in the region 1.2 Å<(d_e+d_i)<1.9 Å, while C···C intermolecular contacts appeared to be merged as an arrow head pointing downwards left in the region of 1.75 Å<(d_e+d_i)<1.76 Å.[12] The results of intermolecular close contacts are shown in Fig. 3(ii), (a) H···H (42.7%), (b) C···H (25.9%), (c) O···H (15.7%), (d) S···H (7.4%) and (e) C···C (5.4%) and a full plot is shown in Fig. 3(f). The 2D fingerprint plots confirmed that H···H, C···H and H···O are the major intercontacts, while others are minor towards contributions in the packing of the Hirshfeld surface. It also plays an important role in the stabilizing the crystal and molecular structure.

Supporting Information

This material is available free of charge on the Web at http://www.jsac.or.jp/xraystruct/.

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