



Synthesis and crystal structure studies of 3,3-bis(methylthio)-1-(4-(trifluoromethyl) phenyl)prop-2-en-1-one and 3,3-bis(methylthio)-1-(thiophen-2-yl)prop-2-en-1-one derivatives

M. Munvar Hussain¹, Shamantha Kumar², A. C. Vinayaka³, T. Bhuvaneswara Babu⁴,
B. G. Devika⁵, B. M Rajesh⁴ and B. H. Doreswamy^{2*}

1. Department of Physics, KNS Institute of Technology, Bangalore 560 064, **INDIA**

2. Department of Physics, SJB Institute of Technology, Kengeri, Bangalore 560 060, **INDIA**

3. Department of Studies in Chemistry, Manasagangotri, University of Mysore, Mysore-570006, Karnataka, **INDIA**

4. Department of Physics, RV College of Engineering, Bangalore 560 059, **INDIA**

5. Department of Physics, SJMIT, Chitradurga, 577 501, **INDIA**

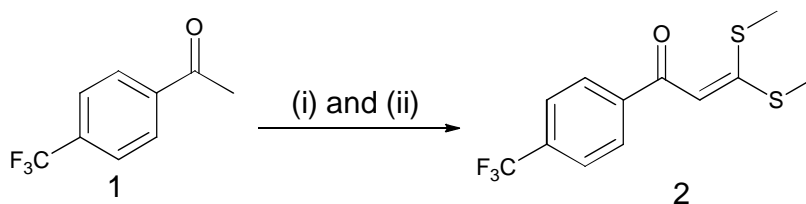
Email: dorephy@gmail.com

Accepted on 16th December 2017, Published online on 27th January 2018

ABSTRACT

The title compounds of 3,3-bis(methylthio)-1-(4-(trifluoromethyl) phenyl)prop-2-en-1-one (J1) and 3,3-bis(methylthio)-1-(thiophen-2-yl)prop-2-en-1-one (J2) were synthesized and characterized by single crystal X-ray diffraction method. The structure was confirmed by X-ray diffraction studies. The compound (J1) crystallizes in monoclinic system under the space group $P2_1/c$, with cell parameters $a = 11.8403(8)\text{\AA}$, $b = 8.5517(7)\text{\AA}$, $c = 11.7986(10)\text{\AA}$, $\beta = 115.158(5)^\circ$ and $Z=4$. Similarly, compound (J2) crystallizes in monoclinic system under the space group $P2_1/n$, with cell parameters $a = 12.962(2)\text{\AA}$, $b = 8.1006(11)\text{\AA}$, $c = 13.348(2)\text{\AA}$, $\beta = 114.945(9)^\circ$ and $Z=4$. Crystal structure (J1 and J2) stabilized by an $C2-H9.O7$ and intramolecular $C(7)-H(6).O(12)$ hydrogen bonds.

Graphical Abstract:

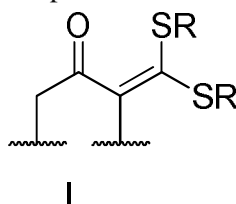


Reaction condition: (i) Potassium-*tert*-butoxide, *tert*-butanol, CS_2 , room temperature, 4h;
(ii) MeI, 0 °C to room temperature, 6h.

Keywords: Ketene dithio acetals, Crystal structure, C-H...O interaction.

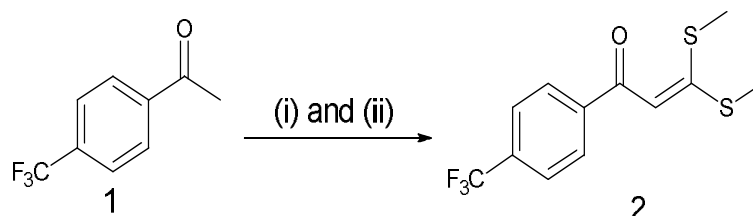
INTRODUCTION

The ketene dithioacetals are the di-sulphur analogue of ketene and ketenes are the organic compounds having functional group $R_1R_2C=C=O$, the simplest example is the ethenone (where R_1 and R_2 are H). The classifications of these organic compounds are mainly based on the type of substituent at α -carbon atom of the ketene dithioacetals. The various type substituents at α -position include functional groups like nitro, cyano, sulfonyl, silyl and carbonyl etc. Among these α -oxo ketene dithioacetals (I) having carbonyl group at α -position are reported as a versatile intermediate and serving as 1,3-electrophilic synthones for the preparation of different kinds of synthetically useful building blocks and biologically significant heterocyclic compounds. The presence of electron releasing (alkyl thio) and electron withdrawing (carbonyl) group in α -oxo ketene dithioacetals are in conjugation with double bond, hence these intermediate undergo reaction with both electrophile and nucleophile reagents.



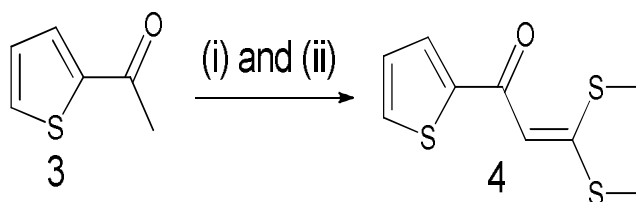
Further, the good leaving property of alkyl thio group in α -oxo-ketene-S,S-acetals provides a reactive site for the substitution or cyclocondensation reaction with various nitrogen nucleophiles such as amines, phenyl hydrazine and hydroxyl amine hydrochloride. The diversity of these intermediates was demonstrated by different type of reactions such as synthesis of multisubstituted pyrroles, [1] annulated aromatic compounds [2], pyrazoles [3], furans [4], thioesters [5] and enaminones [6].

MATERIALS AND METHODS



Reaction condition: (i) Pottasium-*tert*-butoxide, *tert*-butanol, CS_2 , room temeperature, 4h;
(ii) MeI, 0 °C to room temeperature, 6h.

Scheme 1: Synthesis of 3,3-*bis*(methylthio)-1-(4-(trifluoromethyl)phenyl)prop-2-en-1-one(2)



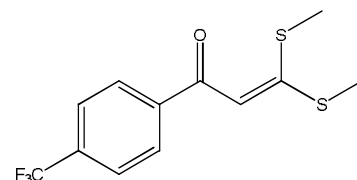
Reaction condition: (i) Pottasium-*tert*-butoxide, *tert*-butanol, CS_2 , 0 °C to room temeperature, 4h;
(ii) MeI, 0 °C to room temeperature, 6h.

Scheme 2: Synthesis of 3,3-*bis*(methylthio)-1-(thiophen-2-yl)prop-2-en-1-one (4)

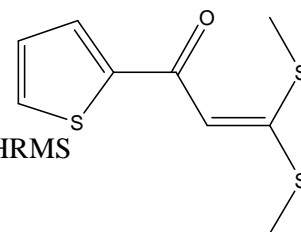
Synthesis of 3,3-bis(methylthio)-1-(4-(trifluoromethyl)phenyl)prop-2-en-1-one(2): A mixture of *p*-(trifluoromethyl) acetophenone (1, 0.01mol) and CS₂ (0.01mol) was added drop wisely to the suspension of potassium-*tert*-butoxide (0.02mol) in *tert*-butanol (20 mL) at room temperature. The reaction mixture was allowed to stir at room temperature for 4 h. To this, methyl iodide (0.025mol) was added drop wisely at 0 °C and stirred vigorously for 6h. After completion of reaction, water and ethyl acetate was added to the reaction mixture and the desired product (2) in organic layer was separated. The obtained organic layer was dried using anhydrous sodium sulphate and solvent was removed under reduced pressure. The crystallization of compound 2 was performed using methanol at room temperature.

Synthesis of 3,3-bis(methylthio)-1-(thiophen-2-yl)prop-2-en-1-one (4): The solution of 2-Acetyl thiophene (3, 0.01mol) and CS₂ (0.01mol) in *tert*-butanol was added slowly to the suspension of potassium-*tert*-butoxide (0.02 mmol) in *tert*-butanol (20 mL) at 0 °C. After completion of addition, reaction mixture was allowed to stir for 4 h at room temperature. Methyl iodide (0.025mol) was then added drop wisely at 0 °C and stirred vigorously for 6h. After completion of reaction, water and chloroform was added to the reaction mixture and the desired product (4) in organic layer was separated. The obtained organic layer was dried using anhydrous sodium sulphate and solvent was removed under reduced pressure. The crystallization of compound 4 was performed using methanol at room temperature.

Characterization data of 3,3-Bis(methylthio)-1-(4-(trifluoromethyl)phenyl)prop-2-en-1-on (2): Yellow solid (74%); mp 96-98 °C; ¹H NMR (400 MHz, CDCl₃) δ = 7.99 (d, *J* = 8.4 Hz, 2H, Ar-H), 7.69 (d, *J* = 8.4 Hz, 2H, Ar-H), 6.72 (s, 1H, HC=C-), 2.57 (s, 3H, -SCH₃), 2.54 (s, 3H, -SCH₃) ppm; HRMS (ESI) *m/z* calcd for C₁₂H₁₁F₃OS₂ [M+H]⁺ 292.0203; Found 292.0200.



Characterization data of 3,3-bis(methylthio)-1-(thiophen-2-yl)prop-2-en-1-one (4): Yellow solid (59%); mp 90-92 °C; ¹H NMR (400 MHz, CDCl₃) δ = 8.41 (d, *J* = 8.6 Hz, 1H, Ar-H), 8.2 (d, *J* = 8.4 Hz, 1H, Ar-H), 7.6 (m, 1H, Ar-H), 6.71 (s, 1H, HC=C-), 2.68 (s, 3H, -SCH₃), 2.66 (s, 3H, -SCH₃) ppm; HRMS (ESI) *m/z* calcd for C₉H₁₀OS₃ [M+H]⁺ 230.3701; Found 230.3703.



Crystal structure determination: A single crystal of the molecules (J1 and J2) with dimensions of 0.30 × 0.25 × 0.20 mm were chosen for X-ray diffraction studies. The crystallographic data were collected at 293K on a Bruker Proteum2 CCD diffractometer equipped with an X-ray generator operating at 45 kV and 10 mA with CuKα radiation (1.54178Å). Raw data was processed and reduced by using SAINT PLUS [7]. The crystal structure was solved by direct methods using SHELXS-97 [8-9]. All non-hydrogen atoms were revealed in the first Fourier map itself. Anisotropic refinement of non-hydrogen atoms was started at this stage. Subsequent refinements were carried out with anisotropic thermal parameters for non-hydrogen atoms and isotropic temperature factors for the hydrogen atoms which were placed at chemically acceptable positions. Full-matrix least squares refinement was carried out using SHELXL-97 [8-9] with a final residual values of R1 = 0.0567 and 0.0621 for molecules J1 and J2, respectively. The thermal ellipsoid plot [10] at 50% probability and packing diagram of the molecules J1 and J2 are represented in figure 1. A summary of experimental and crystallographic data for molecules J1 and J2 are given in Table 1 and 2 respectively.

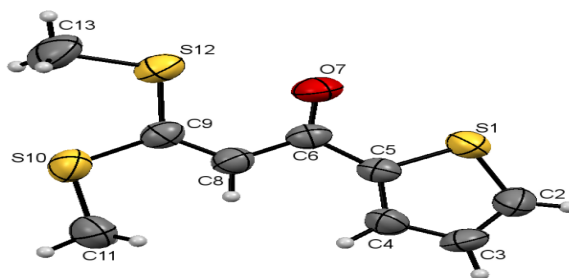


Figure 1: ORTEP of the molecule (J1) with thermal ellipsoids drawn at 50% probability

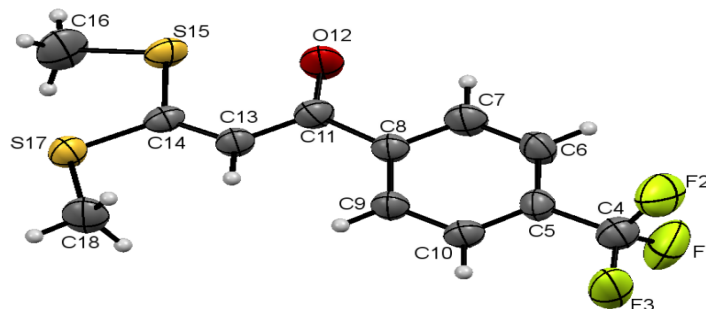


Figure 1: ORTEP of the molecule (J2) with thermal ellipsoids drawn at 50% probability

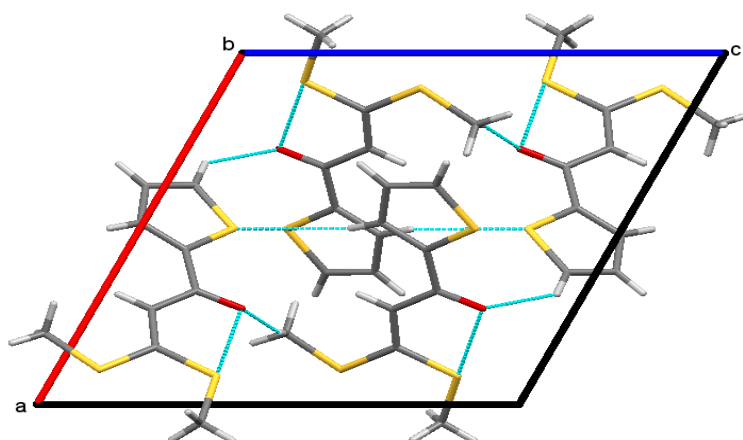


Figure 2: Packing diagram of the molecule (J1) when viewed along bc plane

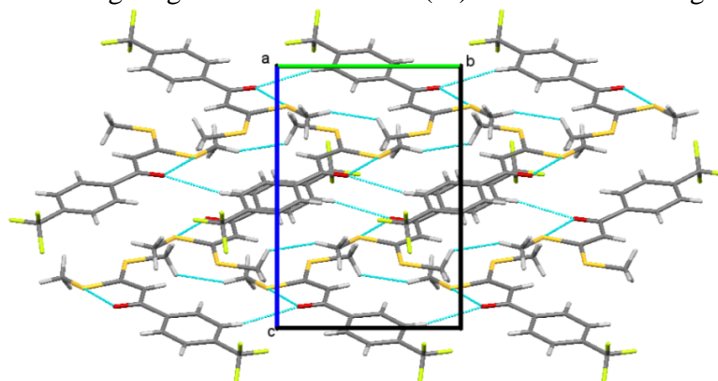


Figure 2: Packing diagram of the molecule (J2) when viewed along ab plane

Table 1a. Crystal data and structure refinement for J1

Identification code	j1
Empirical formula	C ₉ H ₁₀ N ₀ O S ₃
Formula weight	230.35
Temperature	293(2) K
Wavelength	1.54178 Å
Crystal system, space group	Monoclinic, P2 ₁ /c
Unit cell dimensions	a = 11.8403(8) Å, b = 8.5517(7) Å
	c = 11.7986(10) Å, β = 115.158(5)°
Volume	
Z, Calculated density	4, 1.415 Mg/m ³
Absorption coefficient	5.930 mm ⁻¹
F ₀₀₀	480
Crystal size	0.30 x 0.25 x 0.20 mm
Theta range for data collection	6.622 to 64.329°
Limiting indices	-13 ≤ h ≤ 13, -9 ≤ k ≤ 9, -11 ≤ l ≤ 13
Reflections collected / unique	4378 / 1425 [R(int) = 0.0840]
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	1425 / 0 / 120
Goodness-of-fit on F ²	0.874
Final R indices [I > 2σ(I)]	R1 = 0.0567, wR2 = 0.1454
R indices (all data)	R1 = 0.0960, wR2 = 0.1756
Largest diff. peak and hole	0.340 and -0.275 e.Å ⁻³

Table 1b. Bond lengths [Å] and angles [°] for J1

Atoms	Length	Atoms	Length
S(1)-C(2)	1.682(7)	C(2)-S(1)-C(5)	92.8(3)
S(1)-C(5)	1.711(5)	C(9)-S(12)-C(13)	104.9(4)
S(12)-C(9)	1.741(6)	C(9)-S(10)-C(11)	104.1(3)
S(12)-C(13)	1.814(7)	C(4)-C(5)-C(6)	130.2(5)
S(10)-C(9)	1.750(6)	C(4)-C(5)-S(1)	110.3(4)
S(10)-C(11)	1.786(8)	C(6)-C(5)-S(1)	119.5(4)
O(7)-C(6)	1.236(7)	C(9)-C(8)-C(6)	124.0(6)
C(5)-C(4)	1.365(8)	C(9)-C(8)-H(006)	118
C(5)-C(6)	1.469(7)	C(6)-C(8)-H(006)	118
C(8)-C(9)	1.367(7)	C(8)-C(9)-S(12)	121.8(5)
C(8)-C(6)	1.444(8)	C(8)-C(9)-S(10)	122.6(5)
C(8)-H(006)	0.93	S(12)-C(9)-S(10)	115.5(3)
C(4)-C(3)	1.407(8)	C(5)-C(4)-C(3)	113.0(6)
C(4)-H(008)	0.93	C(5)-C(4)-H(008)	123.5

C(2)-C(3)	1.363(10)	C(3)-C(4)-H(008)	123.5
C(2)-H(9)	0.93	O(7)-C(6)-C(8)	123.6(5)
C(3)-H(00B)	0.93	O(7)-C(6)-C(5)	119.4(5)
C(11)-H(00A)	0.96	C(8)-C(6)-C(5)	116.9(5)
C(11)-H(00C)	0.96	C(3)-C(2)-S(1)	111.7(4)
C(11)-H(00D)	0.96	C(3)-C(2)-H(9)	124.1
C(13)-H(00E)	0.96	S(1)-C(2)-H(9)	124.1
C(13)-H(00F)	0.96	C(2)-C(3)-C(4)	112.2(6)
C(13)-H(00G)	0.96	C(2)-C(3)-H(00B)	123.9

Table 1c. Torsion angles [°] for J1

C(2)-S(1)-C(5)-C(4)	0.8(4)
C(2)-S(1)-C(5)-C(6)	-179.7(4)
C(6)-C(8)-C(9)-S(12)	-2.6(7)
C(6)-C(8)-C(9)-S(10)	177.4(4)
C(13)-S(12)-C(9)-C(8)	-179.7(5)
C(13)-S(12)-C(9)-S(10)	0.3(4)
C(11)-S(10)-C(9)-C(8)	-1.3(5)
C(11)-S(10)-C(9)-S(12)	178.7(3)
C(6)-C(5)-C(4)-C(3)	180.0(5)
S(1)-C(5)-C(4)-C(3)	-0.7(6)
C(9)-C(8)-C(6)-O(7)	-2.7(9)
C(9)-C(8)-C(6)-C(5)	179.2(5)
C(4)-C(5)-C(6)-O(7)	174.9(6)
S(1)-C(5)-C(6)-O(7)	-4.4(7)
C(4)-C(5)-C(6)-C(8)	-7.0(8)
S(1)-C(5)-C(6)-C(8)	173.7(4)
C(5)-S(1)-C(2)-C(3)	-0.8(5)
S(1)-C(2)-C(3)-C(4)	0.5(7)
C(5)-C(4)-C(3)-C(2)	0.1(8)

Table 2a. Crystal data and structure refinement for J2.

Identification code	j2
Empirical formula	C ₁₂ H ₁₁ F ₃ O S ₂
Formula weight	292.33
Temperature	293(2) K
Wavelength	1.54178 Å
Crystal system, space group	Monoclinic, P2(1)/n
Unit cell dimensions	a = 12.962(2) Å alpha = 90°.
	b = 8.1006(11) Å beta = 114.945(9)°.

	$c = 13.348(2) \text{ \AA}$ $\gamma = 90^\circ$
Volume	$1270.8(3) \text{ \AA}^3$
Z, Calculated density	4, 1.528 Mg/m^3
Absorption coefficient	4.037 mm^{-1}
F_{000}	600
Crystal size	$0.30 \times 0.25 \times 0.20 \text{ mm}$
Theta range for data collection	6.576 to 63.657°
Limiting indices	$-14 \leq h \leq 11$, $-5 \leq k \leq 9$, $-8 \leq l \leq 15$
Reflections collected / unique	5708 / 1858 [R(int) = 0.0460]
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1858 / 0 / 165
Goodness-of-fit on F^2	1.078
Final R indices [I > 2 σ (I)]	R1 = 0.0621, wR2 = 0.1746
R indices (all data)	R1 = 0.0733, wR2 = 0.1850
Largest diff. peak and hole	0.365 and $-0.501 \text{ e.\AA}^{-3}$

Table 2b. Bond lengths [\AA] and angles [$^\circ$] for J2

Atoms	Length	Atoms	Length
S(15)-C(14)	1.743(3)	C(14)-S(15)-C(16)	103.38(19)
S(15)-C(16)	1.805(5)	C(14)-S(17)-C(18)	104.72(19)
S(17)-C(14)	1.734(4)	C(8)-C(9)-C(10)	120.7(4)
S(17)-C(18)	1.782(4)	C(8)-C(9)-H(2)	119.7
F(3)-C(4)	1.313(4)	C(10)-C(9)-H(2)	119.7
C(9)-C(8)	1.392(5)	C(14)-C(13)-C(11)	123.7(3)
C(9)-C(10)	1.398(5)	C(14)-C(13)-H(8)	118.2
C(9)-H(2)	0.93	C(11)-C(13)-H(8)	118.2
O(12)-C(11)	1.225(4)	C(9)-C(8)-C(7)	117.9(3)
C(13)-C(14)	1.368(5)	C(9)-C(8)-C(11)	124.4(4)
C(13)-C(11)	1.427(5)	C(7)-C(8)-C(11)	117.7(3)
C(13)-H(8)	0.93	O(12)-C(11)-C(13)	121.5(3)
C(8)-C(7)	1.393(5)	O(12)-C(11)-C(8)	118.5(4)
C(8)-C(11)	1.503(5)	C(13)-C(11)-C(8)	120.0(3)
F(2)-C(4)	1.321(5)	C(13)-C(14)-S(17)	123.8(3)
C(6)-C(7)	1.368(5)	C(13)-C(14)-S(15)	120.9(3)
C(6)-C(5)	1.375(5)	S(17)-C(14)-S(15)	115.35(19)
C(6)-H(5)	0.93	C(7)-C(6)-C(5)	120.5(4)
F(1)-C(4)	1.335(5)	C(7)-C(6)-H(5)	119.7
C(10)-C(5)	1.371(5)	C(5)-C(6)-H(5)	119.7
C(10)-H(3)	0.93	C(5)-C(10)-C(9)	119.6(3)
C(5)-C(4)	1.493(5)	C(5)-C(10)-H(3)	120.2

C(7)-H(6)	0.93	C(9)-C(10)-H(3)	120.2
C(18)-H(10A)	0.96	C(10)-C(5)-C(6)	120.1(3)
C(18)-H(10B)	0.96	C(10)-C(5)-C(4)	121.0(3)
C(18)-H(10C)	0.96	C(6)-C(5)-C(4)	118.8(4)
C(16)-H(11A)	0.96	C(6)-C(7)-C(8)	121.1(3)
C(16)-H(11B)	0.96	C(6)-C(7)-H(6)	119.4
C(16)-H(11C)	0.96	C(8)-C(7)-H(6)	119.4

Table 2c. Torsion angles [°] for J2

C(10)-C(9)-C(8)-C(7)	0.7(6)
C(10)-C(9)-C(8)-C(11)	180.0(4)
C(14)-C(13)-C(11)-O(12)	8.9(6)
C(14)-C(13)-C(11)-C(8)	-170.5(4)
C(9)-C(8)-C(11)-O(12)	177.8(4)
C(7)-C(8)-C(11)-O(12)	-2.9(6)
C(9)-C(8)-C(11)-C(13)	-2.8(6)
C(7)-C(8)-C(11)-C(13)	176.4(4)
C(11)-C(13)-C(14)-S(17)	-177.9(3)
C(11)-C(13)-C(14)-S(15)	1.5(5)
C(18)-S(17)-C(14)-C(13)	2.0(4)
C(18)-S(17)-C(14)-S(15)	-177.5(2)
C(16)-S(15)-C(14)-C(13)	172.2(3)
C(16)-S(15)-C(14)-S(17)	-8.3(3)
C(8)-C(9)-C(10)-C(5)	-0.6(6)
C(9)-C(10)-C(5)-C(6)	0.4(6)
C(9)-C(10)-C(5)-C(4)	179.1(4)
C(7)-C(6)-C(5)-C(10)	-0.5(6)
C(7)-C(6)-C(5)-C(4)	-179.1(4)
C(5)-C(6)-C(7)-C(8)	0.6(6)
C(9)-C(8)-C(7)-C(6)	-0.8(6)
C(11)-C(8)-C(7)-C(6)	179.9(4)
C(10)-C(5)-C(4)-F(3)	2.4(6)
C(6)-C(5)-C(4)-F(3)	-178.9(4)
C(10)-C(5)-C(4)-F(2)	-120.2(4)
C(6)-C(5)-C(4)-F(2)	58.4(5)
C(10)-C(5)-C(4)-F(1)	121.7(4)
C(6)-C(5)-C(4)-F(1)	-59.6(5)

RESULTS AND DISCUSSION

The X-ray molecular structure reveals, compound (J1) crystallizes in monoclinic system under the space group $P2_1/c$, with cell parameters $a = 11.8403(8)\text{\AA}$, $b = 8.5517(7)\text{\AA}$, $c = 11.7986(10)\text{\AA}$, $\beta = 115.158(5)^\circ$ and $Z=4$. The bond lengths and bond angles agree with the standard values and are comparable to a related structure. The thiophene unit are essentially coplanar, the maximum deviation from the mean plane being $0.006(5)\text{\AA}$ for atom C5. Thiophene ring makes a torsion angle of $-4.5(7)^\circ$ with the propanone group (S1/C5/C6/O7). Also, The methylthio units are present in a -syn-periplanar (C11/S10/C9/C8) and -anti-periplanar (C13/S12/C9/C8) conformation with respect to the propanone moiety, as indicated by the torsion angle values of $1.3(6)^\circ$ and $-179.7(5)^\circ$, respectively. The crystal structure is stabilized by an C2--H9...O7 hydrogen bonds. The packing diagram of the molecule viewed along bc plane as shown in figure 2. Similarly, crystal structure analysis showed that, compound (J2) crystallizes in monoclinic system under the space group $P2_1/n$, with cell parameters $a = 12.962(2)\text{\AA}$, $b = 8.1006(11)\text{\AA}$, $c = 13.348(2)\text{\AA}$, $\beta = 114.945(9)^\circ$ and $Z=4$. The mean plane of the trifluoromethylphenyl moiety are essentially coplanar, the maximum deviation from the mean plane being $0.024(5)\text{\AA}$ for atom C4. Benzene ring makes a torsion angle of $-2.9(2)^\circ$ with the propanone group (C7/C8/C11/O12). Also, The methylthio units are present in a syn-periplanar (C18/S17/C14/C13) and anti-periplanar (C16/S15/C14/C13) conformation with respect to the propanone moiety, as indicated by the torsion angle values of $2.0(4)^\circ$ and $172.2(4)^\circ$, respectively. There are no classical hydrogen bonds. Further, the crystal structure is stabilized by intramolecular C(7)--H(6)...O(12) hydrogen bonds. The packing diagram of the molecule viewed along ab plane as shown in Figure 2.

APPLICATIONS

This Study is useful to know the crystal structure of the synthesized compound.

ACKNOWLEDGEMENTS

The authors would like to thank the KNS Institute of Technology, SJB Institute of Technology and RV College of Engineering, Bengaluru, for their support.

REFERENCES

- [1] H.S. Prakash Rao, S. Sivakumar, Aroylketene dithioacetal chemistry: Facile synthesis of 4-aryol-3-methylsulfanyl-2-tosylpyrroles from aroylketene dithioacetals and TosMIC, *Beilstein J. Org. Chem.*, **2007**, 3, 31.
- [2] M. P. Balu, G. Singh, H. Ila, H. Junjappa, Cycloaromatization of δ -oxoketendithioacetals with benzylmagnesium chloride: A novel naphthalene annelation reaction, *Tetrahedron Lett.*, **1986**, 27, 117–120.
- [3] S. Kumar, H. Ila, H. Junjappa, Heteroaromatic annulation studies on 10,11-dihydro-11-[bis(methylthio)methylene]dibenzoxepin-10-one: a facile access to novel dibenzoxepino[4,5]-fused heterocycles, *Tetrahedron*, **2007**, 63, 10067–10076.
- [4] X. Yang, F. Hu, H. Di, X. Cheng, D. Li, X. Kan, X. Zou, Q. Zhang, A convenient base-mediated synthesis of 3-aryol-4-methyl (or benzyl)-2-methylthio furans from α -oxo ketene dithioacetals and propargyl alcohols via domino coupling/annulations, *Org. Biomol. Chem.*, **2014**, 12, 8947–8951.
- [5] B.K. Mehta, H. Ila, H. Junjappa, Highly chemoselective and stereoselective conjugate addition of benzyl copper reagents to α -oxoketene dithioacetals, *Tetrahedron Lett.*, **1995**, 36, 1925–1928.
- [6] A.C. Vinayaka, T.R. Swaroop, C. Prasanna Kumara, K.S. Rangappa, M.P. Sadashiva, Transition-metal-free solid phase synthesis of 1,2-disubstituted 4-quinolones via the regiospecific synthesis of enamines, *RSC Adv.*, **2016**, 6, 11528–11535.
- [7] Bruker, APEX2 and SAINT, Bruker AXS Inc, Madison, Wisconsin, USA, **2009**.
- [8] G.M. Sheldrick, *Acta. Cryst. A* 64, 112–122, 2008.

- [9] G.M. Sheldrick, Acta Cryst. C71, 3-8, **2015**.
[10] A. L. Spek, Acta Cryst. D65, 148-155, **2009**.

AUTHOR ADDRESS

1. **B. H. Doreswamy**
Department of Physics,
SJB Institute of Technology,
Kengeri, Bangalore 560 060, India
E-mail: dorephy@gmail.com