SYNTHESIS AND CRYSTAL STRUCTURE OF (*E*)-3-(4-BUTOXYPHENYL)-1-(NAPHTHALEN-1-YL)-PROP-2-EN-1-ONE

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Chalcone derivative (*E*)-3-(4-butoxyphenyl)-1-(naphthalen-1-yl)prop-2-en-1-one (C23H22O2) crystallizes in monoclinic system with space group 'P2$_1$/c ' and unit cell parameters: a = 15.1595(14) Å, b = 7.6644(7) Å, c = 15.8634(15) Å, β = 96.942(7)°. The crystal structure was solved using direct methods and refined by full matrix least squares procédures to a final R-factor of 0.0911 for 1591 observed reflections. The enone moiety adopts *E* conformation with respect to C12=C13 bond.The molecules in the unit cell are linked by weak C–H···O and π-π interactions. The molecule contains two C–H···O intramolecular interactions which stabilizes the crystal structure

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Introduction

Chalcones are main precursors in the biosynthesis of flavonoids and isoflavanoids that are abundant in edible plants.[1] These are valuable intermediates in the synthesis of pyridines,[2] benzodiazepines,[3] pyrazolines,[4] cyclohexene derivatives[5] etc. Chalcones display tremendous pharmacological activities like anticancer,[6] antimalarial,[7] anti-inflammatory,[8] antitubercular,[9] larvicidal,[10] anticonvulsant,[11] cytotoxic,[12] antidepresent,[13] antimicrobial,[14] antiHIV.[15] Chalcone and their derivatives demonstrate wide range of biological activities such as anti-diabetic, anti-neoplastic, anti-hypertensive, anti-retroviral, anti-inflammatory, anti-parasitic, anti-histaminic, anti-malarial, anti-oxidant, anti-fungal, anti-obesity, anti-platelet, anti-tubercular, immunosuppressant, anti-arrhythmic, hypnotic, anti-gout, anxiolytic, anti-spasmodic, anti-nociceptive, hypolipidemic, anti-filarial, anti-angiogenic, anti-protozoal, anti-bacterial, anti-steroidal, cardioprotective, etc.[16-19] Besides this, the chalcone derivative shows non-linear optical (NLO) properties with excellent blue light transmittance and good crystallizability.[20-24]

In view of the extensive biological activities as exhibited by a large variety of chalcone derivatives, we report synthesis and crystal structure of (*E*)-3-(4-butoxyphenyl)-1-(naphthalen-1-yl)prop-2-en-1-one.

Experimental

Synthesis

Sodium hydroxide (0.4 g, 0.01mol) was dissolved in 20 ml of methanol and stirred at room temperature. 1-Acetylnaphthalene (1.7 g, 0.01mol) was dissolved in 20 ml of methanol and added drop wise to the sodium hydroxide solution with constant stirring. 4-Butoxybenzaldehyde (1.78 g, 0.01mol) dissolved in 20 ml methanol and added drop wise to the previous solution and continued stirring for 24 hours. The precipitated product was diluted with cold water and kept aside for 15 minutes. The product was then filtered, washed with distilled water and dried. The pure product was obtained by recrystallization from ethanol. Single crystal of the purified product developed from DMF by slow evaporation method (M.P.: 333-335 K). The complete reaction procedure is given in Figure 1.

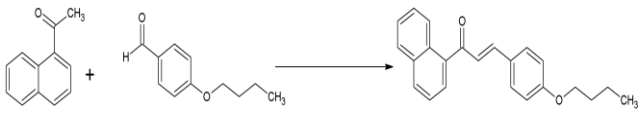

Figure 1. Reaction for the preparation of (*E*)-3-(4-Butoxyphenyl)-1-(naphthalen-1-yl)prop-2-en-1-one

Crystal structure determination

X-ray intensity data of the crystal of dimensions 0.30 x 0.20 x 0.20 mm^3 were collected on *X'calibur* CCD area-detector diffractometer equipped with graphite monochromated MoK$_\alpha$ radiation (λ = 0.71073 Å). X-ray intensity data of 6252 reflections were collected at 293(2) K and out of these reflections 3194 were found unique. The intensities were measured by ω scan mode for θ ranges 3.71° to 24.99°. 1591 reflections with I>2σ(I) were treated as observed.

Data were corrected for Lorentz-polarization and absorption factors. The structure was solved by direct methods using SHELXS97[25] and was refined using SHELXL97.[25] All non-hydrogen atoms of the molecule were located from the map. All the hydrogen atoms were geometrically fixed and allowed to ride on the corresponding carbon with C-H= 0.93-0.97Å and best U_{iso}= 1.2 $U_{eq}(C)$, except for the methyl groups where $U_{iso}(H)$ = 1.5 U_{eq}(C). The final refinement cycles converged to an R-factor of 0.0911 [$wR(F^2)$ = 0.2477] for 1591 observed reflections. Residual electron densities ranges from -0.289 to 0.562 eÅ^{-3}. Geometrical calculations of the molecule was done using the WinGX,[26] PARST[27] and PLATON[28] softwares.

Crystallographic information has been deposited to Cambridge Crystallographic Data Centre with CCDC number 1507484. This data can be accessed free of charge at Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif . Details of crystallographic and X-ray diffraction data are given in Table 1, bond distances and bond angles in Table 2 and torsion angles in Table 3.

Table 1. Crystallographic characteristics, X-ray data collection and structure refinement parameters of $C_{23}H_{22}O_2$.

CCDC No.	1507484
System,	Monoclinic,
Space group,	P 2_1/c
Z	4
a, Å	15.1595(14)
b, Å	7.6644(7)
c, Å	15.8634(15)
β, °	96.942(7)
V, Å^3	1829.63(16)
D_x g.cm^{-3}	1.20
Radiation, λ, Å	0.71073
μ, mm^{-1}	0.075
T, K	293(2)
Sample size, mm^3	0.30*0.20*0.20
Diffractometer	X' calibur Sapphire 3 CCD area-detector
Scan mode	ω scan
Absorption correction,	multi-scan
T_{min}	0.76841,
T_{max}	1.00000
θ range	3.71→24.99
h, *k*, *l* ranges	*h*=-11→18
	k= -8 →9
	l=-17→18
Number of reflections:	
measured/unique (N_1),	1591/3194
R_{int}/with $I > 2\sigma(I)$ (N_2)	0.0430/6252
R_{int}	0.0430
R_{sigma}	0.0790
F(000)	704
R	0.0911
*wR*2	0.2477
$(\Delta/\sigma)_{max}$	0.00
Number of refined parameters	228
S	1.094
$\Delta\rho_{max}/\Delta\rho_{min}$, e/Å^3	0.562/-0.289
Programs	SHELXS97,[25] SHELXL97,[25] PARST,[27] PLATON,[28] ORTEP[29]

Results and discussion

The molecular structure with atomic labeling is shown in Figure 2 (ORTEP).[29] The molecule consists of two aromatic rings (naphthalene and phenyl) bridged by prop-2-en-1-one group. The structural parameters, including bond distances and bond angles show a normal geometry.[30]

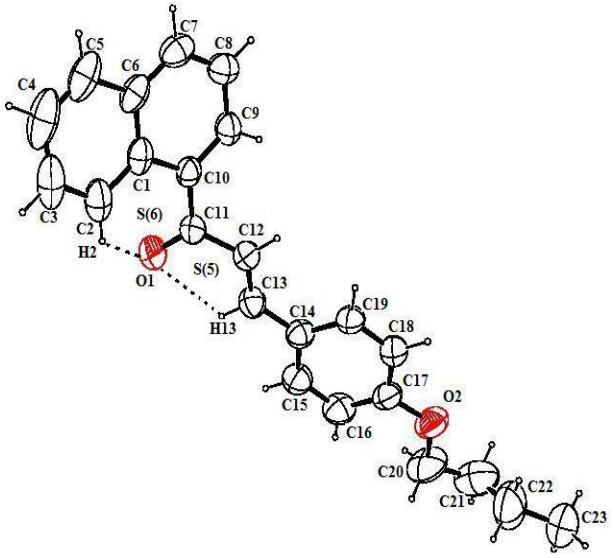

Figure 2. ORTEP view of the molecule at 40% displacement ellipsoid probability level along with atomic labeling scheme. Hydrogen atoms are drawn at arbitrary radii and are not labeled for clarity. The dashed line shows intra-molecular hydrogen bonds.

Table 2. Selected bond distances and bond angles (Å, °)

Bond Distances		Bond Angles	
C1–C2	1.413(5)	C2–C1–C10	122.9(4)
C10–C11	1.482(5)	C13–C14–C15	119.8(3)
C11– C12	1.475(5)	C9–C10–C11	119.3(3)
C20–O2	1.447(6)	C13–C14–C19	122.9(4)
C12–C13	1.317(5)	C1–C10–C11	121.9(3)
C15–C16	1.362(6)	C16–C17–O2	124.7(4)
C11–O1	1.223(4)	O1–C11–C12	121.0(4)
C17–O2	1.364(5)	O2–C17–C18	115.3(4)
C21– H21A	0.9700	O1–C11–C10	120.6(3)
C20– C21	1.484(7)	O2–C20–O21	111.3(5)
C22–H22A	0.9700	C17–O2–C20	117.7(4)
C23– H23A	0.9600	C21–C22–C23	114.0(7)

Table 3. Selected torsion angles (°)

Torsion angles	
C2–C1–C10–C11	-13.6(5)
C8–C9–C10–C11	-169.6(4)
C9–C10–C11–O1	140.8(4)
C1–C10–C11–O1	-32.4(6)
C1–C10–C11–C12	148.6(4)
C12–C13–C14–C19	-1.2(7)
C12–C13–C14–C15	178.1(4)
C20–O2–C17–C16	-3.1(7)
O2–C20–C21–C22	-66.4(8)
C20–O2–C17–C18	175.8(4)

Table 4. Hydrogen bonding geometry (e.s.d.'s in parentheses)

D–H...A	D–H(Å)	H...A(Å)	D...A(Å)	D-H...A(o)
C2–H2...O1	0.93	2.40	2.921(5)	116
C13–H13...O1	0.93	2.44	2.790(4)	102
C18–H18...O1i	0.93	2.56	3.291(5)	136

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

Table 5. Geometry of π-π interactions*

CgI…CgJ	CgI…CgJ(Å)	CgI…P(Å)	α(º)	β(º)	Δ(Å)
Cg2…Cg2^i	3.7179(2)	3.629	0.03	12.56	0.81

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

The bond lengths within the conjugated linear fragment suggests the large degree of localization: the bond length C11–C12 =1.475(5)Å, C12=C13 =1.317(5)Å and C11=O1 =1.223(4)Å, and it agrees well with the corresponding distances in some analogous structures.[31] The naphthalene and phenyl rings are approximately planar and are inclined at a dihedral angle 44.54(1)° with respect to each other. The prop-2-en-1-one group is inclined with the naphthalene ring with a torsion angle (C1-C10-C11-C12) of 148.6(4)°. The prop-2-en-1-one group is approximately linear with phenyl ring having torsion angle (C12-C13-C14-C15) of 178.1(4)°.

The atoms of naphthalene and phenyl rings are almost planar with maximum deviation of 0.0629(5)Å observed for C3 atom and 0.0072(4)Å corresponding to C19 atom for respective rings. The naphthalene ring is significantly folded, even though both the individual rings are almost planar, the dihedral angles between their planes is as high as 3.68(1)°. The butoxy part is tilted with phenyl ring at a dihedral angle of 17.58(2)°. The atoms C21 and C22 are significantly deviated by 0.3024(7)Å and -0.3779(8)Å, respectively, from the mean molecular axis.

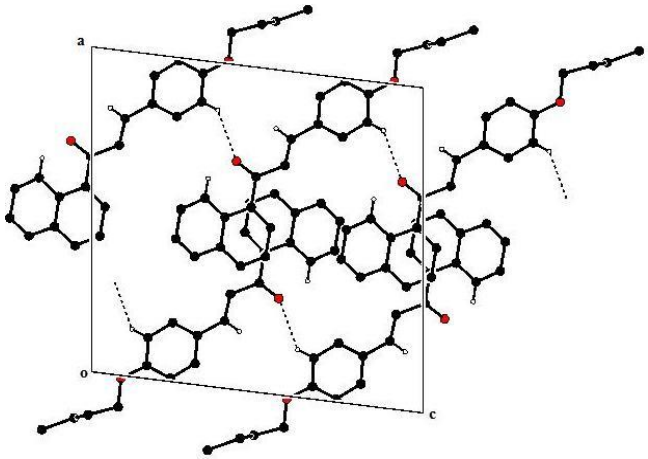

Figure 3. Packing of the molecule along *b-axis*. Intermolecular C–H···O contacts are also shown.

In the crystal structure, presence of C2-H2…O1 and C13-H13…O1 intra-molecular hydrogen bonds respectively results in the formation of S(6) and S(5) graph-set motifs (Figure 2). In the crystal structure, there is only one intermolecular hydrogen bond C18-H18…O1 that links molecule into infinite chains along *y*-direction, and a weak π-π interaction, which stabilize the packing, otherwise determined by the van der Waals interactions and close packing requirements (Figure 3).

Details of intra/inter-molecular hydrogen bonds are given in Table 4 and that of π-π bonding are given in Table 5.

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