

## Synthesis and Crystal Structure of (2E)-1-(Anthracen-9-yl)-3-(3,4-dichlorophenyl)prop-2-en-1-one

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**Abstract:** (2E)-1-(Anthracen-9-yl)-3-(3,4-dichlorophenyl)prop-2-en-1-one.  $C_{23}H_{14}OCl_2$ , crystallizes in the monoclinic space group  $P 2_1/c$  with unit cell parameters,  $a = 13.7669(9)\text{\AA}$ ,  $b = 11.8400(10)\text{\AA}$ ,  $c = 11.3285(8)\text{\AA}$ ,  $\beta = 103.525(6)^\circ$  and number of molecules per unit cell ( $Z$ ) = 4. The crystal structure was solved by direct methods and refined by full matrix least square procedure to a final  $R$ -value of 0.067 for 2233 observed reflections. The dihedral angle between the benzene and anthracene moiety of the molecule is  $89.43(1)^\circ$ . The structure is stabilized by two  $C-H\cdots O$  intermolecular interaction. Aromatic  $\pi$ - $\pi$  stacking interaction has also been observed in the structure.

**Keywords:** X-ray Structure, Intermolecular Interactions, Direct Methods, Graph Set Motif.

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### I. Introduction

Chalcones are an important class of natural compounds and have been widely applied as synthons in synthetic organic chemistry. Naturally occurring chalcones as well as their synthetic analogues have demonstrated interesting biological activities such as, anticancer[1], anti-invasive[2], anti-tuberculosis[3], antimicrobial[4], anti-malarial[5], antitumor[6], antiproliferative[7] and antioxidant activity[8]. The nonlinear optical [NLO] properties of the different chalcone derivatives have also been reported[9-12]. These  $\alpha$ ,  $\beta$ -unsaturated ketones are biosynthetic precursors of various functionalized derivatives[13], pyrazolines[14] and triaryl pyridines[15]. In view of the importance of chalcones derivatives and our past published work[16-18]. The synthesis and crystallographic analysis of (2E)-1-(Anthracen-9-yl)-3-(3,4-dichlorophenyl)prop-2-en-1-one is reported in this paper.

### II. Experimental

#### 2.1 Synthesis of (2E)-1-(Anthracen-9-yl)-3-(3,4-dichlorophenyl)prop-2-en-1-one.

In 9-acetylanthracene (0.01 mol) and 3,4-dichlorobenzaldehyde (0.01 mol) in ethanol (50 mL), 15 mL of 10 % sodium hydroxide solution was added and stirred at 0–5 °C for 3 h. The precipitate formed was collected by filtration and purified by recrystallization from ethanol. Single crystal was grown from DMF by slow evaporation method (M.P.: 449-451 K). The synthetic route of the compound is shown in Fig. 1.

#### 2.2 X-ray data collection and structure refinement.

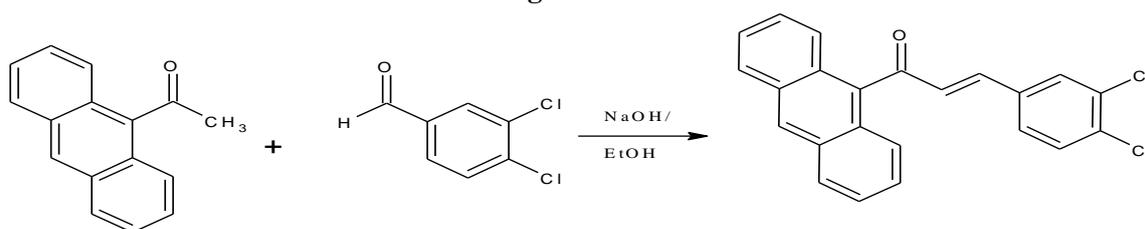
A well defined crystal of dimensions  $0.30 \times 0.20 \times 0.10\text{ mm}^3$  was used for data collection on X'calibur CCD area-detector diffractometer equipped with graphite monochromated  $MoK\alpha$  radiation ( $\lambda = 0.71073\text{\AA}$ ). X-ray intensity data of 7121 reflections were collected at 293(2) K and out of these reflections 3509 were found unique. The intensities were measured by  $\omega$  scan mode for  $\theta$  ranges  $3.61^\circ$  to  $26.00^\circ$ . 2233 reflections were treated as observed using ( $I > 2\sigma(I)$ ) as criterion. Data were corrected for Lorentz-polarization and absorption factors. The structure was solved by direct methods using SHELXS97[19] software. All non-hydrogen atoms of the molecule were located from the best E-map and the hydrogen atoms were geometrically fixed and allowed to ride on the corresponding non-H atoms with  $C-H = 0.93\text{\AA}$  and  $U_{iso} = 1.2 U_{eq}(C)$ . The final refinement cycles converged to an  $R$ -index of 0.0674 ( $wR(F2) = 0.1831$ ) for the 2233 observed reflections. Residual electron densities ranges from  $-0.446$  to  $0.875\text{e}\text{\AA}^{-3}$ . Atomic scattering factors were taken from International Tables for X-ray Crystallography. Allied structural calculations of the molecule were done using the WinGX [20], PARST [21] and PLATON [22] softwares.

Crystallographic information has been deposited to Cambridge Crystallographic Data Centre with CCDC number 1508193. This data can be obtained free of charge at Cambridge Crystallographic Data Centre via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).

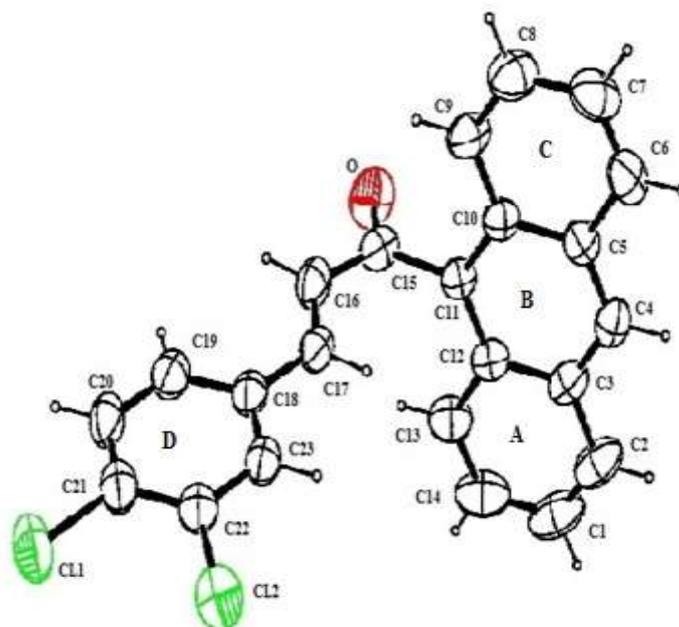
### III. Results And Discussion

The molecular structure containing atomic labeling is shown in Fig. 2 (ORTEP)[23] and the packing diagram is shown in Fig. 3 (PLATON)[22]. The molecule consists of a benzene and anthracene moiety. The structural parameters, including bond distances and angles of the molecule lie within the normal range [24] and are in good agreement with some related structures [25]. The anthracene and benzene rings are essentially planar with maximum deviation (0.0576) Å observed for C8 [anthracene ring] atom and (-0.0049) Å corresponding to C18 [benzene ring] atom. The double bonds C16=C17 and C15=O are confirmed by their respective distances of 1.321(5) Å and 1.218(4) Å, respectively. These values are also consistent with corresponding ones observed in some related structures[25-27]. The anthracene ring system (C1–C14) is twisted at the C11–C15 bond from the (2E)-1-(Anthracen-9-yl)-3-(3,4-dichlorophenyl)acrylaldehyde moiety with the torsion angle [C12—C11—C15—C16] of 93.4(4)°. The benzene and anthracene moiety (C18-C23 and C1-C14 respectively) forms a dihedral angle of 89.43(1)° which makes them inclined at right angles to each other. The oxygen (O) atom attached to carbon atom (C15) adopts the + *anti-clinal* and – *syn-clinal* conformation with torsion angles (O-C15-C11-C10) 91.0(4)° and (O-C15-C11-C12) -86.4(4)° respectively. Molecular packing in the unit cell viewed down the *b*-axis is shown in Fig. 3. The structure is stabilized by the two C–H...O intermolecular interactions, where ‘O’ act as a bifurcated acceptor to C17(via H17) and C23(via H23) atoms (Fig 4.). In the molecular packing, the adjacent molecules are interconnected through C17–H17...O and C23–H23...O hydrogen bond and  $\pi$ - $\pi$  interactions. Details of intermolecular hydrogen bonding and  $\pi$ - $\pi$  interactions are given in Table 3 and 4, respectively.

### IV. Figures And Tables



**Figure 1.** Synthesis of (2E)-1-(Anthracen-9-yl)-3-(3,4-dichlorophenyl)prop-2-en-1-one.



**Figure 2.** ORTEP view of molecules with displacement ellipsoids drawn at 40% probability level. H atoms are shown as small spheres of arbitrary radii.

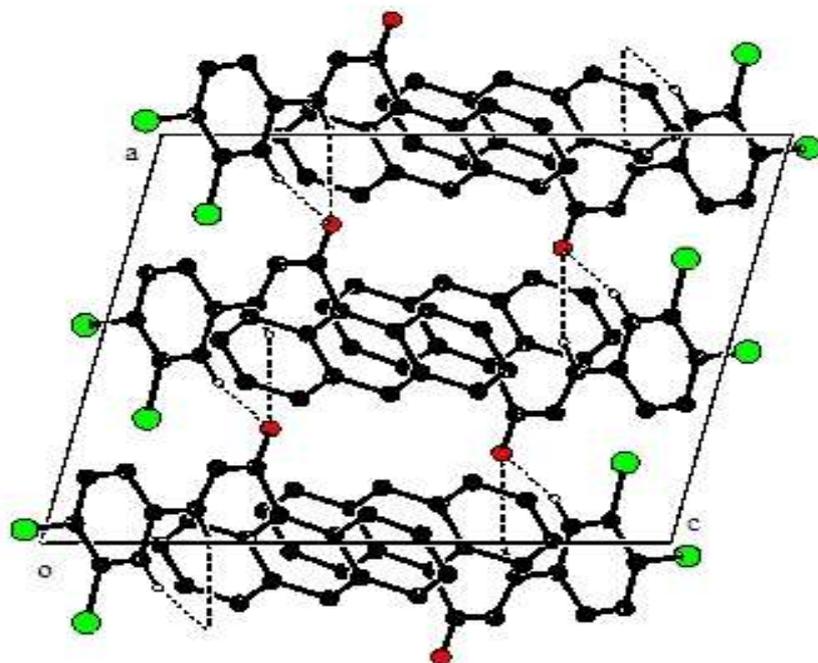


Figure 3. Packing viewed down the *b*-axis.

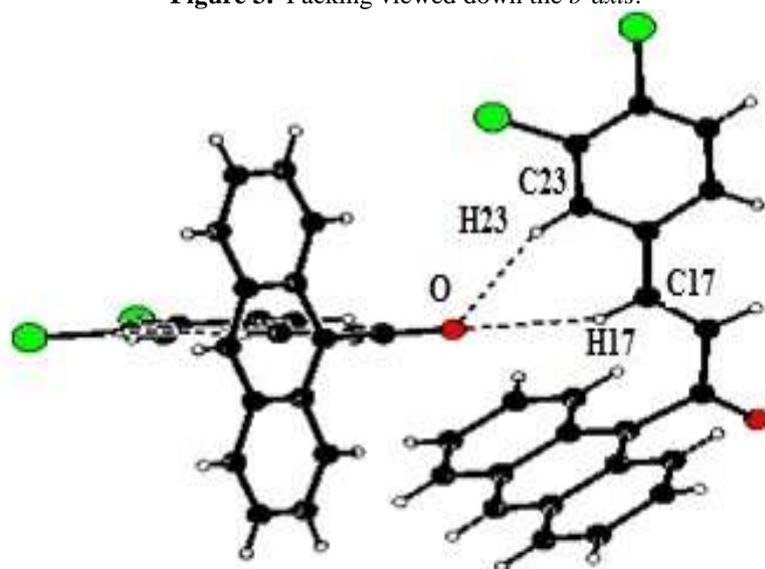


Figure 4. View of bifurcated (acceptor) hydrogen bond and  $R_2^1(6)$  ring motif.

Table 1. Crystal data and other experimental details.

CCDC Number	1508193
Crystal size	0.30x0.20x0.10mm
Empirical formula	$C_{23}H_{14}Cl_2O$
Formula weight	377.24
Radiation, wavelength	MoK $\alpha$ , 0.71073
Unit cell dimensions	a= 13.7669(9) $\text{\AA}$ , b= 11.8400(10) $\text{\AA}$ , c= 11.3285(8) $\text{\AA}$ .
Crystal system	Monoclinic
Space group	P 2 <sub>1</sub> /c
Unit cell volume	1795.3(2) $\text{\AA}^3$
No. of molecule per unit cell, Z	4
Absorption coefficient	0.370mm <sup>-1</sup>
F(000)	776

θ range for entire data collection	3.61 < θ < 26.00
Range of indices	-12 ≤ h ≤ 16 -12 ≤ k ≤ 14 -13 ≤ l ≤ 12
Reflection collected/ unique	7121/3509
Reflection observed (I > 2σ(I))	2233
R int	0.0313
R sigma	0.0567
No. of parameter refined	235
Final R	0.0674
wR(F2)	0.1831
Goodness-of-fit	1.042
Final residual electron density	-0.446 to 0.875 eÅ <sup>-3</sup>

**Table 2.** Selected bond lengths (Å), bond angles (°) and torsion angles (°) for non hydrogen atoms (e.s.d.'s are given in parentheses)

Bond lengths		Bond angles		Torsion angles	
O-C15	1.218(4)	C12-C11-C15	119.1(3)	O-C15-C11-C12	-86.4(4)
C11-C15	1.514(5)	C15-C10-C11	119.4(3)	C11-C15-C16-C17	-6.1(6)
C17-C18	1.460(5)	C11-C12-C13	123.4(4)	C16-C17-C18-C19	6.2(6)
C15-C16	1.447(6)	O-C15-C16	121.7(4)	C16-C17-C18-C23	-174.6(3)
C16-C17	1.321(5)	O-C15-C11	118.3(4)	C18-C23-C22-CL2	179.9(3)
C11-C10	1.402(5)	C22-C21-CL1	120.6(4)	C23-C22-C21-CL1	178.7(3)
C21-CL1	1.726(4)	C23-C22-CL2	118.8(3)	O-C15-C11-C10	91.0(4)
C22-CL2	1.700(4)	C20-C21-CL1	119.8(3)	C16-C15-C11-C10	-89.2(4)
C18-C23	1.394(5)	C21-C22-CL2	121.4(3)	CL1-C21-C20-C19	-179.1(3)

**Table 3.** Hydrogen bonding geometry (e.s.d.'s in parentheses).

D-H...A	D-H(Å)	H...A(Å)	D...A(Å)	D-H...A(°)
C17-H17...O <sup>i</sup>	0.93	2.55	3.410(4)	155
C23-H23...O <sup>i</sup>	0.93	2.54	3.397(5)	153

Symmetry code: (i) x, 1/2-y, 1/2+z.

**Table 4.** Geometry of π-π interactions\*

CgI...CgJ	CgI...CgJ (Å)	CgI...P(Å)	α(°)	β(°)	Δ(Å)
Cg1...Cg3 <sup>i</sup>	3.8620(27)	3.498	2.73	23.89	1.636
Cg2...Cg2 <sup>i</sup>	3.8108(22)	3.460	0.03	24.77	1.597
Cg3...Cg1 <sup>i</sup>	3.8620(27)	3.531	2.73	25.09	1.564
Cg4...Cg4 <sup>ii</sup>	3.7446(23)	3.487	0.02	21.39	1.364

Symmetry code: (i) 1-x, -y, 1-z (ii) -x, 1-y, 1-z

\* Cg1, Cg2, Cg3 and Cg4 represents the centre of gravity of the ring A, B, C and D.

## V. Conclusions

Synthesis of (2E)-1-(Anthracen-9-yl)-3-(3,4-dichlorophenyl)prop-2-en-1-one led to the single crystal grown from DMF by slow evaporation technique (M.P.: 449-451 K) and the molecular and crystal structure was determined using single crystal X-ray diffraction techniques. The structure was refined to final R-factor of 0.067. The dihedral angle [89.43(1)°] between the benzene and anthracene moiety makes them held at right angles to each other. The structure is stabilized by few C-H...O and π-π intermolecular interactions.

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