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Crystal structure analysis of (2'R,3'R,4'R)-3'-(1H-benzo[d]imidazol-2-yl)-4'-(4-bromophenyl)-1'-methyl-2-oxospiro[indoline-3,2'-pyrrolidine]-3'-carbonitrile

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Abstract : The crystal structure of(2'R,3'R,4'R)-3'-(1H-benzo[d]imidazol-2-yl)-4'-(4-bromophenyl)-1'-methyl-2-oxospiro[indoline-3,2'-pyrrolidine]-3'-carbonitrile ($C_{27}H_{26}BrN_5O_3$). The compound crystallizes in Monoclinic, P21/nspace group with unit cell parameters at 296(2) K as follows: a = 18.660 (2) Å, b =7.5092 (7) Å, c =19.001 (2) Å, α = β =90°, γ =110.26°.Crystal data were collected using BRUKER SMART APEX II CCD X-ray diffractometer. The structure was solved by direct methods and refined on F^2 by full-matrix least-squares procedures to the final R_1 of 0.089usingSHELXL programs.
Key Words : oxoindoline, pyrrolidine and crystal structure.

Introduction

Indole containing compounds are best known for their medicinal properties in the pharmaceutical industry. In modern times, analogs based on indole are significant players in a diverse array of markets such as dyes, plastics,agriculture, vitamin supplements, over-the-counterdrugs, flavour enhancers and perfumery[1]. Several indole derivatives, such as sunitinib as tyrosine kinase inhibitor[2] or delavirdine as nonnucleoside reverse transcriptase inhibitor[3], are in clinical use. spiroindole are important heterocyclic compounds with diversebioactivities[4,5].

Experimental

X-ray Structure Determination

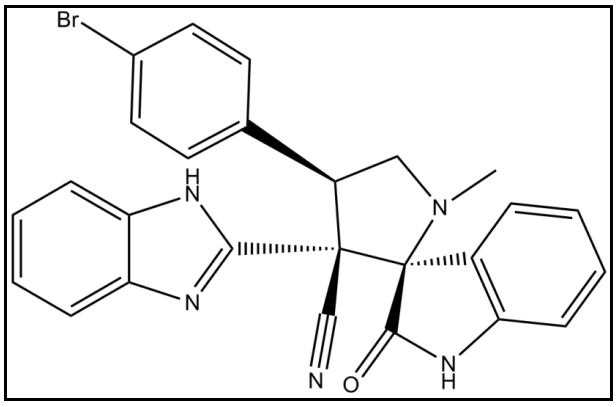
Single crystal of the compound suitable for x-ray diffraction was obtained by slow evaporation method. Three dimensional intensity data were collected on a Bruker[6] SMART APEX CCD Diffractometer using graphite monochromatized Mo-Kα radiation (λ= 0.71073 Å) at Department of chemistry, IIT, Chennai, India. The structure was solved by direct methods and refined on F^2 by full-matrix least-squares procedures using the SHELXL programs[7]. All the non-hydrogen atoms were refined using isotropic and later anisotropic thermal parameters. The hydrogen atoms were included in the structure factor calculation at idealized positions by using a riding model, but not refined. Images were created with ORTEP-3[8].The crystallographic data for the compound are listed in Table 1.

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

Compound	**Parameters**
Empirical formula	C_{27} H_{26} Br N_5 O_3
Formula weight	548.44
Temperature	296(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P21/n
Unit cell dimensions	a = 18.660(2) Å alpha = 90°. b = 7.5092(7) Å beta = 110.267(5) ° c = 19.001(2) Å gamma = 90°.
Volume	2497.6(4) $Å^3$
Z, Calculated density	4, 1.459 Mg/m^3
Absorption coefficient	1.685 mm^{-1}
F(000)	1128
Crystal size	0.25 x 0.20 x 0.15 mm
Theta range for data collection	1.32 to 25.00°.
Limiting indices	-22<=h<=22, -6<=k<=8, -22<=l<=22
Reflections collected / unique	17839 / 4346 [R(int) = 0.0450]
Completeness to theta = 25.00	99.10%
Max. and min. transmission	0.7861 and 0.6780
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4346 / 0 / 347
Goodness-of-fit on F^2	1.181
Final R indices [I>2sigma(I)]	R1 = 0.0603, wR2 = 0.2383
R indices (all data)	R1 = 0.0807, wR2 = 0.2523
Largest diff. peak and hole	0.730 and -0.779 e.$Å^3$

Synthesis of the compound

A mixture of isatin(1 mmol), sarcosine (1 mmol) and-imidazol-2-yl-3-phenylacrylonitrile (1 mmole) in ethanol (3 mL) was refluxed for 5-8 h and cooled to r.t. The crude products were purified by column chromatography to obtain pure product in good yield (75%). . The scheme diagram is given below.



Results and Discussion

The symmetric unit of the title compound is shown in Fig. 1. The pyrrole ring (N3/C9-C11) is Envelope conformation with puckering parameters[9], Q = 0.439Å and ϕ = 186.0(10)°.The pyrrolidine ring is almost orthogonal to the phenyl ring and two indole rings, making a dihedral angle of 34.8(7)°, 3.0(6)° and 74.5(6)°, respectively. The phenylring Br atom is deviating from the mean plane of -0.093Å.

In the crystal, molecules are linked by pairs of N---H...O hydrogen bonds, forming inversion dimers with an $R^2_2(12)$ ring motif, forming chains along [010] (Fig 2& Table 2). The crystal packing is further stabilized by C---H...***π***and ***π***---***π***intermolecular interactions.The selected bond lengths and angles are listed in table 3 and 4, respectively.

Table 2: Hydrogen-bond geometry [Å]

Distance (Å)				Angle (º)
D—H…A	D—H	H…A	D…A	D—H…A
N1---H1…O4^{i}	0.86	2.09	2.876(15)	151
N2---H2…O1^{ii}	0.86	2.14	2.949(13)	157
N3---H3…O1^{iii}	0.86	2.16	2.841(14)	136

Symmetry code: i) 1+x,-1+y,z, ii) 2-x,1/2+y,1/2-z iii) -1/2+x,y,1/2-z

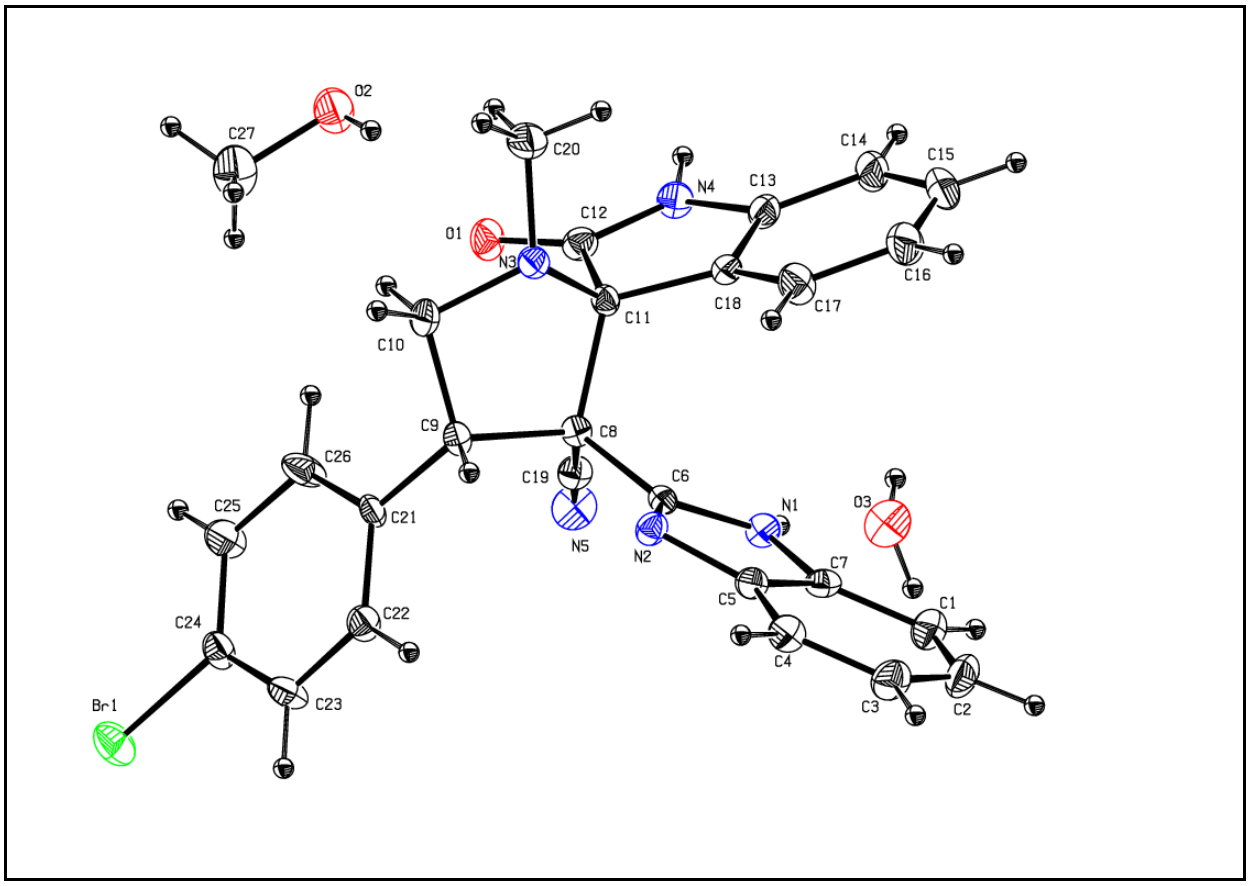


Fig.1.The molecular structure of the title compound, with the atom-numbering scheme. The displacement ellipsoids are drawn at 30% probability level. H atoms are shown as spheres of arbitrary radius.

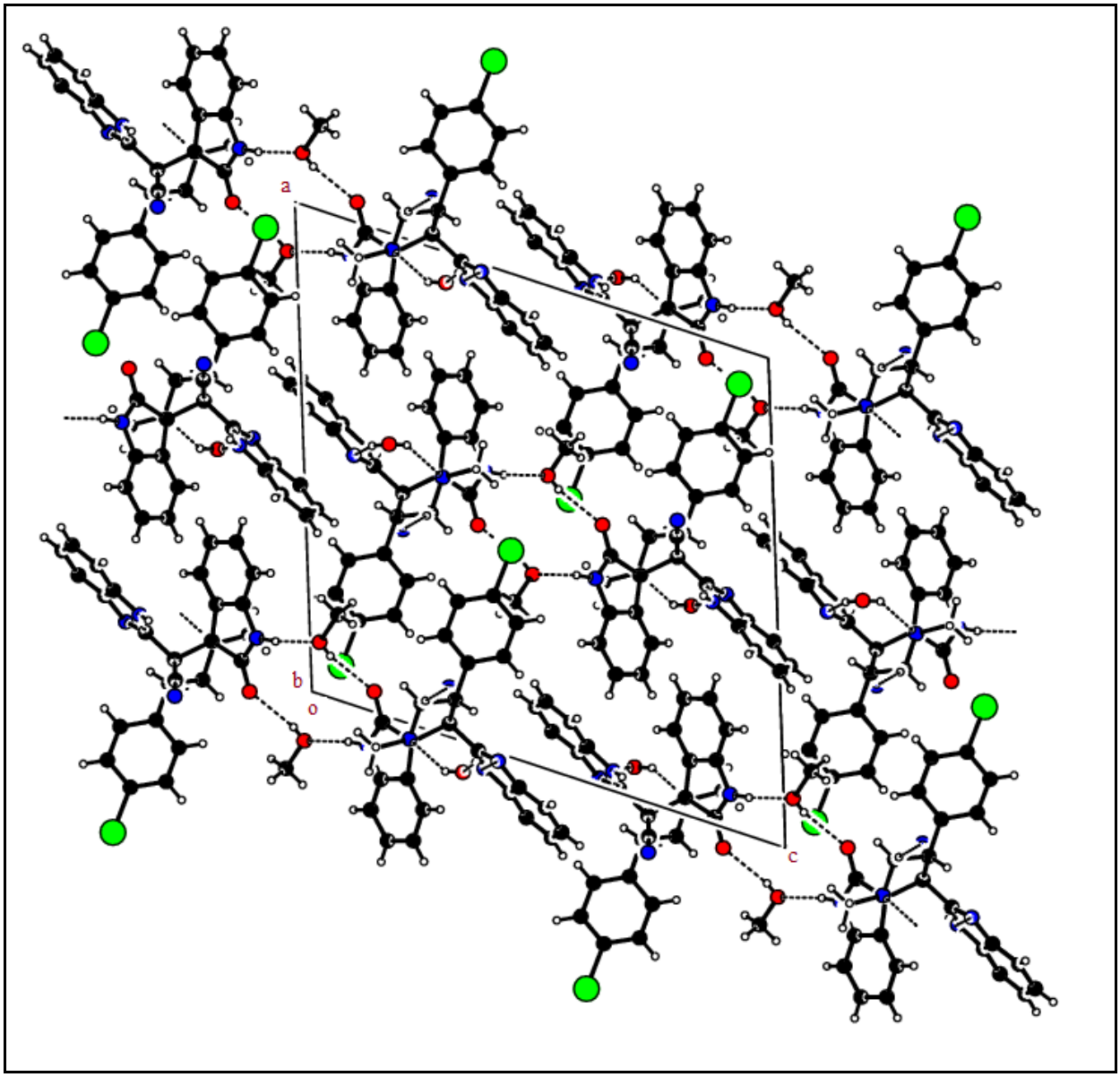


Fig.2. The crystal packing of the title compound, viewed along b axis, showing N---H...O hydrogen bonds.The hydrogen bonds are shown as dashed lines(see Table 2 for details).

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

Bond	Length (Å)
Br(1)-C(24)	1.869(7)
C(1)-C(7)	1.379(10)
C(1)-C(2)	1.384(11)
C(1)-H(1)	0.93
C(2)-C(3)	1.398(12)
C(2)-H(2)	0.93
C(3)-C(4)	1.358(11)
C(3)-H(3)	0.93
C(4)-C(5)	1.405(9)
C(4)-H(4)	0.93
C(5)-N(2)	1.391(9)
C(5)-C(7)	1.397(10)
C(6)-N(2)	1.311(9)
C(6)-N(1)	1.365(9)
C(6)-C(8)	1.521(9)
C(7)-N(1)	1.383(9)
C(8)-C(19)	1.486(10)
C(8)-C(9)	1.577(9)
C(8)-C(11)	1.602(9)
C(9)-C(21)	1.511(9)
C(9)-C(10)	1.519(9)
C(9)-H(9)	0.98
C(10)-N(3)	1.473(9)
C(10)-H(10A)	0.97
C(10)-H(10B)	0.97
C(11)-N(3)	1.461(9)
C(11)-C(18)	1.503(9)
C(11)-C(12)	1.556(9)
C(12)-O(1)	1.228(8)
C(12)-N(4)	1.338(9)
C(13)-C(14)	1.376(10)
C(13)-C(18)	1.390(10)
C(13)-N(4)	1.413(9)
C(14)-C(15)	1.399(12)
C(14)-H(14)	0.93
C(15)-C(16)	1.383(13)

Bond	Angle (º)
C(7)-C(1)-C(2)	116.1(7)
C(7)-C(1)-H(1)	122
C(2)-C(1)-H(1)	122
C(1)-C(2)-C(3)	121.8(7)
C(1)-C(2)-H(2)	119.1
C(3)-C(2)-H(2)	119.1
C(4)-C(3)-C(2)	121.7(7)
C(4)-C(3)-H(3)	119.1
C(2)-C(3)-H(3)	119.1
C(3)-C(4)-C(5)	117.7(7)
C(3)-C(4)-H(4)	121.2
C(5)-C(4)-H(4)	121.2
N(2)-C(5)-C(7)	110.5(6)
N(2)-C(5)-C(4)	129.8(7)
C(7)-C(5)-C(4)	119.7(6)
N(2)-C(6)-N(1)	113.9(6)
N(2)-C(6)-C(8)	123.8(6)
N(1)-C(6)-C(8)	122.2(6)
C(1)-C(7)-N(1)	132.1(7)
C(1)-C(7)-C(5)	122.9(7)
N(1)-C(7)-C(5)	105.0(6)
C(19)-C(8)-C(6)	107.5(5)
C(19)-C(8)-C(9)	112.3(5)
C(6)-C(8)-C(9)	111.1(5)
C(19)-C(8)-C(11)	111.1(5)
C(6)-C(8)-C(11)	111.3(5)
C(9)-C(8)-C(11)	103.5(5)
C(21)-C(9)-C(10)	116.7(6)
C(21)-C(9)-C(8)	115.4(6)
C(10)-C(9)-C(8)	104.4(5)
C(21)-C(9)-H(9)	106.5
C(10)-C(9)-H(9)	106.5
C(8)-C(9)-H(9)	106.5
N(3)-C(10)-C(9)	102.9(5)

Conclusion

The crystal structure analysis of a novel oxoindoline andpyrrolidinecompound was studied using x-ray diffraction method. In the crystal, molecules are linked by pairs of N---H...O hydrogen bonds, forming

inversion dimers with an $R^2_2(12)$ ring motif, forming chains along [010].The crystal packing is further stabilized by C---H...$\pi$and π---π intermolecular interactions.

Supplementary Material

Crystallographic data (excluding structure factors) for thestructure reported in this paper have been deposited withthe Cambridge Crystallographic Data Centre as supplementarypublication no.CCDC-**1020838**. Copies ofavailable material can be obtained, free of charge, onapplication to the Director, CCDC, 12 Union Road, CambridgeCB2 1EZ, UK (fax: +44-1223-336033 or e-mail:deposit@ccdc.cam.ac.uk).

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