

## 含 $\alpha$ -二亚胺配体的硅(IV)配合物的合成、晶体结构与性能研究

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**摘要:** 利用金属单质还原的方法合成了不同取代基的  $\alpha$ -二亚胺配体支持的 2 个硅(IV)配合物  $L(\text{SiMe}_3)_2(\mathbf{2})$  ( $L=[(2,6\text{-}i\text{-Pr}_2\text{C}_6\text{H}_3)\text{NC}(\text{Me})_2]$ ) 和  $L'(\text{SiMe}_3)_2(\mathbf{4})$  ( $L'=[(2,6\text{-}i\text{-Pr}_2\text{C}_6\text{H}_3)\text{NCH}_2]$ )。通过 X-射线单晶衍射测定了配合物的单晶结构,并对其进行了元素分析、<sup>1</sup>H NMR、红外光谱表征,以及紫外-可见光谱和荧光光谱分析。结构分析表明,构成这 2 种化合物中心的 NCCN 骨架呈之字形分布,骨架上三取代的原子接近平面排布。2 种硅配合物在紫外光激发下都具有较好的发光性质。

**关键词:**  $\alpha$ -二亚胺; 晶体结构; 硅(IV)配合物; 光致发光

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### Synthesis, Crystal Structure and Properties of Silicon(IV) Complexes with N-aryl Substituted $\alpha$ -Diimine Ligands

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**Abstract:** Two silicon(IV) compounds with  $\alpha$ -diimine ligands,  $L(\text{SiMe}_3)_2(\mathbf{2})$  ( $L=[(2,6\text{-}i\text{-Pr}_2\text{C}_6\text{H}_3)\text{NC}(\text{Me})_2]$ ),  $L'(\text{SiMe}_3)_2(\mathbf{4})$  ( $L'=[(2,6\text{-}i\text{-Pr}_2\text{C}_6\text{H}_3)\text{NCH}_2]$ ) were synthesized by the reaction of corresponding sodium salts or lithium salts with chloro(trimethyl)silane. The crystal of compound **2** belongs to the monoclinic system, space group  $P2_1/n$  with  $a=0.9585(3)$  nm,  $b=1.8118(5)$  nm,  $c=0.9824(3)$  nm,  $\beta=102.173(3)^\circ$ ,  $V=1.6677(8)$  nm<sup>3</sup>,  $Z=2$ . The crystal of compound **4** belongs to the triclinic system, space group  $P\bar{1}$  with  $a=0.8837(2)$  nm,  $b=0.9675(2)$  nm,  $c=1.1429(4)$  nm,  $\beta=88.94(2)^\circ$ ,  $V=0.8358(4)$  nm<sup>3</sup>,  $Z=1$ . A zig-zag arrangement of the atoms N(1), C(13), C(13<sup>i</sup>), and N(1<sup>i</sup>) is the core of both of the two molecular structures. Both of the two complexes were characterized by single crystal X-ray structural analysis, <sup>1</sup>H NMR, elemental analysis, and FTIR spectra, analyzed by UV-Vis spectrum and fluorescent spectra properties. The compounds exhibit strong fluorescence under ultraviolet light. CCDC: 896958, **2**; 896957, **4**.

**Key words:**  $\alpha$ -diimine; crystal structure; silicon(IV) complex; photoluminescence

## 0 Introduction

Main group metal compounds supported by N, N-chelating  $\alpha$ -diimine ligands successfully used for new olefin polymerization catalysts were widely investigated for their usage in industrial applications<sup>[1-2]</sup>. The

steric and electronic properties of  $\alpha$ -diimine ligands can be readily modified by attaching variable substituents on the carbon and nitrogen atoms of the NCCN backbone. The N, N-chelating  $\alpha$ -diimine agents have been widely adopted for the syntheses of the main group elements<sup>[3]</sup>. Recently, such complexes have

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been shown to be of technological significance<sup>[4-6]</sup>, which shows that certain gallium and magnesium derivatives are highly efficient  $\alpha$ -olefin polymerization catalysts.

N-aryl substituted N-heterocyclic silicon compounds were already reported previously, but the chemistry of these compounds and their use in synthesis is still very limited. In 2010, Zark<sup>[7]</sup> et al. reported on a series of silicon(IV) heterocycles. Doyle<sup>[8]</sup> and coworkers reported on new group 14 element(IV) compounds with  $\beta$ -diiminates in which  $[\text{ArNC}(\text{Me})\text{C}(\text{H})\text{C}(\text{Me})\text{N}(\text{Ar})\text{SiMe}_3]$  was firstly characterized. Efficient preparations of new ligand systems are crucial to the design and development of new catalyst systems. We became interested in monoanionic and dianionic iminoderived ligands with a view to stabilising active catalysts based on main group metals. Recently we used N-aryl-substituted  $\alpha$ -diimine ligands to synthesize a group of compounds with nitrogen-silicon bond by the metal reduction methods<sup>[9-12]</sup>. In these compounds, the ligands appear as the dianionic form. In order to further evaluate the application of  $\alpha$ -diimine ligands in silicon complexes, two silicon compounds were synthesized with  $\alpha$ -diimine ligands bearing different substituents and negative charges. Herein, we report on two silicon complexes with such ligands (**L**, **L'**), **L**( $\text{SiMe}_3$ )<sub>2</sub> (**2**), **L'**( $\text{SiMe}_3$ )<sub>2</sub> (**4**).

## 1 Experimental

### 1.1 General procedures

All manipulations were carried out under a purified nitrogen atmosphere using Schlenk techniques or inside a MB200G Etelux glovebox. All solvents were distilled from Na/benzophenone ketyl prior to use. Commercially available chemicals were purchased from J&K chemical or VAS and used as received. Elemental analyses were performed by the Analytical Instrumentation Center of the Peking University. <sup>1</sup>H NMR spectra were recorded on Bruker AM 400 spectrometers. IR spectra were recorded on a Nicolet iS10 spectrophotometer in 4 000~400  $\text{cm}^{-1}$  region. UV-Vis spectra were taken on a U-3900 spectrophotometer. Luminescence spectra were

measured on a FluoroMax-4 spectrophotometer. Melting points were measured in sealed glass tubes and not corrected.

### 1.2 Synthesis

#### 1.2.1 Synthesis of $[\text{C}(\text{Me})\text{N}(2,6\text{-}^i\text{Pr}_2\text{C}_6\text{H}_3)\text{SiMe}_3]_2$ (**2**)

**L** (0.404 g, 1 mmol) was dissolved in THF (20 mL) and 4 equiv of sodium (0.092 g) was added. The resultant suspension was stirred at room temperature for three days to give a red suspension, which was filtered and the filtrate was cooled to 0 °C. To the cooled solution was added chloro(trimethyl)silane (0.25 mL, 2 mmol) dropwise. The resultant solution was warmed up to room temperature and stirred for 24 h. Then volatiles were removed in vacuo and the residue was extracted into *n*-hexane (20 mL). Concentration of this extract to 15 mL yielded, after cooling at -15 °C for 2 d, colourless crystals of **2** (0.45 g, 81%). Mp: 146~148.6 °C. <sup>1</sup>H NMR (400 MHz,  $\text{C}_6\text{D}_6$ , 25 °C, TMS,  $\delta$  / ppm):  $\delta$  0.215 (s, 18 H,  $\text{Si}(\text{CH}_3)_3$ ), 1.14 (d, <sup>3</sup> $J_{\text{H-H}}=6.8$  Hz, 12 H,  $\text{CH}(\text{CH}_3)_2$ ), 1.42 (d, <sup>3</sup> $J_{\text{H-H}}=6.8$  Hz, 12 H,  $\text{CH}(\text{CH}_3)_2$ ),  $\delta$  2.10 (s, 6 H,  $\text{CCH}_3$ ), 3.59 (sept, <sup>3</sup> $J_{\text{H-H}}=6.8$  Hz, 4 H,  $\text{CH}(\text{CH}_3)_2$ ), 7.06~7.16 (m, 6 H, Ar-H); Anal. Calcd. for  $\text{C}_{34}\text{H}_{58}\text{N}_2\text{Si}_2$ , (551.00, %): C, 74.11; H, 10.61; N, 5.08; Found (%): C, 74.59; H, 10.88; N, 5.34. IR(KBr pellet,  $\text{cm}^{-1}$ ): 3 060(s), 3 020(s), 2 960(s), 2 870(s), 1 660(w), 1 650(w), 1 580(w), 1 460(s), 838(s), 806(s), 769(s). UV-Vis spectrum( $2 \times 10^{-4}$  mol  $\cdot$  L<sup>-1</sup> *n*-Hexane solution):  $\lambda_{\text{max}}$  ( $\epsilon_{\text{max}}$ ) 281 nm (3 292.5  $\text{dm}^3 \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$ ).

#### 1.2.2 Synthesis of $[\text{CHN}(2,6\text{-}^i\text{Pr}_2\text{C}_6\text{H}_3)\text{SiMe}_3]_2$ (**4**)

**L'** (0.376 g, 1 mmol) was dissolved in THF (20 mL) and 4 equiv of lithium powder (0.032 g) was added. The resultant suspension was stirred at room temperature for three days to give a red suspension, which was added anhydrous chloro(trimethyl)silane (0.25 mL, 2 mmol) in THF (8 mL) dropwise at 0 °C. Then it was allowed to warm to room temperature and stirred for 24 h to give a yellow suspension before removal of the solvent and extraction with *n*-hexane (20 mL). It was then filtered and the filtrate concentrated to about 15 mL and stored at -15 °C for several days to yield the product as colorless crystals (0.41 g, 77.5%). Mp: 237.8~240.3 °C. <sup>1</sup>H NMR(400

MHz,  $C_6D_6$ , 25 °C, TMS,  $\delta$  / ppm):  $\delta$  0.213 (s, 18 H,  $SiMe_3$ ), 1.16 (d,  $^3J_{H-H}=6.8$  Hz, 12 H, CH  $(CH_3)_2$ ), 1.44 (d,  $^3J_{H-H}=6.8$  Hz, 12 H, CH $(CH_3)_2$ ), 3.58 (sept,  $^3J_{H-H}=6.8$  Hz, 4 H, CH $(CH_3)_2$ ),  $\delta$  5.18 (s, 2 H, CH=CH), 7.16~7.21 (m, 6 H, Ar-H); Anal. Calcd. for  $C_{32}H_{54}N_2Si_2$ , (522.95, %): C, 73.49; H, 10.41; N, 5.36; Found (%): C, 73.80; H, 10.18; N, 5.72. IR (KBr pellet,  $cm^{-1}$ ): 3 054(s), 3 010(s), 2 970(s), 2 865(s), 1 666(w), 1 625 (w), 1 579(w), 1 463(s), 830(s), 775(s), 754(s). UV-Vis spectrum ( $2 \times 10^{-4}$  mol $\cdot$ L $^{-1}$  *n*-Hexane solution):  $\lambda_{max}(\epsilon_{max})$  283 nm (1 618 dm $^3$  $\cdot$ mol $^{-1}$  $\cdot$ cm $^{-1}$ ).

### 1.3 Single crystal X-ray structure determination and refinement

Crystals suitable for X-ray diffraction analysis of compounds **2** and **4** were grown from their saturated *n*-hexane solution mounted in an inert oil and then

transferred to the cold gas stream of the diffractometer. The crystallographic data for compounds **2** and **4** was collected on a AFC10/Saturn724 + detector system with graphite-monochromated Mo  $K\alpha$  radiation ( $\lambda=0.071\ 073$  nm). The structures were solved by direct methods<sup>[13]</sup> and refined against  $F^2$  using SHELXL-97<sup>[14]</sup>. All non-hydrogen atoms were located by difference Fourier synthesis and refined anisotropically, and hydrogen atoms were included using the riding model with  $U_{iso}$  related to the  $U_{iso}$  of the parent atoms. The crystal data and refinement details for compound **2** and **4** are listed in Table 1, and the selected bond lengths (nm) and angles ( $^\circ$ ) are given in Table 2.

CCDC: 896958, **2**; 896957, **4**.

Table 1 Crystallographic Data for Compounds **2** and **4**

	<b>2</b>	<b>4</b>
Empirical formula	$C_{34}H_{58}N_2Si_2$	$C_{32}H_{54}N_2Si_2$
Formula weight	551.00	522.95
Crystal system	monoclinic	triclinic
Space group	$P2_1/n$	$P\bar{1}$
<i>a</i> / nm	0.958 5(3)	0.883 7(2)
<i>b</i> / nm	1.811 8(5)	0.967 5(2)
<i>c</i> / nm	0.982 4(3)	1.142 9(4)
$\alpha$ / ( $^\circ$ )		73.699(15)
$\beta$ / ( $^\circ$ )	102.173(3)	88.94(2)
$\gamma$ / ( $^\circ$ )		63.876(14)
<i>V</i> / nm $^3$	1667.7(8)	835.8(4)
<i>Z</i>	2	1
<i>D<sub>c</sub></i> / (g $\cdot$ cm $^{-3}$ )	1.097	1.039
$\mu$ / mm $^{-1}$	0.130	0.127
<i>F</i> (000)	608	288
$\theta$ range / ( $^\circ$ )	2.40 to 25.00	2.64 to 25.00
Index ranges	$-11 \leq h \leq 10$ $-11 \leq l \leq 11$ $-11 \leq k \leq 11$	$-21 \leq k \leq 18$ $-10 \leq h \leq 10$ $-13 \leq l \leq 12$
Reflections collected	9 640	6 876
Independent reflections $R_{int}$	2 900 (0.0305)	2 929(0.0812)
Data/restraints/parameters	2 900/0/180	2 929/0/170
GOF/ $F^2$	1.096	0.902
$R_1^a, wR_2^b(I > 2\sigma(I))$	0.037 5, 0.096 0	0.050 5, 0.119 9
$R_1^a, wR_2^b(\text{all data})$	0.044 6, 0.100 4	0.069 9, 0.125 8

Table 2 Selected bond distances (nm) and angles ( $^{\circ}$ ) for compound **2** and **4**

<b>2</b>					
Si(1)-N(1)	0.175 75(13)	N(1)-C(13)	0.145 73(18)	C(13)-C(13i)	0.135 2(3)
C(13)-C(14)	0.150 2(2)	Si(1)-C(15)	0.186 17(16)	Si(1)-C(17)	0.187 12(18)
Si(1)-C(16)	0.186 51(17)				
C(13)-N(1)-C(1)	116.27(11)	C(13)-N(1)-Si(1)	120.78(9)	C(1)-N(1)-Si(1)	119.95(9)
C(13)-C(13)-N(1)	122.20(16)	C(13)-C(13)-C(14)	124.30(17)	N(1)-C(13)-C(14)	113.47(12)
<b>4</b>					
Si(1)-N(1)	0.174 2(2)	N(1)-C(13)	0.140 9(3)	C(13)-C(13i)	0.133 7(4)
Si(1)-C(14)	0.183 4(3)	Si(1)-C(15)	0.183 0(3)	Si(1)-C(16)	0.183 7(3)
C(13)-N(1)-C(1)	116.43(18)	C(13)-N(1)-Si(1)	120.88(15)	C(1)-N(1)-Si(1)	122.66(15)
C(13)-C(13)-N(1)	126.4(3)				

## 2 Results and discussion

### 2.1 Chemistry and $^1\text{H}$ NMR of complex **2** and **4**

The crystalline sodium  $\alpha$ -diketiminate  $\text{LNa}_2$  (**1**) and lithium  $\alpha$ -diketiminate  $\text{LLi}_2$  (**3**) were obtained as precipitate by the metal reduction methods between L with sodium and L' with lithium in THF, respectively. Compound **2** was prepared by the sodium salt elimination of chloro(trimethyl)silane with disodium salt  $\text{LNa}_2$  (Scheme 1). Compound **4** was prepared by reaction of  $\text{LLi}_2$  with chloro(trimethyl)silane.

Compound **2** and **4** were characterized by  $^1\text{H}$  NMR investigation in  $\text{C}_6\text{D}_6$  solution. The  $^1\text{H}$  NMR

spectra of **2** and **4** exhibit one set of resonances for the aryl groups both on nitrogen and on the ligand, indicating a symmetric arrangement. Compound **2** shows the  $\text{CH}(\text{CH}_3)_2$  resonances at  $\delta$  3.59 ppm in a 2:3 ratio to that of the  $\text{CCH}_3$  at  $\delta$  2.10 ppm. Compound **4** exhibits one resonance for the  $\text{CH}=\text{CH}$  proton around  $\delta$  5.18 ppm, and  $\text{CH}(\text{CH}_3)_2$  protons around  $\delta$  3.58 ppm with the intensity of 1:2.

### 2.2 Crystal structure of the crystalline complex **2**

Colorless single crystals of **2** were obtained from saturated *n*-hexane solution at  $-15$   $^{\circ}\text{C}$ . Compound **2** crystallizes in the monoclinic space group  $P2_1/n$ . The molecular structure of crystalline **2** is illustrated in

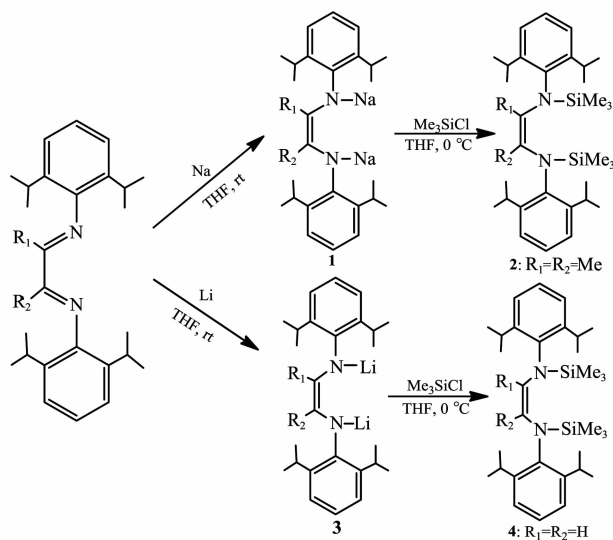
Scheme 1 Preparation of compound **2** and **4**

Fig.1 and selected geometrical parameters are listed in Table 2.

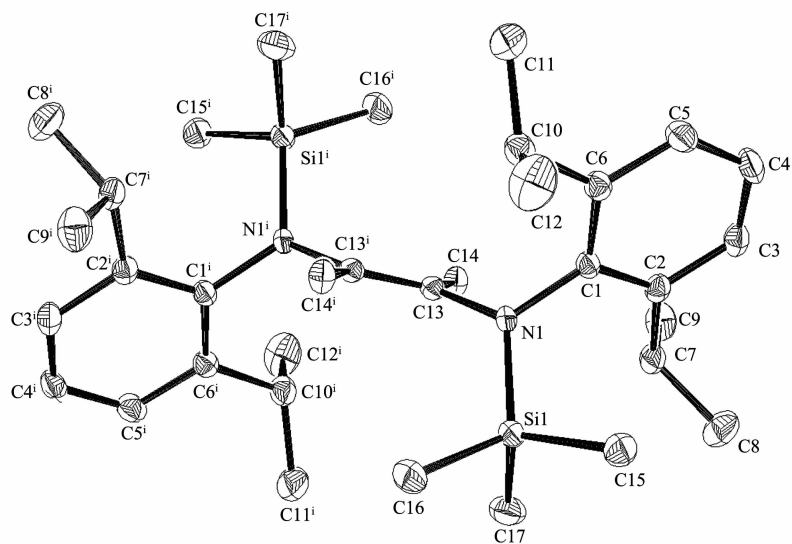
In the structure of compound **2**, a zig-zag arrangement of the atoms N(1), C(13), C(13<sup>i</sup>), and N(1<sup>i</sup>) is the core with mutually trans-methyl substituents at C(13) and C(13<sup>i</sup>) and the Si<sub>i</sub>Me<sub>3</sub> group cisoid to the latter. The C=N and C=C bond lengths are consistent with C(13)-C(13<sup>i</sup>)(0.135 2(3) nm) being double bonds, N(1)-C(13) and N(1<sup>i</sup>)-C(13<sup>i</sup>) (0.145 73(18) nm) being single bonds. The sum of the angles subtended at each of the tri-substituted atoms N(1), C(13), C(13<sup>i</sup>), and N(1<sup>i</sup>) is nearly 360°. The Si(1)-N(1)(0.175 75(13) nm) bonds are much shorter than the Si-N bond length (0.177 88(17) nm) of Si(L)Me<sub>3</sub>(L={N(Ar)C(Me)}<sub>2</sub>CH, Ar=2, 6-<sup>i</sup>Pr<sub>2</sub>C<sub>6</sub>H<sub>3</sub>)<sup>[7]</sup>, showing stronger interaction between SiMe<sub>3</sub> and N-aryl substituted  $\alpha$ -diimine ligand than that of SiMe<sub>3</sub> and  $\beta$ -diiminates because of the steric effect.

### 2.3 Crystal structure of the crystalline complex **4**

Extraction with *n*-hexane followed by crystallization at -15 °C afforded compound **4** as

colorless crystals. Compound **4** crystallizes in the monoclinic space group  $P\bar{1}$ . The molecular structure of the crystalline complex **4** is shown in Fig.2 and selected values for bond lengths and angles are given in Table 2.

From the structure of compound **4**, the  $\alpha$ -diamide ligand adopts a trans-stereochemistry and bridges two terminal SiMe<sub>3</sub> fragments. Similar to compound **2**, a zig-zag arrangement of the atoms N(1), C(13), C(13<sub>i</sub>), and N(1<sub>i</sub>) is the core of the molecular structure. The C=N and C=C bond lengths are consistent with C(13)-C(13<sub>i</sub>)(0.1337(4) nm) being double and N(1)-C(13) and N(1<sub>i</sub>)-C(13<sub>i</sub>) (0.140 9(3) nm) being single bonds. The sum of the angles subtended at each of the three-coordinate atoms N(1) and N(1<sub>i</sub>) is nearly 360°. The Si(1)-N(1)(0.174 2(2) nm) bond length is much shorter than the Si-N bond length (0.17575(3) nm) of compound **2**, showing that the steric effect and electronic effect of methyl groups at the carbon atoms is obvious.



Thermal ellipsoids are drawn at 50% probability level and the hydrogen atoms are omitted for clarity; Symmetry codes: <sup>i</sup> -x+1, -y+1, -z+1

Fig.1 Molecular structure of compound **2**

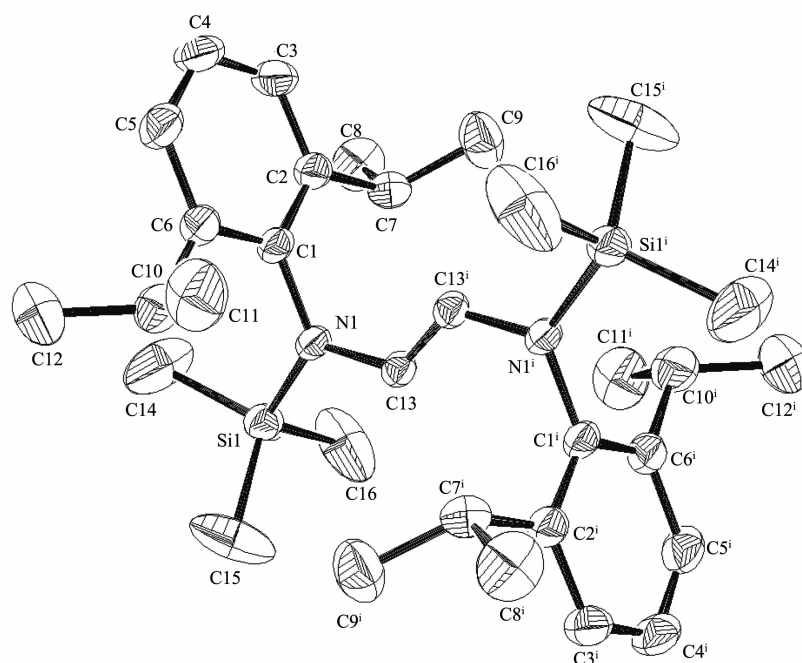
### 2.4 FTIR spectra

The FTIR spectra of complex **2** and **4** in 4 000~400 cm<sup>-1</sup> region is given in Table 3. The two absorption bands of 1 660, 1 650 cm<sup>-1</sup> for complex **2**, 1 666 and 1 625 cm<sup>-1</sup> for complex **4** can be assigned to the stretching of C=C bond. This result is consistent with

the structure determined by X-ray structural analysis. For both complex **2** and **4**, series of very strong absorptions ranged from 890 to 700 cm<sup>-1</sup> are signed to a combination of C-Si bond stretching vibrations.

### 2.5 UV-Vis and fluorescent spectra properties

Complex **2** and **4** are intense purple and UV-Vis



Thermal ellipsoids are drawn at 50% probability level and the hydrogen atoms are omitted for clarity; Symmetry codes:  $i -x+1, -y+1, -z+1$

Fig.2 Molecular structure of compound 4

Table 3 Main IR bonds ( $\text{cm}^{-1}$ ) for Compound 2 and 4

Compound	$\nu(\text{Ar-H, =C-H})$	$\nu(\text{CH}_3)$	$\nu(\text{=C-C})$	benzene ring skeleton	$\nu(\text{Si-C})$
2	3060, 3020	2960, 2870	1660, 1650	1580, 1460	838, 806, 769
4	3054, 3010	2970, 2865	1666, 2865	1579, 1463	830, 775, 754

spectra of both show one band in the ultraviolet region,  $\lambda_{\text{max}}$  281 nm for **2** and  $\lambda_{\text{max}}$  283 nm for **4**, respectively. According to the study of Zark and his coworkers<sup>[6]</sup>, we assume that the chromaticity of the  $\text{SiMe}_3$  group is solely due to the N-aryl substituted moiety.

The luminescent property of compound **2** and **4**

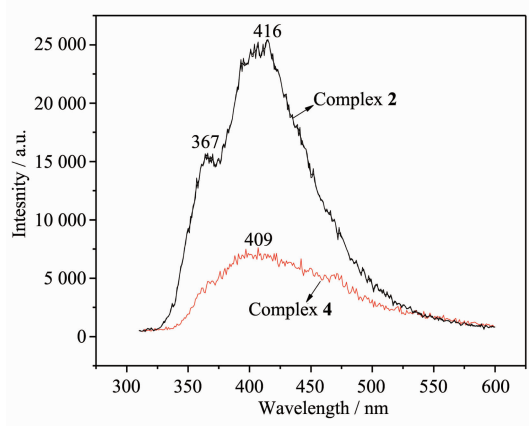


Fig.3 Emission spectroscopy of compound **2** and **4** with excitation at 300 nm

have been studied at room temperature (Fig.3). Excited by 300 nm, the emission maxima peak of complex **2** is at 416 nm plus shoulder peak at 367 nm. And the photoluminescence spectra of complex **4** shows the emission maxima at 409 nm. These emissions may be assigned to  $\pi^*-n$  or  $\pi^*-\pi$  transitions of the N-aryl substituted ligands<sup>[15-16]</sup>. The fluorescent intensity of complex **2** is much stronger than complex **4**, which may be caused by the steric effect and electronic effect of methyl groups on the NCCN framework. For possessing strong fluorescent intensity, they appear to be good candidates for novel potential organic-inorganic photoactive materials.

### 3 Conclusions

In summary, we report on the synthesis and structures of two silicon (IV) compounds with redox noninnocent  $\alpha$ -diimine ligands. Using different substituents on the ligands, reducing agents, resulted in the consistent structures. The difference of Si-N

bond length shows that the steric effect of methyl groups at the carbon atoms is obvious.

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