两个三维柔性功能化双苯并咪唑金属 N-杂环卡宾的合成和结构

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摘要:由 1,1'-双苄基-3,3'-氧双(亚乙基-2,1)-二苯并咪唑六氟磷酸盐(L1)和 1,1'-双(吡啶-2-甲基)-3,3'-氧双(亚乙基-2,1)-二苯并咪唑六氟磷酸盐(L2)为配体合成了[Hg($C_{32}H_{28}N_4O$)](CH_3COO)(PF_6)₂ (1)和[NiCl($C_{30}H_{28}N_6O$)](PF_6)₂ (2),并对其进行了结构表征。配合物 1 是一个罕见的三价态 Hg 配合物,为单斜晶系 $P2_1/c$ 空间群,配合物 2 为单斜晶系 $P2_1/n$ 的配合物。1 和 2 中存在 $O-H\cdots F$, $C-H\cdots F$, $C-H\cdots C1$, $C-H\cdots \pi$, $P-F\cdots \pi$ 氢键作用和 $\pi-\pi$ 堆积作用,并以此分别形成了 3D 超分子结构。

关键词:柔性双苯并咪唑盐;金属 N-杂环卡宾;3D 超分子结构

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Syntheses and Structures of Two 3D Metal NHCs Based on Flexible Functionalized Bibenzimidazole

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Abstract: [Hg($C_{32}H_{28}N_4O$)](CH₃COO)(PF₆)₂ (1) and [NiCl($C_{30}H_{28}N_6O$)]PF₆ (2) have been synthesized based on 1,1'-bisbenzyl-3,3'-oxybis(ethane-2,1-diyl)-dibenzimidazolium-dihexafluorophosphate ligand (L1) and 1,1'-bis (pyridine -2-ylmethyl)-3,3'-oxybis (ethane-2,1-diyl)-dibenzimidazolium-dihexafluorophosphate ligand (L2) and structurally characterized. The complex 1 is a rare three valence state mercury NHC and crystallizes in the $P2_1/c$ space group with monoclinic crystal system. The complex 2 crystallizes in the $P2_1/n$ space group with monoclinic crystal system. There are $O-H\cdots F$, $C-H\cdots F$, $C-H\cdots Cl$, $C-H\cdots \pi$, $P-F\cdots \pi$ hydrogen bonds interactions and $\pi\cdots\pi$ stacking interactions contributing to 3D supramolecular structure of 1 and 2. CCDC: 1038410, 1; 1038411, 2.

Key words: flexible dibenzimidazolium; metal NHC; 3D supramolecular structure

0 Introuduction

Over the past few years, the popularity of metal-NHC complexes has developed exponentially for their very broad applications^[1-4], particularly in the area of sensitizer^[5], biological science^[6] and catalysis^[7-8]. Up to now, there are many reports witnessing the development of metal-NHC complexes from mononuclear to

polynuclear^[9-10], most of these complexes constructed by using azole-based NHCs as auxiliary ligand associating with transition metal^[11-13]. Obviously, functional imidazolium-based ligands as a kind of potential good candidates of bridging ligand^[14-15] have enjoyed great popularity by the scientists in forming novel metal-NHCs, that's just because their own unique structures which can contain unprecedented multidentate *N*-

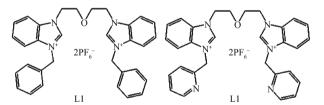
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donations and large π -conjugated system. Meanwhile, the transition metals offer unoccupied orbital as acceptors receiving electron which from carbenedonors, thus, which offering the possibilities of coordination between the metals and NHC-ligands. Furthermore, introducing functional groups^[16] to the imidazole-NHCs becoming multi-NHCs^[17] can not only stabilizes the coordinate system but also affords more chances for obtaining novel structure of metal-NHCs complexes.

In our work, considering the above many factors we have deliberately chosen two dibenzimidazolium NHC ligands modified by diethyl ether chain and functionalized groups, namely, 1,1'-bisbenzyl-3,3'-oxybis (ethane-2,1-diyl)-dibenzimidazolium ligand (L1) and 1,1'-bis (pyridine-2-ylmethyl)-3,3'-oxy-bis(ethane-2,1divl)-dibenzimidazolium-dihexafluorophosphate ligand (L2) (Scheme1), though during the recent year, many researches based on metal and benzimidazole-NHC have made significant achievements [18], particularly, silver who is the leading actor in most of the previous work[19-21], however, the NHC-complexes of trivalent mercury are rare. Herein, we present mercury (III) complex $([Hg(C_{32}H_{28}N_4O)](CH_3COO)(PF_6)_2$ (1)) and nickel(II) complex ($[NiCl(C_{30}H_{28}N_6O)]PF_6$ (2)) as well as their structures.



Scheme 1 Ligands L1 and L2

1 Experimental

1.1 Reagents and physical measurements

All manipulations were performed using Schlenk techniques, and solvents were purified by standard procedures. All the reagents for syntheses and analyses were of analytical grade and without further purification. L1 and L2 were prepared in a manner analogous to the method in the literature^[22-23].

1.2 Synthesis of the complex 1 and 2

For the synthesis of 1, $Hg(Ac)_2$ (0.669 g, 2.10

mmol) was added to the solution of L1 (0.801 g, 1.03 mmol) in CH₃CN (10 mL), and the mixture was heated under argon at 80 °C for 36 h. The resulting solution was filtered and H₂O (100 mL) was added to precipitate a powder. Recrystallization from CH₃CN and Et₂O produced a pale yellow powder. Yield: 75%. Anal. Calcd. for C₃₄H₃₄F₁₂HgN₄O₃P₂ (%): C, 39.37; H, 3.30; N, 5.40. Found(%): C, 39.23; H, 3.18; N, 5.47.

For the synthesis of **2**, similarly, NiCl₂ (0.259 2 g, 2.0 mmol) was added in the solution of L2 (0.788 g, 1.01 mmol) in DMSO, and the mixture was heated under argon at 80 °C for 36 h. Then the resulting mixture was filtered, and a write powder obtained after recrystallization from CH₃CN and Et₂O. Yield, 65%. Anal. Calcd. for C₃₀H₂₈ClF₆N₆NiOP(%): C, 49.52; H, 3.88; N, 11.55. Found (%): C, 49.50; H, 3.76; N, 11.50.

1.3 Crystal structure determination

The crystal date of $\bf 1$ and $\bf 2$ were collected by a Bruker SMART APEX-II CCD diffractometer with graphite monochromatic Mo $K\alpha$ radiation (λ =0.071 073 nm) at room temperature (Table 1). The structures of $\bf 1$ and $\bf 2$ were solved by direct method and refined with the full-matrix least-squares procedures on F^2 using the SHELXS-97 and SHELXL-97 programs [24], respectively. All H atoms were initially located in electron-density difference maps. For all two compounds, H atoms which bonded with C atoms were constrained to idealized positions. The selected bond lengths and angles for $\bf 1$ and $\bf 2$ are listed in Table 2.

CCDC: 1038410, 1; 1038411, 2.

2 Results and discussion

2.1 Crystal structure of 1

The crystal of 1 crystallizes in the $P2_1/c$ space group with monoclinic crystal system, and the coordination environment of 1 contains one Hg (III) cation, a carbene ligand and three counter anions containing one acetate anion and two hexafluorophosphate anions. Each Hg cation was coordinated by one oxygen atom and two carbene carbon atoms forming two adjacent symmetrical six-membered rings adopting distorted boat conformation for the bond

Table 1 Crystal structure data for complexes 1 and 2

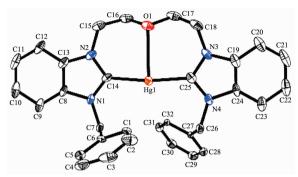
Compound	1	2
Empirical formula	$C_{34}H_{34}F_{12}HgN_4O_3P_2$	C ₃₀ H ₂₈ ClF ₆ N ₆ NiOP
Formula weight	1 037.18	727.71
Temperature / K	298	113
Crystal system	Monoclinic	Monoclinic
Space group	$P2\sqrt{c}$	$P2_1/n$
a / nm	1.034 36(12)	1.369 8(4)
b / nm	1.639 4(2)	1.101 2(3)
c / nm	2.086 7(2)	2.092 6(5)
β / (°)	90.840(2)	94.736(4)
V / nm^3	3.538 1(7)	3.145 7(14)
Z	4	4
$D_{\rm c}$ / (g·cm ⁻³)	1.947	1.537
F(000)	2 032	1 488
Absorption coefficient / mm ⁻¹	4.548	0.824
Range for date collection / (°)	3.02~25.0	1.7~27.9
Reflections collected	23 936	28 430
Ind reflns, $R_{ m int}$	6 217, 0.034	7 465, 0.047
Goodness-of-fit on F^2	1.118	1.017
$R_1 (I > 2\sigma(I))$	0.037 8	0.042 8
$wR_2(I>2\sigma(I))$	0.081 0	0.109 5

Table 2 Selected bond lengths (nm) and bond angles (°) for complexes 1 and 2

1						
C14-Hg1	0.207 2(5)	Hg1-O1	0.265 3(4)	C25-Hg1	0.207 4(5)	
C16-O1-Hg1	106.0(3)	N2-C14-Hg1	127.9(4)	C14-Hg1-C25	172.0(2)	
C17-O1-Hg1	108.9(3)	N4-C25-Hg1	124.0(4)	C14-Hg1-O1	85.53(17)	
N1-C14-Hg1	123.9(4)	N3-C25-Hg1	126.9(4)	C25-Hg1-O1	86.47(18)	
Hg1-O1-H1A	56.2					
	2					
Ni1-C13	0.186 3(2)	Ni1-C23	0.186 5(2)	Ni1-N1	0.193 1(22)	
Ni1-Cl1	0.221 7(97)					
C23-Ni1-N1	173.94(8)	C13-Ni1-Cl1	169.17(7)	C23-Ni1-Cl1	89.17(7)	
N1-Ni1-Cl1	91.29(6)	C1-N1-Ni1	118.38(15)	C5-N1-Ni1	122.07(14)	
N2-C13-Ni1	117.62(15)	N3-CB13-Ni1	136.00(17)	N4-C23-Ni1	122.39(15)	
N5-C23-Ni1	130.63(16)	C13-Ni1-C23	92.56(9)	C13-Ni1-N1	88.11(8)	

angles of C25-Hg1-O1 (86.47(18)°) and C14-Hg1-O1 (85.53(17)°) are almost equal (Fig.1). Two benzene rings are located away from the coordination sphere pointing to the opposite directions to reduce the steric clash, and making dihedral angles of 79.61(10)° (C1 \rightarrow C6 ring) and 62.52(12)° (C27 \rightarrow C32 ring) with linked benzimidazole ring, separately. The dihedral angle

between two benzimidazole planes is 1.14(15)°, indicating that two benzimidazole rings are almost in a plane. The Hg-O bond length is 0.265 3(4) nm, this value is somewhat lower than those (0.270 7(31) nm, 0.282 7(10) nm and 0.279 9(10) nm) reported^[25], however, longer than the regular Hg-O bond (0.184 0 nm) of mercuric oxide. The C25-Hg1-C14 is approxi-

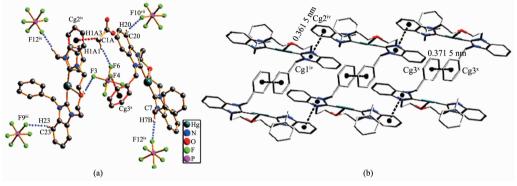


H atoms have been omitted for clarity

Fig.1 Molecular structure of complex 1, showing 50% probability displacement ellipsoids and the atom-numbering scheme

mately linear with the angle of $172.0(2)^{\circ}$ which is larger than those in Hg(II)-NHC complexes of $158.7(5)^{\circ[26]}$, and with the bond distances of Hg1-C14 and Hg1-C25 are 0.207 2(5) nm and 0.207 4(5) nm, respectively, which shorter than the analogical compound of those (0.209 1(13) nm) (Table 2).

The crystal structure of **1** contains $C-H\cdots F$, $O-H\cdots F$ hydrogen bonds and two types of $C-H\cdots \pi$ intermolecular contacts, as well as $\pi\cdots\pi$ stacking interactions (Fig.2), and the data are summarized in Table 3. The $[Hg(C_{32}H_{28}N_4O)]^{3+}$ arrange in four kinds of manner, and every such four adjacent cations



Hydrogen atoms, except those forming hydrogen bonds, are omitted for clarity, and the centers of rings are marked by black balls and signed Cg; Symmetry code: ${}^{\text{vii}}1-x$, -1/2+y, 1/2-z; ${}^{\text{ii}}1+x$, -1+y, z; ${}^{\text{iv}}1+x$, y, z; ${}^{\text{x}}1-x$, -y, -z; ${}^{\text{ix}}-x$, 1-y, -z

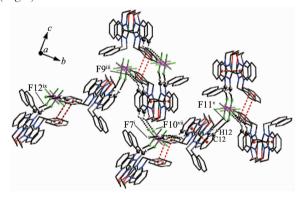
Fig.2 View of C-H···F, O-H···F hydrogen bonds and C-H··· π , P-F··· π intermolecular hydrogen bonds (a) and intermolecular π ··· π stacking (b) of 1

Table 3 Hydrogen bond distances and angles for the complexes 1 and 2

$\mathrm{DH\cdots A}$	d(D-H) / nm	$d(\mathbf{H}\cdots\mathbf{A})$ / nm	$d(\mathbf{D}\cdots\mathbf{A})$ / nm	\angle DHA / (°)
1				
O1-H1…F3	0.093	0.252	0.339 8(2)	158
C1-H1A1…F6	0.096	0.230	0.325 50	174
$\text{C7-H7B}\cdots\text{F12}^{\text{ix}}$	0.097	0.221	0.317	172
C12-H12···F11 ^v	0.093	0.249	0.325 4(6)	140
C15-H15A…F4	0.097	0.251	0.345 5(4)	165
$C20H20\cdots F10^{vii}$	0.093	0.239	0.319 7(9)	146
C23-H23···F9 ⁱⁱⁱ	0.093	0.239	0.310 3	132
C30-H30···F7 ⁱ	0.093	0.248	0.337 1(4)	160
2				
C2-H2···F5 ^{viii}	0.095	0.249	0.334 1(3)	149
C4-H4···F1 ^{xiii}	0.095	0.248	0.341 3(3)	167
C6-H6A···Cl1viii	0.099	0.282	0.352 3(2)	129
C8-H8···F2 ^{xiii}	0.095	0.253	0.335 1(3)	145
$C14H14A\cdots F4^{vi}$	0.099	0.244	0.304 6(1)	119
C24-H24B···F3 ^{xi}	0.099	0.235	0.329 0(3)	158

Symmetry code for 1: ${}^{i}x$, -1+y, z; ${}^{ii}1+x$, -1+y, z; ${}^{v}-x$, -1/2+y, 1/2-z; ${}^{vi}1-x$, -1/2+y, 1/2-z; ${}^{ix}-x$, 1-y, -z; Symmetry code for 2: ${}^{vi}1/2-x$, 1/2+y, 1/2-z; ${}^{vii}3/2-x$, 1/2+y, 1/2-z; ${}^{vii}1-x$, 1-y, 1-z; ${}^{vii}1/2+x$, 3/2-y, -1/2+z

connect together with each other leading to a crisscross cluster through the C-H···F hydrogen bonding interactions, meanwhile, the C1A-H1A3 $\cdots \pi_{\mathbb{C}^{2}}$ and P1 -F4 $\cdots \pi_{\mathbb{C}_{2}^{3}}$ interactions to solidify the structure (Fig.2(a)). The uncoordinated CH₃COO- was linked by C1A-H1A1 ··· F6 hydrogen bond to the cluster as counter anions. The cluster links the same neighbor units with similar way to form a crossing infinite 2D layer. In addition, there are $\pi \cdots \pi$ stacking interactions between rings of C8 \rightarrow C13 (Cg2^{iv}: x=-0.007 5(4), y=0.325 9, z=0.166 4(7)) and N3-C19-C24-N4-C25 $(Cg1^{iv}: x=0.7767(3), y=0.1648(9), z=0.2157)$ with the dihedral angle of 1.321° and ring-centroid separation of 0.361 5(2) nm. The another $\pi \cdots \pi$ stacking interactions exist between the benzene rings of C27→C32 $(Cg3^x: x=0.532\ 3(3), y=-0.001\ 7(6), z=0.087\ 7(8))$ and these rings are parallel to each other with the distances of 0.371 5 nm (Fig.2(b)). The two kinds of stacking extend those layers into a 3D supramolecular structure (Fig.3).



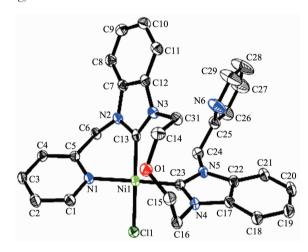
Hydrogen atoms, except those forming hydrogen bonds, are omitted for clarity; Symmetry code: ${}^{i}x$, -1+y, z; ${}^{ii}1+x$, -1+y, z; ${}^{v}-x$, -1/2+y, 1/2-z; ${}^{vi}1-x$, -1/2+y, 1/2-z; ${}^{ix}-x$, 1-y, -z

Fig.3 View of part of 3D supramolecular structure of 1

2.2 Crystal structure of 2

The complex **2** crystallizes in the *P*2₁/*n* space group with monoclinic crystal system. One Ni atom coordinates with one chlorine, two carbon atoms of pyridine forming a square planar, and one hexafluorophosphate anion acts as counter anion in the unit. Unexpectedly, the nitrogen atom of the pyridine ring (C25-C26-C27-C28-C29-N6) is not coordinate with the Ni atom and has moved far away to avoid conflict (Fig.4). The C13-Ni1-C23 is approximately perpen-

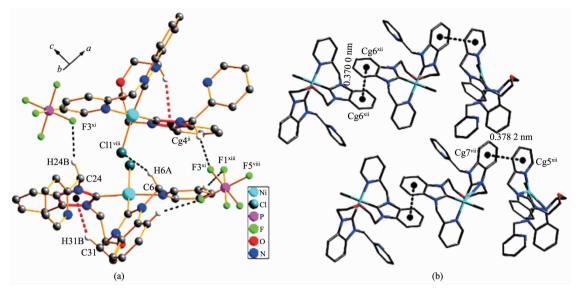
dicular with the angle of 92.56 (9)°, the Ni-C bond distances are 0.186 3(2) nm and 0.186 5(2) nm, respectively, which are quite normal compared with the reported values of the Ni-NHC complex^[27-28]. The Ni-Cl bond length is 0.221 7(97) nm, and the distance of Ni-N1 is 0.193 1(22) nm. The dihedral angle between two benzimidazole planes is 65.13(4)° and those formed of the coordinated pyridine ring (C1-C2-C3-C4-N1) and the benzimidazole planes are 13.28 (9)° (N4-C17-C22-N5-C23 ring) and 57.21(5)° (C13-N2-C7-C12-N3 ring).



H atoms have been omitted for clarity

Fig.4 Molecular structure of **2**, showing 50% probability displacement ellipsoids and the atom-numbering scheme

Similarly with 1, the crystal structure of 2 also exists $C-H\cdots F$, $C-H\cdots Cl$ hydrogen bonds, $C-H\cdots \pi$ (arene) intramolecular force, and two types of $\pi \cdots \pi$ stacking interactions between the hybrid aryl ring (Fig.5), the related data are in Table 3. Two adjacent [NiCl (C₃₀H₂₈N₆O)] + in different arrangement bridge together by C6-H6 ··· Cl1viii and other C-H ··· F hydrogen bonds including C31 -H31B \cdots $\pi_{ce^{-i}}$ intramolecular interaction obtaining a back to back unit (Fig.5(a)). Then, C2-H2···F5viii and C14-H14A··· F4vi hydrogen bonds act as messengers to link such similar adjacent units forming infinite 2D layer. The distances of ring-centroid are 0.37082 nm and 0.3700 nm which indicating there are weak $\pi \cdots \pi$ stacking interactions between the parallel π planes which formed by C7 \rightarrow C12(Cg6^{vii}: x=0.449 3(5), y=1.035 0,



Hydrogen atoms, except those forming hydrogen bonds, are omitted for clarity, and the centers of rings are marked by black balls and signed Cg; Symmetry code: viii 3/2-x, 1/2+y, 1/2-z; viii 1/2+x, 3/2-y, -1/2+z; vi 1-x, 1-y, 1-z; vi 1-x, y, z; vii 1-x, -1/2+y, 1/2-z; vii 1-x, 2-y, -z

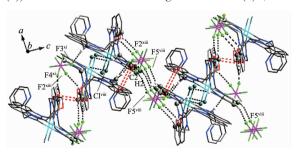
Fig.5 C-H···F, C-H···Cl hydrogen bonds and C-H··· π intramolecular hydrogen bonds (a) and intermolecular π ··· π stacking (b) of **2**

Table 4 C/P-H/F \cdots π interactions for the complexes 1 and 2

С−H/F…π _{сg}	d(C/P-H/F) / nm	$d(H/F\cdots Cg)$ / nm	$d(C/P\cdots Cg)$ / nm	∠C/P−H/F····Cg / (°)
1				
C1A-H1A3···π _{cg2} iv	0.096 0	0.283 1	0.343 4	122
P1-F4 $\cdots\pi_{^{\text{Cg3}}}^{^{\text{x}}}$	0.159 8	0.336 3	0.336 3(6)	124
2				
С31–H31В… $\pi_{c_g4}^{ii}$	0.099 1	0.256 0	0.345 3(1)	150

Symmetry code for 1: $^{\text{iv}}$ 1+x, y, z; $^{\text{x}}$ 1-x, -y, -z; Cg2 $^{\text{iv}}$: C8 \rightarrow C13; Cg3 $^{\text{x}}$: C27 \rightarrow C32; Symmetry code for 2: $^{\text{ii}}$ x, y, z; Cg4 $^{\text{ii}}$: N4-C17-C22-N5-C23

z=0.077 1(8)) with the distances between the two planes are 0.362 1(5) nm. The $\pi \cdots \pi$ stacking interactions exist between rings C17 \rightarrow C22(Cg7 $^{\text{vii}}$: x=0.472 5(2), y=0.494 9(9), z=0.317 4) and N1-C1-C2-C3-C4-C5 (Cg5 $^{\text{vii}}$: x=0.850 2(4), y=0.823 2(8), z=0.067 4(9)) (Fig. 5(b)) with the dihedral angles of 5.954(5) $^{\circ}$, which



Hydrogen atoms, except those forming hydrogen bonds, are omitted for clarity; Symmetry code: v^{iii} 3/2-x, 1/2+y, 1/2-z; v^{iii} 1/2+x, 3/2-y, -1/2+z; v^{ii} 1/2-x, 1/2+y, 1/2-z; v^{ii} 1-x, 1-y, 1-z

Fig.6 View of part of 3D supramolecular structure of ${\bf 2}$

resulting in a 3D supramolecular structure (Fig.6).

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