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A Short Review on Structural Concepts of Metal Complexes of Proton Transfer Salts of Pyridine-2-Carboxylic Acid

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Abstract: In this study, a brief review of the proton transfer salts synthesized from the reaction of piperazine with pyridine-2-carboxylic acid and their metal complexes since 2009 was made. The metals in the studied compounds are mostly d-block metals. In complexes, the anionic component of the salt is coordinated to the metal, while the cationic component is present only as a complementary ion in a complex.

Keywords: Pyridine, piperazine, acid, base, salt.

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1. INTRODUCTION

Pyridine carboxylates containing two functional groups are among the most versatile ligands that can coordinate with metal (1). Studies with such molecules have mainly focused on the development of metal-based compounds containing pyridine-2-carboxylic acid (2-picolinic acid) for dietary supplementation and numerous articles and reviews have been conducted (2). Later, such ligands were used for coordination to different transition metals. These compounds usually form five-membered rings by bonding to the metal *via* carboxylate-O and pyridine-N donor atoms (3, 4) (Figure 1).

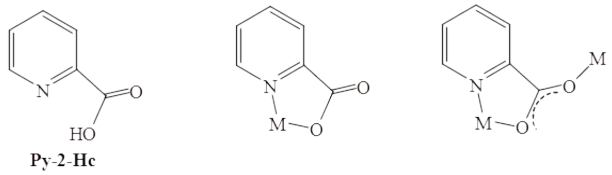


Figure 1: Coordination modes of Py-2-Hc.

As a terminal tryptophan metabolite, 2-picolinic acid is an important structural motif of enzymes and is the active component of many drugs (5, 6). An increase in the number of anticancer agents has been observed with the synthesis of pyridine-2-carboxylic acid type ligands with anti-cancer properties and complexes of different transition metals (7, 8). The complexes with metal ions have been studied as models for broad biological activity spectra such as enzyme activity and DNA (9-11). Proton transfer salts, which consist of a good proton donor-acceptor systems, and their reactions with metals can form supramolecular compounds called Metal-organic frameworks (MOF) (12). It is very important to prepare an effective ion pair that forms the components of the proton transfer salt and accelerates the formation of the metal complexes. Among the proton donor and proton acceptor groups, which are components of the proton transfer salt, carboxylic acids and amines are a good pair. In recent years, the development of new pharmaceutical components with proton transfer reactions has accelerated (13-16). For the synthesis of complexes to be successful, care must be taken to select polydentate ligands and components capable of intermolecular H bonding (17, 18). Piperazines, which are cyclic amines, have many biological activities such as anti-depressant (19), anti-fungal (20), anti-tumor, (21, 22), anti-inflammatory (23), anti-migraine (24) and anti-malarial (25). In this work, an investigation of the transition metal complexes of compounds containing 2-picolinic acid and piperazine as structural units of proton transfer salt was carried out.

2. SYNTHESIS

2.1. Proton Transfer Reaction

The reaction or reaction mechanism in which a proton (H^+) is separated from one species such as an acid and accepted by another species such as a base is called a proton transfer reaction. The product of the proton transfer reaction between donors and acceptors is named differently as the proton transfer salt (26-29) the H-bonded complex (30-33) and the charge transfer complex (34-37). In this study, proton transfer salts of pyridine-2-carboxylic as a proton donor and piperazine as proton acceptor and metal complexes of these salts were investigated.

*2.1.1. Proton transfer salt, (H_2Ppz)(Py-2-c)$_2$ (**1**)*

A mixture of pyridine-2-carboxylic acid (Py-2-Hc) in H_2O and piperazine (Ppz) in THF gave colorless crystals on slow evaporation after one week (38) (Figure 2).

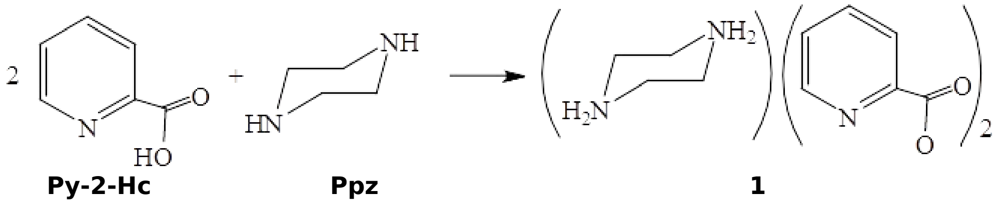


Figure 2: Synthesis of (H_2Ppz)(Py-2-c)$_2$ (**1**).

*2.1.2. Mg(II) (**2**), Zn(II) (**3**) and Cd(II) (**4**) metal complexes of **1***

The complexes, [Mg(Py-2-c)$_2$(H_2O)$_2$]·H_2O (**2**), [Zn(Py-2-c)$_2$(H_2O)$_2$]·H_2O (**3**) and (H_2Ppz)[Cd(Py-2-c)$_3$]·3H_2O (**4**), of proton transfer salt **1** were synthesized (38) (Figure 3).

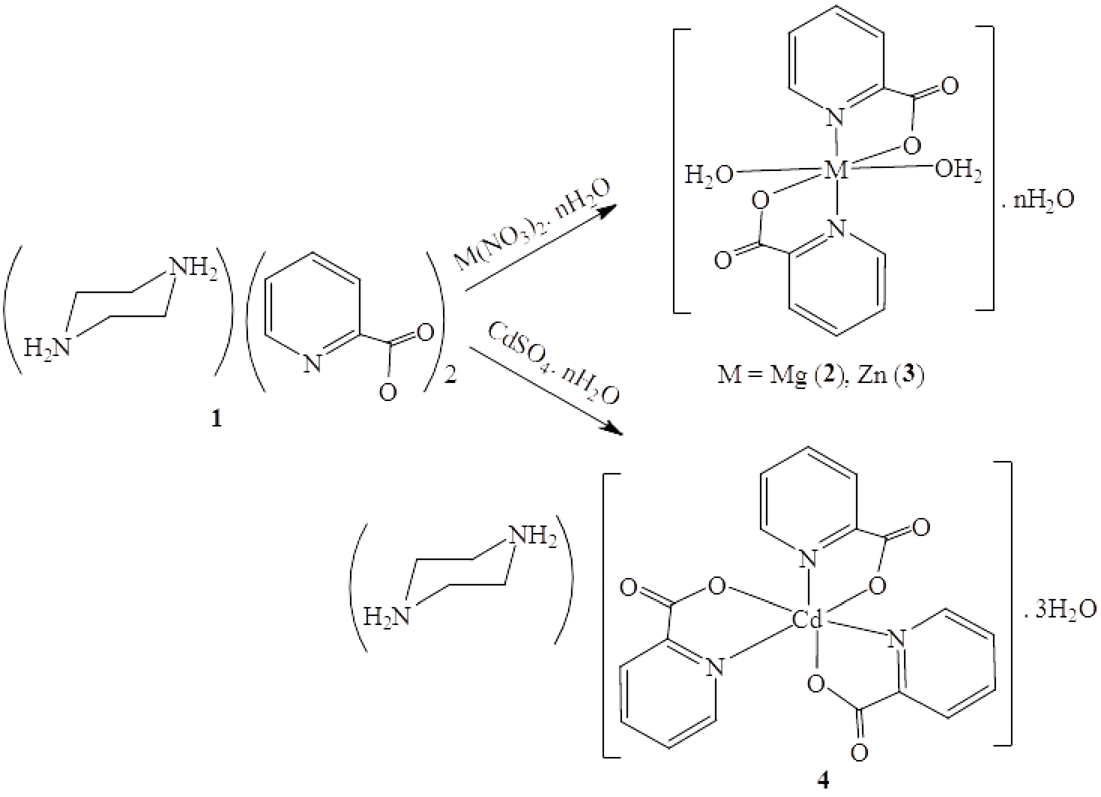


Figure 3: Synthesis of complexes **2**-**4**.

The analyses, crystal systems and space groups of the compounds (**1-4**) reported in the literature are given in Table 1.

Table 1: Proton transfer salt, $(H_2Ppz)(Py\text{-}2\text{-}c)_2$, and its Mg(II) (**2**), Zn(II) (**3**) and Cd(II) (**4**) complexes.

Ref. No	Analyses	Proton Transfer Salt and Complexes	Space Group	Crystal system
(38)	IR and NMR	$(H_2Ppz)(Py\text{-}2\text{-}c)_2$ (**1**)	-	-
(38)	IR and X-ray	$[Mg(Py\text{-}2\text{-}c)_2(H_2O)_2]\cdot H_2O$ (**2**)	*$P2_1/n$*	Monoclinic
(38)	IR and X-ray	$[Zn(Py\text{-}2\text{-}c)_2(H_2O)_2]\cdot H_2O$ (**3**)	*$P2_1/n$*	Monoclinic
(38)	IR and X-ray	$(H_2Ppz)[Cd(Py\text{-}2\text{-}c)_3]\cdot 3H_2O$ (**4**)	*$P2_1/n$*	Monoclinic

In compound **2**, the Mg^{2+} coordinates to two oxygen (O2 and O3) and two nitrogen atoms (N1a and N2) of the two Py-2-c$^-$ ligands, and two oxygen atoms (O6 and O7) of the two H_2O molecules, forming the distorted octahedral geometry. The torsion angles of N2–Mg1–N1–C1 and N2–Mg1–N1–C5 are -84.43(11) and 96.05(8) Å, respectively, indicating that the planes of the two ligands are coplanar (38) (Figure 4a).

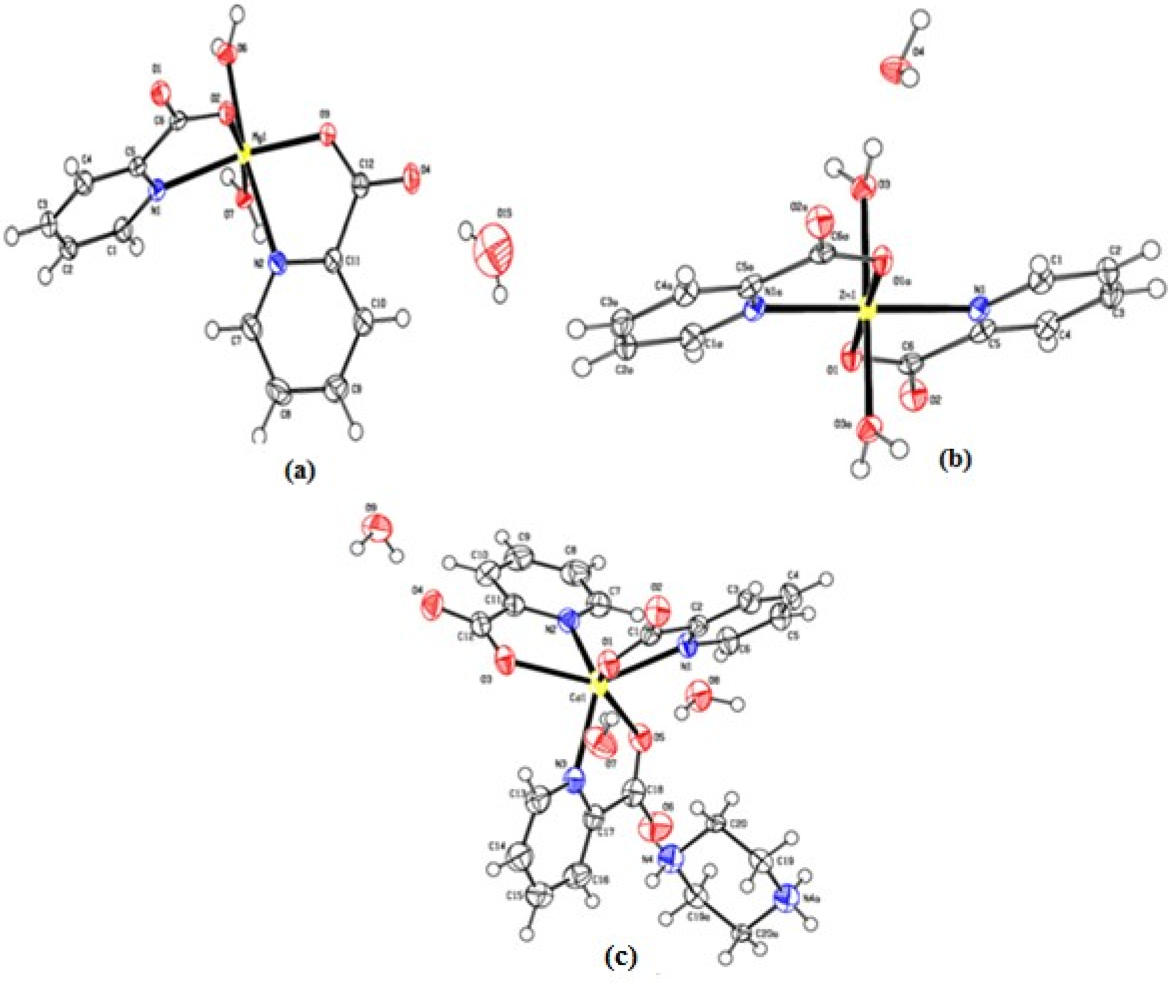


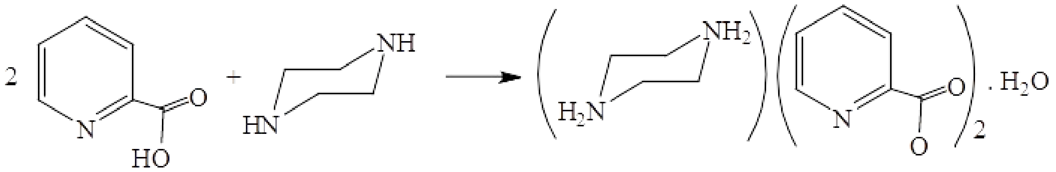
Figure 4: Crystal structures (a) for **2**, (b) for **3** and (c) for **4**.

In $[Zn(Py\text{-}2\text{-}c)_2(H_2O)_2]\cdot H_2O$ (**3**) complex, the N1–Zn1–N1a, O1–Zn1–O1a and O3–Zn1–O3a angles around the Zn^{2+} ion are equivalent to 180.00°, indicating that the geometry is a normal octahedral (Figure 4b). In the Zn(II) (**3**) complex, there are many O-H···O H-bonds between the metal and the coordinated and uncoordinated H_2O molecule (38).

In asymmetric unit of Cd(II) (**4**) compound contains two ionic moieties $[Cd(Py\text{-}2\text{-}c)_3]^{2-}$ anion and $(pipH_2)^{2+}$ cation and three lattice water molecules. The Cd(II) ion, which has six coordination numbers in the complex, is attached to the three oxygen and three nitrogen atoms of the three Py-2-c$^-$ rings (Figure 4c). The extensive hydrogen bond interactions between cationic, anionic moieties, and uncoordinated H_2O molecules along the lattice of the $(H_2Ppz)[Cd(Py\text{-}2\text{-}c)_3]\cdot 3H_2O$ (**4**) complex are of great importance in stabilizing the crystal (38).

2.2. The proton transfer compound, $(H_2Ppz)(Py\text{-}2\text{-}c)_2\cdot H_2O$ (5)

Proton transfer compound, $(H_2Ppz)(Py\text{-}2\text{-}c)_2\cdot H_2O$ (**5**), was prepared (39, 40) from the reaction of Py-2-Hc and Ppz (Figure 5).



Py-2-Hc **Ppz** **5**

Figure 5: Synthesis of $(H_2Ppz)(Py\text{-}2\text{-}c)_2{\cdot}H_2O$ (**5**).

The NMR (^1H and ^{13}C) spectrum data confirmed the formation of the proton transfer salt **5**. ^1H NMR spectrum of $(H_2Ppz)(Py\text{-}2\text{-}c)_2{\cdot}H_2O$ (**5**) exhibited signals at 7.0, 8.0 and 8.1 ppm in the aromatic region for $Py\text{-}2\text{-}c^-$ ring protons and signals at 3.41 ppm for H_2Ppz^{2+} protons. The carbonyl group carbon atom was observed in down field at 169.25 ppm. Five signals in the aromatic region were attributed to the $Py\text{-}2\text{-}c^-$ ring carbons (124.12, 127.51, 143.14, 148.45, 149.15 ppm). The piperazine ring carbons resonated in the high field at 40.49 ppm. From the intensities of the peaks of ^1H NMR spectrum of $(H_2Ppz)(Py\text{-}2\text{-}c)_2{\cdot}H_2O$, the H_2Ppz^{2+} to $Py\text{-}2\text{-}c^-$ molar ratio was found to be 1:2. The proton transfer salt (**5**) contains in its asymmetric unit a H_2Ppz^{2+} cation, two $Py\text{-}2\text{-}c^-$ anions and a H_2O molecule (39) (Figure 6).

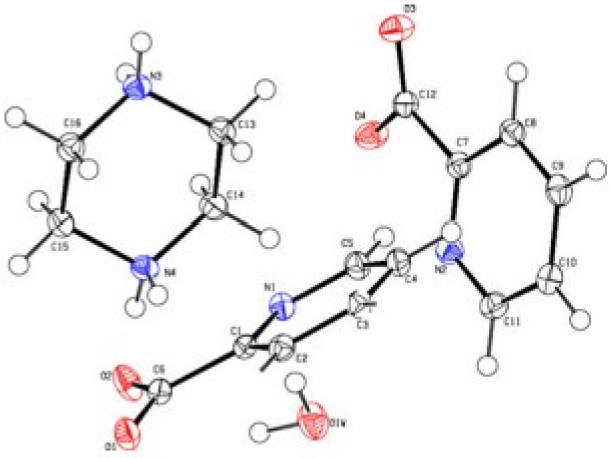


Figure 6: Molecular structure of $(H_2Ppz)(Py\text{-}2\text{-}c)_2{\cdot}H_2O$ (**5**).

This salt is formed through electrostatic attraction between ions $(Py\text{-}2\text{-}c)^-$ and $(H_2Ppz)^{2+}$, different H-bonds (O–H···O, O–H···N, N–H···O, N–H···N, C–H···O), and stacking interactions of C-H···π groups. The crystal structure of **5** exhibits the strongest C–H···π interactions between C5–H5A and N2, the C7–C11 ring and the C11–H11A and N1, C1–C5 ring, with distances of 2.608 Å and 2.627 Å, respectively (39) (Figure 7).

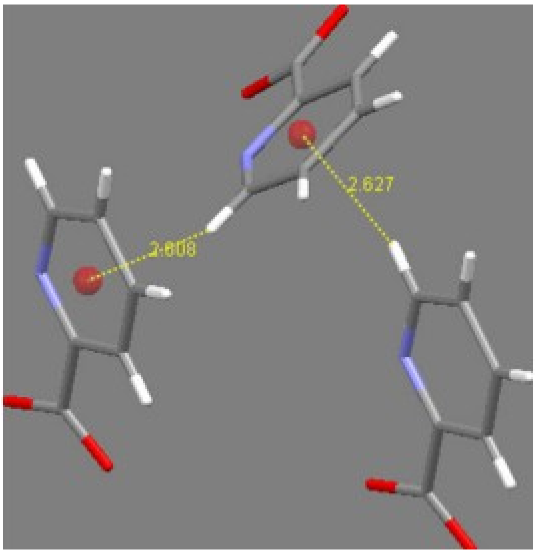


Figure 7: The C-H···π stacking interactions in **5.**

*Mn(II) (**6**), Ni(II) (**7**) and Cu(II) (**8**) complexes of **5***

Complexes of proton transfer salt **5** [Mn(Py-2-c)$_2$(H$_2$O)$_2$]$_2$·2H$_2$O (**6**), [Ni(Py-2-c)$_2$(H$_2$O)$_2$]·H$_2$O (**7**) and [Cu(Py-2-c)$_2$H$_2$O]$_n$ (**8**) were prepared (39) (Figure 8).

The analyzes, crystal systems and space groups of the compounds **5-8** reported in the literature are given in Table 2.

Table 2: Proton transfer salt, (H$_2$Ppz)(Py-2-c)$_2$·H$_2$O (**5**), and its Mn(II) (**6**), Ni(II) (**7**) and Cu(II) (**8**) compounds.

Ref. No	Analyses	Proton Transfer Salt and Complexes	Space Group	Crystal system
(39)	NMR, IR, X-ray and elemental analysis	(H$_2$Ppz)(Py-2-c)$_2$·H$_2$O (**5**)	$P2_1/c$	Monoclinic
(39)	NMR, IR, X-ray and elemental analysis	[Mn(Py-2-c)$_2$(H$_2$O)$_2$]$_2$·2H$_2$O (**6**)	*P 1*	Triclinic
(39)	NMR, IR, X-ray and elemental analysis	[Ni(Py-2-c)$_2$(H$_2$O)$_2$]·H$_2$O (**7**)	$P2_1/n$	Monoclinic
(39)	NMR, IR, X-ray and elemental analysis	[Cu(Py-2-c)$_2$H$_2$O]$_n$ (**8**)	*P 1*	Triclinic

[Mn(Py-2-c)$_2$(H$_2$O)$_2$]$_2$·2H$_2$O (**6**), [Ni(Py-2-c)$_2$(H$_2$O)$_2$]·H$_2$O (**7**) and [Cu(Py-2-c)$_2$H$_2$O]$_n$ (**8**) complexes contain Py-2-c$^-$ ring and water molecule of proton transfer salt (**5**) components. In the molecular structures of the complexes (Figures 9 for **6**, 10a for **7** and 11 for **8**), it was observed that pyridine-2-carboxylate was coordinated to each M(II) ion as a bidentate ligand. In these complexes extensive H-bonds such as O–H···O, O–H···N, N–H···O, N–H···N, C–H···O and C–H···π and C–O···π interactions provides to form a 3-D structure (39).

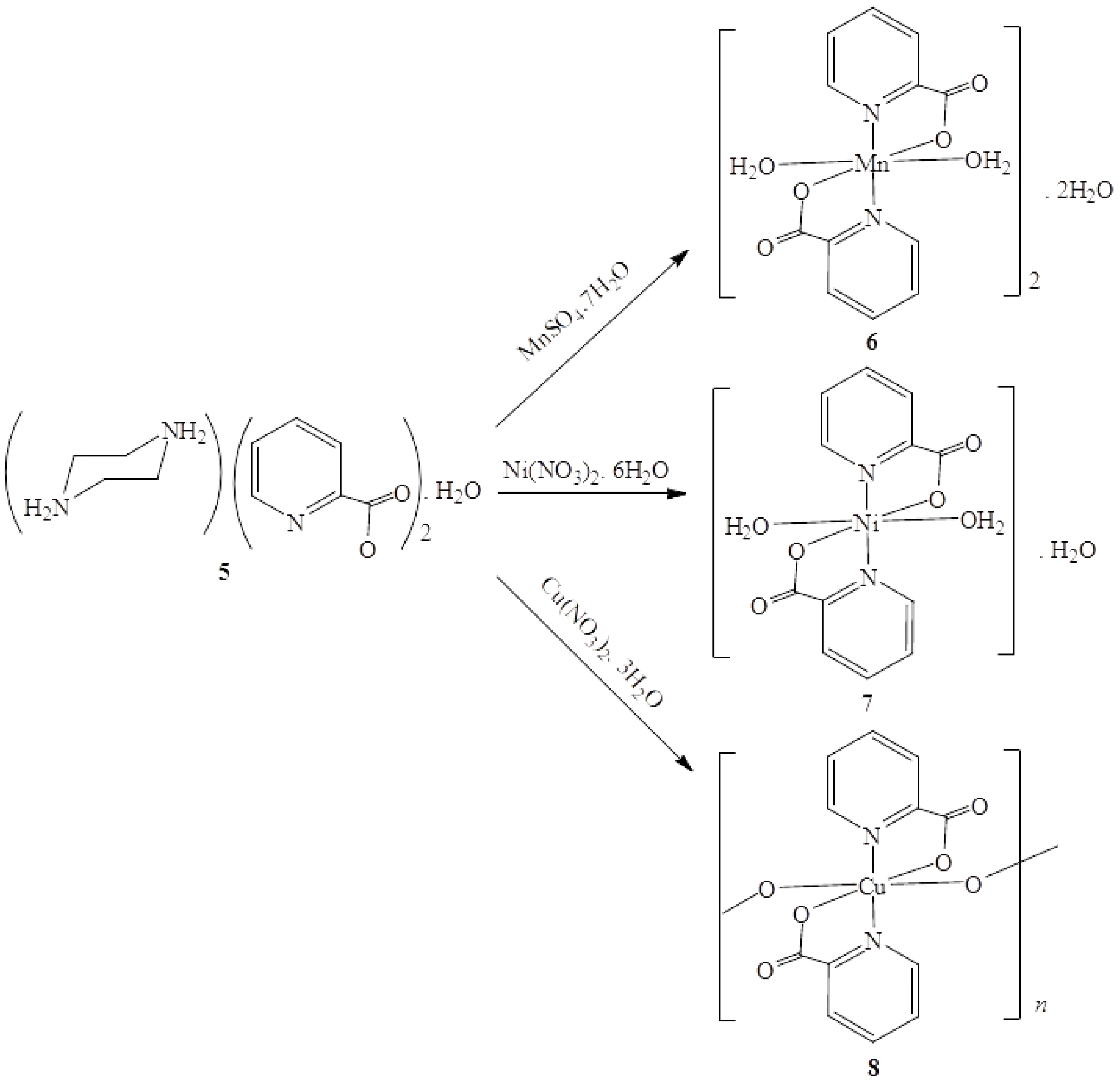


Figure 8: Synthesis of complexes **6**-**8**.

The asymmetric unit of $[Mn(Py\text{-}2\text{-}c)_2(H_2O)_2]_2{\cdot}2H_2O$ (**6**) complex consists of two neutral Mn(II) molecules and H_2O molecules (Figure 9). The two complexes differ slightly in terms of bond angles and bond lengths. Each Mn(II) ion in the complex binds to the nitrogen and oxygen atoms of two Py-2-c-rings, as well as to the oxygens of two H_2O molecules, forming a six-coordinated distorted octahedral geometry. In Mn(II) complex (**6**), it was observed that the O3–Mn1–O5 (164.19(17)°) and O9–Mn2–O11 (167.77(17)°) angles deviated from linearity around Mn^{2+} ions (Mn1 and Mn2). The total bond angles around Mn1 and Mn2 are equal to 362.4°. This means that the atoms form a flattened tetrahedral around the central atom and the Mn^{2+} ion is out of plane (39).

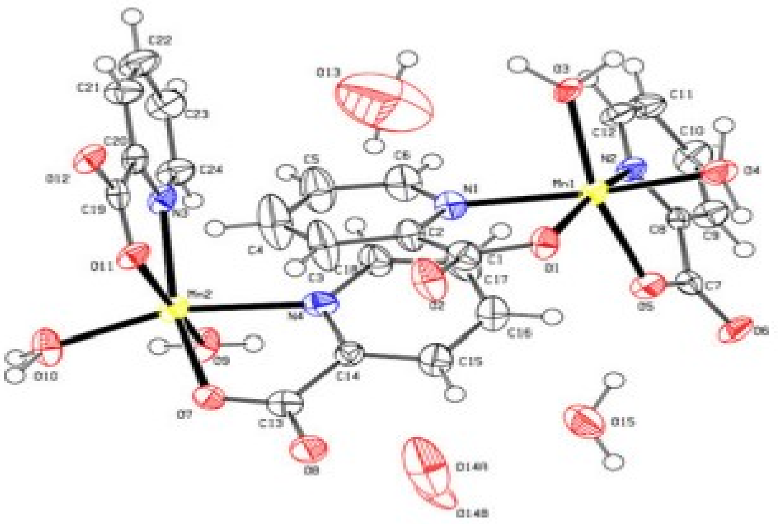
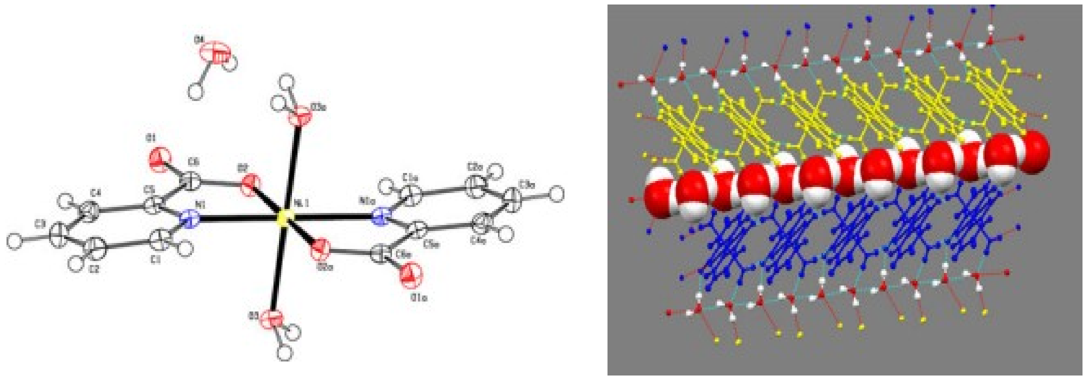


Figure 9: Molecular structure of **6**.

In the [Ni(Py-2-c)$_2$(H$_2$O)$_2$]·H$_2$O (**7**) complex, the Ni(II) ion is coordinated by the two nitrogen and two oxygen atoms of the two Py-2-c$^-$ rings and the oxygens of the two H$_2$O molecules and has a distorted octahedral geometry. The oxygen atoms (O3 and O3a) of the coordinated H$_2$O molecules are in the axial position, while the two oxygen and two nitrogen atoms (O2, O2a, N1 and N1a) of the Py-2-c$^-$ anion are in the equatorial position. The bond angles around Ni(II) (N–Ni–N and O–Ni–O) are 180°, indicating that the coordination around Ni^{2+} is octahedral (Figure 10a). The cavity between the layers of the [Ni(Py-2-c)$_2$(H$_2$O)$_2$] (**7**) part is filled by the H$_2$O molecules (39) (Figure 10b).



(a) (b)

Figure 10: (a) Molecular structure and (b) A layered packing of [Ni(Py-2-c)$_2$(H$_2$O)$_2$]·H$_2$O (**7**).

In the [Cu(Py-2-c)$_2$H$_2$O]$_n$ (**8**) complex, Cu(II) atoms have six coordination environments, with the two nitrogen and two oxygen atoms of the two carboxylate ligands and the two oxygen atoms of the two pyridine 2-carboxylate anions acting as bridges between the two Cu(II) ions.

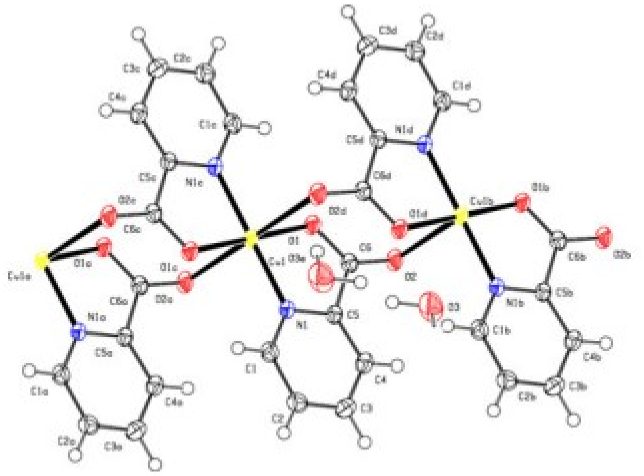


Figure 11: Molecular structure of $[Cu(Py\text{-}2\text{-}c)_2H_2O]_n$ (**8**).

$[Cu(Py\text{-}2\text{-}c)_2H_2O]_n$ (**8**) includes neutral polymeric compound and lattice H_2O molecule (Figure 11). Cu^{2+} ion is coordinated to oxygen [Cu1–O1 1.930(2) Å] and nitrogen Cu1–N1 1.959(2) Å] atoms of two Py-2-c^- anions and two bridging oxygen atoms of the other carboxylate groups. Intramolecular O–H···O and C–H···O H-bonds with D···A ranging from 2.826(4) to 3.202(3) Å stabilize the molecular structure (39).

3. CONCLUSION

In this study, proton transfer salts synthesized from piperazine and pyridine-2-carboxylic acid and their metal complexes from 2009 to the present are presented. It was observed that pyridine-2-carboxylate was coordinated to metal ions but not piperazine in all complexes containing mostly d-block metals. Only one of the studied complexes contains piperazine as a complementary ion.

4. CONFLICT OF INTEREST

The authors declare no conflicts of interest.

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