# Structure report: (2E)-1-(3,4-dimethylphenyl)-3-(3-methylthiophen-2yl)prop-2-en-1-one

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The compound (2E)-1-(3,4-dimethylphenyl)-3-(3-methylthiophen-2-yl) prop-2-en-1-one was synthesized by the reaction of 3-methylthiophene -2-carbaldehyde with 3,4-dimethylacetophenone. The structure of the synthesized compound was characterized by subjecting it to single crystal X-ray diffraction studies. The compound crystallizes in the monoclinic crystal class with space group P 1 21/c 1. The cell parameters are a = 7.1331(4) Å, b = 28.5501(11) Å, c = 7.2354(5) Å, $\beta$ = 116.873(8)°, V = 1314.36(14) Å$^3$ and Z = 4.

## Introduction

Chalcones are precursor of various natural products such as flavonoids, isoflavanoids and key intermediates for synthesis of various heterocyclic scaffolds [1]. Chalcone consists of two aromatic rings joined together by a three carbon $\alpha,\beta$-unsaturated carbonyl system. These compounds have broad range of biological activities such as anticancer, antimalarial activity, anti-TB activity, antiviral, antibacterial, antifilarial activity etc [2]. A vast number of naturally occurring chalcones are polyhydroxylated in the aryl rings. The radical quenching properties of the phenol groups present in many chalcones have raised interest in using the compounds or chalcone rich plant extracts as drugs or food preservatives [3]. Chalcones are finding applications as organic non-linear optical materials (NLO) due to their good SHG conversion efficiencies and higher order nonlinear optical properties [4].Owing to the importance of these chalcones, this new chalcone (2E)-1-(3,4-dimethylphenyl)-3-(3-methylthiophen-2-yl)prop-2-en-1-one was synthesized and its crystal structure is reported.

## Experimental

The compound (2E)-1-(3,4-dimethylphenyl)-3-(3-methylthiophen-2-yl)prop-2-en-1-one was synthesized by the condensation of 3-methylthiophene-2-carbaldehyde (0.01 mol) with 3,4-dimethylacetophenone (0.01 mol) in methanol (60 ml) in the presence of a catalytic amount of sodium hydroxide solution (5 ml, 30%) [5]. After stirring (6 h), the contents of the flask were poured into ice-cold water (500 ml) and left to stand for 5 h. The resulting

crude solid was filtered and dried. The precipitated compound was recrystallized from methanol.

The single crystal X-ray diffraction data was collected on Bruker Kappa Apex using Apex2 software package [6]. The radiation used was graphite monochromatic MoK a radiation. All the data were corrected for Lorentz factor and empirical absorption. The structure was solved by direct method and all the non-hydrogen atoms and hydrogen atoms were found in difference electron density maps. The atomic coordinates and anisotropic temperature factors for non-hydrogen atoms were refined by the full matrix least square method using SHELXTL program package [7].

The molecule exhibits an E configuration with respect to the C10=C11 double bond with the C9—C10—C11—C12 torsion angle being -178.6 (2)°. The bond lengths and bond angles are found to have normal values [8]. The dihedral angle between the benzene and the thiophene ring are 27.79°, indicating that they are non-planar.

In the crystal structure, the molecules are stacked along the *b* axis. Along each axis the adjacent molecules are inverted and arranged in head-to-tail fashion. The parallel molecules between two axes aligned in the same direction. The crystal packing is consolidated by inter molecular C—H···O and C—H···H hydrogen bonding interactions.

## Special Details :

**Geometry:** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**: Refinement of $F^2$ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on $F^2$, conventional R-factors R are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2>\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on $F^2$ are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

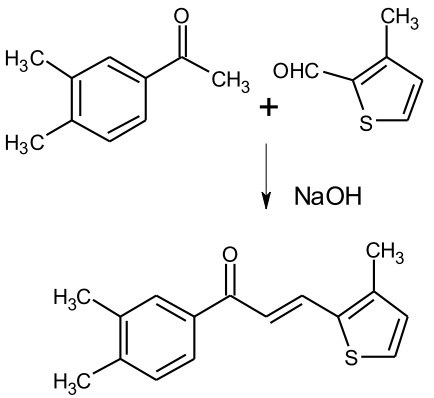


Fig. 1 Reaction Scheme for the title compound

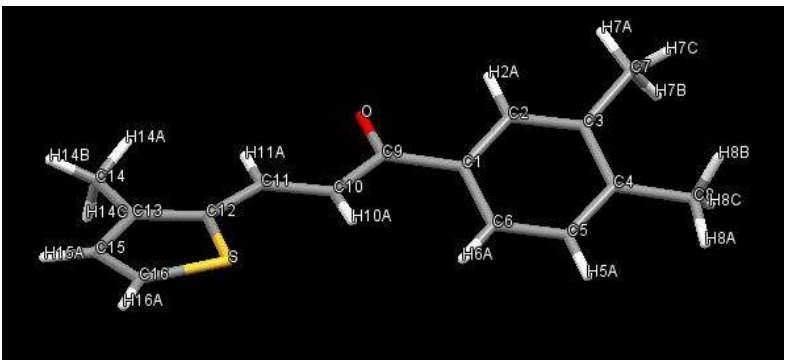


Fig. 2 Molecular structure of the title compound, showing the atom labeling scheme and 50% probability displacement ellipsoids.

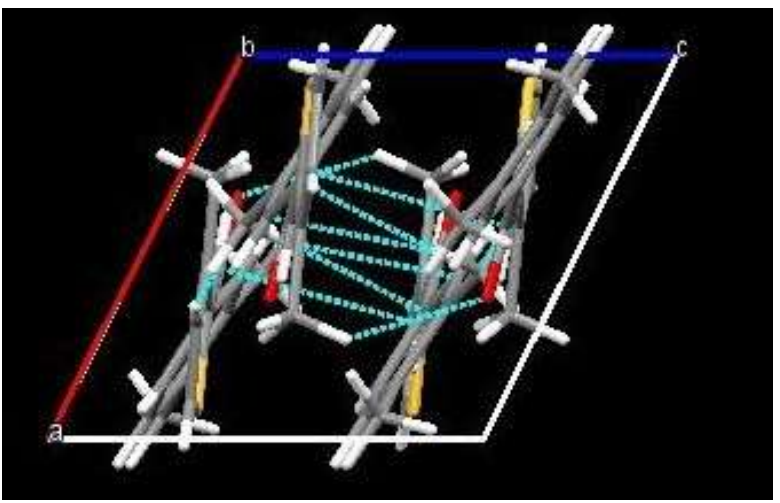


Fig. 3 Packing diagram of the title compound, viewed down the *b* axis. Dashed lines indicate intermolecular bond interaction

Table 1. Crystal data and structure refinement

| | | |
|---|---|---|
| Empirical formula | C16 H16 O S | |
| Formula weight | 256.35 | |
| Temperature | 110(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Monoclinic | |
| Space group | P 1 21/c 1 | |
| Unit cell dimensions | a = 7.1331(4) Å | $\alpha = 90°$. |
| | b=28.5501(11) Å | $\beta = 116.873(8)°$. |
| | c = 7.2354(5)Å | $\gamma = 90°$. |
| Volume | 1314.36(14) $Å^3$ | |
| Z | 4 | |
| Density (calculated) | 1.295Mg/$m^3$ | |
| Absorption coefficient | 0.231 $mm^{-1}$ | |
| F(000) | 544 | |
| Crystal size | 0.46 x 0.34 x 0.14 $mm^3$ | |
| Theta range for data collection | 4.77 to 32.65°. | |
| Index ranges | -10<=h<=10, -41<=k<=36, -10<=l<=9 | |
| Reflections collected | 13232 | |
| Independent reflections | 4397 [R(int) = 0.1711] | |
| Completeness to theta = 25.00° | 99.1 % | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 1.00000 and 0.78949 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 4397 / 0 / 166 | |
| Goodness-of-fit on $F^2$ | 1.151 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0833, wR2 = 0.1625 | |
| R indices (all data) | R1 = 0.1329, wR2 = 0.1759 | |
| Largest diff. peak and hole | 0.567 and -0.395 e.$Å^{-3}$ | |

## Supplementary materials

**Table 2.** Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2x10^3$).U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| S | 721(1) | 2210(1) | 1923(1) | 24(1) |
| O | 6320(3) | 3610(1) | 3452(3) | 28(1) |
| C(1) | 3306(3) | 4057(1) | 2812(3) | 14(1) |
| C(2) | 4215(3) | 4463(1) | 2476(3) | 13(1) |
| C(3) | 3358(3) | 4904(1) | 2378(3) | 13(1) |
| C(4) | 1533(3) | 4948(1) | 2648(3) | 14(1) |
| C(5) | 610(3) | 4541(1) | 2945(3) | 15(1) |
| C(6) | 1463(3) | 4099(1) | 3013(3) | 17(1) |
| C(7) | 4389(3) | 5331(1) | 2021(4) | 17(1) |
| C(8) | 614(3) | 5424(1) | 2637(4) | 18(1) |
| C(9) | 4460(3) | 3602(1) | 3052(4) | 18(1) |
| C(10) | 3316(3) | 3159(1) | 2807(4) | 19(1) |
| C(11) | 4325(4) | 2746(1) | 3094(4) | 19(1) |
| C(12) | 3400(3) | 2287(1) | 2863(4) | 17(1) |
| C(13) | 4443(3) | 1861(1) | 3337(4) | 20(1) |
| C(14) | 6761(4) | 1799(1) | 4173(5) | 35(1) |
| C(15) | 3022(4) | 1479(1) | 2937(4) | 21(1) |
| C(16) | 979(4) | 1615(1) | 2173(4) | 23(1) |

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

| | | | |
|---|---|---|---|
| S-C(16) | 1.711(2) | C(3)-C(4) | 1.407(3) |
| S-C(12) | 1.729(2) | C(3)-C(7) | 1.506(3) |
| O-C(9) | 1.224(3) | C(4)-C(5) | 1.399(3) |
| C(1)-C(6) | 1.392(3) | C(4)-C(8) | 1.506(3) |
| C(1)-C(2) | 1.402(3) | C(5)-C(6) | 1.393(3) |
| C(1)-C(9) | 1.504(3) | C(5)-H(5A) | 0.9500 |
| C(2)-C(3) | 1.387(3) | C(6)-H(6A) | 0.9500 |
| C(2)-H(2A) | 0.9500 | C(7)-H(7A) | 0.9800 |

| | | | |
|---|---|---|---|
| C(7)-H(7B) | 0.9800 | C(6)-C(5)-H(5A) | 119.0 |
| C(7)-H(7C) | 0.9800 | C(4)-C(5)-H(5A) | 119.0 |
| C(8)-H(8A) | 0.9800 | C(1)-C(6)-C(5) | 119.59(17) |
| C(8)-H(8B) | 0.9800 | C(1)-C(6)-H(6A) | 120.2 |
| C(8)-H(8C) | 0.9800 | C(5)-C(6)-H(6A) | 120.2 |
| C(9)-C(10) | 1.473(3) | C(3)-C(7)-H(7A) | 109.5 |
| C(10)-C(11) | 1.347(3) | C(3)-C(7)-H(7B) | 109.5 |
| C(10)-H(10A) | 0.9500 | H(7A)-C(7)-H(7B) | 109.5 |
| C(11)-C(12) | 1.441(3) | C(3)-C(7)-H(7C) | 109.5 |
| C(11)-H(11A) | 0.9500 | H(7A)-C(7)-H(7C) | 109.5 |
| C(12)-C(13) | 1.387(3) | H(7B)-C(7)-H(7C) | 109.5 |
| C(13)-C(15) | 1.427(3) | C(4)-C(8)-H(8A) | 109.5 |
| C(13)-C(14) | 1.492(3) | C(4)-C(8)-H(8B) | 109.5 |
| C(14)-H(14A) | 0.9800 | H(8A)-C(8)-H(8B) | 109.5 |
| C(14)-H(14B) | 0.9800 | C(4)-C(8)-H(8C) | 109.5 |
| C(14)-H(14C) | 0.9800 | H(8A)-C(8)-H(8C) | 109.5 |
| C(15)-C(16) | 1.360(3) | H(8B)-C(8)-H(8C) | 109.5 |
| C(15)-H(15A) | 0.9500 | O-C(9)-C(10) | 121.80(18) |
| C(16)-H(16A) | 0.9500 | O-C(9)-C(1) | 119.29(17) |
| C(16)-S-C(12) | 92.15(10) | C(10)-C(9)-C(1) | 118.91(18) |
| C(6)-C(1)-C(2) | 118.59(17) | C(11)-C(10)-C(9) | 120.3(2) |
| C(6)-C(1)-C(9) | 123.81(17) | C(11)-C(10)-H(10A) | 119.8 |
| C(2)-C(1)-C(9) | 117.51(17) | C(9)-C(10)-H(10A) | 119.8 |
| C(3)-C(2)-C(1) | 122.10(18) | C(10)-C(11)-C(12) | 126.3(2) |
| C(3)-C(2)-H(2A) | 118.9 | C(10)-C(11)-H(11A) | 116.9 |
| C(1)-C(2)-H(2A) | 118.9 | C(12)-C(11)-H(11A) | 116.9 |
| C(2)-C(3)-C(4) | 119.31(17) | C(13)-C(12)-C(11) | 127.03(19) |
| C(2)-C(3)-C(7) | 120.39(18) | C(13)-C(12)-S | 111.10(15) |
| C(4)-C(3)-C(7) | 120.29(17) | C(11)-C(12)-S | 121.87(15) |
| C(5)-C(4)-C(3) | 118.38(17) | C(12)-C(13)-C(15) | 111.58(19) |
| C(5)-C(4)-C(8) | 121.01(19) | C(12)-C(13)-C(14) | 125.17(19) |
| C(3)-C(4)-C(8) | 120.61(17) | C(15)-C(13)-C(14) | 123.25(19) |
| C(6)-C(5)-C(4) | 121.99(18) | C(13)-C(14)-H(14A) | 109.5 |

| | | | |
|---|---|---|---|
| C(13)-C(14)-H(14B) | 109.5 | C(16)-C(15)-H(15A) | 123.3 |
| H(14A)-C(14)-H(14B) | 109.5 | C(13)-C(15)-H(15A) | 123.3 |
| C(13)-C(14)-H(14C) | 109.5 | C(15)-C(16)-S | 111.77(15) |
| H(14A)-C(14)-H(14C) | 109.5 | C(15)-C(16)-H(16A) | 124.1 |
| H(14B)-C(14)-H(14C) | 109.5 | S-C(16)-H(16A) | 124.1 |
| C(16)-C(15)-C(13) | 13.39(18) | | |
| Symmetry transformations used to generate equivalent atoms | | | |

**Table 4.** Anisotropic displacement parameters ($Å^2 x\ 10^3$). The anisotropic displacement factor exponent takes the form: $-2\ \pi 2\ [\ h^2\ a^{*2} U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| S | 18(1) | 16(1) | 33(1) | 3(1) | 7(1) | 1(1) |
| O | 17(1) | 18(1) | 40(1) | 1(1) | 5(1) | 1(1) |
| C(1) | 13(1) | 14(1) | 11(1) | 0(1) | 1(1) | -2(1) |
| C(2) | 10(1) | 17(1) | 8(1) | 0(1) | 1(1) | -1(1) |
| C(3) | 14(1) | 14(1) | 9(1) | 0(1) | 2(1) | -2(1) |
| C(4) | 14(1) | 16(1) | 9(1) | -1(1) | 2(1) | 0(1) |
| C(5) | 11(1) | 22(1) | 11(1) | -1(1) | 4(1) | -3(1) |
| C(6) | 18(1) | 15(1) | 12(1) | 0(1) | 4(1) | -6(1) |
| C(7) | 18(1) | 14(1) | 16(1) | 0(1) | 7(1) | -4(1) |
| C(8) | 18(1) | 19(1) | 14(1) | -2(1) | 4(1) | 3(1) |
| C(9) | 18(1) | 14(1) | 16(1) | 1(1) | 2(1) | -1(1) |
| C(10) | 17(1) | 15(1) | 19(1) | 2(1) | 4(1) | -2(1) |
| C(11) | 21(1) | 15(1) | 20(1) | 1(1) | 7(1) | -2(1) |
| C(12) | 15(1) | 15(1) | 17(1) | 1(1) | 5(1) | -1(1) |
| C(13) | 21(1) | 16(1) | 23(1) | 0(1) | 10(1) | 1(1) |
| C(14) | 20(1) | 27(1) | 56(2) | 1(1) | 17(1) | 1(1) |
| C(15) | 26(1) | 13(1) | 23(1) | -2(1) | 12(1) | -1(1) |
| C(16) | 23(1) | 14(1) | 30(1) | -2(1) | 10(1) | -4(1) |

**Table 5.** Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^3$)

| | x | y | z | U(eg) |
|---|---|---|---|---|
| H(2A) | 5458 | 4435 | 2311 | 16 |
| H(5A) | -636 | 4568 | 3106 | 18 |
| H(6A) | 791 | 3828 | 3196 | 20 |
| H(7A) | 5589 | 5235 | 1795 | 25 |
| H(7B) | 4877 | 5536 | 3236 | 25 |
| H(7C) | 3372 | 5500 | 799 | 25 |
| H(8A) | -649 | 5388 | 2836 | 27 |
| H(8B) | 244 | 5578 | 1307 | 27 |
| H(8C) | 1650 | 5614 | 3762 | 27 |
| H(10A) | 1859 | 3164 | 2445 | 22 |
| H(11A) | 5788 | 2758 | 3486 | 23 |
| H(14A) | 7353 | 2069 | 3783 | 52 |
| H(14B) | 7050 | 1513 | 3600 | 52 |
| H(14C) | 7402 | 1775 | 5687 | 52 |
| H(15A) | 3458 | 1161 | 3181 | 25 |
| H(16A) | -165 | 1405 | 1820 | 28 |

**Table 6.** Torsion angles [°]

| | |
|---|---|
| C(6)-C(1)-C(2)-C(3) | 1.2(3) |
| C(9)-C(1)-C(2)-C(3) | -175.42(19) |
| C(1)-C(2)-C(3)-C(4) | 0.8(3) |
| C(1)-C(2)-C(3)-C(7) | 179.81(19) |
| C(2)-C(3)-C(4)-C(5) | -1.9(3) |
| C(7)-C(3)-C(4)-C(5) | 179.09(18) |
| C(2)-C(3)-C(4)-C(8) | 177.48(19) |
| C(7)-C(3)-C(4)-C(8) | -1.5(3) |
| C(3)-C(4)-C(5)-C(6) | 1.1(3) |
| C(8)-C(4)-C(5)-C(6) | -178.3(2) |
| C(2)-C(1)-C(6)-C(5) | -2.1(3) |

| | |
|---|---|
| C(9)-C(1)-C(6)-C(5) | 174.3(2) |
| C(4)-C(5)-C(6)-C(1) | 1.0(3) |
| C(6)-C(1)-C(9)-O | -158.8(2) |
| C(2)-C(1)-C(9)-O | 17.6(3) |
| C(6)-C(1)-C(9)-C(10) | 20.9(3) |
| C(2)-C(1)-C(9)-C(10) | -162.63(19) |
| O-C(9)-C(10)-C(11) | 2.4(4) |
| C(1)-C(9)-C(10)-C(11) | -177.4(2) |
| C(9)-C(10)-C(11)-C(12) | -178.6(2) |
| C(10)-C(11)-C(12)-C(13) | -173.5(2) |
| C(10)-C(11)-C(12)-S | 6.2(4) |
| C(16)-S-C(12)-C(13) | 0.40(19) |
| C(16)-S-C(12)-C(11) | -179.3(2) |
| C(11)-C(12)-C(13)-C(15) | 179.2(2) |
| S-C(12)-C(13)-C(15) | -0.6(3) |
| C(11)-C(12)-C(13)-C(14) | -0.5(4) |
| S-C(12)-C(13)-C(14) | 179.8(2) |
| C(12)-C(13)-C(15)-C(16) | 0.5(3) |
| C(14)-C(13)-C(15)-C(16) | -179.8(3) |
| C(13)-C(15)-C(16)-S | -0.2(3) |
| C(12)-S-C(16)-C(15) | -0.1(2) |

Symmetry transformations used to generate equivalent atoms

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