



STRUCTURE OF (2*E*,2'*E*)-3,3'-(1,3-PHENYLENE)BIS(1-(ANTHRACENE-9-YL)PROP-2-EN-1-ONE): A CHALCONE DERIVATIVE

Rajni Kant^{[a]*}, Ratika Sharma^[a], Vinutha V. Salian^[b], B. K. Sarojini^[c] and B. Narayana^[b]

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The title compound C₄₀H₂₆O₂ [(2*E*, 2'*E*)-3, 3'-(1,3-Phenylene)-bis(1-(anthracene-9-yl)prop-2-en-1-one)] was prepared from the reaction of isophthalaldehyde and 9-acetylanthracene treated with solution of ethanol and sodium hydroxide. It has been crystallized from DMF using slow evaporation method in the triclinic space group P-1 with Z=2 and unit cell parameters, $a = 9.8276(8)$ $b = 10.7470(8)$, $c = 14.5606(10)$ Å, $\alpha = 99.833(6)$, $\beta = 104.495(6)$, $\gamma = 97.371(6)^\circ$. The architecture of the crystal structure is determined mainly by three intermolecular interactions [C23-H10...O1, C25-H15...O1, C16-H17...O1], here O1 acts as an acceptor to three different carbon atoms, resulting into the formation of a trifurcated hydrogen bond, forming a graph set motif $R^2_2(8)$ through a pair of C25-H15...O1 interactions. These motifs are further extended through another intermolecular hydrogen bond [C3-H6...O2] forming a ladder like network, extending along the c-axis.

* Corresponding Authors

Fax: +91 191 243 2051

E-Mail: rkant.ju@gmail.com

[a] X-ray Crystallography Laboratory, Department of Physics and Electronics, University of Jammu, Jammu Tawi-180 006, India.

[b] Department of Studies in Chemistry, Mangalore University, Mangalagangothri-574 199, India.

[c] Department of Industrial Chemistry, Mangalore University, Mangalagangothri-574 199, India

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.: 523-525 K).

Introduction

Chalcones are an interesting class of compounds which have been reported to possess various useful properties. They have been studied for non-linear optical properties¹, biological activities including anti-inflammatory, antileishmanial, antimicrobial, antioxidant²⁻⁴ and HIV-1 protease inhibitor⁵ as well as fluorescence properties^{6,7}. Chalcones are important intermediates in organic synthesis of variety of pharmacologically important heterocyclic compounds. Due to the presence of enone functionality, chalcones are an interesting target class of compounds which are extensively studied for their broad spectrum of biological activities such as, antileishmanial⁸, anti-invasive^{9,10}, antitubercular¹¹, antifungal¹², antimalarial¹³, anticancer^{14,15} and anti-inflammatory properties¹⁶.

In this paper we report precise synthesis and a detailed X-ray structure of a chalcone derivative containing anthracene moiety and possessing interesting biological properties.

Experimental

Synthesis

The reaction scheme for the synthesis of the title compound is presented in Figure 1. To a mixture of isophthalaldehyde (0.67 g, 0.005 mol) and 9-acetylanthracene (2.20 g, 0.01 mol) in ethanol (50 mL), 15 mL of

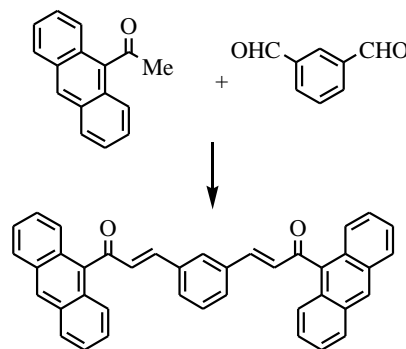


Figure 1. Synthesis of the chalcone derivative

X-Ray Intensity Data Collection

Structure Solution

X-ray intensity data of a crystal having well-defined crystal morphology (0.30 x 0.20 x 0.10 mm) were collected at 293(2)K on X'calibur CCD area-detector diffractometer equipped with graphite monochromated MoK α radiation ($\lambda = 0.71073$ Å). The intensities were measured by employing ω scan mode for the diffraction angle ranging from 3.58 to 26.00°. A total number of 10030 reflections were measured of which 5641 were found to be unique. The criterion ($I > 2\sigma(I)$) was employed to the unique data set and hence 2418 reflections were treated as observed. Data were corrected for Lorentz and Polarization factors. The structure was solved by direct methods using SHELXS97.¹⁷

Table 1. Crystal data and other experimental details.

CCDC Number	1407267
Crystal description	Block
Crystal size	0.30 x 0.20 x 0.10 mm
Empirical formula	C ₄₀ H ₂₆ O ₂
Formula weight	538.61
Radiation, Wavelength	Mo K α , 0.71073 Å
Unit cell dimensions	$a = 9.8276(8)$ Å, $b = 10.7470(8)$ Å $c = 14.5606(10)$ Å $\alpha = 99.833(6)^\circ$ $\beta = 104.495(6)^\circ$ $\gamma = 97.371(6)^\circ$
Crystal system, Space group	Triclinic, <i>P</i> -1
Unit cell volume	14443.25(19) Å ³
No. of molecules per unit cell, Z	2
Absorption coefficient	0.075 mm ⁻¹
F(000)	564
θ range for entire data collection	3.58 < θ < 29.12°
Reflections collected / unique	10030 / 5641
Reflections observed ($I > 2\sigma(I)$)	2418
Range of indices	$h = -12$ to 12 , $k = -13$ to 12 , $l = -17$ to 17
No. of parameters refined	467
Final <i>R</i> -factor	0.0643
w <i>R</i> (<i>F</i> ²)	0.1989
<i>R</i> _{int}	0.0298
<i>R</i> σ	0.0839
Goodness-of-fit	0.989
(Δ/σ) _{max}	0.001
Final residual electron density	-0.199 < $\Delta\rho$ > 0.036 eÅ ⁻³

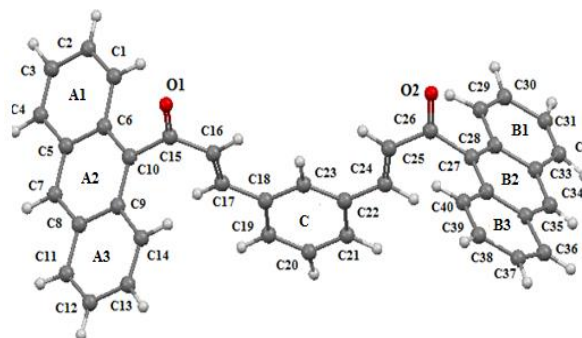
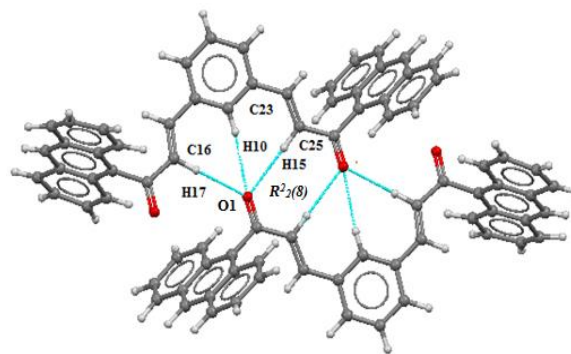
Table 2. Some important bond lengths (Å) and bond angles (°)

S.	Bond lengths(Å)		Bond angles(°)	
1.	O1-C15	1.220	O1-C15-C10	119.70
2.	O2-C15	1.225	O2-C15-C16	119.86
3.	C5-C10	1.413	C16-C15-C10	120.42
4.	C9-C10	1.396	C16-C17-C18	126.46
5.	C10-C15	1.496	C15-C16-C17	125.46
6.	C15-C16	1.462	C26-C25-C24	124.78
7.	C17-C18	1.467	C22-C24-C25	127.00
8.	C27-C28	1.398	C25-C26-O2	120.03
9.	C27-C40	1.404	C27-C26-C27	120.30
10.	C32-C24	1.454	C25-C26-C27	119.66

Structure Refinement

Full-matrix least-squares refinement was carried out using SHELXL97¹⁷. The final refinement cycles converged to $R = 0.0643$ and $wR(F^2) = 0.1989$ for the 2418 observed reflections. Residual electron density ranges from -0.199 to 0.036 eÅ⁻³. Atomic scattering factors were taken from International Tables for X-ray Crystallography (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4). The crystallographic data are summarized in Table 1. Some selected bond angles which plays an important role in collating the structural properties of this molecule with the related structures are presented in Table 2. An ORTEP¹⁸ view of the molecule with atomic

labelling is shown in Figure 2. The geometry of the molecule was calculated using the PLATON¹⁹ and PARST²⁰ software. The Cambridge Crystal Data Centre Number assigned to this structure is CCDC-1407267.

**Figure 2.** ORTEP view of the molecule showing the atom-labelling scheme**Figure 3.** A plot of two molecules of the title compound showing trifurcated intermolecular C-H...O hydrogen bonds (dashed lines) forming $R^2_2(8)$ motif

Result and Discussion

The molecule comprises of two anthracene moieties separated by the chalcone chain system including a phenyl ring labelled as C. The two anthracene moieties are labelled as A (consisting of ring A1, A2 and A3) and B (consisting of ring B1, B2 and B3), respectively (Figure 2). All bond lengths and angles are normal and correspond to those observed in related structures^{21, 22}. The endocyclic bond angles in moiety A and B are very close to 120°, indicating perfect aromatic character in them.²³ C15=O1 and C26=O2 keto distances are confirmed by respective distances of [1.220(3) and 1.220(4) Å] and are slightly longer than the standard value for carbonyl bonds²³ (1.210 Å) probably because atoms O1 and O2 both are involved in C-H...O intermolecular hydrogen bonds. These keto distances are in well agreement with the values observed for related structures.^{24,25} The plane of moiety A forms a dihedral angle of 70.95(6)° with the plane of the moiety B. The ring C is twisted from the plane formed by the ring atoms C1/C6/C10/C9/C14 (in moiety A) and C29/C28/C27/C40/C39 (in moiety B), through the dihedral angles of 69.19(1)° and 76.82(2)°, respectively. The coplanar geometry of ring C with both the carbonyl groups [C15=O1 and C26=O2], confirmed the torsion angles values being close to zero.

Table 3. Geometry of intra and inter molecular hydrogen bonds

D-H...A	D-H (Å)	H...A(Å)	D...A(Å)	∠[DH...A(°)]
C14-H1...O1	0.98(2)	2.53(3)	3.110(4)	118
C3-H6...O2 ⁱ	0.98(3)	2.58(3)	3.773(5)	139
C23-H10...O1 ⁱⁱ	0.94(3)	2.53(2)	3.455(4)	169
C25-H15...O1 ⁱⁱ	1.00(3)	2.51(3)	3.473(4)	161
C16-H17...O1 ⁱⁱ	1.02(3)	2.37(3)	3.392(4)	174

Symmetry codes: (i) $x-1, y, z-1$ (ii) $-x+1, -y, -z+1$

The atom O1 is playing a key role in the stabilization of crystal structure as it participates in three inter and one intra molecular hydrogen bonds out of total five hydrogen bond interactions present in the molecule. The architecture of the crystal structure is determined mainly by three intermolecular interactions in which O1 acts as a hydrogen bond acceptor via hydrogen atoms H6, H15 and H17 to the carbon atoms C23, C25 and C16 respectively, resulting into the formation of a trifurcated hydrogen bonded structure. This generates a graph set motif R22(8)26 through the pair of C25-H15...O1 interactions (Figure 3). The geometry of hydrogen bonding is presented in Table 3.

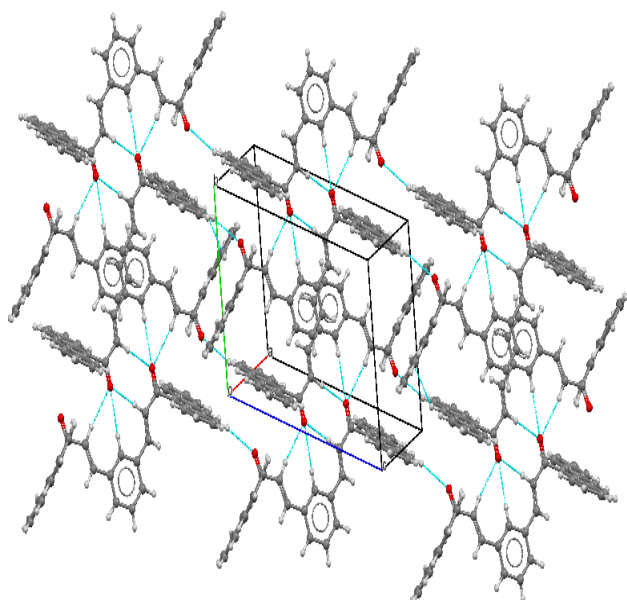


Figure 4. Packing view of the molecule projected along the bc plane

The supramolecular structure formed by the trifurcated hydrogen bonding is further connected by the C3-H6...O2 hydrogen bonds, extending along the c-axis forming a ladder like network as shown in Figure 4.

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