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Synthesis and Crystal Structure of 4-(1,3-Diphenyl-1*H*-pyrazol-5-yl)pyridine

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The title compound, $C_{20}H_{15}N_3$, was synthesized and the structure was investigated by X-ray crystallography. The compound crystallizes in the monoclinic crystal class in the space group $P2_1/c$ with cell parameters a = 10.5700(6)Å, b = 12.2810(13)Å, c = 12.1750(13)Å, β = 94.429(7)° and Z = 4.

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Pyrazoles are a class of important heterocycles that have been attracting immense interest due to their wide range of applications in the pharmaceutical as well as in the agrochemical industry. Many pyrazole derivatives are known to exhibit a wide range of biological properties such as antiviral,[1] antifungal,[2] antiinflammatory,[3] antiarrhythmic and sedative[4] activities. Numerous compounds containing the pyrazole moiety have been shown to exhibit hypoglycemic,[5] inhibitors of Hsp90,[6] and pesticidal[7] properties. Therefore, continuous efforts have been devoted to the development of more general, efficient, and regioselective methods for the synthesis of this class of compounds and their conformational studies.

A mixture of (*E*)-3-phenyl-1(pyridin-4-yl)prop-2-en-1-one (2 g, 0.01 mol) and phenyl hydrazine (1.08 g, 0.01 mol) in acetonitrile (30 ml) was refluxed for 4 h, distilled completly and poured into water and extracted to dichloromethane. The dichloromethane was dried using anhydrous sodium sulphate, filtered, distilled completly and purified using column chromotogrophy (hexane:ethyl acetate). Yield, 71% (yellow color solid); MP, 124°C. Figure 1 is a schematic diagram of the molecule.

A single crystal of the title compound with dimensions 0.27 × 0.25 × 0.23 mm was chosen for the X-ray diffraction study. The data were collected on a DIPLabo Image Plate system equipped with a normal focus, 3 kW sealed X-ray source (graphite monochromated Mo K_α). The crystal to detector distance was fixed at 120 mm with a detector area of 441 × 240 mm^2. Thirty six frames of data were collected at room temperature by the

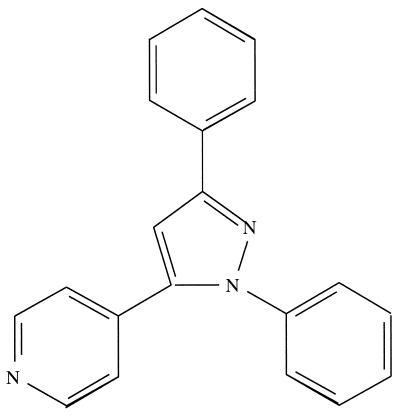

Fig. 1 Schematic diagram.

Table 1 Crystal data and structure refinement table

Empirical formula	$C_{20}H_{15}N_3$
Formula weight	297.35
Temperature	293 K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/c$
Cell dimensions	a = 10.5700(6)Å
	b = 12.2810(13)Å
	c = 12.1750(13)Å
	β = 94.429(7)Å
Volume	1575.7(3)Å^3
Z	4
Density(calculated)	1.253 Mg/m^3
Absorption coefficient	0.076 mm^{-1}
F_{000}	624
Crystal size	0.27 × 0.25 × 0.23 mm
θ range for data collection	2.97° to 25.02°
Index ranges	$-10 \le h \le 11$
	$-14 \le k \le 14$
	$-14 \le l \le 14$
Reflections collected	4811
Independent reflections	2607
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2607/0/209
Goodness-of-fit on F^2	1.051
Final R indices	$R1$ = 0.0447, $wR2$ = 0.1165
R indices (all data)	$R1$ = 0.0572, $wR2$ = 0.1261
Extinction coefficient	0.036(6)
Largest diff. peak and hole	0.127 and –0.144 e.Å^{-3}
Measurement	DIPLabo
Program system	Denzo
Structure determination	SHELXS-97
Refinement	SHELXL-97
CCDC deposition number	788658

Table 2 Selected bond lengths (Å) and bond angles (°)

N1-N2	1.3555(2)	C8-N9	1.331(2)
N1-C5	1.3661(2)	N9-C10	1.334(2)
N1-C12	1.429(2)	C5-C6	1.473(2)
N2-C3	1.3344(2)	C4-C5	1.370(2)
C3-C4	1.404(2)	C3-C18	1.473(2)
N2-N1-C12	119.30(1)	C8-N9-C10	115.48(2)
C5-N1-C12	128.51(1)	C17-C12-N1	119.67(2)
C7-C6-C5	123.57(1)	C13-C12-N1	119.33(1)
N2-C3-C18	119.47(1)	C11-C6-C5	119.72(1)
C4-C3-C18	129.89(1)	C23-C18-C3	120.74(1)
N1-C5-C6	124.31(1)	C19-C18-C3	120.86(1)
C4-C5-C6	129.81(1)		

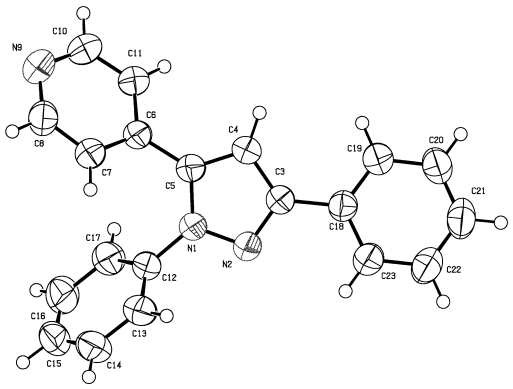

Fig. 2 ORTEP of the molecule with thermal ellipsoids drawn at 50% probability.

oscillation method. Each exposure of the image plate was set to 400 s. Successive frames were scanned in steps of 5° per minute with an oscillation range of 5°. Image processing and data reduction were done using Denzo.[8] The reflections were merged with Scalepack.[9] All of the frames could be indexed using a primitive monoclinic lattice. An absorption correction was not applied.

The structure was solved by direct methods using SHELXS-97.[10] The structure was refined by a full matrix least-squares method with anisotropic temperature factors for non-hydrogen atoms using SHELXL-97.[10] The hydrogen atoms were placed at chemically acceptable positions and were allowed to ride on the parent atoms; 209 parameters were refined with 2607 unique reflections which saturated the residuals to $R_1 = 0.0447$.

The details of the crystal data and refinement are given in Table 1. The selected bond lengths and bond angles of non-hydrogen atoms are given in Table 2. Figure 2 represents an ORTEP diagram of the molecule with thermal ellipsoids drawn at 50% probability.

The dihedral angle between the planes of pyrazole ring and the pyridine ring are defined by atoms N1–N2–C3–C4–C5 and N9–C8–C7–C6–C11–C10 is 39.87(8)°. This angle is large because pyrazole ring is affected from adjacent phenyl ring. The bond distance between atoms C5–C6 is 1.473(2)Å. This value is comparable with bond length between pyrazole and pyridine rings reported for compound[11] which is 1.464(4)Å. The pyrazole ring is almost planar. The N1 and N2 atoms of the pyrazole ring deviates from the Cremer and Pople plane by –0.0004(13)Å and –0.0005(14)Å, respectively. The torsion angle about atoms C12–N1–C5–C6 is 3.3(2)°. The structure exhibits an intermolecular hydrogen bond of the type C–H···N. The inter molecular hydrogen bond C21–H21···N2 has a length of 3.468(2)Å and an angle of 157° with symmetry code 1–*x*, 1/2+*y*, 1/2–*z*.

Acknowledgements

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References

1. P. G. Baraldi, S. Manfredini, R. Romagnoli, L. Stevanato, A. N. Zaid, and R. Manservigi, *Nucleosides Nucleotides*, **1998**, *17*, 2165.
2. H. S. Chen and Z. M. Li, *Chem. J. Chin. Univ.*, **1998**, *19*, 572.
3. S. R. Smith, G. Denhardt, and C. Terminelli, (2001). *Eur. J. Pharmacol.*, **2001**, *432*, 107.
4. O. Bruno, F. Bondavalli, A. Ranise, P. Schenone, C. Losasso, L. Cilenti, C. Matera, and E. Marmo, *Farmaco.*, **1990**, *45*, 147.
5. B. Cottineau, P. Toto, C. Marot, A. Pipaud, and J. Chenault, *Bioorg. Med. Chem.*, **2002**, *12*, 2105.
6. P. A. Brough, X. Barril, and M. Beswick, *Bioorg. Med. Chem. Lett.*, **2005**, *15*, 5197.
7. M. Londershausen, *Pestic. Sci.*, **1996**, *48*, 269.
8. Z. Otwinowski and W. Minor, *Macromelec. Crystallogr.*, **1997**, *276*: part A, ed., C. M. Carter, Jr. and R. M. Sweet, Academic Press, 307-326.
9. S. Mackay, C. J. Gilmore, C. Edwards, N. Stewart, and K. Shankland, "maXus, Computer Program for the Solution and Refinement of Crystal Structures", **1999**, Bruker Nonius, The Netherlands, Mac-Science, Japan and The University of Glasgow.
10. G. M. Sheldrick, *Acta Cryst.*, **2008**, *A64*, 112.
11. Baker Jawabrah Al-Hourani, Klaus Banert, Bernhard Walfort, and Heinrich Lang, *X-ray Struct. Anal. Online*, **2006**, *22*, x275.