

# *N*-苯基-1,7-二硒杂-4-氮杂[7]二茂铁环蕃的合成、晶体结构及其光电性质研究

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**摘要:** 合成了 *N*-苯基-1,7-二硒杂-4-氮杂-[7]二茂铁环蕃(**1**), 并用核磁、质谱、单晶 X-射线衍射进行了表征。晶体结构测试结果表明 **1** 是属于正交晶系的 *Ibca* 空间群, 晶胞参数  $a=0.954\ 74(2)\ \text{nm}$ ,  $b=0.959\ 58(2)\ \text{nm}$ ,  $c=7.859\ 0(2)\ \text{nm}$ , 其中晶胞参数  $c$  值很长, 这种现象在 N-Se 杂大环中很少见。光谱研究显示 **1** 仅对  $\text{Hg}^{2+}$  和  $\text{Cu}^{2+}$  的加入产生响应, 表现为紫外最大吸收峰的红移和荧光淬灭。

**关键词:** 硒-氮杂二茂铁环蕃; 晶体结构; 汞离子; 铜离子

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## Preparation, Structure, Spectral and Electrochemical Properties of *N*-phenyl-1,7-diselenena-4-aza-[7]ferrocenophane

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**Abstract:** *N*-phenyl-1,7-diselenena-4-aza-[7]ferrocenophane (**1**) has been successfully prepared. The compound has been characterized by <sup>1</sup>H NMR spectroscopy, mass spectrum, X-ray crystallography. **1** crystallizes in the orthorhombic space group *Ibca*,  $a=0.954\ 74(2)\ \text{nm}$ ,  $b=0.959\ 58(2)\ \text{nm}$ ,  $c=7.859\ 0(2)\ \text{nm}$ . Unit cell parameter  $c$  of **1** is rare in selenena-aza macrocycles. **1** displays a red shift of absorption maxima and a selective fluorescence quenching upon addition of  $\text{Hg}^{2+}$  and  $\text{Cu}^{2+}$ . CCDC: 861107.

**Key words:** selenena-aza ferrocenophane; crystal structure; mercury ion; cupric ion

## 0 Introduction

The design of novel chemosensors capable of signalling the presence of hazardous metal ions is an interesting subject for chemists. Thereinto, dual channel chemosensor for signalling transition metal cations are particularly charming<sup>[1]</sup>. In the array of signalling methods, chromogenical-fluorogenical signal

is competitive for some advantages, for example, it is easily miniaturizable and can avoid electrical interference<sup>[2]</sup>. As a good receptor unit in chemosensor, the mixed donor macrocycles are more attractive for their highly selective nature for heavy metal ions<sup>[3]</sup>. To date, a great number of polyoxa<sup>[4-6]</sup>, polyaza<sup>[7-12]</sup>, oxa-aza macrocycles<sup>[13-15]</sup>, have been studied as dual channel chemosensors for the detection of alkali and hard

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metal ions. However, less attention has been paid to thia-aza<sup>[16-19]</sup> and seleno-aza macrocycles<sup>[20-24]</sup>, which may show strong affinity to both hard and soft transition-metal ions. Some seleno-phospha ferrocenophane and seleno-arsena ferrocenophane have been successfully synthesized by Osborne group<sup>[25]</sup>. Unfortunately, their ion sensing property was not studied. To the best of our knowledge, no seleno-aza ferrocenophane has ever been reported. Aniline group is selected first to construct seleno-aza ferrocenophane, as a fluorescent unit, it is expected to give chromogenical-fluorogenical signal in combining with target cations<sup>[26-29]</sup>. Pursuing these clues, a novel macrocyclic ferrocenophane, *N*-phenyl-1,7-diselena-4-aza-[7]ferrocenophane (**1**) has been synthesized. Its crystal structure and electrochemical study was investigated. Cation recognition properties of **1** were also analyzed via two channels, chromogenically and fluorogenically.

## 1 Experimental

### 1.1 Materials, general procedures and physical measurements

All the reactions were carried out under nitrogen using standard Schlenk techniques. THF was distilled from K alloy, and ethanol was degassed before use. Mass spectra were recorded using positive ion electrospray (ES);  $m/z$  values have been rounded to the nearest integer or half-integer. Assignments are based on isotopomers containing <sup>1</sup>H, <sup>12</sup>C, <sup>14</sup>N, <sup>56</sup>Fe, <sup>80</sup>Se. Cyclic voltammetry (CV) was performed at room temperature in a dry MeCN solution containing 0.1 M [NBu<sub>4</sub>]PF<sub>6</sub> electrolyte using CHI660D potentiostat system. The sweep rate was 100 mV · s<sup>-1</sup> (CV). A three-electrode arrangement was used with a Pt working electrode, a Pt wire counter electrode and a Ag/Ag<sup>+</sup> (0.01 mol · L<sup>-1</sup> AgNO<sub>3</sub> in MeCN) reference electrode. All the E values listed below are with respect to FcH/[FcH]<sup>+</sup>, the separation of E<sub>a</sub> and E<sub>c</sub> is given in brackets. Absorption spectra were recorded on a HITACHI spectrophotometer UV-3900. Fluorescence emission spectra were recorded on a HITACHI spectrophotometer F-7000. For all measurements, excitation was at 290 nm. Both excitation and

emission slit widths were 10 nm. UV-Vis and fluorescence studies of **1** were carried out using 10<sup>-4</sup> mol · L<sup>-1</sup> dichloromethane solution.

### 1.2 Synthesis of *N*-phenyl-1,7-diselena-4-aza-[7]ferrocenophane (**1**)

To a solution of fcSe<sub>3</sub>, (0.428 g, 1 mmol, fc=[Fe(η<sup>5</sup>-C<sub>5</sub>H<sub>4</sub>)(η<sup>5</sup>-C<sub>5</sub>H<sub>4</sub>)] in 150 mL ethanol dried by anhydrous magnesium sulfate, sodium borohydride (0.300 g, 8 mmol) was added in ice-bath. The reaction was stirred and maintained at 0 °C for 10 min, and then rose to room temperature. After 2 h, a solution of *N,N*-bis(bromoethyl)aniline (0.307 g, 1 mmol) in 5 mL tetrahydrofuran was added, which was prepared according to literature<sup>[30]</sup>. The reaction mixture left to stir for 3 h at room temperature, and then the solvent was removed by evaporation under reduced pressure. The residue was treated with water (25 mL) and extracted with CH<sub>2</sub>Cl<sub>2</sub> (3×25 mL). The red extract was dried over MgSO<sub>4</sub>, evaporated to dryness, and then purified by column chromatography ( $V_{\text{hexane}}/V_{\text{CH}_2\text{Cl}_2}=1:1$ ) on silica gel (0.211 g, 43%). <sup>1</sup>H NMR (CDCl<sub>3</sub>): 7.26 (C<sub>6</sub>H<sub>5</sub>, H<sub>3+5</sub>, t, 2H), 6.81 (C<sub>6</sub>H<sub>5</sub>, H<sub>2+6</sub>, d, 2H), 6.76 (C<sub>6</sub>H<sub>5</sub>, H<sub>4</sub>, t, 1H), 4.25 (C<sub>5</sub>H<sub>4</sub>, H<sub>2+5</sub>, s, 4H), 4.19 (C<sub>5</sub>H<sub>4</sub>, H<sub>3+4</sub>, s, 4H), 3.94 (NCH<sub>2</sub>, t, 4H), 3.40 (fcSeCH<sub>2</sub>, t, 4H). ES-MS: 490 ([M+1]<sup>+</sup>).

### 1.3 Single crystal structure determination

Data for the compound was collected by using a Bruker Apex 2 CCD single-crystal diffractometer with Mo *K*α radiation (λ=0.071 073 nm) at room temperature using the ω-2θ scan method. The single crystal of **1** was chosen onto a thin glass ber by epoxy glue in air for data collection. The structure was solved by direct methods and refined on *F*<sup>2</sup> by full-matrix least-squares methods using the SHELX97<sup>[31]</sup> program package. All non-H atoms were refined anisotropically, while the H atoms of the organic moieties were included in calculated positions, assigned isotropic displacement parameters, and allowed to ride on their parent atoms. A summary of the crystallographic data and structural determination for **1** is provided in Table 1 and selected bond lengths and angles are listed in Table 2.

CCDC: 861107.

**Table 1** Crystal data and structure refinement for **1**

Empirical formula	C <sub>20</sub> H <sub>21</sub> FeNSe <sub>2</sub>	<i>F</i> (000)	3 872
Formula weight	489.15	Crystal size / mm	0.20×0.16×0.14
Temperature / K	296(2)	$\theta$ range for data collection / (°)	1.04~25.00
Wavelength / nm	0.071 073	Limiting indices	$-11 \leq h \leq 11, -11 \leq k \leq 11, -93 \leq l \leq 93$
Crystal system	Orthorhombic	Reflections collected	22 952
Space group	<i>Ibca</i>	Independent reflections ( <i>R</i> <sub>int</sub> )	3 170 (0.117 8)
<i>a</i> / nm	0.954 74(2)	Observed reflections ( <i>I</i> >2 $\sigma$ ( <i>I</i> ))	2 518
<i>b</i> / nm	0.959 58(2)	Data / restraints / parameters	3 170 / 0 / 221
<i>c</i> / nm	7.859 0(2)	Goodness-of-fit on <i>F</i> <sup>2</sup>	1.168
Volume / nm <sup>3</sup>	7.2(2)	Final <i>R</i> indices ( <i>I</i> >2 $\sigma$ ( <i>I</i> ))	<i>R</i> <sub>1</sub> =0.061 4, <i>wR</i> <sub>2</sub> =0.150 8
<i>Z</i>	16	<i>R</i> indices (all data)	<i>R</i> <sub>1</sub> =0.076 2, <i>wR</i> <sub>2</sub> =0.158 0
Calculated density / (g·cm <sup>-3</sup> )	1.805	Largest diff. peak and hole / (e·nm <sup>-3</sup> )	792 and -992
Absorption coefficient / mm <sup>-1</sup>	4.88		

**Table 2** Selected bond lengths (nm), bond angles (°) and dihedral angles (°) for **1**

Se1-C5	0.191 0(8)	Se1-C6	0.195 1(7)	N1-C(7)	0.145 5(7)
N1-C8	0.136 6(1)	Se2-C26	0.195 7(7)	N2-C27	0.145 7(7)
N2-C28	0.138 0(1)	Se2-C25	0.191 4(8)		
C5-Se1-C6	100.1(3)	C8-N1-C7	121.8(4)	C7-N1-C7A	116.5(8)
N1-C8-C9	122.3(4)	C25-Se2-C26	100.3(3)	C28-N2-C27	122.0(4)
C27-N2-C27A	115.9(8)	N2-C28-C29	122.0(4)		
C5-Se1-C6-C7	68.22	C8-N1-C7-C6	86.82	C25-Se2-C26-C27	69.51
C28-N-C27-C26	-87.15				

 Symmetry code: A: 1/2-*x*, -*y*, 1/2-*z*

## 2 Results and discussion

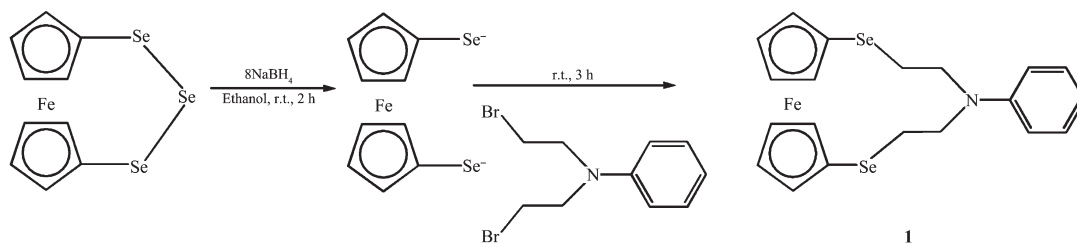
### 2.1 Synthesis and characterization

**1** was synthesized as shown in scheme 1. The reaction of *N,N*-bis(bromoethyl)aniline with one equivalent of 1,1'-bis(3-bromopropylseleno)ferrocene in an ethanol solution gave **1** (43%) with nucleophilic substitution process. It suggests that ferrocene has the correct geometry for ring closure via a [1+1] cyclisation process. In <sup>1</sup>H NMR spectra, the protons of the Cp (cyclopentadiene) rings in **1** appeared as two well

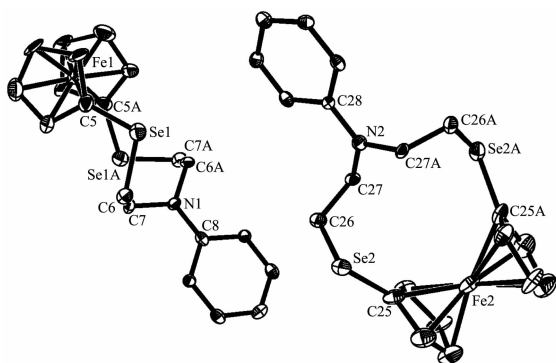
separated singlets at about  $\delta$  4.25 and 4.19 (4:4).

### 2.2 Single crystal structure determination

Crystals of **1** were obtained as red blocks from CH<sub>2</sub>Cl<sub>2</sub>/hexanes. **1** crystallizes in the orthorhombic space group of *Ibca* with two different semimolecules in the asymmetric unit. The dihedral angles between cyclopentadienyl ring plane and phenyl ring plane of two molecules are different, one is 47.33° and the other is 56.98°. It's worth noting that **1** has a large *c* value of 7.859(2) nm, no similar phenomenon has been observed among aza-selena macrocycles<sup>[20-24]</sup>.


 Scheme 1 Synthetic route to **1**

As shown in Fig.1, all molecules adopt an exo conformation with the Se atoms pointing out of the cyclic cavity in **1**. The dihedral angles between the two cyclopentadienyl ring planes ( $9.38^\circ$  and  $8.54^\circ$  in **1**) are larger than that in 1,5-diselena[5]ferrocenophane ( $8.52^\circ$ )<sup>[32]</sup> and 1,3-diselena-2-phenylphosphino[3]ferrocenophane ( $0.6^\circ$ )<sup>[25]</sup>, which may be ascribed to the lack of flexibility in **1**. In **1**, the ferrocene moiety adopts a staggered conformation with the torsion angle  $49.97^\circ$  ( $C5\cdots X1\cdots X2\cdots C5A$ ) and  $50.59^\circ$  ( $C25\cdots X3\cdots X4\cdots C25A$ ) ( $X$  is the centroid of each Cp ring). The phenyl and p-tolyl rings occupy the sterically less crowded exo-position on N in **1**. The N-C(aryl) bond distance ( $0.136\ 6\ \text{nm}$  and  $0.138\ 0\ \text{nm}$  in **1**) is significantly shorter than those of N-CH<sub>2</sub> bonds ( $0.145\ 5\ \text{nm}$  and  $0.145\ 7\ \text{nm}$  in **1**). In **1**, the adjacent asymmetric units are connected through weak C9-H $\cdots\pi$  ( $0.360\ 5\ \text{nm}$ ) and C31-H $\cdots$ Se1 interactions ( $0.374\ 1\ \text{nm}$ ), forming 3D supramolecular framework (Fig.2).



Hydrogen atoms are omitted for clarity; Symmetry codes: A:  $1/2-x, -y, 1/2+z$ ; B:  $-x, 1/2+y, 1/2-z$ ; C:  $1/2+x, 1/2-y, -z$

Fig.1 Asymmetric unit of **1** with the atomic numbering scheme adopted with thermal ellipsoids drawn at 30% probability level

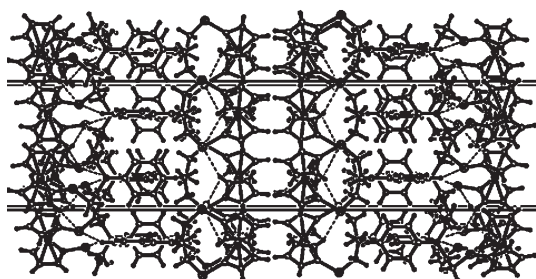


Fig.2 3D supramolecular framework of **1**

## 2.3 Electrochemical study

Cyclic voltammetry was used to investigate the

electrochemistry of **1** in acetonitrile solution. As depicted in Fig.3, **1** exhibits the reversible redox process at  $E_{1/2} = -26.5\ \text{mV}$  ( $|E_{pa} - E_{pc}| = 87$ ). The value is more negative than that in 1,2,3-triselena[3]ferrocenophane ( $E_{1/2} = 213\ \text{mV}$ )<sup>[33]</sup> and *N*-(4-butylphenyl)-2-aza[3]ferrocenophane ( $E_{1/2} = 100\ \text{mV}$ )<sup>[34]</sup>. Ferrocenyl unit is easily oxidized, meaning the decrease of the ring strain and the weakened interaction between the nitrogen atom in the bridge and the Fe(II) ion. There is irreversible oxidation process at  $E_{1/2} = 712\ \text{mV}$  (**1**), corresponding to the oxidation of amine<sup>[34-38]</sup>.

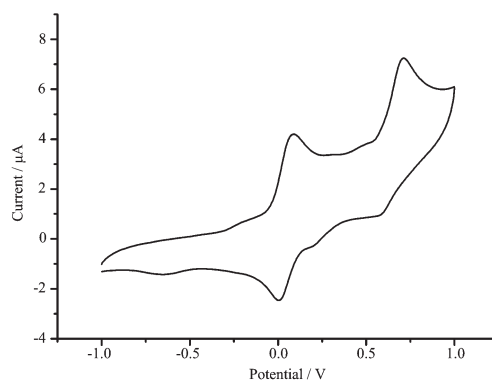
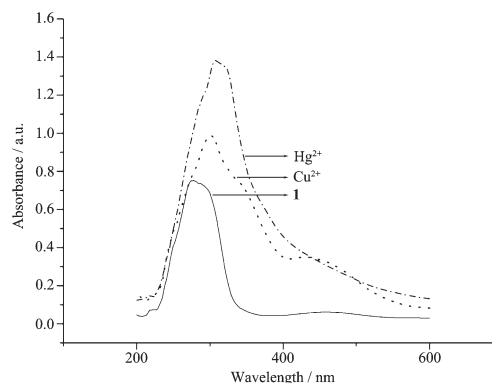


Fig.3 Cyclic voltammogram of **1**

## 2.4 UV-Vis absorption and fluorescent studies

The UV-Vis spectrum of **1** in CH<sub>2</sub>Cl<sub>2</sub> solution ( $c_1 = 1 \times 10^{-4}\ \text{mol} \cdot \text{L}^{-1}$ ) exhibits a prominent band at  $\lambda_{\text{max}} = 274\ \text{nm}$ , which can safely be ascribed to a high-energy ligand-center  $\pi-\pi^*$  electronic transition. The weak absorption at  $460\ \text{nm}$  can be assigned to localized excitation with a lower energy produced by either of two nearly degenerate transitions: an Fe(II) d-d



$c_1 = 1 \times 10^{-4}\ \text{mol} \cdot \text{L}^{-1}$ ;  $c_{\text{Cu}^{2+}} = 3 \times 10^{-2}\ \text{mol} \cdot \text{L}^{-1}$ ;  $c_{\text{Hg}^{2+}} = 3 \times 10^{-2}\ \text{mol} \cdot \text{L}^{-1}$

Fig.4 UV-Vis spectral changes of **1** in dichloromethane-acetonitrile (1:1, V/V) solution upon addition of Cu<sup>2+</sup> and Hg<sup>2+</sup>

transition or by a metal-ligand charge transfer (MLCT) process ( $d_{\pi}-\pi^*$ ) (LE band)<sup>[39-40]</sup>.

The metal recognition property of **1** towards  $\text{Na}^+$ ,  $\text{Ni}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Eu}^{3+}$ ,  $\text{Hg}^{2+}$  and  $\text{Pb}^{2+}$  metal cations was investigated by UV-Vis and fluorescence measurements. Its chemosensor behaviour in the presence of the above-mentioned set of metal ions indicated that only  $\text{Cu}^{2+}$  and  $\text{Hg}^{2+}$  ions promoted a notable response in its absorption spectra, while the other metal ions tested induced negligible responses. Notably, upon addition of  $\text{Cu}^{2+}$  and  $\text{Hg}^{2+}$  ions induced an increase and a red shift of the initial HE band at 274 nm ( $\text{Cu}^{2+}$ ,  $\Delta\lambda=26$  nm;  $\text{Hg}^{2+}$ , 35 nm). Simultaneously, there is an increase in the intensity of the band at 460 nm, and a new band at around 350 nm was gradually developed upon addition of  $\text{Cu}^{2+}$ . All these changes can be assigned to complexation<sup>[40-41]</sup>.

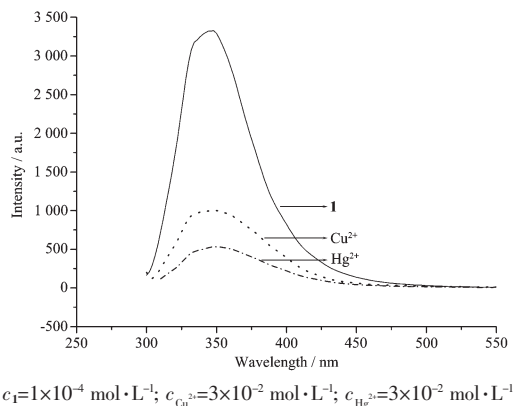


Fig.5 Fluorescence spectra change of **1** in dichloromethane-acetonitrile (1:1, V/V) solution upon addition of  $\text{Cu}^{2+}$  and  $\text{Hg}^{2+}$

In fluorescence spectra, **1** exhibits a relatively intense fluorescence in  $\text{CH}_2\text{Cl}_2$  ( $c_1=1\times 10^{-4} \text{ mol}\cdot\text{L}^{-1}$ ) when excited at  $\lambda_{\text{ex}}=290$  nm. The emission spectrum displays one well-defined emission band at 346 nm, which can be ascribed to the emission of aniline group. Upon addition of  $\text{Cu}^{2+}$  and  $\text{Hg}^{2+}$ , a remarkable quenching of the emission band at 346 nm was observed, probably due to PET process.

### 3 Conclusions

**1** crystallizes in the orthorhombic space group *Ibca* with a large *c* of 7.859(2) nm, which has never been observed in analogous aza-selena molecules. The

metal recognition property of **1** was evaluated by UV-vis and fluorescence measurements with the set of cations previously mentioned. Addition of  $\text{Hg}^{2+}$  and  $\text{Cu}^{2+}$  to **1** led to an increase and a red shift of the absorption maxima and fluorescent quenching.

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