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SYNTHESIS, CHARACTERIZATION, CRYSTAL AND MOLECULAR STRUCTURE STUDIES OF A NOVEL (*E*)-3-(2, 3-Dichloropheny)-1-(p-tolyl)prop-2-en-1-one

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ABSTRACT

The title molecule $C_{16}H_{12}Cl_2O$ is a new chalcone derivative synthesized and grown by slow evaporation technique. X-ray diffraction studies endorsed the crystallization of the compound in primitive monoclinic system in $P2_1/c$ space group with one molecule in the asymmetric unit . The unit cell parameters are a = 16.1126 (8) Å, b = 8.0557 (4) Å, c = 10.7586 (5) Å, and β= 102.963 (2)° with Z=8. The R-factor converged to 0.0447 for 2222 reflections. The molecule exists in trans conformation about the double bond in the central enone group. The crystal structure is stabilized by the presence C–H···O interactions and vanderwaals forces. The bond length, bond angles, torsion angles and plane orientations of the moieties in the molecule are discussed.

KEYWORDS: Crystal structure, Chalcone, Trans conformation, X-ray diffraction,Trigonalplane, Hydrogen bonds

Chalcones form the central core for the construction of a variety of bioactive compounds. The usual method for the synthesis of chalcones involves the condensation of aromatic aldehyde and aromatic ketone in the presence of aqueous alkaline bases (Jadav et al., 2015).Chalcones and their derivatives demonstrate wide range of biological activities such as anti-diabetic, antineoplastic, antitubercular, antiarrhythmic, hypnotic, antiangiogenic, antiprotozoal, antibacterial, antisteroidal, cardioprotective, etc. (Mahapatraetal., 2015). α,β-unsaturated carbonyl system of chalcones made them as useful building blocks in organic synthesis. The presence of conjugated double bond in addition to a completely delocalized π-electron system on both benzene rings enables it to be an intermediate for the preparations of many therapeutic compounds (Straub, 1995 , Sandler and Karo, 1972, Bergman etal., 1959) Various heterocyclic compounds can be synthesized from this compound (Geiger and Conn, 1945) which exhibit ample biological activities (Dimmock et al., 1999,Opletalova and Sedivy1999).They have been efficiently employed as a precursor in the syntheses of biologically potent isoxazoles (Ajay Kumar etal., 2010). In view of these and in continuation of our work on chalcones, we herein report the synthesis, single crystal X-ray diffraction studies of (*E*)-3-(2,3-dichlorophenyl)-1-(p-tolyl)prop-2-en-1-one.

MATERIALS AND METHODS

Synthesis of (E)-3-(2, 3-Dichlorophenyl)-1-(p-tolyl)prop-2-en-1-one

A mixture of 2,3-dichlorobenzaldehyde (5 mmol), 1-(p-tolyl)ethanone (5 mmol) and sodium hydroxide (5 mmol) in 95% ethyl alcohol (25 mL) was stirred at room temperature for 3 hrs. The progress of the reaction was monitored by TLC. After the completion of the reaction, the mixture was poured in to ice cold water and kept in the refrigerator for overnight. The solid formed was filtered, and washed with cold hydrochloric acid (5%). Yellow rectangular slab like crystal of (*E*)-3-(2,3-dichlorophenyl)-1-(p-tolyl)prop-2-en-1-one were obtained from methyl alcohol in a slow solvent evaporation technique. Yield 91%, M.P. 95-96°C.Scheme 1 shows the reaction scheme of the title compound.

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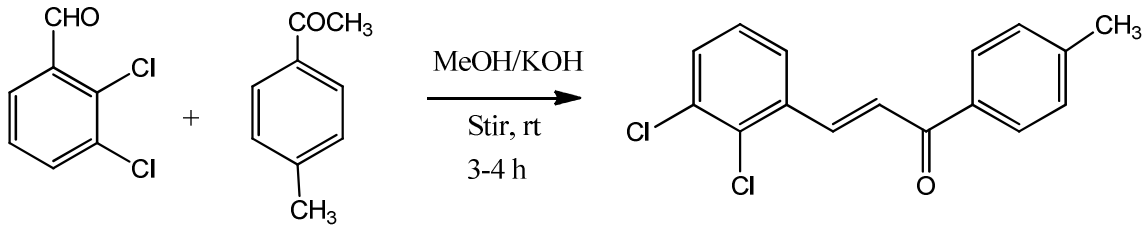

Scheme 1: Reaction Scheme of the title compound

DATA COLLECTION

A colorless block shaped single crystal of dimensions 0.24 x 0.26 x 0.28 mm of the title compound was chosen for an X-ray diffraction study. X-ray intensity data were collected for the title compound at temperature 293 K on a Bruker X8 Proteumdiffractometer usingCuKα radiation of wavelength 1.54178 Å. A complete data set was processed using SAINT(Bruker, 2013).The structure was solved by direct methods and refined by full-matrix least squares method on F^2 using SHELXS -97 and SHELXL-97 (Sheldrick, 2008) programs. All the non-hydrogen atoms were revealed in the first difference Fourier map itself. All the hydrogen atoms were positioned geometrically (C-H = 0.93 A) and refined using a riding model with $U_{iso}(H) = 1.2\ U_{eq}(C)$ abd$U_{iso}(H) = 1.5\ U_{eq}(O)$ for hydroxyl group. After several cycles of refinement, the final difference Fourier map showed peaks of no chemical significance and the residual is saturated. The geometrical calculations were carried out using the program PLATON. The molecular and packing diagrams were generated using the software MERCURY. The details of the crystal structure and data refinement are given in Table 1. The list of bond lengths and bond angles of the non-hydrogen atoms are given in Table 2 and 3. Figure 1 represents the ORTEP diagram of the molecule with thermal ellipsoids drawn at 50% probability.

RESULTS AND DISCUSSION

The mean square plane of the phenyl rings in the molecular structure linked by propenone chain bisects each other as indicated by the dihedral angle of 23.74(10)°.The trans conformation about the C7-C8 double bond in the central enone group is confirmed by the C7=C8-C9–C10 torsion angle of -176.18(18)°. The variation of bond lengths C8-C9 (1.483(3)Å) and C9-C10 (1.496(3)Å) from the standard values can be attributed to the steric hindrance caused by the oxygen atom of the keto group. The distorted trigonal plane about C9 atoms are confirmed by the bond angle values 120.6°, 120.48° and 118.93° which are slightly different from the bond angle values of 122.1°, 120.6° and 117.33° reported for 1-(2'-Thiophen)-3-(2,3,5-trichlorophenyl)-2-propen-1-one (Manjunath etal, 2011). The crystal structure is stabilized by the presence of intermolecular C–H···O interactions. The molecular are linked through $R^2_2(7)$ to form one dimensional chain down the *b* axis as shown in figure 2.

SUMMARY

(*E*)-3-(2,3-Dichlorophenyl)-1-(p-tolyl)prop-2-en-1-onewas synthesized and characterized by single crystal X-ray diffraction studies.The bond lengths around the keto group are distorted due to the steric hindrance caused by the oxygen atoms. The crystal structure is stabilized by the intermolecular hydrogen bonds.

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Table 1: Crystal data and structure refinement table

CCDC	1568342
Empirical Formula	$C_{16}\ H_{12}\ Cl_2O$
Formula Weight	291.16
Temperature	296 K
Wavelength	1.54178 Å
Crystal System	Monoclinic
Space Group	$P2_1/c$
Cell dimension	a = 16.1126(8) Å
	b = 8.0557(4) Å
	c = 10.7586(5) Å
	β = 102.963(2)°
Volume	1360.86(12) Å^3
Z	4
Density	1.421 g/cm^3
F_{000}	600
Crystal size	0.25 x 0.27 x 0.29 mm
Theta range for data collection	6.2° to 64.4°
Index ranges	-18 ≤ h ≤ 18
	-9 ≤ k ≤ 9
	-12 ≤ l ≤ 10
Independent reflections	2222 [R(int) = 0.044]
Absorption method	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2222 / 00 /173
Goodness-of-fit on F^2	1.06
Final R indices [$I > 2\sigma(I)$]	$R1$ = 0.0447, $\omega R2$ = 0.1313
Largest diff. peak and hole	-0.38 and 0.49 e. Å^3

Figure 1: ORTEP diagram of the molecule at 50% probability of displacement ellipsoids for non-hydrogen atoms.

Table 2: Bond Lengths [Å] for Non-Hydrogen atoms

Atoms	Length	Atoms	Length
Cl1-C6	1.739(2)	C7-C8	1.336(3)
Cl2-C1	1.733(2)	C8-C9	1.483(3)
O1-C9	1.228(3)	C9-C10	1.496(3)
C1-C6	1.391(3)	C10-C15	1.395(3)
C1-C2	1.404(3)	C10-C11	1.394(3)
C2-C3	1.406(3)	C11-C12	1.384(3)
C2-C7	1.467(3)	C12-C13	1.392(3)
C3-C4	1.381(3)	C13-C14	1.390(3)
C4-C5	1.389(3)	C13-C16	1.504(3)
C5-C6	1.375(3)	C14-C15	1.384(3)

Table :3: **Bond Angles [°]for Non-Hydrogen atoms**

Atoms	Angle	Atoms	Angle
C6-C1-C2	120.55(1)	O1-C9-C8	120.60(1)
C6-C1-Cl2	118.86(1)	O1-C9-C10	120.47(1)
C2-C1-Cl2	120.60(1)	C8-C9-C10	118.94(1)
C1-C2-C3	117.59(1)	C15-C10-C11	118.16(1)
C1-C2-C7	120.52(1)	C15-C10-C9	123.10(1)
C3-C2-C7	121.89(1)	C11-C10-C9	118.73(1)
C4-C3-C2	121.00(2)	C12-C11-C10	120.77(1)
C3-C4-C5	120.70(2)	C11-C12-C13	121.02(1)
C6-C5-C4	119.12(1)	C14-C13-C12	118.20(1)
C5-C6-C1	121.06(1)	C14-C13-C16	120.10(1)
C5-C6-Cl1	118.54(1)	C12-C13-C16	121.66(1)
C1-C6-Cl1	120.39(1)	C15-C14-C13	121.0(2)
C8-C7-C2	126.75(1)	C14-C15-C10	120.82(1)
C7-C8-C9	120.00(1)		

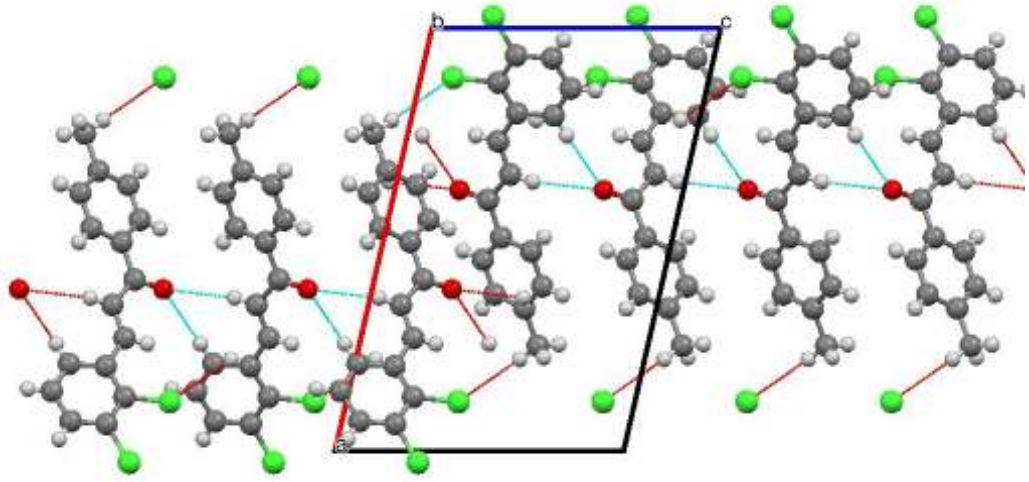

Figure 2: Packing diagram of the molecule down 'b' axis

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