International Journal of ChemTech Research
CODEN (USA): IJCRGG, ISSN: 0974-4290, ISSN(Online):2455-9555
Vol.11 No.01, pp 260-264, 2018

Crystal structure analysis of (E) - (2-chlorophenyl) (phenyl) methanone O-benzyl oxime

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Abstract : The crystal structure of(E) - (2-chlorophenyl) (phenyl) methanone O-benzyl oxime($C_{20}H_{16}ClNO$). The compound crystallizes in MonoclinicP21/nspace group with unit cell parameters at 296(2) K as follows: a = 11.3109(7)Å, b =6.0701(4)Å, c =24.4544(15)Å, α =90°, β= 91.258(5)° γ = 90°.Crystal data were collected using BRUKER SMART APEX II CCD X-ray diffractometer. The structure was solved by direct methods and refined on F^2 by full-matrix least-squares procedures to the final R_1 of 0.054usingSHELXL programs.
Key Words : chlorophenyl, methanone, oxime and crystal structure.

Introduction

The conformation of the oxime six-membered ring is halfchair, very similar to that observed in the structure of cyclohexanoneoxime itself[1]. The geometrical parameters of the oxime fragment show standard values for oximes[2]. The most interesting feature of this structure is the system of hydrogen bonds. The oximehydrogen bonds were first classified[3]and divided into three groups. Oxime-type compounds are great important ligands in modern coordination chemistry[4,5]. Structures of oxime-type compounds derived from substituted benzaldehydes and 1-(4-aminophenyl)ethanone haven't been reported so far[6]. In view of this importance and in continuation of our work on the crystal structure analysis of Oxime derivatives, the crystal structure of the title compound has been carried out and the results are presented here.

Experimental

X-ray Structure Determination

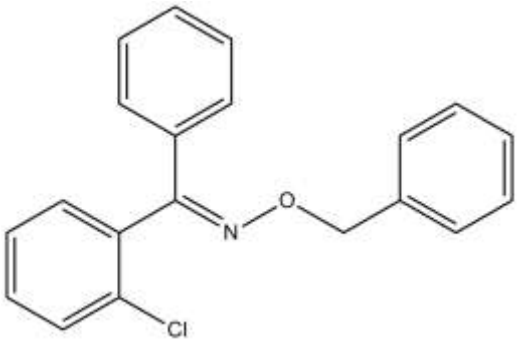
Single crystal of the compound suitable for x-ray diffraction was obtained by slow evaporation method. Three dimensional intensity data were collected on a Bruker[7] SMART APEX CCD Diffractometer using graphite monochromatized Mo-Kα radiation (λ= 0.71073 Å) at Department of chemistry, IIT, Chennai, India. The structure was solved by direct methods and refined on F^2 by full-matrix least-squares procedures using the SHELXL programs[8]. All the non-hydrogen atoms were refined using isotropic and later anisotropic thermal parameters. The hydrogen atoms were included in the structure factor calculation at idealized positions by using a riding model, but not refined. Images were created with ORTEP-3[9].The crystallographic data for the compound are listed in Table 1.

Table 1: Crystal data and structure refinement of the titled compound

Compound	**Parameters**
Empirical formula	$C_{20}H_{16}ClNO$
Formula weight	214.53
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic,P21/n
Unit cell dimensions	a = 11.3109(7)Å alpha = 90°. b = 6.0701(4)Å beta = 91.258(5)°. c = 24.4544(15)Å gamma = 90°
Volume	1678.59(18)$Å^3$
Z, Calculated density	6, 1.273Mg/m^3
Absorption coefficient	0.231 mm^{-1}
F(000)	672
Crystal size	0.35 x 0.20 x 0.16 mm
Theta range for data collection	3.46 to 29.15°.
Limiting indices	-15<=h<=14, -8<=k<=8, -31<=l<=33
Reflections collected / unique	10666 / 3907 [R(int) = 0.0297]
Completeness to theta = 25.00	100.00%
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3907 / 0 / 271
Goodness-of-fit on F^2	1.035
Final R indices [I>2sigma(I)]	**R1 = 0.0549**, wR2 = 0.1250
R indices (all data)	R1 = 0.1091, wR2 = 0.1540
Largest diff. peak and hole	0.159 and -0.266 e. $Å^{-3}$

Synthesis of the compound

To a suspension of NaH (4 mmol) in THF, solution of (E) - (2-chlorophenyl) (phenyl) methadone oxime (2 mmol) was added drop wise. After addition the reaction mixture was stirred for 1 hour. Then benzyl bromide (2 mmol) in THF was added dropwise at 0°C. The resulting mixture was stirred overnight at rt and then quenched with saturated aqueous NH_4Cl solution and extracted with ether (100 mL). The organic solvent were evaporated under vacuum and the residue was subjected to column chromatography to get the pure product. A mixture of ethylacetate and methanol (1:1) used for the crystallization under slow evaporation method.



Results and Discussion

In the title compound (Fig.1), is an aromatic schiff base having an O-benzyl oxime substituent. The dihedral angle between the planes of the two benzene (C1-C6 and C9-C14) rings is 55.28(11)°. The oxime group is tilted by 38.2(2)° with respect to the mean plane of the benzene (C1-C6) ring. The Cl1 deviates from the plane of the benzene (C15-C20) ring by -0.065(2) Å. The C18-C19-C20-Cl1 torsion angle of -175.5(11)° indicates that the chlorine atom Cl1 is not quite coplanar with the phenyl ring. The dihedral angle between the two chlorophenyl (C15-C20 and C21-C26) rings is 1.7(5)°. There are no conventionals hydrogen bonds binding the molecules. The molecules are further linked by C-H...π stacking interactions, forming a three dimensional network. The benzene group is disordered over two orientations, with an occupancy ratio of 0.717 (12):0.283 (12).

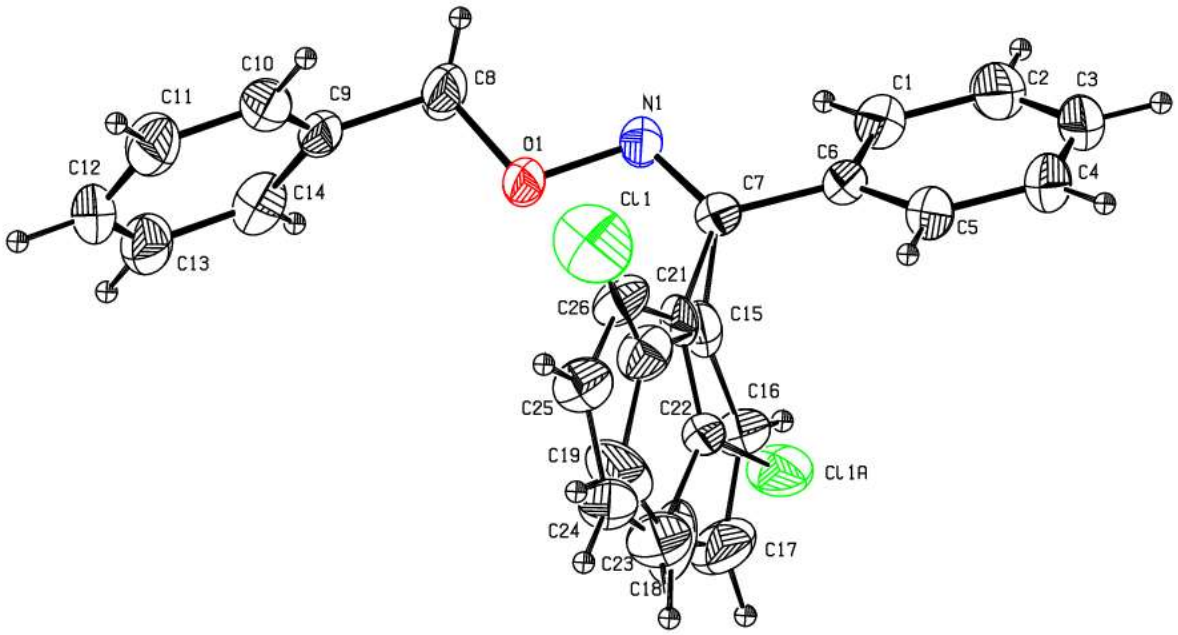


Fig.1.The molecular structure of the title compound, with the atom-numbering scheme. The displacement ellipsoids are drawn at 30% probability level. H atoms are shown as spheres of arbitrary radius.

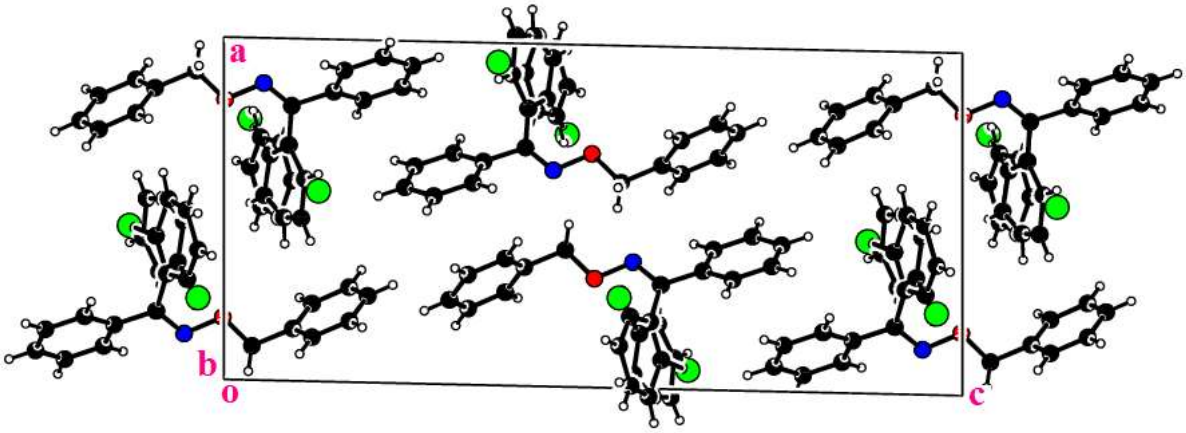


Fig.2. The crystal packing of the title compound, viewed along a axis.

Table 2: Selected Bond lengths (Å)Table 4: Selected Bond angles (º)

Bond	Length (Å)	Bond	Angle (°)
C(7)-N(1)	1.275(2)	N(1)-C(7)-C(15)	128.5(4)
C(7)-C(15)	1.370(1)	N(1)-C(7)-C(6)	116.28(2)
C(7)-C(6)	1.484(2)	C(15)-C(7)-C(6)	113.7(4)
C(7)-C(21)	1.582(8)	N(1)-C(7)-C(21)	120.7(3)
C(9)-C(10)	1.371(3)	C(15)-C(7)-C(21)	21.1(4)
C(9)-C(14)	1.378(3)	C(6)-C(7)-C(21)	122.5(3)
C(9)-C(8)	1.499(3)	C(10)-C(9)-C(14)	118.55(2)
C(6)-C(5)	1.384(3)	C(10)-C(9)-C(8)	120.6(2)
C(6)-C(1)	1.385(3)	C(14)-C(9)-C(8)	120.9(2)
C(1)-C(2)	1.376(3)	C(5)-C(6)-C(1)	118.76(2)
C(4)-C(3)	1.366(4)	C(5)-C(6)-C(7)	120.05(2)
C(4)-C(5)	1.381(3)	C(1)-C(6)-C(7)	121.19(2)
C(14)-C(13)	1.374(3)	C(2)-C(1)-C(6)	120.6(2)
C(13)-C(12)	1.361(3)	C(3)-C(4)-C(5)	120.1(2)
C(10)-C(11)	1.372(3)	C(13)-C(14)-C(9)	120.6(2)
C(2)-C(3)	1.377(3)	C(12)-C(13)-C(14)	120.0(2)
C(11)-C(12)	1.365(3)	C(4)-C(5)-C(6)	120.5(2)
O(1)-N(1)	1.406(2)	C(9)-C(10)-C(11)	120.7(2)
O(1)-C(8)	1.422(2)	C(1)-C(2)-C(3)	119.9(2)
C(21)-C(22)	1.336(1)	C(12)-C(11)-C(10)	120.2(2)
C(21)-C(26)	1.339(8)	C(4)-C(3)-C(2)	120.2(2)
Cl(1A)-C(22)	1.731(5)	C(13)-C(12)-C(11)	119.9(2)
C(22)-C(23)	1.597(1)	N(1)-O(1)-C(8)	108.48(1)
Cl(1)-C(20)	1.721(7)	C(7)-N(1)-O(1)	112.30(1)
C(23)-C(24)	1.25(3)	O(1)-C(8)-C(9)	107.45(12)
C(24)-C(25)	1.414(1)	C(22)-C(21)-C(26)	115.8(6)
C(26)-C(25)	1.382(6)	C(22)-C(21)-C(7)	122.9(5)
C(17)-C(18)	1.23(2)	C(26)-C(21)-C(7)	121.2(7)
C(17)-C(16)	1.372(1)	C(21)-C(22)-C(23)	121.4(9)
C(16)-C(15)	1.447(1)	C(21)-C(22)-Cl(1A)	119.8(4)
C(15)-C(20)	1.320(8)	C(23)-C(22)-Cl(1A)	118.7(8)
C(20)-C(19)	1.457(10)	C(24)-C(23)-C(22)	113.7(13)
C(19)-C(18)	1.31(3)	C(23)-C(24)-C(25)	126.8(8)

Conclusion

The crystal structure analysis of a novel oxime compound was studied using x-ray diffraction methodthere are no conventionals hydrogen bonds binding the molecules. The molecules are further linked by C-H...π stacking interactions, forming a three dimensional network. The benzene group is disordered over two orientations, with an occupancy ratio of 0.717 (12):0.283 (12).

Acknowledgments

R. A. gratefully acknowledges the DST-SERB for young scientist start-up research grant (YSS/2014/000561) and DST-FIST for providing NMR facilities to the department.

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