ANNEXURE-III

UNIVERSITY GRANTS COMMISSION
BAHADUR SHSH ZAFAR MARG
NEW DELHI- 110 002

STATEMENT OF EXPENDITURE IN RESPECT OF MAJOR RESEARCH PROJECT

1. Name of Principal Investigator : Dr.Kamni
2. Deptt. Of Principal Investigator : Physics
   University/College: Shri Mata Vaishno Devi University
3. UGC approval letter No. and Date: F.No-43-525/2014(SR) dated: Oct., 2015
4. Title of the Research Project: Biological Activity Predictions….Medicinal Importance
5. Effective date of starting the project: 01.07.2015
6. a. Period of Expenditure : From 01.07.2015 to 30.06.2018
   b. Details of Expenditure:

S. No.	Item	Amount Approved(Rs.)	Amount Received as Ist installment	Expenditure Incurred(Rs.)
i.	Books & Journals	10,000/-	10,000/-	7456/-
ii.	Equipment	4,00,000/-	4,00,000/-	3,30,291/-
iii.	Contingency	80,000/-	40,000/-	39,976/-
iv	Field work/Travel(give details in the proforma at annexure IV)	50,000/-	25,000/-	6,438/-
v	Hiring Services	NA	NA	NA
vi	Chemicals & Glassware	50,000/-	25,000/-	23,277/-
viii	Overhead	13,000/-	13,000/-	13,000/-
viii	Any other items(Please specify)(Interest)	----------------	83896/-	1000/-(Paid as Registration fee to present research work in J & K Women Science Congress)

Finance Officer   Registrar
Authorised Signatories
SMVD University

Amar[illegible] Singh & Co.
Chartered Accountants

(Dr. KAMNI)
AP, SOP
SMVDU

c . Staff

Date of Appointment — NA —

S.No	Items	From	To	Amount Approved (Rs.)	Expenditure incurred (Rs.)
1.	Honorarium to PI (Retired Teachers) @ Rs. 18,000/-p.m.	—	—	—	—
2.	**Project fellow:** i) **NET/GATE qualified**-Rs. 16,000/- p.m. for initial 2 years and Rs. 18,000/- p.m. for the third year. ii) **Non-GATE/Non-NET**- Rs. 14,000/- p.m. for initial 2 years and Rs. 16,000/- p.m. for the third year.	—	—	NA	—

1. It is certified that the appointment(s) have been made in accordance with the terms and conditions laid down by the Commission.

2. If as a result of check or audit objection some irregularly is noticed at later date, action will be taken to refund, adjust or regularize the objected amounts.

3. Payment @ revised rates shall be made with arrears on the availability of additional funds.

4. It is certified that the grant of Rs. 5,13,000/- (Rupees FIVE LAKH THIRTEEN THOUSAND only) received from the University Grants Commission under the scheme of support for Major Research Project entitled Biological Activity Predictions - & - Importance vide UGC letter No. F. No: 43-525/2014(SR) dated Oct, 2015 has been fully utilized for the purpose for which it was sanctioned and in accordance with the terms and conditions laid down by the University Grants Commission.

SIGNATURE OF PRINCIPAL INVESTIGATOR

REGISTRAR/PRINCIPAL
(Seal)

Finance Officer Registrar
Authorised Signatories
SMVD University

SIGNATURE OF THE CO-INVESTIGATOR — NA —

Annexure – VII

UNIVERSITY GRANTS COMMISSION
BAHADUR SHAH ZAFAR MARG
NEW DELHI – 110 002

MAJOR RESEARCH PROJECT COPY OF THE SPECIMEN OF HOUSE RENT FOR PROJECT FELLOW

Certified that Shri/Dr. — NA — is paying House Rent of Rs. — and is eligible to draw House Rent Allowances @ — as per University Rules.

Registrar/Principal
(Signature with Seal)

Finance Officer Registrar
Authorised Signatories
SMVD University

Certified that Shri/Dr. — NA — is not staying independently and therefore is eligible to draw House Rent @ of Rs. ________p.m. minimum admissible to a Lecturer as per University Rules.

Registrar/Principal
(Signature with Seal)

Finance Officer Registrar
Authorised Signatories
SMVD University

Certified that Shri/Dr. — NA — has been provided accommodation in the Hostel. But he/she could not be provided with single seated flat type accommodation as recommended by the Commission, Hostel fee @ Rs.________ per month w.e.f. ________ is being charged from him/her.

Finance Officer Registrar
Authorised Signatories
SMVD University

Registrar/Principal
(Signature with Seal)

Annexure - IV

UNIVERSITY GRANTS COMMISSION
BAHADUR SHAH ZAFAR MARG
NEW DELHI – 110 002

STATEMENT OF EXPENDITURE INCURRED ON FIELD WORK

Name of the Principal Investigator:

Name of the Place visited	Duration of the Visit		Mode of Journey	Expenditure Incurred (Rs.)
	From	To		
UGC New Delhi	Jammu New Delhi	New Delhi Jammu	By Air	6,438/-

Certified that the above expenditure is in accordance with the UGC norms for Major Research Projects.

Kamni

SIGNATURE OF PRINCIPAL INVESTIGATOR
(Dr. KAMNI)

REGISTRAR/PRINCIPAL
(Seal)

Finance Officer Registrar
Authorised Signatories
SMVD University

SIGNATURE OF THE CO-INVESTIGATOR – Nil –

Annexure - V

UNIVERSITY GRANTS COMMISSION

BAHADUR SHAH ZAFAR MARG

NEW DELHI – 110 002

Utilization certificate

Certified that the grant of Rs. 5,13,000/- (Rupees Five Lac thirteen thousand only only) received from the University Grants Commission under the scheme of support for Major Research Project entitled Biological-Activity-Predictions --- Medicinal Importance vide UGC letter No. F.No: 43-525/2014(SR) dated Oct., 2015 has been fully utilized for the purpose for which it was sanctioned and in accordance with the terms and conditions laid down by the University Grants Commission. The balance Unspent amount of Rs.1,75,458/- after incurring expenditure of Rs.4,21,438/- has been refunded to UGC vide ch.No./RTGS No. 015744 (UTR No: JAKA20021 74094591) Dt. 10.02.20.

Kamni

SIGNATURE OF THE PRINCIPAL INVESTIGATOR

(Dr. KAMNI)

REGISTRAR/PRINCIPAL

(Seal)

Finance Officer Registrar

Authorised Signatories

SMVD University

STATUTORY AUDITOR

(Seal)

Amarjeet Singh & Co.

FRN-10094N

SIGNATURE OF THE CO-INVESTIGATOR –Nil–

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Annexure-VI

PROFORMA FOR SUPPLYING THE INFORMATION IN RESPECT OF THE STAFF APPOINTED UNDER THE SCHEME OF MAJOR RESEARCH PROJECT

UGC FILE NO. F.______________(HRP)   YEAR OF COMMENCEMENT ☐☐ ☐☐ ☐☐☐☐

TITLE OF THE PROJECT :

1.	Name Of the Principal Investigator :	Prof./Dr. ———				
2.	Name of the University/College	———				
3.	Name of the Research Personnel appointed	— NA —				
4.	Academic qualification	S.No.	Qualifications	Year	Marks	%age
		1.	M.A./M.Sc./M.Tech.			
		2.	M.Phil			
		3.	Ph.D.			
5.	Date of joining	———				
6.	Date of Birth of Research Personnel	———				
7.	Amount of HRA, if drawn	———				
8.	Number of Candidate applied for the post	———				

CERTIFICATE

This is to certify that all the rules and regulations of UGC Major Research Project outlined in the guidelines have been followed. Any lapse on the part of the University will liable to terminate of said UGC project.

Kamini
Principal Investigator

Head of the Deptt.

Registrar/Principal

Finance Officer   Registrar
Authorised Signatories
SMVD University

45

ANNEXURE-A

Project Title : **Biological-Activity-Predictions, X-ray Structure Analysis and Molecular Interactions in Some Organic Compounds of Medicinal Importance**

(i) Brief objectives of the work

The research proposal was envisaged with the following objectives:

(i) **Literature survey**: A general survey of the Cambridge Crystal Data Centre (CCDC, UK) and literature, to know if there exists any published reference with regard to the growth and crystallography of the materials chosen for the present work.

(ii) **Growth**: Several techniques are available for crystallization of single crystals of a variety of organic molecules such as slow evaporation, slow cooling, diffusion methods etc.

(iii) **Crystal Selection**: external morphological features of the crystals can be investigated by using Lieca Polarizing Microscope.

(iv) **Crystal Mounting**: Crystal whose structure is to be determined must be a single crystal without defects. Before crystal data collection each compound mounted on the goniometer will be viewed in color on the video screen of the camera and this shall help us to know which particular orientation is desired for data collection.

(v) **Data Collection**: Three-dimensional intensity data will be collected from the Single Crystal X-ray Diffractometer (SCXRD) which provides (h k l) reflections and their corresponding intensities that serves as an input for undertaking computation of the three-dimensional structure.

(vi) **Structure Determination:** Molecular structure elucidation of the organic compounds and refinement of the same by using a variety of crystallographic softwares. It is proposed to make detailed calculations for various geometrical, structural and conformational parameters viz. bond distances, bond angles, torsion angles, dihedral angles, least square planes, ring conformation etc.

(vii) **Structure-Activity-Predictions**: The structure-activity-predictions is proposed to be carried in detail for each organic compound by using some standard mechanisms. The

4. Comparative crystallographic analysis and role of intermolecular interactions in molecular assemblies in 5th International conference on recent trends and advancements in engineering and technology (ICRTAET), SMVDU 25-26th October, 2018

Kamni

(Dr. KAMNI)
Principal Investigator
MRP-MAJOR-PHYS-2013-26952

ANNEXURE-B

FINAL REPORT

(ii) Work Done so far and results achieved and publications

The project titled **"Biological-Activity-Predictions, X-ray Structure Analysis and Molecular Interactions in Some Organic Compounds of Medicinal Importance"**, presents the molecular and crystal structure of some medicinally important organic compounds by using single crystal X-ray diffraction techniques. The compounds undertaken in the present investigations belong to the following classes of organic materials: Imidazole, Chalcone, Quinoline and Thiophene. In view of the wide-ranging applications of the field of small molecule crystallography, a detailed analysis of the crystal structures, molecular interactions and activity has been reported for various compounds and this forms the main structure of the Ph.D. thesis. The single crystal X-ray diffraction data for each molecule were collected by using an in-house DST-XRD National Facility [Oxford Diffraction X'calibur, Sapphire 3 Diffractometer] which is equipped with graphite-monochromated Mo*Kα* radiation (λ = 0.71073 Å). The crystal structures as reported in the thesis were solved by Direct methods using WINGX Suite- a platform which provides access to a variety of crystallographic soft wares for the computation of the three-dimensional molecular and crystal structure of various types and classes of materials in solid form. All structures as reported in the thesis have been determined with precision and the data has been presented in tabular form and also in the form of diagrams, including the placement of molecules along various crystallographic axis. The references have been appended at the end of each chapter. The thesis has been designed in seven chapters. A brief description of each chapter is presented below:

➢ Synthesis and X-ray crystallographic analysis of two imidazole derivatives

Imidazole derivatives are important building blocks in biologically active compounds and have been reported to have important biological activities including potential α-adrenergic receptor agonist, anti-microbial, anti-parasitic [Neves *et al.*, 2010], anti-convulsant, anti-inflammatory, analgesic [Khan & Chawla, 2002] etc. In view of the importance of naturally occurring

- In addition to this, C-H…π and π…π interactions play a crucial role in assembling the molecules into three dimensional pattern.

Research work presented here shall certainly help the crystallographers to understand the architecture of various medicinally important organic compounds and use this knowledge for drug designing. Study of intermolecular interactions and their control is very important in the area of crystal engineering, to design new materials of desirable properties and also for crystal structure predictions. Thus it may be stated that the crystallographic studies play a very important role in the field of drug designing, as the results from X-ray crystallographic studies provide accurate and reliable three-dimensional structural parameters.

The crystallography research of small molecules is in place for the last over eight decades and the way its scope is expanding day by day especially with regard to using small molecule data, it is expected that this area of research is going to stay for all times to come.

Note:

1. SHELXS97, SHELXL97 [Sheldrick, 2008], PLATON [Spek, 2009], PARST [Nardelli, 1995] softwares have been employed for the computation of three dimensional molecular and crystal structures and PASS [Geronikaki *et al.*, 2008; Filz & Poroikov, 2012] software was used extensively for making some possible predictions about the possible biological activity of the investigated molecules.

2. Crystallographic Information File and Supplementary Data for each structure presented in the thesis have been deposited at Cambridge Crystallographic Data Centre against a deposition number and the same can be obtained on request from ***http://www.ccdc.cam.ac.uk/deposit@ccdc.cam.ac.uk***

Figure 2: ORTEP view of molecules (II) with displacement ellipsoids drawn at 40% probability level. H atoms are shown as small spheres of arbitrary radii.

Compound-**I** [$C_{24}H_{21}N_3OS$] crystallizes in the monoclinic crystal system with space group $P2_1/c$ having following unit cell parameters a = 15.8392 (13) Å, b = 12.9075 (10) Å, c = 10.3997 (8) Å, α = 90° β = 103.354(8)°, γ = 90° and Z = 4. The compound has been refined to an R-value of 0.074.

All the four rings are planar and the sum of bond angle around N2 is 358.3°, which confirms that the atom N2 is in sp^2 hybridized state. The bond distance of C7 = O1 is 1.218(5) Å which is somewhat longer than the standard value for a carbonyl group (1.192 Å); lengthening of this double bond may be due to the involvement of this oxygen atom as a hydrogen bond acceptor. The imidazole ring subtends dihedral angles of 4.6 (1)° and 20.2 (1)° with the thiophene and benzene rings, respectively. The plane of the imidazole ring forms a dihedral angle of 39.9 (1)° with the phenyl ring. An intramolecular C—H...N hydrogen bond closes an S(6) graph set motif in the ring (Fig. 2). In the crystal, there exist a C—H...O hydrogen bond which link molecules into inversion dimers featuring $R_2^2(10)$ graph-set motifs (Fig. 4). All the geometrical parameters of the compound are presented in the form of tables. Data Centre (CCDC) numbers against which the data for this structure has been deposited is: **1472955.** The findings as presented in this chapter has been published [**IUCr Data.** 1 (2016), x160587].

Compound-**II** [$C_{24}H_{21}N_3OS$] Crystallizes in the monoclinic system with space group $P2_1/c$ having following unit cell parameters: a = 20.6769(18), b = 5.40046(4), c = 17.2537(16) Å, α = 90° β = 104.299(9)°, γ = 90° and Z = 4 . The number of observed reflections and final reliability index for the compound are 2215 and 0.050, respectively.

All the aromatic rings are essentially planar. The bond lengths and bond angles have normal values. The methoxy group is essentially planar with the benzene ring to which it is

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2002] etc. Among quinoline derivatives, the tetrahydroquinolines (THQs) are important structural subunits of natural and synthetic products and many THQ derivatives exhibit interesting biological and pharmaceutical activities. Hydroxyquinolines are good substrates for glucoronidation by UDP-glucoronosyl-transferase [Kanou *et al*., 2002]. Backed-up by the immense importance of these derivatives, structure of the following two quinoline derivatives have been reported in this chapter:

I 3-[6-(4-methoxyphenyl)-2-methylpyrimidin-4-yl]-2-(4-methylpiperazin-1-yl) quinoline.

II tert-butyl4-(3-(4-methoxyphenyl) pyrimidinequinolin-2-yl) piperazine-1-carboxylate

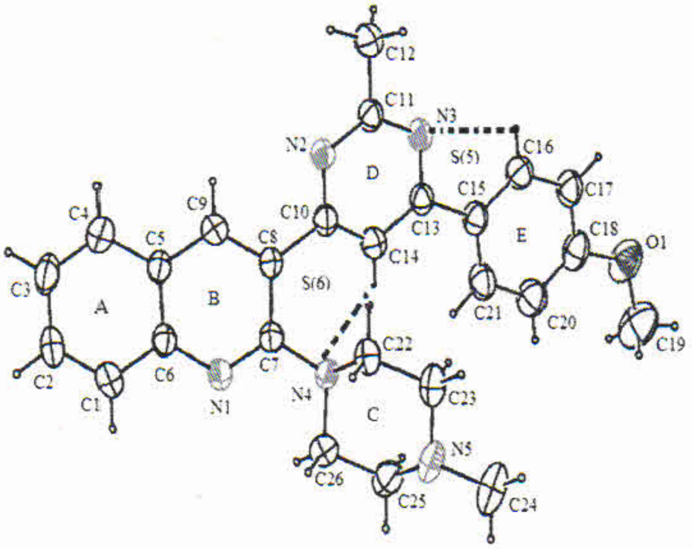

Figure : ORTEP view of the compound-I

Note:

1. SHELXS97, SHELXL97 [Sheldrick, 2008], PLATON [Spek, 2009], PARST [Nardelli, 1995] softwares have been employed for the computation of three dimensional molecular and crystal structures and PASS [Geronikaki *et al.*, 2008; Filz & Poroikov, 2012] software was used extensively for making some possible predictions about the possible biological activity of the investigated molecules.

2. Crystallographic Information File and Supplementary Data for each structure presented in the thesis have been deposited at Cambridge Crystallographic Data Centre against a deposition number and the same can be obtained on request from

http://www.ccdc.cam.ac.uk/deposit@ccdc.cam.ac.uk

References

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interactions. All the geometrical parameters of the compound are presented in the form of tables. Data Centre (CCDC) numbers against which the data for this structure has been deposited is: **1814854**. The findings as presented in this chapter has been published [**IUCr Data.** 3 (2018), x180030].

While compound-**II** crystallizes in the triclinc, $P\bar{1}$ space group with unit cell parameters, a = 11.0894(6) Å, b = 12.6875(6) Å, c = 20.8953(9) Å and Z = 4. The number of observed reflections and the final reliability index for structures are 5244 and 0.065, respectively.

There are two crystallographically independent molecules (A and B), in the asymmetric unit cell. Each molecule consists of four rings, a quinoline ring, piperazinyl ring, pyrimidine ring and methoxy phenyl rings. All bond lengths and bond angles are in normal ranges (Allen *et al.*, 1987). The quinoline ring system form dihedral angles of 30.68(11)° and 37.76(1)° with the plane of the pyrimidine ring in molecules A and B, respectively. In both the molecules, piperazine ring adopts chair conformation. The best mirror plane passes through atoms N4A and N5A and the best two-fold rotational axis bisects the N5A—C23A bond [asymmetry parameter C_S(N4A) = 2.53 and C_2(N5A—C23A) = 1.91] . In molecule B, the best mirror plane passes through atoms N4B and N5B and the best two-fold rotational axis bisects the N5B—C24B bond [asymmetry parameter C_S(N4B) = 0.54 and C_2(N5B—C24B) = 5.2] (Duax & Norton, 1975). The quinoline units are essentially planar with a maximum deviation of 0.0851 (1) Å for atom C21A and 0.0607 (1) Å for C21B. All the geometrical parameters of the compound are presented in the form of tables. Data Centre (CCDC) numbers against which the data for this structure has been deposited is: **1829297**. The findings as presented in this chapter has been published [**IUCr Data.** 3 (2018), x180427].

➢ Synthesis and X-ray crystallography of a thiophen derivative

Thiophene has been found to possess an array of biological activities such as anti-microbial, anti-tumour and anti-inflammatory activity [Iqbal *et al.*, 2012]. These are widely used as building blocks in many agro-chemicals and pharmaceuticals [Sabnis *et al.*, 1999]. In view of some wide range medicinally importance of thiophene derivatives, crystal structure

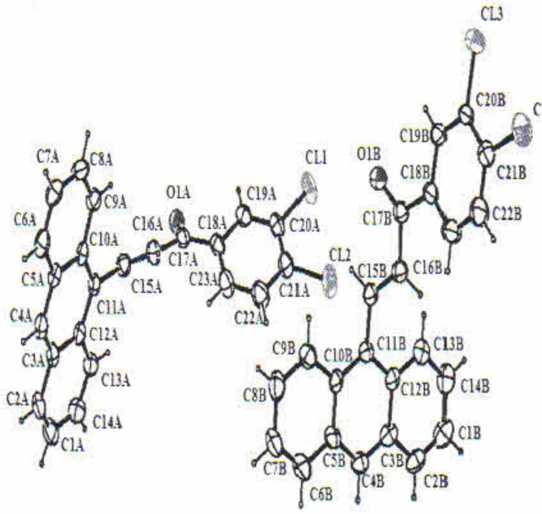

Figure 6 : ORTEP view of the compound - I

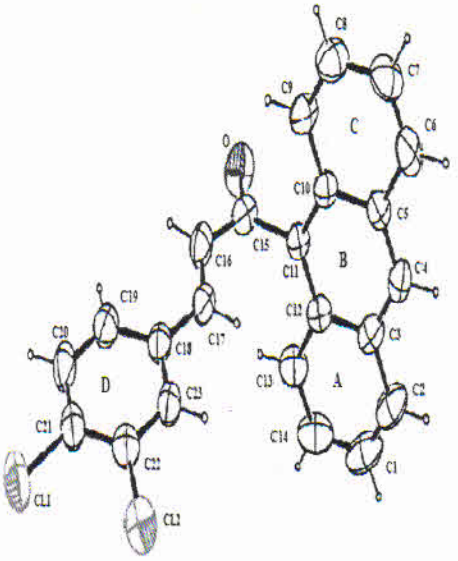

Figure 7 : ORTEP view of the compound - II

Compound-**I** crystallizes in the monoclinic system with space group $P2_1$ having unit cell parameters, $a = 8.7235(7)$ Å, $b = 14.4981(13)$ Å, $c = 14.1480(12)$ Å, $\beta = 95.239(6)^\circ$ and Z = 4. The structure has been refined to a reliability index (R-value) of 0.055.

There are two crystallographically independent molecules (A and B), in the asymmetric unit cell. Bond distances and bond anglesof both the molecules (A and B) lie within the normal ranges. Each molecule consists of two rings, an anthracene and a dichlorophenyl rings. Both the rings are attached by bridge C15A-C16A-C17A-C18A in molecule A and C15B-C16B-C17B-C18B in molecule B. Both asymmetric molecules are almost planar [maximum deviations = 0.048(4) Å and 0.049(4) Å, respectively, for the O1 atoms]. The terminal benzene [C18A–C23A/C18B-C23B] and anthracene [C1A–C14A/C1B-C14B] ring systems form a dihedral angle of 86.51(12)° and 76.42(13)°, respectively. No classical hydrogen bonds were observed but for the van der Waals forces which help stabilize the crystal packing**.** All the geometrical parameters of the compound are presented in the form of tables. Data Centre (CCDC) numbers against which the data for this structure has been deposited is: **1493090**. The interactions C9–H9B···O2, C10–H10A···O1 and C15–H15C···O2 further provide the stability to the molecules within themselves leading to the formation of S(5), S(5) and S(6) graph set motifs (Fig. 7). The two intermolecular hydrogen bonds link the molecules into infinite chains. All the geometrical parameters of the compound are presented in the form of tables. CCDC number assigned to this structure is **1543225.** A part of the work reported in this chapter has been communicated for publication.

➢ Synthesis and structural analysis of two biological important chalcone derivatives

Chalcones constitute an important family of substances belonging to flavonoids, a large group of natural and synthetic products with interesting physicochemical properties, biological activity and structural characteristics [Mohammed *et al.*, 2011]. Chalcones are abundant in edible plants and are considered to be precursors of flavonoids and isoflavonoids which are common substances in plants [Avila *et al.*, 2008] and exhibits interesting pharmacological activities [Mojzis *et al.*, 2008]. Chalcone derivatives are reported to possess a broad spectrum of biological; properties such as antimalarial [Xue *et al.*, 2004], antiangiogenic and antitumor [Lee *et al.*, 2006], antihyperglycemic [Satyanarayana *et al.*, 2004] and anti-inflammatory [Won *et al.*, 2005] activities. Chalcones are important compounds not only because of their biological properties but also because they serve as important intermediates for the synthesis of a large number of heterocyclic systems. In view of the above, the structures of the following two Chalcones derivatives have been reported in this chapter:

I [(2E)-3-(anthrance-9-yl)-1-(3,4-dichlorophenyl) prop-2-en-1-one]

II [(2E)-1-(anthracen-9-yl)-3-(3,4-dichlorophenyl) prop-2-en-1-one].

a thiophen derivative [1-(tert-butyl) 3-ethyl 3-(hydroxy(thiophen-2-yl) methyl) piperidine-1,3-dicarboxylate] has been reported in this chapter:

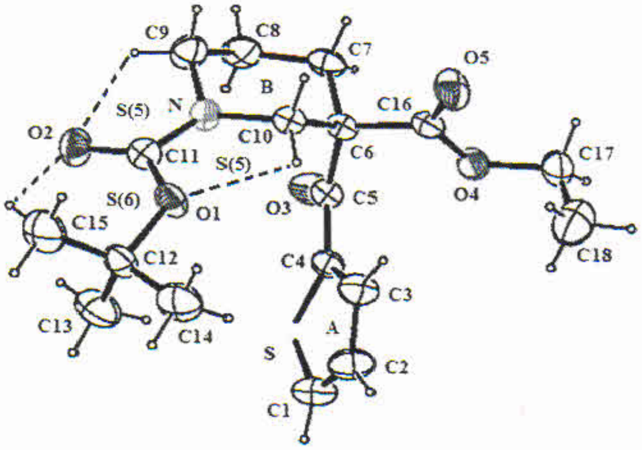

Figure 5: ORTEP view of molecules with displacement ellipsoids drawn at 40% probability level. H atoms are shown as small spheres of arbitrary radii. The broken lines show the intramolecular hydrogen bonding.

The compound crystallizes in the orthorhombic crystal system with space group $P\,c\,a\,2_1$ having following unit cell parameters: a = 19.4502(13), b = 6.3571(4), c =15.2577(10) Å, β = 90.00(0)° and Z= 4. The crystal structure was solved to final Reliability index of 0.035 for 2251 observed reflections.

The molecule consists of a five membered, thiophen ring (ring A) and a six membered piperidine ring (ring B) bridged by carbonyl group. The structural parameters, including bond distances and angles are in normal range (Allen *et al.*, 1987). Piperidine ring deviates significantly from planarity as indicated by the large values of torsion angles in it and adopts the *chair* conformation with mirror plane passing through atoms C8 and C10 [with asymmetry parameter $\Delta C_s(C8) = 4.211$] and best two fold rotational axis bisecting the bonds C7-C8 and N1-C10 [with the asymmetry parameter $\Delta C_2(C7\text{-}C8) = 1.303$].

The two intermolecular C–H···O hydrogen bonds and five intramolecular C–H···O hydrogen bonds links the molecule into a three dimensional network. Intra-molecular findings as presented in this chapter has been published [**Crystallography Reports.** 62 (2017), 7].

Compound-(**II**) crystallizes in the monoclinic system with space group $P2_1$ having unit cell parameters, a = 13.7669(9) Å, b = 11.8400(10) Å, c = 11.3285(8) Å, β = 103.525(6)° and Z = 4. The structure has been refined to a reliability index (R-value) of 0.067, respectively

The molecule consists of a benzene and anthracene moiety. The anthracene and benzene rings are nearly planar with maximum deviation (0.0576) Å observed for C8 [anthracene ring] atom and (-0.0049) Å corresponding to C18 [benzene ring] atom. The anthracene ring system (C1-C14) is twisted at C11-C15 bond from the (2E-1-(anthracene-9-yl)-3-(3,4-dichlorophenyl) acrylaldehyde moiety with the torsion angle [C12-C11-C15-C16] of 93.4(4)°]. The benzene and anthracene moiety for a dihedral angle of 89.43(1)° which makes them inclined at right angles to each other. The O atom attached to C15 adopts +anti-clinical and –syn-clinical conformation with the torsion angles(O-C15-C11-C10) 91.0(4)° and (O-C15-C11-C12) -86.4(4)°, respectively. The structure is stabilized by the two C–H···O intermolecular interactions, where 'O' act as a bifurcated acceptor to C17 (via H17) and C23 (via H23) atoms (Fig. 10). In the molecular packing, the adjacent molecules are interconnected through C17–H17···O and C23–H23···O hydrogen bond and π-π interactions. The intermolecular interactions C–H···O, forming the $R_2^1(6)$ ring set-motif. Data Centre (CCDC) numbers against which the data for this structure has been deposited is: **1508193**. The findings as presented in this chapter has been published [**IOSR Journal of Applied Chemistry.** 10 (2017), 53-57].

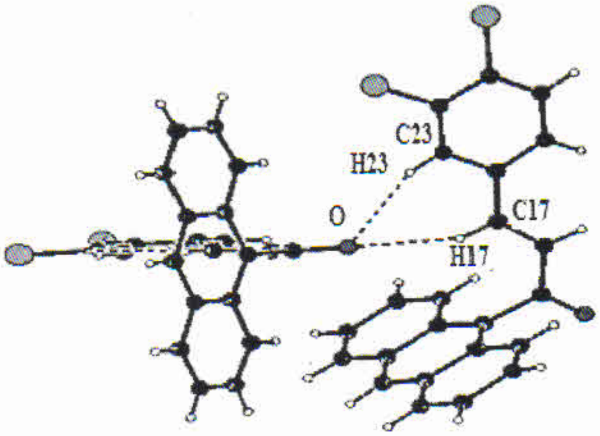

Figure 8: A view of the bifurcated (acceptor) hydrogen bond and $R_2^1(6)$ ring motif.

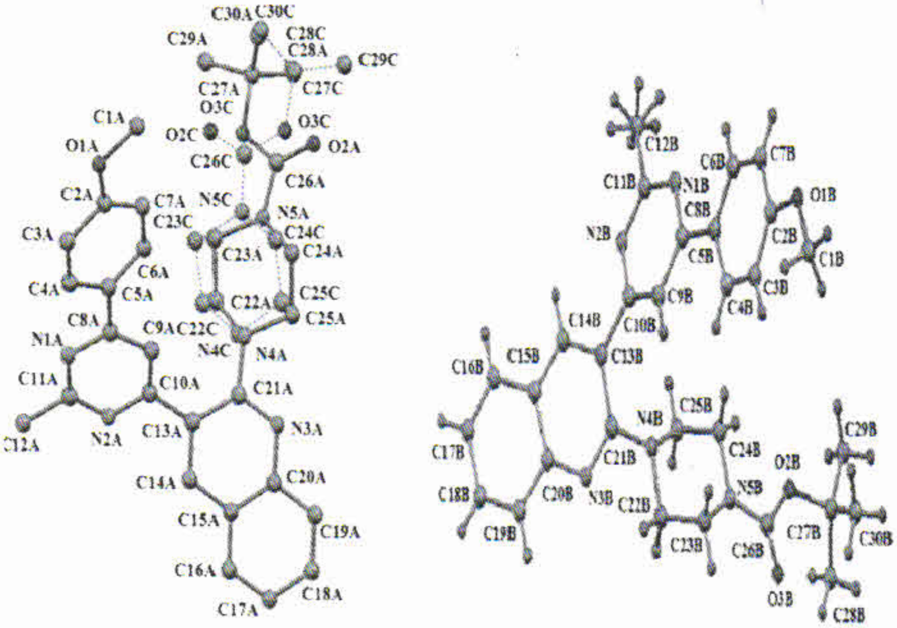

Figure 4 : ORTEP view of the compound-II
(Two molecules in the asymmetric unit)

Compound-**I** crystallizes in the monoclinic system with space group $P2_1/c$ having unit cell parameters, a = 11.9896(9) Å, b = 18.4215(13) Å, c = 10.9903(10) Å, β = 110.153(9)°, Z = 4. The number of observed reflections and the final reliability index for the structures are: 2193, and 0.064, respectively.

In compound-**I**, the mean plane of dihydropyridine ring is nearly co-planar with the mean plane of the cyclohexene ring [dihedral angle = 3.36(1)°]. The bond angles in the methoxyphenyl ring (ring E) vary from 116.9(3)° to 122.3(3)° with an average value of 120.0(3)° which coincides exactly with the theoretical value of sp^2 hybridization. In the piperazinyl ring (ring C) bond angles vary from 108.3(2)° to 110.8(2)° with an average of 110.0(2)°, which is an indication of a perfect aromatic character. The piperazine ring adopts a chair conformation with best mirror plane passing through atoms N4 and N5 [asymmetry parameter C_S (N4) = 1.03] and the best two fold rotational axis bisecting the N4—C25 and N5—C23 bonds [asymmetry parameter C_2 (N5—C25) = 1.27; Duax & Norton, 1975]. The quinoline ring system is essentially planar with a maximum deviation of 0.0583(1)Å for atom C21. In the crystal, molecules are consolidated in the crystal packing by weak C—H…π interactions and π—π stacking

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attached, with a torsion angle (C22-O2-C19-C18) of -0.8(4)°. The imidazole ring subtends dihedral angles of 9.2(2)°, 10.9(2)° and 12.5(2)° with the thiophene, phenyl and methoxy substituted benzene rings, respectively. There are two intramolecular C—H...N hydrogen bonds forming S(5) and S(6) rings and one intramolecular C—H...O hydrogen bond forming an S(6) ring (Fig. 3). The torsion angle C22—O2—C19—C18 is -0.8(4)^0 which conforms that the methoxy group is essentially planar to the benzene ring. All the geometrical parameters of the compound are presented in the form of tables. Data Centre (CCDC) numbers against which the data for this structure has been deposited is: **1472471.** The findings as presented in this chapter has been published [**IUCr Data.** 1 (2016), x160566].

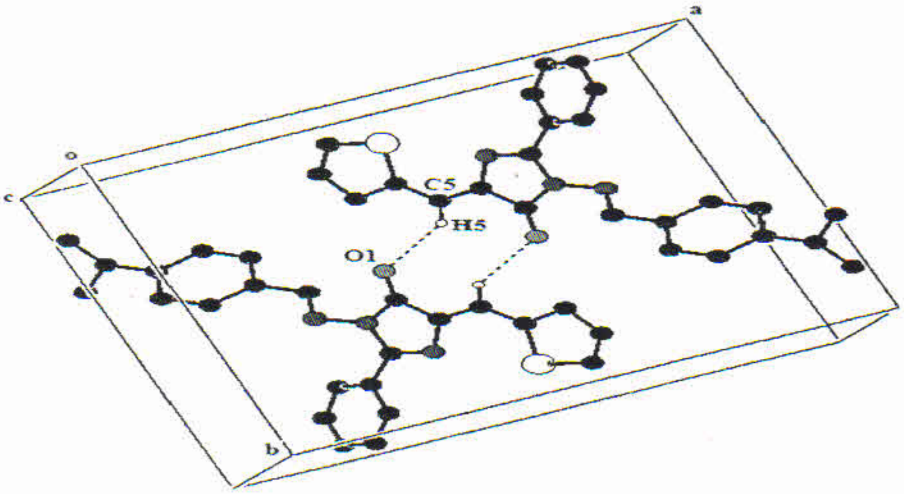

Figure 3 : Molecular plot showing the formation of a dimer through C5-H5-O1 hydrogen bond forming an $R_2^2(10)$ loop.

➤ Structure elucidation and conformational analysis of quinoline derivatives

Quinoline derivatives are privileged drugs due to their wide range of biological activities. Quinolines are found in natural products [Morimoto *et al.*, 1991; Michael, 1997], numerous commercial products including fragrances, dyes [Padwa *et al.*, 1999] and the biologically active compounds [Markees *et al.*, 1970; Campbell *et al.*, 1988], and exhibit diverse range of pharmacological activities such as anti-cancer, anti-bacterial, anti-fungal,[Chauhan & Srivastava, 2001; Chen *et al.*, 2001; Mogilaiah *et al.*, 2001], cholinesterase [Isomae *et al.*,

a][1,8]naphthyridine-6-carboxamides, new compounds with anti-aggressive and potent anti-inflammatory activities *Eur.J. Med. Chem.* 35, pp 1021.

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imidazoles in biological systems, a vast number of synthetic imidazoles are reported to have been prepared as potential pharmacological agents. Few structural studies on imidazole derivatives have also been found to exist in the literature [Jones *et al*., 1996]. In view of the immense importance of these derivatives, the structures of the following two imidazole derivatives has been elucidated in this chapter:

I (4Z)-2-Phenyl-1-{(E)-[4-(propan-2-yl)benzylidene]-amino}-4-[(thiophen-2-yl) methylidene]-1H-imidazol-5(4H)-one.

II (4Z)-1-[(E)-(4-Methoxybenzylidene)-amino]-2-phenyl-4-[(thiophen-2-yl) methylidene]-1H-imidazol-5(4H)-one.

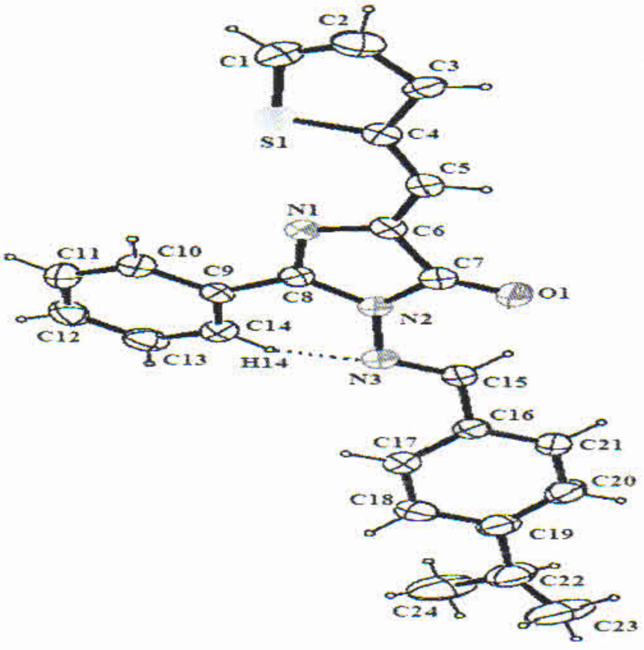
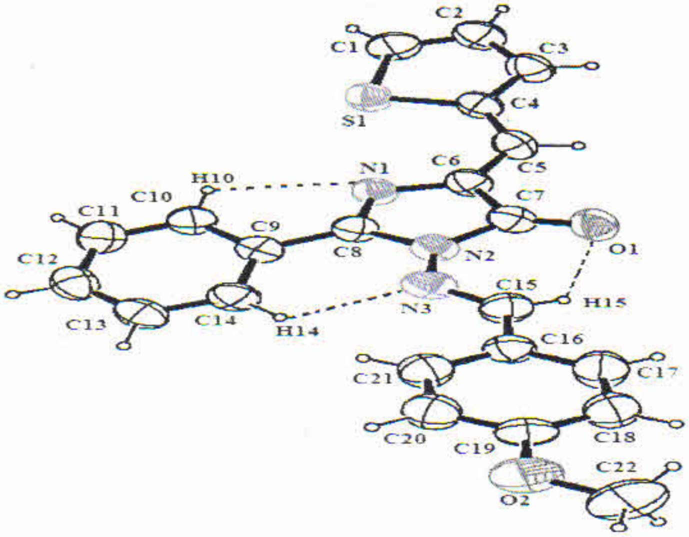


Figure 1: ORTEP view of molecules (I) with displacement ellipsoids drawn at 40% probability level. H atoms are shown as small spheres of arbitrary radii.



ANNEXURE-C

Contribution to the society

Seven biologically important organic compounds have been included in this project. Based on their single crystal growth and X-ray crystallographic studies, it has been observed that these organic compounds have shown their preference to exist in monoclinic space groups, followed by orthorhombic and triclinic. Chemically synthesized and characterized molecules of varied nature whose three-dimensional molecular structures have been presented in the thesis provide a scientific scale to access the impact of various geometrical parameters on the structure and properties of compounds having medicinal importance. Studies of crystal structures of organic molecules by X-ray diffraction provide valuable information about the spatial arrangement of atoms in molecules and their packing in the unit cell. Although some broad conclusions across the whole range of compounds can be drawn, most of the conclusions are specific to a compound or series of compounds undertaken in the present work. All such compounds whose structures have been reported in the thesis are biologically active. The predictions with regard to the activity/inactivity of a molecule have been made on the basis of qualitative presentation of the molecular fragments which allows integration of the information concerning biologically active compounds collected from many different sources into the general PASS software. These compounds have shown their preference to exist in monoclinic followed by orthorhombic and triclinic crystal system. There exist plenty of intra and intermolecular interactions which plays a decisive role in determining the three-dimensional molecular structures. Precisely, the following conclusions can be drawn from analysis of hydrogen bonding interactions present in the investigated structures:

- Presence of intramolecular hydrogen bonds locks the molecular conformation and removes the conformational flexibility.
- A functional group form interactions with other groups but its exact behavior during crystallization depends on the nature and position of functional groups in the molecule.
- The C–H...O hydrogen bonds are specific interactions with distinct structural consequences and may be considered to be structure determining.

predominance of one type of activity over another will be compared for all the investigated compounds.

(viii) **Hydrogen Bonding**: The non-covalent intra- and intermolecular interactions are expected to be analyzed in detail. The molecular packing will also be studied in more details, with special emphasis on the role of hydrogen bonding in molecular entities of these kinds.

(ix) To publish the results in journals of National/ International repute.

Shri Mata Vaishno Devi University, Katra
Faculty of Sciences
School of Physics

No.: SMVDU/SoP/19/ Dated:12 Sept., 2019.

ANNEXURE-XI

Minutes of the Evaluation Committee meeting for final assessment/evaluation of the Project RP-88

Ref.: No. SMVDU/A&R/R&D/15/366 dated: 23rd Nov., 2015.

With reference to above cited reference, the meeting of Evaluation Committee for final assessment/evaluation of the project RP-88 entitled, "Biological activity predictions, X-ray structure Analysis and molecular interactions in some Organic Compounds of medicinal importance" was held on 12 Sept., 2019 at 11:00 pm in the School of Physics. Following members were present:

1) Prof A K Sharma, Head, SoP	Chairman
2) Prof. Rajnikant Dept. of Physics, University of Jammu	External Expert
3) Prof. K K Kapoor Dept. of Chemistry, University of Jammu	External Expert
4) Dr. Kamni, AP, SoP	P.I. of the Project

The committee assessed/evaluated the final report of the above titled project. It was found that all the objectives of the project had been achieved. Based on the project work, 06 research papers have been published in International Journals of repute. The remaining work is submitted for publication in reputed international journals. An amount of Rs. 603000.00 was approved out of which Rs.513000.00 has been received as Ist instalment. Only Ist instalment was received. The total expenditure till 30th June, 2018 is Rs. 337542.00.

The committee assessed/evaluated the final report of the project and found it satisfactory.

Kamni

(Dr. Kamni)
P.I.

(Prof. Rajnikant)
External Expert

(Prof. K K Kapoor)
External Expert

(Prof. A K Sharma)
Chairman

It is also certified that final report, Executive Summary of the report, Research documents, monographic papers under MRP have been posted on the website of Shri Mata Vaishno Devi University.

Kamni
(PI-UGC-MRP)

Dean R&D

Dean (R&D)

28/08/2020
Registrar

Annexure – IX

UNIVERSITY GRANTS COMMISSION
BAHADUR SHAH ZAFAR MARG
NEW DELHI – 110 002

PROFORMA FOR SUBMISSION OF INFORMATION AT THE TIME OF SENDING THE FINAL REPORT OF THE WORK DONE ON THE PROJECT

1. Title of the Project Biological-Activity-Predictions--- Med. Importa
2. NAME AND ADDRESS OF THE PRINCIPAL INVESTIGATOR Dr. KAMNI, School of Physics
3. NAME AND ADDRESS OF THE INSTITUTION Shri Mata Vaishno Devi Univ., KATR
4. UGC APPROVAL LETTER NO. AND DATE 43-525/2014(SR) dt: oct., 2015
5. DATE OF IMPLEMENTATION ....Nov., 2015............
6. TENURE OF THE PROJECT 01.07.2015 - 30.06.2018
7. TOTAL GRANT ALLOCATED ..Rs. 6,03,000/-.........
8. TOTAL GRANT RECEIVED ..Rs. 5,13,000/- (Interest Earned ₹83,896/
9. FINAL EXPENDITURE ....Rs 4,21,438/-....
10. TITLE OF THE PROJECT Biological-Activity-Predictions --- Med. Impor
11. OBJECTIVES OF THE PROJECT ..Attached as Annexure - A
12. WHETHER OBJECTIVES WERE ACHIEVED Yes, Objectives have been achiev
    (GIVE DETAILS) as described in the publicati
13. ACHIEVEMENTS FROM THE PROJECT Quality Publications in Journals of R
14. SUMMARY OF THE FINDINGS Please see Annexure - B
    (IN 500 WORDS)
15. CONTRIBUTION TO THE SOCIETY Please see Annexure - C
    (GIVE DETAILS)
16. WHETHER ANY PH.D. ENROLLED/PRODUCED OUT OF THE PROJECT...Yes.....
17. NO. OF PUBLICATIONS OUT OF THE PROJECT ..06 (Reprints Attached)
    (PLEASE ATTACH)

Kamni
(PRINCIPAL INVESTIGATOR)
(Dr. KAMNI)
AP, School of Physics
Shri Mata Vaishno Devi Univ.
SMVDU - KATRA

(CO-INVESTIGATOR) —— NA——

(REGISTRAR/PRINCIPAL)
Finance Officer Registrar
Authorised Signatories
SMVD University
(Seal)

(4*Z*)-1-[(*E*)-(4-Methoxybenzylidene)amino]-2-phenyl-4-[(thiophen-2-yl)methylidene]-1*H*-imidazol-5(4*H*)-one

ISSN 2414-3146

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Keywords: crystal structure; imidazole; hydrogen bonding.

CCDC reference: 1472471

Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{22}H_{17}N_3O_2S$, the imidazole ring forms dihedral angles of 9.2 (2), 10.9 (2) and 12.5 (2)° with the thiophene, phenyl and methoxy-substituted benzene rings, respectively. There are two intramolecular C—H···N hydrogen bonds forming *S*(5) and *S*(6) rings and one intramolecular C—H···O hydrogen bond forming an *S*(6) ring.

Structure description

Compounds containing the imidazolone as well as the imine moiety exhibit pharmaceutical activities such as antimicrobial (Suthakaran *et al.*, 2008; Patel *et al.*, 2006), antioxidant (Suhasini *et al.*, 2014), anticonvulsant (Mohamed *et al.*, 2012), anthelmintic (Patel *et al.*, 2010) antibacterial (Solankee *et al.*, 2011) analgesic (Sridhar & Ramesh, 2001), anti-inflammatory (Viveka *et al.*, 2015) and antitumor (Hodnett & Dunn, 1970). Apart from their biological activity, Schiff bases have assumed importance as ligands in coordination chemistry (Özkar *et al.* 2004, Pouralimardan *et al.*, 2007; Patti *et al.*, 2009). Structural information on the title compound is useful in developing the coordination properties of Schiff bases and to investigate new ligands.

The molecular structure of the title compound is shown in Fig. 1. The bond lengths and angles have normal values and all aromatic rings are essentially planar. The imidazole ring forms dihedral angles of 9.2 (2), 10.9 (2) and 12.5 (2)° with the thiophene (S1/C1–C4), phenyl (C9–C14) and methoxy-substituted benzene (C16–C21) rings, respectively. The methoxy group is essentially planar with the benzene ring to which it is attached, with

http://dx.doi.org/10.1107/S2414314616005666

②

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10···N1	0.93	2.46	2.792 (4)	101
C14—H14···N3	0.93	2.32	2.942 (4)	124
C15—H15···O1	0.93	2.21	2.867 (3)	127

a C22—O2—C19—C18 torsion angle of −0.8 (4)°. There are two intramolecular C—H···N hydrogen bonds (Table 1), forming $S(5)$ and $S(6)$ rings, and one intramolecular C—H···O hydrogen bond forming an $S(6)$ ring (Fig. 1).

Synthesis and crystallization

A mixture of 3-hydrazinyl-3-oxo-1-(thiophen-2-yl)prop-1-en-2-yl]benzamide (0.01 mol) in 2-propanol (30 ml) with 4-methoxybenzaldehyde (0.01 mol) in presence of one to two drops of sulfuric acid were heated under reflux for 8 h. The reaction mixture was cooled to ambient temperature and poured on cold water. The solid mass obtained was collected by filtration and washed with cold water. It was crystallized at ambient temperature in the presence of air from a mixture of methanol and *N*,*N*-dimethylformamide(1:1 *v*/*v*) (m.p. 421 K).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

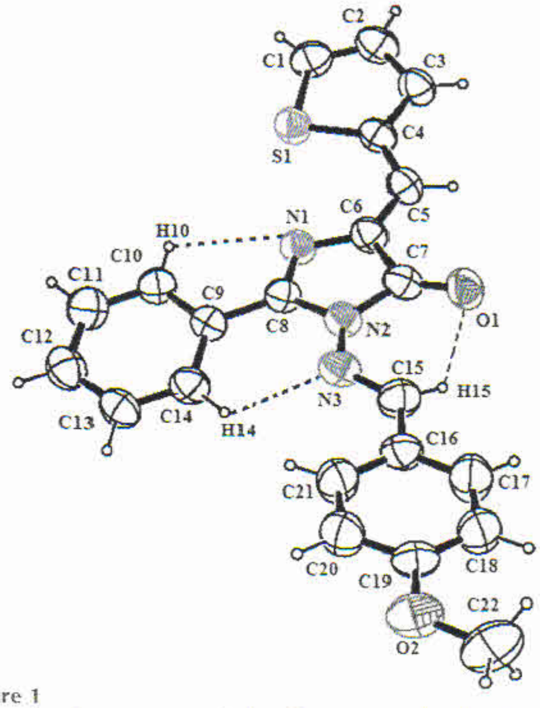

Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 40% probability level. H atoms are shown as small spheres of arbitrary radii. Dashed lines indicate weak intramolecular hydrogen bonds.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{22}H_{17}N_3O_2S$
M_r	387.45
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, b, c (Å)	20.6796 (18), 5.4004 (4), 17.2537 (16)
β (°)	104.299 (9)
V (Å^3)	1867.2 (3)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.20
Crystal size (mm)	0.30 × 0.20 × 0.20
Data collection	
Diffractometer	Oxford Diffraction Xcalibur Sapphire3
Absorption correction	Multi-scan (*CrysAlis RED*; Oxford Diffraction, 2010)
$T_{\min}$, $T_{\max}$	0.769, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7300, 3663, 2215
$R_{\rm int}$	0.028
$(\sin \theta/\lambda)_{\max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.050, 0.136, 1.05
No. of reflections	3663
No. of parameters	255
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å^{-3})	0.24, −0.23

Computer programs: *CrysAlis PRO* (Oxford Diffraction, 2010), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009).

Acknowledgements

RK acknowledges the Indian Council of Medical Research, New Delhi, for Financial Support under Research Project No. BIC/12 (14)/2012. SNK acknowledges the Department of Chemistry, Shri MadhwaVadiraja Institute of Technology, Bantakal (VTU Belgam), for providing research facilities.

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④

full crystallographic data

IUCrData (2016). **1**, x160566 [doi:10.1107/S2414314616005666]

(4*Z*)-1-[(*E*)-(4-Methoxybenzylidene)amino]-2-phenyl-4-[(thiophen-2-yl)methylidene]-1*H*-imidazol-5(4*H*)-one

Vikram D. Singh, Kamni, K. N. Subbulakshmi, B. Narayana, B. K. Sarojini, Sumati Anthal and Rajni Kant

(4*Z*)-1-[(*E*)-(4-Methoxybenzylidene)amino]-2-phenyl-4-[(thiophen-2-yl)methylidene]-1*H*-imidazol-5(4*H*)-one

Crystal data

$C_{22}H_{17}N_3O_2S$
$M_r = 387.45$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 20.6796\ (18)$ Å
$b = 5.4004\ (4)$ Å
$c = 17.2537\ (16)$ Å
$\beta = 104.299\ (9)°$
$V = 1867.2\ (3)$ Å^3
$Z = 4$
$F(000) = 808$
$D_x = 1.378$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1894 reflections
$\theta = 4.2$–$27.1°$
$\mu = 0.20$ mm^{-1}
$T = 293$ K
Rectangular, white
0.30 × 0.20 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.1049 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2010)
$T_{\min} = 0.769$, $T_{\max} = 1.000$
7300 measured reflections
3663 independent reflections
2215 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\max} = 26.0°$, $\theta_{\min} = 3.9°$
$h = -21 \rightarrow 25$
$k = -6 \rightarrow 4$
$l = -21 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.136$
$S = 1.05$
3663 reflections
255 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0436P)^2 + 0.2269P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.24$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

Special details

Experimental. Absorption correction: *CrysAlis PRO*, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27–08-2010 CrysAlis171. NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$
S1	0.44505 (4)	0.95712 (13)	0.26907 (4)	0.0566 (3)
N1	0.33090 (10)	0.8287 (4)	0.12524 (13)	0.0476 (6)
C9	0.28224 (12)	1.0201 (5)	−0.00327 (15)	0.0435 (6)
N2	0.23366 (10)	0.6575 (4)	0.05940 (13)	0.0469 (5)
O1	0.22389 (9)	0.3399 (4)	0.14907 (11)	0.0633 (6)
C7	0.25357 (14)	0.5141 (5)	0.12958 (16)	0.0498 (7)
C8	0.28221 (12)	0.8389 (5)	0.06065 (15)	0.0444 (6)
N3	0.17324 (11)	0.6492 (4)	0.00114 (13)	0.0518 (6)
C10	0.33009 (13)	1.2060 (5)	0.01438 (17)	0.0508 (7)
H10	0.3591	1.2132	0.0650	0.061*
C5	0.35065 (13)	0.5749 (5)	0.24616 (16)	0.0499 (7)
H5	0.3359	0.4344	0.2678	0.060*
C14	0.23988 (13)	1.0125 (5)	−0.07990 (17)	0.0539 (7)
H14	0.2079	0.8885	−0.0930	0.065*
O2	−0.11004 (10)	0.3630 (4)	−0.22410 (13)	0.0745 (6)
C4	0.40723 (13)	0.6972 (5)	0.29599 (15)	0.0473 (7)
C1	0.50041 (14)	0.9677 (6)	0.36046 (17)	0.0613 (8)
H1	0.5338	1.0866	0.3749	0.074*
C13	0.24499 (15)	1.1877 (5)	−0.13666 (17)	0.0595 (8)
H13	0.2164	1.1812	−0.1875	0.071*
C12	0.29240 (15)	1.3713 (5)	−0.11775 (18)	0.0600 (8)
H12	0.2957	1.4896	−0.1558	0.072*
C2	0.49050 (14)	0.7853 (6)	0.40935 (18)	0.0618 (8)
H2	0.5161	0.7651	0.4614	0.074*
C6	0.31598 (13)	0.6373 (5)	0.17177 (16)	0.0479 (7)
C16	0.07441 (14)	0.4331 (5)	−0.06258 (16)	0.0527 (7)
C15	0.13843 (14)	0.4562 (5)	−0.00441 (17)	0.0601 (8)
H15	0.1540	0.3241	0.0297	0.072*
C19	−0.04988 (14)	0.3744 (5)	−0.16912 (17)	0.0543 (7)
C21	0.05040 (15)	0.6050 (5)	−0.12224 (18)	0.0634 (8)
H21	0.0762	0.7426	−0.1268	0.076*
C3	0.43762 (14)	0.6291 (5)	0.37355 (16)	0.0555 (7)
H3	0.4243	0.4939	0.3992	0.067*

C11	0.33506 (14)	1.3800 (5)	−0.04245 (18)	0.0580 (8)
H11	0.3673	1.5035	−0.0299	0.070*
C17	0.03512 (17)	0.2308 (6)	−0.0599 (2)	0.0823 (11)
H17	0.0507	0.1090	−0.0217	0.099*
C18	−0.02719 (16)	0.2015 (6)	−0.1122 (2)	0.0762 (10)
H18	−0.0532	0.0639	−0.1081	0.091*
C20	−0.01077 (15)	0.5760 (6)	−0.17477 (19)	0.0674 (9)
H20	−0.0259	0.6935	−0.2145	0.081*
C22	−0.15119 (16)	0.1525 (6)	−0.2217 (2)	0.0901 (12)
H22A	−0.1300	0.0068	−0.2357	0.135*
H22B	−0.1937	0.1750	−0.2590	0.135*
H22C	−0.1576	0.1342	−0.1687	0.135*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0543 (5)	0.0598 (5)	0.0512 (5)	−0.0039 (4)	0.0044 (4)	0.0050 (3)
N1	0.0426 (13)	0.0533 (13)	0.0432 (13)	−0.0023 (10)	0.0038 (11)	0.0027 (10)
C9	0.0389 (15)	0.0493 (15)	0.0421 (15)	0.0057 (12)	0.0096 (12)	0.0014 (12)
N2	0.0385 (12)	0.0565 (13)	0.0431 (13)	−0.0050 (11)	0.0051 (10)	−0.0003 (10)
O1	0.0606 (13)	0.0725 (13)	0.0539 (13)	−0.0183 (11)	0.0089 (11)	0.0084 (10)
C7	0.0466 (16)	0.0599 (18)	0.0420 (16)	−0.0003 (14)	0.0092 (13)	−0.0004 (13)
C8	0.0377 (14)	0.0510 (16)	0.0440 (16)	−0.0024 (13)	0.0090 (13)	−0.0023 (12)
N3	0.0378 (13)	0.0688 (15)	0.0450 (13)	−0.0054 (12)	0.0032 (11)	−0.0018 (11)
C10	0.0443 (16)	0.0549 (17)	0.0515 (17)	−0.0014 (13)	0.0089 (14)	−0.0028 (13)
C5	0.0461 (16)	0.0572 (16)	0.0457 (16)	0.0024 (13)	0.0103 (13)	0.0079 (13)
C14	0.0468 (16)	0.0619 (18)	0.0491 (17)	−0.0001 (14)	0.0042 (14)	0.0011 (14)
O2	0.0502 (13)	0.0910 (15)	0.0701 (15)	−0.0045 (12)	−0.0083 (11)	−0.0081 (12)
C4	0.0437 (15)	0.0558 (16)	0.0413 (15)	0.0073 (13)	0.0087 (13)	0.0036 (12)
C1	0.0500 (18)	0.0685 (19)	0.0581 (19)	−0.0053 (15)	−0.0002 (15)	−0.0074 (16)
C13	0.0559 (19)	0.070 (2)	0.0478 (17)	0.0097 (16)	0.0038 (15)	0.0091 (15)
C12	0.063 (2)	0.0571 (18)	0.063 (2)	0.0128 (16)	0.0204 (17)	0.0121 (15)
C2	0.0523 (18)	0.079 (2)	0.0469 (17)	0.0021 (16)	−0.0008 (15)	0.0030 (16)
C6	0.0422 (15)	0.0557 (16)	0.0431 (15)	−0.0012 (13)	0.0055 (13)	0.0026 (13)
C16	0.0495 (17)	0.0588 (17)	0.0459 (16)	−0.0063 (15)	0.0045 (14)	−0.0035 (14)
C15	0.0549 (18)	0.0609 (18)	0.0566 (19)	−0.0094 (16)	−0.0008 (15)	−0.0005 (15)
C19	0.0455 (16)	0.0651 (19)	0.0477 (17)	−0.0018 (15)	0.0029 (14)	−0.0114 (15)
C21	0.0567 (19)	0.0653 (19)	0.063 (2)	−0.0126 (16)	0.0040 (16)	0.0053 (16)
C3	0.0524 (17)	0.0639 (18)	0.0459 (17)	0.0007 (15)	0.0039 (14)	0.0076 (14)
C11	0.0522 (17)	0.0547 (17)	0.067 (2)	0.0003 (14)	0.0147 (16)	0.0052 (16)
C17	0.082 (2)	0.080 (2)	0.066 (2)	−0.027 (2)	−0.0183 (19)	0.0186 (18)
C18	0.071 (2)	0.080 (2)	0.064 (2)	−0.0310 (19)	−0.0078 (18)	0.0067 (18)
C20	0.063 (2)	0.069 (2)	0.061 (2)	−0.0026 (17)	−0.0006 (17)	0.0091 (16)
C22	0.054 (2)	0.098 (3)	0.105 (3)	−0.015 (2)	−0.005 (2)	−0.022 (2)

Geometric parameters (Å, º)

S1—C1	1.705 (3)	C1—H1	0.9300
S1—C4	1.725 (3)	C13—C12	1.376 (4)
N1—C8	1.306 (3)	C13—H13	0.9300
N1—C6	1.389 (3)	C12—C11	1.379 (4)
C9—C10	1.390 (3)	C12—H12	0.9300
C9—C14	1.395 (3)	C2—C3	1.398 (4)
C9—C8	1.475 (3)	C2—H2	0.9300
N2—N3	1.398 (3)	C16—C17	1.369 (4)
N2—C8	1.399 (3)	C16—C21	1.384 (4)
N2—C7	1.410 (3)	C16—C15	1.456 (4)
O1—C7	1.215 (3)	C15—H15	0.9300
C7—C6	1.475 (3)	C19—C18	1.353 (4)
N3—C15	1.257 (3)	C19—C20	1.374 (4)
C10—C11	1.380 (4)	C21—C20	1.371 (4)
C10—H10	0.9300	C21—H21	0.9300
C5—C6	1.349 (3)	C3—H3	0.9300
C5—C4	1.431 (3)	C11—H11	0.9300
C5—H5	0.9300	C17—C18	1.388 (4)
C14—C13	1.384 (4)	C17—H17	0.9300
C14—H14	0.9300	C18—H18	0.9300
O2—C19	1.367 (3)	C20—H20	0.9300
O2—C22	1.426 (3)	C22—H22A	0.9600
C4—C3	1.380 (3)	C22—H22B	0.9600
C1—C2	1.345 (4)	C22—H22C	0.9600
C1—S1—C4	91.37 (14)	C1—C2—H2	123.6
C8—N1—C6	106.9 (2)	C3—C2—H2	123.6
C10—C9—C14	118.4 (2)	C5—C6—N1	126.4 (2)
C10—C9—C8	116.6 (2)	C5—C6—C7	123.5 (3)
C14—C9—C8	125.0 (2)	N1—C6—C7	110.0 (2)
N3—N2—C8	123.0 (2)	C17—C16—C21	117.0 (3)
N3—N2—C7	128.3 (2)	C17—C16—C15	119.4 (3)
C8—N2—C7	108.4 (2)	C21—C16—C15	123.6 (3)
O1—C7—N2	127.0 (3)	N3—C15—C16	122.0 (3)
O1—C7—C6	130.8 (3)	N3—C15—H15	119.0
N2—C7—C6	102.1 (2)	C16—C15—H15	119.0
N1—C8—N2	112.5 (2)	C18—C19—O2	124.4 (3)
N1—C8—C9	121.6 (2)	C18—C19—C20	119.6 (3)
N2—C8—C9	125.9 (2)	O2—C19—C20	115.9 (3)
C15—N3—N2	118.3 (2)	C20—C21—C16	121.2 (3)
C11—C10—C9	120.7 (3)	C20—C21—H21	119.4
C11—C10—H10	119.7	C16—C21—H21	119.4
C9—C10—H10	119.7	C4—C3—C2	112.7 (3)
C6—C5—C4	128.3 (3)	C4—C3—H3	123.6
C6—C5—H5	115.8	C2—C3—H3	123.6
C4—C5—H5	115.8	C12—C11—C10	120.2 (3)

C13—C14—C9	120.7 (3)	C12—C11—H11	119.9
C13—C14—H14	119.6	C10—C11—H11	119.9
C9—C14—H14	119.6	C16—C17—C18	122.2 (3)
C19—O2—C22	117.6 (2)	C16—C17—H17	118.9
C3—C4—C5	124.6 (3)	C18—C17—H17	118.9
C3—C4—S1	110.5 (2)	C19—C18—C17	119.6 (3)
C5—C4—S1	124.9 (2)	C19—C18—H18	120.2
C2—C1—S1	112.6 (2)	C17—C18—H18	120.2
C2—C1—H1	123.7	C21—C20—C19	120.4 (3)
S1—C1—H1	123.7	C21—C20—H20	119.8
C12—C13—C14	119.9 (3)	C19—C20—H20	119.8
C12—C13—H13	120.0	O2—C22—H22A	109.5
C14—C13—H13	120.0	O2—C22—H22B	109.5
C13—C12—C11	120.0 (3)	H22A—C22—H22B	109.5
C13—C12—H12	120.0	O2—C22—H22C	109.5
C11—C12—H12	120.0	H22A—C22—H22C	109.5
C1—C2—C3	112.9 (3)	H22B—C22—H22C	109.5

N3—N2—C7—O1	7.1 (4)	C4—C5—C6—N1	4.4 (5)
C8—N2—C7—O1	−180.0 (3)	C4—C5—C6—C7	−172.6 (2)
N3—N2—C7—C6	−170.7 (2)	C8—N1—C6—C5	−174.8 (3)
C8—N2—C7—C6	2.3 (3)	C8—N1—C6—C7	2.5 (3)
C6—N1—C8—N2	−1.0 (3)	O1—C7—C6—C5	−3.1 (5)
C6—N1—C8—C9	−179.0 (2)	N2—C7—C6—C5	174.5 (2)
N3—N2—C8—N1	172.4 (2)	O1—C7—C6—N1	179.5 (3)
C7—N2—C8—N1	−1.0 (3)	N2—C7—C6—N1	−2.9 (3)
N3—N2—C8—C9	−9.6 (4)	N2—N3—C15—C16	179.1 (2)
C7—N2—C8—C9	177.0 (2)	C17—C16—C15—N3	−172.5 (3)
C10—C9—C8—N1	−10.9 (4)	C21—C16—C15—N3	8.1 (5)
C14—C9—C8—N1	166.7 (2)	C22—O2—C19—C18	−0.8 (4)
C10—C9—C8—N2	171.4 (2)	C22—O2—C19—C20	178.1 (3)
C14—C9—C8—N2	−11.1 (4)	C17—C16—C21—C20	1.6 (5)
C8—N2—N3—C15	166.7 (2)	C15—C16—C21—C20	−179.0 (3)
C7—N2—N3—C15	−21.3 (4)	C5—C4—C3—C2	−178.0 (2)
C14—C9—C10—C11	0.6 (4)	S1—C4—C3—C2	−0.3 (3)
C8—C9—C10—C11	178.3 (2)	C1—C2—C3—C4	−0.1 (4)
C10—C9—C14—C13	−0.7 (4)	C13—C12—C11—C10	−0.5 (4)
C8—C9—C14—C13	−178.2 (2)	C9—C10—C11—C12	0.0 (4)
C6—C5—C4—C3	176.1 (3)	C21—C16—C17—C18	−2.4 (5)
C6—C5—C4—S1	−1.3 (4)	C15—C16—C17—C18	178.1 (3)
C1—S1—C4—C3	0.5 (2)	O2—C19—C18—C17	179.3 (3)
C1—S1—C4—C5	178.2 (2)	C20—C19—C18—C17	0.4 (5)
C4—S1—C1—C2	−0.6 (2)	C16—C17—C18—C19	1.5 (6)
C9—C14—C13—C12	0.2 (4)	C16—C21—C20—C19	0.2 (5)
C14—C13—C12—C11	0.4 (4)	C18—C19—C20—C21	−1.2 (5)
S1—C1—C2—C3	0.5 (3)	O2—C19—C20—C21	179.8 (3)

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Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10···N1	0.93	2.46	2.792 (4)	101
C14—H14···N3	0.93	2.32	2.942 (4)	124
C15—H15···O1	0.93	2.21	2.867 (3)	127

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(4Z)-2-Phenyl-1-{(E)-[4-(propan-2-yl)benzylidene]-amino}-4-[(thiophen-2-yl)methylidene]-1H-imidazol-5(4H)-one

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In the title molecule, $C_{24}H_{21}N_3OS$, the imidazole ring subtends dihedral angles of 4.6 (1) and 20.2 (1)° with the thiophene and iso-propylbenzene rings, respectively. The plane of the imidazole ring forms a dihedral angle of 39.9 (1)° with the phenyl ring. An intramolecular C—H⋯N hydrogen bond closes an $S(6)$ ring. In the crystal, pairs of C—H⋯O hydrogen bonds link molecules into inversion dimers featuring $R_2^2(10)$ graph-set motifs. Aromatic π–π stacking interactions are observed between the thiophene and imidazole rings [centroid–centroid distance = 3.570 (2) Å] and thiophene and benzene rings [centroid–centroid distance = 3.889 (2) Å]. Weak C—H⋯π interactions are also observed.

Structure description

Imidazolones are nitrogen analogues of azlactones bearing an exocyclic double bond at the fourth position, usually called unsaturated 2,4-disubstituted 2-imidazolin-5-ones. Compounds containing imidazolone as well as imine moieties exhibit a range of pharmaceutical activities, such as antimicrobial (Suthakaran *et al.* 2008; Patel *et al.* 2006), anti-oxidant (Suhasini *et al.*2014) and anticonvulsant activity (Mohamed *et al.* 2012).

In the title compound (Fig. 1), the imidazole ring subtends dihedral angles of 4.6 (1) and 20.2 (1)° with the thiophene and benzene rings, respectively. The dihedral angle between the phenyl and imidazole rings is 39.9 (1)°. The sum of bond angles around N2 is 358.3°, which confirms that the atom N2 is in an sp^2 hybridized state. The C7═O1 bond distance is 1.218 (5) Å which is somewhat longer than the standard value for a carbonyl

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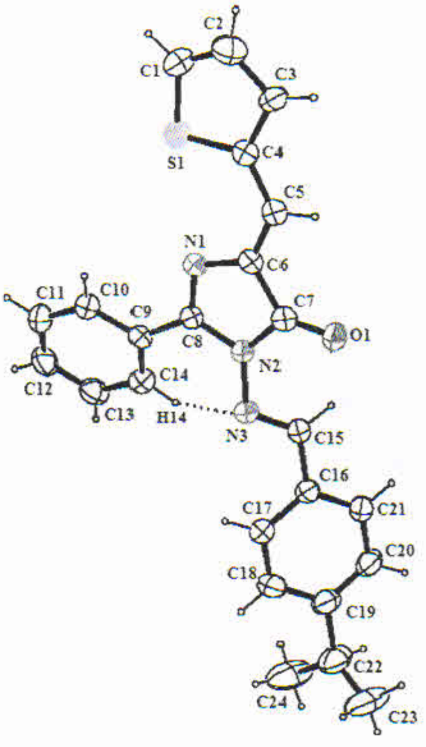

Figure 1
ORTEP view of the title molecule with displacement ellipsoids drawn at the 40% probability level. Dashed lines indicate the intramolecular hydrogen bond.

group (1.192 Å); the lengthening of this double bond may be due to the involvement of this oxygen atom as a hydrogen-bond acceptor.

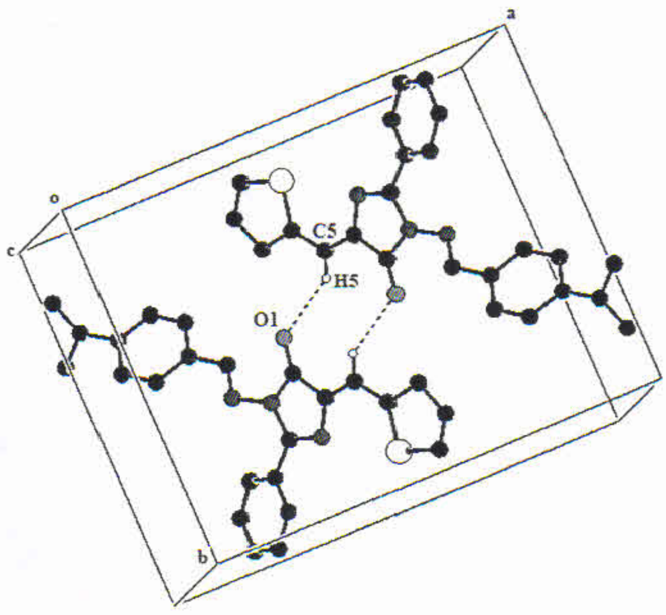

Figure 2
A dimer of molecules of the title compound linked by a pair of C—H⋯O hydrogen bonds forming an $R_2^2(10)$ loop.

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C14—H14⋯N3	0.93	2.38	2.960 (5)	120
C5—H5⋯O1^{i}	0.93	2.44	3.221 (5)	142

Symmetry code: (i) $-x+1, -y+2, -z+2$.

In the crystal, centrosymmetric dimeric aggregates (Fig. 2) are formed by pairs of C—H⋯O hydrogen bonds (Table 1), forming $R_2^2(10)$ ring motifs. π–π interactions are observed between the thiophene and imidazole rings [centroid separation = 3.570 (2) Å, interplanar spacing = 3.528 Å and centroid shift = 0.55 Å] and the thiophene and benzene rings [centroid separation= 3.889 (2) Å, interplanar spacing = 2.976 Å and centroid shift = 2.50 Å]. Weak C—H⋯π interactions are also observed.

Synthesis and crystallization

A mixture of 3-hydrazinyl-3-oxo-1-(thiophen-2-yl)prop-1-en-2-yl]benzamide (0.01 mol) in 2-propanol (30 ml) with 4-(propan-2-yl)benzaldehyde (0.01 mol) in the presence of one or two drops of sulfuric acid was heated under reflux for 8 h. The reaction mixture was then cooled to room temperature and poured on cold water; the solid mass obtained was

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{24}H_{21}N_3OS$
M_r	399.50
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, b, c (Å)	15.8392 (13), 12.9075 (10), 10.3997 (8)
β (°)	103.354 (8)
V (Å^3)	2068.7 (3)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.18
Crystal size (mm)	0.30 × 0.20 × 0.20
Data collection	
Diffractometer	Oxford Diffraction Xcalibur, Sapphire3
Absorption correction	Multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010
T_{min}, T_{max}	0.636, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8085, 4053, 2412
R_{int}	0.033
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.074, 0.247, 1.05
No. of reflections	4053
No. of parameters	264
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.43, −0.34

Computer programs: *CrysAlis PRO* (Oxford Diffraction,2010), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009).

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collected by filtration, washed with cold water and recrystallized at room temperature from a mixture of methanol and *N*,*N*-dimethyl formamide (1:1 *v*/*v*) giving yellow block-like crystals [m.p. 449 K]

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C—H distances of 0.93–0.96 A; and with $U_{\rm iso}(\rm H) = 1.2U_{\rm eq}(\rm C)$, except for the methyl groups where $U_{\rm iso}(\rm H) = 1.5U_{\rm eq}(\rm C)$.

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x160587 [doi:10.1107/S2414314616005873]

(4*Z*)-2-Phenyl-1-{(*E*)-[4-(propan-2-yl)benzylidene]amino}-4-[(thiophen-2-yl)methylidene]-1*H*-imidazol-5(4*H*)-one

Kamni, Vikram D. Singh, K. N. Subbulakshmi, B. Narayana, B. K. Sarojini, Sumati Anthal and Rajni Kant

(4*Z*)-2-Phenyl-1-{(*E*)-[4-(propan-2-yl)benzylidene]amino}-4-[(thiophen-2-yl)methylidene]-1*H*-imidazol-5(4*H*)-one

Crystal data

$C_{24}H_{21}N_3OS$
$M_r = 399.50$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 15.8392$ (13) Å
$b = 12.9075$ (10) Å
$c = 10.3997$ (8) Å
$\beta = 103.354$ (8)°
$V = 2068.7$ (3) Å^3
$Z = 4$
$F(000) = 840$
$D_x = 1.283$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1816 reflections
$\theta = 4.1$–25.6°
$\mu = 0.18$ mm^{-1}
$T = 293$ K
Block, yellow
0.30 × 0.20 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur, Sapphire3 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.1049 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010
$T_{min} = 0.636$, $T_{max} = 1.000$
8085 measured reflections
4053 independent reflections
2412 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 26.0°$, $\theta_{min} = 3.4°$
$h = -19 \rightarrow 19$
$k = -15 \rightarrow 15$
$l = -10 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.074$
$wR(F^2) = 0.247$
$S = 1.05$
4053 reflections
264 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1066P)^2 + 1.5103P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

Special details

Experimental. *CrysAlis PRO*, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27–08-2010 CrysAlis171. NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.48932 (8)	0.61536 (9)	0.93165 (13)	0.0810 (5)
N3	0.2515 (2)	0.9237 (3)	1.1960 (3)	0.0596 (8)
N1	0.37039 (19)	0.7338 (2)	1.0619 (3)	0.0565 (8)
N2	0.31414 (19)	0.8713 (2)	1.1460 (3)	0.0586 (8)
O1	0.3822 (2)	1.0076 (2)	1.0621 (3)	0.0852 (10)
C9	0.2618 (2)	0.6909 (3)	1.1846 (4)	0.0521 (9)
C17	0.1300 (3)	1.0434 (3)	1.2985 (4)	0.0661 (11)
H17	0.1079	0.9851	1.2497	0.079*
C8	0.3151 (2)	0.7636 (3)	1.1301 (4)	0.0535 (9)
C5	0.4681 (2)	0.8271 (3)	0.9533 (4)	0.0639 (10)
H5	0.4867	0.8930	0.9363	0.077*
C16	0.2157 (2)	1.0712 (3)	1.3090 (4)	0.0557 (9)
C6	0.4073 (2)	0.8230 (3)	1.0243 (4)	0.0601 (10)
C14	0.2251 (3)	0.7125 (3)	1.2905 (4)	0.0634 (10)
H14	0.2293	0.7789	1.3263	0.076*
C4	0.5077 (2)	0.7429 (3)	0.9007 (4)	0.0602 (10)
C15	0.2759 (3)	1.0096 (3)	1.2529 (4)	0.0606 (10)
H15	0.3322	1.0331	1.2588	0.073*
C19	0.1081 (3)	1.1886 (4)	1.4336 (5)	0.0773 (13)
C7	0.3709 (3)	0.9153 (3)	1.0755 (4)	0.0646 (11)
C13	0.1821 (3)	0.6353 (4)	1.3432 (4)	0.0736 (12)
H13	0.1591	0.6496	1.4158	0.088*
C10	0.2515 (3)	0.5916 (3)	1.1311 (5)	0.0702 (12)
H10	0.2744	0.5763	1.0587	0.084*
C21	0.2456 (3)	1.1606 (3)	1.3806 (4)	0.0687 (11)
H21	0.3025	1.1822	1.3874	0.082*
C18	0.0771 (3)	1.1004 (4)	1.3589 (5)	0.0765 (13)
H18	0.0197	1.0802	1.3502	0.092*
C3	0.5704 (3)	0.7537 (3)	0.8164 (4)	0.0612 (10)
H3	0.5913	0.8153	0.7894	0.073*
C20	0.1924 (3)	1.2173 (3)	1.4413 (5)	0.0796 (14)
H20	0.2140	1.2765	1.4886	0.096*
C1	0.5561 (3)	0.5752 (4)	0.8383 (5)	0.0809 (13)

H1	0.5669	0.5056	0.8249	0.097*
C11	0.2084 (3)	0.5161 (4)	1.1823 (5)	0.0862 (15)
H11	0.2028	0.4501	1.1455	0.103*
C2	0.5925 (3)	0.6525 (4)	0.7848 (5)	0.0877 (15)
H2	0.6302	0.6403	0.7300	0.105*
C22	0.0515 (4)	1.2478 (4)	1.5091 (7)	0.112 (2)
H22	0.0963	1.2742	1.5832	0.135*
C12	0.1736 (3)	0.5381 (4)	1.2884 (5)	0.0834 (14)
H12	0.1441	0.4870	1.3233	0.100*
C23	0.0029 (5)	1.1847 (5)	1.5790 (6)	0.137 (3)
H23A	−0.0227	1.2276	1.6352	0.206*
H23B	0.0408	1.1350	1.6318	0.206*
H23C	−0.0421	1.1492	1.5169	0.206*
C24	0.0168 (5)	1.3427 (5)	1.4508 (7)	0.138 (3)
H24A	−0.0288	1.3286	1.3744	0.207*
H24B	0.0617	1.3818	1.4251	0.207*
H24C	−0.0060	1.3818	1.5136	0.207*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0782 (8)	0.0620 (8)	0.1081 (10)	−0.0024 (6)	0.0322 (7)	−0.0085 (6)
N3	0.0539 (18)	0.0530 (19)	0.076 (2)	0.0012 (15)	0.0237 (16)	−0.0084 (16)
N1	0.0487 (17)	0.0492 (18)	0.074 (2)	−0.0024 (14)	0.0204 (16)	−0.0050 (16)
N2	0.0534 (18)	0.0491 (19)	0.079 (2)	−0.0051 (14)	0.0273 (16)	−0.0111 (16)
O1	0.085 (2)	0.0509 (19)	0.135 (3)	−0.0133 (15)	0.056 (2)	−0.0143 (18)
C9	0.0396 (18)	0.053 (2)	0.062 (2)	−0.0018 (16)	0.0080 (16)	−0.0053 (18)
C17	0.060 (2)	0.055 (2)	0.085 (3)	−0.0010 (19)	0.020 (2)	−0.009 (2)
C8	0.0459 (19)	0.051 (2)	0.063 (2)	−0.0013 (16)	0.0104 (17)	−0.0071 (18)
C5	0.056 (2)	0.054 (2)	0.085 (3)	−0.0081 (18)	0.025 (2)	−0.007 (2)
C16	0.057 (2)	0.048 (2)	0.065 (2)	0.0024 (17)	0.0205 (18)	0.0003 (18)
C6	0.048 (2)	0.057 (2)	0.077 (3)	−0.0055 (18)	0.0181 (19)	−0.010 (2)
C14	0.066 (2)	0.057 (2)	0.069 (2)	0.0005 (19)	0.019 (2)	−0.0049 (19)
C4	0.054 (2)	0.057 (2)	0.071 (2)	−0.0005 (18)	0.0173 (19)	−0.002 (2)
C15	0.055 (2)	0.052 (2)	0.080 (3)	−0.0061 (18)	0.025 (2)	−0.009 (2)
C19	0.089 (3)	0.057 (3)	0.099 (3)	0.016 (2)	0.049 (3)	0.010 (2)
C7	0.056 (2)	0.054 (3)	0.087 (3)	−0.0052 (18)	0.024 (2)	−0.009 (2)
C13	0.064 (3)	0.082 (3)	0.081 (3)	0.002 (2)	0.029 (2)	0.009 (3)
C10	0.065 (3)	0.060 (3)	0.089 (3)	−0.007 (2)	0.025 (2)	−0.014 (2)
C21	0.068 (3)	0.055 (2)	0.089 (3)	−0.008 (2)	0.031 (2)	−0.009 (2)
C18	0.057 (2)	0.072 (3)	0.107 (3)	0.005 (2)	0.033 (2)	0.004 (3)
C3	0.068 (2)	0.048 (2)	0.073 (2)	0.0073 (19)	0.026 (2)	−0.0071 (19)
C20	0.099 (4)	0.052 (3)	0.101 (3)	0.000 (2)	0.050 (3)	−0.010 (2)
C1	0.087 (3)	0.067 (3)	0.093 (3)	0.008 (3)	0.027 (3)	−0.017 (3)
C11	0.085 (3)	0.056 (3)	0.126 (4)	−0.017 (2)	0.044 (3)	−0.012 (3)
C2	0.085 (3)	0.101 (4)	0.085 (3)	0.005 (3)	0.037 (3)	0.000 (3)
C22	0.131 (5)	0.075 (4)	0.160 (5)	0.017 (3)	0.094 (5)	0.000 (4)
C12	0.068 (3)	0.072 (3)	0.115 (4)	−0.011 (2)	0.031 (3)	0.014 (3)

C23	0.188 (7)	0.120 (5)	0.145 (5)	0.066 (5)	0.123 (5)	0.049 (4)
C24	0.196 (7)	0.094 (4)	0.164 (6)	0.077 (5)	0.126 (6)	0.050 (4)

Geometric parameters (Å, °)

S1—C1	1.675 (5)	C19—C22	1.526 (6)
S1—C4	1.715 (4)	C13—C12	1.372 (7)
N3—C15	1.274 (5)	C13—H13	0.9300
N3—N2	1.397 (4)	C10—C11	1.366 (6)
N1—C8	1.306 (4)	C10—H10	0.9300
N1—C6	1.388 (5)	C21—C20	1.376 (6)
N2—C8	1.400 (5)	C21—H21	0.9300
N2—C7	1.405 (5)	C18—H18	0.9300
O1—C7	1.218 (5)	C3—C2	1.410 (6)
C9—C14	1.387 (5)	C3—H3	0.9300
C9—C10	1.392 (5)	C20—H20	0.9300
C9—C8	1.461 (5)	C1—C2	1.336 (7)
C17—C18	1.372 (6)	C1—H1	0.9300
C17—C16	1.384 (5)	C11—C12	1.372 (7)
C17—H17	0.9300	C11—H11	0.9300
C5—C6	1.343 (5)	C2—H2	0.9300
C5—C4	1.426 (6)	C22—C24	1.419 (7)
C5—H5	0.9300	C22—C23	1.429 (7)
C16—C21	1.395 (5)	C22—H22	0.9800
C16—C15	1.463 (5)	C12—H12	0.9300
C6—C7	1.476 (6)	C23—H23A	0.9600
C14—C13	1.389 (6)	C23—H23B	0.9600
C14—H14	0.9300	C23—H23C	0.9600
C4—C3	1.474 (5)	C24—H24A	0.9600
C15—H15	0.9300	C24—H24B	0.9600
C19—C20	1.369 (6)	C24—H24C	0.9600
C19—C18	1.402 (6)		
C1—S1—C4	91.8 (2)	C9—C10—H10	119.2
C15—N3—N2	115.4 (3)	C20—C21—C16	121.1 (4)
C8—N1—C6	106.8 (3)	C20—C21—H21	119.5
N3—N2—C8	123.5 (3)	C16—C21—H21	119.5
N3—N2—C7	126.4 (3)	C17—C18—C19	121.1 (4)
C8—N2—C7	108.4 (3)	C17—C18—H18	119.4
C14—C9—C10	118.1 (4)	C19—C18—H18	119.4
C14—C9—C8	124.3 (4)	C2—C3—C4	106.8 (4)
C10—C9—C8	117.5 (3)	C2—C3—H3	126.6
C18—C17—C16	121.1 (4)	C4—C3—H3	126.6
C18—C17—H17	119.5	C19—C20—C21	121.4 (4)
C16—C17—H17	119.5	C19—C20—H20	119.3
N1—C8—N2	112.5 (3)	C21—C20—H20	119.3
N1—C8—C9	122.8 (3)	C2—C1—S1	113.6 (4)
N2—C8—C9	124.7 (3)	C2—C1—H1	123.2

C6—C5—C4	128.0 (4)	S1—C1—H1	123.2
C6—C5—H5	116.0	C10—C11—C12	119.7 (5)
C4—C5—H5	116.0	C10—C11—H11	120.2
C17—C16—C21	117.6 (3)	C12—C11—H11	120.2
C17—C16—C15	123.0 (4)	C1—C2—C3	116.1 (4)
C21—C16—C15	119.4 (3)	C1—C2—H2	121.9
C5—C6—N1	126.2 (4)	C3—C2—H2	121.9
C5—C6—C7	123.8 (4)	C24—C22—C23	120.5 (5)
N1—C6—C7	110.0 (3)	C24—C22—C19	115.4 (5)
C9—C14—C13	120.1 (4)	C23—C22—C19	115.2 (5)
C9—C14—H14	119.9	C24—C22—H22	100.1
C13—C14—H14	119.9	C23—C22—H22	100.1
C5—C4—C3	124.9 (4)	C19—C22—H22	100.1
C5—C4—S1	123.4 (3)	C13—C12—C11	120.3 (4)
C3—C4—S1	111.6 (3)	C13—C12—H12	119.9
N3—C15—C16	120.2 (3)	C11—C12—H12	119.9
N3—C15—H15	119.9	C22—C23—H23A	109.5
C16—C15—H15	119.9	C22—C23—H23B	109.5
C20—C19—C18	117.7 (4)	H23A—C23—H23B	109.5
C20—C19—C22	121.0 (5)	C22—C23—H23C	109.5
C18—C19—C22	121.3 (5)	H23A—C23—H23C	109.5
O1—C7—N2	125.8 (4)	H23B—C23—H23C	109.5
O1—C7—C6	132.0 (4)	C22—C24—H24A	109.5
N2—C7—C6	102.2 (3)	C22—C24—H24B	109.5
C12—C13—C14	120.2 (4)	H24A—C24—H24B	109.5
C12—C13—H13	119.9	C22—C24—H24C	109.5
C14—C13—H13	119.9	H24A—C24—H24C	109.5
C11—C10—C9	121.6 (4)	H24B—C24—H24C	109.5
C11—C10—H10	119.2		

C15—N3—N2—C8	153.1 (4)	N3—N2—C7—C6	−168.3 (3)
C15—N3—N2—C7	−43.6 (5)	C8—N2—C7—C6	−2.9 (4)
C6—N1—C8—N2	−2.2 (4)	C5—C6—C7—O1	3.8 (8)
C6—N1—C8—C9	178.7 (3)	N1—C6—C7—O1	−176.9 (5)
N3—N2—C8—N1	169.3 (3)	C5—C6—C7—N2	−177.6 (4)
C7—N2—C8—N1	3.4 (4)	N1—C6—C7—N2	1.7 (4)
N3—N2—C8—C9	−11.6 (6)	C9—C14—C13—C12	−1.9 (6)
C7—N2—C8—C9	−177.6 (4)	C14—C9—C10—C11	−1.6 (6)
C14—C9—C8—N1	157.6 (4)	C8—C9—C10—C11	174.9 (4)
C10—C9—C8—N1	−18.7 (5)	C17—C16—C21—C20	1.6 (6)
C14—C9—C8—N2	−21.4 (6)	C15—C16—C21—C20	−176.2 (4)
C10—C9—C8—N2	162.3 (4)	C16—C17—C18—C19	−0.2 (7)
C18—C17—C16—C21	−1.5 (6)	C20—C19—C18—C17	1.7 (7)
C18—C17—C16—C15	176.3 (4)	C22—C19—C18—C17	−175.6 (5)
C4—C5—C6—N1	0.3 (7)	C5—C4—C3—C2	−179.2 (4)
C4—C5—C6—C7	179.4 (4)	S1—C4—C3—C2	2.1 (4)
C8—N1—C6—C5	179.5 (4)	C18—C19—C20—C21	−1.5 (7)
C8—N1—C6—C7	0.3 (4)	C22—C19—C20—C21	175.8 (5)

C10—C9—C14—C13	2.2 (6)	C16—C21—C20—C19	−0.2 (7)
C8—C9—C14—C13	−174.1 (4)	C4—S1—C1—C2	0.4 (4)
C6—C5—C4—C3	176.0 (4)	C9—C10—C11—C12	0.6 (7)
C6—C5—C4—S1	−5.4 (6)	S1—C1—C2—C3	0.9 (6)
C1—S1—C4—C5	179.8 (4)	C4—C3—C2—C1	−1.9 (6)
C1—S1—C4—C3	−1.4 (3)	C20—C19—C22—C24	78.6 (8)
N2—N3—C15—C16	180.0 (3)	C18—C19—C22—C24	−104.2 (7)
C17—C16—C15—N3	−3.1 (6)	C20—C19—C22—C23	−133.9 (6)
C21—C16—C15—N3	174.6 (4)	C18—C19—C22—C23	43.3 (8)
N3—N2—C7—O1	10.4 (7)	C14—C13—C12—C11	0.9 (7)
C8—N2—C7—O1	175.8 (4)	C10—C11—C12—C13	−0.2 (8)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14···N3	0.93	2.38	2.960 (5)	120
C5—H5···O1^{i}	0.93	2.44	3.221 (5)	142
C3—H3···O1^{i}	0.93	2.74	3.349 (5)	124

Symmetry code: (i) $-x+1, -y+2, -z+2$.

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Synthesis and Crystal Structure of a Chalcone Derivative[1]

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Abstract—(2E)-3-(anthrance-9-yl)-1-(3,4-dichlorophenyl)prop-2-en-1-one $[C_{23}H_{14}OCl_2]$ is synthesized and its crystal structure is determined by single X-ray diffraction. There exist two molecules in the asymmetric unit. The dihedral angle between the benzene and anthracene moiety of the molecule *A* and *B* is 86.51(12)° and 76.42(13)°, respectively. No classical hydrogen bonds are observed and only van der Waals forces stabilize the crystal packing.

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INTRODUCTION

Chalcone derivatives are reported to possess a broad spectrum of biological properties such as anticancer [1, 2], antimalarial [3, 4], anti-inflammatory [5, 6], antioxidant [7, 8], antimicrobial [9, 10], antifilarial [11], antifungal [12, 13], larvicidal [14], and anticonvulsant [15] activities. Apart from being biologically important compounds, chalcone derivatives also show non-liner optical (NLO) properties [16, 17]. Chalcones have also been studied for their wide range of applications including laser activity [18] and fluorescence properties [19]. As a part of our studies on chalcone derivatives [20–22], we report the synthesis and crystal structure of a new compound, (2*E*)-3-(anthrance-9-yl)-1-(3,4-dichlorophenyl)prop-2-en-1-one.

EXPERIMENTAL

Synthesis

To the mixture of 9-anthraldehyde (0.01 mol) and 3,4-dichloroacetophenone (0.01 mol) in ethanol (50 mL), 15 mL of 10% sodium hydroxide solution was added and stirred at 0–5°C for 3 h. The precipitate formed was collected by filtration and purified by recrystallization from ethanol. Single crystal was grown from mixture of absolute alcohol and DMF by slow evaporation (m. p. 407–409 K). Scheme showing the synthesis of title compound is shown in Fig. 1.

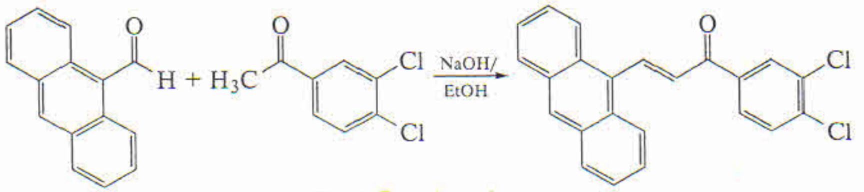

Fig. 1. Reaction scheme.

Crystal Structure Determination

Crystallographic characteristics and the X-ray-data collection and structure-refinement parameters are given in Table 1. The structure was solved by direct methods. Multisolution tangent refinement was used. All non-hydrogen atoms of the molecule were located in the best *E*-map and refined in anisotropic approximation. All the hydrogen atoms were geometrically

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[1] The article is published in the original.

Table 1. Crystallographic characteristics and the X-ray-data collection and structure-refinement parameters for $C_{23}H_{14}Cl_2O$

Crystal system, sp. gr., Z	Monoclinic, $P2_1$, 4
a, b, c, Å	8.7235(7), 14.4981(13), 14.1480(12)
β, deg	95.239(6)
V, Å^3	1781.9(3)
D_x, g cm^{-3}	1.370
Radiation, λ, Å	MoK_α, 0.71073
μ, mm^{-1}	0.380
T, K	293(2)
Sample size, mm	0.3 × 0.2 × 0.1
Diffractometer	X' calibur CCD
Scan mode	ω
Absorption correction, $T_{\min}$, $T_{\max}$	Multi-scan, 0.808, 1.000
$\theta_{\max}$, deg	26.00
h, k, l ranges	$-10 \le h \le 10$, $-17 \le k \le 15$, $-15 \le l \le 17$
Number of reflections: measured/unique ($N1$), $R_{\rm int}$/with $I > 2\sigma(I)$ ($N2$)	7074/5129, 0.023/4029
Refinement method	Full_matrix least_squares on F^2
Number of refined parameters	469
$wR2$ relative to $N1$	0.1396
$R1$ relative to $N2$	0.055
S	1.044
$\Delta\rho_{\max}/\Delta\rho_{\min}$, e/Å^3	0.639/−0.334
Programs	SHELX97 [23], ORTEP [24], PARST [25], PLATON [26]

Table 2. Selected bond distances d (Å) and bond angles ω (deg)

Bond	d	Bond angle	ω
O1*A*–C17*A*	1.215(5)	C11*A*–C12*A*–C13*A*	123.6(4)
O1*B*–C17*B*	1.232(6)	C11*B*–C12*B*–C13*B*	123.7(4)
C15*A*–C16*A*	1.292(7)	C16*A*–C15*A*–C11*A*	126.2(4)
C15*B*–C16*B*	1.324(7)	C16*B*–C15*B*–C11*B*	127.2(4)
C18*A*–C23*A*	1.385(7)	C16*A*–C17*A*–C18*A*	117.5(4)
C18*B*–C23*B*	1.389(7)	C16*B*–C17*B*–C18*B*	119.7(4)
C20*A*–C21*A*	1.378(7)	C23*A*–C18*A*–C17*A*	121.8(5)

fixed and allowed to ride on the corresponding non-H atoms with C–H = 0.93 Å, and $U_{\rm iso}$(H) = 1.5 $U_{\rm eq}$ of the attached C atoms for methyl groups and 1.2 $U_{\rm eq}$ for other H atoms. Crystallographic data have been deposited at Cambridge Crystallographic Data Centre, CCDC number 1493090. These data can be obtained free of charge from Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

RESULTS AND DISCUSSION

The structure contains two molecules in the asymmetric unit. Bond distances and bond angles of both the molecules (*A* and *B*) lie within the normal ranges [27]. The double bonds C17*A* = O1*A* and C17*B* = O1*B* are confirmed by their distances of 1.215(5) and 1.232(6) Å, respectively. These values are also consistent with the corresponding ones observed in some related structures [28, 29]. The anthracene ring system (C1*A*–C14*A*/C1*B*–C14*B*) is twisted at the (C11–C15) bond from the acrylaldehyde moiety [maximal deviation is −0.128(3) Å and −0.162(3) Å for atom O1 (*A* and *B*, respectively)] with a C10–C11–C15–C16 torsion angle of −52.9(7)° for molecule A and −129.7(5)° for molecule B. The terminal benzene [C18*A*–C23*A*/C18*B*–C23*B*] and anthracene ring systems form a dihedral angle of 86.51(12)° and 76.42(13)°, respectively, while it is −61.4(2)° in the crystal structure of (*E*)-1-(anthracen-9-yl)-3-(2-chloro-6-fluorophenyl)-prop-2-en-1-one [27]. The anthracene moiety is almost planar in both the asymmetric molecules with maximum deviation of −0.081(5) Å [C11*A*]

Table 3. Torsion angle τ (deg)

Torsion angle	τ
C12*A*–C11*A*–C15*A*–C16*A*	123.6(5)
C12*B*–C11*B*–C15*A*–C16*A*	46.7(7)
C16*A*–C17*A*–C18*A*–C23*A*	−26.0(6)
C16*B*–C17*B*–C18*B*–C23*B*	18.6(7)
C10*A*–C11*A*–C15*A*–C16*A*	−52.9(7)
C10*B*–C11*B*–C15*B*–C16*B*	−129.7(5)
C15*A*–C16*A*–C17*A*–C18*A*	155.3(5)
C15*B*–C16*B*–C17*B*–C18*B*	−161.7(4)
C18*A*–C19*A*–C20*A*–Cl1	178.2(3)
C18*B*–C19*B*–C20*B*–Cl3	−179.1(3)
C23*A*–C22*A*–C21*A*–Cl2	178.1(4)
C23*B*–C22*B*–C21*B*–Cl4	−179.1(4)

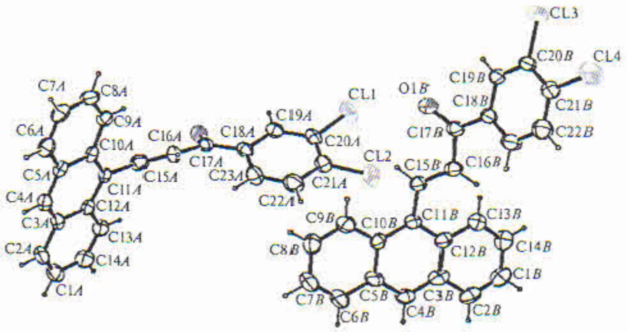

Fig. 2. ORTEP view of the molecules with displacement ellipsoids drawn at 50% probability level.

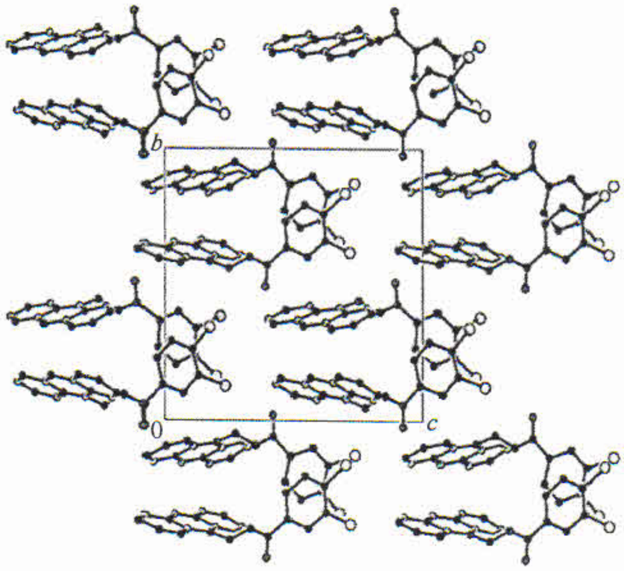

Fig. 3. The crystal packing viewed down the *a*-axis.

and −0.076(5) Å [C11*B*], respectively. No classical hydrogen bonds were observed. The crystal packing is stabilized only by van der Waals forces.

CONCLUSIONS

Synthesis of the compound was achieved by reaction of 9-anthraldehyde (0.01 mol) and 3,4-dichloroacetophenone. Single crystal was grown by slow evaporation from the mixture of absolute alcohol and DMF. The molecular and crystal structure of the compound was characterized by single crystal X-ray diffraction. Crystal packing is stabilized by weak van der waals interactions.

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3-[6-(4-Methoxyphenyl)-2-methylpyrimidin-4-yl]-2-(4-methylpiperazin-1-yl)quinoline

Vikram. D. Singh, Sumati Anthal, N. R. Desai, D. B. Arunakumar, S. Sreenivasa, Kamni and Rajni Kant

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3-[6-(4-Methoxyphenyl)-2-methylpyrimidin-4-yl]-2-(4-methylpiperazin-1-yl)quinoline

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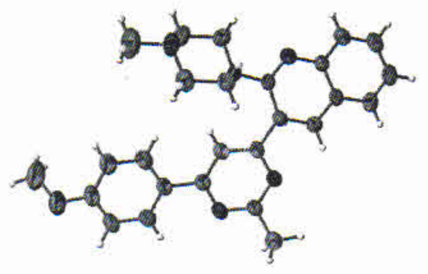
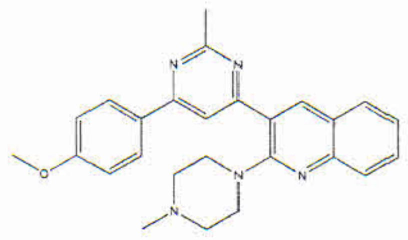
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{26}H_{27}N_5O$, the piperazine ring adopts a chair conformation. The pyrimidine ring makes a dihedral angle of 1.5 (1)° with the methoxyphenyl ring and 33.1 (1)° with the quinoline ring system. In the crystal, molecules are consolidated in the crystal packing by weak C—H$\cdots\pi$ interactions and π–π stacking interactions.

3D view



Chemical scheme



Structure description

Quinoline derivatives are important owing to their wide occurrence in natural products and in biologically active compounds (Markees *et al.*, 1970; Campbell *et al.*, 1988; Kalluraya & Sreenivasa, 1998). The pyrimidine nucleus also plays an important role in the medicinal chemistry and is reported to possess a broad spectrum of biological activities such as antimicrobial, anti-inflammatory, anthelmintic, anti HIV, antitubercular properties (Prasad *et al.*, 2008; Vaidya & Mathias 2005; Virsodia *et al.*, 2008). In a continuation of structural studies of these derivatives (Sharma *et al.*, 2017; Kaiser *et al.*, 2009; Prasath *et al.*, 2010) the title compound was investigated.

In the title molecule (Fig. 1), bond lengths are comparable with those in related structures (Prasath *et al.*, 2010, 2011; Sharma *et al.*, 2017). The piperazine ring adopts a chair conformation with best mirror plane passing through atoms N4 and N5 [asymmetry parameter C_s(N4) = 1.03] and the best twofold rotational axis bisecting the N4—C25 and N5—C23 bonds [asymmetry parameter C_2(N5—C25) = 1.27; Duax & Norton, 1975]. The quinoline ring system is essentially planar with a maximum deviation of 0.0583 (1) Å for atom C21. The methoxyphenyl ring makes a dihedral angle of 1.5 (1)° with the pyrimi-

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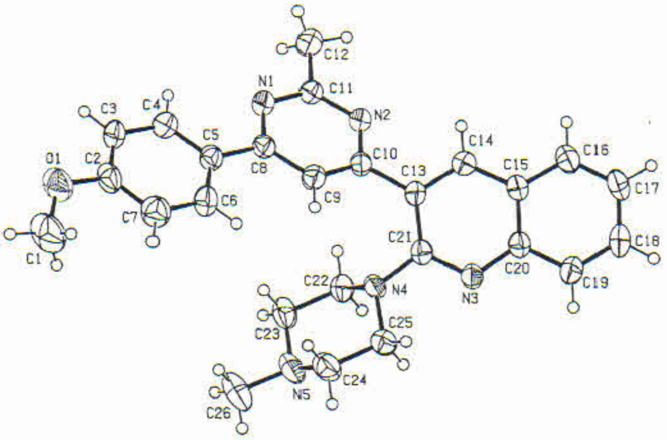

Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 40% probability level. H atoms are shown as small spheres of arbitrary radii.

dine ring while the dihedral angle between the pyrimidine ring and quinoline ring system is 33.1 (1)°.

In the crystal, π–π stacking interactions are observed between the pyrimidine ring and methoxyphenyl ring [centroid–centroid separation = 3.628 (1) Å, interplanar spacing = 3.588 Å and centroid shift = 0.54 Å; symmetry code: $1 - x, -y, -z$]. Weak C—H$\cdots\pi$ interactions are also observed (Table 1). The above interaction combine to assemble the molecules into a three-dimensional network (Fig. 2).

Synthesis and crystallization

(2*E*)-3-[2-(4-Methylpiperazin-1-yl)quinolin-3-yl]-1-(4-methoxyphenyl)prop-2-en-1-one (1 g, 0.0026 mmol), acetamidine hydrochloride (243 mg, 0.0026 mmol) and NaOH (309 mg, 0.0077 mmol) were refluxed for 12 h in ethanol. The progress of reaction was monitored by thin layer chromatography. After the completion of reaction, the reaction mixture was poured into water and extracted with ethyl acetate. The organic layer was washed with water, dried and concentrated under vacuum using a rotary evaporator. The compound was purified by column chromatography by using 60–120 mesh silica gel and it was recrystallized from an ethyl acetate–petroleum ether mixture (7:3 *v*:*V*).

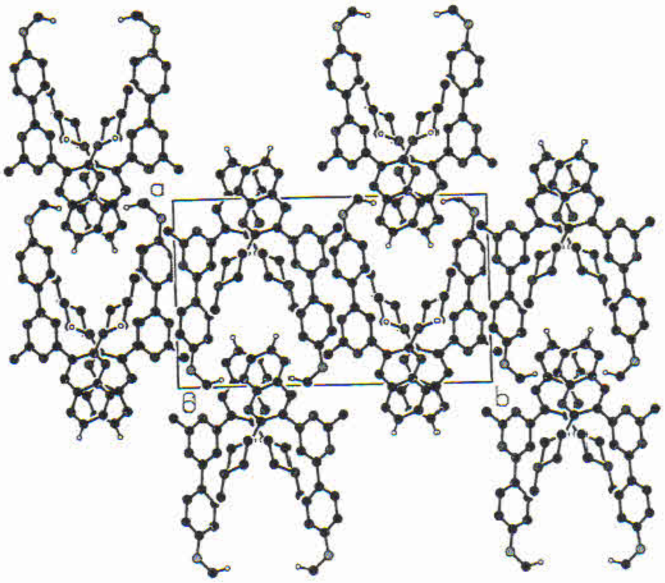

Figure 2
Part of the crystal structure. Only H atoms involved in the C—H$\cdots\pi$ interactions are shown.

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1, *Cg*2 and *Cg*3 are the centroids of the C15–C20, C8–C10/N2/C11/N2 and C2–C7 rings, respectively.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C1—H1$A\cdots Cg1^{i}$	0.96	3.10	3.867 (2)	138
C25—H25$A\cdots Cg2^{ii}$	0.97	2.96	3.706 (2)	134
C17—H17$\cdots Cg3^{iii}$	0.93	3.08	3.759 (2)	131

Symmetry codes: (i) $x - 1, y, z - 1$; (ii) $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (iii) $x + 1, y, z + 1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{26}H_{27}N_5O$
M_r	425.52
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, b, c (Å)	11.9896 (9), 18.4215 (13), 10.9903 (10)
β (°)	110.153 (9)
V (Å^3)	2278.8 (3)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.30 × 0.20 × 0.20
Data collection	
Diffractometer	Oxford Diffraction Xcalibur Sapphire3
Absorption correction	Multi-scan (*CrysAlis RED*; Oxford Diffraction, 2010)
$T_{\min}$, $T_{\max}$	0.776, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9015, 4461, 2193
$R_{\rm int}$	0.045
$(\sin \theta/\lambda)_{\max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.064, 0.165, 1.00
No. of reflections	4461
No. of parameters	293
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å^{-3})	0.18, −0.17

Computer programs: *CrysAlis PRO* (Oxford Diffraction, 2010), *SHELXS97* (Sheldrick, 2008), *SHELXL2016* (Sheldrick, 2015) and *PLATON* (Spek,2009).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2018). **3**, x180030 [https://doi.org/10.1107/S2414314618000305]

3-[6-(4-Methoxyphenyl)-2-methylpyrimidin-4-yl]-2-(4-methylpiperazin-1-yl)quinoline

Vikram. D. Singh, Sumati Anthal, N. R. Desai, D. B. Arunakumar, S. Sreenivasa, Kamni and Rajni Kant

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Crystal data

$C_{26}H_{27}N_5O$
$M_r = 425.52$
Monoclinic, $P2_1/c$
$a = 11.9896$ (9) Å
$b = 18.4215$ (13) Å
$c = 10.9903$ (10) Å
$\beta = 110.153$ (9)°
$V = 2278.8$ (3) Å^3
$Z = 4$
$F(000) = 904$
$D_x = 1.240$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1944 reflections
$\theta = 3.9$–27.5°
$\mu = 0.08$ mm^{-1}
$T = 293$ K
Block, white
0.30 × 0.20 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 6.1049 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2010)
$T_{\min} = 0.776$, $T_{\max} = 1.000$
9015 measured reflections
4461 independent reflections
2193 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.045$
$\theta_{\max} = 26.0°$, $\theta_{\min} = 3.6°$
$h = -14 \rightarrow 10$
$k = -22 \rightarrow 21$
$l = -13 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.165$
$S = 1.00$
4461 reflections
293 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0527P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}
Extinction correction: SHELXL2016 (Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0065 (11)

full crystallographic data

IUCrData (2018). **3**, x180030 [https://doi.org/10.1107/S2414314618000305]

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Extinction coefficient: 0.0065 (11)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. All H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C—H distances of 0.93–0.97 Å; and with U_{iso}(H) = 1.2U_{eq}(C), except for the methyl groups where U_{iso}(H) = 1.5U_{eq}(C).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
N3	0.1267 (2)	0.72304 (11)	0.7034 (2)	0.0458 (6)
N2	0.16616 (19)	0.92591 (10)	0.9859 (2)	0.0472 (6)
N4	0.29090 (19)	0.72812 (11)	0.8926 (2)	0.0441 (6)
N1	0.3478 (2)	0.97474 (11)	1.1300 (2)	0.0499 (6)
C15	−0.0198 (2)	0.81946 (14)	0.6399 (3)	0.0459 (7)
C13	0.1555 (2)	0.83286 (13)	0.8309 (3)	0.0425 (7)
C5	0.5412 (3)	0.93685 (14)	1.1382 (3)	0.0484 (7)
C11	0.2301 (2)	0.97043 (13)	1.0800 (3)	0.0477 (7)
C10	0.2289 (2)	0.88089 (13)	0.9372 (3)	0.0429 (7)
C8	0.4107 (2)	0.93117 (13)	1.0799 (3)	0.0446 (7)
C21	0.1884 (2)	0.76059 (13)	0.8064 (3)	0.0404 (6)
C14	0.0510 (3)	0.85949 (14)	0.7484 (3)	0.0498 (7)
H14	0.025655	0.905167	0.763886	0.060*
C20	0.0242 (2)	0.75182 (14)	0.6186 (3)	0.0438 (7)
O1	0.9073 (2)	0.95927 (12)	1.3186 (3)	0.0891 (8)
C9	0.3520 (3)	0.88340 (13)	0.9812 (3)	0.0476 (7)
H9	0.394618	0.853403	0.945043	0.057*
C4	0.5930 (3)	0.98535 (14)	1.2376 (3)	0.0596 (9)
H4	0.544180	1.014562	1.266880	0.072*
C22	0.2925 (2)	0.71665 (14)	1.0246 (3)	0.0502 (8)
H22A	0.240969	0.676301	1.025978	0.060*
H22B	0.262964	0.759659	1.054419	0.060*
C19	−0.0409 (3)	0.71172 (16)	0.5084 (3)	0.0582 (8)
H19	−0.011218	0.667858	0.490842	0.070*
N5	0.4637 (2)	0.63727 (13)	1.0676 (3)	0.0655 (7)
C25	0.3320 (3)	0.66256 (14)	0.8466 (3)	0.0590 (8)
H25A	0.329712	0.669435	0.758258	0.071*
H25B	0.280934	0.621952	0.847932	0.071*
C2	0.7879 (3)	0.94946 (17)	1.2531 (3)	0.0628 (9)
C16	−0.1296 (3)	0.84345 (16)	0.5544 (3)	0.0603 (8)
H16	−0.159657	0.887878	0.568982	0.072*
C18	−0.1473 (3)	0.73667 (17)	0.4274 (3)	0.0667 (9)
H18	−0.190151	0.709160	0.355491	0.080*
C3	0.7132 (3)	0.99188 (15)	1.2944 (3)	0.0655 (9)
H3	0.744853	1.025074	1.361203	0.079*
C7	0.7395 (3)	0.90055 (17)	1.1532 (3)	0.0651 (9)
H7	0.788756	0.871573	1.124200	0.078*

C17	−0.1932 (3)	0.80266 (17)	0.4500 (3)	0.0657 (9)
H17	−0.266549	0.818794	0.394481	0.079*
C6	0.6171 (3)	0.89510 (15)	1.0968 (3)	0.0608 (8)
H6	0.585050	0.862413	1.029274	0.073*
C24	0.4585 (3)	0.64721 (17)	0.9351 (3)	0.0660 (9)
H24A	0.487508	0.603801	0.905819	0.079*
H24B	0.509439	0.687366	0.931018	0.079*
C23	0.4171 (3)	0.70077 (16)	1.1133 (3)	0.0633 (9)
H23A	0.467516	0.742353	1.115965	0.076*
H23B	0.417257	0.692039	1.200358	0.076*
C12	0.1623 (3)	1.02123 (15)	1.1351 (3)	0.0710 (10)
H12A	0.178956	1.010145	1.224980	0.106*
H12B	0.078716	1.015809	1.088726	0.106*
H12C	0.185668	1.070310	1.126857	0.106*
C26	0.5859 (3)	0.6232 (2)	1.1512 (4)	0.0978 (13)
H26A	0.614351	0.580052	1.122464	0.147*
H26B	0.588714	0.616714	1.238847	0.147*
H26C	0.635150	0.663593	1.147165	0.147*
C1	0.9855 (3)	0.9098 (2)	1.2957 (4)	0.1088 (15)
H1A	0.963790	0.861485	1.311432	0.163*
H1B	1.064890	0.919991	1.352491	0.163*
H1C	0.981739	0.913863	1.207268	0.163*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N3	0.0408 (15)	0.0517 (13)	0.0405 (14)	0.0014 (11)	0.0086 (12)	−0.0049 (12)
N2	0.0432 (15)	0.0419 (12)	0.0480 (15)	−0.0009 (11)	0.0049 (13)	−0.0035 (11)
N4	0.0398 (15)	0.0507 (13)	0.0366 (13)	0.0098 (10)	0.0067 (12)	0.0003 (11)
N1	0.0438 (15)	0.0470 (13)	0.0503 (15)	−0.0057 (11)	0.0053 (13)	−0.0045 (12)
C15	0.0377 (17)	0.0517 (16)	0.0431 (17)	0.0011 (13)	0.0071 (15)	0.0009 (14)
C13	0.0352 (16)	0.0453 (15)	0.0420 (17)	−0.0023 (12)	0.0069 (14)	−0.0034 (13)
C5	0.0416 (18)	0.0468 (16)	0.0515 (19)	−0.0065 (13)	0.0091 (16)	0.0027 (15)
C11	0.0429 (18)	0.0408 (15)	0.0520 (19)	−0.0039 (13)	0.0070 (16)	−0.0004 (14)
C10	0.0395 (18)	0.0440 (15)	0.0401 (17)	−0.0028 (13)	0.0072 (15)	0.0010 (13)
C8	0.0463 (19)	0.0405 (15)	0.0411 (16)	−0.0022 (13)	0.0074 (15)	0.0050 (13)
C21	0.0367 (16)	0.0480 (15)	0.0363 (16)	−0.0003 (12)	0.0123 (14)	−0.0005 (13)
C14	0.0494 (19)	0.0477 (15)	0.0474 (18)	0.0026 (14)	0.0103 (16)	−0.0046 (15)
C20	0.0369 (17)	0.0514 (16)	0.0412 (17)	−0.0012 (13)	0.0111 (14)	−0.0017 (13)
O1	0.0451 (15)	0.0878 (16)	0.120 (2)	−0.0029 (12)	0.0106 (16)	0.0083 (16)
C9	0.0446 (19)	0.0483 (16)	0.0474 (18)	−0.0039 (13)	0.0128 (16)	−0.0034 (14)
C4	0.052 (2)	0.0491 (17)	0.068 (2)	−0.0036 (15)	0.0075 (18)	−0.0041 (16)
C22	0.049 (2)	0.0590 (17)	0.0398 (17)	−0.0035 (14)	0.0116 (16)	0.0015 (14)
C19	0.054 (2)	0.0626 (18)	0.048 (2)	−0.0024 (15)	0.0058 (17)	−0.0125 (16)
N5	0.0454 (17)	0.0750 (16)	0.0623 (18)	0.0086 (13)	0.0009 (15)	0.0191 (15)
C25	0.055 (2)	0.0588 (17)	0.058 (2)	0.0109 (15)	0.0121 (18)	−0.0059 (16)
C2	0.042 (2)	0.064 (2)	0.072 (2)	−0.0084 (16)	0.0058 (19)	0.0144 (18)
C16	0.048 (2)	0.0679 (19)	0.055 (2)	0.0085 (16)	0.0059 (18)	−0.0003 (16)

C18	0.055 (2)	0.081 (2)	0.048 (2)	−0.0054 (18)	−0.0043 (18)	−0.0075 (18)
C3	0.048 (2)	0.0594 (19)	0.070 (2)	−0.0101 (16)	−0.0034 (19)	−0.0057 (17)
C7	0.047 (2)	0.078 (2)	0.069 (2)	−0.0017 (17)	0.018 (2)	−0.0049 (19)
C17	0.049 (2)	0.078 (2)	0.053 (2)	0.0070 (17)	−0.0042 (17)	0.0073 (18)
C6	0.047 (2)	0.073 (2)	0.056 (2)	−0.0067 (16)	0.0108 (18)	−0.0074 (17)
C24	0.054 (2)	0.074 (2)	0.066 (2)	0.0158 (16)	0.0149 (19)	0.0020 (18)
C23	0.058 (2)	0.076 (2)	0.0449 (19)	0.0001 (17)	0.0038 (17)	0.0116 (16)
C12	0.053 (2)	0.0680 (19)	0.078 (3)	0.0034 (16)	0.0060 (19)	−0.0248 (18)
C26	0.063 (3)	0.125 (3)	0.085 (3)	0.019 (2)	−0.001 (2)	0.032 (2)
C1	0.052 (3)	0.127 (3)	0.146 (4)	0.009 (2)	0.031 (3)	0.011 (3)

Geometric parameters (Å, º)

N3—C21	1.315 (3)	C19—H19	0.9300
N3—C20	1.368 (3)	N5—C24	1.448 (4)
N2—C11	1.336 (3)	N5—C23	1.458 (4)
N2—C10	1.348 (3)	N5—C26	1.460 (4)
N4—C21	1.402 (3)	C25—C24	1.520 (4)
N4—C22	1.459 (3)	C25—H25A	0.9700
N4—C25	1.459 (3)	C25—H25B	0.9700
N1—C11	1.328 (3)	C2—C3	1.378 (4)
N1—C8	1.343 (3)	C2—C7	1.383 (4)
C15—C16	1.400 (4)	C16—C17	1.363 (4)
C15—C20	1.404 (3)	C16—H16	0.9300
C15—C14	1.410 (4)	C18—C17	1.392 (4)
C13—C14	1.360 (3)	C18—H18	0.9300
C13—C21	1.440 (3)	C3—H3	0.9300
C13—C10	1.488 (3)	C7—C6	1.387 (4)
C5—C6	1.382 (4)	C7—H7	0.9300
C5—C4	1.382 (4)	C17—H17	0.9300
C5—C8	1.477 (4)	C6—H6	0.9300
C11—C12	1.498 (4)	C24—H24A	0.9700
C10—C9	1.386 (3)	C24—H24B	0.9700
C8—C9	1.385 (3)	C23—H23A	0.9700
C14—H14	0.9300	C23—H23B	0.9700
C20—C19	1.404 (4)	C12—H12A	0.9600
O1—C2	1.376 (3)	C12—H12B	0.9600
O1—C1	1.391 (4)	C12—H12C	0.9600
C9—H9	0.9300	C26—H26A	0.9600
C4—C3	1.364 (4)	C26—H26B	0.9600
C4—H4	0.9300	C26—H26C	0.9600
C22—C23	1.504 (4)	C1—H1A	0.9600
C22—H22A	0.9700	C1—H1B	0.9600
C22—H22B	0.9700	C1—H1C	0.9600
C19—C18	1.360 (4)		
C21—N3—C20	119.3 (2)	N4—C25—H25B	110.0
C11—N2—C10	115.8 (2)	C24—C25—H25B	110.0

C21—N4—C22	116.2 (2)	H25A—C25—H25B	108.4
C21—N4—C25	116.2 (2)	O1—C2—C3	115.5 (3)
C22—N4—C25	109.5 (2)	O1—C2—C7	125.2 (3)
C11—N1—C8	117.5 (2)	C3—C2—C7	119.3 (3)
C16—C15—C20	119.3 (3)	C17—C16—C15	121.0 (3)
C16—C15—C14	123.8 (3)	C17—C16—H16	119.5
C20—C15—C14	117.0 (3)	C15—C16—H16	119.5
C14—C13—C21	117.2 (2)	C19—C18—C17	121.1 (3)
C14—C13—C10	118.0 (2)	C19—C18—H18	119.4
C21—C13—C10	124.8 (2)	C17—C18—H18	119.4
C6—C5—C4	116.9 (3)	C4—C3—C2	120.2 (3)
C6—C5—C8	122.5 (3)	C4—C3—H3	119.9
C4—C5—C8	120.6 (3)	C2—C3—H3	119.9
N1—C11—N2	127.0 (3)	C2—C7—C6	119.4 (3)
N1—C11—C12	116.3 (2)	C2—C7—H7	120.3
N2—C11—C12	116.7 (3)	C6—C7—H7	120.3
N2—C10—C9	120.9 (2)	C16—C17—C18	119.4 (3)
N2—C10—C13	114.7 (2)	C16—C17—H17	120.3
C9—C10—C13	124.4 (2)	C18—C17—H17	120.3
N1—C8—C9	119.7 (3)	C5—C6—C7	121.9 (3)
N1—C8—C5	116.1 (2)	C5—C6—H6	119.1
C9—C8—C5	124.2 (3)	C7—C6—H6	119.1
N3—C21—N4	117.4 (2)	N5—C24—C25	110.8 (2)
N3—C21—C13	122.6 (3)	N5—C24—H24A	109.5
N4—C21—C13	120.1 (2)	C25—C24—H24A	109.5
C13—C14—C15	121.5 (3)	N5—C24—H24B	109.5
C13—C14—H14	119.2	C25—C24—H24B	109.5
C15—C14—H14	119.2	H24A—C24—H24B	108.1
N3—C20—C19	119.1 (2)	N5—C23—C22	110.2 (2)
N3—C20—C15	122.1 (3)	N5—C23—H23A	109.6
C19—C20—C15	118.8 (3)	C22—C23—H23A	109.6
C2—O1—C1	117.6 (3)	N5—C23—H23B	109.6
C8—C9—C10	119.1 (3)	C22—C23—H23B	109.6
C8—C9—H9	120.4	H23A—C23—H23B	108.1
C10—C9—H9	120.4	C11—C12—H12A	109.5
C3—C4—C5	122.3 (3)	C11—C12—H12B	109.5
C3—C4—H4	118.8	H12A—C12—H12B	109.5
C5—C4—H4	118.8	C11—C12—H12C	109.5
N4—C22—C23	110.0 (2)	H12A—C12—H12C	109.5
N4—C22—H22A	109.7	H12B—C12—H12C	109.5
C23—C22—H22A	109.7	N5—C26—H26A	109.5
N4—C22—H22B	109.7	N5—C26—H26B	109.5
C23—C22—H22B	109.7	H26A—C26—H26B	109.5
H22A—C22—H22B	108.2	N5—C26—H26C	109.5
C18—C19—C20	120.3 (3)	H26A—C26—H26C	109.5
C18—C19—H19	119.8	H26B—C26—H26C	109.5
C20—C19—H19	119.8	O1—C1—H1A	109.5
C24—N5—C23	110.9 (2)	O1—C1—H1B	109.5

C24—N5—C26	109.9 (3)	H1A—C1—H1B	109.5
C23—N5—C26	110.3 (3)	O1—C1—H1C	109.5
N4—C25—C24	108.3 (2)	H1A—C1—H1C	109.5
N4—C25—H25A	110.0	H1B—C1—H1C	109.5
C24—C25—H25A	110.0		

C8—N1—C11—N2	1.6 (4)	C14—C15—C20—C19	−177.2 (2)
C8—N1—C11—C12	−178.2 (2)	N1—C8—C9—C10	−0.8 (4)
C10—N2—C11—N1	−0.1 (4)	C5—C8—C9—C10	177.6 (2)
C10—N2—C11—C12	179.7 (2)	N2—C10—C9—C8	2.3 (4)
C11—N2—C10—C9	−1.8 (4)	C13—C10—C9—C8	179.1 (2)
C11—N2—C10—C13	−178.9 (2)	C6—C5—C4—C3	−0.7 (4)
C14—C13—C10—N2	34.1 (3)	C8—C5—C4—C3	179.7 (3)
C21—C13—C10—N2	−147.7 (2)	C21—N4—C22—C23	−164.8 (2)
C14—C13—C10—C9	−142.9 (3)	C25—N4—C22—C23	61.0 (3)
C21—C13—C10—C9	35.3 (4)	N3—C20—C19—C18	175.4 (3)
C11—N1—C8—C9	−1.0 (4)	C15—C20—C19—C18	−2.9 (4)
C11—N1—C8—C5	−179.5 (2)	C21—N4—C25—C24	165.0 (2)
C6—C5—C8—N1	179.7 (2)	C22—N4—C25—C24	−60.8 (3)
C4—C5—C8—N1	−0.7 (4)	C1—O1—C2—C3	169.6 (3)
C6—C5—C8—C9	1.2 (4)	C1—O1—C2—C7	−9.3 (5)
C4—C5—C8—C9	−179.1 (3)	C20—C15—C16—C17	−1.0 (4)
C20—N3—C21—N4	178.1 (2)	C14—C15—C16—C17	179.0 (3)
C20—N3—C21—C13	−3.1 (4)	C20—C19—C18—C17	1.0 (5)
C22—N4—C21—N3	−119.1 (3)	C5—C4—C3—C2	0.1 (5)
C25—N4—C21—N3	12.0 (3)	O1—C2—C3—C4	−178.7 (3)
C22—N4—C21—C13	62.1 (3)	C7—C2—C3—C4	0.3 (5)
C25—N4—C21—C13	−166.7 (2)	O1—C2—C7—C6	178.8 (3)
C14—C13—C21—N3	5.6 (4)	C3—C2—C7—C6	−0.1 (5)
C10—C13—C21—N3	−172.6 (2)	C15—C16—C17—C18	−0.9 (5)
C14—C13—C21—N4	−175.7 (2)	C19—C18—C17—C16	0.9 (5)
C10—C13—C21—N4	6.1 (4)	C4—C5—C6—C7	0.9 (4)
C21—C13—C14—C15	−2.8 (4)	C8—C5—C6—C7	−179.5 (3)
C10—C13—C14—C15	175.5 (2)	C2—C7—C6—C5	−0.5 (5)
C16—C15—C14—C13	178.0 (3)	C23—N5—C24—C25	−57.3 (3)
C20—C15—C14—C13	−2.0 (4)	C26—N5—C24—C25	−179.5 (2)
C21—N3—C20—C19	179.7 (2)	N4—C25—C24—N5	59.3 (3)
C21—N3—C20—C15	−2.1 (4)	C24—N5—C23—C22	56.2 (3)
C16—C15—C20—N3	−175.4 (2)	C26—N5—C23—C22	178.2 (2)
C14—C15—C20—N3	4.6 (4)	N4—C22—C23—N5	−57.9 (3)
C16—C15—C20—C19	2.9 (4)		

Hydrogen-bond geometry (Å, °)

Cg1, Cg2 and Cg3 are the centroids of the C15–C20, C8–C10/N2/C11/N2 and C2–C7 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···*Cg*1^{i}	0.96	3.10	3.867 (2)	138

C25—H25A···$Cg2^{ii}$	0.97	2.96	3.706 (2)	134
C17—H17···$Cg3^{iii}$	0.93	3.08	3.759 (2)	131

Symmetry codes: (i) $x-1, y, z-1$; (ii) $x, -y+1/2, z+1/2$; (iii) $x+1, y, z+1$.

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tert-Butyl 4-{3-[6-(4-methoxyphenyl)-2-methylpyrimidin-4-yl]quinolin-2-yl}piperazine-1-carboxylate

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The asymmetric unit of the title compound, $C_{30}H_{33}N_5O_3$, contains two independent molecules, *A* and *B*. In molecule *A*, the *tert*-butyl 4-methylpiperazine-1-carboxylate group was refined as disordered over two sets of sites with an occupancy ratio of 0.821 (4):0.179 (4). The piperazine ring adopts a chair conformation in both molecules. The dihedral angles between the pyrimidine ring and methoxyphenyl ring are 20.27 (14) and 8.72 (11)° for molecules *A* and *B*, respectively. The quinoline ring system makes dihedral angles of 30.68 (11) and 37.76 (10)° with the pyrimidine ring in molecules *A* and *B*, respectively. In the crystal, C—H···O hydrogen bonds link the molecules into chains along [100].

Structure description

Pyrimidine and its derivatives have a diverse array of biological and pharmacological activities including anticonvulsant, antibacterial, antifungal, antiviral and anticancer properties (Jain *et al.*, 2006). An SAR study of the pyrimidine nucleus revealed that substitution at different positions may lead to compounds with a variation in their biological activities (Khanage *et al.*, 2012). Compounds with a pyrimidine ring substituted at the 2 and 4 positions with keto or amino or mixed keto–amino groups have shown anticancer, antiviral, antibacterial and antifungal activity and are used in the treatment of respiratory tract infections and liver disorders. Substitution at the 5 and 6 positions with a heterocyclic ring or a substituted aryl ring leads to anticancer, antiviral, antibacterial and

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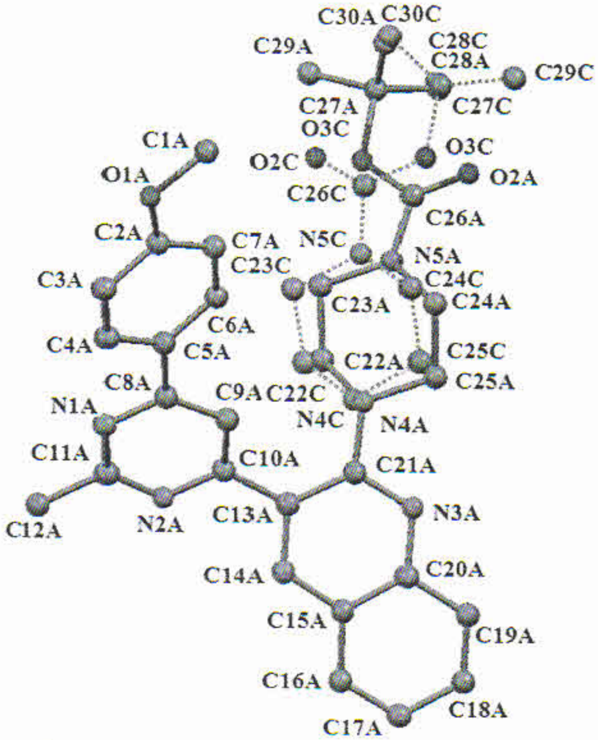

Figure 1
The molecular structure of molecule *A* showing 30% probability displacement ellipsoids. The minor disorder component is shown with green dashed bonds. H atoms are omitted for clarity.

vasodilation activity and such compounds are also used in the treatment of urinary tract infections (Selvam *et al.*, 2012). On the other hand, quinoline derivatives also find importance owing to their wide occurrence in natural products and in biologically active compounds (Markees *et al.*, 1970; Campbell *et al.*, 1988; Kalluraya & Sreenivasa, 1998). As part of our studies in this area, we now describe the synthesis and structure of the title compound.

The asymmetric unit of the title compound comprises two independent molecules, *A* and *B* (Figs. 1 and 2). In both the molecules, the piperazine ring adopts a chair conformation. In the major occupancy component of disordered molecule *A* [0.821 (5) occupancy], the best mirror plane passes through atoms N4*A* and N5*A* and the best twofold rotational axis bisects the N5*A*—C23*A* bond [asymmetry parameter C_s(N4*A*) = 2.53 and C_2(N5*A*—C23*A*) = 1.91] whereas for the minor [0.179 (5)] occupancy component, the best mirror plane passes through atoms C23*C* and C25*C* and the best twofold rotational axis bisects the C22*C*—C23*C* bond [asymmetry parameter C_s(C23*C*) = 1.06 and C_2(C22*C*—C23*C*) = 0.71]. In molecule *B*, the best mirror plane passes through atoms N4*B* and N5*B* and the best twofold rotational axis bisects the N5*B*—C24*B* bond [asymmetry parameter C_s(N4*B*) = 0.54 and C_2(N5*B*—C24*B*) = 5.2] (Duax & Norton, 1975). All bond lengths are in normal ranges and are comparable with those in previously reported related structures (Prasath *et al.*, 2010, 2011; Sharma *et al.*, 2017; Singh *et al.*, 2018). The dihedral angle between the pyrimidine and methoxyphenyl rings is 20.27 (14)° in molecule *A* and 8.72 (11)° in molecule *B* while the pyrimidine ring makes a dihedral angle of 30.68 (11)° in *A* and 37.76 (10)° in *B* with the quinoline ring system. The quinoline units are essentially planar with a maximum deviation of 0.0851 (1) Å for atom C21*A* and 0.0607 (1) Å for C21*B*.

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7*B*—H7*BA*···O2*B*^{i}	0.93	2.40	3.141 (2)	136

Symmetry code: (i) *x* + 1, *y*, *z*.

In the crystal, adjacent molecules are linked by C—H···O hydrogen bonds, forming chains along [100] (Table 1 and Fig. 3).

Synthesis and crystallization

2-(4-N-Boc-piperazin-1-yl) quinoline chalcone (1000 mg, 0.00258 mmol), acetamidine hydrochloride (199 mg, 0.002211 mmol) and NaOH (253 mg, 0.00633 mmol) were refluxed for 12 h in ethanol. The progress of the reaction was

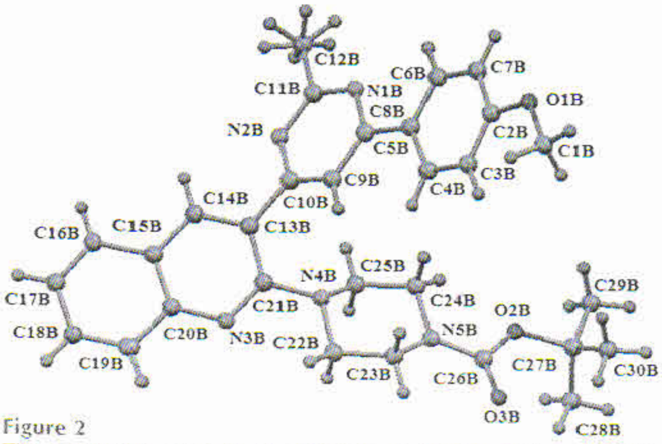

Figure 2
The molecular structure of molecule *B* showing 30% probability displacement ellipsoids.

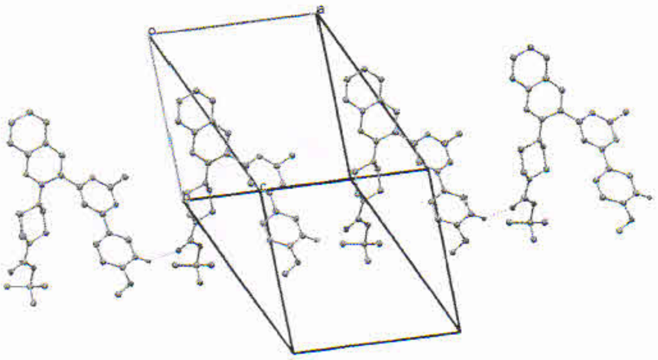

Figure 3
Partial packing plot. Dashed lines indicate the C—H···O intermolecular interactions forming chains along [100]. H atoms not involved in these interactions have been omitted for clarity.

monitored by thin layer chromatography. After the completion of reaction, the reaction mixture was poured into water and extracted with ethyl acetate. The organic layer was washed with water, dried and concentrated under vacuum using a rotary evaporator to obtain a solid that was purified by column chromatography using 60–120 mesh silica gel. Crystals of the title compound were obtained by recrystallization from ethyl acetate solution.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Disorder was modeled for the *tert*-butyl 4-methyl piperazine-1-carboxylate group in molecule *A* with an occupancy ratio of 0.821 (4):0.179 (4). In addition, the methyl H atoms attached to C12*A* and C12*B* are also disordered with occupancy ratios of 0.68 (4):0.32 (4) and 0.53 (4):0.47 (4), respectively.

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Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{30}H_{33}N_5O_3$
M_r	511.61
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	293
a, b, c (Å)	11.0894 (6), 12.6875 (6), 20.8953 (9)
α, β, γ (°)	83.663 (4), 75.335 (4), 86.259 (4)
V (Å^3)	2824.6 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.30 × 0.20 × 0.20
Data collection	
Diffractometer	Oxford Diffraction Xcalibur Sapphire3
Absorption correction	Multi-scan (*CrysAlis RED*; Oxford Diffraction, 2010)
$T_{\min}, T_{\max}$	0.869, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19560, 9886, 5244
R_{int}	0.042
$(\sin \theta/\lambda)_{\max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.065, 0.181, 1.03
No. of reflections	9886
No. of parameters	751
No. of restraints	32
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\max}, \Delta\rho_{\min}$ (e Å^{-3})	0.27, −0.21

Computer programs: *CrysAlis PRO* (Oxford Diffraction, 2010), *SHELXL97* (Sheldrick, 2008), *SHELXL2016* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009).

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full crystallographic data

IUCrData (2018). **3**, x180427 [https://doi.org/10.1107/S2414314618004273]

tert-Butyl 4-{3-[6-(4-methoxyphenyl)-2-methylpyrimidin-4-yl]quinolin-2-yl}piperazine-1-carboxylate

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tert-Butyl 4-{3-[6-(4-methoxyphenyl)-2-methylpyrimidin-4-yl]quinolin-2-yl}piperazine-1-carboxylate

Crystal data

$C_{30}H_{33}N_5O_3$
$M_r = 511.61$
Triclinic, $P\bar{1}$
$a = 11.0894$ (6) Å
$b = 12.6875$ (6) Å
$c = 20.8953$ (9) Å
$\alpha = 83.663$ (4)°
$\beta = 75.335$ (4)°
$\gamma = 86.259$ (4)°
$V = 2824.6$ (2) Å^3

$Z = 4$
$F(000) = 1088$
$D_x = 1.203$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4885 reflections
$\theta = 3.8–26.6°$
$\mu = 0.08$ mm^{-1}
$T = 293$ K
Block, white
0.30 × 0.20 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 6.1049 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2010)
$T_{\min} = 0.869$, $T_{\max} = 1.000$

19560 measured reflections
9886 independent reflections
5244 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.042$
$\theta_{\max} = 25.0°$, $\theta_{\min} = 3.6°$
$h = -13 \rightarrow 13$
$k = -15 \rightarrow 15$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.181$
$S = 1.03$
9886 reflections
751 parameters
32 restraints
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.056P)^2 + 0.4039P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}
Extinction correction: SHELXL2016 (Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0055 (7)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. All H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C—H distances of 0.93–0.97 Å; and with $U_{\rm iso}$(H) = 1.2Ueq(C), except for the methyl groups where $U_{\rm iso}$(H) = 1.5Ueq(C).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$	Occ. (<1)
O1A	0.4112 (2)	0.24904 (15)	0.89015 (12)	0.0782 (7)	
N1A	0.4291 (2)	0.74137 (18)	0.90707 (12)	0.0547 (7)	
N2A	0.3208 (2)	0.90807 (18)	0.89406 (12)	0.0549 (7)	
N3A	−0.0740 (2)	0.95687 (18)	0.83629 (12)	0.0535 (7)	
C1A	0.3435 (4)	0.1902 (2)	0.8577 (2)	0.0910 (13)	
H1AA	0.364378	0.115925	0.864713	0.137*	
H1AB	0.364741	0.212412	0.810897	0.137*	
H1AC	0.255635	0.202571	0.875508	0.137*	
C2A	0.3901 (3)	0.3566 (2)	0.88660 (16)	0.0557 (8)	
C3A	0.4511 (3)	0.4105 (2)	0.92207 (17)	0.0631 (9)	
H3AA	0.503422	0.373218	0.945828	0.076*	
C4A	0.4352 (3)	0.5183 (2)	0.92263 (16)	0.0577 (8)	
H4AA	0.477179	0.553336	0.946796	0.069*	
C5A	0.3575 (3)	0.5764 (2)	0.88772 (13)	0.0439 (7)	
C6A	0.2974 (3)	0.5202 (2)	0.85269 (15)	0.0553 (8)	
H6AA	0.244421	0.556792	0.829052	0.066*	
C7A	0.3135 (3)	0.4115 (2)	0.85170 (16)	0.0607 (9)	
H7AA	0.272351	0.375925	0.827392	0.073*	
C8A	0.3410 (3)	0.6926 (2)	0.88887 (13)	0.0441 (7)	
C9A	0.2394 (3)	0.7501 (2)	0.87395 (13)	0.0461 (7)	
H9AA	0.177416	0.716377	0.862080	0.055*	
C10A	0.2319 (3)	0.8586 (2)	0.87707 (13)	0.0447 (7)	
C11A	0.4142 (3)	0.8467 (2)	0.90806 (16)	0.0573 (8)	
C12A	0.5128 (3)	0.9007 (3)	0.9286 (2)	0.0883 (12)	
H12D	0.523039	0.866999	0.970447	0.132*	0.68 (4)
H12E	0.488240	0.974079	0.932971	0.132*	0.68 (4)
H12F	0.590317	0.895735	0.895541	0.132*	0.68 (4)
H12X	0.544692	0.957543	0.895526	0.132*	0.32 (4)
H12Y	0.579490	0.850463	0.933001	0.132*	0.32 (4)
H12Z	0.477414	0.928807	0.970432	0.132*	0.32 (4)
C13A	0.1206 (3)	0.9268 (2)	0.86981 (14)	0.0453 (7)	
C14A	0.0897 (3)	1.0104 (2)	0.90780 (14)	0.0514 (8)	
H14B	0.144325	1.027588	0.931943	0.062*	
C15A	−0.0226 (3)	1.0711 (2)	0.91138 (14)	0.0517 (8)	
C16A	−0.0554 (3)	1.1607 (2)	0.94699 (15)	0.0657 (9)	
H16B	−0.000588	1.182985	0.969415	0.079*	
C17A	−0.1658 (4)	1.2153 (3)	0.94927 (17)	0.0763 (11)	

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H17B	−0.185662	1.275867	0.971721	0.092*	
C18A	−0.2483 (4)	1.1787 (3)	0.91735 (18)	0.0811 (12)	
H18B	−0.325076	1.214441	0.919940	0.097*	
C19A	−0.2206 (3)	1.0926 (3)	0.88262 (17)	0.0693 (10)	
H19B	−0.278427	1.070025	0.862175	0.083*	
C20A	−0.1046 (3)	1.0368 (2)	0.87717 (15)	0.0534 (8)	
C21A	0.0360 (3)	0.9066 (2)	0.83122 (14)	0.0471 (7)	
N4A	0.0650 (6)	0.8296 (5)	0.7862 (3)	0.046 (2)	0.821 (4)
N5A	0.1156 (4)	0.7165 (3)	0.67279 (16)	0.0595 (10)	0.821 (4)
C22A	0.1768 (6)	0.8494 (5)	0.7325 (3)	0.0569 (17)	0.821 (4)
H22A	0.243242	0.869194	0.750750	0.068*	0.821 (4)
H22B	0.159489	0.908089	0.701780	0.068*	0.821 (4)
C23A	0.2187 (5)	0.7528 (4)	0.6955 (2)	0.0584 (12)	0.821 (4)
H23A	0.288056	0.769940	0.657650	0.070*	0.821 (4)
H23B	0.246730	0.696709	0.724492	0.070*	0.821 (4)
C24A	0.0010 (5)	0.7023 (3)	0.7245 (2)	0.0648 (12)	0.821 (4)
H24A	0.012995	0.643983	0.756743	0.078*	0.821 (4)
H24B	−0.064906	0.684583	0.705132	0.078*	0.821 (4)
C25A	−0.0367 (5)	0.8018 (4)	0.7589 (3)	0.0580 (14)	0.821 (4)
H25A	−0.054339	0.859274	0.727514	0.070*	0.821 (4)
H25B	−0.111648	0.790413	0.794376	0.070*	0.821 (4)
O2A	0.0461 (3)	0.6256 (2)	0.60292 (15)	0.0806 (10)	0.821 (4)
O3A	0.2454 (3)	0.6779 (3)	0.57806 (16)	0.0736 (10)	0.821 (4)
C26A	0.1296 (4)	0.6692 (3)	0.6160 (2)	0.0618 (12)	0.821 (4)
C27A	0.2787 (4)	0.6395 (4)	0.51153 (19)	0.0716 (13)	0.821 (4)
C28A	0.1984 (12)	0.6966 (7)	0.4686 (3)	0.115 (5)	0.821 (4)
H28D	0.112601	0.680592	0.487811	0.173*	0.821 (4)
H28E	0.224041	0.673604	0.424767	0.173*	0.821 (4)
H28F	0.207761	0.771734	0.466098	0.173*	0.821 (4)
C29A	0.4124 (4)	0.6716 (4)	0.4865 (2)	0.1048 (19)	0.821 (4)
H29D	0.460597	0.638089	0.515867	0.157*	0.821 (4)
H29E	0.415776	0.747231	0.484935	0.157*	0.821 (4)
H29F	0.445958	0.649747	0.442710	0.157*	0.821 (4)
C30A	0.2686 (11)	0.5201 (6)	0.5179 (5)	0.122 (6)	0.821 (4)
H30D	0.308103	0.488397	0.551669	0.182*	0.821 (4)
H30E	0.308971	0.493401	0.476254	0.182*	0.821 (4)
H30F	0.182220	0.502811	0.529939	0.182*	0.821 (4)
N4C	0.085 (2)	0.836 (2)	0.7815 (14)	0.047 (12)*	0.179 (4)
N5C	0.1755 (15)	0.7272 (14)	0.6637 (7)	0.060 (6)*	0.179 (4)
C22C	0.206 (2)	0.8579 (18)	0.7344 (14)	0.031 (6)*	0.179 (4)
H22E	0.262662	0.880194	0.758237	0.037*	0.179 (4)
H22F	0.194262	0.915838	0.701871	0.037*	0.179 (4)
C23C	0.2630 (15)	0.763 (2)	0.6988 (11)	0.072 (10)*	0.179 (4)
H23E	0.340864	0.781375	0.667239	0.087*	0.179 (4)
H23F	0.280618	0.705894	0.730595	0.087*	0.179 (4)
C24C	0.0558 (16)	0.7045 (16)	0.7084 (10)	0.058 (8)*	0.179 (4)
H24E	0.065108	0.644200	0.739854	0.070*	0.179 (4)
H24F	−0.000913	0.685885	0.683268	0.070*	0.179 (4)

C25C	0.0015 (17)	0.7984 (18)	0.7456 (13)	0.050 (8)*	0.179 (4)
H25E	−0.017292	0.855714	0.714419	0.060*	0.179 (4)
H25F	−0.076294	0.779032	0.777063	0.060*	0.179 (4)
O2C	0.3282 (13)	0.6920 (11)	0.5751 (7)	0.073 (5)*	0.179 (4)
O3C	0.1338 (15)	0.6351 (13)	0.5861 (7)	0.083 (6)*	0.179 (4)
C26C	0.2201 (14)	0.6809 (16)	0.6060 (7)	0.046 (6)*	0.179 (4)
C27C	0.1653 (14)	0.5955 (12)	0.5196 (7)	0.070 (6)*	0.179 (4)
C28C	0.204 (4)	0.6879 (19)	0.4671 (11)	0.065 (13)*	0.179 (4)
H28A	0.224656	0.662758	0.423825	0.098*	0.179 (4)
H28B	0.136505	0.739899	0.470207	0.098*	0.179 (4)
H28C	0.275499	0.719316	0.474140	0.098*	0.179 (4)
C29C	0.0406 (14)	0.5576 (13)	0.5176 (9)	0.076 (6)*	0.179 (4)
H29A	0.047717	0.529127	0.476050	0.114*	0.179 (4)
H29B	0.013994	0.503444	0.553492	0.114*	0.179 (4)
H29C	−0.019517	0.616014	0.522018	0.114*	0.179 (4)
C30C	0.264 (3)	0.507 (2)	0.5163 (17)	0.10 (2)*	0.179 (4)
H30A	0.284381	0.482174	0.473014	0.145*	0.179 (4)
H30B	0.337996	0.533877	0.524238	0.145*	0.179 (4)
H30C	0.234172	0.450245	0.549386	0.145*	0.179 (4)
O1B	0.4659 (2)	1.16589 (17)	0.28836 (13)	0.0853 (8)	
N1B	0.4623 (2)	0.66522 (19)	0.30835 (12)	0.0557 (7)	
N2B	0.3734 (2)	0.52099 (18)	0.27451 (12)	0.0551 (7)	
N3B	0.0359 (2)	0.55788 (18)	0.16688 (12)	0.0520 (6)	
N4B	0.0356 (2)	0.66254 (17)	0.25152 (11)	0.0498 (6)	
N5B	−0.1124 (2)	0.7906 (2)	0.34538 (12)	0.0630 (8)	
C1B	0.3905 (4)	1.2467 (3)	0.26348 (19)	0.0943 (13)	
H1BA	0.422597	1.314778	0.264531	0.141*	
H1BB	0.306494	1.243604	0.290543	0.141*	
H1BC	0.391224	1.236430	0.218548	0.141*	
C2B	0.4366 (3)	1.0626 (2)	0.28821 (16)	0.0606 (9)	
C3B	0.3370 (3)	1.0326 (2)	0.26817 (17)	0.0663 (9)	
H3BA	0.282413	1.083486	0.254246	0.080*	
C4B	0.3181 (3)	0.9249 (2)	0.26880 (16)	0.0619 (9)	
H4BA	0.249447	0.904575	0.255800	0.074*	
C5B	0.3986 (3)	0.8478 (2)	0.28824 (14)	0.0488 (7)	
C6B	0.4954 (3)	0.8812 (2)	0.31091 (15)	0.0598 (9)	
H6BA	0.548875	0.830766	0.326207	0.072*	
C7B	0.5143 (3)	0.9874 (2)	0.31135 (16)	0.0647 (9)	
H7BA	0.579421	1.008000	0.327225	0.078*	
C8B	0.3852 (3)	0.7341 (2)	0.28273 (14)	0.0471 (7)	
C9B	0.3023 (3)	0.6982 (2)	0.25148 (14)	0.0506 (8)	
H9BA	0.250663	0.745740	0.232552	0.061*	
C10B	0.2974 (3)	0.5898 (2)	0.24882 (13)	0.0468 (7)	
C11B	0.4525 (3)	0.5623 (2)	0.30273 (15)	0.0577 (9)	
C12B	0.5392 (4)	0.4855 (3)	0.33093 (18)	0.0829 (12)	
H12A	0.494706	0.424178	0.353327	0.124*	0.53 (4)
H12B	0.571196	0.519033	0.361898	0.124*	0.53 (4)
H12C	0.607200	0.464357	0.295566	0.124*	0.53 (4)

H12H	0.620695	0.514201	0.320534	0.124*	0.47 (4)
H12I	0.544205	0.419346	0.311963	0.124*	0.47 (4)
H12J	0.508201	0.474022	0.378295	0.124*	0.47 (4)
C13B	0.2175 (3)	0.5448 (2)	0.21209 (14)	0.0460 (7)	
C14B	0.2663 (3)	0.4644 (2)	0.17423 (14)	0.0513 (8)	
H14A	0.342992	0.432813	0.177178	0.062*	
C15B	0.2034 (3)	0.4276 (2)	0.13054 (15)	0.0507 (8)	
C16B	0.2510 (3)	0.3443 (2)	0.09124 (16)	0.0642 (9)	
H16A	0.326080	0.309344	0.094095	0.077*	
C17B	0.1869 (4)	0.3149 (3)	0.04889 (18)	0.0759 (10)	
H17A	0.217222	0.258515	0.023653	0.091*	
C18B	0.0765 (4)	0.3686 (3)	0.04325 (18)	0.0782 (11)	
H18A	0.034490	0.348840	0.013448	0.094*	
C19B	0.0286 (3)	0.4503 (3)	0.08085 (16)	0.0684 (9)	
H19A	−0.045362	0.485812	0.076405	0.082*	
C20B	0.0911 (3)	0.4806 (2)	0.12621 (15)	0.0505 (8)	
C21B	0.0952 (3)	0.5863 (2)	0.20887 (14)	0.0459 (7)	
C22B	−0.0774 (3)	0.7163 (2)	0.23884 (15)	0.0597 (9)	
H22C	−0.146901	0.669642	0.254401	0.072*	
H22D	−0.066387	0.734848	0.191477	0.072*	
C23B	−0.1044 (3)	0.8159 (2)	0.27487 (15)	0.0661 (10)	
H23C	−0.038772	0.865391	0.255923	0.079*	
H23D	−0.182533	0.849538	0.269037	0.079*	
C24B	−0.0071 (3)	0.7270 (3)	0.36064 (16)	0.0706 (10)	
H24C	−0.025227	0.704896	0.407932	0.085*	
H24D	0.066415	0.769326	0.349080	0.085*	
C25B	0.0178 (3)	0.6312 (2)	0.32272 (14)	0.0609 (9)	
H25C	0.091885	0.592583	0.330594	0.073*	
H25D	−0.051874	0.584688	0.338109	0.073*	
O2B	−0.2597 (2)	0.9194 (2)	0.37400 (12)	0.0920 (9)	
O3B	−0.1626 (2)	0.8354 (2)	0.44866 (12)	0.0929 (9)	
C26B	−0.1844 (3)	0.8539 (3)	0.38876 (17)	0.0651 (9)	
C27B	−0.2333 (5)	0.8896 (4)	0.5058 (2)	0.0995 (14)	
C28B	−0.3655 (5)	0.8573 (5)	0.5203 (3)	0.181 (3)	
H28G	−0.401764	0.886628	0.484821	0.271*	
H28H	−0.411978	0.883339	0.561224	0.271*	
H28I	−0.367602	0.781332	0.524297	0.271*	
C29B	−0.1713 (6)	0.8443 (5)	0.5595 (2)	0.182 (3)	
H29G	−0.082931	0.853015	0.544378	0.274*	
H29H	−0.187609	0.770219	0.569941	0.274*	
H29I	−0.203714	0.880877	0.598506	0.274*	
C30B	−0.2271 (7)	1.0048 (4)	0.4932 (3)	0.186 (3)	
H30G	−0.276743	1.030462	0.462799	0.280*	
H30H	−0.142085	1.023623	0.474388	0.280*	
H30I	−0.258472	1.036247	0.534293	0.280*	

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.0893 (18)	0.0390 (12)	0.108 (2)	0.0004 (12)	−0.0272 (16)	−0.0079 (12)
N1A	0.0546 (16)	0.0462 (14)	0.0688 (17)	−0.0004 (12)	−0.0213 (14)	−0.0169 (12)
N2A	0.0581 (16)	0.0428 (14)	0.0682 (18)	−0.0056 (12)	−0.0198 (14)	−0.0126 (12)
N3A	0.0581 (17)	0.0504 (14)	0.0517 (16)	0.0005 (13)	−0.0136 (13)	−0.0055 (12)
C1A	0.101 (3)	0.048 (2)	0.125 (4)	−0.014 (2)	−0.020 (3)	−0.023 (2)
C2A	0.059 (2)	0.0412 (18)	0.065 (2)	−0.0049 (15)	−0.0103 (17)	−0.0049 (15)
C3A	0.063 (2)	0.0474 (18)	0.085 (2)	0.0040 (16)	−0.0331 (19)	−0.0003 (17)
C4A	0.060 (2)	0.0477 (18)	0.072 (2)	−0.0038 (15)	−0.0272 (18)	−0.0056 (16)
C5A	0.0491 (18)	0.0383 (15)	0.0447 (17)	−0.0046 (13)	−0.0109 (14)	−0.0051 (13)
C6A	0.064 (2)	0.0456 (17)	0.062 (2)	0.0003 (15)	−0.0264 (17)	−0.0083 (15)
C7A	0.079 (2)	0.0421 (17)	0.068 (2)	−0.0072 (16)	−0.0286 (19)	−0.0107 (15)
C8A	0.0508 (18)	0.0411 (16)	0.0408 (16)	−0.0022 (14)	−0.0095 (14)	−0.0093 (12)
C9A	0.0554 (19)	0.0400 (16)	0.0481 (17)	−0.0066 (14)	−0.0209 (15)	−0.0056 (13)
C10A	0.0523 (18)	0.0391 (16)	0.0438 (17)	−0.0012 (14)	−0.0125 (15)	−0.0070 (13)
C11A	0.057 (2)	0.0511 (19)	0.070 (2)	−0.0077 (16)	−0.0217 (18)	−0.0173 (16)
C12A	0.076 (3)	0.063 (2)	0.142 (4)	−0.0108 (19)	−0.044 (3)	−0.032 (2)
C13A	0.0521 (18)	0.0370 (15)	0.0469 (17)	−0.0064 (13)	−0.0117 (15)	−0.0024 (13)
C14A	0.060 (2)	0.0443 (16)	0.0505 (18)	−0.0061 (15)	−0.0131 (16)	−0.0057 (14)
C15A	0.063 (2)	0.0421 (16)	0.0436 (18)	0.0016 (15)	−0.0038 (16)	−0.0019 (13)
C16A	0.087 (3)	0.0550 (19)	0.051 (2)	0.0082 (18)	−0.0082 (19)	−0.0168 (15)
C17A	0.095 (3)	0.066 (2)	0.058 (2)	0.019 (2)	−0.003 (2)	−0.0138 (17)
C18A	0.083 (3)	0.080 (3)	0.068 (3)	0.028 (2)	−0.001 (2)	−0.010 (2)
C19A	0.063 (2)	0.075 (2)	0.064 (2)	0.0113 (18)	−0.0084 (19)	−0.0092 (18)
C20A	0.057 (2)	0.0494 (18)	0.0446 (18)	0.0043 (15)	−0.0003 (16)	0.0020 (14)
C21A	0.0544 (19)	0.0398 (16)	0.0439 (17)	−0.0049 (14)	−0.0067 (15)	−0.0009 (13)
N4A	0.049 (3)	0.047 (3)	0.044 (3)	−0.007 (2)	−0.0084 (18)	−0.0108 (17)
N5A	0.061 (3)	0.071 (2)	0.046 (2)	−0.012 (2)	−0.004 (2)	−0.0205 (17)
C22A	0.059 (4)	0.062 (3)	0.053 (3)	−0.008 (3)	−0.014 (3)	−0.0139 (19)
C23A	0.056 (3)	0.074 (3)	0.045 (3)	−0.001 (3)	−0.008 (2)	−0.016 (2)
C24A	0.065 (3)	0.064 (3)	0.066 (3)	−0.015 (2)	−0.007 (3)	−0.021 (2)
C25A	0.050 (3)	0.066 (3)	0.059 (3)	−0.007 (2)	−0.009 (3)	−0.017 (2)
O2A	0.088 (3)	0.092 (2)	0.068 (2)	−0.0160 (19)	−0.0164 (19)	−0.0331 (16)
O3A	0.073 (3)	0.101 (2)	0.047 (2)	−0.0036 (19)	−0.0081 (19)	−0.0239 (18)
C26A	0.066 (3)	0.059 (3)	0.062 (3)	−0.006 (2)	−0.017 (3)	−0.010 (2)
C27A	0.076 (3)	0.100 (3)	0.038 (2)	0.011 (3)	−0.011 (2)	−0.021 (2)
C28A	0.115 (7)	0.174 (9)	0.064 (4)	0.007 (5)	−0.044 (4)	−0.002 (4)
C29A	0.092 (4)	0.163 (5)	0.057 (3)	0.006 (4)	−0.005 (3)	−0.039 (3)
C30A	0.159 (9)	0.108 (7)	0.103 (7)	0.031 (4)	−0.031 (4)	−0.061 (5)
O1B	0.108 (2)	0.0496 (14)	0.108 (2)	−0.0091 (13)	−0.0438 (17)	−0.0097 (13)
N1B	0.0653 (17)	0.0537 (15)	0.0557 (16)	0.0090 (13)	−0.0282 (14)	−0.0135 (12)
N2B	0.0672 (18)	0.0518 (15)	0.0516 (15)	0.0078 (13)	−0.0257 (14)	−0.0075 (12)
N3B	0.0561 (16)	0.0524 (14)	0.0489 (15)	−0.0057 (12)	−0.0149 (13)	−0.0043 (12)
N4B	0.0564 (16)	0.0491 (14)	0.0442 (14)	0.0047 (12)	−0.0156 (13)	−0.0025 (11)
N5B	0.0701 (18)	0.0679 (17)	0.0520 (17)	0.0253 (14)	−0.0206 (15)	−0.0134 (13)
C1B	0.130 (4)	0.049 (2)	0.106 (3)	0.004 (2)	−0.039 (3)	0.002 (2)

C2B	0.069 (2)	0.050 (2)	0.065 (2)	−0.0040 (17)	−0.0184 (19)	−0.0080 (16)
C3B	0.073 (2)	0.053 (2)	0.078 (2)	0.0089 (17)	−0.029 (2)	−0.0076 (17)
C4B	0.063 (2)	0.059 (2)	0.072 (2)	0.0022 (17)	−0.0307 (18)	−0.0136 (16)
C5B	0.0519 (19)	0.0482 (17)	0.0494 (18)	0.0014 (14)	−0.0178 (15)	−0.0083 (14)
C6B	0.060 (2)	0.058 (2)	0.065 (2)	0.0051 (16)	−0.0235 (18)	−0.0092 (16)
C7B	0.067 (2)	0.054 (2)	0.081 (2)	−0.0037 (17)	−0.033 (2)	−0.0084 (17)
C8B	0.0467 (18)	0.0489 (17)	0.0456 (17)	0.0016 (14)	−0.0130 (15)	−0.0030 (14)
C9B	0.0528 (19)	0.0492 (18)	0.0518 (18)	0.0018 (14)	−0.0193 (16)	−0.0008 (14)
C10B	0.0510 (18)	0.0490 (17)	0.0394 (16)	−0.0004 (14)	−0.0090 (14)	−0.0062 (13)
C11B	0.069 (2)	0.058 (2)	0.0498 (19)	0.0130 (17)	−0.0233 (17)	−0.0114 (15)
C12B	0.101 (3)	0.070 (2)	0.094 (3)	0.027 (2)	−0.056 (2)	−0.018 (2)
C13B	0.0490 (18)	0.0441 (16)	0.0457 (17)	−0.0028 (13)	−0.0143 (15)	−0.0007 (13)
C14B	0.0511 (19)	0.0492 (17)	0.0535 (19)	−0.0024 (14)	−0.0122 (16)	−0.0058 (14)
C15B	0.0499 (19)	0.0531 (18)	0.0503 (18)	−0.0103 (15)	−0.0113 (16)	−0.0072 (14)
C16B	0.067 (2)	0.061 (2)	0.066 (2)	−0.0021 (17)	−0.0137 (19)	−0.0208 (17)
C17B	0.077 (3)	0.079 (2)	0.074 (3)	−0.006 (2)	−0.012 (2)	−0.0313 (19)
C18B	0.084 (3)	0.089 (3)	0.071 (2)	−0.015 (2)	−0.024 (2)	−0.032 (2)
C19B	0.064 (2)	0.082 (2)	0.067 (2)	−0.0052 (18)	−0.0238 (19)	−0.0199 (19)
C20B	0.053 (2)	0.0516 (17)	0.0480 (18)	−0.0096 (15)	−0.0121 (16)	−0.0074 (14)
C21B	0.0525 (19)	0.0441 (16)	0.0416 (17)	−0.0030 (14)	−0.0139 (15)	0.0002 (13)
C22B	0.058 (2)	0.070 (2)	0.056 (2)	0.0096 (17)	−0.0245 (17)	−0.0090 (16)
C23B	0.081 (2)	0.065 (2)	0.054 (2)	0.0225 (18)	−0.0256 (19)	−0.0060 (16)
C24B	0.078 (2)	0.082 (2)	0.051 (2)	0.027 (2)	−0.0206 (19)	−0.0126 (17)
C25B	0.068 (2)	0.063 (2)	0.0486 (19)	0.0104 (17)	−0.0133 (17)	0.0002 (16)
O2B	0.099 (2)	0.0923 (18)	0.0857 (19)	0.0430 (16)	−0.0306 (16)	−0.0226 (14)
O3B	0.108 (2)	0.112 (2)	0.0537 (15)	0.0459 (17)	−0.0173 (15)	−0.0233 (14)
C26B	0.070 (2)	0.062 (2)	0.060 (2)	0.0113 (18)	−0.013 (2)	−0.0101 (17)
C27B	0.107 (4)	0.125 (4)	0.063 (3)	0.032 (3)	−0.012 (3)	−0.037 (2)
C28B	0.119 (5)	0.277 (8)	0.124 (5)	−0.027 (5)	0.031 (4)	−0.060 (5)
C29B	0.210 (7)	0.267 (7)	0.069 (3)	0.102 (6)	−0.041 (4)	−0.060 (4)
C30B	0.289 (9)	0.119 (4)	0.166 (6)	0.028 (5)	−0.067 (6)	−0.073 (4)

Geometric parameters (Å, °)

O1A—C2A	1.367 (3)	O2C—C26C	1.218 (10)
O1A—C1A	1.423 (4)	O3C—C26C	1.325 (9)
N1A—C11A	1.337 (3)	O3C—C27C	1.480 (10)
N1A—C8A	1.342 (3)	C27C—C29C	1.505 (10)
N2A—C11A	1.328 (4)	C27C—C30C	1.511 (12)
N2A—C10A	1.341 (3)	C27C—C28C	1.518 (12)
N3A—C21A	1.325 (3)	C28C—H28A	0.9600
N3A—C20A	1.368 (4)	C28C—H28B	0.9600
C1A—H1AA	0.9600	C28C—H28C	0.9600
C1A—H1AB	0.9600	C29C—H29A	0.9600
C1A—H1AC	0.9600	C29C—H29B	0.9600
C2A—C7A	1.365 (4)	C29C—H29C	0.9600
C2A—C3A	1.378 (4)	C30C—H30A	0.9600
C3A—C4A	1.369 (4)	C30C—H30B	0.9600

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C3A—H3AA	0.9300	C30C—H30C	0.9600
C4A—C5A	1.391 (4)	O1B—C2B	1.371 (4)
C4A—H4AA	0.9300	O1B—C1B	1.419 (4)
C5A—C6A	1.383 (4)	N1B—C11B	1.338 (4)
C5A—C8A	1.476 (3)	N1B—C8B	1.346 (3)
C6A—C7A	1.380 (4)	N2B—C11B	1.335 (4)
C6A—H6AA	0.9300	N2B—C10B	1.337 (3)
C7A—H7AA	0.9300	N3B—C21B	1.314 (3)
C8A—C9A	1.386 (4)	N3B—C20B	1.379 (3)
C9A—C10A	1.382 (3)	N4B—C21B	1.405 (3)
C9A—H9AA	0.9300	N4B—C22B	1.457 (3)
C10A—C13A	1.491 (4)	N4B—C25B	1.463 (3)
C11A—C12A	1.500 (4)	N5B—C26B	1.347 (4)
C12A—H12D	0.9600	N5B—C23B	1.454 (4)
C12A—H12E	0.9600	N5B—C24B	1.458 (4)
C12A—H12F	0.9600	C1B—H1BA	0.9600
C12A—H12X	0.9600	C1B—H1BB	0.9600
C12A—H12Y	0.9600	C1B—H1BC	0.9600
C12A—H12Z	0.9600	C2B—C3B	1.365 (4)
C13A—C14A	1.368 (4)	C2B—C7B	1.375 (4)
C13A—C21A	1.434 (4)	C3B—C4B	1.394 (4)
C14A—C15A	1.411 (4)	C3B—H3BA	0.9300
C14A—H14B	0.9300	C4B—C5B	1.379 (4)
C15A—C16A	1.404 (4)	C4B—H4BA	0.9300
C15A—C20A	1.406 (4)	C5B—C6B	1.384 (4)
C16A—C17A	1.360 (4)	C5B—C8B	1.481 (4)
C16A—H16B	0.9300	C6B—C7B	1.378 (4)
C17A—C18A	1.390 (5)	C6B—H6BA	0.9300
C17A—H17B	0.9300	C7B—H7BA	0.9300
C18A—C19A	1.353 (5)	C8B—C9B	1.379 (4)
C18A—H18B	0.9300	C9B—C10B	1.388 (4)
C19A—C20A	1.412 (4)	C9B—H9BA	0.9300
C19A—H19B	0.9300	C10B—C13B	1.486 (4)
C21A—N4A	1.397 (4)	C11B—C12B	1.503 (4)
C21A—N4C	1.422 (9)	C12B—H12A	0.9600
N4A—C22A	1.461 (5)	C12B—H12B	0.9600
N4A—C25A	1.464 (5)	C12B—H12C	0.9600
N5A—C26A	1.358 (5)	C12B—H12H	0.9600
N5A—C24A	1.452 (6)	C12B—H12I	0.9600
N5A—C23A	1.461 (6)	C12B—H12J	0.9600
C22A—C23A	1.505 (4)	C13B—C14B	1.360 (4)
C22A—H22A	0.9700	C13B—C21B	1.437 (4)
C22A—H22B	0.9700	C14B—C15B	1.413 (4)
C23A—H23A	0.9700	C14B—H14A	0.9300
C23A—H23B	0.9700	C15B—C20B	1.395 (4)
C24A—C25A	1.504 (5)	C15B—C16B	1.403 (4)
C24A—H24A	0.9700	C16B—C17B	1.362 (4)
C24A—H24B	0.9700	C16B—H16A	0.9300

C25A—H25A	0.9700	C17B—C18B	1.387 (5)
C25A—H25B	0.9700	C17B—H17A	0.9300
O2A—C26A	1.211 (5)	C18B—C19B	1.368 (4)
O3A—C26A	1.332 (5)	C18B—H18A	0.9300
O3A—C27A	1.474 (5)	C19B—C20B	1.406 (4)
C27A—C29A	1.509 (6)	C19B—H19A	0.9300
C27A—C30A	1.515 (9)	C22B—C23B	1.516 (4)
C27A—C28A	1.516 (9)	C22B—H22C	0.9700
C28A—H28D	0.9600	C22B—H22D	0.9700
C28A—H28E	0.9600	C23B—H23C	0.9700
C28A—H28F	0.9600	C23B—H23D	0.9700
C29A—H29D	0.9600	C24B—C25B	1.496 (4)
C29A—H29E	0.9600	C24B—H24C	0.9700
C29A—H29F	0.9600	C24B—H24D	0.9700
C30A—H30D	0.9600	C25B—H25C	0.9700
C30A—H30E	0.9600	C25B—H25D	0.9700
C30A—H30F	0.9600	O2B—C26B	1.211 (3)
N4C—C25C	1.464 (10)	O3B—C26B	1.327 (4)
N4C—C22C	1.468 (10)	O3B—C27B	1.466 (4)
N5C—C26C	1.364 (9)	C27B—C30B	1.460 (6)
N5C—C24C	1.442 (10)	C27B—C28B	1.495 (7)
N5C—C23C	1.474 (10)	C27B—C29B	1.502 (6)
C22C—C23C	1.508 (9)	C28B—H28G	0.9600
C22C—H22E	0.9700	C28B—H28H	0.9600
C22C—H22F	0.9700	C28B—H28I	0.9600
C23C—H23E	0.9700	C29B—H29G	0.9600
C23C—H23F	0.9700	C29B—H29H	0.9600
C24C—C25C	1.504 (10)	C29B—H29I	0.9600
C24C—H24E	0.9700	C30B—H30G	0.9600
C24C—H24F	0.9700	C30B—H30H	0.9600
C25C—H25E	0.9700	C30B—H30I	0.9600
C25C—H25F	0.9700		

C2A—O1A—C1A	117.2 (3)	C24C—C25C—H25F	109.0
C11A—N1A—C8A	116.4 (3)	H25E—C25C—H25F	107.8
C11A—N2A—C10A	116.2 (2)	C26C—O3C—C27C	119.5 (11)
C21A—N3A—C20A	118.1 (3)	O2C—C26C—O3C	126.0 (12)
O1A—C1A—H1AA	109.5	O2C—C26C—N5C	119.8 (11)
O1A—C1A—H1AB	109.5	O3C—C26C—N5C	113.9 (10)
H1AA—C1A—H1AB	109.5	O3C—C27C—C29C	100.6 (10)
O1A—C1A—H1AC	109.5	O3C—C27C—C30C	110.0 (12)
H1AA—C1A—H1AC	109.5	C29C—C27C—C30C	113.1 (15)
H1AB—C1A—H1AC	109.5	O3C—C27C—C28C	109.0 (11)
C7A—C2A—O1A	124.7 (3)	C29C—C27C—C28C	110.5 (14)
C7A—C2A—C3A	119.6 (3)	C30C—C27C—C28C	112.9 (14)
O1A—C2A—C3A	115.7 (3)	C27C—C28C—H28A	109.5
C4A—C3A—C2A	120.5 (3)	C27C—C28C—H28B	109.5
C4A—C3A—H3AA	119.7	H28A—C28C—H28B	109.5

C2A—C3A—H3AA	119.7	C27C—C28C—H28C	109.5
C3A—C4A—C5A	121.2 (3)	H28A—C28C—H28C	109.5
C3A—C4A—H4AA	119.4	H28B—C28C—H28C	109.5
C5A—C4A—H4AA	119.4	C27C—C29C—H29A	109.5
C6A—C5A—C4A	117.0 (3)	C27C—C29C—H29B	109.5
C6A—C5A—C8A	122.4 (3)	H29A—C29C—H29B	109.5
C4A—C5A—C8A	120.7 (3)	C27C—C29C—H29C	109.5
C7A—C6A—C5A	122.1 (3)	H29A—C29C—H29C	109.5
C7A—C6A—H6AA	119.0	H29B—C29C—H29C	109.5
C5A—C6A—H6AA	119.0	C27C—C30C—H30A	109.5
C2A—C7A—C6A	119.6 (3)	C27C—C30C—H30B	109.5
C2A—C7A—H7AA	120.2	H30A—C30C—H30B	109.5
C6A—C7A—H7AA	120.2	C27C—C30C—H30C	109.5
N1A—C8A—C9A	120.5 (2)	H30A—C30C—H30C	109.5
N1A—C8A—C5A	116.4 (2)	H30B—C30C—H30C	109.5
C9A—C8A—C5A	123.1 (3)	C2B—O1B—C1B	117.6 (3)
C10A—C9A—C8A	118.7 (3)	C11B—N1B—C8B	116.8 (3)
C10A—C9A—H9AA	120.7	C11B—N2B—C10B	116.5 (2)
C8A—C9A—H9AA	120.7	C21B—N3B—C20B	118.3 (3)
N2A—C10A—C9A	121.1 (3)	C21B—N4B—C22B	117.4 (2)
N2A—C10A—C13A	115.1 (2)	C21B—N4B—C25B	116.1 (2)
C9A—C10A—C13A	123.4 (3)	C22B—N4B—C25B	110.0 (2)
N2A—C11A—N1A	127.1 (3)	C26B—N5B—C23B	118.7 (2)
N2A—C11A—C12A	116.7 (3)	C26B—N5B—C24B	121.9 (3)
N1A—C11A—C12A	116.2 (3)	C23B—N5B—C24B	114.9 (2)
C11A—C12A—H12D	109.5	O1B—C1B—H1BA	109.5
C11A—C12A—H12E	109.5	O1B—C1B—H1BB	109.5
H12D—C12A—H12E	109.5	H1BA—C1B—H1BB	109.5
C11A—C12A—H12F	109.5	O1B—C1B—H1BC	109.5
H12D—C12A—H12F	109.5	H1BA—C1B—H1BC	109.5
H12E—C12A—H12F	109.5	H1BB—C1B—H1BC	109.5
C11A—C12A—H12X	109.5	C3B—C2B—O1B	124.4 (3)
H12D—C12A—H12X	141.1	C3B—C2B—C7B	120.3 (3)
H12E—C12A—H12X	56.3	O1B—C2B—C7B	115.3 (3)
H12F—C12A—H12X	56.3	C2B—C3B—C4B	119.3 (3)
C11A—C12A—H12Y	109.5	C2B—C3B—H3BA	120.3
H12D—C12A—H12Y	56.3	C4B—C3B—H3BA	120.3
H12E—C12A—H12Y	141.1	C5B—C4B—C3B	121.5 (3)
H12F—C12A—H12Y	56.3	C5B—C4B—H4BA	119.2
H12X—C12A—H12Y	109.5	C3B—C4B—H4BA	119.2
C11A—C12A—H12Z	109.5	C4B—C5B—C6B	117.4 (3)
H12D—C12A—H12Z	56.3	C4B—C5B—C8B	121.4 (3)
H12E—C12A—H12Z	56.3	C6B—C5B—C8B	121.2 (3)
H12F—C12A—H12Z	141.1	C7B—C6B—C5B	121.6 (3)
H12X—C12A—H12Z	109.5	C7B—C6B—H6BA	119.2
H12Y—C12A—H12Z	109.5	C5B—C6B—H6BA	119.2
C14A—C13A—C21A	116.9 (3)	C2B—C7B—C6B	119.7 (3)
C14A—C13A—C10A	117.3 (3)	C2B—C7B—H7BA	120.1

C21A—C13A—C10A	125.5 (2)	C6B—C7B—H7BA	120.1
C13A—C14A—C15A	121.8 (3)	N1B—C8B—C9B	120.4 (3)
C13A—C14A—H14B	119.1	N1B—C8B—C5B	116.2 (3)
C15A—C14A—H14B	119.1	C9B—C8B—C5B	123.3 (2)
C16A—C15A—C20A	119.9 (3)	C8B—C9B—C10B	118.7 (3)
C16A—C15A—C14A	123.9 (3)	C8B—C9B—H9BA	120.6
C20A—C15A—C14A	116.2 (3)	C10B—C9B—H9BA	120.6
C17A—C16A—C15A	121.2 (4)	N2B—C10B—C9B	121.1 (3)
C17A—C16A—H16B	119.4	N2B—C10B—C13B	116.5 (2)
C15A—C16A—H16B	119.4	C9B—C10B—C13B	122.2 (3)
C16A—C17A—C18A	118.6 (3)	N2B—C11B—N1B	126.4 (3)
C16A—C17A—H17B	120.7	N2B—C11B—C12B	116.8 (3)
C18A—C17A—H17B	120.7	N1B—C11B—C12B	116.8 (3)
C19A—C18A—C17A	122.1 (4)	C11B—C12B—H12A	109.5
C19A—C18A—H18B	118.9	C11B—C12B—H12B	109.5
C17A—C18A—H18B	118.9	H12A—C12B—H12B	109.5
C18A—C19A—C20A	120.5 (4)	C11B—C12B—H12C	109.5
C18A—C19A—H19B	119.7	H12A—C12B—H12C	109.5
C20A—C19A—H19B	119.7	H12B—C12B—H12C	109.5
N3A—C20A—C15A	123.1 (3)	C11B—C12B—H12H	109.5
N3A—C20A—C19A	119.2 (3)	H12A—C12B—H12H	141.1
C15A—C20A—C19A	117.6 (3)	H12B—C12B—H12H	56.3
N3A—C21A—N4A	115.7 (4)	H12C—C12B—H12H	56.3
N3A—C21A—N4C	122.0 (13)	C11B—C12B—H12I	109.5
N3A—C21A—C13A	123.2 (3)	H12A—C12B—H12I	56.3
N4A—C21A—C13A	121.0 (4)	H12B—C12B—H12I	141.1
N4C—C21A—C13A	114.5 (12)	H12C—C12B—H12I	56.3
C21A—N4A—C22A	114.4 (5)	H12H—C12B—H12I	109.5
C21A—N4A—C25A	116.2 (5)	C11B—C12B—H12J	109.5
C22A—N4A—C25A	109.8 (4)	H12A—C12B—H12J	56.3
C26A—N5A—C24A	118.7 (4)	H12B—C12B—H12J	56.3
C26A—N5A—C23A	124.3 (4)	H12C—C12B—H12J	141.1
C24A—N5A—C23A	114.2 (3)	H12H—C12B—H12J	109.5
N4A—C22A—C23A	111.3 (4)	H12I—C12B—H12J	109.5
N4A—C22A—H22A	109.4	C14B—C13B—C21B	116.9 (3)
C23A—C22A—H22A	109.4	C14B—C13B—C10B	118.3 (3)
N4A—C22A—H22B	109.4	C21B—C13B—C10B	124.6 (3)
C23A—C22A—H22B	109.4	C13B—C14B—C15B	121.7 (3)
H22A—C22A—H22B	108.0	C13B—C14B—H14A	119.1
N5A—C23A—C22A	109.9 (4)	C15B—C14B—H14A	119.1
N5A—C23A—H23A	109.7	C20B—C15B—C16B	120.3 (3)
C22A—C23A—H23A	109.7	C20B—C15B—C14B	116.7 (3)
N5A—C23A—H23B	109.7	C16B—C15B—C14B	123.0 (3)
C22A—C23A—H23B	109.7	C17B—C16B—C15B	119.8 (3)
H23A—C23A—H23B	108.2	C17B—C16B—H16A	120.1
N5A—C24A—C25A	110.7 (4)	C15B—C16B—H16A	120.1
N5A—C24A—H24A	109.5	C16B—C17B—C18B	120.3 (3)
C25A—C24A—H24A	109.5	C16B—C17B—H17A	119.9

N5A—C24A—H24B	109.5	C18B—C17B—H17A	119.9
C25A—C24A—H24B	109.5	C19B—C18B—C17B	121.0 (4)
H24A—C24A—H24B	108.1	C19B—C18B—H18A	119.5
N4A—C25A—C24A	109.0 (4)	C17B—C18B—H18A	119.5
N4A—C25A—H25A	109.9	C18B—C19B—C20B	120.0 (3)
C24A—C25A—H25A	109.9	C18B—C19B—H19A	120.0
N4A—C25A—H25B	109.9	C20B—C19B—H19A	120.0
C24A—C25A—H25B	109.9	N3B—C20B—C15B	122.7 (3)
H25A—C25A—H25B	108.3	N3B—C20B—C19B	118.6 (3)
C26A—O3A—C27A	120.1 (4)	C15B—C20B—C19B	118.7 (3)
O2A—C26A—O3A	125.9 (4)	N3B—C21B—N4B	117.5 (3)
O2A—C26A—N5A	123.2 (4)	N3B—C21B—C13B	123.2 (3)
O3A—C26A—N5A	110.9 (4)	N4B—C21B—C13B	119.2 (3)
O3A—C27A—C29A	101.2 (4)	N4B—C22B—C23B	109.1 (3)
O3A—C27A—C30A	109.8 (4)	N4B—C22B—H22C	109.9
C29A—C27A—C30A	111.7 (6)	C23B—C22B—H22C	109.9
O3A—C27A—C28A	110.4 (5)	N4B—C22B—H22D	109.9
C29A—C27A—C28A	110.7 (6)	C23B—C22B—H22D	109.9
C30A—C27A—C28A	112.5 (6)	H22C—C22B—H22D	108.3
C27A—C28A—H28D	109.5	N5B—C23B—C22B	110.7 (2)
C27A—C28A—H28E	109.5	N5B—C23B—H23C	109.5
H28D—C28A—H28E	109.5	C22B—C23B—H23C	109.5
C27A—C28A—H28F	109.5	N5B—C23B—H23D	109.5
H28D—C28A—H28F	109.5	C22B—C23B—H23D	109.5
H28E—C28A—H28F	109.5	H23C—C23B—H23D	108.1
C27A—C29A—H29D	109.5	N5B—C24B—C25B	110.4 (3)
C27A—C29A—H29E	109.5	N5B—C24B—H24C	109.6
H29D—C29A—H29E	109.5	C25B—C24B—H24C	109.6
C27A—C29A—H29F	109.5	N5B—C24B—H24D	109.6
H29D—C29A—H29F	109.5	C25B—C24B—H24D	109.6
H29E—C29A—H29F	109.5	H24C—C24B—H24D	108.1
C27A—C30A—H30D	109.5	N4B—C25B—C24B	110.4 (2)
C27A—C30A—H30E	109.5	N4B—C25B—H25C	109.6
H30D—C30A—H30E	109.5	C24B—C25B—H25C	109.6
C27A—C30A—H30F	109.5	N4B—C25B—H25D	109.6
H30D—C30A—H30F	109.5	C24B—C25B—H25D	109.6
H30E—C30A—H30F	109.5	H25C—C25B—H25D	108.1
C21A—N4C—C25C	119.0 (17)	C26B—O3B—C27B	122.9 (3)
C21A—N4C—C22C	119.2 (17)	O2B—C26B—O3B	124.8 (3)
C25C—N4C—C22C	108.6 (12)	O2B—C26B—N5B	123.2 (3)
C26C—N5C—C24C	123.0 (11)	O3B—C26B—N5B	112.0 (3)
C26C—N5C—C23C	120.0 (11)	C30B—C27B—O3B	112.3 (4)
C24C—N5C—C23C	111.6 (11)	C30B—C27B—C28B	110.3 (5)
N4C—C22C—C23C	112.8 (12)	O3B—C27B—C28B	107.3 (4)
N4C—C22C—H22E	109.0	C30B—C27B—C29B	112.9 (5)
C23C—C22C—H22E	109.0	O3B—C27B—C29B	102.1 (3)
N4C—C22C—H22F	109.0	C28B—C27B—C29B	111.7 (5)
C23C—C22C—H22F	109.0	C27B—C28B—H28G	109.5

H22E—C22C—H22F	107.8	C27B—C28B—H28H	109.5
N5C—C23C—C22C	109.2 (12)	H28G—C28B—H28H	109.5
N5C—C23C—H23E	109.8	C27B—C28B—H28I	109.5
C22C—C23C—H23E	109.8	H28G—C28B—H28I	109.5
N5C—C23C—H23F	109.8	H28H—C28B—H28I	109.5
C22C—C23C—H23F	109.8	C27B—C29B—H29G	109.5
H23E—C23C—H23F	108.3	C27B—C29B—H29H	109.5
N5C—C24C—C25C	111.2 (12)	H29G—C29B—H29H	109.5
N5C—C24C—H24E	109.4	C27B—C29B—H29I	109.5
C25C—C24C—H24E	109.4	H29G—C29B—H29I	109.5
N5C—C24C—H24F	109.4	H29H—C29B—H29I	109.5
C25C—C24C—H24F	109.4	C27B—C30B—H30G	109.5
H24E—C24C—H24F	108.0	C27B—C30B—H30H	109.5
N4C—C25C—C24C	113.0 (12)	H30G—C30B—H30H	109.5
N4C—C25C—H25E	109.0	C27B—C30B—H30I	109.5
C24C—C25C—H25E	109.0	H30G—C30B—H30I	109.5
N4C—C25C—H25F	109.0	H30H—C30B—H30I	109.5

C1A—O1A—C2A—C7A	−3.2 (5)	C23C—N5C—C24C—C25C	55 (2)
C1A—O1A—C2A—C3A	176.0 (3)	C21A—N4C—C25C—C24C	−165 (3)
C7A—C2A—C3A—C4A	0.1 (5)	C22C—N4C—C25C—C24C	54 (2)
O1A—C2A—C3A—C4A	−179.2 (3)	N5C—C24C—C25C—N4C	−55 (2)
C2A—C3A—C4A—C5A	0.1 (5)	C27C—O3C—C26C—O2C	−2 (3)
C3A—C4A—C5A—C6A	0.0 (4)	C27C—O3C—C26C—N5C	171.3 (17)
C3A—C4A—C5A—C8A	179.8 (3)	C24C—N5C—C26C—O2C	−170 (2)
C4A—C5A—C6A—C7A	−0.4 (4)	C23C—N5C—C26C—O2C	−18 (3)
C8A—C5A—C6A—C7A	179.8 (3)	C24C—N5C—C26C—O3C	16 (3)
O1A—C2A—C7A—C6A	178.8 (3)	C23C—N5C—C26C—O3C	168.1 (19)
C3A—C2A—C7A—C6A	−0.4 (5)	C26C—O3C—C27C—C29C	−176.8 (17)
C5A—C6A—C7A—C2A	0.5 (5)	C26C—O3C—C27C—C30C	64 (2)
C11A—N1A—C8A—C9A	−1.7 (4)	C26C—O3C—C27C—C28C	−61 (2)
C11A—N1A—C8A—C5A	179.9 (2)	C1B—O1B—C2B—C3B	−2.6 (5)
C6A—C5A—C8A—N1A	−160.9 (3)	C1B—O1B—C2B—C7B	178.5 (3)
C4A—C5A—C8A—N1A	19.3 (4)	O1B—C2B—C3B—C4B	178.6 (3)
C6A—C5A—C8A—C9A	20.8 (4)	C7B—C2B—C3B—C4B	−2.7 (5)
C4A—C5A—C8A—C9A	−159.0 (3)	C2B—C3B—C4B—C5B	−1.0 (5)
N1A—C8A—C9A—C10A	1.1 (4)	C3B—C4B—C5B—C6B	3.7 (5)
C5A—C8A—C9A—C10A	179.4 (2)	C3B—C4B—C5B—C8B	−174.0 (3)
C11A—N2A—C10A—C9A	−0.6 (4)	C4B—C5B—C6B—C7B	−2.9 (4)
C11A—N2A—C10A—C13A	172.7 (2)	C8B—C5B—C6B—C7B	174.9 (3)
C8A—C9A—C10A—N2A	0.1 (4)	C3B—C2B—C7B—C6B	3.5 (5)
C8A—C9A—C10A—C13A	−172.7 (2)	O1B—C2B—C7B—C6B	−177.6 (3)
C10A—N2A—C11A—N1A	0.0 (5)	C5B—C6B—C7B—C2B	−0.7 (5)
C10A—N2A—C11A—C12A	−178.7 (3)	C11B—N1B—C8B—C9B	−1.0 (4)
C8A—N1A—C11A—N2A	1.2 (5)	C11B—N1B—C8B—C5B	−178.9 (3)
C8A—N1A—C11A—C12A	179.9 (3)	C4B—C5B—C8B—N1B	−174.6 (3)
N2A—C10A—C13A—C14A	−29.4 (4)	C6B—C5B—C8B—N1B	7.8 (4)
C9A—C10A—C13A—C14A	143.8 (3)	C4B—C5B—C8B—C9B	7.6 (4)

N2A—C10A—C13A—C21A	157.0 (3)	C6B—C5B—C8B—C9B	−170.1 (3)
C9A—C10A—C13A—C21A	−29.9 (4)	N1B—C8B—C9B—C10B	1.9 (4)
C21A—C13A—C14A—C15A	2.8 (4)	C5B—C8B—C9B—C10B	179.7 (3)
C10A—C13A—C14A—C15A	−171.5 (2)	C11B—N2B—C10B—C9B	0.3 (4)
C13A—C14A—C15A—C16A	−176.7 (3)	C11B—N2B—C10B—C13B	174.7 (3)
C13A—C14A—C15A—C20A	4.0 (4)	C8B—C9B—C10B—N2B	−1.6 (4)
C20A—C15A—C16A—C17A	0.1 (4)	C8B—C9B—C10B—C13B	−175.6 (2)
C14A—C15A—C16A—C17A	−179.2 (3)	C10B—N2B—C11B—N1B	0.7 (5)
C15A—C16A—C17A—C18A	2.2 (5)	C10B—N2B—C11B—C12B	−179.2 (3)
C16A—C17A—C18A—C19A	−2.0 (5)	C8B—N1B—C11B—N2B	−0.4 (5)
C17A—C18A—C19A—C20A	−0.5 (5)	C8B—N1B—C11B—C12B	179.6 (3)
C21A—N3A—C20A—C15A	3.1 (4)	N2B—C10B—C13B—C14B	−37.4 (4)
C21A—N3A—C20A—C19A	179.1 (3)	C9B—C10B—C13B—C14B	136.9 (3)
C16A—C15A—C20A—N3A	173.4 (3)	N2B—C10B—C13B—C21B	148.4 (3)
C14A—C15A—C20A—N3A	−7.2 (4)	C9B—C10B—C13B—C21B	−37.4 (4)
C16A—C15A—C20A—C19A	−2.6 (4)	C21B—C13B—C14B—C15B	3.9 (4)
C14A—C15A—C20A—C19A	176.8 (3)	C10B—C13B—C14B—C15B	−170.8 (2)
C18A—C19A—C20A—N3A	−173.4 (3)	C13B—C14B—C15B—C20B	2.4 (4)
C18A—C19A—C20A—C15A	2.8 (4)	C13B—C14B—C15B—C16B	−179.5 (3)
C20A—N3A—C21A—N4A	−176.8 (4)	C20B—C15B—C16B—C17B	−0.3 (5)
C20A—N3A—C21A—N4C	−168.5 (17)	C14B—C15B—C16B—C17B	−178.2 (3)
C20A—N3A—C21A—C13A	4.4 (4)	C15B—C16B—C17B—C18B	1.7 (5)
C14A—C13A—C21A—N3A	−7.3 (4)	C16B—C17B—C18B—C19B	−1.4 (5)
C10A—C13A—C21A—N3A	166.4 (2)	C17B—C18B—C19B—C20B	−0.2 (5)
C14A—C13A—C21A—N4A	173.9 (4)	C21B—N3B—C20B—C15B	3.7 (4)
C10A—C13A—C21A—N4A	−12.4 (6)	C21B—N3B—C20B—C19B	−179.6 (3)
C14A—C13A—C21A—N4C	166.0 (16)	C16B—C15B—C20B—N3B	175.4 (3)
C10A—C13A—C21A—N4C	−20.3 (17)	C14B—C15B—C20B—N3B	−6.5 (4)
N3A—C21A—N4A—C22A	119.7 (5)	C16B—C15B—C20B—C19B	−1.3 (4)
C13A—C21A—N4A—C22A	−61.4 (6)	C14B—C15B—C20B—C19B	176.8 (3)
N3A—C21A—N4A—C25A	−9.8 (7)	C18B—C19B—C20B—N3B	−175.3 (3)
C13A—C21A—N4A—C25A	169.0 (4)	C18B—C19B—C20B—C15B	1.6 (5)
C21A—N4A—C22A—C23A	167.0 (6)	C20B—N3B—C21B—N4B	−177.9 (2)
C25A—N4A—C22A—C23A	−60.2 (6)	C20B—N3B—C21B—C13B	3.3 (4)
C26A—N5A—C23A—C22A	147.7 (5)	C22B—N4B—C21B—N3B	−9.6 (4)
C24A—N5A—C23A—C22A	−51.5 (5)	C25B—N4B—C21B—N3B	123.5 (3)
N4A—C22A—C23A—N5A	54.0 (6)	C22B—N4B—C21B—C13B	169.3 (2)
C26A—N5A—C24A—C25A	−144.1 (4)	C25B—N4B—C21B—C13B	−57.7 (3)
C23A—N5A—C24A—C25A	54.0 (5)	C14B—C13B—C21B—N3B	−7.0 (4)
C21A—N4A—C25A—C24A	−167.2 (6)	C10B—C13B—C21B—N3B	167.3 (2)
C22A—N4A—C25A—C24A	61.0 (5)	C14B—C13B—C21B—N4B	174.1 (2)
N5A—C24A—C25A—N4A	−57.5 (5)	C10B—C13B—C21B—N4B	−11.5 (4)
C27A—O3A—C26A—O2A	4.9 (6)	C21B—N4B—C22B—C23B	−162.8 (2)
C27A—O3A—C26A—N5A	−175.2 (4)	C25B—N4B—C22B—C23B	61.5 (3)
C24A—N5A—C26A—O2A	8.5 (6)	C26B—N5B—C23B—C22B	−152.0 (3)
C23A—N5A—C26A—O2A	168.5 (4)	C24B—N5B—C23B—C22B	51.4 (4)
C24A—N5A—C26A—O3A	−171.4 (3)	N4B—C22B—C23B—N5B	−55.4 (4)
C23A—N5A—C26A—O3A	−11.4 (6)	C26B—N5B—C24B—C25B	153.4 (3)

C26A—O3A—C27A—C29A	176.5 (4)	C23B—N5B—C24B—C25B	−50.8 (4)
C26A—O3A—C27A—C30A	−65.3 (6)	C21B—N4B—C25B—C24B	161.7 (3)
C26A—O3A—C27A—C28A	59.2 (7)	C22B—N4B—C25B—C24B	−61.9 (3)
N3A—C21A—N4C—C25C	−15 (3)	N5B—C24B—C25B—N4B	54.8 (4)
C13A—C21A—N4C—C25C	171.5 (19)	C27B—O3B—C26B—O2B	3.2 (6)
N3A—C21A—N4C—C22C	121 (2)	C27B—O3B—C26B—N5B	−176.7 (3)
C13A—C21A—N4C—C22C	−52 (3)	C23B—N5B—C26B—O2B	13.4 (5)
C21A—N4C—C22C—C23C	163 (3)	C24B—N5B—C26B—O2B	168.3 (3)
C25C—N4C—C22C—C23C	−56 (2)	C23B—N5B—C26B—O3B	−166.7 (3)
C26C—N5C—C23C—C22C	149 (2)	C24B—N5B—C26B—O3B	−11.8 (5)
C24C—N5C—C23C—C22C	−56 (2)	C26B—O3B—C27B—C30B	−57.1 (6)
N4C—C22C—C23C—N5C	58 (2)	C26B—O3B—C27B—C28B	64.2 (5)
C26C—N5C—C24C—C25C	−151 (2)	C26B—O3B—C27B—C29B	−178.2 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7*B*—H7*BA*···O2*B*^{i}	0.93	2.40	3.141 (2)	136

Symmetry code: (i) *x*+1, *y*, *z*.

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Spectroscopic, thermodynamic properties and Fukui Function analysis of (4Z)-2-phenyl-1-{(E)-[4-propan-2-yl)benzylidene]amino}-4-[(thiophen-2-yl)methylidene]-1H-imidazol-5(4H)-one}

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ABSTRACT

In the present work, the compound (4Z)-2-Phenyl-1-{(E)-[4-propan-2-yl) benzylidene]amino)-4-[(thiophen-2-yl)methylidene]-1H-imidazol-5(4H)-one} is studied experimentally and theoretically. The calculations have been performed to compute optimized geometry, atomic charges and thermodynamic properties using Hartree Fock Theory (HF) and Density Functional Theory (DFT) with 6-311G basis set. The TD-DFT calculations were carried out using B3LYP with 6-311G basis set to determine the maximum absorption wavelength of the UV–Vis spectra for the title compound and have been compared with the experiment. Natural Bond orbital analysis has been carried out to study the stability of the molecule arises from hyperconjugative interactions; charge delocalization. The calculated HOMO-LUMO energies show that the charge transfer occurs within the molecule and its derivative. The HOMO-LUMO energies, the temperature dependence of the thermodynamic properties and the Fukui functions of the title compound are also studied. The vibrational analysis of the compound has also been carried out.

1. Introduction

The term imidazole was coined by the German chemist Arthur Rudolf Hantzsch in 1889 [1]. Imidazole (1, 3-diaza-2, 4-cyclopentadiene) is a planar five-member ring system with three carbon atoms and two nitrogen atoms and is classified as a diazole having non-adjacent nitrogen atoms. Imidazole ($C_3H_4N_2$) is an organic compound and it is white or colourless solid which is soluble in water and other polar solvents. It exists in two equivalent tautomeric forms because the hydrogen atom can be located on either of the two nitrogen atoms. It is a highly polar compound as evidenced by a calculated electric dipole of 3.67D [2]. It is included into many important biological molecules and the most common is the amino acid "histidine". Imidazole containing drugs have a broader scope in remedying various dispositions in clinical medicine [3]. Imidazole has become an important part of many pharmaceuticals such as certain antifungal drugs, the nitroimidazole series of antibiotics and the sedative midazolam [4–8]. Its synthesis used glyoxal and formaldehyde in ammonia to form imidazole [9] and its derivatives are very important in the field of medicinal chemistry. Medicinal properties of imidazoles include antibacterial, anticancer, antitubercular, antifungal, analgesic, antidiabetic and antimalarial [10–16]. One of the most vital features of imidazole derivatives is their use as material for treatment of denture stomatities [17,18]. The high beneficial properties of the imidazole associated drugs have encouraged the medicinal chemists to prepare a large number of new chemotherapeutic materials based on imidazole nucleus [19]. Imidazole derivatives are reported to be pharmacologically and physiologically active and it is used in the treatment of several diseases. Imidazoles as constituent are found in component in a large number of natural products and clinical active molecules i.e., a large numbers of medicines contain the imidazole ring, containing ketoconazole which are used to treat bacterial infections, gastric ulcers and fungal infections [20,21]. Due to their significance, it has become a suitable target for the synthetic and clinical uses. Because of its importance and no theoretical data available, a complete analysis of the title compound (4Z)-2-Phenyl-1-{(E)-[4-propan-2-yl)benzylidene]amino}-4-[(thiophen-2-yl)methylidene]-1H-imidazol-5(4H)-one} has been carried out in this paper.

2. Experimental details

The infrared spectrum of the compound was recorded in the

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spectral range of 400–4000 cm^{-1} by Shimadzu IR-Tracer with spectral resolution of 4 cm^{-1}. The ultraviolet absorption spectrum of title compound was carried out in the range 190–800 nm using Shimadzu UV-2600 Double beam spectrophotometer.

3. Computational details

The HF and DFT [22] methods are carried out using the basis set 6-311G on (4Z)-2-Phenyl-1-{(E)-[4-propan-2-yl)benzylidene] amino}-4-[(thiophen-2-yl)methylidene]-1H-imidazol-5(4H)-one}. All the theoretical calculations were performed using Gaussian 09 W [23] program. The molecular structural optimization of title compound and corresponding vibrational frequencies were computed by using Becke's three-parameter hybrid functional(B3) [24,25] for the exchange part and the Lee-Yang-Parr (LYP) correlation function [26] at 6-311G basis set. The optimized structural parameters were used for calculating vibrational frequencies, Mulliken charges, thermodynamic parameters and several other molecular properties. The electronic properties of the title compound such as HOMO-LUMO energies, absorption wavelengths and oscillator strengths were calculated by using time-dependent DFT method (TD-DFT) [27–29]. All computational calculations were compared with the observed ones.

4. Results and discussion

4.1. Geometric structure

The most optimized structural parameters (bond lengths and bond angles) of(4Z)-2-Phenyl-1-{(E)-[4-propan-2-yl) benzylidene]-imidazol-5(4H)-one} were calculated with HF and DFT methods using 6-311G basis set are shown in Table 1 and Table 2 along with the atom numbering scheme as shown in Fig. 1. We have compared our results with the reported results of Kamni et al. [30] obtained from X-ray diffraction method and tabulated in Tables 1 and 2. Tables 1 and 2 reveals that the theoretical values are in good agreement with the experimental values. The minimum energies obtained for structure optimization of title molecule calculated by HF/6-311G and B3LYP/6-311G are about −1556.47531471a.u. and − 1564.77813876 a.u. respectively. The

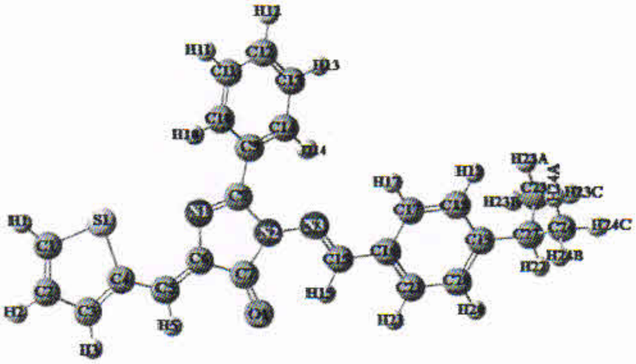

Fig. 1. Optimized structure of (4Z)-2-Phenyl-1-{(E)-[4-propan-2-yl)benzylidene] amino}-4-[(thiophen-2-yl)methylidene]-1H-imidazol-5(4H)-one}.

Table 1
Calculated Bond lengths (Å) of the molecule using HF/6-311G and B3LYP/6-311G basis set.

Atom no.	XRD	HF/6-311G	B3LYP/6-311G	Atom no.	XRD	HF/6-311G	B3LYP/6-311G
C2–C1	1.336	1.3438	1.3672	C14–C13	1.389	1.3863	1.3958
C3–C4	1.474	1.3552	1.3838	C13–C12	1.372	1.3859	1.3963
C1–S1	1.675	1.7797	1.7953	C12–C11	1.372	1.3878	1.3996
C5–C6	1.343	1.3309	1.3587	N3–C15	1.274	1.2716	1.3001
C6–N1	1.388	1.4061	1.4042	C15–C16	1.463	1.4679	1.4626
C7–O1	1.218	1.2239	1.248	C21–C20	1.376	1.3877	1.3948
C7–N2	1.405	1.385	1.4221	C21–C16	1.395	1.3883	1.4047
C8–N1	1.306	1.2851	1.3201	C21–H21	0.93	1.072	1.083
C8–N2	1.400	1.4103	1.4233	C19–C18	1.402	1.3982	1.4086
C8–C9	1.461	1.4683	1.4643	C18–C17	1.372	1.3797	1.3896
N2–N3	1.397	1.3741	1.3894	C18–H18	0.93	1.0708	1.0825
C10–C9	1.392	1.3952	1.4115	C22–C23	1.429	1.5371	1.5452
C10–C11	1.366	1.3829	1.3907	C22–C24	1.419	1.5371	1.5448
C9–C14	1.387	1.3927	1.4078	C22–H22	0.98	1.0837	1.0951

Table 2
Calculated Bond Angles of the molecule using HF/6-311G and B3LYP/6-311G basis set.

Structural parameters	XRD	HF/6-311G	B3LYP/6-311G	Structural parameters	XRD	HF/6-311G	B3LYP/6-311G(d,p)
C3–C2–C1	116.1	113.023	113.3744	N1–C8–C9	122.8	123.0759	122.415
C2–C3–C4	106.8	114.6109	114.8518	N2–C8–C9	124.7	125.388	126.4266
C4–C3–H3	126.6	122.2984	121.6533	C6–N1–C8	106.8	108.1382	107.929
C3–C4–S1	111.6	110.3785	109.9768	C7–N2–C8	108.4	108.6297	108.2419
C3–C4–C5	124.9	124.5397	125.8449	C7–N2–N3	126.4	129.0555	128.8795
S1–C4–C5	123.4	125.0818	124.1783	C8–N2–N3	123.5	121.7555	122.5191
C2–C1–S1	113.6	112.4775	112.5474	C9–C10–C11	121.6	120.4452	120.5899
C4–S1–C1	91.8	89.51	89.2497	C8–C9–C14	124.3	123.4228	124.1227
C4–C5–C6	128.0	128.9631	128.636	C12–C11–H11	120.2	120.2025	120.101
C5–C6–C7	123.8	124.1884	124.2456	N2–N3–C15	115.4	121.7101	120.1089
C5–C6–N1	126.2	127.63	126.2301	N3–C15–C16	120.2	120.3692	120.1381
C7–C6–N1	110.0	108.1812	109.5233	C21–C20–C19	121.4	121.0854	121.1497
C6–C7–O1	132.0	129.9192	130.5937	C20–C19–C22	121.0	120.6803	120.7171
C6–C7–N2	102.2	103.5165	103.1533	C18–C19–C22	121.3	121.4823	121.3844
O1–C7–N2	125.8	126.562	126.251	C18–C17–C16	121.1	120.4788	120.5304
N1–C8–N2	112.5	111.5342	111.1514	C21–C16–C17	117.6	118.5225	118.388

difference in energy between these two basis sets is very small.

4.2. Atomic net charges

The charge distribution on the molecule has an important influence on the vibrational spectrum. The total atomic charges of (4Z)-2-Phenyl-1-{(E)-[4-propan-2-yl)benzylidene]amino}-4-[(thiophen-2-yl)methylidene]-1H-imidazol-5(4H)-one} obtained by Mulliken and NBO methods [31and32] using HF/6-311G and B3LYP/6-311G level of theory are shown in Table 3. The atomic Mulliken charges have been obtained by the Mulliken population analysis and are based on the linear combination of atomic orbitals and therefore, wavefunction of the molecule. The details for the calculations of Mulliken charges can be found elsewhere [31] and the NBO charges can be found in Ref. [32]. It has been found that the atomic charges, Mulliken and NBO on all the hydrogen atoms exhibit positive charge values. The range of hydrogen atom charges in case of HF/6-311G for both the charges (Mulliken and NBO) is from 0.164 to 0.267 where as the range of hydrogen atom charges in case of B3LYP/6-311G are from 0.150 to 0.236 which are acceptor atoms. The charge distribution for the oxygen atom (O1) and nitrogen atoms (N1, N2, N3) present in the molecule have negative charge values which are donor atoms for both the methods.

4.3. FTIR spectroscopy and vibrational analysis

Molecule has 50 atoms so there are 144 vibrational modes. All the vibrational frequency calculations were carried out after optimizing the structure of the molecule to its minimum energy. The high accuracy prediction of vibrational calculations for a large number of compounds is done by DFT method [33]. Only those calculated frequencies are given in Table 4 for which the corresponding experimental frequencies are observed. Besides, theoretical vibrational spectra of the title compound were interpreted by means of potential energy distributions (PED) using VEDA program [34]. Electron correlations effects, inappropriate basis sets

Table 3
Mulliken and nbo charges using HF and DFT theory.

Atom NO.	MullikenHF/6-311G	MulikenDFT/6-311G	NBOHF/6-311G	NBODFT/6-311G
C2	−0.090089	−0.073359	−0.25672	−0.26250
C3	−0.062119	−0.024426	−0.17784	−0.20026
C4	−0.363059	−0.381713	−0.19792	−0.17699
C1	−0.475161	−0.471952	−0.29779	−0.31631
H3	0.194708	0.178697	0.20898	0.22124
H1	0.211584	0.198978	0.21516	0.22265
H2	0.193959	0.178658	0.21164	0.22253
S1	0.418157	0.441241	0.34102	0.37927
C5	0.050046	0.032247	−0.07679	−0.13475
H5	0.212080	0.193897	0.22047	0.23186
C6	−0.117349	−0.121126	0.01706	0.04380
C7	0.804678	0.587712	0.73707	0.59322
C8	0.494264	0.382273	0.54826	0.45276
O1	−0.544981	−0.397477	−0.68798	−0.59318
N1	−0.517034	−0.400335	−0.56160	−0.50970
N2	−0.885010	−0.725080	−0.36901	−0.28108
C10	−0.081643	−0.053336	−0.12544	−0.14839
C9	0.018975	−0.040613	−0.12628	−0.10690
C14	−0.037074	−0.006310	−0.13699	−0.16408
C13	−0.215765	−0.207544	−0.20600	−0.20442
C12	−0.116758	−0.104296	−0.15986	−0.17914
C11	−0.197209	−0.186361	−0.20389	−0.20421
H10	0.224982	0.214230	0.21943	0.23038
H14	0.217792	0.210454	0.21797	0.23468
H13	0.164432	0.150404	0.19520	0.20472
H12	0.167838	0.153886	0.19336	0.20323
H11	0.166857	0.152707	0.19639	0.20596
N3	−0.224477	−0.176136	−0.31222	−0.29031
C15	0.082322	−0.018257	0.14111	0.07352
H15	0.267050	0.236017	0.20953	0.21824
C21	−0.182748	−0.152853	−0.14642	−0.16399
C20	−0.227108	−0.184290	−0.20978	−0.20700
C19	0.131297	0.109416	0.01126	0.00203
C18	−0.186165	−0.154010	−0.20977	−0.20777
C17	−0.097696	−0.081255	−0.13868	−0.16085
C16	−0.072084	−0.053279	−0.11347	−0.09551
H21	0.176766	0.163522	0.19535	0.20679
H20	0.167121	0.153230	0.19417	0.20445
H17	0.205446	0.181747	0.21286	0.21985
H18	0.172755	0.157007	0.19314	0.20361
C22	−0.334766	−0.333182	−0.18027	−0.21059
C23	−0.456698	−0.484263	−0.50757	−0.56591
C24	−0.456885	−0.485628	−0.50755	−0.56644
H23A	0.168449	0.175106	0.17620	0.19681
H23B	0.165431	0.172677	0.17162	0.19175
H23C	0.176636	0.182453	0.18198	0.20178
H24A	0.168680	0.175334	0.17635	0.19720
H24B	0.175806	0.182118	0.18151	0.20161
H24C	0.164840	0.171766	0.17131	0.19119
H22	0.178927	0.181302	0.17146	0.19515

Table 4
Experimental and calculated (scaled) wavenumbers (in cm^{-1}) of (4Z)-2-Phenyl-1-{(E)-[4-propan-2-yl)benzylidene]-imidazol-5(4H)-one).

UNSCALED (B3LYP/6-311G)	SCALED (by 0.967)	Experimental	IR Intensities	Assignments and TED(≥10%, given in parenthesis)
3197	3088.302	3091	15.75	νCH (95)
3178	3069.948	3072	26.37	νCH (96)
3149	3041.934	3031	3.91	νCH (100)
3076	2971.416	2965	8.36	νCH (98)
3014	2911.524	2927	37.39	νCH (91)
3002	2899.932	2872	24.39	νCH (95)
1689	1631.574	1632	99.45	νCC (51)
1661	1604.526	1601	100	νOC (61)
1652	1595.832	1599	33.85	νCC(61)+ β HCC(18)
1615	1560.09	1567	5.16	νCC(62)
1564	1510.824	1519	7.28	νCC(60)+ β HCC(16)
1542	1489.572	1490	19.71	β HCH(71)+ τ HCCC(18)
1523	1471.218	1463	65.19	β HCC(35)+ νCC(17)
1488	1437.408	1448	35.85	β HCC(49)+ νCC(20)
1461	1411.326	1418	13.96	νCC(32)+ β HCC(45)
1437	1388.142	1389	27.8	β HCH(95)
1410	1362.06	1362	24.9	β HCC(52)+ νCC(12)
1387	1339.842	1341	31.66	β HCC(74)
1364	1317.624	1321	10.84	TORHCCC(78)
1291	1247.106	1256	41.88	νCC(33)+ β HCC(13)
1251	1208.466	1208	3.7	νCC(41)
1213	1171.758	1179	2.62	β HCC(77)
1167	1127.322	1120	55.82	νNC(34)
1130	1091.58	1096	6.62	νCC(40)
1114	1076.124	1078	12.39	νCC(29)+ β HCC(23)
1058	1022.028	1031	19.93	νCC(22)+ β HCC(21)
1045	1009.47	1010	23.38	τ HCNN(72)
1014	979.524	978	45.4	νNC(34)
994	960.204	966	6.99	τ HCCC(78)
979	945.714	945	12.59	τ HCCC(74)
953	920.598	918	5.46	τ HCCC(66)
899	868.434	874	12.35	ν CC(80)
884	853.944	853	12.92	τ HCCC(84)
858	828.828	832	7.94	β CCC(66)
814	786.324	798	27.16	νCC(11)+ τ HCCC(22)
807	779.562	776	36.37	τ HCCC(49)
740	714.84	723	40.07	β CCC(34)
711	686.826	687	18.51	β CCC(23)+ τ HCCC(16)
686	662.676	661	17.08	β CCC(34)+ β CCO(23)
649	626.934	619	7.91	β CCC(61)
601	580.566	584	13.44	τ CCCC(74)
437	441.462	450	3.98	β CCO(49)

ν – stretching, β – bending, γ→ out-of-plane deformation, τ → torsion.

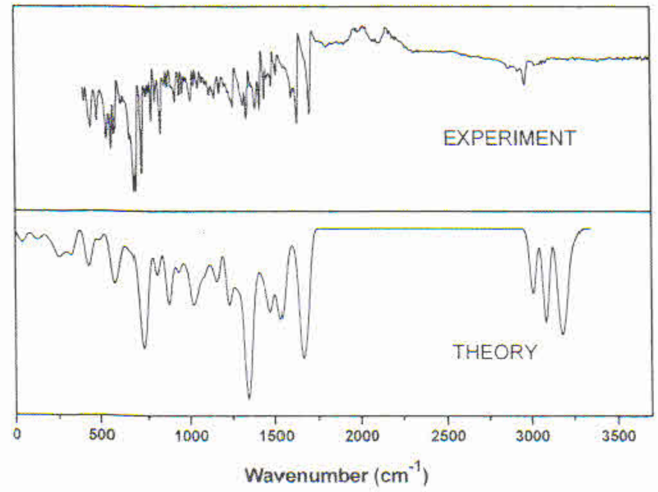

Fig. 2. Experimental and calculated FTIR spectra of (4Z)-2-Phenyl-1-[(E)-[4-propan-2-yl)benzylidene]amino}-4-[(thiophen-2-yl)methylidene]-1H-imidazol-5(4H)-one).

and anharmonicity are some of the important factors which results in higher values of calculated wavenumbers than the corresponding experimental ones. These discrepancies are removed either by introducing scalar field or by direct scaling of the calculated wavenumbers with a proper scaling factor [35,36]. The computed harmonic frequencies in the present study have been scaled by 0.966 in case of DFT/6-311G.The calculated frequency values are in close agreement with the experimental ones as shown in Table 4.

The experimental FTIR spectrum is presented in Fig. 2 along with calculated FTIR spectra using DFT method. The FT-IR spectra contain some characteristic bands of the stretching vibrations of the C–H, C–C, O–C, N–C groups. The aromatic structure shows the presence of C–H stretching vibrations in the region 3150–2900 cm^{-1}, which is the characteristic region for the identification of the C–H stretching vibrations. In this region, the band is not affected by the nature of the substituent [37]. In the present work, the experimental C–H stretching vibration were observed in the range 3087–2864 cm^{-1} and the computed scaled wavenumbers in the range 3088–2899 cm^{-1}. The carbonyl group has a strong C=O stretching vibration around 1850-1550 cm^{-1} [38]. In the present work experimental C=O stretching vibration is at

Table 5
Secor [illegible]rder perturbation theory analysis of Fock matrix in NBO basic corresponding to the intermolecular bonds of (4Z)-2-Phenyl-1-{(E)-[4-propan-2-yl)benzylidene]-imidaz[illegible]-5(4H)-one}.

Donor (i)	Type	ED(i)(e)	Acceptor (j)	Type	ED(j)(e)	E(2) (KJ/mol)[a]	E(j) - E(i) (a.u)[b]	F(i,j) (a.u)[c]
C2–C3	σ	1.97527	C2–C1	σ^*	0.01590	2.69	1.26	0.052
			C4–C5	σ^*	0.02254	4.73	1.20	0.067
C2–C1	π	1.98575	C2–C3	π^*	0.01533	2.84	1.26	0.053
		1.83684	C3–C4	π^*	0.35479	16.43	0.29	0.065
C2–H2	σ	1.97224	C3–C4	σ^*	0.01950	2.64	1.09	0.048
			C1–S1	σ^*	0.02913	5.50	0.68	0.055
C3–C4	π	1.97937	C4–C5	π^*	0.02254	4.10	1.25	0.064
		1.76205	C2–C1	π^*	0.28079	15.86	0.28	0.060
		1.76205	C5–C6	π^*	0.24561	22.39	0.30	0.074
C3–H3	σ	1.97070	C4–S1	σ^*	0.04695	6.50	0.66	0.059
C4–S1	σ	1.97446	C3–H3	σ^*	0.01662	4.11	1.05	0.059
C4–C5	σ	1.97809	C5–C6	σ^*	0.02145	5.13	1.32	0.074
C5–H5	σ	1.95950	C6–N1	σ^*	0.02547	7.90	0.94	0.077
C5–C6	π	1.97574	C4–C5	π^*	0.02254	4.48	1.27	0.067
		1.74145	C3–C2	π^*	0.35479	14.38	0.29	0.059
		1.74145	C7–O1	π^*	0.34771	26.88	0.25	0.076
		1.74145	C8–N1	π^*	0.31651	12.82	0.27	0.054
C6–C7	σ	1.97317	N2–N3	σ^*	0.03349	5.39	0.99	0.065
C6–N1	π	1.97561	C8–C9	π^*	0.03536	6.16	1.23	0.078
C8–N1	σ	1.86674	C5–C6	σ^*	0.24561	19.76	0.34	0.076
			C9–C14	σ^*	0.37624	8.30	0.35	0.051
S1	LP(2)	1.64548	C3–C4	π^*	0.35479	18.05	0.25	0.060
O1	LP(2)	1.86798	C7–N2	σ^*	0.10533	28.34	0.61	0.118
N2	LP(1)	1.60027	C7–O1	π^*	0.34771	52.18	0.26	0.105
C7–O1	π^*	0.34771	C5–C6	π^*	0.24561	42.37	0.05	0.076
C8–N1	σ^*	0.31651	C5–C6	σ^*	0.24561	32.11	0.03	0.053
			C9–C14	σ^*	0.37624	59.86	0.04	0.070
N3–C15	π^*	0.18905	C21–C16	π^*	0.37886	49.27	0.03	0.067

[a] E(2) means energy of hyperconjugative interactions.
[b] Energy difference between donor and acceptor i and j NBO orbitals.
[c] F(i,j) is the Fock matrix element between i and j NBO orbitals.

1606 cm^{-1} is very well coincide with the calculated wavenumber at 1604 cm^{-1} with 61% contribution. The bending vibrations at 660 cm^{-1}and 447 cm^{-1} experimentally and matches well with calculated at 662 cm^{-1} and 441 cm^{-1} respectively as shown in Table 4. The C=C stretching modes of the phenyl group are expected in the range from 1650 to 1200 cm^{-1} [39]. Therefore, the strong vibrational frequency bands at 1629, 1597, 1561, 1515, 1460, 1444, 1416, 1360, 1241, 1207 cm^{-1} are assigned to C→C stretching vibrations experimentally which coincides well with the calculated vibrational bands at 1631, 1595, 1560, 1510, 1471, 1437, 1411, 1362, 1247, 1208 cm^{-1}. The medium intensity band experimentally at 1096, 1075, 1027, 870 cm^{-1} are also attributed to C→C stretching and matches well with the calculated frequency value at 1091, 1076, 1022, 868 cm^{-1}. The FTIR bands appearing at 1119 and 974 cm^{-1} are assigned to the C–N stretching vibration and corresponding calculated wavenumbers at 1127 and 979 cm^{-1} respectively with contribution 34% as shown in Table 4.

4.4. Natural bond orbital analysis

NBO program implemented in the Gaussian 09 package at the DFT/B3LYP level was used for natural bond orbital analysis to understand various second-order interactions between the filled orbitals of one subsystem and vacant orbitals of another subsystem, which is a measure of the delocalization or hyperconjugation. The hyperconjugative interaction energy was deduced from the second-order perturbation approach [40]. Natural bond analysis provides an efficient method for studying intra and intermolecular bonding interactions among bonds. It also provides a convenient source for investigating charge transfer or conjugative interactions in molecular systems. It gives useful information about interactions in both filled and virtual orbital spaces, which could enhance the analysis of intra and intermolecular interactions [41]. For each donor NBO (i) and acceptor NBO (j), the stabilization energy E(2) associated with electron delocalization i→j is estimated as

$$E(2) = \Delta E_{ij} = q_i \ \frac{F(i,j)^2}{E_j \ - E_i}$$

Where q_i is the donor orbital occupancy, E_i, E_j are diagonal elements (orbital energies) and F(i,j) is the off-diagonal NBO Fock matrix element. Larger the E(2) value, the more intensive is the interaction between electron donors and electron acceptors i.e. the more donating tendency from electron donors to electron acceptors of the whole system. In this compound, the intramolecular interaction are formed by the orbital overlap between the bonding π (C2–C1) and the antibonding π* (C3–C4) orbital which results in intramolecular charge transfer (ICT) causing stabilization of the system. These interactions are observed as an increase in the electron density (ED) in the C5–C6 antibonding orbital that weakens the respective bond. The strong intramolecular hyperconjugation interaction of the π electrons from C3–C4 to the π* antibonding orbital of C5–C6 and π (C5–C6) to the π* antibonding orbital C7–O1 leads to the stabilization energy of 22.39 kJ/mol and 26.88 kJ/mol respectively. The most important interaction energies related to the resonance in the molecule, is the electron donating from the lone pair LP O1 atom to σ*(C7–N2) and LP N2 atom to π* (C7–O1) which leads to a stabilization energy of 28.34 kJ/mol and 52.18 kJ/mol respectively as shown in Table 5. Hence the charge transfer interactions explained above are responsible for the pharmaceutical and biological properties (Amoebicidal drugs, Antifungal drugs, Antithyroid drugs etc.) [42–45] of $C_{24}H_{21}N_3OS$.

4.5. UV–Vis spectral analysis

The most of the absorption spectroscopy of the organic

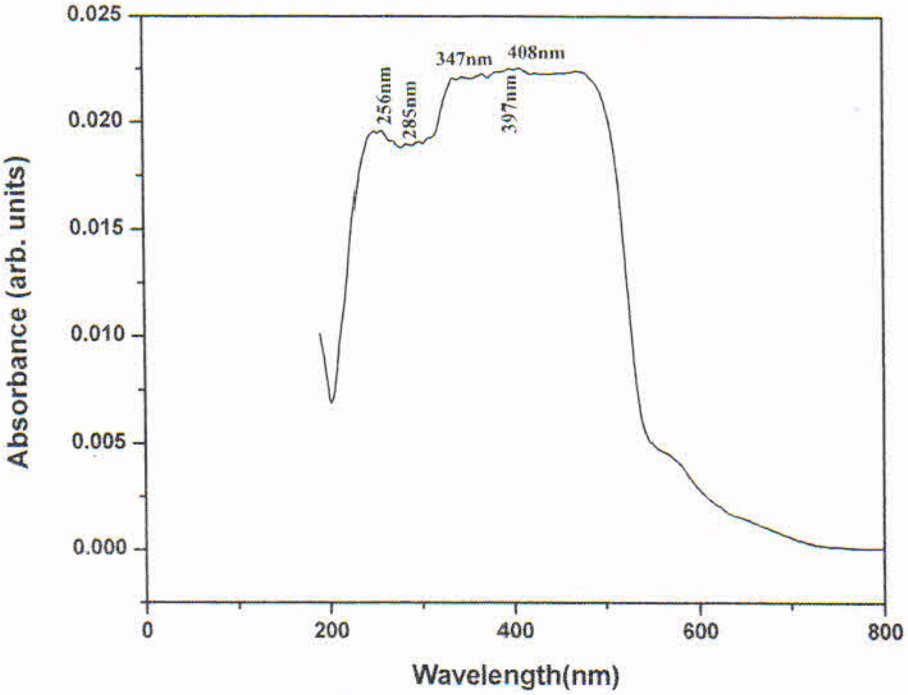

Fig. 3. Experimental UV–Vis of (4Z)-2-Phenyl-1-{(E)-[4-propan-2-yl)benzylidene]amino}-4-[(thiophen-2-yl)methylidene]-1H-imidazol-5(4H)-one}.

Table 6
Theoretical and experimental electronic absorption spectra values of (4Z)-2-Phenyl-1-{(E)-[4-propan-2-yl)benzylidene]-imidazol-5(4H)-one}using TD-DFT/6-311G method and their assignments.

Wavelength λ(**nm**)		Energy(eV)	Oscillator strength(f)	Assignments [58]
Experiment	Theory	Theory	Theory	
408	408.3	2.81	0.2086	HOMO →2 → LUMO+0 (+92%) +
397	382.3	3.24	0.6169	HOMO → 1 → LUMO+0 (+74%) + HOMO → 0 → LUMO+0 (+23%)
347	343.3	3.61	0.1302	HOMO → 0 → LUMO+1 (+91%)
	306.3	4.05	0.0054	HOMO → 2 → LUMO+1 (+82%)
285	285.1	4.35	0.0432	HOMO → 8 → LUMO+0(+63%) + HOMO → 7 → LUMO+0(+23%)
256	258.7	4.79	0.0047	HOMO → 3 → LUMO+1(+78%)

molecules are based on transitions $\pi \rightarrow \pi^*$ and $\sigma \rightarrow \sigma^*$ in the UV–Vis region [46,47]. The UV–Vis absorption spectrum of title molecule is shown in Fig. 3 in the spectral range 190–800 nm. The significant absorption peaks observed are at 256, 285, 347, 397 and 408 nm. The most intense UV bands are observed due to absorption from Highest Occupied Molecular Orbital and Lowest Unoccupied Molecular Orbital (HOMO to LUMO). In order to understand electronic transitions of compound, TD-DFT calculations on electronic absorption spectra were performed. Experimentally observed peaks are in good agreement with the theoretical values calculated by TD-DFT method. The values of wavelength, electronic excitation energies and oscillator strength calculated by TD-DFT method are tabulated in Table 6.

4.6. Thermodynamic properties

The density functional theory is a very well-established and efficient tool to predict various statistical thermodynamic properties of molecules which are important for the understanding the chemical processes. The values of some thermodynamic parameters (such as zero point vibrational energy, thermal energy, heat capacity, entropy, rotational constants, and dipole moment) of $C_{24}H_{21}N_3OS$ at 298.15 K in ground state are listed in Table 7. The ZPVE is lower by the DFT method than by the HF method. The biggest value of ZPVE of $C_{24}H_{21}N_3OS$ is 266.67652 kcal/mol obtained at HF/6-311G whereas the smallest value is 248.50302 kcal/mol obtained at B3LYP/6-311G. On the basis of vibrational analysis at B3LYP/6-311G level, the standard statistical thermodynamic functions: heat capacity($C^0_{p.m}$), entropy (S^0_m) and enthalpy(H^0_m) for the title compound were calculated within the temperature range from 100 to 700 K. From Fig. 4, it can be observed that these thermodynamic functions are increasing with temperature due to the fact that the molecular vibrational intensities increase with temperature [48]. The correlation equations between these thermodynamic functions and temperatures were fitted by quadratic formulas, and R is the corresponding fitting factors (R^2) for the thermodynamic properties. The corresponding fitting equations are as follows and the correlation graph of those show in Fig. 4.

$$C^0_{p.m} = 5.3411 + 0.34942T - 1.17588 \times 10^{-4}T^2 \quad \left(R^2 = 0.99845\right)$$

Table 7

Calculated thermodynamic parameters with HF and DFT theory using B3LYP function and 6-311G basis set.

Thermodynamic Parameters (298 K)	HF/6-311G	B3LYP/6-311G
SCF energy	−1556.47531472	−1564.77813876
Total energy (Thermal), E_{Total}(Kcal mol^{-1})	281.648	264.450
Vibrational energy, E_{vib}(Kcal mol^{-1})	279.870	262.673
Zero point vibrational energy, E_0(Kcal mol^{-1})	266.67652	248.50302
Heat Capacity, C_v(Cal/Mol-Kelvin)	90.443	97.753
Entropy, S (Cal/Mol-Kelvin)	173.604	180.117
Rotational Constants (GHZ)		
A	0.30114	0.29404
B	0.06185	0.06112
C	0.05227	0.05138
Dipole moment (Debye)		
μ_x	0.8104	0.3022
μ_y	0.5017	0.6935
μ_z	0.0527	0.0559
μ_{total}	0.9546	0.7585

$$S_m^0 = 68.59444 + 0.4036T - 9.76915 \times 10^{-5}T^2 \quad \left(R^2 = 0.99967\right)$$

$$H_m^0 = 247.84772 + 0.01659T + 1.34085 \times 10^{-4}T^2 \quad \left(R^2 = 0.99983\right)$$

4.7. HOMO-LUMO analysis

The highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) are known as frontier molecular orbitals (FMOs). Chemical stability is mainly influenced by the frontier orbitals (HOMO and LUMO). The FMOs plays an important role in the optical and electrical properties as well as in quantum chemistry [49]. The HOMO represents electron-donating ability, while the LUMO represents electron accepting ability [50]. The HOMO and LUMO energies are very useful for physicists and chemists and are very important terms in quantum chemistry [51]. The pictorial representation of energies of molecular orbitals with molecular formula ($C_{24}H_{21}N_3OS$) is shown in Fig. 5 (positive phase is represented by the red colour and negative phase represented in green colour). The HOMO lying at − 5.82 eV and spreads approximately over the entire molecule whereas the LUMO located at −2.79 eV with large anti-bonding character which shows that the eventual charge transfer occurs within the molecule and the frontier orbital energy gap is 3.04 eV.

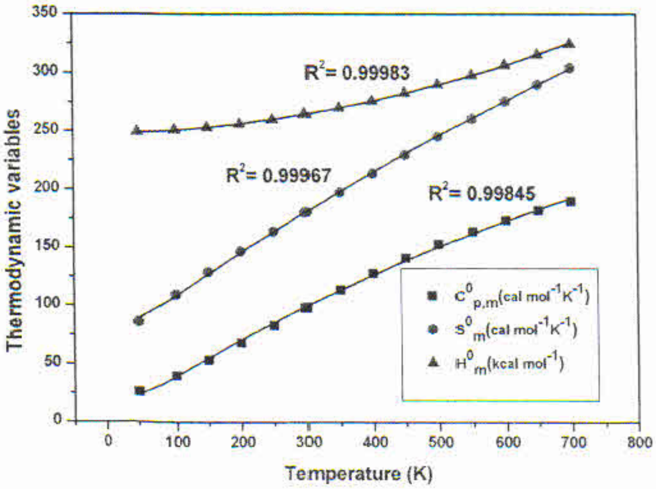

Fig. 4. Correlation graph between thermodynamic variables with temperature.

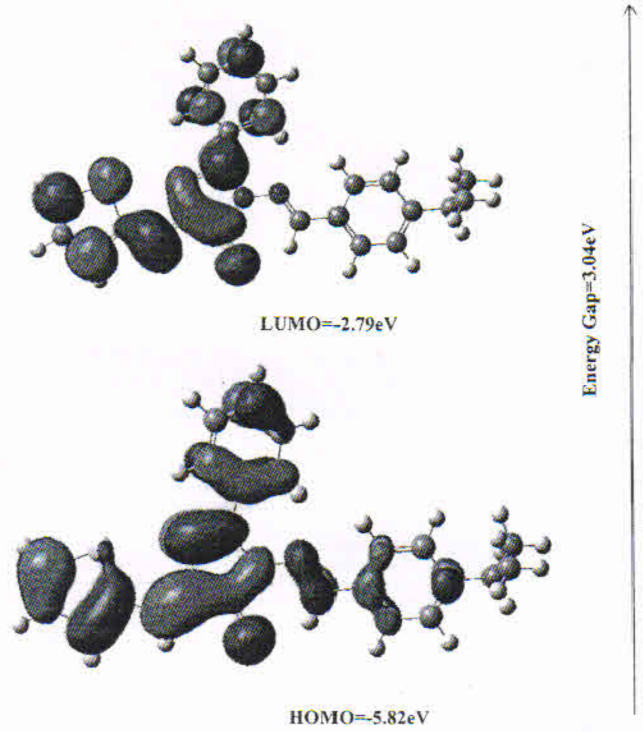

Fig. 5. HOMO-LUMO diagrams of (4Z)-2-Phenyl-1-{(E)-[4-propan-2-yl)benzylidene] amino}-4-[(thiophen-2-yl)methylidene]-1H-imidazol-5(4H)-one}.

The HOMO and LUMO studies of the present compound have been compared with its another derivative namely, (4Z)-1-[(E)-[4-methoxybenzylidene]amino}-2-phenyl-4-[(thiophen-2-yl)methylidene]-1H-imidazol-5(4H)-one}($C_{22}H_{17}N_3O_2S$). It has been found that for $C_{22}H_{17}N_3O_2S$, the HOMO lying at −5.76eV and LUMO located at −2.76eV and the frontier orbital energy gap is 3.0eV.

As both the HOMO and LUMO orbitals are the main orbital that helps to describe the chemical reactivity and kinetic stability of molecule. The HOMO and LUMO energies, the energy gap (ΔE), the ionization potential (I), the electron affinity (A), global hardness (η) chemical potential (μ), global electrophilicity (ω) for the title

Table 8
HOMO-LUMO and other related molecular properties of title compound (4Z)-2-Phenyl-1-{(E)-[4-propan-2-yl)benzylidene]amino}-4-[(thiophen-2-yl)methylidene]-1H-imidazol-5(4H)-one}.

Molecular parameters (eV)	B3LYP/6-311G
E_{LUMO}	−2.79
E_{HOMO}	−5.82
E_{LUMO}- E_{HOMO}	3.04
Ionization potential (I)	5.82
Electron affinity (A)	2.79
Global hardness (η)	1.52
Chemical potential (μ)	4.3
Global Electrophilicity (ω)	6.08

molecule and its derivative (4Z)-1-[(E)-[4-methoxybenzylidene] amino}-2-phenyl-4-[(thiophen-2-yl)methylidene]-1H-imidazol-5(4H)-one} have been calculated at DFT/6-311G level and the results are given in Table 8 and Table 9 respectively. There is a small decrease in all the above mentioned values for the derivative as compared to the title compound.

4.8. Fukui Function

Density Functional Theory is a powerful tool for the study of reactivity and selectivity in a molecule [52]. The most basic and commonly used local reactivity parameter is the Fukui function indicates tendency of electron density to deform at a given position upon accepting or donating electrons [53,54]. Fukui function gives us information about electrophilic/nucleophilic power of a given atomic site in a molecule. The condensed Fukui functions on the jth atom site can be expressed as:

Table 9
HOMO-LUMO and other related molecular properties of its derivative (4Z)-1-[(E)-[4-methoxybenzylidene]amino}-2-phenyl-4-[(thiophen-2-yl) methylidene]-1H-imidazol-5(4H)-one}.

Molecular parameters (eV)	B3LYP/6-311G
E_{LUMO}	−2.76
E_{HOMO}	−5.76
E_{LUMO}- E_{HOMO}	3.0
Ionization potential (I)	5.76
Electron affinity (A)	2.76
Global hardness (η)	1.5
Chemical potential (μ)	4.26
Global Electrophilicity (ω)	6.05

$$f_j^+ = q_j(N+1) - q_j(N)$$

$$f_j^0 = \frac{1}{2}\left[q_j(N+1) - q_j(N-1)\right]$$

$$f_j^- = q_j(N) - q_j(N-1)$$

where, f_j^+ for nucleophilic attack, f_j^- for electrophilic attack and f_j^0 for free radical. In these equations, q_j is the atomic charge at the jth atomic site in the neutral (N), anionic (N+1) or cationic (N-1) chemical species. The atomic charges either calculated by natural population analysis (NPA) or by Mulliken population analysis (MPA) have been used to calculate the Fukui function. In the present study the values of Fukui Function calculated from the NBO charges. Dual descriptor Δf(r) [55] for the calculation of nucleophilicity and electrophilicity is defined as the difference between the nucleophilic and electrophilic Fukui function and is given by the equation:

$$\Delta f(r) = f_j^+ - f_j^-$$

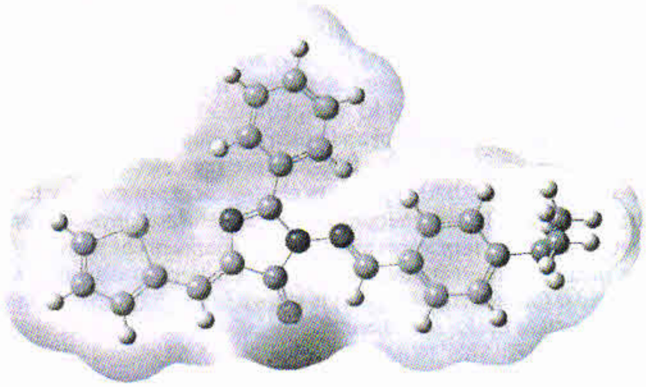

Fig. 6. Molecular electrostatic potential map of the title compound (4Z)-2-Phenyl-1-{(E)-[4-propan-2-yl)benzylidene]amino}-4-[(thiophen-2-yl)methylidene]-1H-imidazol-5(4H)-one}.

Table 10
Fukui indices for nucleophilic and electrophilic attacks on atoms calculated from natural population analysis at DFT/6-311G.

Atom	f^+	f^-	f^0	Δf(r)
C2	0.16632	−0.14712	0.0096	0.31344
C3	−0.04946	−0.09208	−0.07077	0.04262
C4	0.13748	−0.07265	0.032415	0.21013
C1	0.02053	−0.13522	−0.05735	0.15575
H3	−0.11767	0.10097	−0.00835	−0.21864
S1	−0.25445	0.16504	−0.04471	−0.41949
C5	−0.14624	−0.06668	−0.10646	−0.07956
H5	−0.122	0.10712	−0.00744	−0.22912
C6	−0.03633	0.0424	0.003035	−0.07873
O1	0.2072	−0.25529	−0.02405	0.46249
N1	0.25142	−0.1938	0.02881	0.44522
N2	0.15536	−0.13022	0.01257	0.28558
C10	0.03047	−0.07171	−0.02062	0.10218
C9	0.0745	−0.03736	0.01857	0.11186
N3	0.15412	−0.12371	0.015205	0.27783
C15	−0.05571	0.01617	−0.01977	−0.07188
C33	−0.0149	0.00076	−0.00707	−0.01566
C23	0.28301	−0.2812	0.000905	0.56421
C24	0.2833	−0.28155	0.000875	0.56485
H23A	−0.10316	0.09171	−0.00573	−0.19487

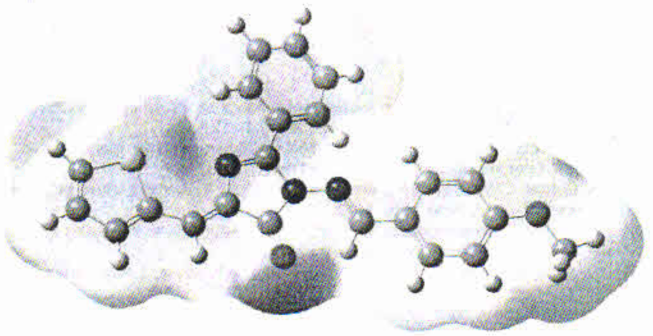

Fig. 7. Molecular electrostatic potential map of its derivative (4Z)-1-[(E)-[4-methoxybenzylidene]amino]-2-phenyl-4-[(thiophen-2-yl)methylidene]-1H-imidazol-5(4H)-one}.

If $\Delta f(r) > 0$, then the site is prone for nucleophilic attack, if $\Delta f(r) < 0$, then the site is for electrophilic attack. In the title compound, the order of the reactive sites for electrophilic attack, nucleophilic attack and free radical attacks is given in Table 10. For this molecule, C2,C3,C4,C1,C9,C23,C24, O1 and all nitrogen atoms are prone for nucleophilic attack as $\Delta f(r) > 0$ whereas hydrogen atoms, S1 and C5,C6,C15, C19 are prone for electrophilic attack as $\Delta f(r) < 0$.

4.9. Molecular electrostatic potential

Molecular electrostatic potential map is constructed using DFT/6-311G level of theory for the title compound and its derivative (4Z)-1-[(E)-[4-methoxybenzylidene]amino}-2-phenyl-4-[(thiophen-2-yl)methylidene]-1H-imidazol-5(4H)-one} and are shown pictorial in Fig. 6 and Fig. 7. The MEP is a 3D-plot of electrostatic potential mapped onto electron density surface which depict the size, shape, charge density and site of chemical reactivity of the molecules. In the colour scheme of MEPs, red represents electron rich, partially negative charge which is the preferred site for electrophilic attack, blue corresponds to electron deficient, partially positive charge which is preferred site for nucleophilic attack, yellow for slightly electron rich region; green for neutral respectively. Molecular electrostatic potential mapping is very useful in the investigation of the molecular structure with its physiochemical property relationships [56,57]. The colour code of this map is in the range between -5.177×10^{-2}(deepest red) to 5.177×10^{-2} (deepest blue). It can be seen that the negative regions are mainly over the oxygen atom and nitrogen atoms and the positive potential sites are around the hydrogen atoms.

The MEP map of the present compound have been compared with its derivative namely, (4Z)-1-[(E)-[4-methoxybenzylidene] amino}-2-phenyl-4-[(thiophen-2-yl)methylidene]-1H-imidazol-5(4H)-one}. It has been found that for its derivative ($C_{22}H_{17}N_3O_2S$), the colour code of this map is in the range between -5.187×10^{-2}(deepest red) to 5.187×10^{-2} (deepest blue). The maps for the derivative of the title compound show similar nature (negative regions over the oxygen atom and on the nitrogen atoms and the positive regions around the hydrogen atoms).

5. Conclusions

(4Z)-2-Phenyl-1-{(E)-[4-propan-2-yl)benzylidene]amino}-4-[(thiophen-2-yl)methylidene]-1H-imidazol-5(4H)-one}have been characterized by FTIR, UV–Vis and XRD. The complete structural, vibrational, NBO, HOMO-LUMO analysis, and the thermodynamic properties of the title molecule were performed and analyzed by HF and DFT methods at B3LYP/6-311G basis set. NBO analyses exhibit hyper conjugative interaction and stabilization of the molecule. The correlation graphs show that the heat capacities, entropies and enthalpies increase with increasing temperature. The Fukui functions are evaluated to describe the possible sites of nucleophilic and electrophilic attacks in the molecule. Electrostatic potential map shows that the oxygen atom show negative MEP while all the hydrogen atoms show positive MEP.

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