

A Manganese Coordination Polymer and a Palladium Molecular Compound of 3-Pyridinepropionic acid (HL): $[\text{MnL}_2(\text{H}_2\text{O})_2]_\infty$ and *trans*- $[\text{Pd}(\text{HL})_2\text{Cl}_2]$

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Three coordination polymers, $[\text{ML}_2(\text{H}_2\text{O})_2]$ ($\text{M} = \text{Co}$ (**1**), Ni (**2**), Mn (**3**)), were prepared from metal acetates ($\text{M}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$) and 3-pyridinepropionic acid ($\text{HL} = (3\text{-py})\text{-CH}_2\text{CH}_2\text{COOH}$) by solvent-layer methods. By contrast, a discrete molecular compound, *trans*- $[\text{Pd}(\text{HL})_2\text{Cl}_2]$ (**4**), was synthesized by replacing benzonitrile (PhCN) ligands in *trans*- $[\text{Pd}(\text{PhCN})_2\text{Cl}_2]$ with HL under microwave-heating conditions. Compounds **1–3** have a 2D framework, and compound **4** contains a square-planar Pd metal.

Key Words : Pyridyl-carboxylate terminals, Coordination polymer

Introduction

Coordination polymers are currently under intensive study because of their remarkable applications to various fields, including luminescence, catalysis, host–guest chemistry, magnetism, conductivity, biomedicine, molecular adsorption–desorption, and gas storage.^{1–11} Bis(pyridyl)- or multicarboxylate-type linking ligands are typically employed for the preparation of coordination polymers.^{11–13} Our research group continually reported several linking ligands containing pyridyl–pyridyl, pyridyl–amine, furan–furan, or thiophene–thiophene terminals and their coordination polymers.^{14–31}

Recently, several asymmetric linking ligands, in terms of different terminal groups such as carboxylate and pyridyl or imidazole, were utilized to create intriguing coordination polymers, especially those containing both *d*- and *f*-block metals within their frameworks.^{32–40} Consistent with the HSAB (hard–soft acid–base) concept, the relatively softer *N*-terminal is coordinated to the *d*-block metal, and the relatively harder *O*-terminal is bound to the *f*-block metal in such polymers. Since 2010, our group has prepared a couple of pyridyl–carboxylate-type asymmetric linking ligands (L1–L6 in Chart 1) and their coordination polymers.^{41–48}

Coordination polymers are commonly prepared by various heating methods such as hydrothermal, hydro(solvo)thermal, solvothermal, and reflux methods, which typically require long reaction times. To overcome such vigorous conditions, microwave was recently employed to quickly bring about high heating effects.^{49–57} For example, Filipe's group utilized microwave-heating to synthesize lanthanide coordination polymers.⁵⁰ In addition, Zheng and co-workers prepared 3*d*–4*f* polynuclear metal clusters under microwave-heating conditions.⁵⁴ We also reported on the microwave-assisted preparation of Ag–4*f*⁴² and lanthanide (4*f*)^{43,49} coordination polymers.

3-Pyridinepropionic acid (HL in Chart 1) is a pyridyl–carboxylate-type linking ligand that contains an intervening ethylene ($-\text{CH}_2\text{CH}_2-$) fragment between the two terminal

linking groups. Because of the intervening fragment, this compound resists the hydrolysis during the hydrothermal or hydro(solvo)thermal reaction. For example, Suen's group utilized the 3-pyridinepropionic acid to create three coordination polymers: $\{[\text{AgL}] \cdot (\text{H}_2\text{O})\}$, $[\text{ZnL}_2(\text{H}_2\text{O})_2]$, and $\{[\text{CuL}_2] \cdot 2(\text{H}_2\text{O})\}$.⁵⁸ In addition, LaDuca and co-workers prepared two coordination polymers by employing the same acid: $[\text{CoL}_2(\text{H}_2\text{O})_2]$ and $[\text{NiL}_2(\text{H}_2\text{O})_2]$.⁵⁹ We also prepared a Cd–L coordination polymer, $[\text{CdL}_2(\text{H}_2\text{O})_2]$.⁴⁸ As a continuous work, we attempted to prepare new coordination polymers of HL by different synthetic methods. Herein, we report the synthesis and structures of three coordination polymers, $[\text{ML}_2(\text{H}_2\text{O})_2]$ ($\text{M} = \text{Co}$ (**1**), Ni (**2**), Mn (**3**)), and a discrete Pd(II) complex, *trans*- $[\text{Pd}(\text{HL})_2\text{Cl}_2]$ (**4**). Compounds **1–3** were prepared by the solvent-layering method, whereas compound **4** by the microwave-heating method.

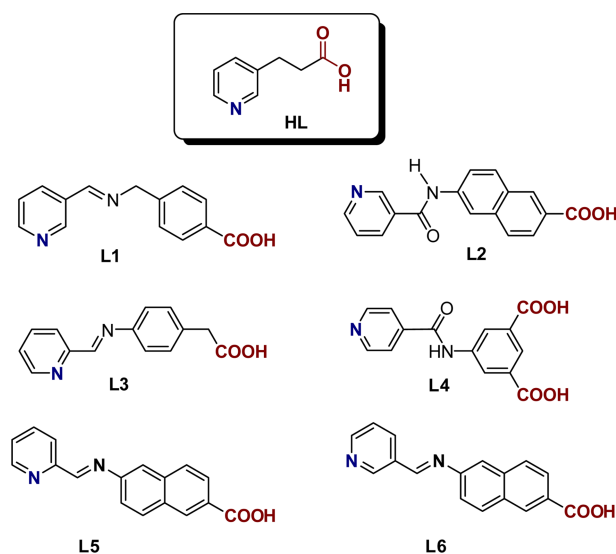


Chart 1. Pyridyl–carboxylate-type linking ligands used by our research group.

Experimental Section

All solid chemicals were purified by recrystallization, and solvents were distilled and stored under argon. IR spectra were obtained in the range of 400–4000 cm^{-1} on a Nicolet 320 FTIR spectrophotometer. Thermogravimetric analysis (TGA) was performed on a TA4000/SDT 2960 instrument at the Cooperative Center for Research Facilities (CCRF) in Sungkyunkwan University. Elemental analyses were carried out by means of an Elementar Vario EL cube (CCRF). For the synthesis of complex **4**, the microwave reactor Anton Paar (No.80868835) was employed.

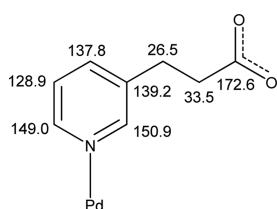
Preparation of Coordination Polymers $[\text{ML}_2(\text{H}_2\text{O})_2]$ ($\text{M} = \text{Co}$ (1**), Ni (**2**), Mn (**3**)).** Compounds **1–3** were synthesized in the same way. 1 N NaOH (0.2 mL, 0.2 mmol) was added to an H_2O (3 mL) solution containing HL (30 mg, 0.2 mmol) to give a colorless solution. Onto the top of this solution was layered a methanol (3 mL) solution containing $\text{Co}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ (29 mg, 0.1 mmol). After 48 h, the resulting pink crystals were separated by filtration, washed with H_2O (10 mL \times 3) and ethanol (10 mL \times 3), and then dried under vacuum to give compound **1**. For the preparation of compounds **2** and **3**, $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ and $\text{Mn}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ were used, respectively.

Data for Compound 1: 87% yield (34 mg, 0.87 mmol). IR (KBr, cm^{-1}): 3259, 1667, 1403, 1307, 1204, 963, 850, 810, 768, 652, 613. Anal. Calc. for $\text{C}_{16}\text{H}_{20}\text{CoN}_2\text{O}_6$: C, 48.62; H, 5.10; N, 7.09. Found: C 48.91; H 4.89; N 6.96.

Data for Compound 2: 82% yield. IR (KBr, cm^{-1}): 3258, 2916, 2660, 2371, 2074, 1788, 1553, 1403, 1306, 1205, 1122, 1030, 963, 922, 853, 782, 707. Anal. Calc. for $\text{C}_{16}\text{H}_{20}\text{NiN}_2\text{O}_6$: C, 48.65; H, 5.10; N, 7.09. Found: C 46.38; H 5.17; N 5.52.

Data for Compound 3: 74% yield. IR (KBr, cm^{-1}): 3173, 2966, 2659, 1664, 1403, 1307, 1249, 1204, 1120, 1047, 962, 708, 650. Anal. Calc. for $\text{C}_{16}\text{H}_{20}\text{MnN}_2\text{O}_6$: C, 49.11; H, 5.15; N, 7.16. Found: C 48.64; H 5.01; N 6.89.

Preparation of *trans*- $[\text{Pd}(\text{HL})_2\text{Cl}_2]$ (4**).** A mixture of HL (30 mg, 0.2 mmol) and *trans*- $[\text{Pd}(\text{PhCN})_2\text{Cl}_2]$ (38 mg, 0.1 mmol) in H_2O (2 mL) was sealed in a 10 mL reusable high-performance reaction vial. The mixture was microwave-heated at 70 $^\circ\text{C}$ for 1 h, and then slowly air-cooled. The resulting yellow crystals were filtered, washed with H_2O (10 mL \times 3) and ethanol (10 mL \times 3), and then dried under vacuum to give compound **4** (10 mg, 0.124 mmol, 61%). mp 188–190 $^\circ\text{C}$. ^1H NMR (DMSO, δ): 8.63–8.58 (m, 2H, aromatic protons), 7.92–7.90 (m, 1H, aromatic proton), 7.49–5.46 (m, 1H aromatic proton), 2.86 (m, 2H, $-\text{CH}_2-$), 2.60 (m, 2H, $-\text{CH}_2-\text{COO}-$), $^{13}\text{C}\{^1\text{H}\}$ NMR, 172.6, 150.9,



149.0, 139.2, 137.8, 128.9, 33.5, 26.5. IR (KBr, cm^{-1}): 3624, 3177, 2925, 2372, 2072, 1733, 1572, 1483, 1433, 1394, 1276, 1166, 912, 813, 769, 695, 572, 418. Anal. Calc. for $\text{C}_{16}\text{H}_{18}\text{Cl}_2\text{N}_2\text{O}_4\text{Pd}$: C, 40.06; H, 3.78; N, 5.84. Found: C 40.43; H 4.30; N 5.81.

X-Ray Structure Determination. All X-ray data were collected with a Bruker Smart APEX2 diffractometer equipped with a Mo X-ray tube (CCRF).⁶⁰ Absorption corrections were made by SADABS on the basis of the Laue symmetry of equivalent reflections.⁶¹ All calculations were carried out with SHELXTL programs.⁶² A colorless crystal of compound **3** (block, 0.40 \times 0.40 \times 0.22 mm^3) and a yellow crystal of compound **4** (plate, 0.10 \times 0.10 \times 0.02 mm^3) were used for crystal- and intensity-data collection. All structures were solved by direct methods.

All non-hydrogen atoms in both compounds were refined anisotropically. In compound **3**, the hydrogen atoms in the aqua ligands were located and refined freely, and the remaining hydrogen atoms were generated in idealized positions and refined in a riding model. In compound **4**, all hydrogen atoms were generated in idealized positions and refined in a riding model. Details on crystal data, intensity collection, and refinement details are given in Table 1. Selected bond lengths and bond angles are given in Table 2.

Table 1. X-ray data collection and structure refinement details

	Compound 3	Compound 4
Empirical formula	$\text{C}_{16}\text{H}_{20}\text{MnN}_2\text{O}_6$	$\text{C}_{16}\text{H}_{18}\text{Cl}_2\text{N}_2\text{O}_4\text{Pd}$
Formula weight	391.28	479.62
Temperature, K	296(2)	296(2)
Crystal system	monoclinic	monoclinic
Space group	$P2_1/n$	$C2/c$
<i>a</i> , Å	9.6670(2)	23.347(2)
<i>b</i> , Å	8.8341(2)	4.3604(4)
<i>c</i> , Å	10.0645(2)	17.696(1)
α , $^\circ$	90	90
β , $^\circ$	104.300(1)	91.742(8)
γ , $^\circ$	90	90
<i>V</i> , Å ³	832.87(3)	1800.7(3)
<i>Z</i>	2	4
<i>D</i> _{calc} , g cm ^{−3}	1.560	1.769
μ , mm	0.829	1.351
<i>F</i> (000)	406	960
θ range $^\circ$	2.62–28.39	1.75–28.32
<i>T</i> _{max}	0.8387	0.9735
<i>T</i> _{min}	0.7328	0.8768
No. of reflections measured	13468	12997
No. of reflections unique	2063	2207
No. of reflections with $I > 2\sigma(I)$	1921	1401
No. of parameters	123	116
Max., in $\Delta\rho$ (e Å ^{−3})	0.304	0.683
Min., in $\Delta\rho$ (e Å ^{−3})	−0.168	−0.741
GOF on <i>F</i> ²	1.060	0.933
<i>R</i> ₁ ^a	0.0216	0.0431
<i>wR</i> ₂ ^b	0.0564	0.0973

^a $R = \sum[|F_o| - |F_c|]/\sum|F_o|$. ^b $wR_2 = \sum[w(F_o^2 - F_c^2)^2]/\sum[w(F_o^2)^2]^{1/2}$

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

Compound 3					
Mn1–O1#2	2.1587(7)	Mn1–O3	2.2027(8)	Mn1–N1	2.2917(9)
O1#2–Mn1–O1#3	180.00(4)	O1#2–Mn1–O3	87.90(3)	O1#3–Mn1–O3	92.10(3)
O1#3–Mn1–N1	91.37(3)	O3–Mn1–N1	89.73(3)		
Compound 4					
Pd1–N1	2.014(3)	Pd1–Cl1	2.313(1)		
N1#5–Pd1–N1	180.000(1)	N1#5–Pd1–Cl1	89.1(1)	N1–Pd1–Cl1	90.9(1)

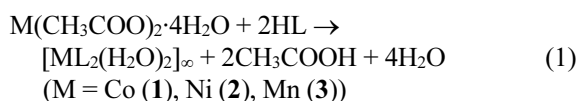
Symmetry transformations used to generate equivalent atoms: #1 = $-x, -y, -z + 1$; #2 = $-x + 1/2, y - 1/2, -z + 1/2$; #3 = $x - 1/2, -y + 1/2, z + 1/2$; #4 = $-x + 1/2, y + 1/2, -z + 1/2$; #5 = $-x - 1/2, -y + 3/2, -z + 1$.

CCDC 943602 and 943603 contain the supplementary crystallographic data for compounds **3** and **4**, and respectively. These data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html> or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

Results and Discussion

Preparation. As mentioned in Introduction, our research group previously prepared a 2D cadmium coordination polymer by employing HL and $\text{Cd}(\text{NO}_3)_2 \cdot 6(\text{H}_2\text{O})$ under hydrothermal conditions.⁴⁸ On the basis of this result, a mixture of a metal acetate tetrahydrate $\text{M}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ and HL in water was heated at 170 °C for 48 h in the presence of NaOH. However, these reactions did not produce crystalline products for X-ray diffraction study, and therefore our synthetic strategy was changed from the hydrothermal method to the solvent-layering method.

Compounds **1–3** were synthesized by layering a methanol solution containing a metal acetate to an aqueous solution containing HL and NaOH. All products were obtained as crystals in relatively high yield (74–87%) (eq. 1), and characterized by IR, elemental analysis, and single X-ray crystallography.



Compounds **1** and **2** were previously prepared in the presence of NaOH under hydrothermal conditions by LaDuca and co-workers.⁵⁹ The former was prepared from cobalt(II) *nitrate* $\{\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}\}$ and HL and the latter **2** from nickel(II) *thiocyanate* $\{\text{Ni}(\text{SCN})_2\}$ and HL. The chemical compositions and structures of our compounds were clearly confirmed by comparing their IR spectra, elemental analysis

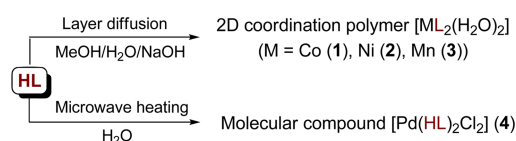
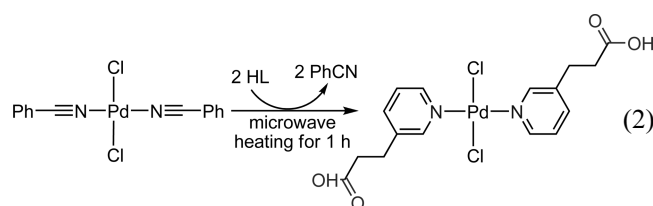
values, and X-ray structures with those in the literature. Consequently, despite the difference in starting metal compounds and synthetic methods, our compounds turned out to be identical to those prepared by the LaDuca's group.

To prepare a Pd–L coordination polymer, the layer-diffusion method was initially attempted by using HL and *trans*- $[\text{Pd}(\text{PhCN})_2\text{Cl}_2]$. However, the reaction gave only powder products whose colors depended on the amount of NaOH used: (i) a black powder in the larger amount of NaOH and (ii) a yellow powder in the smaller amount of NaOH. On the basis of these results, we decided to change the synthetic method from the layer-diffusion method to the microwave-heating method. An aqueous solution containing HL and *trans*- $[\text{Pd}(\text{PhCN})_2\text{Cl}_2]$ was microwave-heated at 70 °C for 1 h to give a discrete molecular species, *trans*- $[\text{Pd}(\text{HL})_2\text{Cl}_2]$ (**4**) (eq. 2). In this reaction, the HL compound replaced the benzonitrile ligands in the *trans*- $[\text{Pd}(\text{PhCN})_2\text{Cl}_2]$.

Compound **4** was fully characterized by spectroscopy (^1H NMR, $^{13}\text{C}\{^1\text{H}\}$ NMR, and IR) and X-ray diffraction. The IR spectrum of compound **4** displays a peak at 3624 cm^{-1} assignable to the OH (COOH in HL) absorption band, without the CN (benzonitrile) stretching peak at $2240\text{--}2260\text{ cm}^{-1}$ of the starting compound *trans*- $[\text{Pd}(\text{PhCN})_2\text{Cl}_2]$.

The structures of compounds **1–4** tell us that the HL ligand has different coordination modes for the first-row metals (Co, Ni, Mn) and the second-row metal (Pd). For the relatively hard Co, Ni, and Mn metals, the HL ligand utilizes both its terminal groups to form 2D coordination polymers. By contrast, for the relatively soft Pd metal, it coordinates to the metal *via* the softer pyridyl terminal and its harder carboxylate terminal remains uncoordinated (Scheme 1).

Structure of a Polymeric Mn(II) Compound (3). Compounds **3** is isostructural with $\{[\text{CuL}_2] \cdot 2(\text{H}_2\text{O})\}_\infty$,⁵⁸ $[\text{ZnL}_2 \cdot (\text{H}_2\text{O})_2]_\infty$,⁵⁸ $[\text{CoL}_2(\text{H}_2\text{O})_2]_\infty$,⁵⁹ $[\text{NiL}_2(\text{H}_2\text{O})_2]_\infty$,⁵⁹ and $[\text{CdL}_2 \cdot (\text{H}_2\text{O})_2]_\infty$ ⁴⁸ in literature. An asymmetric of compound **3** is given in Figure 1, which shows one-half Mn^{2+} ion, one anionic ligand (L), and one aqua ligand. The local coordination environment of the Mn^{2+} ion is given Figure 2, which

**Scheme 1.** Formation of compounds **1–4**, depending on the coordination modes of HL.

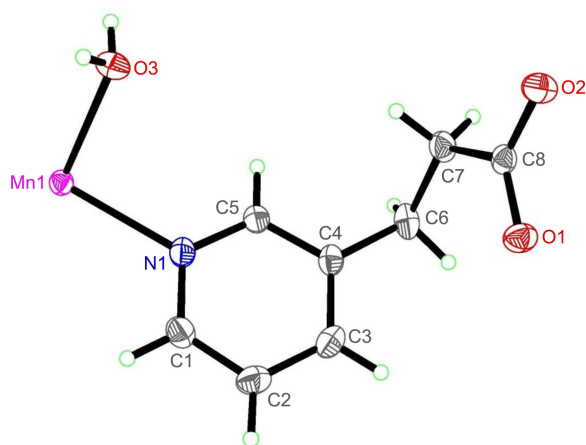


Figure 1. Asymmetric unit of compound **3**.

shows an octahedral Mn^{2+} ion. The Mn^{2+} ion is located on the crystallographic center of symmetry, and the remaining atoms occupy general positions. The Mn^{2+} ion is coordinated to four oxygen atoms and two nitrogen atoms. Two oxygen atoms in the carboxylate terminal behave differently; that is, one is coordinated to the Mn^{2+} ion and the other participates

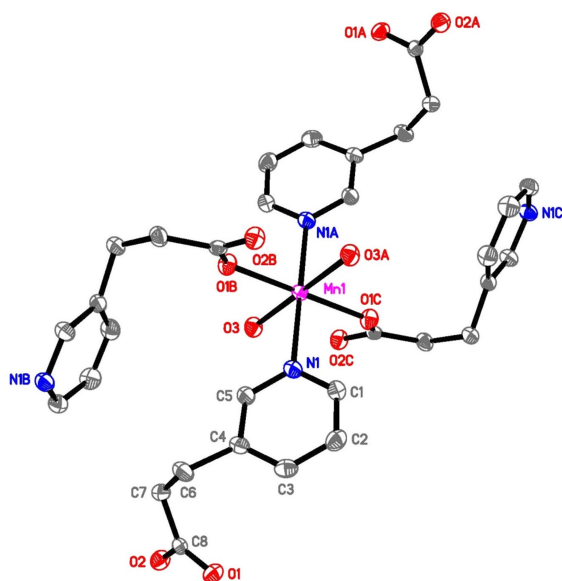


Figure 2. Local coordination environment around the Mn^{2+} ion in compound **3**.

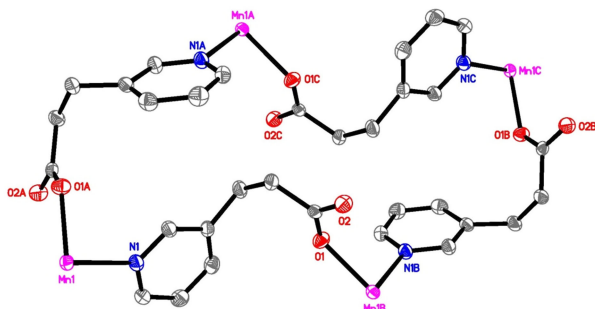


Figure 3. Repeat unit in compound **3**.

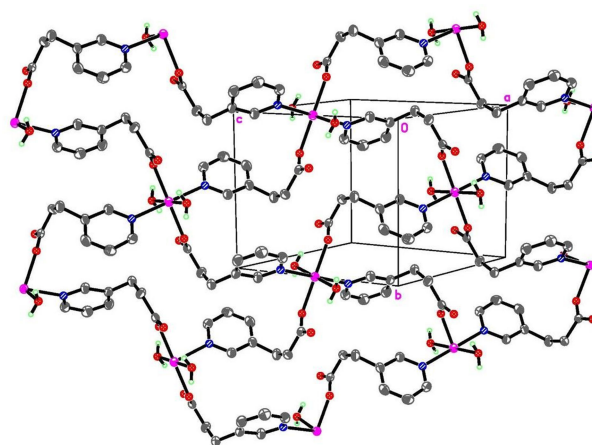


Figure 4. Linking of the repeat units of compound **3** by edge-sharing to form a 2D framework.

in the $\text{O-H}\cdots\text{O}$ hydrogen bond.

Figure 3 illustrates the repeat unit in compound **3**, which consists of four Mn^{2+} ions and four ligands. The repeat unit is a 36-membered ring (four Mn^{2+} ions and 32 ligand atoms), and the $\text{Mn}^{2+}\cdots\text{Mn}^{2+}$ separation is 8.9561(1) Å. The repeat units are linked by edge-sharing in the [101] direction to form a 2-D framework (Figure 4). The $\text{O-H}\cdots\text{O}$ hydrogen bonds connect the 2-D frameworks to create a three-dimensional network (Figure 5).

To examine the thermal stability of compound **3**, TGA (thermogravimetric analysis) was performed. The TGA curve displays two rather well-defined weight-loss steps. The first weight loss in the wide range of 132–190 °C may be assigned to the elimination of the two aqua ligands (calculated: 10.0%; observed: 10.5%). The resulting framework appears to be stable from 190 to 303 °C, above which it decomposes rapidly.

Structure of a Discrete Pd(II) Compound (4). The

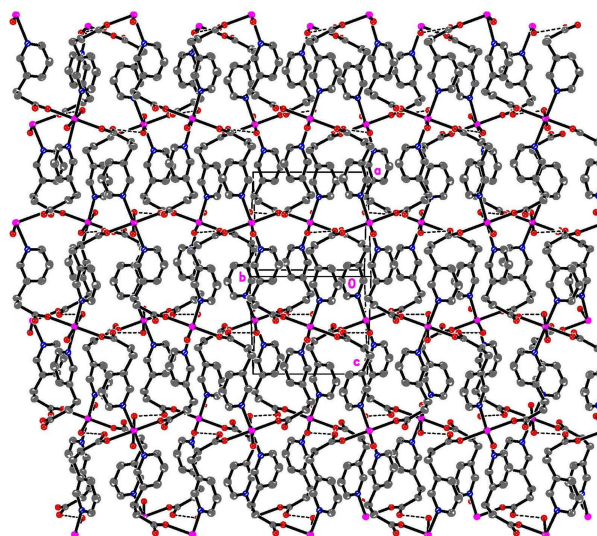


Figure 5. Packing diagram perpendicular to the (101) plane, showing a three-dimensional network. The dotted lines denote intermolecular $\text{O-H}\cdots\text{O}$ hydrogen bonds.

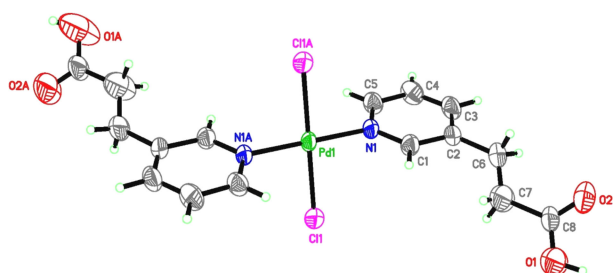


Figure 6. Molecular structure of compound **4** showing the atomic numbering and 40% probability thermal ellipsoids.

molecular structure of compound **4** with the atom-numbering scheme is shown in Figure 6. Only half the molecule consisting of one half Pd(II) atom, one HL ligand, and one Cl ligand forms an asymmetric unit. The Pd atom lies on the crystallographic inversion center, and the remaining atoms occupy general positions. The coordination sphere of each Pd(II) atom can be described as square planar, with the HL ligands *trans* to each other. The molecular plane, PdN₂Cl₂, is perfectly planar due to its generation of the inversion operation.

The HL ligand is bonded to the Pd atom through the pyridyl terminal, and the carboxylate terminal is not. The bonding modes of the ligand contrasts with those found in compound **3**, in which both its pyridyl and carboxylate terminals are bonded to the metal (Mn²⁺). Compound **4** may act as a secondary building unit for the preparation of *d-f* coordination polymers, because it has two uncoordinated carboxylate terminals available for bonding to the *f*-block metals. This synthetic strategy is currently under study. As a comparison, we recently reported a two-step synthetic route to Ag-4*f* coordination polymers by using the 4*f*-L complexes ([Ln₂L₆(H₂O)₄]·{[Ln₂L₄(H₂O)₈](NO₃)₂}) (Ln = Eu, Tb, Nd) as secondary building units.⁴²

In summary, three coordination polymers, [ML₂(H₂O)₂]_∞ (M = Co (**1**), Ni (**2**), Mn (**3**)), were prepared from metal acetates (M(CH₃COO)₂·4H₂O) and 3pyridinepropionic acid (HL = (3-py)-CH₂CH₂COOH) by solvent-layer methods. By contrast, this acid replaced the benzonitrile (PhCN) ligands in the Pd-bis(benzonitrile) complex *trans*-[Pd(PhCN)₂Cl₂] under microwave-heating conditions to give a discrete molecular compound, *trans*-[Pd(HL)₂Cl₂] (**4**). Compounds **1** and **2** were previously synthesized from different starting metal compounds by different synthetic methods. Compound **3** has a two-dimensional framework. Compound **4** contains a square-planar Pd metal.

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