Article

# Synthesis, Structures, and Magnetism of Four One-Dimensional Complexes Using $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$ and Macrocyclic Metal Complexes 

Guangchuan Ou *, Qiong Wang, Yingzhi Tan and Qiang Zhou

College of Chemistry and Bioengineering, Hunan University of Science and Engineering, Yongzhou 425199, China

* Correspondence: ogcouguangchuan@huse.cn; Tel.: +86-74-6638-1164

Citation: Ou, G.; Wang, Q.; Tan, Y.; Zhou, Q. Synthesis, Structures, and Magnetism of Four One-Dimensional Complexes Using $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$ and Macrocyclic Metal Complexes.
Molecules 2023, 28, 4529. https:/ / doi.org/10.3390/molecules28114529

Academic Editors: Takashiro Akitsu and Yu G. Gorbunova

Received: 6 April 2023
Revised: 9 May 2023
Accepted: 29 May 2023
Published: 2 June 2023


Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/).


#### Abstract

Four one-dimensional complexes, denoted as $\left[\mathrm{NiL}_{1}\right]\left[\mathrm{Ni}(\mathrm{CN})_{4}\right](\mathbf{1}),\left[\mathrm{CuL}_{1}\right]\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]$ (2), $\left[\mathrm{NiL}_{2}\right]\left[\mathrm{Ni}(\mathrm{CN})_{4}\right] \cdot 2 \mathrm{H}_{2} \mathrm{O}(3)$, and $\left[\mathrm{CuL}_{2}\right]\left[\mathrm{Ni}(\mathrm{CN})_{4}\right] \cdot 2 \mathrm{H}_{2} \mathrm{O}(4)\left(\mathrm{L}_{1}=1,8\right.$-dimethyl-1,3,6,8,10,13-hexaazacyclotetradecane; $\mathrm{L}_{2}=1,8$-dipropyl-1,3,6,8,10,13-hexaazacyclotetradecane) were synthesized by reacting nickel/copper macrocyclic complexes with $\mathrm{K}_{2}\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]$. Subsequently, the synthesized complexes were characterized using elemental analysis, infrared spectroscopy analysis, thermogravimetric analysis, and X-ray powder diffraction. Single-crystal structure analysis revealed that the $\mathrm{Ni}(\mathrm{II}) / \mathrm{Cu}(\mathrm{II})$ atoms were coordinated by two nitrogen atoms from $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$ with four nitrogen atoms from a macrocyclic ligand, forming a six-coordinated octahedral coordination geometry. Nickel/copper macrocyclic complexes were bridged by $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$ to construct one-dimensional chain structures in 1-4. The characterization results showed that the four complexes obeyed the Curie-Weiss law with a weak antiferromagnetic exchange coupling.


Keywords: macrocyclic metal complexes; tetracyanonicolate; magnetism

## 1. Introduction

The rational construction of one to three-dimensional porous coordination complexes has attracted considerable attention because of their potential applications in gas adsorption and separation, catalysis, and magnetism [1-4]. The design and assembly of different dimensional cyano-bridged complexes have been extensively researched in recent decades. Cyano-bridged complexes with superior magnetic properties can be constructed using cyano complexes, such as $\left[\mathrm{Ag}(\mathrm{CN})_{2}\right]^{-},\left[\mathrm{Cu}(\mathrm{CN})_{3}\right]^{2-},\left[\mathrm{M}(\mathrm{CN})_{4}\right]^{2-}(\mathrm{M}=\mathrm{Cd}, \mathrm{Ni}, \mathrm{Pd}$, and $\mathrm{Pt})$, and $\left[\mathrm{M}(\mathrm{CN})_{6}\right]^{3-}(\mathrm{M}=\mathrm{W}, \mathrm{Mo}$, and Fe$)[5-15]$. Studies on tetracyanometallate anions $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$ exhibiting bridging characteristics with either one or two of the cyano groups have been reported [16-28].

In previous reports, a few cyano-bridged complexes with one-dimensional helical chains were prepared and structurally characterized using transition metal macrocyclic complexes $\left[\mathrm{NiL}_{0}\right]^{2+}\left(\mathrm{L}_{0}=5,5,7,12,12,14\right.$-hexamethyl-1,4,8,11-tetraazacyclo-tetradecane) and $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}[24]$. As a continuation of the above research, we used different macrocyclic ligands $\left(\mathrm{L}_{1}=1,8\right.$-dimethyl-1,3,6,8,10,13-hexaazacyclotetradecane, $\mathrm{L}_{2}=1,8$-dipropyl-1,3,6,8,10,13hexaazacyclotetradecane, represented in Scheme 1) for the construction of cyano-bridged complexes. In this study, we synthesized four cyano-bridged complexes: $\left[\mathrm{NiL}_{1}\right]\left[\mathrm{Ni}(\mathrm{CN})_{4}\right](\mathbf{1})$, $\left.[\mathrm{CuL}]_{1}\right]\left[\mathrm{Ni}(\mathrm{CN})_{4}\right](2),\left[\mathrm{NiL}_{2}\right]\left[\mathrm{Ni}(\mathrm{CN})_{4}\right] \cdot 2 \mathrm{H}_{2} \mathrm{O}(3)$, and $\left[\mathrm{CuL}_{2}\right]\left[\mathrm{Ni}(\mathrm{CN})_{4}\right] \cdot 2 \mathrm{H}_{2} \mathrm{O}(4)$, which were isolated from the reactions of $[\mathrm{ML}]\left(\mathrm{ClO}_{4}\right)_{2}$ with $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$. In addition, the structures and magnetism of four cyano-bridged complexes were analyzed.


Scheme 1. Structural formula of macrocyclic complexes $\left(R_{1}=\mathrm{CH}_{3}, \mathrm{R}_{2}=\mathrm{C}_{3} \mathrm{H}_{7}\right.$, and $\mathrm{M}=\mathrm{Ni}$ or Cu$)$.

## 2. Results and Discussion

### 2.1. Description of Structures

Figure 1 shows the similarity between the structures of complexes 1 and 2. One asymmetric unit of the structure of complex 1 contained one $\left[\mathrm{NiL}_{1}\right]^{2+}$ cation and one $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$ anion. Each $\mathrm{Ni}(1)$ was on an inversion center and coordinated with four nitrogen atoms, namely $\mathrm{N}(1), \mathrm{N}(2), \mathrm{N}(1 \mathrm{~A})$, and $\mathrm{N}(2 \mathrm{~A})$ (symmetry code: $\mathrm{A}, 1-x, 1-y$, and $1-z)$, of the macrocyclic ligand in the equatorial plane and two nitrogen atoms, namely, $\mathrm{N}(4)$ and $\mathrm{N}(4 \mathrm{~B})$ (symmetry code: $\mathrm{B},-x,-y, 1-z)$, of $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$ in axial positions, forming a six-coordinated octahedral geometry in 1 . The distances [2.0498(16) and 2.0712(16) A] of $\mathrm{Ni}(1)-\mathrm{N}$ (macrocycle) were shorter than that of the $\mathrm{Ni}(1)-\mathrm{N}($ cyano $)$ [2.1489(17) $\AA$ ] , and they were longer than that of the $\mathrm{Ni}(2)-\mathrm{C}($ cyano $)[1.858(2)-1.866(2) \AA]$ (Table 1). The $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$ anion bridged the macrocyclic complex $\left[\mathrm{NiL}_{1}\right]^{2+}$ cation in trans-positions to form a one-dimensional chain along the $b$-axis (Figure 1). One-dimensional linear chains were formed when trans-M(II) tetradentate macrocyclic complexes were used because of steric hindrance. Four possible helical isomers can be formed when cis-M(II) tetradentate macrocyclic complexes and $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$ building blocks are used $[18,19,24]$.


Figure 1. (I) The structure of complexes 1 and $2(M=N i$ or Cu$)$ and the symmetry codes for the generated atoms: $\mathrm{A}(1-x, 1-y, 1-z), \mathrm{B}(-x,-y, 1-z)$; (II) The $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$ bridges macrocyclic complex $\left[\mathrm{ML}_{1}\right]^{2+}$ to form a one-dimensional chain along the $b$-axis.

The six-coordinated $\mathrm{Cu}(\mathrm{II})$ ions of complex 2 displayed a distorted octahedral geometry by coordinating with four nitrogen atoms, namely, $\mathrm{N}(1), \mathrm{N}(2), \mathrm{N}(1 \mathrm{~A})$, and $\mathrm{N}(2 \mathrm{~A})$ (symmetry code: A, 1-x, 1-y, 1-z), of macrocyclic ligand in the equatorial plane and two nitrogen atoms, namely, $\mathrm{N}(5)$ and $\mathrm{N}(5 \mathrm{~B})$ (symmetry code: $\mathrm{B},-x,-y, 1-z)$ of $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$ in axial positions (Figure 1). The $\mathrm{Cu}(1)-\mathrm{N}($ macrocycle) distances [2.0009(15) and $2.0240(16) \AA$ ] ] were shorter than the $\mathrm{Cu}(1)-\mathrm{N}$ (cyano) distances $[2.563(2) \AA$ ] due to the Jahn-Teller effect, but longer than the distances $[1.861(2)-1.865(2) \AA]$ of $\mathrm{Ni}(1)-\mathrm{C}\left(\right.$ cyano ) (Table 1). The $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$ anion bridged the macrocyclic complex $\left[\mathrm{CuL}_{1}\right]^{2+}$ cation in trans-positions to form a onedimensional chain along the $b$-axis in 2 (Figure 1). Complexes 3 and 4 exhibited similar onedimensional structures. The asymmetric unit of complex $3 / 4$ contained one $\left[\mathrm{Ni} / \mathrm{CuL}_{2}\right]^{2+}$ cation, one $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$ anion, and two water molecules (Figure 2). The $\mathrm{Ni}(\mathrm{II}) / \mathrm{Cu}(\mathrm{II})$ ions were coordinated by four nitrogen atoms of ligands along the equatorial plane and two nitrogen atoms of $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$ in axial positions. The $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$ anion bridged the
macrocyclic complex $\left[\mathrm{Ni} / \mathrm{CuL}_{2}\right]^{2+}$ cation in trans-positions to form a one-dimensional chain along the $b$-axis in 3/4 (Figure 2).

Table 1. Selected bond distances ( $\AA$ ) and angles $\left({ }^{\circ}\right)$.

| 1 |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: |
| Bond | Length | Bond | Length | Bond | Length |
| $\mathrm{Ni}(1)-\mathrm{N}(1)$ | $2.0498(16)$ | $\mathrm{Ni}(1)-\mathrm{N}(2)$ | 2.0712(16) | $\mathrm{Ni}(1)-\mathrm{N}(4)$ | 2.1489 (17) |
| $\mathrm{Ni}(2)-\mathrm{C}(6)$ | 1.866(2) | $\mathrm{Ni}(2)-\mathrm{C}(7)$ | 1.858(2) |  |  |
| Bond | Angle | Bond | Angle | Bond | Angle |
| $\mathrm{N}(1)-\mathrm{Ni}(1)-\mathrm{N}(2)$ | 94.15(7) | $\mathrm{N}(1)-\mathrm{Ni}(1)-\mathrm{N}(2){ }^{\# 1}$ | 85.85(7) | $\mathrm{N}(1)-\mathrm{Ni}(1)-\mathrm{N}(4)^{\# 1}$ | 90.27(6) |
| $\mathrm{N}(2)-\mathrm{Ni}(1)-\mathrm{N}(4){ }^{\# 1}$ | 88.33(6) | $\mathrm{N}(1)-\mathrm{Ni}(1)-\mathrm{N}(4)$ | 89.73(6) | $\mathrm{N}(2)-\mathrm{Ni}(1)-\mathrm{N}(4)$ | 91.67(6) |
| $\mathrm{N}(5)-\mathrm{C}(7)-\mathrm{Ni}(2)$ | 176.0(2) | $\mathrm{N}(4)-\mathrm{C}(6)-\mathrm{Ni}(2)$ | 171.6(7) | $\mathrm{C}(6)-\mathrm{N}(6)-\mathrm{Ni}(1)$ | 149.7(5) |
| 2 |  |  |  |  |  |
| Bond | Length | Bond | Length | Bond | Length |
| $\mathrm{Cu}(1)-\mathrm{N}(1)$ | 2.0009 (15) | $\mathrm{Cu}(1)-\mathrm{N}(2)$ | 2.0240 (16) | $\mathrm{Cu}(1)-\mathrm{N}(5)$ | 2.563(2) |
| $\mathrm{Ni}(1)-\mathrm{C}(7)$ | 1.865(2) | $\mathrm{Ni}(1)-\mathrm{C}(6)$ | 1.861(2) |  |  |
| Bond | Angle | Bond | Angle | Bond | Angle |
| $\mathrm{N}(1)-\mathrm{Cu}(1)-\mathrm{N}(2)$ | 93.72(7) | $\mathrm{N}(1)-\mathrm{Cu}(1)-\mathrm{N}(2){ }^{\# 1}$ | 86.28(7) | $\mathrm{C}(6)-\mathrm{Ni}(1)-\mathrm{C}(7)$ | 88.08(9) |
| $\mathrm{C}(6)-\mathrm{Ni}(1)-\mathrm{C}(7)^{\# 2}$ | 91.92(9) | $\mathrm{N}(5)-\mathrm{C}(7)-\mathrm{Ni}(1)$ | 175.7(9) | $\mathrm{N}(4)-\mathrm{C}(6)-\mathrm{Ni}(1)$ | 177.3(2) |
| $\mathrm{C}(7)-\mathrm{N}(5)-\mathrm{Cu}(1)$ | 127.8(8) |  |  |  |  |
| 3 |  |  |  |  |  |
| Bond | Length | Bond | Length | Bond | Length |
| $\mathrm{Ni}(1)-\mathrm{N}(1)$ | 2.061(2) | $\mathrm{Ni}(1)-\mathrm{N}(2)$ | 2.068(2) | $\mathrm{Ni}(1)-\mathrm{N}(4)$ | 2.118(2) |
| $\mathrm{Ni}(2)-\mathrm{C}(8)$ | 1.864(3) | $\mathrm{Ni}(2)-\mathrm{C}(9)$ | 1.862(3) |  |  |
| Bond | Angle | Bond | Angle | Bond | Angle |
| $\mathrm{N}(1)-\mathrm{Ni}(1)-\mathrm{N}(2){ }^{\# 1}$ | 85.66(10) | $\mathrm{N}(1)-\mathrm{Ni}(1)-\mathrm{N}(2)$ | 94.34(10) | $\mathrm{N}(1)-\mathrm{Ni}(1)-\mathrm{N}(4)$ | 92.09(10) |
| $\mathrm{N}(2)-\mathrm{Ni}(1)-\mathrm{N}(4)$ | 91.21(9) | $\mathrm{N}(1)-\mathrm{Ni}(1)-\mathrm{N}(4){ }^{\text {\#1 }}$ | 87.91(10) | $\mathrm{N}(2)-\mathrm{Ni}(1)-\mathrm{N}(4)^{\# 1}$ | 88.79(9) |
| $\mathrm{C}(9)-\mathrm{Ni}(2)-\mathrm{C}(8)$ | 91.14(11) | $\mathrm{C}(9)-\mathrm{Ni}(2)-\mathrm{C}(8){ }^{\# 3}$ | 88.86(11) | $\mathrm{N}(4)-\mathrm{C}(8)-\mathrm{Ni}(2)$ | 177.1(2) |
| $\mathrm{N}(5)-\mathrm{C}(9)-\mathrm{Ni}(2)$ | 178.9(3) | $\mathrm{C}(8)-\mathrm{N}(4)-\mathrm{Ni}(1)$ | 148.4(2) |  |  |
| 4 |  |  |  |  |  |
| Bond | Length | Bond | Length | Bond | Length |
| $\mathrm{Cu}(1)-\mathrm{N}(1)$ | 2.016(2) | $\mathrm{Cu}(1)-\mathrm{N}(2)$ | 2.0159(19) | $\mathrm{Cu}(1)-\mathrm{N}(4)$ | 2.410(2) |
| $\mathrm{Ni}(1)-\mathrm{C}(8)$ | 1.864(3) | $\mathrm{Ni}(1)-\mathrm{C}(9)$ | 1.865(3) |  |  |
| Bond | Angle | Bond | Angle | Bond | Angle |
| $\mathrm{N}(1)-\mathrm{Cu}(1)-\mathrm{N}(2){ }^{\# 4}$ | 85.93(8) | $\mathrm{N}(1)-\mathrm{Cu}(1)-\mathrm{N}(2)$ | 94.07(8) | $\mathrm{N}(1)-\mathrm{Cu}(1)-\mathrm{N}(4){ }^{\text {\# }}$ | 91.93(8) |
| $\mathrm{N}(2)-\mathrm{Cu}(1)-\mathrm{N}(4){ }^{\# 4}$ | 91.37(9) | $\mathrm{N}(1)-\mathrm{Cu}(1)-\mathrm{N}(4)$ | 88.07(8) | $\mathrm{N}(2)-\mathrm{Cu}(1)-\mathrm{N}(4)$ | 88.63(9) |
| $\mathrm{C}(8)-\mathrm{Ni}(1)-\mathrm{C}(9)$ | 90.18(11) | $\mathrm{C}(8)-\mathrm{Ni}(1)-\mathrm{C}(9){ }^{\# 5}$ | 89.82(11) | $\mathrm{N}(4)-\mathrm{C}(8)-\mathrm{Ni}(1)$ | 178.0(2) |
| $\mathrm{N}(5)-\mathrm{C}(9)-\mathrm{Ni}(1)$ | 178.8(3) | $\mathrm{C}(8)-\mathrm{N}(4)-\mathrm{Cu}(1)$ | 160.8(2) |  |  |

Symmetry transformation: ${ }^{\# 1}-x+1,-y+1,-z+1$; $^{\# 2}-x,-y+1,-z+1 ;{ }^{\# 3}-x+2,-y+1,-z ;{ }^{\# 4}-x+1,-y+1$,
$-z+2 ;^{\# 5}-x+2,-y+1,-z+1$.


Figure 2. (I) The structure of complexes 3 and $4(M=N i$ or Cu$)$ and the symmetry codes for the generated atoms: $\mathrm{A}(1-x, 1-y, 1-z), \mathrm{B}(-x,-y, 1-z)$; (II) The $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$ bridges macrocyclic complex $\left[\mathrm{ML}_{2}\right]^{2+}$ to form a one-dimensional chain along the $b$-axis.

Due to the Jahn-Teller effect, the axial $\mathrm{Cu}-\mathrm{N}$ (cyano) bonds were considerably longer [2.563(2) in 2 and 2.410(2) A in 4 (Table 1)] than those of the equatorial with mean values
of $\mathrm{Cu}-\mathrm{N}($ macrocycle) bonds of $2.0125(15)$ and $2.0159(19) \AA$ in 2 and 4, respectively. The five-membered intra-chelate $\mathrm{N}-\mathrm{M}-\mathrm{N}$ angles had similar values [85.85(7), 86.28(7), 85.66(10), and $85.93(8) \AA$ for $\mathbf{1}-4$, respectively]. The $\mathrm{Ni}-\mathrm{C}-\mathrm{N}$ angles slightly deviated from linearity, and the maximum deviation was observed for the $\mathrm{Ni}-\mathrm{C}-\mathrm{N}$ angle $\left[171.6(7)^{\circ}\right]$ in complex 1 , and the $\mathrm{Ni}-\mathrm{C}-\mathrm{N}$ angles of coordinated cyano groups $\left[171.6(7)^{\circ}, 175.7(9)^{\circ}, 177.1(2)^{\circ}\right.$, and $178.0(2)^{\circ}$ for $\mathbf{1 - 4}$, respectively] were smaller than those of uncoordinated cyano groups [176.0(2) ${ }^{\circ}, 177.3(2)^{\circ}, 178.9(3)^{\circ}$, and $178.8(3)^{\circ}$ for $1-4$, respectively]. However, the M-C-N angles significantly deviated from linearity. The $\mathrm{M}-\mathrm{C}-\mathrm{N}$ angles of the $\mathrm{L}_{1}$ ligand $\left[149.7(5)^{\circ}\right.$ and $127.8(8)^{\circ}$ for $\mathbf{1}$ and 2 , respectively] were smaller than the $\mathrm{M}-\mathrm{C}-\mathrm{N}$ angles of $\mathrm{L}_{2}$ ligand [168.4(2) ${ }^{\circ}$ and $160.8(2)^{\circ}$ for 3 and 4, respectively], shortening the distances of neighboring macrocyclic metal center [9.834(2), 9.906(2), 10.187(2), and 10.647(2) Å for 1-4].

### 2.2. IR Spectra

The infrared spectra (Figures S1-S4) of complexes 3 and 4 had broad absorption bands near $3400 \mathrm{~cm}^{-1}$, and the absorption was attributed to the stretching vibration of $\mathrm{O}-\mathrm{H}$. The $\mathrm{N}-\mathrm{H}$ absorption bands appeared near $3200 \mathrm{~cm}^{-1}$, and the absorption bands at 2136 (s) and 2125 (s), 2123 (s) and 2119 (s), 2152 (s) and 2131 (s), and 2136 (s) and 2129 (s) $\mathrm{cm}^{-1}$ were assigned to $v(\mathrm{M}-\mathrm{CN}-\mathrm{M})$ and $v(\mathrm{M}-\mathrm{CN})(\mathrm{M}=\mathrm{Ni}$ or Cu$)$ in complexes $\mathbf{1 - 4}$.

## 2.3. $X R D$ and $T G$

X-ray powder diffraction measurements for 1-4 (Figure 3) showed that the peaks in the measured patterns for both complexes closely matched those in the simulated patterns generated from single-crystal diffraction data, indicating that single phases were formed.


Figure 3. XRD patterns of complexes 1-4.
Figure 4 shows the thermogravimetric analysis (TGA) curves of both complexes. The TGA of complex 1 revealed that a weight loss of approximately $24.2 \%$ occurred from room temperature to 613 K , corresponding to the release of adsorbed water from the air ( $2.3 \%$ ) and four $\mathrm{CN}^{-}$(calcd 22.5\%).


Figure 4. TGA curves of complexes 1-4.
The decomposition of the macrocyclic structure was observed after the further heating of the macrocyclic ligand. The TGA curve for 2 showed the first weight loss from room temperature to 523 K , and the observed weight loss of $22.8 \%$ was related to the release of four $\mathrm{CN}^{-}$(calcd 22.7\%). Then, the macrocyclic structure began to decompose after the macrocyclic ligand was further heated.

The TGA curve for complex 3 revealed that a weight loss of approximately $27.2 \%$ occurred from room temperature to 630 K , which was attributed to the release of adsorbed water from the air ( $2.3 \%$ ), two lattice water molecules (calcd $6.5 \%$ ), and four $\mathrm{CN}^{-}$(calcd $18.6 \%$ ). The weight loss was attributed to the release of structural water molecules and four $\mathrm{CN}^{-}$. The TGA curve of complex 4 was similar to that of complex 3, which revealed that an initial weight loss of $25.3 \%$ (calcd $25.5 \%$ ) occurred from room temperature to 496 K , corresponding to the release of structural water molecules and four $\mathrm{CN}^{-}$.

### 2.4. Magnetism

Magnetic susceptibility measurements were performed to investigate the magnetic behaviors of complexes $\mathbf{1 - 4}$ at 1000 G within the temperature range of $2-300 \mathrm{~K}$. Plots of $\chi_{\mathrm{M}}$ vs. $T$ and $\mu_{\text {eff }} / \mu_{\mathrm{B}}$ vs. T of the complexes within the temperature range of $2-300 \mathrm{~K}$ are shown in Figure 5. Complexes 1 and 3 exhibited similar magnetic properties, and their $\mu_{\text {eff }} / \mu_{\mathrm{B}}$ values within the temperature range of 7-300 K were close to the theoretical value expected for two unpaired d electrons in $\mathrm{Ni}(\mathrm{II})$ ions. In addition, complexes 2 and 4 exhibited similar magnetic properties, and their $\mu_{\mathrm{eff}} / \mu_{\mathrm{B}}$ values within the temperature range of 7-300 K were close to the theoretical value expected for an unpaired d electron in Cu (II) ions.

The magnetic susceptibility measurements between 2 and 300 K yielded $C=1.35 \mathrm{~cm}^{3} \cdot \mathrm{~K} \cdot \mathrm{~mol}^{-1}$ and $\theta=-1.85 \mathrm{~K}$ for 1 , while the corresponding values were $C=0.56 \mathrm{~cm}^{3} \cdot \mathrm{~K} \cdot \mathrm{~mol}^{-1}$ and $\theta=-2.21 \mathrm{~K}$ for $2, C=1.15 \mathrm{~cm}^{3} \cdot \mathrm{~K} \cdot \mathrm{~mol}^{-1}$ and $\theta=-1.79 \mathrm{~K}$ for 3, and $C=0.43 \mathrm{~cm}^{3} \cdot \mathrm{~K} \cdot \mathrm{~mol}^{-1}$ and $\theta=-2.20 \mathrm{~K}$ for 4 . The antiferromagnetic interactions were confirmed because of the negative Weiss constants $\theta$ in complexes $\mathbf{1}-\mathbf{4}$. Weak antiferromagnetic interactions were also observed in analogous complexes $\mathrm{Cu}($ cyclam $) \mathrm{M}(\mathrm{CN})_{4}$ [cyclam $=1,4,8,11$-tetraazacyclotetradecane, $\mathrm{M}=\mathrm{Ni}, \mathrm{Pd}$ ], and the reported values of the Weiss constants $\theta$ were -0.79 K for $\mathrm{M}=\mathrm{Ni}$ [21]. The characterization results showed
that the four complexes 1-4 obeyed the Curie-Weiss law with a weak antiferromagnetic exchange coupling.


Figure 5. Plots of $\chi_{\mathrm{M}}$ vs. $T$ and $\mu_{\text {eff }} / \mu_{\mathrm{B}}$ vs. $T$ (inset) of complexes 1-4 in the temperature range of 2-300 K.

## 3. Materials and Methods

The $\mathrm{Ni}(\mathrm{II})$ and $\mathrm{Cu}(\mathrm{II})$ macrocycle complexes were prepared following the previous report procedure [29]. All of the chemicals used in this work were commercially available and were used without further purification. Elemental analyses were carried out using an Elementar Micro Cube elemental analyzer. Infrared spectra were recorded in the $4000-400 \mathrm{~cm}^{-1}$ region using KBr pellets and a Bruker EQUINOX 55 spectrometer (Bruker, Germany). Thermogravimetric analyses were performed using a Netzsch STA 449F3 instrument (Netzsch, Germany) in flowing air at a heating rate of $10^{\circ} \mathrm{C} \cdot \mathrm{min}^{-1}$. X-ray powder diffraction data were recorded using a Bruker D8 ADVANCE X-ray powder diffractometer ( $\mathrm{Cu} \mathrm{K} \alpha$ radiation, $\lambda=1.5418 \AA$, Bruker, Germany). Magnetic susceptibility measurements were conducted to determine the magnetic behaviors of both complexes at 1000 G in a temperature range of $2-300 \mathrm{~K}$.

## Preparation of the Compounds

$\left[\mathrm{NiL}_{1}\right]\left[\mathrm{Ni}(\mathrm{CN})_{4}\right](1):$ An aqueous solution $(20 \mathrm{~mL})$ of $\mathrm{K}_{2}\left[\mathrm{Ni}(\mathrm{CN})_{4}\right](0.024 \mathrm{~g}, 0.1 \mathrm{mmol})$ was layered on an acetonitrile solution $(20 \mathrm{~mL})$ of $\mathrm{NiL}_{1}\left(\mathrm{ClO}_{4}\right)_{2}(0.048 \mathrm{~g}, 0.1 \mathrm{mmol})$. After a few days, green prism crystals of 1 with $\sim 37 \%$ yield were obtained. Elemental Anal. Found: C, 37.62; H, 5.65; N, 31.25\%. Calcd for $\mathrm{C}_{7} \mathrm{H}_{13} \mathrm{NiN}_{5}$ : C, 37.22; H, 5.80; N, 31.00\%. IR (KBr): 3226 (s), 2946 (m), 2870 (m), 2136 (s), 2125 (s), 1435 (m), 1283 (s), 1059 (m), 1015 (m), 834 (m), and $603(\mathrm{~m}) \mathrm{cm}^{-1}$.
$\left[\mathrm{CuL}_{1}\right]\left[\mathrm{Ni}(\mathrm{CN})_{4}\right](2)$ : An aqueous solution $(20 \mathrm{~mL})$ of $\mathrm{K}_{2}\left[\mathrm{Ni}(\mathrm{CN})_{4}\right](0.024 \mathrm{~g}, 0.1 \mathrm{mmol})$ was layered on an acetonitrile solution $(20 \mathrm{~mL})$ of $\mathrm{CuL}_{1}\left(\mathrm{ClO}_{4}\right)_{2}(0.049 \mathrm{~g}, 0.1 \mathrm{mmol})$. After some weeks, red prism crystals of 2 with $\sim 42 \%$ yield were obtained. Elemental Anal. Found: C, 36.45 ; H, $5.98 ; \mathrm{N}, 30.92 \%$. Calcd for $\mathrm{C}_{14} \mathrm{H}_{26} \mathrm{CuN}_{10} \mathrm{Ni}$ : C, 36.82; H, 5.74; N, 30.67\%. IR
(KBr): 3247 (m), 3180 (m), 2960 (w), 2123 ( s$), 2119$ ( s$), 1430$ (m), 1286 (m), 1090 (m), 1007 (s), $840(\mathrm{~m}), 612(\mathrm{~m}) \mathrm{cm}^{-1}$.
$\left[\mathrm{NiL}_{2}\right]\left[\mathrm{Ni}(\mathrm{CN})_{4}\right] \cdot 2 \mathrm{H}_{2} \mathrm{O}$ (3): Crystals of complex 3 with $\sim 28 \%$ yield were prepared following the similar synthetic method of complex 1; however, $\left[\mathrm{NiL}_{2}\right]\left(\mathrm{ClO}_{4}\right)_{2}(0.054 \mathrm{~g}$, 0.1 mmol ) was used in place of $\left[\mathrm{NiL}_{1}\right]\left(\mathrm{ClO}_{4}\right)_{2}$. Elemental Anal. Found: C, 39.92; H, 7.28; $\mathrm{N}, 25.52 \%$. Calcd. for $\mathrm{C}_{18} \mathrm{H}_{38} \mathrm{Ni}_{2} \mathrm{~N}_{10} \mathrm{O}_{2}$ : C, 39.75; H, 7.04; $\mathrm{N}, 25.75 \%$. IR (KBr): 3590 (m), 3405 (m), 3222 (m), 2870 (w), 2152 (s), 2131 (s), 1430 (w), 1284 (w), 1080 (m), 1019 (s), 908 (m), and $620(\mathrm{~m}) \mathrm{cm}^{-1}$.
$\left[\mathrm{CuL}_{2}\right]\left[\mathrm{Ni}(\mathrm{CN})_{4}\right] \cdot 2 \mathrm{H}_{2} \mathrm{O}(4)$ : Crystals of complex 4 with $\sim 35 \%$ yield were synthesized using a similar synthetic method of complex 1; however, $\left[\mathrm{CuL}_{2}\right]\left(\mathrm{ClO}_{4}\right)_{2}(0.055 \mathrm{~g}, 0.1 \mathrm{mmol})$ was used in place of $\left[\mathrm{NiL}_{1}\right]\left(\mathrm{ClO}_{4}\right)_{2}$. Elemental Anal. Found: C, 39.65; H, 6.65; N, 25.35\%. Calcd. for $\mathrm{C}_{18} \mathrm{H}_{38} \mathrm{CuNiN}_{10} \mathrm{O}_{2}$ : C, 39.39; H, 6.98; N, 25.52\%. IR (KBr): 3599 (m), 3372 (m), 3181 (m), 2925 (w), 2136 (s), 2129 (s), 1426 (w), 1285 (w), 1087 (m), 1012 (s), 842 (m), and 627 (m) cm ${ }^{-1}$.

Crystal Structure Determination. Single-crystal data for 1-4 were collected using a Bruker Smart Apex II diffractometer (Bruker, Germany) with Mo-K $\alpha$ radiation ( $\lambda=0.71073 \AA$ ). All empirical absorption corrections were applied using the SADABS program [30]. All structures were solved using direct methods, which yielded the positions of all nonhydrogen atoms. The positions were first refined isotropically, then anisotropically. All the hydrogen atoms of the ligands were placed in calculated positions with fixed isotropic thermal parameters and included in the structure factor calculations in the final stage of full-matrix least-squares refinement. All calculations were performed using the SHELXTL 5.1 software package [31]. For complexes 3 and 4, the hydrogen atoms bonded to oxygen were introduced at idealized positions and refined as riders with isotropic displacement parameters assigned 1.2 times the $U_{\text {eq }}$ value of the corresponding bonding partner. Selected bond lengths and angles are listed in Table 1. The crystallographic data of 1-4 are summarized in Table 2.

Table 2. Crystallographic data.

| Compound | $\left[\mathrm{NiL}_{1}\right]\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]$ | $\left[\mathrm{CuL}_{1}\right]\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]$ | $\left[\mathrm{NiL}_{2}\right]\left[\mathrm{Ni}(\mathrm{CN})_{4}\right] \cdot 2 \mathrm{H}_{2} \mathrm{O}$ | $\left[\mathrm{CuL}_{2}\right]\left[\mathrm{Ni}(\mathrm{CN})_{4}\right] \cdot 2 \mathrm{H}_{2} \mathrm{O}$ |
| :---: | :---: | :---: | :---: | :---: |
| Empirical formula | $\mathrm{C}_{14} \mathrm{H}_{26} \mathrm{Ni}_{2} \mathrm{~N}_{10}$ | $\mathrm{C}_{14} \mathrm{H}_{26} \mathrm{CuNiN}_{10}$ | $\mathrm{C}_{18} \mathrm{H}_{38} \mathrm{Ni}_{2} \mathrm{O}_{2} \mathrm{~N}_{10}$ | $\mathrm{C}_{18} \mathrm{H}_{38} \mathrm{CuNiN}_{10} \mathrm{O}_{2}$ |
| Formula weight | 451.86 | 456.70 | 544.00 | 548.83 |
| Temperature (K) | 296(2) | 296(2) | 296(2) | 296(2) |
| Crystal system | Monoclinic | Monoclinic | Triclinic | Triclinic |
| Space group | P2(1)/n | P2(1)/n | P-1 | P-1 |
| $a / \AA$ | 9.834(3) | 9.906(4) | 8.588(8) | 8.400(4) |
| $b / \AA$ | 9.167(3) | 9.110(4) | 8.873(8) | 8.771(4) |
| $c / \AA$ | 10.910(4) | 10.785(5) | 10.001(9) | 10.098(4) |
| $\alpha /{ }^{\circ}$ | 90 | 90 | 80.930(11) | 81.180(4) |
| $\beta 1{ }^{\circ}$ | 95.177(4) | 92.268(4) | 65.954(10) | 69.590(4) |
| $\gamma /^{\circ}$ | 90 | 90 | 67.544(10) | 67.991(4) |
| $V / \AA^{3}$ | 979.5(6) | 972.6(7) | 643.1(10) | 646.2(5) |
| Z | 4 | 2 | 2 | 1 |
| $D_{\text {c }} / \mathrm{Mg} \mathrm{cm}^{-3}$ | 1.532 | 1.559 | 1.405 | 1.410 |
| $\mu / \mathrm{mm}^{-1}$ | 1.943 | 2.082 | 1.498 | 1.585 |
| $F(000)$ | 472 | 474 | 288 | 289 |
| Crystal size (mm) | $0.36 \times 0.28 \times 0.23$ | $0.46 \times 0.36 \times 0.28$ | $0.28 \times 0.23 \times 0.15$ | $0.38 \times 0.26 \times 0.20$ |
| $\theta$ range | 2.67-27.46 | 2.74-27.47 | 2.23-27.44 | 2.15-27.53 |
| Collected/unique | 10687/2221(0.022) | 10594/2206(0.021) | 7227/2838(0.0295) | 6873/2872(0.0306) |
| Completeness to $\theta$ | 99.3\% | 99.1\% | 97.0\% | 96.3\% |
| Goodness-of-fit on $F^{2}$ | 1.041 | 1.012 | 0.979 | 0.991 |
| Final $R$ indices $[I>2 \sigma$ <br> (I)] | 0.0279, 0.0721 | 0.0268, 0.0716 | 0.0340, 0.0828 | 0.0356, 0.0893 |
| $R$ indices (all data) | 0.0411, 0.0799 | 0.0417, 0.0792 | 0.0554, 0.0910 | 0.0551, 0.0981 |
| Max. peak/hole (e. $\left.\AA^{-3}\right)$ | 0.290/-0.173 | 0.196/-0.178 | 0.460/-0.464 | 0.605/-0.379 |

## 4. Conclusions

In this work, four one-dimensional linear chains were successfully obtained between the reactions of macrocyclic nickel/copper complexes and $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$. All complexes exhibited one-dimensional linear chain structures, which were formed by bridging $[\mathrm{NiL}]^{2+} /[\mathrm{CuL}]^{2+}$ with $\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]^{2-}$ moieties. The magnetic susceptibilities revealed Curie-Weiss behavior for complexes 1-4 and the existence of weak antiferromagnetic exchange coupling.

Supplementary Materials: The following supporting information can be downloaded at: https:/ /www.mdpi.com/article/10.3390/molecules28114529/s1, Figure S1: The infrared spectra of complex 1; Figure S2: The infrared spectra of complex 2; Figure S3: The infrared spectra of complex 3; Figure S4: The infrared spectra of complex 4.
Author Contributions: Conceptualization, G.O.; methodology, G.O.; software, G.O.; validation, G.O.; formal analysis, Q.Z.; investigation, Q.Z.; resources, Q.W.; data curation, Q.Z.; writing-original draft preparation, Q.W. and Y.T.; writing-review and editing, G.O.; visualization, Y.T.; supervision, G.O.; project administration, G.O.; funding acquisition, G.O. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the Scientific Research Fund of Hunan Provincial Education Department, grant number 20A210, and the National Natural Science Foundation of China, grant number 51772091.
Institutional Review Board Statement: Not applicable.
Informed Consent Statement: Not applicable.
Data Availability Statement: Crystallographic data for 1-4 have been deposited with the Cambridge Crystallographic Data Center as supplemental publication numbers CCDC 2178844, 2178845, 2178848, and 2178849, respectively. Copies of the data can be obtained free of charge via http:/ / www.ccdc. cam.ac.uk (accessed on 30 August 2022).

Acknowledgments: We acknowledge the National Natural Science Foundation of China and the Scientific Research Fund of Hunan Provincial Education Department.

Conflicts of Interest: The authors declare no conflict of interest.
Sample Availability: Samples of the compounds are available from the authors.

## References

1. Schneemann, A.; Bon, V.; Schwedler, I.; Senkovska, I.; Kaskel, S.; Fischer, R.A. Flexible metal-organic frameworks. Chem. Soc. Rev. 2014, 43, 6062-6096. [CrossRef] [PubMed]
2. Miras, H.N.; vilà-Nadal, L.; Cronin, L. Polyoxometalate based open-frameworks (POM-OFs). Chem. Soc. Rev. 2014, 43, 5679-5699. [CrossRef] [PubMed]
3. Zeng, F.; Xiao, X.S.; Gong, S.F.; Yuan, L.; Tang, L.L. An electron-deficient supramolecular macrocyclic host for the selective separation of aromatics and cyclic aliphatics. Org. Chem. Front. 2022, 9, 4829-4833. [CrossRef]
4. Ding, M.H.; Tang, L.L.; Liao, J.; Ou, G.C.; Zeng, F. High-yield synthesis of a novel water-soluble macrocycle for selective recognition of naphthalene. Chin. Chem. Lett. 2021, 5, 1665-1668. [CrossRef]
5. Mudoi, P.P.; Sarma, B.; Choudhury, A.; Gogoi, N. Evidence of protonation induced intra-molecular metal-to-metal charge transfer in a highly symmetric cyanido bridged $\left\{\mathrm{Fe}_{2} \mathrm{Ni}_{2}\right\}$ molecular square. Dalton Trans. 2021, 50, 2057-2066. [CrossRef] [PubMed]
6. Azhar, A.; Young, C.; Kaneti, Y.V.; Yamauchi, Y.; Badjah, A.Y.; Naushad, M.; Habila, M.; Wabaidur, S.; Alothman, Z.A.; Kim, J. Cyano-Bridged Cu-Ni Coordination Polymer Nanoflakes and Their Thermal Conversion to Mixed Cu-Ni Oxides. Nanomaterials 2018, 8, 968. [CrossRef] [PubMed]
7. Kawabata, S.; Nakabayashi, K.; Imoto, K.; Klimke, S.; Renz, F.; Ohkoshi, S.I. Second harmonic generation on chiral cyanido-bridged $\mathrm{Fe}^{\mathrm{II}} \mathrm{Nb}^{\mathrm{IV}}$ spin-crossover complexes. Dalton Trans. 2021, 50, 8524-8532. [CrossRef]
8. Kawabata, S.; Nakabayashi, K.; Imoto, K.; Ohkoshi, S. Spin crossover phenomenon in a three-dimensional cyanido-bridged $\mathrm{Fe}^{\mathrm{II}} \mathrm{Mo}^{\mathrm{IV}}$ assembly. J. Appl. Phys. 2021, 129, 105501. [CrossRef]
9. Reczynski, M.; Pinkowicz, D.; Nakabayashi, K.; Nather, C.; Stanek, J.; Koziel, M.; Kalinowska-Tluscik, J.; Sieklucka, B.; Ohkoshi, S.I.; Nowicka, B. Room-temperature bistability in a Ni-Fe chain: Electron transfer controlled by temperature, pressure, light, and humidity. Angew. Chem. Int. Edit. 2021, 60, 2330-2338. [CrossRef]
10. Chorazy, S.; Zakrzewski, J.J.; Korzeniak, T.; Nowicka, B.; Pinkowicz, D.; Podgajny, R.; Sieklucka, B. Octacyanidometallates for multifunctional molecule-based materials. Chem. Soc. Rev. 2020, 49, 5945-6001. [CrossRef]
11. Jankowski, R.; Reczynski, M.; Chorazy, S.; Zychowicz, M.; Arczynski, M.; Koziel, M.; Ogorzaly, K.; Makowski, W.; Pinkowicz, D.; Sieklucka, B. Guest-dependent pressure-induced spin crossover in $\mathrm{Fe}_{4}{ }^{\mathrm{II}}\left[\mathrm{M}^{\mathrm{IV}}(\mathrm{CN})_{8}\right]_{2}(\mathrm{M}=\mathrm{Mo}, \mathrm{W})$ cluster-based material showing persistent solvent-driven structural transformations. Chem. Eur. J. 2020, 26, 11187-11198. [CrossRef] [PubMed]
12. Pai, T.Y.; Stefanczyk, O.; Kumar, K.; Mathoniere, C.; Sieklucka, B.; Ohkoshi, S.I. Experimental and theoretical insights into the photomagnetic effects in trinuclear and ionic $\mathrm{Cu}^{\mathrm{II}}-\mathrm{Mo}^{\mathrm{IV}}$ systems. Inorg. Chem. Front. 2022, 9, 771-783. [CrossRef]
13. Magott, M.; Pinkowicz, D. Chiral porous CN-bridged coordination polymer mimicking MOF-74 and showing magnetization photoswitching. Chem. Commun. 2021, 57, 9926-9929. [CrossRef] [PubMed]
14. Qi, X.H.; Guionneau, P.; Lafon, E.; Perot, S.; Kauffmann, B.; Mathoniere, C. New photomagnetic ionic salts based on $\left[\mathrm{Mo}(\mathrm{CN})_{8}\right]^{4-}$ and $\left[\mathrm{W}(\mathrm{CN})_{8}\right]^{4-}$ anions. Magnetochemistry 2021, 7, 97. [CrossRef]
15. Sellin, M.; Rupf, S.M.; Abram, U.; Malischewski, M. Eightfold electrophilic methylation of octacyanotungstate $\left[\mathrm{W}(\mathrm{CN})_{8}\right]^{4-/ 3-}$ : Preparation of homoleptic, eight-coordinate methyl isocyanide complexes $\left[\mathrm{W}(\mathrm{CNMe})_{8}\right]^{4+/ 5+}$. Inorg. Chem. 2021, 60, 5917-5924. [CrossRef]
16. Ali, M.; Ray, A.; Sheldrick, W.S.; Mayer-Figge, H.; Gao, S.; Sahmes, A.I. Synthesis, crystal structure, EPR and magnetic properties of a cyano-bridged CuII-NiII heterobimetallic complex: An unusual structure with long-range ferromagnetic exchange through hydrogen bonding. New J. Chem. 2004, 28, 412-417. [CrossRef]
17. Yeung, W.F.; Kwong, H.K.; Lau, T.C.; Gao, S.; Szeto, L.; Wong, W.T. Cyano-bridged molecular squares: Synthesis and structures of $[\mathrm{Ni}(\text { cyclen })]_{2}\left[\mathrm{Pt}(\mathrm{CN})_{4}\right]_{2} \cdot 6 \mathrm{H}_{2} \mathrm{O},[\mathrm{Ni}(\text { cyclen })]_{2}\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]_{2} \cdot 6 \mathrm{H}_{2} \mathrm{O}$ and $[\mathrm{Mn}(\text { cyclen })]_{2}\left[\mathrm{Ni}(\mathrm{CN})_{4}\right]_{2} \cdot 6 \mathrm{H}_{2} \mathrm{O}$. Polyhedron 2006, 25, 1256-1262. [CrossRef]
18. Jiang, L.; Lu, T.B.; Feng, X.L. Two supramolecular isomers of molecular squares and 1D helical chains with alternating right- and left-handed chirality. Inorg. Chem. 2005, 44, 7056-7062. [CrossRef]
19. Jiang, L.; Feng, X.L.; Su, C.Y.; Chen, X.M.; Lu, T.B. Interchain-solvent-induced chirality change of 1D helical chains: From achiral to chiral crystallization. Inorg. Chem. 2007, 46, 2637-2644. [CrossRef]
20. Curtis, N.F.; Flood, K.; Robinson, W.T. N-rac-(5,7,7,12,12,14-hexamethyl-1,4,8,11-tetraazacyclotetradeca-4,14(1)-diene)copper(II): Structures of the perchlorate salt and a trinuclear tetracyanonickelato-bridged compound. Polyhedron 2006, 25, 1579-1584. [CrossRef]
21. Černák, J.; Kuchár, J.; Stolárová, M.; Kajnaková, M.; Vavra, M.; Potocǹák, I.; Falvello, L.R.; Tomás, M. Preparation, spectroscopic and magnetic characterization of $\mathrm{Cu}(\mathrm{cyclam}) \mathrm{M}(\mathrm{CN})_{4}$ complexes exhibiting one-dimensional crystal structures (cyclam =1,4,8,11tetraazacyclotetradecane, $\mathrm{M}=\mathrm{Ni}, \mathrm{Pd}, \mathrm{Pt})$. Transit. Met. Chem. 2010, 35, 737-744. [CrossRef]
22. Kartal, Z.; Sahin, O.; Yavuz, A. Synthesis of Hofmann-type $\mathrm{Zn}\left(\mathrm{H}_{2} \mathrm{O}\right)_{2} \mathrm{Ni}(\mathrm{CN})_{4} \cdot \mathrm{nG}(\mathrm{G}=$ water and 1,4-dioxane) clathrates and the determination of their structural properties by various spectroscopic methods. Turk. J. Chem. 2019, 43, 1608-1621. [CrossRef]
23. Zhang, D.P.; Si, W.J.; Wang, P.; Chen, X.; Jiang, J.Z. 1D to 3D Heterobimetallic complexes tuned by cyanide precursors: Synthesis, crystal structures, and magnetic properties. Inorg. Chem. 2014, 53, 3494-3502. [CrossRef] [PubMed]
24. Ou, G.C.; Zhang, M.; Li, Z.Z.; Yuan, X.Y. Construction of one-dimensional homochiral helical supramolecular stereoismers using chiral macrocyclic nickel(II) complexes as building blocks. Inorg. Chim. Acta 2014, 411, 11-16. [CrossRef]
25. Pinkowicz, D.; Podgajny, R.; Nowicka, B.; Chorazy, S.; Reczynski, M.; Sieklucka, B. Magnetic clusters based on octacyanidometallates. Inorg. Chem. Front. 2015, 2, 10-27. [CrossRef]
26. Jiang, X.; Tao, B.L.; Yu, X.; Wang, Y.H.; Xia, H. Syntheses, crystal structures and properties of three cyano-bridged one-dimensional coordination polymers based on macrocyclic metallic tectons. RSC Adv. 2015, 5, 19034. [CrossRef]
27. Vafazadeh, R.; Dehghani-Firouzabadi, A.; Willis, A.C. Synthesis of hetero- and homo-multinuclear complexes with a tetracyanonickelate anion: Structural characterization $\left[\mathrm{Cu}(\mathrm{bcen}) \mathrm{Ni}(\mathrm{CN})_{4}\right]_{2}$. Acta Chim Slov. 2017, 64, 686-691. [CrossRef]
28. Azhar, A.; Zakaria, M.B.; Kim, J.; Na, J.; Kaneti, Y.V.; Fatehmulla, A.; Aldhafiri, A.M.; Farooq, W.A.; Bando, Y.; Yamauchi, Y. Single crystal growth of two-dimensional cyano-bridged coordination polymer of $\mathrm{Co}\left(\mathrm{H}_{2} \mathrm{O}\right)_{2} \mathrm{Ni}(\mathrm{CN})_{4} \cdot 4 \mathrm{H}_{2} \mathrm{O}$ using trisodium citrate dihydrate. Bull. Chem. Soc. Jpn. 2019, 92, 1263-1267. [CrossRef]
29. Suh, M.P.; Kang, S.G. Synthesis and properties of nickel(II) and copper(II) complexes of 14-membered hexaaza macrocycles, 1,8-dimethyl- and 1,8-diethyl-1,3,6,8,10,13-hexaazacyclotetradecane. Inorg. Chem. 1988, 27, 2544-2546. [CrossRef]
30. Sheldrick, G.M. SADABS, Program for Empirical Absorption Correction of Area Detector Data; University of Göttingen: Göttingen, Germany, 1996.
31. Sheldrick, G.M. Crystal structure refinement with SHELXL. Acta Cryst. A 2015, 71, 3-8. [CrossRef]

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and / or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.

