

Syntheses, spectral and crystallographic studies of novel monometallic Co(II) and Zn(II) complexes with phenyl-(5-pyridin-4-yl[1,3,4]thiadiazol-2-yl)-amine

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ABSTRACT

Two novel monometallic complexes $[\text{Co}(\text{ppt})(\text{SO}_4)(\text{H}_2\text{O})_4]\cdot\text{H}_2\text{O}$ (**1**) and $[\text{Zn}(\text{ppt})_2(\text{CH}_3\text{COO})_2(\text{H}_2\text{O})_2]$ (**2**) of ppt = phenyl-(5-pyridin-4-yl[1,3,4]thiadiazol-2-yl)-amine have been synthesized and their structures investigated by elemental analyses, IR, UV-Vis., magnetic susceptibility and single crystal X-ray. The geometry of both complexes are distorted octahedral. The π - π and C-H... π interactions play important role along with hydrogen bonding to form host-guest cavity. The hydrogen-bonding interactions between adjacent layers extend these structures to overall 3D supramolecular architectures.

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1. Introduction

The derivatives of 2-amino-[1,3,4]thiadiazole, depending on the nature of substituents can be found as a structural fragment of drugs and herbicides with a wide spectrum of activity due to the presence of the toxiphobic $-\text{N}=\text{C}-\text{S}-$ linkage [1]. They are known as the carbonic anhydrase inhibitors and some of them possess antimycobacterial, anesthetic, antidepressant, anxiolytic [2–7], anti-inflammatory [8], anticonvulsant [9,10], antihypertensive [11] and anticancer [12] activities. They also find applications in agriculture, material chemistry [13] and are also useful as corrosion inhibitors [14]. Derivatives of 1,3,4-thiadiazoles were synthesized by intramolecular cyclization of substituted thiosemicarbazides under acidic condition. Due to the presence of aromatic pyridine, phenyl and 1,3,4-thiadiazole ring in the ligand under investigation, π - π stacking is envisaged in the present system [15]. The bent organic compounds such as 2,5-bis(4-pyridyl)-[1,3,4]thiadiazole, a member of crystal engineering family, has recently focused interest because of several characteristics: (i) the bent ligand has flexible coordination modes and (ii) longer ligand will lead to larger voids. The phenyl-(5-pyridin-4-yl[1,3,4]thiadiazol-2-yl)-amine (ppt) being investigated here is longer than those containing planar centers and has potential biological activities due to the presence of thiadiazole ring [16]. With an aim of obtaining information about the chemical and structural properties of the metal complexes, especially in reference to stereochemistry and the effects of coordination

on the conformation of the thiadiazole, we have undertaken the studies of two new complexes of Co(II) and Zn(II) obtained from a new ligand phenyl-(5-pyridin-4-yl[1,3,4]thiadiazol-2-yl)-amine (ppt).

2. Experimental

2.1. Chemical and starting materials

Commercial reagents were used without further purification and all experiments were carried out in open atmosphere. Isonicotinic acid hydrazide (Sigma-Aldrich) and phenyl isothiocyanate (Fluka Chemicals, Switzerland) were used as received. All solvents were purchased from Merck Chemicals, India, dried and distilled before use by following standard procedure.

2.2. Physical measurements

Carbon, hydrogen and nitrogen contents were estimated on a CHN Model CE-440 Analyzer and on an Elementar Vario EL III Carlo Erba 1108. By following standard procedures, the complexes were analysed for their metal content, after decomposition with a mixture of conc. HNO_3 and HCl , followed by conc. H_2SO_4 [17]. Magnetic susceptibility measurements were performed at room temperature on a Cahn Faraday balance using $\text{Hg}[\text{Co}(\text{NCS})_4]$ as the calibrant and electronic spectra were recorded on SIMADZU 1700 UV-Vis. spectrophotometer in DMSO. IR spectra were recorded in the 4000–400 cm^{-1} region as KBr pellets on a Varian Excalibur 3100 FT-IR spectrophotometer. ^1H and ^{13}C NMR spectra were recorded in

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DMSO- d_6 on a JEOL AL300 FT NMR spectrometer using TMS as internal reference.

2.3. X-ray crystallography

The X-ray data collection for **1** and **2** were performed on Oxford Xcaliber diffractometer equipped with a Crys Alis Pro, using graphite monochromated Mo $K\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$) source. The structures were solved by direct methods (SHELXL-2008) and refined against all data by full matrix least-squares on F^2 using anisotropic displacement parameters for all non-hydrogen atoms. All the hydrogen atoms were included in the refinement at geometrically ideal positions and refined with a riding model [18]. The MERCURY package, DIAMOND and ORTEP-3 programs were used for generating molecular structures [19–21].

2.4. Synthesis

2.4.1. Synthesis of phenyl-(5-pyridin-4-yl-[1,3,4]thiadiazole-2-yl)-amine (ppt)

A mixture of isonicotinic acid hydrazide (2.74 g, 20 mmol) and phenyl isothiocyanate (2.4 mL, 20 mmol) in absolute ethanol was refluxed for 8 h at 65 °C. The solid 1-isonicotinoyl-4-phenyl thiosemicarbazide obtained upon cooling was filtered off, washed successively with water and diethyl ether, dried and crystallized from ethanol. 1-Isonicotinoyl-4-phenyl thiosemicarbazide (2.72 g, 10 mmol) was added portion wise in 10 ml conc. H_2SO_4 and stirred for 2 h with cooling. The mixture was poured over crushed ice and the precipitated solid was filtered off, washed twice with cold water (50 ml) and dried. The yellow crystals of ppt suitable for X-ray analysis were obtained by slow crystallization in methanol for 15 days. Yield: 85%.

Anal. Found: C, 61.35; H, 3.93; N, 21.95; S, 12.58%. Calc. for $C_{13}H_{10}N_4S$ (254.31): C, 61.40; H, 3.96; N, 12.60; S, 20.60%. M.p. 140 °C; IR (KBr, cm^{-1}): 3003 $\nu(NH)$, 1484 $\nu(C=N)$, 685 (pyridine) and 826 (phenyl). 1H NMR (DMSO- d_6 , δ , ppm): [8.86 (2H), 8.19 (2H)] pyridine; [7.60 (2H), 7.37 (1H), 7.11 (2H)] phenyl; 4.84 (1H, NH). ^{13}C NMR (DMSO- d_6 , δ , ppm): [146.42 C(4,5), 140.01 C(2), 122.93 C(3,6)] pyridine; [141.58 C(8), 129.30 C(10,12), 122.93 C(11), 122.07 C(9,13)] phenyl and [166.60 C(7), 153.88 C(1)] thiadiazole carbons of ppt. The tentative structure of ppt is depicted in Fig. 1.

2.4.2. Synthesis of complex **1**

A methanol solution (15 mL) of $CoSO_4 \cdot 7H_2O$ (0.281 g, 1 mmol) was stirred with a methanol solution (15 mL) of ppt (0.250 g, 1 mmol). The resulting clear brown solution was filtered off, where upon the single crystals of **1** suitable for X-ray analyses were obtained by slow evaporation of the solution over a period of 15 days. Yield: 55%.

Anal. Found: C, 31.75; H, 2.43; N, 11.38; S, 13.00; Co, 11.94%. Calc. for $C_{13}H_{12}CoN_4O_9S_2$ (491.32): C, 31.77; H, 2.46; N, 11.40; S, 13.05; Co, 11.99%. M.p. 235 °C, $\mu_{eff} = 5.20$ BM; IR (KBr, cm^{-1}): 3622 $\nu(OH)$; 2985 $\nu(NH)$; 1564 $\nu(S=O)$ sulphate; 1433 $\nu(C=N)$; 677 (pyridine) and 818 (phenyl). UV–Vis. [λ_{max} , DMSO, nm]: 867, 441, 388, 358 and 332.

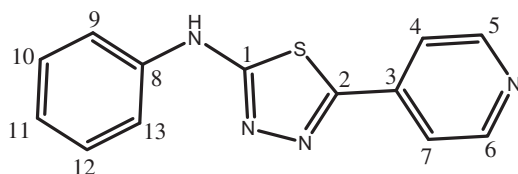


Fig. 1. Phenyl-(5-pyridin-4-yl-[1,3,4]thiadiazole-2-yl)-amine.

2.4.3. Synthesis of complex **2**

A solution of ppt (0.5 g, 2 mmol) in MeOH (15 mL) was added to a solution of $Zn(OAc)_2 \cdot 2H_2O$ (0.219 g, 1 mmol) in MeOH (15 mL) and the mixture was stirred for 4 h at room temperature. The resulting clear solution was filtered off and kept for crystallization. Colorless crystals of **2** suitable for X-ray analyses were obtained by slow evaporation of its solution over a period of 21 days. Yield: 57%.

Anal. Found: C, 49.72; H, 3.59; N, 15.43; S, 8.82; Zn, 8.97%. Calc. for $C_{30}H_{26}N_8O_6S_2Zn$ (724.10): C, 49.76; H, 3.62; N, 15.47; S, 8.86; Zn, 9.03%. M.p. 250 °C; IR (KBr, cm^{-1}): 3607 $\nu(OH)$; 2963 $\nu(NH)$; 1691 $\nu(C=O)$, 1429 $\nu(C=N)$; 663 (pyridine) and 825 (phenyl). UV–Vis. [λ_{max} , DMSO, nm]: 396, 354, 329 and 313. Scheme 1 gives a general method of synthesis of the ligand and its complexes.

3. Results and discussion

A methanol solution of Co(II) and Zn(II) salts react with ppt and yielded complexes **1** and **2**, respectively. The single crystal X-ray diffraction studies indicate that the ligand (ppt) adopts the monodentate binding nature in both the complexes. All the complexes are air stable, non-hygroscopic crystalline solids, which are partially soluble in common organic solvents but soluble in DMF and DMSO and can be kept in desiccators over a prolonged period without any sign of decomposition.

3.1. IR spectra

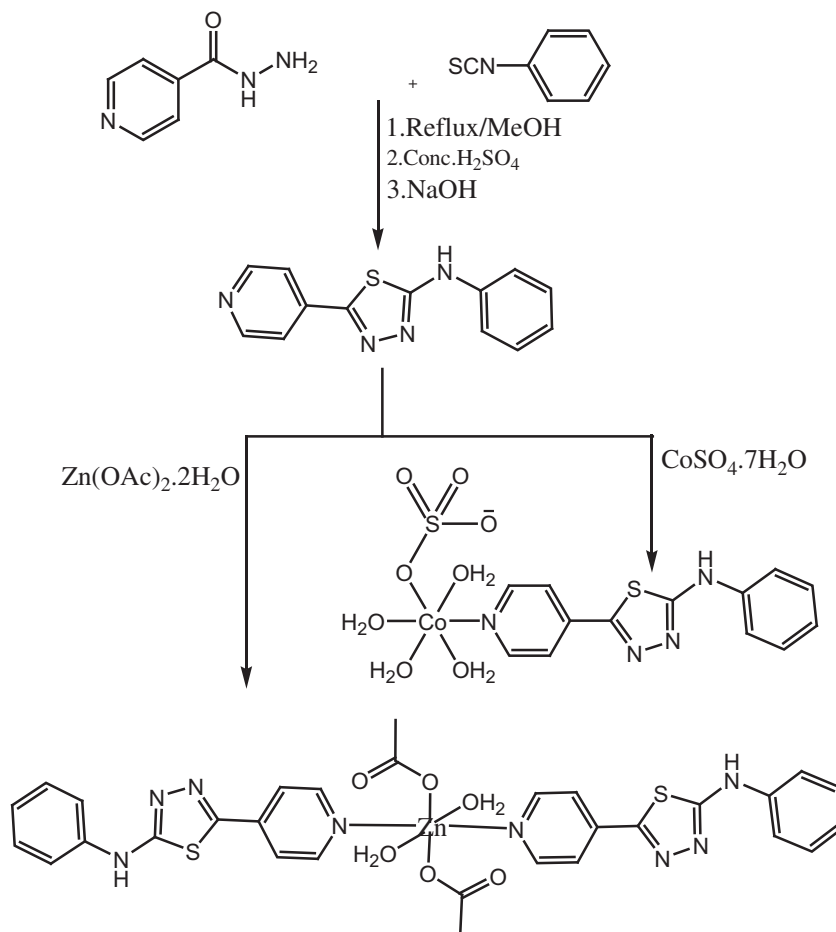
The bands at 3003 and 1484 cm^{-1} in the free ligand may be assigned to the $\nu(NH)$ of the imine group adjacent to the thiadiazole moiety and $\nu(C=N)$ of thiadiazole group, respectively. The spectrum of compound **1** reveals the characteristic bands due to $\nu(S=O)$ sulphate, $\nu(C=N)$ thiadiazole and pyridine rings at 1564, 1433 and 677 cm^{-1} , respectively, suggesting bonding of sulphate and pyridine nitrogen to metal ion. The compound **2** shows a negative shift of 22 cm^{-1} in the pyridine ring vibration on complexation in comparison to the free ligand suggesting bonding via pyridine nitrogen with Zn(II). A band at 1691 cm^{-1} corresponding to $\nu(C=O)$ of acetate indicates the presence of monodentate acetate in the complex. The persistence of a band due to $\nu(NH)$ in the spectra of both the complexes indicates the non-involvement of this group in coordination. A broad band in the region 3622–3607 cm^{-1} indicates the presence of coordinated/uncoordinated water molecule in the complexes **1** and **2**.

3.2. Electronic spectra and magnetic moment

Complex **1** shows a magnetic moment of 5.2 BM and the presence of a broad d–d band around 867 nm assigned to the $^4T_{1g}(F) \rightarrow ^4T_{2g}(F)$ transition, suggest its octahedral geometry. Other high energy bands observed at 441, 388, 358 and 332 nm may be due to charge transfer/intraligand transitions [22]. The higher energy bands at 396, 354, 329 and 313 nm in compound **2** arise because of the charge transfer/intraligand transitions from coordinated and uncoordinated nitrogen atoms to the aromatic ring and imine of the ppt.

3.3. Crystal structure description of complexes **1** and **2**

Crystallographic data and structural refinement details related to complexes **1** and **2** are given in Table 1 and selected bond lengths and angles in Table 2. The X-ray analysis of **1** reveals the presence of four water molecules, monodentate sulphate and a ppt ligand coordinated to Co(II), resulting in a distorted octahedral geometry (Fig. 2). There is one uncoordinated interstitial water



Scheme 1. Preparation of ppt and Co(II) and Zn(II) complexes.

molecule in every unit of structure **1**. The crystallographic unit (Fig. 3) of compound **2** consists of one Zn(II) ion, two acetates, two ppt and two water molecules and adopts an octahedral geometry in which Zn atom and basal atoms are coplanar. The X-ray structure of complex **2** shows the presence of three independent complexes in the asymmetric unit and in each unit the Zn atom is on a centre of inversion.

A nitrogen bonded ppt and a water molecule are present at the axial positions of complex **1** bonded through Co(1)–N(4) and Co(1)–O(3w) at distances of 2.133(7) and 2.066(6) Å, respectively. The four equatorial sites are occupied by the oxygen of three water molecules and remaining coordination site by one oxygen atom of sulphate group. Three Co–O bond lengths occupied by H₂O molecules Co(1)–O(1w), Co(1)–O(2w) and Co(1)–O(4w) are nearly equal being 2.102(5), 2.087(6) and 2.186(5) Å, respectively and fourth one Co(1)–O(1) (sulphate) is found to be 2.080(5) Å. The shortest inter chain Co···Co and Zn···Zn separations between almost superimposable pairs are 6.248 and 5.613 Å, respectively. The coordination environment of **2** is fulfilled by two axial ppt ligands at *trans* positions bonded through Zn–N(4) and Zn–N(5) at distances of 2.190(5) and 2.187(5) Å, respectively; two out of four equatorial sites Zn–O(1) and Zn–O(3) are occupied by two acetates at distances of 2.072(4) and 2.084(4) Å, so is the case with other two units. The remaining two equatorial sites are occupied by Zn–O(1w1) and Zn–O(1w2) at distances of 2.147(5) and 2.132(4) Å, respectively.

The pyridine and phenyl rings of two different molecules in **1** and **2** are approximately parallel ($\angle ABC = 89.21^\circ$ and 84.24°) but displaced with respect to each other (Figs. 4 and 5). The displace-

ment as measured by the angle formed between the ring centroid BC and the ring normal BA to the pyridine plane are 0.79 and 5.76 Å, with a displacement angle of 20.74° and 22.23° , respectively. The dihedral angles between the planes formed by the thiazole and pyridine ring and between thiazole and phenyl ring are 33.78° and 26.98° , respectively. The face to face interactions of rings C(4)···C(13) or π – π and edge to edge C(5)–H(5)···C(7) contacts at distances of 3.374 and 2.824 Å are well within the reported range affording extra stability to the complex **1**. The shortest centroid–centroid separation found between the two parallel phenyl and pyridine rings of nearby molecules is 3.704 Å. The face to face interactions of rings C(12)···C(24) or π – π contacts at distance of 3.383 Å and edge to edge C(8)–H(8)···C(14B) are well within the reported range [15] affording extra stability to the molecule structure of **2**.

3.4. Crystal packing of complexes **1** and **2**

The hydrogen bonding parameters and intermolecular interactions of **1** and **2** are compiled in Tables 3 and 4. The molecular structure of **1** is stabilized by means of extended H-bonds O(5w)–H(2w)···S(2), O(5w)–H(1w)···S(1), O(5w)–H(2w)···O(2), C(10)–H(10)···O(3), C(11)–H(11)···O(4) and C(7)–H(7)···O(1). In addition to this, the most noteworthy structural feature of **1** is the formation of host–guest model and cavities by O(5w)–H(2w)···S(1), O(5w)–H(2w)···S(2), O(5w)–H(1w)···N(2), O(5w)–H(2w)···O(2) and C(7)–H(7)···O(1) by weak interactions in which the lattice water molecule is held as a guest (Fig. 6). Guest

Table 1
Crystallographic data and structure refinement for **1** and **2**.

Empirical formula	C ₁₃ H ₁₂ CoN ₄ O ₉ S ₂	C ₃₀ H ₂₆ N ₈ O ₆ S ₂ Zn
Formula weight	491.34	724.12
Color	Brown	White
Crystal system, space group	Monoclinic, P 21/c	Triclinic, P-1
T (K)	296(2)	293(2)
λ (Mo Kα) (Å)	0.71073	0.71073
a (Å)	7.602(5)	11.2262(4)
b (Å)	11.780(5)	16.3673(5)
c (Å)	21.614(5)	17.9275(6)
α (°)	90	72.973(3)
β (°)	99.241(5)	80.980(3)
γ (°)	90	89.970(3)
V (Å ³)	1910.4(16)	3107.11(19)
Z	4	4
ρ _{calc} (Mg/m ³)	1.708	1.548
μ (mm ⁻¹)	1.173	0.983
F(0 0 0)	996.0	1488.0
Sample size/mm	0.25 × 0.23 × 0.21	0.27 × 0.25 × 0.22
θ range (°)	1.91–32.08	2.02–28.88
Index ranges	–11 ≤ 10 –17 ≤ 16 –32 ≤ 31	–15 ≤ 11 –22 ≤ 21 –24 ≤ 22
Reflection collected	18,444	22,155
Independent reflections	3762	16,325
Data/restraints/parameters	3762/0/262	16,325/0/850
Final R indices	0.0804	0.0372
wR ₂ [I > 2σ(I)](R _{int})	0.0970	0.1056
Final R indices (all data)	0.2368	0.0926
data	0.2482	0.1134
Goodness-of-fit on F ²	1.243	0.879

$$^a wR_1 = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}$$

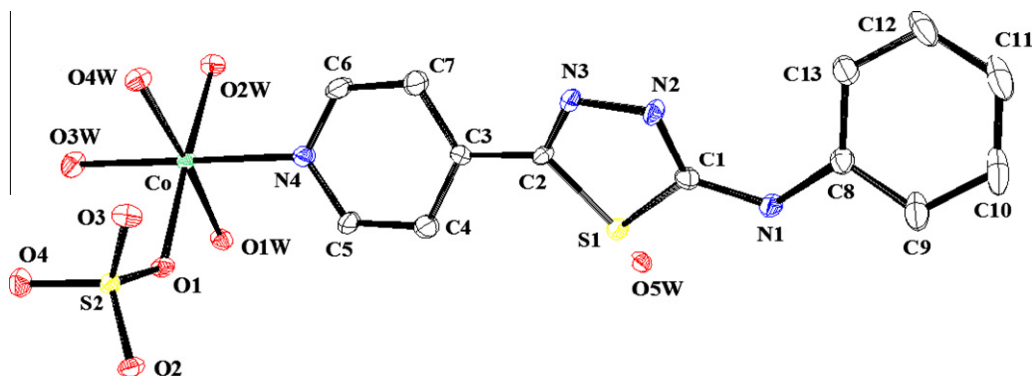
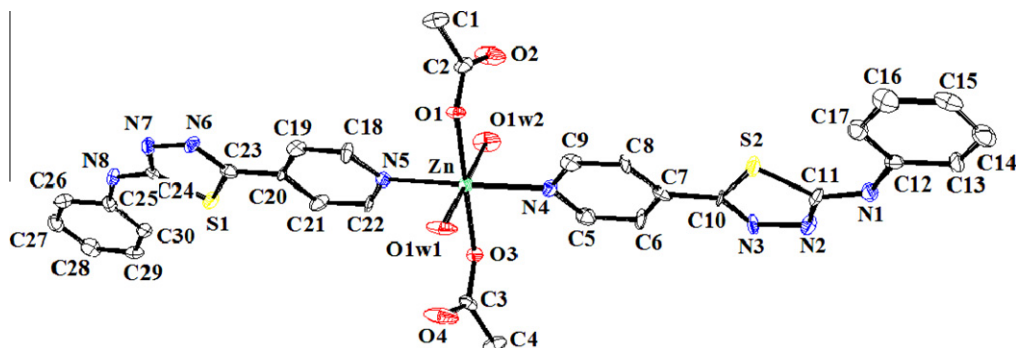
$$^b wR_2 = \left[\frac{\sum w(|F_o|^2 - |F_c|^2)^2}{\sum w|F_o|^2} \right]^{1/2}$$

Table 2
Interatomic distances (Å) and bond angles (°) for **1** and **2**.

Co(1)—O(1)	2.080(5)	Zn—O(1)	2.072(4)
Co(1)—N(4)	2.133(7)	Zn—O(3)	2.084(4)
S(2)—O(1)	1.471(5)	Zn—N(4)	2.190(5)
S(2)—O(2)	1.466(6)	Zn—N(5)	2.187(5)
S(2)—O(3)	1.476(6)	Zn—O(1w1)	2.147(5)
S(2)—O(4)	1.469(6)	Zn—O(1w2)	2.132(4)
Co(1)—O(1w)	2.102(5)	O(1)—C(2)	1.275(7)
Co(1)—O(2w)	2.087(6)	O(3)—C(3)	1.290(6)
Co(1)—O(3w)	2.066(6)	C(2)—O(2)	1.219(8)
Co(1)—O(4w)	2.186(5)	C(3)—O(4)	1.234(7)
O(1)—Co(1)—N(4)	89.7(2)	O(1)—Zn—O(3)	179.5(2)
O(3w)—Co(1)—O(1)	89.5(2)	O(1)—Zn—O(1w2)	89.34(18)
O(3w)—Co(1)—O(1w)	90.1(2)	O(3)—Zn—O(1w1)	90.43(16)
O(3w)—Co(1)—O(2w)	91.9(2)	O(3)—Zn—O(1w2)	90.96(18)
O(1)—Co(1)—O(1w)	82.5(2)	O(1w2)—Zn—(1w1)	178.4(2)
O(2w)—Co(1)—O(1w)	94.2(2)	O(1)—Zn—N(4)	90.83(19)
O(2w)—Co(1)—N(4)	89.1(3)	O(3)—Zn—N(5)	92.08(18)
O(1w)—Co(1)—N(4)	92.0(2)	N(5)—Zn—N(4)	179.2(3)
O(3w)—Co(1)—O(4w)	86.9(2)		
O(1)—Co(1)—O(4w)	94.4(2)		
O(2w)—Co(1)—O(4w)	89.0(2)		
N(4)—Co(1)—O(4w)	90.8(2)		

size and the effectiveness of space filling by the guest are likely to play the main role in stability of compound **1**.

Additionally, the elements of the structure are joint to each other in the packing diagram of **2** by means of extended C(26)—H(26)···N(6), N(8)—H(20)···N(7), C(1A)—H(1AB)···O(2B), C(1A)—H(1AB)···O(3w1), O(3w1)—H(1AB)···N(2), O(1)—H(18)···N(4) and N(5)—H(10)···O(1) H-bonds which plays an important

**Fig. 2.** ORTEP diagram of complex **1** with 50% probability level.**Fig. 3.** ORTEP diagram of complex **2** with 30% probability level.

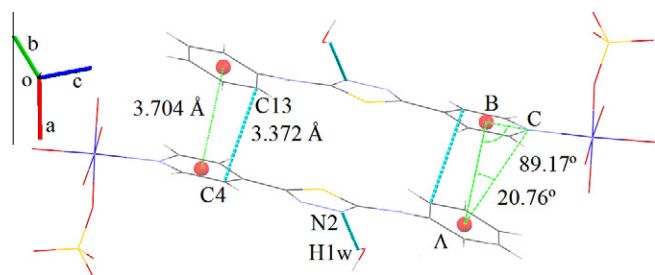


Fig. 4. Mercury view of complex **1** showing π - π , O-H \cdots N interactions between pyridine rings along with relative shifts.

role in contributing to the stability of 3D network of the complex. In addition to this, the most noteworthy structural feature of complex **2** is the formation of close cavities [23–25] formed by C(11B)–H(11B) \cdots N(2B), N(4B)–H(10B) \cdots N(3B), N(1)–H(10) \cdots N(2), C(4)–H(2C) \cdots O(2), C(1A)–H(1AB) \cdots O(3W1), C(1A)–H(1AB) \cdots O(2B) interactions (Fig. 7). As a result of all these interactions, each chain is linked to other chains by hydrogen bond interactions, forming a three dimensional network in **1** and **2**. The supramolecular association of thiadiazole and water in arrays is presumably enforced by the size and the orientation of the interior of the host's channel.

Table 3

Hydrogen bond parameters for **1** (Å and °).

D–H \cdots A	d(D–H)	d(H \cdots A)	d(D \cdots A)	\angle (D–H \cdots A)
O(5W)–H(1W) \cdots N(2)#2	0.97	2.08	2.9054	143
O(5W)–H(1W) \cdots O(4)#1	0.97	1.82	2.7559	162
N(1)–H(8) \cdots O(2)#1	0.86	1.99	2.8381	168
C(7)–H(7) \cdots O(1) #3	0.93	2.46	3.3813	169
C(10)–H(10) \cdots O(3)#2	0.93	2.53	3.3347	144
C(11)–H(11) \cdots O(2)#2	0.93	2.55	3.3785	149

#1 = $x, 3/2 - y, -1/2 + z$.

#2 = $1 - x, 1 - y, -z$.

#3 = $2 - x, -1/2 + y, 1/2 - z$.

Table 4

Hydrogen bond parameters for **2** (Å and °).

D–H \cdots A	d(D–H)	d(H \cdots A)	d(D \cdots A)	\angle (D–H \cdots A)
N(1)–H(10) \cdots N(2)#1	0.86	2.09	2.9488	172
N(4A)–H(10A) \cdots N(3A)#2	0.86	2.09	2.9472	171
N(4B)–H(10B) \cdots N(3B)#3	0.86	2.05	2.9040	172
N(8)–H(20) \cdots N(7)#4	0.86	2.06	2.9087	169

#1 = $1 - x, -y, 1 - z$.

#2 = $-x, 1 - y, 1 - z$.

#3 = $1 - x, 1 - y, 1 - z$.

#4 = $2 - x, 2 - y, -1 - z$.

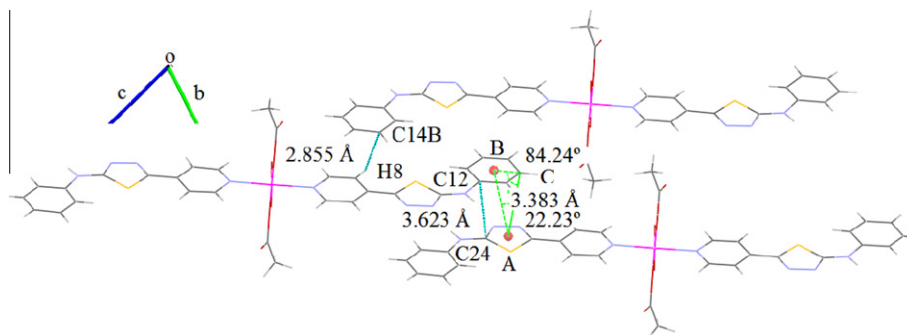


Fig. 5. Mercury view of **2** showing π - π , C–H \cdots π interactions between thiadiazole and phenyl rings along with relative shifts.

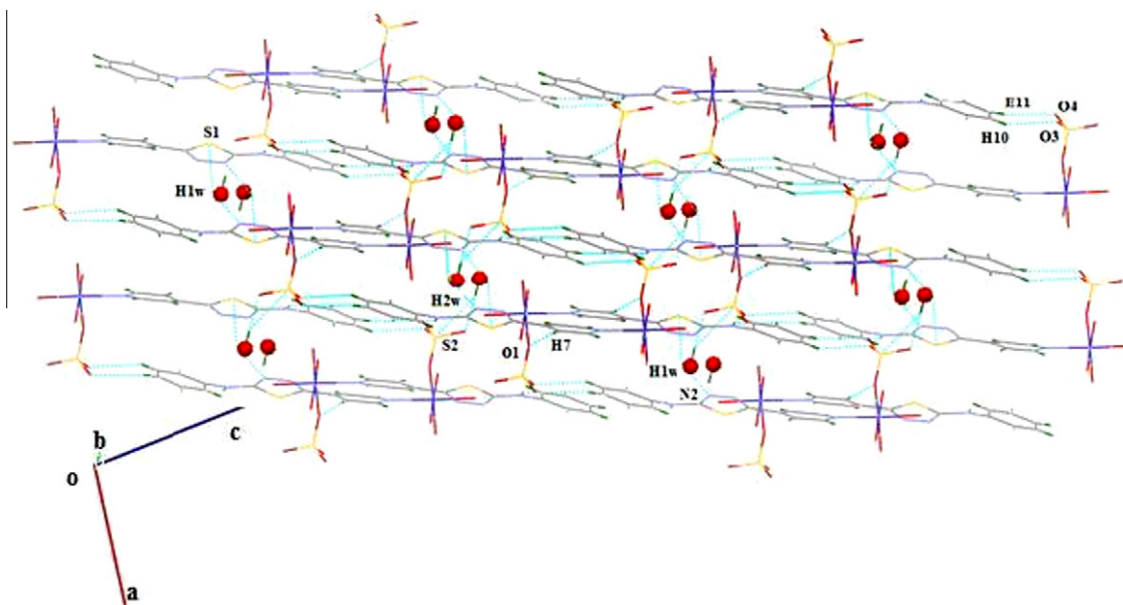


Fig. 6. Host-guest model of complex **1** due to N(2) \cdots H(1w) and S(1) \cdots H(1w) interactions with water molecule inside the close cavity.

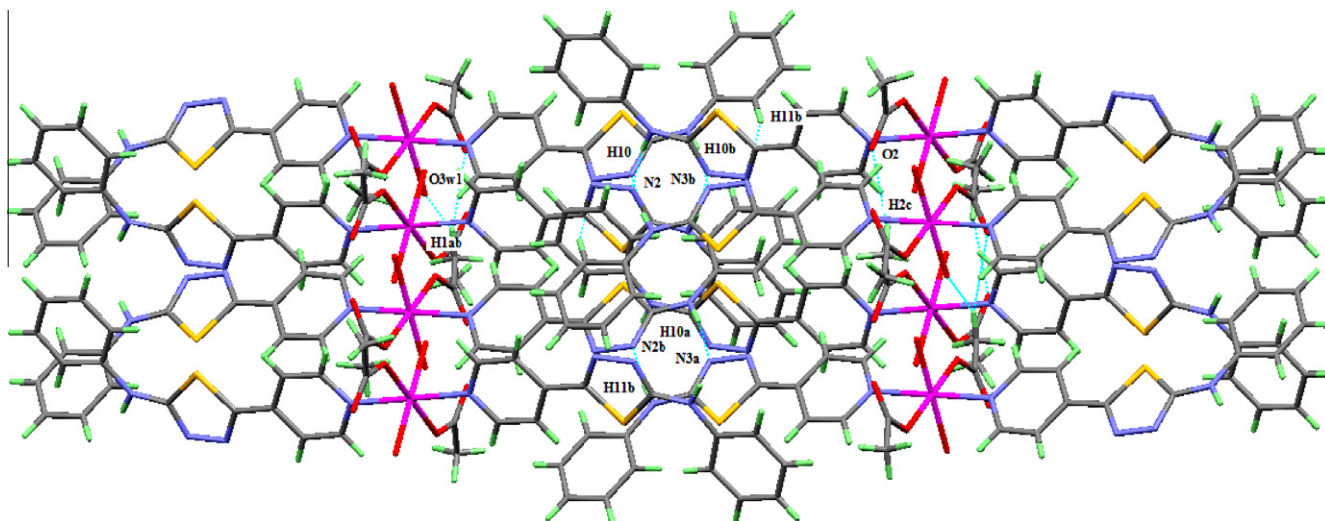


Fig. 7. Packing diagram of complex **2** showing close cavity and 3D supramolecular network along *b* axis.

4. Conclusion

This paper reports the syntheses, characterization and crystal structure investigation of two novel complexes $[\text{Co}(\text{ppt})(\text{SO}_4)(\text{H}_2\text{O})_4] \cdot \text{H}_2\text{O}$ and $[\text{Zn}(\text{ppt})_2(\text{CH}_3\text{COO})_2(\text{H}_2\text{O})_2]$ containing a new ligand phenyl-(5-pyridin-4-yl)[1,3,4]thiadiazol-2-yl)-amine. In the crystalline state both complexes involve π - π and C-H \cdots π interactions, which play important role along with hydrogen bonding to form host-guest cavity. The hydrogen-bonding interactions between adjacent layers extend the structures of the complexes to overall 3D supramolecular architectures.

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Appendix A. Supplementary material

CCDC 757912 and 57913 contains the supplementary crystallographic data for the complexes **1** and **2**. These data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html>, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336 033; or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.molstruc.2010.09.012.

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