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NOTE

Hydrothermal Synthesis and Crystal Structure of (4-Piperidine-2-pyrimidine) Ferrocene

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One new complexes containing N-donor ferrocenyl ligand has been successfully synthesized. Compound shows a one-dimensional framework. The 3D supramolecular structure is formed *via* hydrogen bonding connection.

Keywords: Coordination polymer, Crystal structure, Ferrocene.

Palladacycles containing a Pd-C bond intramolecularly stabilized by one donor atom were first reported in the middle 1960s[1]. After 40 years, palladacycles are one of the most developed and studied classes of precatalysts because of their structural versatility and high catalytic activity[2]. Since the first report on the use of palladacycles for the coupling reactions by Herrmann[3], a wide variety of palladacycles have been successfully employed in these reactions. Among them, cyclopalladated complexes containing N-donor ferrocenyl ligand have been studied extensively in the past two decades[4]. We have studied the cyclopalladation of ferrocenyl ligand containing N-donor and coupling reaction of chloromercuriferrocene[5].

All reagent and solvents employed were commercially available and used as received without further purification.

General procedure: The compound was obtained from the coupling reaction of chloromercuriferrocene and pyrimidine (1 mmol), piperidine (1 mmol) as described in and recrystallized from dichloromethane/petroleum followed by slow cooling to room temperature. Red crystals of the compound formed.

Detection method: Diffraction intensity data of the single crystal of the five compounds were collected on a Bruker SMART APEXII CCD diffractometer equipped with a graphite monochromated MoK$_\alpha$ radiation (λ = 0.71073 Å) by using a ω-scan mode. All the structures were solved by direct methods and refined by full-matrix least-squares methods on F^2 using the program SHEXL 97. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were located by geometrically calculations and their positions and thermal parameters were fixed during the structure refinement. The molecular structure of the title compound is shown in Fig. 1. The crystallographic data and experimental details of structural analyses for coordination polymers are summarized in Table-1. Selected bond and angle parameters are listed in Table-2. CCDC: 910922.

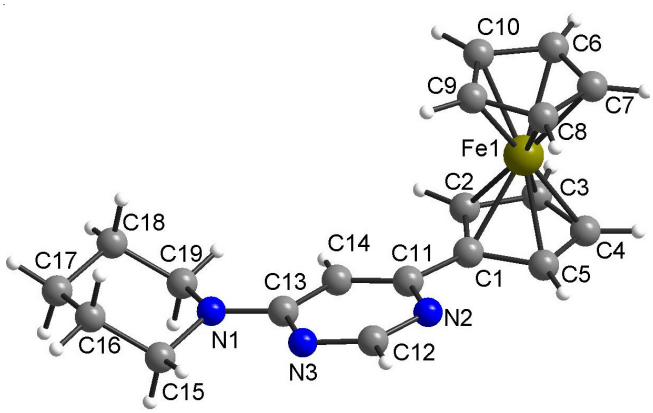

Fig. 1. Molecular structure of the title compound at 30 % probability displacement

X-ray diffraction analysis revealed that the fundamental building unit consists of two cyclopentadienyl rings in the title crystal structure are almost parallel with dihedral angles of 1.14°. The dihedral angle between the pyrimidine and substituted cyclopentadienyl ring is 11.57°. In the crystal there exist intermolecular C-H…N hydrogen bonds between N atom and the adjacent C-H group of substituted cyclopentadienyl ring , which are attributed to construct the chain structure of the title compound. The 1D chains are further assembled by the intermolecular hydrogen bonding interaction leading to the formation of a 3D framework (Fig. 2).

TABLE-1
CRYSTALLOGRAPHIC DATA AND STRUCTURE REFINEMENT SUMMARY FOR COMPLEX

Empirical formula	$C_{19}H_{21}N_3Fe$	Z, Calculated density (mg/m^3)	8,1.394
Formula weight	347.24	Absorption coefficient (mm^{-1})	0.914
Crystal system space group	Orthorhombic, Pbcn	F(000)	1344
Unit cell dimensions	a = 30.19(3) Å	Limiting indices	$-36 \le h \le 36$
	b = 10.145(10) Å		$-12 \le k \le 12$
	c = 10.803(11) Å		$-13 \le l \le 13$
Volume ($Å^3$)	3309(6)	Largest diff. peak and hole ($e/Å^3$)	0.241 and -0.265
θ range for data collection	2.75-25.50	Goodness-of-fit on F^2	1.009
Final R indices [$I > 2\sigma(I)$]	R_1= 0.0426 w, R_2 = 0.0909	R indices (all data)	R_1 = 0.0853, wR_2 = 0.1119

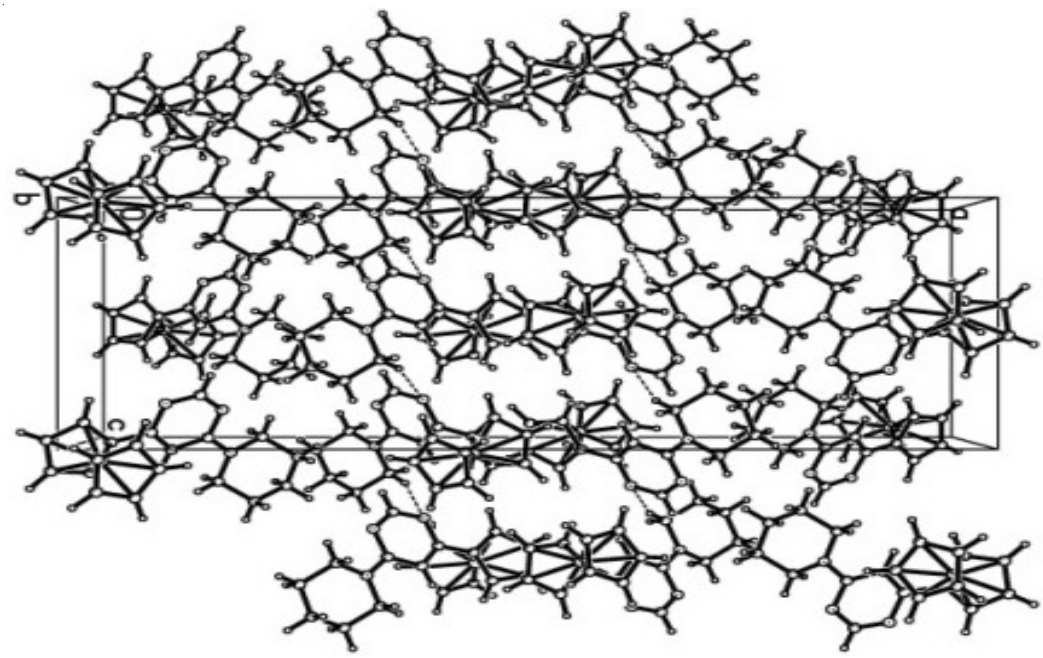

Fig. 2. 3D structure formed *via* hydrogen bonding interactions

TABLE-2
SELECTED BOND LENGTHS (Å)
AND ANGLES (°) FOR COMPLEX

N1–C19	1.460 (4)	Fe1–C9	2.026 (4)
N1–C15	1.469 (5)	Fe1–C5	2.035 (4)
Fe1–C1	2.056 (4)	Fe1–C2	2.046 (4)
C13–N1–C19	123.1 (3)	N2–C11–C1	115.2 (3)
C13–N1–C15	120.9 (3)	C14–C11–C1	122.6 (3)
N1–C13–N3	116.5 (3)	C2–C1–C11	107.0 (3)
N1–C13–C14	123.7 (3)	C5–C1–C11	124.6 (3)

Symmetry codes: (i)

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