Available online at www.joac.info

ISSN: 2278-1862

Journal of Applicable Chemistry

2017, 6 (3): 400-409

(International Peer Reviewed Journal)

Synthesis, Characterization, Crystal Structure and Hirshfeld Surface Analysis of (1*E*)-1-Phenylethanone (1-Isobutyl-1*H*-Imidazo [4,5-*C*] Quinolin-4-Yl)Hydrazone

K.N. Chethan Prathap[1], Reshma Kayarmar[2], S. Naveen[3], Manjunath Bhat[2], G.K.Nagaraja[2] and N.K. Lokanath[1*]

1. Department of Studies in Physics, University of Mysore, Manasagangotri, Mysuru 570 006, **INDIA**
2. Department of Chemistry, Mangalore University, Mangalore 574 199, **INDIA**
3. Instiution of Excellence, University of Mysore, Manasagangotri, Mysuru 570 006, **INDIA**

Email: lokanath@physics.uni-mysore.ac.in

Accepted on 18th May 2017, Published online on 27th May 2017

ABSTRACT

The compound (1E)-1-phenylethanone (1-isobutyl-1H-imidazo [4,5-c] quinoline-4-yl) hydrazone was synthesized by condensation of 4-hydrazino-1-isobutyl-1H-imidazo[4,5-c]quinoline with acetophenone. The resultant compound was crystallized using ethanol by slow evaporation technique. The structure was investigated by FTIR, ^{1}H NMR, ^{13}C NMR and confirmed by single crystal X-ray diffraction studies. The title compound crystallizes in the monoclinic crystal system, in the space group $P2_1/c$ with cell parameters a=10.3426(7) Å, b=18.4489(13) Å, c=11.7160(9) Å, β=115.409(4)°, Z=4 and V=2013.3(3) Å^3. The structure adopts an E-conformation with respect to C=N bond. The structure exhibits both inter and intra molecular hydrogen bonds of type N—H…N, C—H…N, O—H…N and C—H…O. The Hirshfeld surface analysis for visually analyzing intermolecular interaction in the crystal structure was carried out. H-H (57.9%) interactions play a prominent role in stabilizing the crystal structure.

Keywords: Imidazo-quinoline; Hydrazone; Crystal structure; FTIR spectrum; *E* conformation.

INTRODUCTION

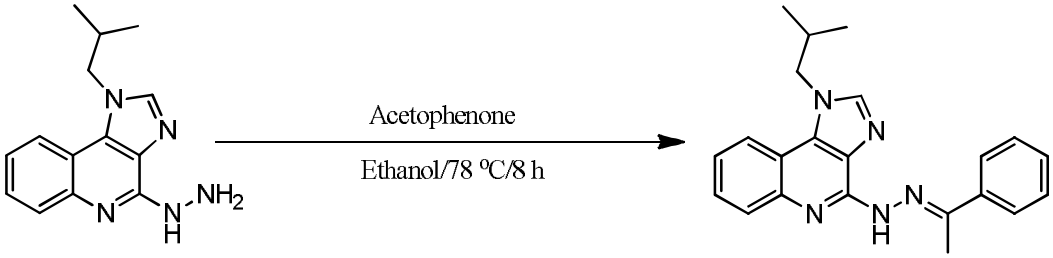
Hydrazones have been identified to possess, antimicrobial, antifungal, analgesic, anti-inflammatory, antimalarial, antitumoral, antiviral, antiplatelet, vasodilator [1-3], anti-HIV [4], antiproliferative [5] and many more biological activities. Of many procedures that have been reported for the synthesis of hydrazones, condensation of appropriate substituted hydrazines/ hydrazides with aldehydes/ketones [6] in an acidic medium is widely used. Another route for the synthesis of hydrazones is using aryldiazonium salts via substitution by elimination of an active hydrogen compound [7]. On the other hand molecules with imidazo-quinoline moiety have been demonstrated to possess antiviral [8], antiallergic [9], antitumor [10], PI3K inhibition [11], immune response modification activity [12]. Dactolisib, an imidazo-quinoline derivative known for its PI3K/mTOR inhibition activity, is being investigated for the treatment of cancer [13] and Alzheimer's disease [14].

Hydrazide-hydrazones compounds are not only intermediates but they are also very effective organic compounds. When they are used as intermediates, coupling products can be synthesized by using the active hydrogen component of –CONHN=CH- azomethine group [15]. N-Alkyl hydrazides can be synthesized by reduction of hydrazones with NaBH4 [16], substituted 1,3,4- oxadiazolines can be synthesized when hydrazones are heated in the presence of acetic anhydride [17, 18]. 2-Azetidinones can be synthesized when hydrazones react with trietylamine chloro acetyl chloride [19]. 4-Thiazolidinones are synthesized when hydrazones react with thioglycolic acid/ thiolactic acid [20]. Encouraged by these pharmacological properties of Imidazoquinoline and Hydrazones, and as a part of our ongoing research on biologically active Imidazoquinoline analogs [21-24], we have synthesized the title compound. The compound obtained was characterized using spectroscopic techniques and finally the structure was confirmed using by X-ray diffraction studies.

MATERIALS AND METHODS

The IR spectra were obtained in KBr disk on a Shimadzu FT-IR 4100 Type. ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker WH-200 or on a Perkin-Elmer EM-390 (100 MHz, 400 MHz, and 300 MHz) in DMSO-d_6 and $CDCl_3$ as solvent and using TMS as an internal standard. Chemical shift values were recorded in δ ppm. The elemental analyses (CHN) were performed on CHNS-O analyzer Flash EA 1112 series.

Synthesis of Compound (1E)-1-phenylethanone(1-isobutyl-1H-imidazo[4,5-c]quinolin-4-yl) hydrazone: A mixture of 4-hydrazino-1-isobutyl-1*H*-imidazo[4,5-*c*]quinoline (0.01 mol), acetophenone (0.01 mol) in absolute ethanol (10 mL) containing acetic acid (0.5mL) was heated under reflux for 8h. The solid separated was filtered washed, dried, and recrystallized from ethanol. The synthetic strategy of the title compound is depicted in scheme 1. The product obtained was yellow crystals (4.0 g, 80%) [m.p. 200°C–204°C].



Scheme 1

X-ray diffraction studies: A good block shaped single crystal of dimensions 0.21x0.20x0.23 mm of the title compound was chosen for an X-ray diffraction study. X-ray intensity data were collected at a temperature of 296 K on a Bruker Proteum2 CCD diffractometer using CuK_α radiation of wavelength 1. 54178 *Å*. Data collection was carried out with different settings of φ (0° to 90°), keeping the scan width of 0.5°, exposure time of 5 s, the sample to detector distance of 45.10 mm and θ value ranging between 4.794° and 64.337°. The complete data set was processed using *SAINT PLUS* [25]. The structure was solved by direct methods and refined by full-matrix least squares method on F^2 using *SHELXS* and *SHELXL* programs [26].

Table-1: Crystal structure and refinement details

Parameter	Value
CCDC deposit number	1539509
Empirical Formula	$C_{22}H_{23}N_5 \cdot H_2O$
Molecular Weight	373.45
Temperature	293(2)° K
Wavelength	1.54178Å
Crystal system, space group	Monoclinic, $P2_1/c$
Unit cell dimensions	a = 10.3426(7)Å b = 18.4489(13)Å c = 11.7160(9)Å β = 115.409(4)°
Volume	2019.3(3) Å^3
Z, Calculated density	4, 1228.0(8) kgm^{-3}
Absorption coefficient	0.626
F_{000}	792
Crystal size (Max, mid & min)	0.45, 0.4 & 0.3 mm
θ range for data collection	4.794° to 64.337°
Limiting indices	$-11 \leq h \leq 9$ $-21 \leq k \leq 20$ $-12 \leq l \leq 12$
Reflections collected/unique	3196/2641
Completeness to θ = 64.42^0	94.6 %
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	3196/0/263
Goodness of fit on F^2	1.046
Final R indices [$I > 2\sigma(I)$]	R_1 = 0.0421 ; wR_2 = 0.1070
R indices [all data]	R_1 = 0.0524 ; wR_2 = 0.1137
Largest diff. peak and hole	0.200 and -0.153 eÅ^{-3}

All the non-hydrogen atoms were revealed in the first difference Fourier map itself. All hydrogen atoms were positioned geometrically (C-H = 0.93 *Å*, O-H = 0.82 *Å* and N-H = 0.86 *Å*) and refined using a riding model with U_{iso}(H) = 1.5 U_{eq} (C,O) and U_{iso}(H) = 1.2 U_{eq} (N). After several cycles of refinement, the final difference Fourier map showed peaks of no chemical significance. About 263 parameters were refined with 2641 unique reflections which saturated the residuals to R_1 = 0.0421 and wR_2=0.1070. The geometrical calculations were carried out using the program PLATON [27]. The molecular and packing diagrams were generated using MERCURY [28]. The details of the crystal structure and data refinement are given in table-1. The list of selected bond lengths, bond angles and torsion angles are given in table-3. Table-4 lists the hydrogen bond geometry. Figure 4 represents the ORTEP of the molecule with thermal ellipsoids drawn at 50% probability.

RESULTS AND DISCUSSION

Elemental Analysis: In order to confirm the chemical composition of the synthesized compound Carbon (C),Hydrogen (H) and Nitrogen (N) analysis was carried out. The experimental and calculated percentages of C, H, and N are given in table 2. The experimental and calculated percentages of C, H, and N were very close to each other within the experimental errors. This confirms the formation of the product in the stoichiometric proportion.

Table 2: Elemental analysis for $C_{22}H_{23}N_5$.

	Element Experimental (%)	**Calculated (%)**
Carbon	73.92	73.90
Nitrogen	19.59	19.57
Hydrogen	6.49	6.47

FT-IR Spectral Analysis: The FT-IR spectrum of the compound is shown in figure 1. The sharp band appeared at 3351 cm^{-1} corresponds to the NH stretching vibration. The characteristic bands at 1583 cm^{-1} and 1475 cm^{-1} are due C=N and C=C 1488 cm−1 stretching. The absorption band at 1377 cm^{-1} assigned for the aromatic amines (C-N). The above mentioned functional groups are in good agreement with the title compound.

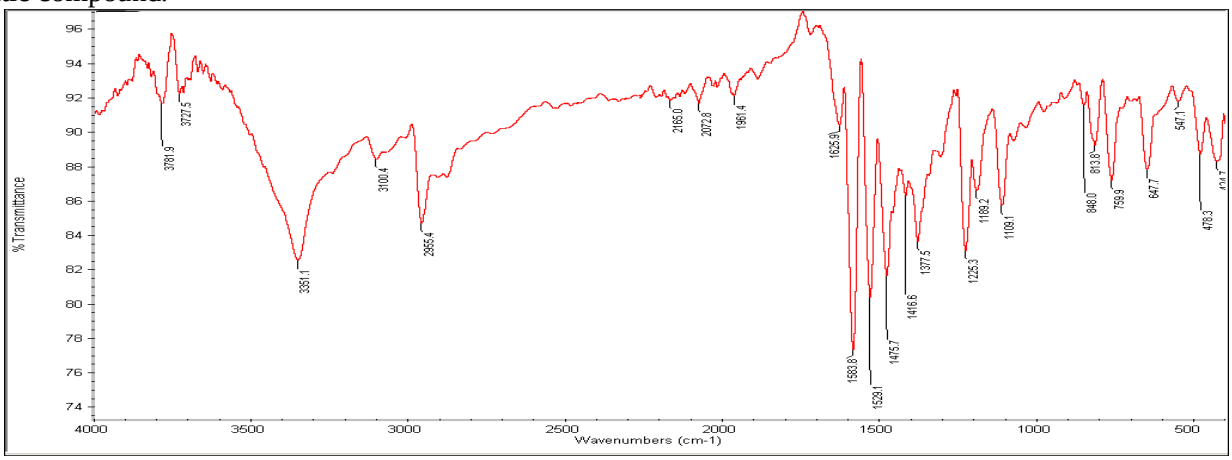


Figure-1: IR spectrum of the compound *(1E)-1-phenylethanone(1-isobutyl-1H-imidazo[4,5-c]quinolin-4-yl)hydrazone.*

^{1}H **Spectral Analysis:** *^{1}H* of the compound is shown in figure 3. The ^{1}H NMR of *(1E)-1-phenylethanone(1-isobutyl-1H-imidazo[4,5-c]quinolin-4-yl)hydrazone* (Figure-1) was characterized by the presence of CH=N protons which appeared as a singlet at δ 9.75 ppm. The nine protons of isobutyl chain of *(1E)-1-phenylethanone (1-isobutyl-1H-imidazo [4,5-c]quinolin-4-yl)hydrazine* appeared as two doublet at δ 1.03 ppm with *J*=6.4 Hz for its $2CH_3$, 4.25 ppm with *J*=7.6 Hz for its CH_2 and one multiplet at δ 2.40-2.30 ppm for its CH protons. The five protons of imidazoquinoline ring appeared as two doublets at δ 7.65 ppm with *J*=8.0 Hz and 8.14 with *J*=7.6 Hz for its two protons, one triplet at δ 7.85 ppm for two protons and one singlet at δ 9.02 ppm for one proton of imidazole. The four protons of phenyl ring appeared as one triplet at 7.28 ppm for three protons, two doublets at δ 7.35 ppm with *J*=7.2 Hz and 7.55 ppm with *J*=8.0 Hz. Methyl group attached to azometine carbon appear as singlet at δ 1.89 ppm. NH of azometine appears as singlet at δ 7.12 ppm.

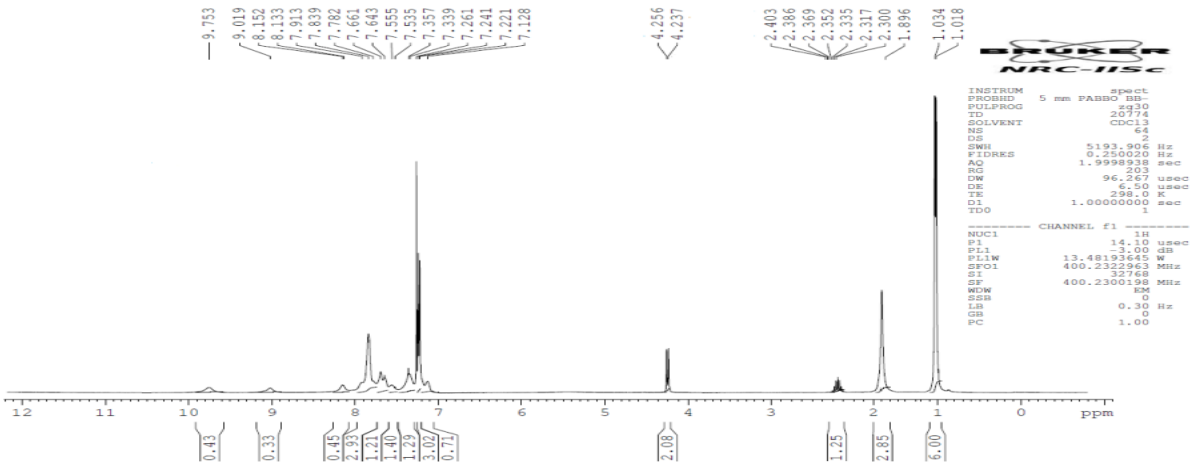


Figure-2: *^{1}H NMR* spectrum of the compound *(1E)-1-phenylethanone (1-isobutyl-1H-imidazo[4,5-c]quinolin-4-yl)hydrazone.*

Crystal structure studies: The imidazole ring and the quinoline rings are planar. The dihedral angle between imidazole ring with atoms C19-C20-N21-N22-N23 and the quinoline moiety with atoms C11-N12-C13…C19-C20 is 3.91(8)° indicating that the imidazole and quinoline rings are almost coplanar. The phenyl ring connected to imidazoquinoline ring through N-N=C hydrazone link makes a dihedral angle of 6.24(8)° indicating that they are coplanar. Also the torsion angles for C1-C6-C7-C8 = 0.4(3) °, N9-N10-C11-N12 = 2.0(2) ° and N10-C11-C20-N21 = 0.0(3) ° indicate that the hydrazone bridge lies in the plane containing both imidazoquinoline and phenyl rings. The torsion angle C7-N9-N10-C11 = 174.51(15)° also indicates that the C=N is in an *E*-conformation. The torsion angle N23-C24-C25-C26 = 60.5(2)° suggests that the side chain atoms linked to fused imidazoquinoline ring adopt gauche staggered conformation.

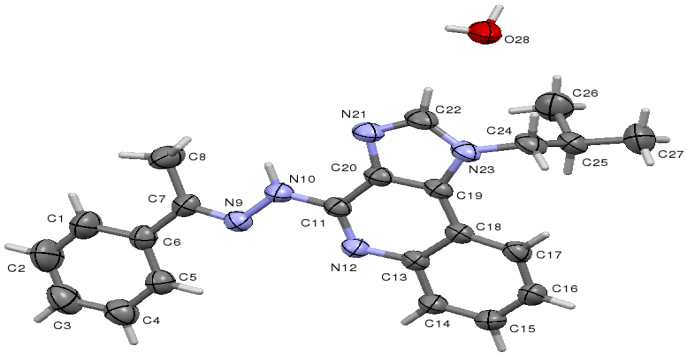


Figure-3: ORTEP diagram of the molecule with thermal ellipsoids drawn at 50 % probability

The structure exhibits both inter and intra-molecular hydrogen bond interactions of the type N—H…N, C—H…N, O—H…N and C—H…O. The inter molecular hydrogen bonds O28—H28A…N21 & O28—H28B…N12 has a length of 2.916(2) *Å* & 2.955(2) *Å*, and angles of 177.66° & 166.94° with symmetry codes *1-x, -y, 1-z* & *1+x, y, z* respectively.

Table 3: Selected bond lengths, bond angles and torsion angles

Bond length in Å			
N(10) - C(11)	1.366(2)	N(12) - C(11)	1.312(2)
N(9) - C(7)	1.287(2)	N(21) - C(22)	1.311(3)
C(6) > C(7)	1.485(3)	N(23) - C(22)	1.357(2)
C(13) - C(18)	1.426(2)	C(19) - C(20)	1.374(2)
N(23) - C(24)	1.467(2)	C(11) - C(20)	1.425(2)
Bond Angle in degrees			
N(10) - N(9) - C(7)	118.08(15)	N(9) - N(10) - C(11)	120.05(14)
C(11) - N(12) - C(13)	119.03(15)	C(20) - N(21) - C(22)	103.29(15)
C(22) - N(23) - C(24)	124.67(16)	N(9) - C(7) - C(6)	115.74(16)
N(10) - C(11) - N(12)	120.80(16)	N(10) - C(11) - C(20)	118.15(15)
N(23) - C(19) - C(20)	105.20(14)	N(21) - C(22) - N(23)	114.24(18)
Torsion angle in degrees			
C(6)-C(7)-N(9)-N(10)	179.50(14)	C(7)-N(9)-N(10)-C(11)	-174.51(15)
C(8)-C(7)-N(9)-N(10)	0.4(2)	N(10)-C(11)-C(20)-N(21)	0.0(3)
N(10)-C(11)-N(12)-C(13)	178.27(14)	C(19)-C(20)-N(21)-C(22)	0.66(19)
C(20)-N(21)-C(22)-N(23)	0.6(2)	C(17)-C(18)-C(19)-N(23)	4.9(3)
C(22)-N(23)-C(24)-C(25)	103.5(2)	C(18)-C(19)-N(23)-C(24)	9.2(3)

There are two intra-molecular hydrogen bonds N10—H10…N21 between the hydrazone moiety & the imidazole moiety with a distance of 2.932(2) Å & an angle of 102.90° and C5—H5…N9 between the benzene ring and hydrazone bridge with distance 2.757(3) Å & an angle of 99.61°. The packing diagram of the molecules viewed down the *a* and *c* axes (Figure 5 and 6 respectively) shows that the molecules exhibit layered stacking and are interconnected by the intermolecular hydrogen bonds to form a one dimensional zigzag chain along *b*-axis.

Table 4: Hydrogen bond geometry.

Number	Type	Scheme			Distances		
		Donor	H	Acceptor	D-H	H-A	D…A
1	Intra	N(10)	H(10)	N(21)	0.86	2.62	2.932(2)
2		O(28)	H(28A)	N(21)	0.84(4)	2.08(4)	2.916(2)
3		O(28)	H(28B)	N(12)	0.82(4)	2.15(4)	2.955(2)
4	Intra	C(5)	H(5)	N(9)	0.93	2.45	2.757(3)
5		C(22)	H(22)	O(28)	0.93	2.5	3.360(3)

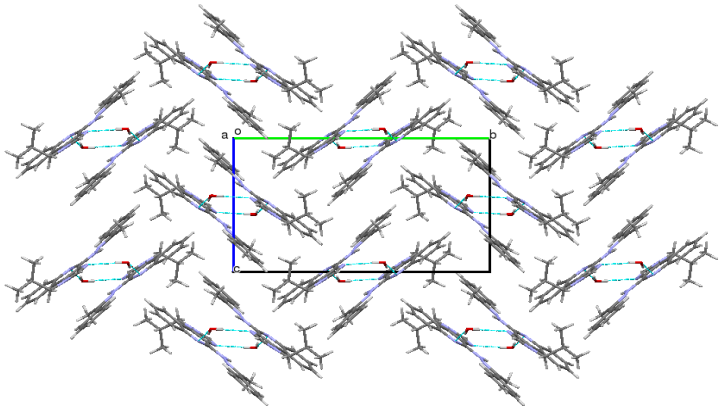


Figure 4: Packing diagram of the title molecule when viewed down along the *a* axis

Hirshfeld surface analysis: The close contacts in the crystal structure were visualized by carrying out the Hirshfeld surface [29-32] analysis. The normalized contact distance d_{norm} based on the distance from a point on the surface to the nearest nucleus outside the surface, d_e and the distance from a point on the surface to the nearest nucleus inside the surface, d_i enables the identification of the regions of particular importance to the intermolecular interactions. The Hirshfeld surface of the title molecule of volume 496.01 $Å^3$, area 447.03 $Å^2$, mapped over a d_{norm} range of -0.542 to 1.661 Å and shown as transparent to allow visualization of all the atoms of the molecule as shown in figure 6.

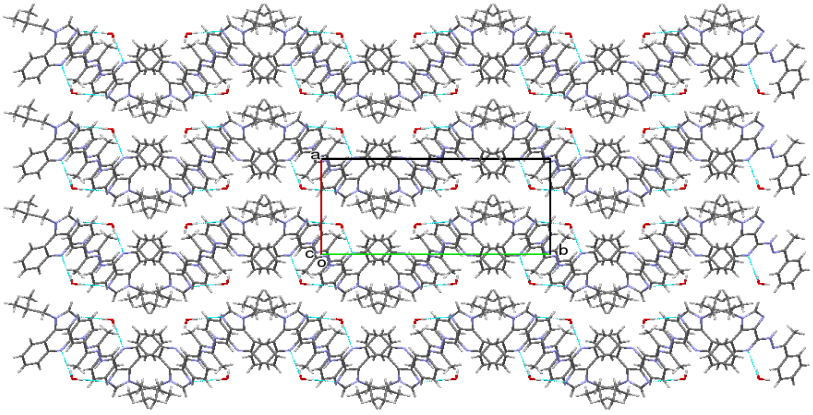


Figure 5: Packing diagram of the title molecule when viewed down along the *c* axis.

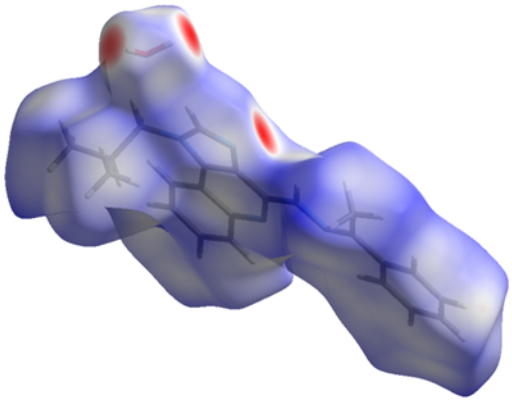


Figure 6: d_{norm} mapped on the Hirshfeld surface of the title molecule.

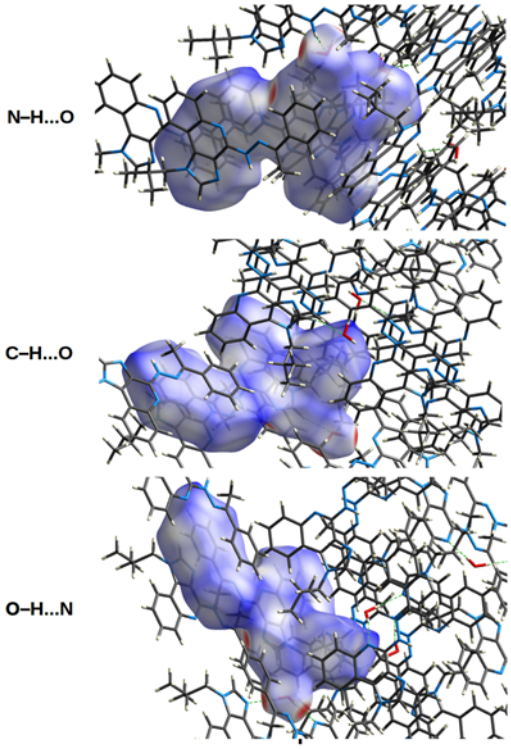


Figure 7: The visualization of N−H···O, C−H···O and O−H···N hydrogen bond interactions in the crystal lattice.

The hydrogen bond interactions discussed in X-ray diffraction studies can be visualized through dark red spots obtained on the Hirshfeld surface due to hydrogen bond acceptors of the types C−H···O, N−H···O and O−H···N are shown in figure 7.

The combination of d_e and d_i in the form of two-dimensional fingerprint plot [33] gives the summary of intermolecular contacts in the crystal lattice. Fingerprint plots for the title molecule and inter molecular close contacts are shown in figure 8a and 8b respectively. The fingerprint plot can be decomposed to highlight the close contacts of a particular pair of atoms. This decomposition enables the separation of contributions from different interaction types which overlap in the full fingerprint.

The H···H short contacts appear as a blunt spike of sky blue colour in the region 1.10 Å < (*d*e + *d*i) < 1.10 Å. The intermolecular close contacts, H···H (57.9%), C···H (19.9%) and N···H (12.8%) at distances shorter than 3.4 Å, play a vital role in the formation of Hirshfeld surface. The close contacts C···C, C···N and O···H contributed 4.1 %, 2.5 % and 2.5 % respectively, in the stabilization of crystal and molecular structure.

CONCLUSIONS

Keeping in view the role the hydrazones and imidazoquinolines, and because of their diverse biological properties, the title compound was synthesized. X-ray diffraction studies reveal that the title compound crystallizes in monoclinic crystal system, in the space group $P2_1/c$. The structure adopts an *E-conformation* with respect to C=N bond. The phenol ring, imidazoquinoline moiety and Hydrazone Bridge that connects them all are nearly coplanar. The crystal structure exhibits both intra and intermolecular hydrogen bonds of type N—H…N, O—H…N, C—H…N and C-H…O. The hydrogen bond interactions

were visualized through dark red spots on the Hirshfeld surface. The intermolecular close contacts, $H \cdots H$ (57.9%) play a prominent role in the stabilization of crystal and molecular structure of the title molecule.

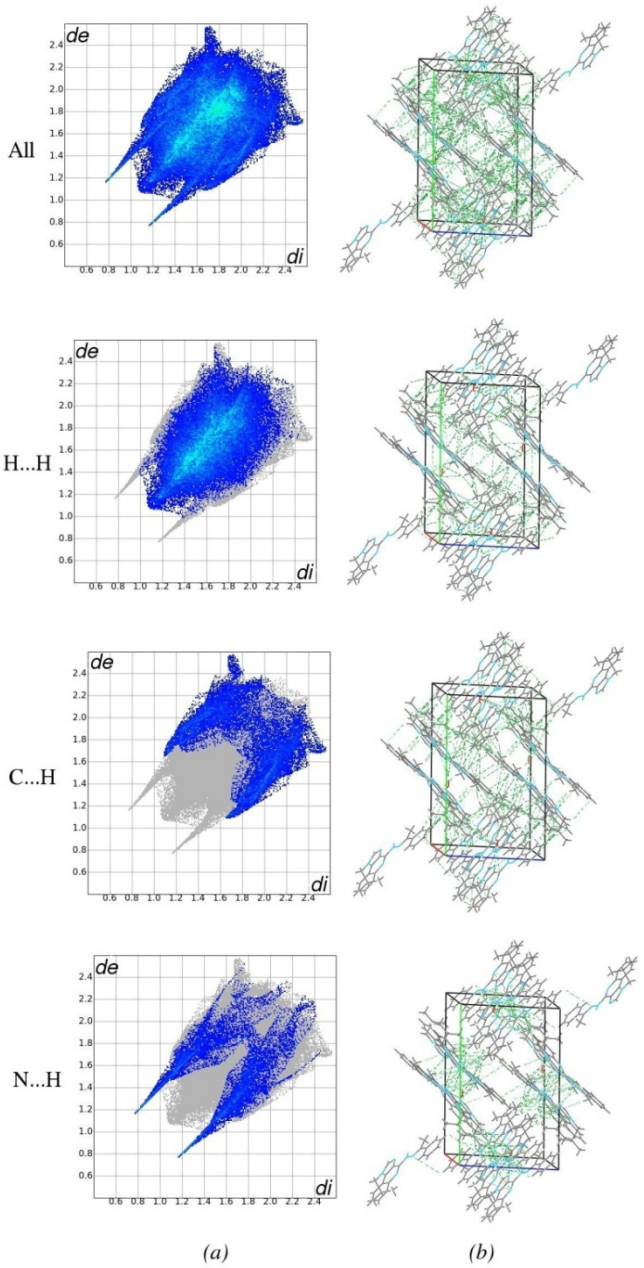


Figure 9: (**a**) Finger print plots of the molecule. (**b**) Inter molecular close contacts.

ACKNOWLEDGEMENTS

The authors are thankful to the Institution of Excellence, Vijnana Bhavana, University of Mysore, Manasagangotri, Mysore, for providing the single-crystal X-ray diffraction data.

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AUTHORS' ADDRESSES

1. **N.K. Lokanath**
Department of Studies in Physics,
University of Mysore,
Manasagangotri, Mysuru 570 006, India
E-mail: lokanath@physics.uni-mysore.ac.in
2. **K.N. Chetan Prathap**
Department of Studies in Physics,
University of Mysore,
Manasagangotri, Mysuru 570 006, India
3. **Reshma Kayarmar**
Department of Chemistry,
Mangalore University,
Mangalore 574 199, India
4. **Manjunath Bhat**
Department of Chemistry,
Mangalore University,
Mangalore 574 199, India
5. **G.K.Nagaraja**
Department of Chemistry,
Mangalore University,
Mangalore 574 199, India
6. **S. Naveen**
Instiution of Excellence,
University of Mysore,
Manasagangotri, Mysuru 570 006, India