



X-ray and infrared spectroscopic studies of the molecular structure of 3-(2,4-dichlorophenyl)-1-phenylprop-2-en-1-one

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Abstract The title compound $C_{15}H_{10}Cl_2O$ is derived from acetophenone and 2,4-dichlorobenzaldehyde. There are two molecules in the asymmetric unit of the molecular structure of the compound. The two aromatic rings exhibit a non-coplanar geometry, which is due to the presence of two chlorine atoms in one aromatic ring. The compound was further characterized by infrared spectroscopy where a band at 1661 cm^{-1} indicates the presence of the carbonyl group.

Keywords Chalcone, Infrared spectroscopy, X-ray diffraction, single crystal

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1. Introduction

Chalcones are the important group of natural products [1] which have wide pharmacological properties. Some chalcones possess anticancer [2], antimalarial [3], antimicrobial [4], and anti-inflammatory [5] activities. A new chalcone derivative, namely 3-(2,4-dichlorophenyl)-1-phenylprop-2-en-1-one, has been synthesized by condensation of acetophenone and 2,4-dichlorobenzaldehyde in alkaline medium. The molecular structure of the synthesized chalcone was determined by analyzing the single crystal X-ray diffraction data [6]. Further characterization of the compound was done by using infrared spectroscopy.

2. Experimental details

2.1. Synthesis of 3-(2,4-dichlorophenyl)-1-phenylprop-2-en-1-one :

2,4-Dichlorobenzaldehyde (8.74 g, 0.05 mol), acetophenone (6 g, 0.05 mol) and sodium hydroxide (0.1 g, 0.0025 mol) were dissolved in 50 mL methanol. The reaction mixture

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was stirred at 298 K for 24 hours. The solution is cooled and the solid precipitated out was collected. A second crop was obtained by evaporating a part of the solvent. Finally, the product was recrystallized from methanol to give colourless needle like crystals (yield: 59%, mp. 345-346 K).

2.2. X-ray Measurements :

The X-ray diffraction measurements were carried out in Bruker SMART CCD diffractometer at room temperature. The crystal structure was solved and refined by using SHELX97 [7]. The crystal data and details of the X-ray analysis are given in Table 1.

Table 1. Crystal data and structure refinement.

Molecular Formula	C ₁₅ H ₁₀ Cl ₂ O	Radiation	Mo K α
Formula weight	277.13	Calculated Density	1.426 Mg m ⁻³
Temperature	293 (2) K	Adsorption coefficient	0.49 mm ⁻¹
Crystal system	Triclinic	F(000)	568 0
space group	P $\bar{1}$	Crystal size	0.42 × 0.15 × 0.12 mm
Unit cell dimensions		Reflections collected /unique	16672/5889
a	3.9712 (1) Å	θ Range for data collection	0.7 - 28.4°
b	11.7738 (4) Å	Completeness to θ = 28.4	90 %
c	27.7962 (10) Å	Refinement method:	Full-matrix least-squares on F ²
α	90.748 (2)°	Data/parameters	5889/325
β	91.268 (2)°	Goodness-of-fit on F ²	1.040
γ	96.492 (2)°	Final R indices	R1 0.064, wR2 0.162
Volume	1290.83 (7) Å ³	R indices (all data)	R1 0.118, wR2 0.189
Z	4	Largest diff. peak and hole	0.416 and -0.245 eÅ ⁻³

2.3. Infrared Spectroscopic measurements :

The IR spectra of 3-(2,4-dichlorophenyl)-1-phenylprop-2-en-1-one was recorded in KBr matrix at 300 °K using a Perkin Elmer RX1 FT-IR spectrophotometer.

3. Results and discussions

3.1. X-Ray Crystallography :

The structure of 3-(2,4-dichlorophenyl)-1-phenylprop-2-en-1-one with atomic labelling is given in Figure 1. The crystals are triclinic, space group P $\bar{1}$, with four formula units per unit-cell. The molecular structure of the compound contains two molecules in the asymmetric unit (Figure 1). Two benzene rings derived from acetophenone and 2,4-dichlorobenzaldehyde exhibit a non-coplanar geometry, making dihedral angles of 38.74 (9)° and 33.22 (11)° in the two molecules. This is in contrast to a previously reported derivative of chalcone, 2'-

hydroxy-4"-dimethylaminochalcone, in which a dihedral angle of 10.32° is observed between the rings [8]. The non-coplanarity of the two rings is due to the presence of two chlorine atoms on one ring. The mean C—C bond length of 1.3739 (5) Å in the rings of the molecule of the title compound shown in Figure 1 compares well with other reported derivatives of chalcone (1.34 Å : Liu *et al* [8]; 1.39 Å : Murafuji *et al* [9]).

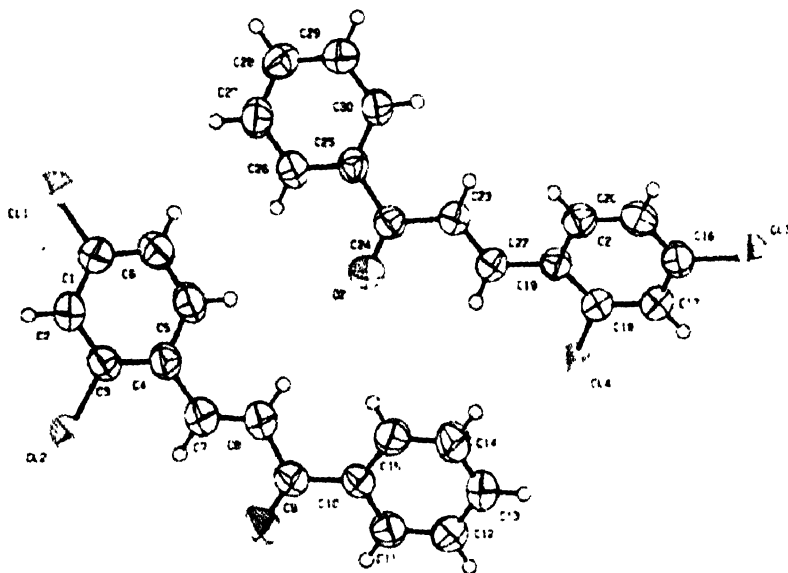


Figure 1. Two asymmetric units of the compound in the crystal lattice

The double-bond character of the bond between C7 and C8 is deduced from the short bond distance [1.314 (5) Å]. [The value in the other molecule is 1.319 (4) Å for C22—C23]. The average C—Cl and C—O bond lengths of 1.7375 (4) and 1.2185 (4) Å, respectively, for the first molecule (Figure 1) are also well within the expected range. The orientation of the rings with respect to the double bond may be described by the torsion angles $C5—C4—C7—C8 = 10.5 (7)^\circ$ and $C8—C9—C10—C15 = 18.5 (6)^\circ$. The torsion angles for the second molecule are $C20—C19—C22—C23 = 24.8 (5)$ and $C23—C24—C25—C30 = -1.6 (5)^\circ$.

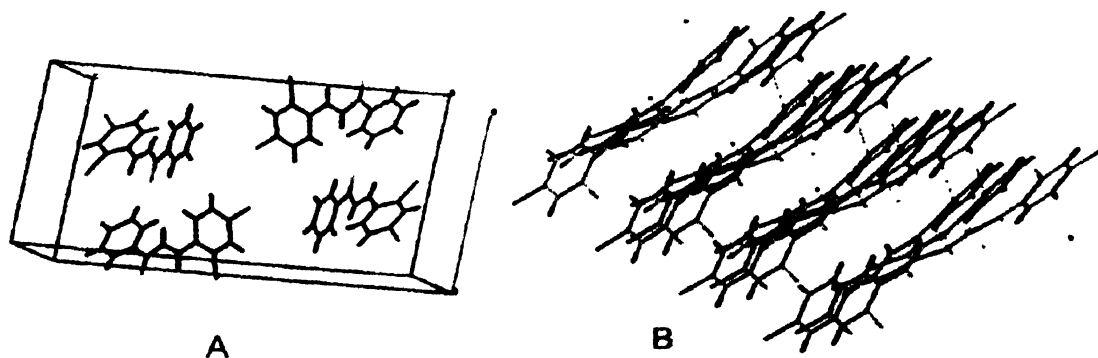


Figure 2. Unit Packing diagram (A) and supramolecular assembly (B) of the compound

A supramolecular assembly of 3-(2,4-dichlorophenyl)-1-phenylprop-2-en-1-one (Figure 2) is formed due to C-H-O interaction of the oxygen atom of a chalcone with one hydrogen atom of another molecule. The second factor which is influencing the supramolecular architecture is the $\pi - \pi$ interactions of the phenyl rings in the molecule. The compound exists in an assembly of sheets due to these C-H....O and $\pi - \pi$ interactions.

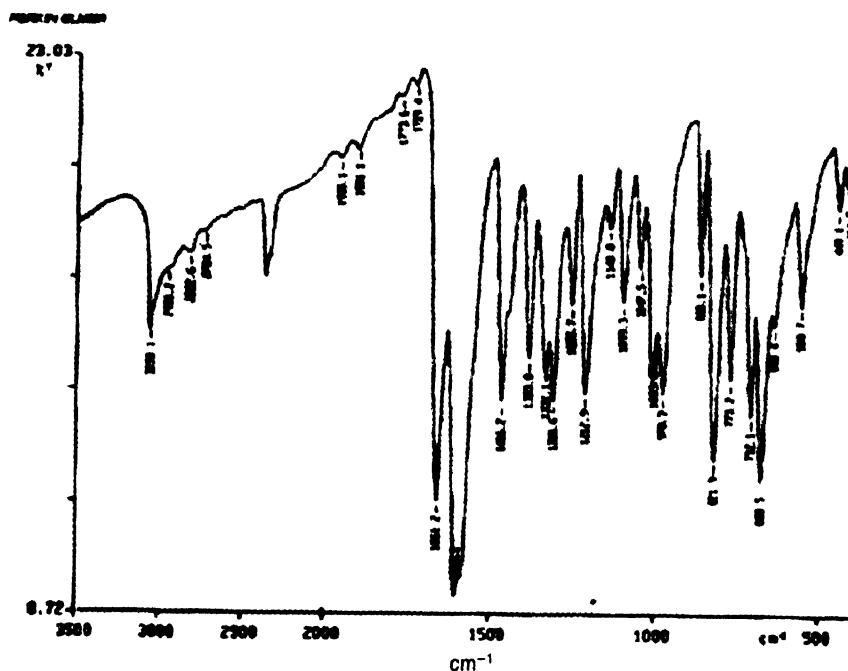


Figure 3. IR spectrum of the compound

3.2. Infrared Spectroscopy :

IR spectrum of the compound (Figure 3) is in excellent agreement with the results of the crystal structure analysis. In this spectrum two prominent bands around 1661 cm^{-1} are due to the presence of the C = O group. A broad band at 1603 cm^{-1} (in the form of a doublet) is due to phenyl rings as well as the olefinic bond. Bands in the range $2724\text{--}3058\text{ cm}^{-1}$ are due to C-H stretching vibrations whereas C-H bending vibrations appear at 1384 cm^{-1} and 1466 cm^{-1} . Strong bands in the range $712\text{--}978\text{ cm}^{-1}$ is due to C-H bending vibrations of the phenyl ring. A prominent peak at 680 cm^{-1} is due the presence of C-Cl bond.

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