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A STUDY ON THE SYNTHESIS AND CRYSTALLOGRAPHIC CHARACTERIZATION OF AN ETHOXYCHALCONE CRYSTAL

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Abstract **- Base catalyzed Claisen-Schmidt reaction was involved in the synthesis of an ethoxychalcone. Slow evaporation solution method was associated with the development of the ether based-chalcone crystals. Its structure was characterized by making use of crystallographic study. This derivative of the chalcone crystallizes in monoclinic system with a noncentro-symmetric space group triclinic P2 (1)/n. The unit cell parameters a = 5.7634(2) Å, b = 8.2218(2) Å c = 12.0915(3) Å, α = 105.5413(14) °, β = 102.5395(16)°, γ = 97.4291(14)°, Z = 2 and V = 528.03(3) Å^3. From the data obtained Oak Ridge thermal ellipsoid plot (ORTEP) and packing fraction were derived. In view of the fact that this crystalline ethoxychalcone is noncentrosymmetric it may materialize as non linear optical (NLO) material.**

Keywords: **Ethoxychalcone, Crystal, Crystallography, Packing Fraction.**

I. INTRODUCTION

Chalcones belonging to the flavonoid family constitute an important group of natural products due to their unforeseen pharmacological potential. Chemically they consist of open-chain flavonoids in which the two aromatic rings are joined by a three-carbon alpha,beta-unsaturated carbonyl system. The radical quenching properties of the phenolic groups present in many chalcones have raised interest in using the compounds or chalcone-rich plant extracts as drugs or food preservatives. Chalcones have been reported to possess many exciting activities which include anti-inflammatory,antimicrobial, antifungal, antioxidant, cytotoxic, anti-tumor and anticancer (Nowakowska, 2007). A number of chalcones having hydroxy or alkoxy groups in different position have been observed to possess vasodilatory (Ram et al., 2000), antimitotic (Khatib et al., 2005) and antimalarial activities (Papo & Shai, 2003).

II. PROCEDURE

A. Experimental

The title compound was synthesized by the base catalyzed Claisen-Schmidt reaction according to the published procedures(Shettigar et al.,2006;Patil et al 2007).In a 250ml round-bottom flask acetone (0.79 ml) and 4-Ethoxybenzaldehyde (0.02 ml) were placed and 120 ml of absolute alcohol were added. The mixture was stirred at room temperature for 5 min. Then 10% Sodium hydroxide solution was added and the mixture was stirred for 2 h. The precipitate generated by adding a sufficient amount of dilute hydrochloric acid was filtered, washed with water and dried. The crude product was recrystallised twice from absolute alcohol yielding clear colourless block-like crystals (yield 78%; M.p.296 K)

B. Refinement

All H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C-H distances 0.93-0.96 Å.

C. Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2009).

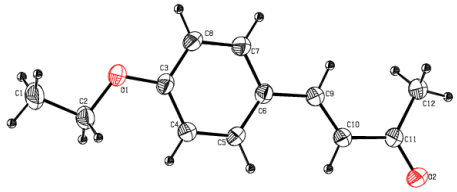

Figure 1 ORTEP view of the molecule with the atom – labeling scheme. The displacement ellipsoids are drawn at the 40% probability level.

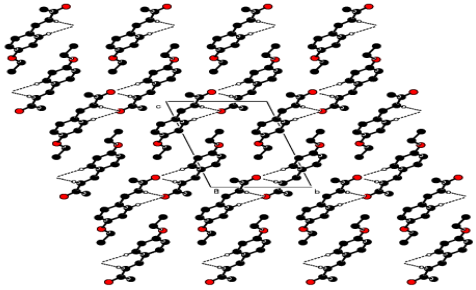

Figure.2 The packing arrangement of molecules viewed down the b axis Hydrogen bonds are shown with dashed lines.

Table-1
Crystal data of (3*E*)-4-(4-ethoxyphenyl) but-3-en-2-one

Empirical formula	C_{12} H_{14} O_2
Formula weight	190.23
Wavelength	0.71073 A
Crystal system	triclinic
space group	P -1
Unit cell dimensions	*a* = 5.7634(2) Å, α = 105.5413(14)° b = 8.2218(2) Å β = 102.5395(16)° c = 12.0915(3) Å, γ = 97.4291(14)° *Z = 2 and* *V* = 528.03(3) Å^3
F(000)	204
Crystal size	0.30 x 0.25 x 0.25 mm
Theta range for data collection	1.81 to 25.00 deg
Limiting indices	-6<=h<=6, -9<=k<=9, -14<=l<=14
Reflections collected / unique	7360 / 1842 [R(int) = 0.0198]
Completeness to theta	25.00 99.1 %
Absorption correction	None
Max. and min. transmission	0.9802 and 0.9763
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1842 / 0 / 130
Goodness-of-fit on F^2	1.031
Final R indices [I>2sigma (I)]R1	0.0391
wR2	0.1129

II. Results and Discussion

In the title compound, the C-C bond lengths of phenyl ring is in the range of 1.372(1)-1.393(1) Å and are in good agreement with similar reported structure (Vasanthi et al., (2014). The C-O bond lengths 1.361(2), 1.428(2) Å and 1.217(2) Å, respectively, indicates the single and double bond characters and are comparable with the literature values (Allen et al., (1987). The C9-C10 bond distance is 1.332(1) Å, it indicates the double bond character and is comparable with the value reported previously (Sathya et al., (2014). The torsion angle of C7-C6-C9-C10=-179.84(1)˚ indicates prop-2-en-1-one group is in -anti-periplanar(-ap) orientation with the phenyl ring. The torsion angle of C7-C8-C3-O1=-178.41(1)˚ indicates ethoxy group is in -anti-periplanar(-ap) orientation with the phenyl ring. The prop-2-en-one group makes dihedral angle of 5.16(1)˚ with the phenyl ring and the ethoxy group makes dihedral angle of 4.80(1)˚ with the phenyl ring. The prop-2-en-one group and ethoxy group are in axial orientation with the phenyl group.

In the packing of the title compound, the crystal packing is stabilized by C-H...O type bonds and the atom O acts as a bifurcated being involved in the intermolecular interactions. C-H...O hydrogen bonds forming inversion dimmers with an $R^2_2(8)$ ring motif. In the two dimensional view, the symmetry related molecules forming a small structural unit and running parallel to 'b' axis (101).

Table 2

Geometric Parameters (Å, °)

C1-C2	1.503(2)	C1-H1A	0.96
C1-H1B	0.96	C1-H1C	0.96
C2-O1	1.4277(18)	C2-H2A	0.97
C2-H2B	0.97	C3-O1	1.3612(17)
C3-C8	1.383(2)	C3-C4	1.387(2)
C4-C5	1.386(2)	C4-H4	0.93
C5-C6	1.3921(19)	C6-C9	1.4655(19)
C7-C8	1.372(2)	C7-H7	0.93
C8-H8	0.93	C9-C10 1	.3215(19)
C9-H9	0.93	C10-C11	1.4649(19)
C10-H10	0.93	C11-O2 1	.2171(17)
C11-C12	1.4965(19)	C12-H12A	0.96
C12-H12B	0.96	C12-H12C	0.96
C2-C1-H1A	109.5	C2-C1-H1B	109.5
H1A-C1-H1B	109.5	C2-C1-H1C	109.5
H1A-C1-H1C	109.5	H1B-C1-H1C	109.5
O1-C2-C1	106.73(13)	O1-C2-H2A	110.4
C1-C2-H2A	110.4	O1-C2-H2B	110.4
C1-C2-H2B	110.4	H2A-C2-H2B	108.6
O1-C3-C8	115.85(13)	O1-C3-C4	124.88(13)
C8-C3-C4	119.27(13)	C5-C4-C3	119.84(13)
C5-C4-H4	120.1	C3-C4-H4	120.1
C4-C5-C6	121.55(13)	C4-C5-H5	117.25(13)
C5-C6-C9	123.61(13)	C7-C6-C9	119.14(13)
C8-C7-C6	121.84(14)	C8-C7-H7	119.1
C6-C7-H7	119.1	C7-C8-C3	120.23(13)
C7-C8-H8	119.9	C3-C8-H8	119.9
C10-C9-C6	127.99(13)	C10-C9-H9	116.0
C6-C9-H9	116.0	C9-C10-C11	125.15(13)
C9-C10-H10	117.4	C11-C10-H10	117.4
O2-C11-C10	119.43(13)	O2-C11-C12	119.77(13)
C10-C11-C12	120.80(12)	C11-C12-H12A	109.5
C11-C12-H12B	109.5	H12A-C12-H12B109.5	
C11-C12-H12C	109.5	H12A-C12-H12C109.5	
H12B-C12-H12C	109.5	C3-O1-C2	119.18(12)
O1-C3-C4-C5	178.53(13)	C8-C3-C4-C5	-1.6(2)
C3-C4-C5-C6	0.2(2)	C4-C5-C6-C7	1.1(2)
C4-C5-C6-C9	-178.39(12)	C5-C6-C7-C8	-1.0(2)
C9-C6-C7-C8	178.50(13)	C6-C7-C8-C3	-0.4(2)
O1-C3-C8-C7	-178.42(13)	C4-C3-C8-C7	1.7(2)
C5-C6-C9-C10	-0.4(2)	C7-C6-C9-C10	-179.84(14)
C6-C9-C10-C11	-179.57(13)	C9-C10-C11-O2	174.48(14)
C9-C10-C11-C12	-5.5(2)	C8-C3-O1-C2	175.45(13)
C4-C3-O1-C2	-4.7(2)	C1-C2-O1-C3	-176.23(13)

Table 3

Hydrogen –bond geometry (Å, °)

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(5)-H(5)...O(2)#1	0.93	2.50	3.4298(17)	178.0
C(10)-H(10)...O(2)#1	0.93	2.57	3.5006(17)	179.2

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