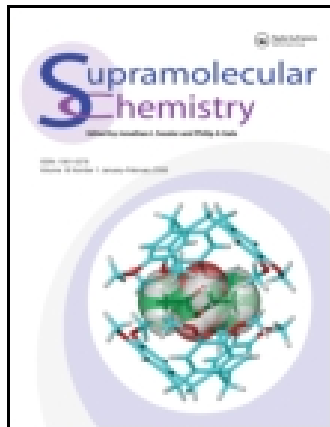


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Synthesis and Structural Elucidation of N,N'-Ditosyl-1,11-diaza-4,8,14,18-tetraselena-cycloicosane and its Copper and Platinum Complexes

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Synthesis and Structural Elucidation of *N,N'*-Ditosyl-1,11-diaza-4,8,14,18-tetraselena-cycloicosane and its Copper and Platinum Complexes

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The reaction of 1,2-diselenacyclopentane with *N,O,O*-tri-(toluene-*p*-sulphonate)-diethanolamine afforded a new seleno-azacrown ether, i.e. *N,N'*-ditosyl-1,11-diaza-4,8,14,18-tetraselena cycloicosane (**1**), in 19% yield, which was comprehensively characterized by elemental analysis, UV-Vis, ¹H NMR and mass spectroscopy. The reaction of **1** with copper(II) perchlorate (Cu(ClO₄)₂) and platinum(IV) tetrachloride (PtCl₄) gave its corresponding copper (**2**) and platinum complexes (**3**), respectively. The crystallographic investigations showed that the disparity of metal ion led not only to the distinct crystal system and space group, i.e. monoclinic system (C2/c) for **2** and triclinic system (P-1) for **3**, but also the different coordination modes of copper and platinum ions with **1**, i.e. normal coordination mode for **2** and ring-contracted coordination mode for **3**. Moreover, the metal ions in the crystals **2** and **3** were found in Cu(I) and Pt(II) forms, respectively, although Cu(II) and Pt(IV) were used at the initial stage of coordination reaction.

Keywords: Crown ether; Crystal structure; Synthesis; Transition metal complex

INTRODUCTION

The chemistry of seleno- and telluro-ethers has been drastically developed since 1970s [1–4], because these compounds usually show a great affinity for transition metal cations and heavy metal cations (for example Pd²⁺, Ag⁺, Hg²⁺, Ru²⁺, etc.) [5–11]. Among them, transition metal complexes involving a chalcogen atom such as sulfur or selenium are of particular importance as functional materials exhibiting peculiar electromagnetic properties [12,13] and active sites of a metal enzyme [14,15] participating in some vital functions. As two crucial properties

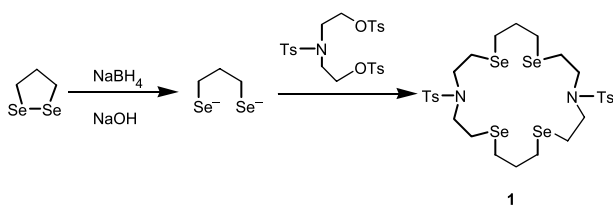
of these complexes, the coordination behavior and the redox chemistry between metal ions and sulfur-, selenium-, or tellurium-based ligands have attracted intense interests [16–31]. For example, Pinto *et al.* reported the redox behaviors of selenium coronands and their complexes with copper(I) and copper(II), and demonstrated the possible mechanism involving the electron transfer reaction between selenium coronands and copper ions [32–34]. Morley *et al.* reported that Pd²⁺ and Pt²⁺ exhibited different complexation behaviors with seleno-ethers. The reaction of cyclohepteno-1,4-diselenin with [PtCl₂(PhCN)₂] proceeded *via* an unprecedented oxidative addition of a C-Se bond to yield a platinum(IV) product, but its reaction with [PdCl₂(PhCN)₂] formed *trans*-[PdCl₂L₂] with diselenin bonded *via* one selenium only [35]. In the present work, we wish to report the synthesis and characterization of a new seleno-azacrown ether, i.e. *N,N'*-ditosyl-1,11-diaza-4,8,14,18-tetraselenacycloicosane (**1**), as well as the investigations on the crystallographic studies of its complexes with copper(II) and platinum(IV), which will serve our further understanding on the molecular design of chalcogen-containing crown ethers and their transition metal complexes.

EXPERIMENTAL SECTION

General

Melting points, measured with an XT-4 apparatus, were uncorrected. ¹H NMR spectra were recorded on a Varian Mercury VX300 instrument in CDCl₃

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SCHEME 1 The preparation processes of compound 1.

solution, using tetramethylsilane as an internal reference. UV-Vis spectra were recorded on a Shimadzu UV-2401/PC instrument. Elemental analyses were performed on a Perkin-Elmer 2400C instrument. Mass spectra were measured by using a JEOLJMS-DX-303 instrument. The molecular modeling study was performed with the CAChe 3.2 program (Oxford Molecular Co., 1999), and the energy of the structure is minimized using the PM3 force field.

Materials

Starting materials were commercially available unless noted otherwise. 1,2-Ddiselena-cyclopentane [36] and *N,O,O*-tri-(toluene-*p*-sulphonate)-diethanolamine [37] were prepared according to the literature procedures.

Synthesis of *N,N'*-Ditosyl-1,11-diaza-4,8,14,18-tetraselenacycloicosane (1)

1,2-Diselena-cyclopentane (0.2 g, 1 mmol) was dissolved in absolute ethanol (60 mL) containing sodium hydroxide (0.12 g, 3 mmol) and sodium borohydride (0.114 g, 3 mmol). After the ethanol

solution became colorless, *N,O,O*-tri-(toluene-*p*-sulphonate)-diethanolamine (1 mmol) in absolute ethanol (10 mL) was added to the solution under a nitrogen atmosphere, and the reaction was carried out under reflux for 6 h. After cooling, the solvent was removed *in vacuo*. The residue obtained was added to a mixture of water (100 mL) and chloroform (60 mL). The resultant binary mixture was stirred vigorously, and the chloroform layer was separated and dried over MgSO_4 . The residue obtained after the evaporation of chloroform was purified on a silica column chromatogram (eluted with dichloromethane), then was recrystallized from ethanol to give colorless crystals of **1** (110 mg, 19% yield): mp. 159–161°C; UV-Vis λ_{max} (CHCl_3)/nm ($\epsilon/\text{M}^{-1}\text{cm}^{-1}$): 243 (16400); MS m/z 850.0(M^+); ^1H NMR(CDCl_3): δ 2.00 (m, 4H), 2.04 (m, 4H), 2.43 (s, 6H), 2.65 (t, 8H), 2.77(t, 8H), 3.38(t, 8H), 7.32(d, 4H, $J = 8.1$ Hz), 7.70(d, 4H, $J = 8.1$ Hz); Anal. Calcd for $\text{C}_{28}\text{H}_{42}\text{S}_2\text{O}_4\text{Se}_4\text{N}_2$: C, 39.54; H, 4.98; N, 3.29. Found: C, 39.44; H, 5.01; N, 3.31.

1-Copper Complex (2)

A solution of $\text{Cu}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$ (14.8 mg, 0.04 mmol) in 10 mL of CH_3NO_2 was added to a solution of **1** (34 mg, 0.04 mmol) in 10 mL of CH_3NO_2 . The reaction mixture was stirred for 30 min at ambient temperature. The diffusion of diethyl ether vapor into the mixture solution yielded **2** as colorless crystals suitable for X-ray crystallography (yield 45%).

1-Platinum Complex (3)

A solution of PtCl_4 (13.5 mg, 0.04 mmol) in 10 mL of CH_3CN was added to a solution of **1** (34 mg,

TABLE I Crystallographic data and data collection parameters for 2 and 3.

Data	2	3
Empirical formula	$\text{C}_{30}\text{H}_{48}\text{Cl}\text{Cu}\text{N}_4\text{O}_{12}\text{S}_2\text{Se}_4$	$\text{C}_{14}\text{H}_{21}\text{Cl}_2\text{N}\text{O}_2\text{Pt}\text{S}\text{Se}_2$
Formula weight	1135.67	691.29
T (K)	293(2) K	293(2) K
Crystal system, space group	Monoclinic, $\text{C2}/c$	Triclinic, P-1
Unit cell dimensions	$a = 28.462(8) \text{ \AA}$ $b = 10.771(3) \text{ \AA}$ $c = 13.947(4) \text{ \AA}$	$a = 8.645(4) \text{ \AA}$ $b = 10.879(5) \text{ \AA}$ $c = 11.702(6) \text{ \AA}$
Unit cell dimensions	$\alpha = 90^\circ$ $\beta = 100.049(4)^\circ$ $\gamma = 90^\circ$	$\alpha = 68.161(7)^\circ$ $\beta = 84.950(7)^\circ$ $\gamma = 77.500(8)^\circ$
$V \text{ \AA}^3$	4210(2) \AA^3	1997.3(9) \AA^3
Z	4	2
D_c mg/m ³	1.792	2.302
$F(000)$	2264	648
Crystal size	0.22 × 0.18 × 0.10 mm	0.20 × 0.18 × 0.12 mm
Theta range for data collection	2.03 to 26.41°	2.22 to 26.38°
Data / restraints / parameters	4303 / 212 / 304	3983 / 0 / 209
Reflections collected / unique	11777 / 4303 [R(int) = 0.0350]	5670 / 3983 [R(int) = 0.0340]
Goodness-of-fit on F^2	1.055	1.012
R (wR_2) [$I > 2\sigma(I)$]	0.0332 (0.0755)	0.0437 (0.1204)
R (wR_2) (all data)	0.0610 (0.0864)	0.0542 (0.1278)
Largest diff. peak and hole e. \AA^{-3}	0.452, -0.482	3.528, -1.579

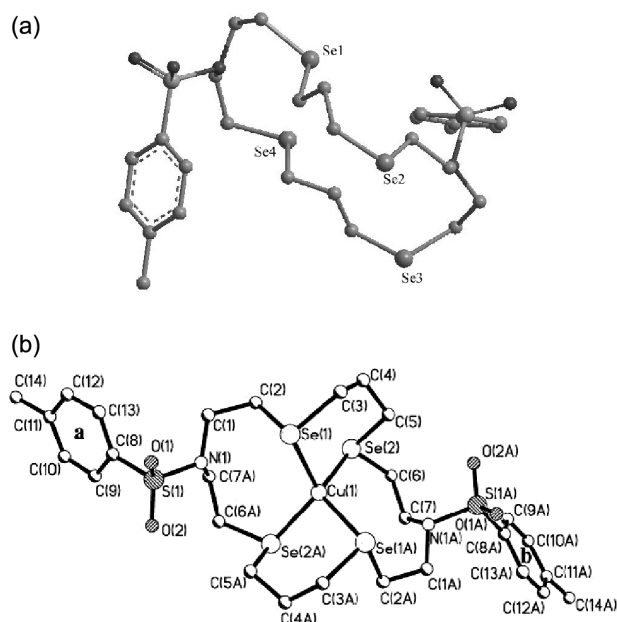


FIGURE 1 Molecular structures of (a) 1 and (b) 2.

0.04 mmol) in 10 mL of CH_3CN . The reaction mixture was stirred for 30 min at ambient temperature. The diffusion of diethyl ether vapor into the mixture solution yielded **3** as yellow crystals suitable for X-ray crystallography (yield 41%) Scheme 1.

X-RAY CRYSTALLOGRAPHIC STUDY

The X-ray intensity data of **2** and **3** were collected on a standard Siemens SMART CCD Area Detector System equipped with a normal-focus molybdenum-target X-ray tube ($\lambda = 0.71073 \text{ \AA}$) operated at 2.0 kW (50 kV, 40 mA) and a graphite monochromator. The structures were solved by the direct method and refined employing the full-matrix least squares on F^2 (Siemens, SHELXTL, version 5.04). The obtained crystallographic data for **2** and **3** were summarized in Table I. The crystallographic data (excluding structure factors) have been deposited to the Cambridge

Crystallographic Data Center with No. CCDC-247403 for **2** and CCDC-247404 for **3**.

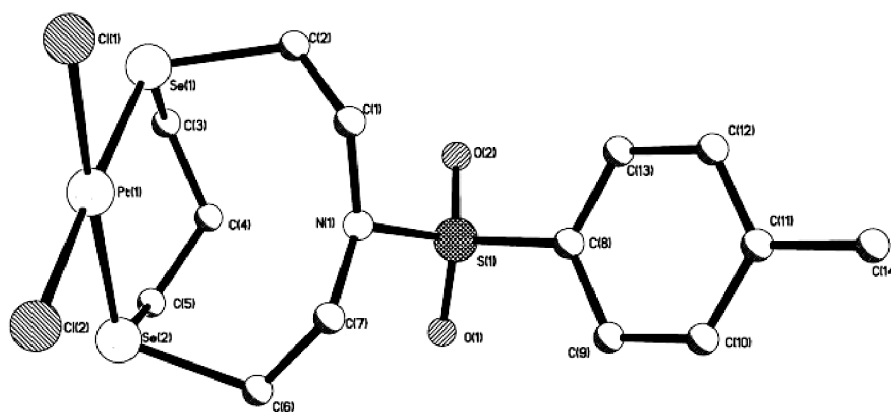
RESULTS AND DISCUSSION

Coordination Reaction of 1 with Copper(II)

It should be noted that, although we attempted to react copper(II) ion with seleno-azacrown ether **1** in various solvents, only Cu(I) complex was obtained. The X-ray crystallographic analysis showed that each asymmetric unit of **2** contained three components, i.e. a complex cation $[\mathbf{1}\text{-Cu}]^+$, a counteranion ClO_4^- and a solvent molecule CH_3NO_2 , and the latter two of them were disordered. The molecular structure of **2** was illustrated in Figure 1b, where the hydrogen atoms, perchlorate anion and solvent molecules were omitted for clarity. Moreover, a computed structure of **1** based on the PM3 calculation was also plotted in Figure 1a. Seen from Figure 1b, the copper was coordinated by four selenium atoms with cis angles in the range of $97.84\text{--}118.37^\circ$ and Cu–Se bond lengths $2.3775\text{--}2.3978 \text{ \AA}$ to complete a distorted-tetrahedral geometry. The complex conformation displayed a 2-fold rotation symmetry around an axis which intersected the midpoint of the Se(1) to Se(1A) vector and copper center. Moreover, the observed weighted-mean Cu–Se bond lengths, i.e. Cu–Se(1) $2.3978(7) \text{ \AA}$, Cu–Se(2) $2.3775(6) \text{ \AA}$, Cu–Se(1A) $2.3978(7) \text{ \AA}$, Cu–Se(2A) $2.3978(7) \text{ \AA}$, were shorter than the corresponding values of the reported complex $[\text{Cu}(\mathbf{16}\text{Se}_4)]\text{SO}_3\text{CF}_3$ (Cu–Se $2.42\text{--}2.52 \text{ \AA}$) [38], indicating a stronger Cu–Se coordination for the present Cu–Se complex. In addition, the obviously shortened Se–Se distances in **2** (Se1–Se1A = 3.924 \AA , Se2–Se2A = 4.027 \AA , Se1–Se2A = Se2–Se1A = 4.101 \AA , Se1–Se2 = Se1A–Se2A = 3.600 \AA) as compared with those in **1** (Se1–Se2 = 4.967 \AA , Se1–Se3 = 6.857 \AA , Se1–Se4 = 4.166 \AA , Se2–Se3 = 5.543 \AA , Se2–Se4 = 7.108 \AA , Se3–Se4 = 5.341 \AA) also verified the efficient Cu–Se combinations. The selected bond lengths and angles were given in Table II. No $\pi\text{--}\pi$ interactions between the adjacent complex units could be observed in the crystal structure of **2**. On the other hand, the copper in **2** was found to exist in Cu(I) form, although Cu(II) were used in the initial

TABLE II Selected bond lengths and angles of 2

Cu(1)–Se(2)	$2.3775(6) \text{ \AA}$	Se(1)–C(3)	$1.959(4) \text{ \AA}$
Cu(1)–Se(1)	$2.3978(7) \text{ \AA}$	Se(2)–C(6)	$1.943(4) \text{ \AA}$
Se(1)–C(2)	$1.945(4) \text{ \AA}$	Se(2)–C(5)	$1.947(4) \text{ \AA}$
Se(2)–Cu(1)–Se(2)#1	$115.73(4)^\circ$	C(2)–Se(1)–C(3)	$98.63(18)^\circ$
Se(2)–Cu(1)–Se(1)	$97.84(2)^\circ$	C(2)–Se(1)–Cu(1)	$101.97(14)^\circ$
Se(2)#1–Cu(1)–Se(1)	$118.37(2)^\circ$	C(3)–Se(1)–Cu(1)	$104.23(13)^\circ$
Se(2)–Cu(1)–Se(1)#1	$118.37(2)^\circ$	C(6)–Se(2)–C(5)	$101.21(17)^\circ$
Se(2)#1–Cu(1)–Se(1)#1	$97.84(2)^\circ$	C(6)–Se(2)–Cu(1)	$116.21(12)^\circ$
Se(1)–Cu(1)–Se(1)#1	$109.81(4)^\circ$	C(5)–Se(2)–Cu(1)	$97.77(13)^\circ$

FIGURE 2 Molecular structure of **3**.

stage of the coordination reaction. In order to explore the possible reason for this phenomenon, we also calculated the atom charge distribution of the Se atoms in **1** using a PM3 method, and the obtained result showed that the average charge intensity of Se atoms in **1** was -0.152 ($q\text{\AA}^{-1}$), which indicated that these selenium atoms could easily lose their electrons in the reaction process to form positive ions or radical positive ions. Therefore, we deduce that an electron transfer process from Se atoms to the coordinated copper center may be responsible to this redox reaction of copper with **1**.

Coordination Reaction of **1** with Platinum(IV)

By switching the central copper ion of complex **2** to a platinum ion, we obtained a ring-contracted complex **3**; that is, the seleno-azacrown ether unit was contracted from a twenty-membered ring in **1** to a ten-membered ring in **3**. The X-ray crystallographic structure of **3** was given in Figure 2, and the selected bond lengths and angles were listed in Table III. As can be seen in Figure 2, the platinum center was coordinated to two selenium atoms and two terminal chlorine atoms with *cis* angles in the range of $83.26\text{--}100.31^\circ$ to complete a square planar geometry, and no interactions between platinum and nitrogen could be observed.

In addition, the Se(1)–Se(2) distance (3.656 \AA) was found less than the sum of the van der Waal radii of two Se atoms (ca. 4 \AA), indicating the existence of transannular interactions between Se(1) and Se(2).

Figure 3 illustrates the packing structure of **3**, which displayed several types of interactions among the complex monomers. As can be seen in Figure 3a, the centroid distance between the benzene ring C8HB–C9HB–C10D–C11D–C12D–C13D and the benzene ring C8EC–C9EC–C10E–C11E–C12E–C13E was 3.919 \AA , and the dihedral angle was 0° , which gave an appreciable π – π interaction. Moreover, the distance between C14D and the benzene ring C8EA–C9EA–C10A–C11A–C12A–C13A was 3.599 \AA , which would result in a relatively strong C–H– π interaction. In addition, a number of intermolecular Pt–Pt, Se–Pt, and Se–Cl interactions (dotted lines) were also observed, which further stabilized the intermolecular linkage among the complex monomers. Attributed to a joint contribution of these interactions, the discrete monomers of **3** were self-assembled to form a layered aggregate in the solid state as shown in Figure 3b. Moreover, Pt(IV) ion was also changed to Pt(II) ion after coordinated with **1**, which may be attributed to a more complicated electron transfer mechanism than that for **2**. Researches

TABLE III Selected bond lengths and angles of **3**

Pt(1)–Cl(1)	2.348(2) Å	Se(1)–C(2)	1.973(11) Å
Pt(1)–Cl(2)	2.352(2) Å	Se(1)–C(3)	1.979(11) Å
Pt(1)–Se(1)	2.3783(12) Å	Se(2)–C(5)	1.968(10) Å
Pt(1)–Se(2)	2.3832(12) Å	Se(2)–C(6)	1.990(9) Å
Cl(1)–Pt(1)–Cl(2)	92.35(10)°	C(2)–Se(1)–C(3)	99.2(6)°
Cl(1)–Pt(1)–Se(1)	83.77(8)°	C(2)–Se(1)–Pt(1)	102.9(3)°
Cl(2)–Pt(1)–Se(1)	174.57(7)°	C(3)–Se(1)–Pt(1)	112.9(4)°
Cl(1)–Pt(1)–Se(2)	173.83(6)°	C(5)–Se(2)–C(6)	99.2(5)°
Cl(2)–Pt(1)–Se(2)	83.26(8)°	C(5)–Se(2)–Pt(1)	113.7(3)°
Se(1)–Pt(1)–Se(2)	100.31(5)°	C(6)–Se(2)–Pt(1)	102.4(3)°

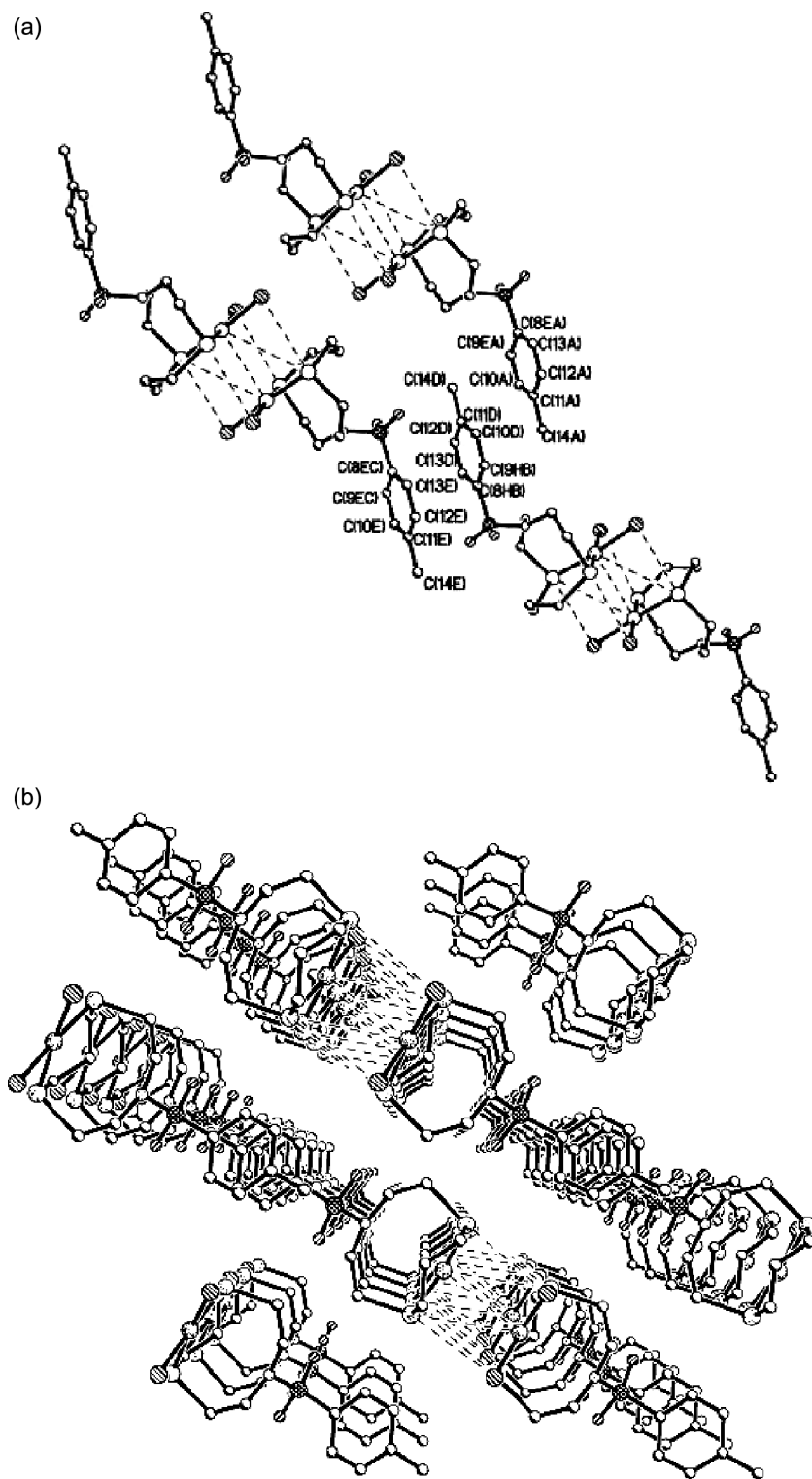


FIGURE 3 (a) Partial view and (b) top view of the assembly by 3.

about the detailed redox mechanism between **1** and these transition metal ions are still in progress.

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