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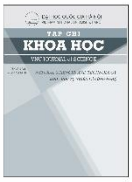

Synthesizing and Studying the Structure of Ni(II) Complexes Containing 2-alkyliminomethyl Pyrene Ligands

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Abstract: The 1(Ni) and Ni(II) complex was synthesized based on the reaction between N,O-bidentate ligand 2-((methylimino)methyl) pyrene-1-ol (1) and nickel acetate in a mixed solvent of toluene, ethanol and NaOAc. The characters of the complex, such as elemental analysis (EA), IR, NMR, MS spectroscopy and single crystal X-ray diffraction, were investigated. With respect to the nickel atom, 1(Ni) complex adopts a distorted square planar *trans*-coordination geometry. Concerning the ligand arrangement, 1(Ni) complex has a step configuration with an angle of 35.5° between the mean planes of the pyrene rings in the two independent molecules and step distance S = 1.93 Å, whereas the referent nickel(II) bis(salicylaldiminato) takes a much smaller step configuration with an angle of 17.6° and step distance S = 0.845 Å (Chart 1).

Keywords: Coordination chemistry, Nickel, Pyrene, expanded ligand, salicylaldimine

1. Introduction

Much attention has been paid to transition-metal-catalyzed polymerization catalysts bearing salicylaldiminato ligands since both electronic and steric parameters of the ligands can be systematically tunable by introducing various substituents on the aromatic ring [1-15]. Among them, nickel complexes bearing the salicylaldimine ligands were investigated as catalysts for polymerization of ethylene, methyl acrylate, acrylonitrile, and norbornene [2, 6-8, 16-23]. Recently, S. Mecking has developed many types of neutral nickel(II) salicylaldiminato complexes to generate branched oligoethylenes [24], ideal polyethylene nanocrystals [25], ethylene polymerization [26-28], copolymerization [29, 30].

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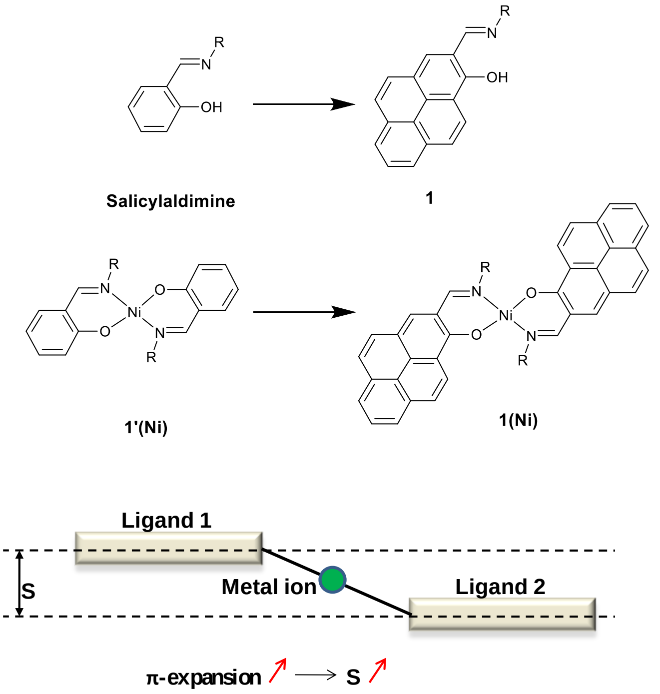


Chart 1.

Although, a lot of salicylaldiminato nickel(II) complexes with different structures have been synthesized and characterized, less attention has been focused on the π-expanded ligands and corresponding nickel(II) complexes. Moreover, recently, Saito group has reported several naphthalene-based salicylaldiminato-type nickel(II) complexes exhibiting cold crystallization as the first attempts at developing heat-storage materials [31, 32]. These have encouraged us to continue the development of salicylaldiminato-type metal complexes enhancing catalytic activities for various reactions and developing heat-storage materials as well as MOCVD precursors.

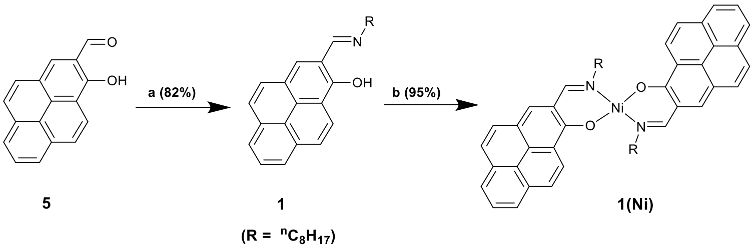
As part of our research program on the syntheses and characterizations of π-expansion-based salicylaldimine-type ligands and corresponding metal complexes [33, 34], in this part, we describe the synthesis, characterizations and crystal structure of new nickel(II) complex 1(Ni) with our reported ligand, 1-hydroxy-2-[(octylimino)methyl]-pyrene.

2. Results and discussion

Synthesis and MS Analysis

The syntheses of the ligand (**1**) and the corresponding nickel(II) complex (**1(Ni)**) are shown in Scheme 1. The reaction of $Ni(OAc)_2$ with the ligand **1** in a solvent mixture of toluene and ethanol was heated in the presence of a base, CH_3COONa, at room temperature for 1 hour under ambient atmosphere, and the complex **1(Ni)** was obtained by washing of the precipitate with methanol to remove acetate salts.

The addition of base is also crucial to prevent **1** from being decomposed in an acid environment that was created when adding metal cation into the solution as mentioned in above items [33, 34]. **1(Ni)** was obtained from the reaction mixture as a yellow solid with a high yield of ~95 %. This complex has black color that obtained from slow evaporation of its solution of chloroform or slow diffusion of a solvent system of chloroform and hexane.



^{a}(a) n-octylamine, CH_2Cl_2, r.t., 1 h; (b) $Ni(CH_3COO)_2.H_2O$, CH_3COONa, 1:5 PhMe:EtOH, r.t., 1 h.

Scheme 1. Syntheses of the pyrene-based ligand 1, its nickel complexes 1(Ni) ^{a}

These show that the complex has two polymorphs: black form and yellow form. It should be noted that the new complex **1(Ni)** is stable under ambient condition and/or toward usual manipulations such as silica-gel chromatography and recrystallization from hot solvents, *e.g.*, boiling ethyl acetate, under the air and room light. The reference complex **1'(Ni)** was prepared according to the literature reported for the similar complex having another alkyl group [35].

After being purified by filtration, the nickel(II) complex **1(Ni)** went through analysis by mass spectroscopy (MS). The parent peak was observed by HR-MS at m/z 771.34 $[M^+]$, while m/z 771.34 was calculated for $C_{50}H_{52}N_2O_2Ni$. The theoretical value and the experimental value are perfectly consistent. Besides, all compounds were also characterized by elemental analysis.

Diffraction study

The molecular structures of the complex **1(Ni)** (R = $^{n}C_8H_{17}$) was established by single crystal X-ray diffraction. Additionally, the reference complex **1'(Ni)** (R = $^{n}C_4H_9$) was also presented to compare their structural characterizations [36]. The structures of the two complexes are shown in Figure 1. Details of the crystallization procedures can be found in the experimental section, while full CIFs are accessible in the SI and the relevant reference.

The crystal structure of **1(Ni)** (R = $^{n}C_8H_{17}$) is in the P-1 space group, whereas the crystal structure of **1'(Ni)** (R = $^{n}C_4H_9$) is in the $P2_1/c$. These possibly result from the different electronic structures of pyrene core and the CH-π interactions between alkyl chain and pyrene ring. In general, a diamagnetic bis(salicylaldiminato) nickel(II) complex has a square planar geometry or tetrahedral geometry around nickel in solution [37-39]. In this research, these complexes **1(Ni)** and **1'(Ni)** have the coordination of a distorted *trans*-square planar geometry around nickel with no deflection from planarity. The four coordination sites are occupied by the two imines and the pyrenolate groups for **1(Ni)** and phenolate groups for **1'(Ni)**. For the complex **1(Ni)**, the Ni-N bonds were recorded at 1.9127(10) Å while the Ni-O distances are at 1.8529(7) Å. The bond distances of Ni-N are generally shorter than those observed for the complex **1'(Ni)** whereas the bond distances of Ni-O are generally longer than those observed for **1'(Ni)** (1.927 Å for the Ni-N bonds and 1.828 for the Ni-O bonds).

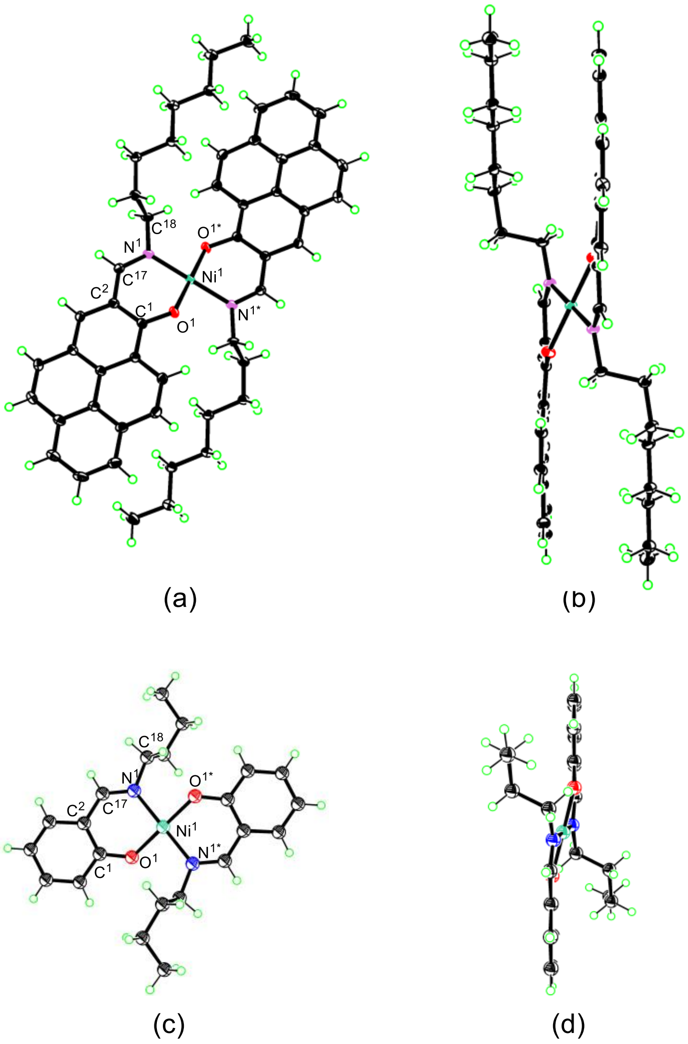


Figure 1. ORTEP view of the two complexes **1(Ni)** and **1’(Ni)** as obtained by single crystal X-ray diffraction: (a) **1(Ni)** top view, (b) **1(Ni)** side view, (c) **1’(Ni)** top view, and (d) **1’(Ni)** side view. Atomic displacement ellipsoids are draw at the 50% probability level. Element (color): nickel (green), carbon (black), nitrogen (blue), oxygen (red) and hydrogen (yellow green).

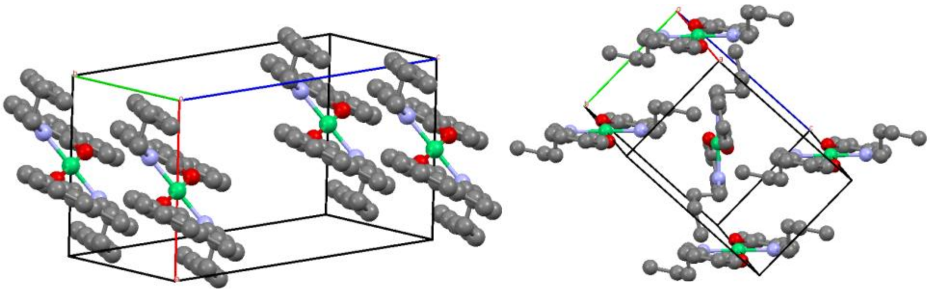
As shown in Figure 1, both the complexes **1(Ni)** and **1’(Ni)** are not co-planar, but are stepped as commonly seen in similar molecules, i.e. the two benzene rings are parallel, but their planes are separated by 0.845 Å. In **1(Ni)**, the two pyrene rings are also parallel and their planes are separated by 1.930 Å, approximately 2.3 times as much as that in **1’(Ni)**. Therefore, the dihedral angle between pyrene ring and the plane of N1-O1-O1*-N1* was measured at 35.5^{o}, about 2 times as much as that in **1’(Ni)** (17.6^{o}). Another notable point is that the plane of seven carbon atoms of the long alkyl chains of **1(Ni)** is nearly parallel to pyrene ring (5.8^{o}) whereas four carbon atoms of the long alkyl chains of **1’(Ni)** arranges randomly. This difference can be caused by the fact that pyrene has a big size and much more π-electron that created the interaction CH-π. Table 2 displays some selected geometric parameters for the available experimental data from X-ray diffraction analysis.

Table 1. Crystal data and structure refinement details for **1(Ni)** and **1’(Ni)**

	1(Ni) (R = $^{n}C_8H_{17}$)	**1’(Ni)** (R = $^{n}C_4H_9$)
Mol. formula	$C_{50}H_{52}NiN_2O_2$	$C_{22}H_{28}NiN_2O_2$
Mol. Weight	771.67	411.17
Crystal habit	Black, block	Dark blue, block
Crystal dimens./mm	0.31 x 0.11 x 0.00	0.18 x 0.22 x 0.28
Crystal system	Triclinic	Monoclinic
Space group	P-1	$P2_1/c$
a (Å)	8.0138(16)	10.8573(13)
b (Å)	9.7210(18)	7.2521(8)
c (Å)	12.246(3)	14.7043(13)
α (deg)	98.379(3)	90
β (deg)	98.341(2)	119.352(6)
γ (deg)	91.845(3)	90
V (Å^3)	932.4(3)	1009.16
Z	1	2
μ(Mo Kα) (cm^{-1})	5.666	9.800
T/K	123(1)	298
$2\theta_{max}$ (deg)	55.0	53.0
Radiation MoKa (l = 0.71075 Å)	MoKα	MoKα
R_{int}	0.0299	0.0332

The molecular packing diagrams for both complexes **1(Ni)** and **1’(Ni)** are displayed in Figure 2. These complexes exhibit different packings. The packing of the complex **1(Ni)** were configured in a ladder-like network and the molecules formed parallel strands, a “single ladder” type network, Figure 2a. The packing of the complexes **1’(Ni)** arranged into two series of ladders that penetrate each other, a “cross-ladder” type network, Figure 2b [40].

Some hydrogen atoms in alkyl chains of **1(Ni)** have close contacts with pyrene rings: implying intra- and inter-molecular CH-π interactions. These crystal structures suggest that the spatial extension of π-system was suitable for unidirectional alignment of the ladders, and there was a space for the alkyl chains to fluctuate to some extents between the running ladders.



(a)

(b)

Figure 2. Crystal packings of the nickel complexes (a) **1(Ni)** (R = $^{n}C_8H_{17}$); (b) **1’(Ni)** (R = $^{n}C_4H_9$); (c) . Hydrogen atoms are omitted for clarity.

Table 2. Comparison of selected geometric parameters coming from X-ray diffraction analysis

1(Ni) (R = $^{n}C_8H_{17}$)		**1'(Ni)** (R = $^{n}C_4H_9$)	
N1-Ni1	1.9127(10)	N1-Ni1	1.927(1)
N1^{i}-Ni1	1.9127(10)	N1^{i}-Ni1	1.927(1)
O1-Ni1	1.8529(7)	O1-Ni1	1.828(1)
O1^{i}-Ni1	1.8529(7)	O1^{i}-Ni1	1.828(1)
O1-C1	1.3122(14)	O1-C1	1.313(3)
N1-C18	1.4842(14)	N1-C18	1.482(2)
N1-C17	1.2926(15)	N1-C17	1.293(3)
O1^{i} -Ni1-N1^{i}	91.03(4)	O1^{i} -Ni1-N1^{i}	92.69
O1-Ni1-N1	91.03(4)	O1-Ni1-N1	92.69
O1^{i}-Ni1-N1	88.97(4)	O1^{i}-Ni1-N1	87.31
O1-Ni1-N1^{i}	88.97(4)	N1-Ni1-N1^{i}	87.31

3. Conclusions

A salicylaldiminato-type nickel(II) complex of pyrene was designed and synthesized by a six-step synthesis. The coordination of the ligand **1** to Ni(II) metal centre gave stable neutral square-planar complex **1(Ni)**, which was characterized by elemental analysis, IR spectroscopy, X-ray diffraction analysis. The complex **1(Ni)** is not co-planar, but is stepped as is commonly observed in similar complex **1'(Ni)**. However, the arrangement of carbon atoms of long alkyl chains is different between **1(Ni)** and **1'(Ni)**. The packing of **1(Ni)** exhibits a ladder-like network and the molecules formed parallel strands of the crystal structure that exists in mononuclear complex with the long distances of Ni···Ni separations. The authors are currently investigating deeply on the supercooling process of the polymorphs **1(Ni)** and applying the complex **1(Ni)** for catalysts, precursor of MOCVD and energy storage materials.

4. Experimental

General Procedures. General experimental details have already been reported in previous items [33, 34].

Synthesis.

Bis[2-[(octylimino)methyl]-1-pyrenolato-N,O] nickel(II) 1(Ni). A mixture of **1** (20.0 mg, 56 μmol, 2.0 eq.), anhydrous sodium acetate (17.2 mg, 210 μmol, 7.5 eq., Junsei Chemical Co.,Ltd.), nickel acetate tetrahydrate (10.5 mg, 42 μmol, 1.5 eq., Wako Pure Chemical Industries, Ltd.), 1 mL of PhMe, and 3 mL of EtOH was stirred at room temperature for 5 min. Then, the reaction suspension was added 2 mL of ethanol and stirred at room temperature for 55 min. The formed precipitate was collected and washed with MeOH to give the product **1(Ni)** as a brown solid, 20.4 mg (95%). M.p. 210 ºC; ^{1}H NMR (500 MHz, $CDCl_3$, TMS): δ = 13.17 (s, 2H), 8.32 (d, J = 9.08 Hz, 2H), 7.96 (s, 2H), 7.92-7.84 (m, 4H), 7.81-7.74 (m, 6H), 7.42 (d, J = 9.08 Hz, 2H), 5.71 (m, 4H), 2.30 (m, 4H) 1.67 (m, 4H), 1.44-1.00 (m, 16H), 0.77 (t, J = 6.60 Hz 6H); IR (KBr, cm^{-1}) 3035(w), 2954(m), 2925(m), 2855(m), 1614 (s, $\nu_{C=N}$), 1545(m), 1473(m), 1458(m), 1432(m), 1421(m), 1412(m), 1375(m), 1224(m), 1193(m), 1037(w), 959(w), 841(m), 836(m), 754(m), 740(m), 723(m), 683(m), 598(w), 502(w), 419(w); MS (APCI): m/z (%): 771.34 (100) ($[M+H]^+$); elemental analysis calcd (%) for $C_{50}H_{52}NiN_2O_2$: C, 77.82; H, 6.79; N, 3.63. Found: C, 77.59; H, 7.07; N, 3.49; A sample for diffraction study was prepared by slow diffusion of a solvent system of chloroform and hexane.

Reference complex 1'(Ni): Bis[2-[(octylimino)methyl]phenolato-N,O] Nickel(II) 1'(Ni). *The complex has been prepared as is described in ref. 35.* A cold concentrated solution of the nickel(II) acetate tetrahydrate (0.5 mmol) in 2 mL of water was treated with the salicylaldehyde (1 mmol) in methanol (2 mL). The resulting suspension was stirred at room temperature for 1 h, and filtered. The solid bis(salicylaldehydato) nickel(II) was then stirredat room temperature in methanol (1 mL) with an excess of n-octylamine (1.26 mmol) in 1 mL of methanol for 1 h. The formed precipitate was filtered, washed with methanol and dried under reduced pressure to obtain the product **1’(Ni)** (93 mg, 36%) as a green powder. m.p. 102 ºC; ^{1}H NMR (500 MHz, $CDCl_3$, TMS): δ = 9.21 (2H, s), 7.17 (m, 2H), 7.09 (m, 2H), 6.50 (d, J= 8.11 Hz, 2H), 6.40 (t, J = 7.31 Hz, 2H), 3.86 (t, J = 6.84 Hz, 4H) 1.89 (m, 4H), 1.46-1.21 (m, 20H), 0.87 (t, J = 6.84 Hz, 6H); IR (KBr): 3052(w), 3029(w), 2921(s), 1615(s, $\nu_{C=N}$), 1543(s), 1471(s), 1452(s), 1398(m), 1353(s), 1333(s), 1226(m), 1206(m), 1148(s), 1126(m), 1013(w), 965(w), 914(m), 849(w), 823(w), 753(s), 738(s), 722(w), 600(w), and 462(m) cm^{-1}; MS (APCI): m/z (%): 523.30 ([M+H]$^+$); elemental analysis calcd (%) for $C_{30}H_{44}N_2O_2Ni$: C, 68.85; H, 8.47; N, 5.35. Found: C, 68.57; H, 8.46; N, 5.27.

Appendix A. Supplementary material

CCDC 1872192 contains the supplementary crystallographic data for 2018/10/09. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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Tổng Hợp và Nghiên Cứu Cấu Trúc Phức Chất Ni(II) Chứa Phối Tử 2-alkyliminomethyl Pyren

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Tóm tắt: Phức Ni(II) (1(Ni)) được tổng hợp dựa trên phản ứng giữa phối tử hai càng 2 ((methylimino)methyl)pyren-1-ol (1) chứa N và O với niken axêtát ở trong hỗn hợp dung môi của toluene, rượu etylic và NaOAc. Các đặc trưng của phức này, như phân tích nguyên tố (EA), phổ hồng ngoại (IR), phổ cộng hưởng từ hạt nhân (NMR), phổ khối lượng (MS) và nhiễu xạ tia X đơn tinh thể (XRD) được khảo sát. Đối với nguyên tử niken, phức 1(Ni) có hình học phối trí loại trans- vuông phẳng biến dạng. Về mặt sắp xếp phối tử, phức 1(Ni) có cấu hình bậc thang với góc giữa các mặt phẳng của vòng pyren trong hai phân tử độc lập là 35,5^{o} và khoảng cách bậc S = 1.93 Å. Trong khi, niken(II) bis(salicylaldiminato), phức niken đối chiếu, có cấu hình bậc thang nhỏ hơn nhiều với giá trị góc là 17,6^{o} và khoảng cách bậc S = 0.845 Å (biểu đồ 1).

Từ khóa: Hóa học Phức Chất, Niken, Pyren, Cấu Tử tạo Phức có Giàu Điện tử, Salicylaldimin.