

## Synthesis and Characterization of Nickel(II) Tetraaza Macrocyclic Complex with 1,1-Cyclohexanediacetate Ligand

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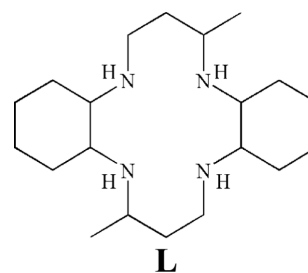
**ABSTRACT.** The reaction of  $[\text{Ni}(\text{L})\text{Cl}_2 \cdot 2\text{H}_2\text{O}]$  ( $\text{L} = 3,14\text{-dimethyl-}2,6,13,17\text{-tetraazatricyclo}[14,4,0^{1,18},0^{7,12}]\text{docosane}$ ) with 1,1-cyclohexanediacetic acid ( $\text{H}_2\text{cda}$ ) yields mononuclear nickel(II) complex,  $[\text{Ni}(\text{L})(\text{Hcda})_2]$  (**1**). This complex has been characterized by X-ray crystallography, electronic absorption, cyclic voltammetry and thermogravimetric analyzer. The crystal structure of **1** exhibits a distorted octahedral geometry with four nitrogen atoms of the macrocycle and two 1,1-cyclohexanediacetate ligands. It crystallizes in the triclinic system  $P\bar{1}$  with  $a = 11.3918(7)$ ,  $b = 12.6196(8)$ ,  $c = 12.8700(8)$  Å,  $V = 1579.9(2)$  Å<sup>3</sup>,  $Z = 2$ . Electronic spectrum of **1** also reveals a high-spin octahedral environment. Cyclic voltammetry of **1** undergoes one wave of a one-electron transfer corresponding to  $\text{Ni}^{\text{II}}/\text{Ni}^{\text{III}}$  process. TGA curve for **1** shows three-step weight loss. The electronic spectra, electrochemical and TGA behavior of the complex are significantly affected by the nature of the axial  $\text{Hcda}^-$  ligand.

**Key words:** Crystal structure, Nickel(II) complex, Octahedral complex, Tetraaza macrocycle, 1,1-cyclohexanediacetic acid

### INTRODUCTION

The transition metal(II) polyaza macrocyclic complexes with axial ligands have attracted considerable attention because of their structural and chemical properties, which are often quite different from those complexes with uncoordinated axial sites.<sup>1-16</sup> Recently, we reported the synthesis and crystal structure of  $[\text{Ni}(\text{L})(\text{ONO})_2] \cdot 2\text{H}_2\text{O}$ <sup>3</sup>, in which the nickel ion reveals a distorted octahedral geometry with four nitrogen atoms of the macrocycle and two axial nitrito groups. The compound  $[\text{Ni}(\text{Me}_8\text{cyclam})(\text{h}^2\text{-NO}_2)]\text{Cl} \cdot \text{H}_2\text{O}$  ( $\text{Me}_8\text{cyclam} = 3,5,7,7,10,12,14,14\text{-octamethyl-}1,4,8,11\text{-tetraazacyclotetradecane}$ )<sup>4</sup> shows that the coordination geometry of the nickel atom is six-coordinated bicapped square-pyramidal with two oxygen atoms of the bidentate nitrito group. In isocyanato nickel(II) complexes  $[\text{Ni}(\text{cyclam})(\text{NCO})(\text{H}_2\text{O})](\text{ClO}_4)$  and  $[\text{Ni}(\text{Me}_4\text{cyclam})(\text{NCO})](\text{ClO}_4)$  ( $\text{Me}_4\text{cyclam} = 1,4,8,11\text{-tetramethyl-}1,4,8,11\text{-tetraazacyclotetradecane}$ )<sup>5</sup> the coordination environment around the nickel(II) ion show a distorted octahedral and square pyramidal geometry, respectively. Furthermore, the aliphatic oxalate-bridged nickel(II) complexes  $[\text{Ni}(\text{L})(\text{oxalato})]_n \cdot n\text{H}_2\text{O}$ <sup>6</sup> and  $[\text{Ni}_2(\text{cyclam})_2(\text{oxalato})](\text{NO}_3)_2$ <sup>7</sup> exhibit one-dimensional polymeric and dinuclear structures with nickel centers and bridging oxalate ligand. It was thought that the

different molecular topologies in the complexes may be due to the stereochemical rigidity of the macrocycle and different coordination modes of the axial ligands. In order to better understand some aspects of different molecular topologies, we investigated the synthesis, properties and crystal structure of mononuclear nickel(II) complex  $[\text{Ni}(\text{L})(\text{Hcda})_2]$  (**1**) ( $\text{L} = 3,14\text{-dimethyl-}2,6,13,17\text{-tetraazatricyclo}[14,4,0^{1,18},0^{7,12}]\text{docosane}$ ;  $\text{H}_2\text{cda} = 1,1\text{-cyclohexanediacetic acid}$ ).



### EXPERIMENTAL

#### Materials and Physical Measurements

All chemicals used in the synthesis were of reagent grade and were used without further purification. The complex  $[\text{Ni}(\text{L})\text{Cl}_2 \cdot 2\text{H}_2\text{O}]$  was prepared according to literature method.<sup>17</sup> IR spectra were recorded as KBr pellets on a

Perkin-Elmer Paragon 1000 FT-IR spectrometer. The solution electronic and diffuse reflectance spectra were obtained on a Jasco V-550 spectrophotometer. Electrochemical measurements were accomplished with a three electrode potentiostat BAS-100BW system. A 3-mm Pt disk was used as the working electrode. The counter electrode was a coiled Pt wire and a Ag/AgCl electrode was used as a reference electrode. Cyclic voltammetric data were obtained in DMSO solution with 0.10 M tetraethylammonium perchlorate (TEAP) as supporting electrolyte at 20.0±0.1 °C. The solution was degassed with high purity N<sub>2</sub> prior to carrying out the electrochemical measurements. DSC and TGA were performed under flowing nitrogen at a heating rate of 10 min<sup>-1</sup> using an SDT 2960 Thermogravimetric Analyzer. Elemental analyses (C, H, N) were carried out on a Perkin-Elmer CHN-2400 analyzer.

### Synthesis of [Ni(L)(Hcda)<sub>2</sub>] (1)

1,1-cyclohexanediactic acid (200 mg, 1 mmol) was added to a water solution (20 mL) of [Ni(L)]Cl<sub>2</sub>·2H<sub>2</sub>O (251 mg, 0.5 mmol) and the mixture was refluxed for 1 h. The solution was filtered and allowed to stand for a few days to precipitate a quantity of purple crystals. The product was filtered and recrystallized from a hot H<sub>2</sub>O/CH<sub>3</sub>CN (1:1 v/v, 10 mL) mixture. Yield: 68%. Calc. (found) for C<sub>30</sub>H<sub>54</sub>N<sub>4</sub>NiO<sub>4</sub>: C, 60.72 (60.81); H, 9.17 (9.24); N, 9.44 (9.54)%. IR (KBr, cm<sup>-1</sup>): 3414(s), 3216(m), 2925(s), 2856(m), 1570(s), 1447(w), 1380(m), 1303(w), 1270(w), 1210(w), 1151(w), 1113(m), 996(w), 947(w), 894(w), 738(m), 678(w), 520(w).

### Crystallography

Single crystal X-ray diffraction measurement for **1** was carried out on a Bruker APEX II CCD diffractometer using graphite-monochromated Mo-Kα radiation (λ = 0.71073 Å).

**Table 1.** Crystallographic data

	1
Compound	[Ni(L)(Hcda) <sub>2</sub> ]
Color/shape	Purple/block
Chemical formula	C <sub>30</sub> H <sub>54</sub> N <sub>4</sub> NiO <sub>4</sub>
Formula weight	593.48
Temperature	296(2) K
Crystal system	Triclinic
Space group	<i>P</i> -1
Unit cell dimensions	
<i>a</i> (Å)	11.3918(7)
<i>b</i> (Å)	12.6196(8)
<i>c</i> (Å)	12.8700(8)
α(°)	69.572(4)
β(°)	83.516(4)
γ(°)	65.753(4)
Volume (Å <sup>3</sup> )	1579.9(2)
<i>Z</i>	2
Density (calculated, mg/m <sup>3</sup> )	1.248
Absorption coefficient (mm <sup>-1</sup> )	0.653
<i>F</i> (000)	644
Crystal size (mm)	0.14×0.08×0.06
θ range for data collection (°)	1.69 to 28.53
Index ranges	-15 ≤ <i>h</i> ≤ 15, -15 ≤ <i>k</i> ≤ 16, 0 ≤ <i>l</i> ≤ 17
Reflections collected/unique	7837/7837 (R <sub>int</sub> = 0.0000)
Absorption correction	SADABS
Max./min. transmission	0.9619 and 0.9142
Refinement method	Full-matrix least-squares on <i>F</i> <sup>2</sup>
Data/restraints/parameters	7837/0/356
Goodness of fit on <i>F</i> <sup>2</sup>	1.071
Final <i>R</i> indices [ <i>I</i> > 2σ( <i>I</i> )]	<i>R</i> <sub>1</sub> = 0.0737, <i>wR</i> <sub>2</sub> = 0.2323
<i>R</i> indices (all data)	<i>R</i> <sub>1</sub> = 0.1250, <i>wR</i> <sub>2</sub> = 0.2755
Weighting scheme	<i>w</i> = 1/[σ <sup>2</sup> ( <i>F</i> <sub>o</sub> <sup>2</sup> ) + (0.1639 <i>P</i> ) <sup>2</sup> + 0.2660 <i>P</i> ] with <i>P</i> = ( <i>F</i> <sub>o</sub> <sup>2</sup> + 2 <i>F</i> <sub>c</sub> <sup>2</sup> )/3
Largest difference peak and hole (eÅ <sup>-3</sup> )	1.951 and -0.510

Note. *R*<sub>1</sub> = ∥*F*<sub>o</sub> - |*F*<sub>c</sub>||/∥*S*||*F*<sub>o</sub>||. *wR*<sub>2</sub> = {*S*[*w*(*F*<sub>o</sub><sup>2</sup> - *F*<sub>c</sub><sup>2</sup>)<sup>2</sup>]/*S*[*w*(*F*<sub>o</sub><sup>2</sup>)<sup>2</sup>]}<sup>1/2</sup>.

Intensity data were measured at 100(2) K by  $\omega$ -2 $\theta$  technique. Accurate cell parameters and an orientation matrix were determined by the least-squares fit of 25 reflections. The intensity data were corrected for Lorentz and polarization effects. An empirical absorption correction was applied with the SADABS program.<sup>18</sup> The structure was solved by direct methods<sup>19</sup> and the least-squares refinement of the structure was performed by the SHELXL-97 program.<sup>20</sup> All atoms except all hydrogen atoms were refined anisotropically. The hydrogen atoms were placed in calculated positions allowing them to ride on their parent C atoms with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C or N})$ . The crystallographic data, conditions used for the intensity collection, and some features of the structure refinement are listed in Table 1. Crystallographic data for the structural analysis have been deposited with Cambridge Crystallographic Data Center, CCDC No. 1864424 for compound **1**. Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge, CB2, 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

## RESULTS AND DISCUSSION

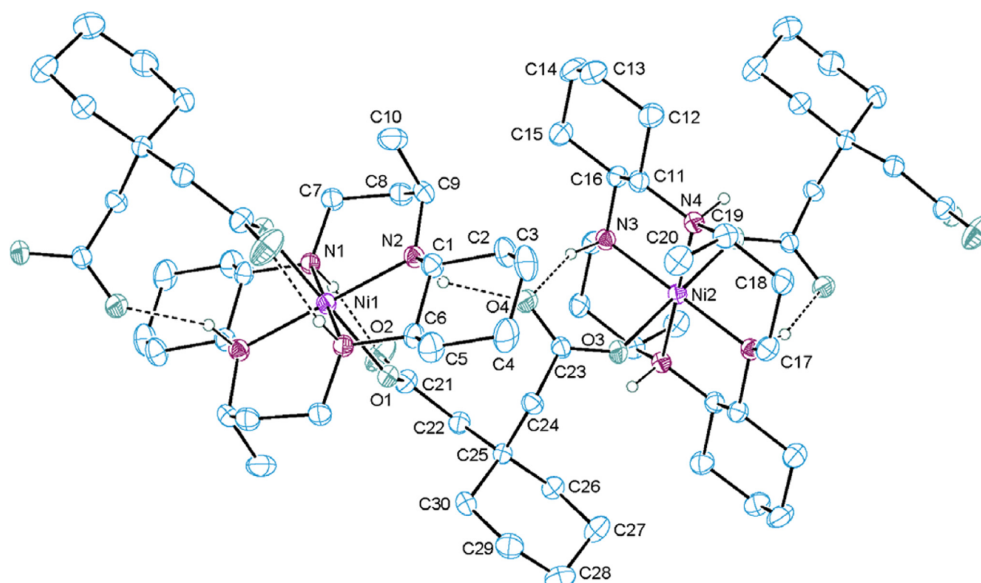
An ORTEP drawing<sup>21</sup> of  $[\text{Ni}(\text{L})(\text{Hcda})_2](\mathbf{1})$  with the atomic numbering scheme is shown in Fig. 1. Selected bond lengths and angles are listed in Table 2. The structure of **1** shows that the central nickel(II) ion is coordinated axially by two Hcda<sup>-</sup> ligands. The macrocyclic ligand skeleton in **1** takes the most stable *trans*-III (R,R,S,S) conformation

**Table 2.** Selected bond distances (Å) and angles (°) for  $[\text{Ni}(\text{L})(\text{cda})_2](\mathbf{1})$

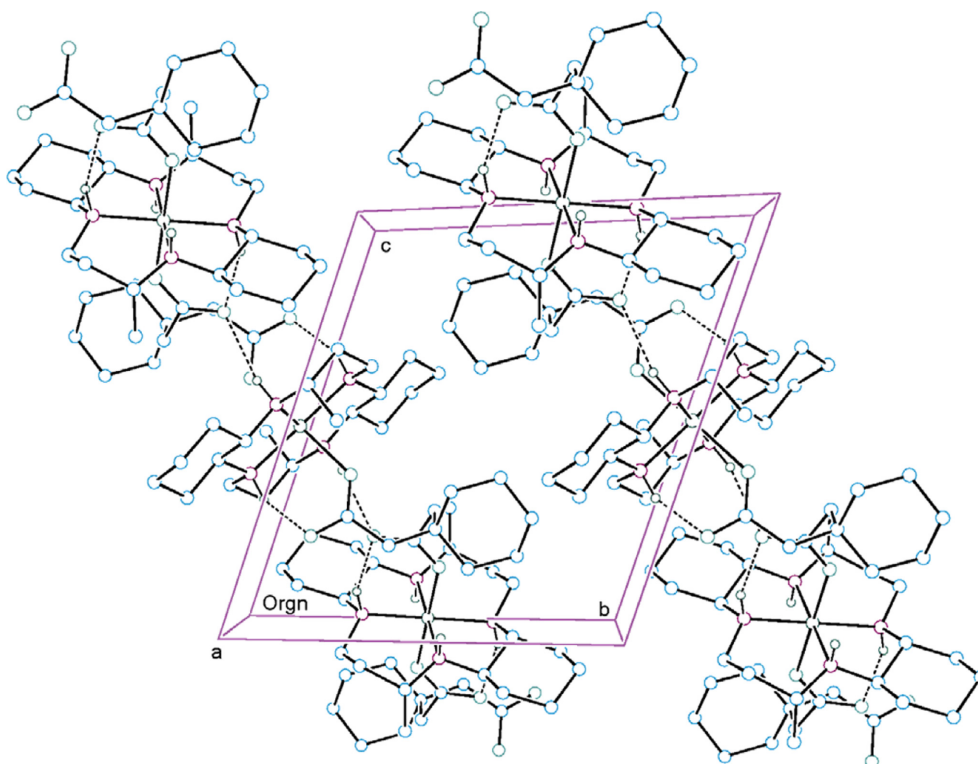
<i>Bond lengths</i>			
Ni(1)-N(1)	2.064(3)	Ni(2)-N(3)	2.054(4)
Ni(1)-N(2)	2.097(4)	Ni(2)-N(4)	2.096(3)
Ni(1)-O(1)	2.211(3)	Ni(2)-O(3)	2.150(3)
C(21)-O(1)	1.271(5)	C(23)-O(3)	1.264(5)
C(21)-O(2)	1.248(6)	C(23)-O(4)	1.257(5)
<i>Bond angles</i>			
N(1)-Ni(1)-N(2)	95.5(1)	N(3)-Ni(2)-N(4)	84.0(1)
N(1)-Ni(1)-N(2) <sup>i</sup>	84.5(1)	N(3) <sup>ii</sup> -Ni(2)-N(4)	96.0(1)
N(1)-Ni(1)-O(1)	91.2(1)	N(3)-Ni(2)-O(3)	92.0(1)
N(1) <sup>i</sup> -Ni(1)-O(1)	88.8(1)	N(3) <sup>ii</sup> -Ni(2)-O(3)	88.0(1)
N(2)-Ni(1)-O(1)	82.6(1)	N(4)-Ni(2)-O(3)	97.9(1)
N(2) <sup>i</sup> -Ni(1)-O(1)	97.4(1)	N(4) <sup>ii</sup> -Ni(2)-O(3)	82.1(1)
Ni(1)-O(1)-C(21)	130.8(3)	Ni(2)-O(3)-C(23)	133.4(3)
O(1)-C(21)-O(2)	123.4(4)	O(3)-C(23)-O(4)	123.4(4)

Symmetry codes: (i)  $-x+1, -y, -z+1$  (ii)  $-x, -y+1, -z$ .

as usual. Both independent molecules lie on a center of inversion. The coordination environment around the central nickel(II) ion reveals a distorted octahedron with four Ni-N and two Ni-O bonds. The nickel atom and the four nitrogen atoms of the macrocycle are exactly in a plane. The average Ni-N distance of 2.078(3) Å is significantly longer than in the square-planar geometry of  $[\text{Ni}(\text{L})\text{Cl}_2 \cdot 2\text{H}_2\text{O}]$  [1.948(4) Å],<sup>17</sup> but is similar to those observed for high-spin octahedral nickel (II) complexes with 14-membered tetraaza macrocyclic ligands.<sup>9-11</sup> The average Ni-O distance of 2.181(3) Å is longer than the NiN<sub>4</sub> plane (2.078(3) Å) to give an axially elongated octahedron. The N-Ni-N



**Figure 1.** An ORTEP view of  $[\text{Ni}(\text{L})(\text{Hcda})_2](\mathbf{1})$  with the atomic numbering scheme (30% probability ellipsoids shown).



**Figure 2.** A Parking diagram of  $[\text{Ni}(\text{L})(\text{Hcda})_2]$  (**1**). The hydrogen bonds are shown as dashed lines.

angles of the six-membered chelate rings are larger than those of the five-membered chelate rings. The dihedral angles between the plane of the carboxylate group and  $\text{NiN}_4$  plane involving Ni(1) and Ni(2) are  $82.7(1)$  and  $89.6(1)^\circ$ , respectively. The two Ni(1)-O(1) and Ni(2)-O(3) linkages are bent slightly off the perpendicular to the  $\text{NiN}_4$  plane by  $1.2\text{--}7.4^\circ$  and  $2.0\text{--}7.9^\circ$ , respectively. The two Ni-O-C angles related to the Hcda<sup>-</sup> ligands are Ni(1)-O(1)-C(21) [ $130.8(3)^\circ$ ] and Ni(2)-O(3)-C(23) [ $133.4(3)^\circ$ ]. Interestingly, the secondary amines of the macrocycle are hydrogen bonded to the carboxylate oxygen and nitrogen atoms of the Hcda<sup>-</sup> ligand [N(1)-H(1)⋯O(2) 2.785(5) Å,  $156.6^\circ$ ; N(5)-H(33)⋯O(4)<sup>v</sup> 2.856(6) Å,  $154.0^\circ$ ; N(2)-H(32)⋯N(8)<sup>vi</sup> 3.263(6) Å,  $167.7^\circ$ ; symmetry codes (v) x, y, z; (vi) -x+1, -y+1, -z+1]. It was also observed that the secondary amines of the macrocycle N(2) and N(3) form intermolecular hydrogen bonds with the uncoordinated carboxylate oxygen atom O(4) of the Hcda<sup>-</sup> ligand [N(2)-H(2)

⋯O(4) 3.127(5) Å,  $155.8^\circ$ ; N(3)-H(3)⋯O(4) 2.888(5) Å,  $152.8^\circ$ ]. This interaction gives rise to a 1D hydrogen-bonded infinite chain (Fig. 2 and Table 3).

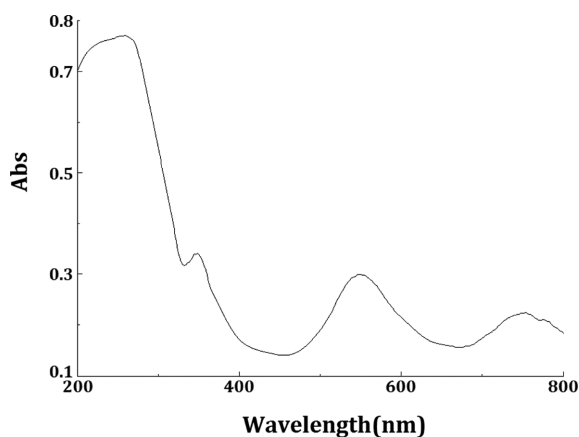
The IR spectra of **1** shows a band at ca.  $3216\text{ cm}^{-1}$ , which is assigned to the  $\nu(\text{N-H})$  of the coordinated secondary amines of the macrocycle. Two strong  $\nu(\text{COO})$  band at  $1570\text{ cm}^{-1}$  are associated with the coordinated Hcda<sup>-</sup> ligand, which is consistent with the crystal structure of **1**. The UV-Vis spectral data and solid state spectra data of **1** are shown in Table 4 and Fig. 3. The UV spectrum of **1** shows an absorption maximum at 262 nm attributed to a ligand-metal charge transfer associated with nitrogen and oxygen donors.<sup>22,23</sup> As shown in Fig. 3, the solid electronic spectrum of **1** in the visible region reveals three absorption bands at 348, 544, and 754 nm assignable to the  ${}^3\text{B}_{1g} \rightarrow {}^3\text{E}_g(\text{T}_{1g}(\text{P}))$ ,  ${}^1\text{B}_{2g} \rightarrow {}^1\text{B}_{1g}$ ,  ${}^3\text{B}_{1g} \rightarrow {}^3\text{B}_{2g}(\text{T}_{2g}(\text{F}))$  transitions, which are characteristic spectrum expected a high-spin  $d^8$  nickel(II) ion in  $D_{4h}$  environment.<sup>24,25</sup> However, the vis-

**Table 3.** Hydrogen bonding parameters (Å, °) for  $[\text{Ni}(\text{L})(\text{Hcda})_2]$  (**1**)

D-H⋯A	D-H (Å)	H⋯A (Å)	D⋯A (Å)	D-H⋯A (°)
N(1)-H(1)⋯O(2)	0.93	1.91	2.785(5)	156.6
N(2)-H(2)⋯O(4)	0.93	2.26	3.127(5)	155.8
N(3)-H(3)⋯O(4)	0.93	2.03	2.888(5)	152.8

**Table 4.** Electronic spectral data<sup>a</sup>

Complex	State	$\lambda_{\max}$ , nm ( $\epsilon = \text{M}^{-1} \text{cm}^{-1}$ )
[Ni(L)](ClO <sub>4</sub> ) <sub>2</sub> <sup>b</sup>	H <sub>2</sub> O	459(70)
	MeNO <sub>2</sub>	463(73)
[Ni(L)(Hcda <sup>-</sup> ) <sub>2</sub> ] ( <b>1</b> )	H <sub>2</sub> O	243(7.3×10 <sup>3</sup> ), 462(68)
	Solid	262, 348, 544, 754

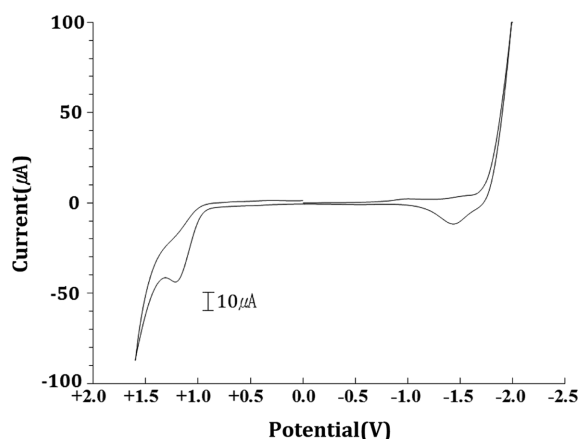
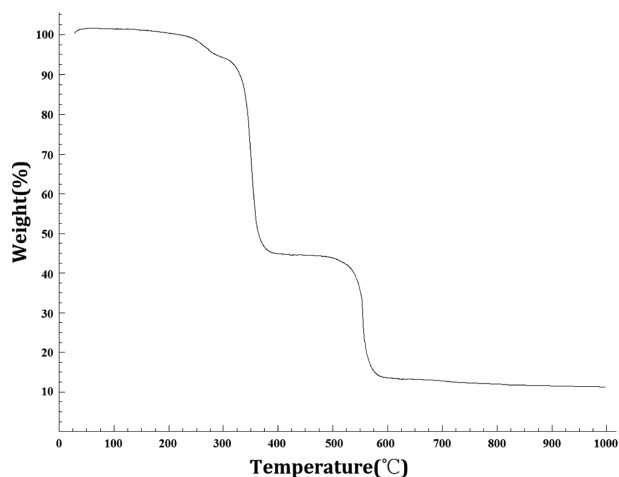
<sup>a</sup>Solution = H<sub>2</sub>O at 20±0.1 °C; solid = diffuse reflectance.<sup>b</sup>Ref. [26].**Figure 3.** Solid state electronic absorption spectra of [Ni(L)(Hcda<sup>-</sup>)<sub>2</sub>] (**1**) by the diffuse reflectance method at 20.0 ± 0.1 °C.

ible spectrum of **1** in water solution displays a broad band 462 nm, which has a low-spin d<sup>8</sup> nickel(II) ion in a square-planar environment of [Ni(L)](ClO<sub>4</sub>)<sub>2</sub> (463 nm).<sup>26</sup> This fact can be understood in terms of dissociation of the Hcda<sup>-</sup> ligand at water solution.

Cyclic voltammetric data for the nickel(II) complexes in 0.10 M TEAP-DMSO solution are listed in Table 5. Cyclic voltammogram of **1** is shown in Fig. 4. The complex **1** exhibits one one-electron wave corresponding to Ni<sup>II</sup>/Ni<sup>III</sup> process. The oxidation potential for **1** are considerably more positive than that for the square-planar [Ni(L)](ClO<sub>4</sub>)<sub>2</sub>,<sup>26</sup> indicating that this complex makes the oxidation of Ni(II) to Ni(III) easily. This fact may be attributed to the coordination of the axial Hcda<sup>-</sup> ligand, which is in agreement with the crystal structure of **1**. Similar results are also observed for tetraaza macrocyclic

**Table 5.** Cyclic voltammetric data<sup>a</sup>

Complex	Potentials (V) versus	
	Ni(II)/Ni(III)	Ag/AgCl Ni(II)/Ni(I)
[Ni(L)](ClO <sub>4</sub> ) <sub>2</sub> <sup>b</sup>	+0.73	-1.63
[Ni(L)(Hcda <sup>-</sup> ) <sub>2</sub> ] ( <b>1</b> )	+1.20(i) <sup>c</sup>	

<sup>a</sup>Measured in 0.10 M TEAP-DMSO solution at 20.0±0.1 °C.<sup>b</sup>Ref. [26]. These values are reduced from those of Ag/AgCl reference electrodes.<sup>c</sup>i = irreversible.**Figure 4.** Cyclic voltammogram of [Ni(L)(Hcda<sup>-</sup>)<sub>2</sub>] (**1**) in 0.1 M TEAP-DMSO solution at 20.0 ± 0.1 °C. The scan rate is 100 mV/s.**Figure 5.** Thermogravimetric curve of [Ni(L)(Hcda<sup>-</sup>)<sub>2</sub>] (**1**)

nickel(II) complexes containing axial groups.<sup>5,13</sup>

The TGA diagram of compound **1** is shown in Fig. 5. The compound was heated in the temperature range 30–1000 °C in nitrogen gas. TGA curve for **1** shows a first weight loss of 56.5% (calculated 56.7%) over ca. 242–431 °C, which is due to the loss of the macrocycle. A second weight loss corresponding to the 1,1-cyclohexanediacetate ligands (observed 31.2%, calculated 33.4%) is found in the temperature range 489–656 °C. A final residue (observed 11.2%, calculated 9.9%) was remained above 656 °C with NiO composition.

## CONCLUSION

The crystal structure of **1** has a six-coordinated octahedral geometry with bonds from the nickel(II) ion to the secondary amines of the macrocycle and to the two 1,1-

cyclohexanediacetate ligands. The intermolecular hydrogen bonds for **1** give rise to a 1D hydrogen-bonded infinite chain. The solid electronic spectra of **1** in the visible region exhibit three absorption bands. However, the visible spectrum of **1** in water solution displays a broad band 458 nm, which has a low-spin  $d^8$  nickel(II) ion in a square-planar environment. The cyclic voltammogram for **1** reveals one-electron wave. TGA curve for **1** shows three-step weight loss. The electronic spectrum, electrochemical and TGA behaviors of the complex **1** are significantly affected by the nature of the axial  $Hcda^-$  ligand.

**Supporting Information.** Additional supporting information is available in the online version of this article.

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