

# ChemTech

International Journal of ChemTech Research

CODEN (USA): IJCRGG, ISSN: 0974-4290, ISSN(Online):2455-9555 Vol.10 No.13, pp 175-178, **2017** 

# Crystal structure analysis of 4,5-diphenyl-3,5dihydroisochromeno[8,1-ab]phenazine

# K. Elumalai, K. Sakthi Murugesan\*

Department of Physics, Presidency College (Autonomous), Chennai-600 005, India

**Abstract** : The title compound,  $C_{30}H_{18}N_2O$ , crystalized with two independent molecules (A and B) in the asymmetric unit. In the molecule A, the plane of the central chromene ring forms dihedral angles of 66.4(2) and 41.7(2)°, with that of the therminal two benzene rings, and the dihedral angle between planes of the terminal phenazine and chromene rings s  $1.04(11)^\circ$ . The corresponding values for molecules B are 65.9(2), 41.6(2) and  $0.30(13)^\circ$ , respectively. The layers stack with interactions of the type (benzene) C...H... $\pi$  (outer-C8 ring of the fused the system) connections them. The crystal packing also features  $\pi$ ... $\pi$  interactions. There are no classical hydrogen atoms present.Crystal data were collected using BRUKER SMART APEX II CCD X-ray diffractometer. The structure was solved by direct methods and refined on F<sup>2</sup> by full-matrix least-squares procedures to the final R<sub>1</sub> of 0.065usingSHELXL programs. **Key Words :** Isochromene, phenazine and crystal structure.

# Introduction

Chromene derivatives are very important heterocyclic compounds that have a variety of industrial, biological and chemical synthesis applications<sup>1,2</sup>. They exhibit a number of pharmacological activities such as anti-HIV, anti-inflammatory, anti-bacterial, anti-allergic, anti-cancer, etc<sup>3,4</sup>.

# 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<sup>5</sup> SMART APEX CCD Diffractometer using graphite monochromatized Mo-K $\alpha$  radiation ( $\lambda$ = 0.71073 Å) at Department of chemistry, IIT, Chennai, India. The structure was solved by direct methods and refined on F<sup>2</sup> by full-matrix least-squares procedures using the SHELXL programs<sup>6</sup>. 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<sup>7</sup>.The crystallographic data for the compound are listed in Table 1.

Compound	Parameters
Empirical formula	C30 H18 N2 O
Formula weight	422.46
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	monoclinic, P21/n
	$a = 16.2577(6)$ Å $alpha = 90.00^{\circ}$ .
	b = 10.3688(4)Å beta = 95.868(2)°.
Unit cell dimensions	c = 25.4340(2)Å gamma = 90.00°
Volume	$4265.0(2)\text{\AA}^3$
Z, Calculated density	4, $1.3159(1)$ Mg/m <sup>3</sup>
Absorption coefficient	$0.080 \text{ mm}^{-1}$
F(000)	1760
Crystal size	25 x 30 x 20 mm
Theta range for data collection	1.48 to 28.58°.
Limiting indices	-21<=h<=21, -13<=k<=11, -33<=l<=33
Reflections collected / unique	38393 / 10595 [R(int) = 0.0614]
Completeness to theta $= 25.00$	100.00%
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	10595 / 0 / 596
Goodness-of-fit on F <sup>2</sup>	0.853
Final R indices [I>2sigma(I)]	R1 = 0.0456, wR2 = 0.099
R indices (all data)	R1 = 0.2365, WR2 = 0.1669
Largest diff. peak and hole	$0.159 \text{ and } -0.144 \text{ e. } \text{Å}^{-3}$

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

#### Synthesis of the compound

To an dried 50 mL round bottom flask fitted with a reflux condenser were added 2-hydroxybenzoic acid 1 (0.3mmol), 1,2-bis(4-methoxyphenyl)ethyne 2 (0.3mmol),  $[RuCl_2(p-cymene)]2$  (5.0mol%), AgOAc (1.0equiv) and AgSbF<sub>6</sub> (20mol%) in 1,2-DCE. The reaction mixture was refluxed for 12 hours. After cooling to ambient temperature, the reaction mixture was diluted with CH<sub>2</sub>Cl<sub>2</sub>, filtered through Celite and the filtrate was concentrated under reduced pressure. The crude rest was purified through a silica gel column using hexane and ethyl acetate as eluent. To give afford desired product 3 was obtained in 65 % yield. The compound was crystallized slow evaporation of chloroform.



#### **Results and Discussion**

The symmetric unit of the title compound is shown in Fig. 1. The title compound crystallized with two molecules (A and B) in the asymmetric unit (Fig. 1). In both molecules, the C-O bond lengths are in the range 1.373(6)-1.393(6)Å and represent single-bond character. In molecules A, the torsion angles C6-N2-C7-C8 = 179.6(5) and C9-O1-C10-C11° show that the ethylidenemethanamine and methoxyethanegroups have a +ap and +sp orientation with respect to the phenazine. The arrangement in molecules B is slightly different, with torsion angles C55-C56-C57-C58 = 0.9(9) and C59-C60-C55-C40 =  $177.6(5)^\circ$ .

The layers stack with interactions of the type (benzene) C-H... $\pi$ (outer-c8 ring of the fused the system) connections them. The crystal packing also features $\pi$ ... $\pi$  interactions. There are no classical hydrogen atoms present.

# Table 2: Hydrogen-bond geometry [Å]

	Angle (°)			
D—HA	D—H	HA	DA	D—HA
С8Н8Сg26	0.93	2.730	3.834(4)	164

Symmetry code: x, y, z



Fig.1.The molecular structure of the two independent molecules of the title compound, showing the atom labeling. Displacement ellipsoids are drawn at the 30% probability level.



Fig.2. The packing view of the title compound along the c axis. The dashed lines represent the hydrogen bonds. For clarity sake, hydrogen atoms not involved in hydrogen bonding have been omitted.

Atom	Angles	Atom	Length
C(45)-C(46)-C(47)	118.1(5)	C(46)-C(45)	1.400(6)
C(45)-C(46)-C(48)	123.6(5)	C(46)-C(47)	1.412(6)
C(47)-C(46)-C(48)	118.3(5)	C(46)-C(48)	1.457(6)
C(38)-C(39)-O(2)	118.3(5)	C(39)-C(38)	1.341(6)
C(38)-C(39)-C(47)	123.1(5)	C(39)-O(2)	1.372(5)
O(2)-C(39)-C(47)	118.5(5)	C(39)-C(47)	1.438(6)
N(4)-C(36)-C(35)	119.8(6)	C(36)-N(4)	1.348(6)
N(4)-C(36)-C(31)	121.7(6)	C(36)-C(35)	1.421(7)
C(35)-C(36)-C(31)	118.5(5)	C(36)-C(31)	1.417(7)
C(60)-C(59)-C(58)	120.1(6)	C(59)-C(60)	1.380(6)
C(58)-C(57)-C(56)	120.9(6)	C(59)-C(58)	1.388(8)
C(51)-C(52)-C(53)	119.4(6)	C(57)-C(58)	1.373(7)
C(27)-C(28)-C(29)	120.4(6)	C(57)-C(56)	1.380(6)
C(9)-O(1)-C(10)	119.7(4)	C(52)-C(51)	1.374(7)
C(18)-N(1)-C(1)	116.7(5)	C(52)-C(53)	1.391(7)
C(16)-C(17)-C(12)	122.5(5)	C(28)-C(27)	1.358(7)
C(16)-C(17)-C(9)	118.2(5)	C(28)-C(29)	1.368(7)
C(12)-C(17)-C(9)	119.3(5)	O(1)-C(9)	1.375(5)
C(10)-C(11)-C(12)	118.0(5)	O(1)-C(10)	1.392(6)
C(10)-C(11)-C(19)	120.7(5)	N(1)-C(18)	1.323(6)
C(12)-C(11)-C(19)	121.2(5)	N(1)-C(1)	1.358(6)
C(15)-C(16)-C(17)	118.7(5)	C(17)-C(16)	1.397(6)
C(15)-C(16)-C(18)	121.1(5)	C(17)-C(12)	1.404(7)
C(17)-C(16)-C(18)	120.2(5)	C(17)-C(9)	1.430(7)
N(2)-C(7)-C(8)	118.4(5)	C(11)-C(10)	1.340(6)
N(2)-C(7)-C(18)	122.2(5)	C(11)-C(12)	1.455(6)
C(8)-C(7)-C(18)	119.4(5)	C(11)-C(19)	1.481(7)
C(13)-C(12)-C(17)	117.4(5)	C(16)-C(15)	1.397(6)
C(13)-C(12)-C(11)	123.2(5)	C(16)-C(18)	1.458(6)
C(17)-C(12)-C(11)	119.4(5)	C(7)-N(2)	1.339(6)

Table 3: Selected Bond angles (°)Table 4: Selected Bond lengths (Å)

## Conclusion

The crystal structure analysis of a novel isochromene compound was studied using x-ray diffraction method. The crystal packing also features  $\pi$ ... $\pi$  interactions. There are no classical hydrogen atoms present.

## Acknowledgments

The authors thank the Department of chemistry, IIT, Chennai, India, for X-ray intensity data collection.

### References

- 1. Ercole, F., Davis, T. P. & Evans, R. A. (2009). Macromolecules, 42, 1500--1511.
- 2. Farrugia, L. J. (2012). J. Appl. Cryst. 45, 849--854.
- 3. Geen, G. R., Evans, J. M. &Vong, A. K. (1996). *Comprehensive Heterocyclic Chemistry*, 1st ed., edited by A. R. Katrizky, Vol. 3, pp. 469--500. New York:Pergamon.
- 4. Khan, K. M., Ambreen, N., Mughal, U. R., Jalil, S., Perveen, S. & Choudhary, M. I. (2010). *Eur. J. Med. Chem.* 45.4058--4064.
- 5. Raj, T., Bhatia, R. K., Kapur, A., Sharma, M., Saxena, A. K. &Ishar, M. P. S. (2010). Eur. J. Med. Chem. 45, 790--794.
- 6. Bruker (2008), APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, US.
- 7. Sheldrick, G. M. (2008). ActaCryst. A64, 112–122.
- 8. Farrugia, L. J. (2012). J. Appl. Cryst. 45, 849--854.
- 9. Cremer, D. & Pople, J. A. (1975). J. Am. Chem. Soc. 97, 1354–1358.