

# Synthesis of polysiloxane-supported ferrocenyl selenoether platinum complexes and their catalytic behavior in the hydrosilylation of olefins with triethoxysilane

Jun-Zhi Yao, Yuan-Yin Chen<sup>\*</sup>, Bing-Shou Tian

*Department of Chemistry, Wuhan University, Wuhan 430072, People's Republic of China*

Received 14 August 1996; revised 16 October 1996

## Abstract

Polysiloxane-supported ferrocenyl selenoether platinum complex was prepared from 1,1'-bis(undecenylseleno)ferrocene via hydrosilylation with triethoxysilane, followed by immobilization on fumed silica and then reaction with potassium chloro-platinate. Fumed silica-bound (undecenylseleno)- or 1,1'-bis(undecenylseleno)ferrocene platinum complexes were also synthesized from triethoxysilane and (undecenylseleno)- or 1,1'-bis(undecenylseleno)ferrocene platinum complex directly. The platinum complexes of ferrocenyl selenoether and polymer-supported ferrocenyl selenoether were efficient catalysts for hydrosilylation of olefins with triethoxysilane. The catalytic activity of polysiloxane-supported platinum complexes (prepared by the method of immobilization after coordination) was similar to those of the corresponding ferrocenyl selenoether platinum complexes and higher than those of catalysts prepared by the method of coordination after immobilization.

*Keywords:* Ferrocenyl selenoether; Polysiloxane; Platinum complex; Hydrosilylation; Supported catalysts

## 1. Introduction

Polymer-supported metal complexes have the advantages of high activity and selectivity of homogeneous catalysts and ease of separation of heterogeneous catalysts. These 'third generation' catalysts [1,2] have received much attention [3–11], and several reviews on the area are available [1,12,13]. In the preparation of supported catalysts a wide variety of support materials have been used including cross-linked polymers [14,15], silica [4], and high surface area glasses [16]. Inorganic supports possess a rigid structure which is not deformed by solvent swelling during catalytic reactions. Fumed silica is a better support because they have large surface area, high mechanical strength, heat and chemical stability.

The chemistry of ferrocene and its derivatives has attracted much interest, mainly because of their stability and unusual reactivity. Ferrocenyl selenoethers are among the interesting derivatives of ferrocene [17]. It has been used to prepare Pd, Pt complexes that are

catalytically active for hydrogenation, isomerization, Grignard cross-coupling and hydrosilylation [17–19]. Thus it is interesting to immobilize these complexes. No paper concerned with supporting them has been reported until now. Cullen and coworkers [20] have prepared cross-linked polystyrene-supported ferrocene derivatives from lithioferrocenes  $\text{Fe}(\eta^5\text{-C}_5\text{H}_4\text{Li})(\eta^5\text{-C}_5\text{H}_5)$ ,  $\text{Fe}(\eta^5\text{-C}_5\text{H}_4\text{Li})_2$ ,  $\text{Fe}[\eta^5\text{-C}_5\text{H}_3(\text{CHMeNMe}_2)\text{Li-1,2}][\eta^5\text{-C}_5\text{H}_5]$ , and lithium ferrocenylphosphines.

The catalytic activity and selectivity of polymer-supported complexes are dependent on their intrinsic structures, which is related to the method of preparation. We have used two different methods to prepare silica-supported ferrocenyl selenoether platinum complexes. The hydrosilylation catalytic properties of the polymer-supported complexes were investigated. Since symmetrically 1,1'-bis(undecenylseleno)ferrocene is a rigid chelating ligand, the structure around the metal center of the polymer-supported platinum complex may be similar to the 1,1'-bis(undecenylseleno)ferrocene platinum complex. It is possible for us to obtain more information about the influences of immobilization. So we investigated the catalytic behavior of both the plat-

<sup>\*</sup> Corresponding author.

inimum complexes of ferrocenyl selenoethers and polymer-supported ferrocenyl selenoethers. The conclusions may be applicable to other silica-attached catalytic systems prepared by using linking agents.

## 2. Experimental

Infrared spectra, nuclear magnetic resonance spectra, mass spectra, and X-ray photoelectron energy spectra were obtained on a PE-983 IR spectrometer, a Bruker ARX-300 NMR spectrometer, a ZAB HF-HF mass spectrometer and a KRATOS XSAM 800 electron energy spectrometer respectively. Elemental analysis was performed on a MOD 1106 elemental auto-analyzer. GLC was carried out on a 102G model chromatograph using a thermal conductivity detector, SE-30 stationary phase and silylanized 101 white matrix.

Air-sensitive reagents were manipulated in a pre-purified argon atmosphere by using standard Schlenk techniques. All solvents were carefully dried and freshly distilled under nitrogen before use.

Undecenyl diselenide was prepared according to Syper and Mlochowski's procedure [21], 92% yield. MS:  $m/e$  466 ( $M^+$ ), 233 ( $CH_2=CH(CH_2)_9Se^+$ ), 149, 135, 109, 95, 81, 69, 55, 41.  $^1H$  NMR ( $CCl_4$ , TMS)  $\delta$  (ppm): 1.23–1.99 (m, 32 H), 2.66–2.88 (t, 4 H), 4.67, 4.87, 4.95 (m, 4 H), 5.34–5.97 (m, 2 H). IR ( $cm^{-1}$ ): 3075 ( $=C < H$ ), 2926 ( $-CH_2-$ ), 2853 ( $-CH_2-$ ), 1640 ( $> C=C <$ ), 1460 ( $-CH_2-$ ), 992, 909, 720.

### 2.1. Synthesis of 1,1'-bis(undecenylseleno)ferrocene (1) and (undecenylseleno)ferrocene (2)

Ferrocene (4.65 g, 25 mmol) was added to a 500 ml round-bottomed flask equipped with a side-arm and a magnetic stirrer. After the neck was sealed with a rubber septum, the flask was evacuated and refilled with argon. A solution of *n*-butyllithium in hexane (26 ml, 1.93 M, 50 mmol) was added to the flask via a cannula. The suspension was rapidly stirred and freshly distilled *N,N,N',N'*-tetramethylethylenediamine (TMEDA) (5.8 g, 50 mmol) was added dropwise. The reaction mixture was stirred for 3 h at room temperature, and then a solution of undecenyl diselenide (23.3 g, 50 mmol) in 100 ml of benzene was added dropwise at  $-40^\circ C$ . The mixture was then stirred for 12 h at room temperature and filtered. The filtrate was washed with  $H_2O$ , and the organic layer was separated and evaporated to give a red-brown oil. The oil was separated on a silica gel column with petroleum ether ( $30-60^\circ C$ ) as the eluent to give a red-brown oil (2.7 g, monosubstituted compound 2) and an orange-yellow solid (9.4 g, disubstituted compound 1, m.p.  $39-40^\circ C$ ). 2:  $^1H$  NMR  $\delta$  (ppm): 1.26–1.34 (m, 12 H), 1.54–1.62 (m, 2 H), 1.99–2.06 (q, 2 H), 2.55–2.59 (t, 2 H), 4.18, 4.28 (d, 9 H),

4.91–5.02 (m, 2 H), 5.73–5.87 (m, 1 H). MS:  $m/e$  418 ( $M^+$ ), 322 ( $M - C_7H_{12}$ ) $^+$ , 265 ( $C_5H_5FeC_5H_4Se$ ) $^+$ . 1:  $^1H$  NMR  $\delta$  (ppm): 1.27–1.35 (m, 24 H), 1.53–1.62 (m, 4 H), 1.98–2.08 (q, 4 H), 2.56–2.61 (t, 4 H), 4.21, 4.31 (d, 8 H), 4.92–5.00 (m, 4 H), 5.73–5.87 (m, 2 H). MS:  $m/e$  650 ( $M^+$ ), 554 ( $M - C_7H_{12}$ ) $^+$ , 342 ( $Fe(C_5H_4Se)_2$ ) $^+$ . Anal. Found: C, 59.06; H, 7.88.  $C_{32}H_{50}Se_2Fe$  Calc.: C, 59.27; H, 7.77%.

### 2.2. Synthesis of dichloro[1,1'-bis(undecenylseleno)ferrocene]platinum(II) (3)

A mixture of 1,1'-bis(undecenylseleno)ferrocene (0.713 g, 1.1 mmol) and potassium chloro-platinite (0.415 g, 1.0 mmol) in 20 ml acetone was stirred at  $65^\circ C$  for 36 h under nitrogen. The yellow precipitate formed was collected by filtration, washed with distilled water, benzene and petroleum ether, and dried in vacuum. The product was purified by column chromatography on silica gel with dichloromethane as the eluent. MS (FAB):  $m/e$  916 ( $M + 1$ ) $^+$ , 879 ( $M - HCl$ ) $^+$ , 843 ( $M - 2 HCl$ ) $^+$ , 789 ( $M - C_9H_{18}$ ) $^+$ , 650 ( $M - PtCl_2$ ) $^+$ . Anal. Found: C, 41.86; H, 5.45; Cl, 8.01.  $C_{32}H_{50}Cl_2Se_2FePt$  Calc.: C, 42.03; H, 5.51; Cl, 7.76%.

### 2.3. Synthesis of dichloro[(undecenylseleno)ferrocene]platinum(II) (4)

A mixture of (undecenylseleno)ferrocene (0.417 g, 1.00 mmol) and potassium chloro-platinite (0.436 g, 1.05 mmol) in 20 ml acetone was stirred at  $65^\circ C$  for 36 h under nitrogen. The brown-yellow precipitate formed was collected by filtration, washed with distilled water, and dried in vacuum. The product was purified by column chromatography on silica gel with dichloromethane as the eluent. MS (FAB):  $m/e$  418 ( $M - PtCl_2$ ) $^+$ , 265 ( $PtCl_2$ ) $^-$ . Anal. Found: C, 36.86; H, 4.35; Cl, 10.00.  $C_{21}H_{30}Cl_2SeFePt$  Calc.: C, 36.92; H, 4.42; Cl, 10.38%.

### 2.4. Synthesis of silica-bound 1,1'-bis(undecenylseleno)ferrocene platinum complex (5), coordination after immobilization

A mixture of 1,1'-bis(undecenylseleno)ferrocene (0.40 g, 0.62 mmol), triethoxysilane (4.9 g, 30 mmol), tetra(triphenylphosphine)platinum complex (2.0 mg,  $1.6 \times 10^{-3}$  mmol) was stirred at  $90^\circ C$  for 60 h under nitrogen atmosphere. The cooled mixture was passed through a sinter glass filter in which an alumina layer 2 cm thick was packed. The alumina was washed three times with absolute ether. The filtrate and washings were combined and then concentrated. No peak of proton attached to the double bond (4.8–5.8 ppm) was found and a new peak around 0.5 ppm due to the Si- $CH_2$  proton appeared in the NMR spectra of the residue. The residue

was added to a suspension of 0.5 g of fumed silica ( $150\text{--}200\text{ m}^2\text{ g}^{-1}$ ) in 10 ml toluene, stirred at  $100^\circ\text{C}$  for 10 h, and then an additional 5 h after addition of 5 ml of distilled water. The solid was filtered, washed three times with acetone, dried under nitrogen at  $90^\circ\text{C}/26.7\text{ Pa}$ , then transferred to a Soxhlet apparatus and extracted with acetone for 24 h. After drying at  $90^\circ\text{C}/26.7\text{ Pa}$  for 4 h, the product was ground to powder. Silica-bound 1,1'-bis(undecenylseleno)ferrocene was obtained as a black–brown powder. Anal. Found: Fe, 1.15%.

A mixture of silica-bound 1,1'-bis(undecenylseleno)ferrocene (391 mg),  $\text{K}_2\text{PtCl}_4$  (37 mg, 0.089 mmol) and acetone (50 ml) was stirred under argon at reflux temperature for 72 h. The solid product was filtered by suction, washed with acetone, distilled water and acetone successively and dried at  $90^\circ\text{C}/26.7\text{ Pa}$  under argon for 3 h. **5** was obtained as a black–brown powder. Anal. Found: Pt, 2.01; Fe, 1.12%.

#### 2.5. Synthesis of silica-bound 1,1'-bis(undecenylseleno)ferrocene platinum complex (**6**), immobilization after coordination

Dichloro[1,1'-bis(undecenylseleno)ferrocene]platinum(II) (**3**) (17.2 mg, 0.019 mmol) was allowed to react with triethoxysilane (1.7 g, 10.4 mmol) at  $80^\circ\text{C}$  for 10 h under nitrogen atmosphere. Distillation under reduced pressure to remove extra triethoxysilane provided 110 mg of brown–yellow oil. The disappearance of a specific peak of the carbon–carbon double bond ( $1642\text{ cm}^{-1}$ ) indicated that the carbon–carbon double bond had reacted.

Toluene (4 ml) and fumed silica (100 mg) were added to the above resulting oil. The mixture was stirred at  $90^\circ\text{C}$  for 45 h under nitrogen atmosphere, and then an additional 5 h after addition of distilled water. The cooled mixture was filtered by suction, washed with acetone and dichloromethane, dried at  $90^\circ\text{C}/26.7\text{ Pa}$  under argon for 3 h. **6** was obtained as a grey powder. Anal. Found: Pt, 3.63; Fe, 1.10%.

#### 2.6. Synthesis of silica-bound (undecenylseleno)ferrocene platinum complex (**7**), immobilization after coordination

Dichloro(undecenylselenoferrocene)platinum(II) (**4**) (11.5 mg, 0.0168 mmol) was allowed to react with triethoxysilane (0.8 g, 4.9 mmol) at  $80^\circ\text{C}$  for 12 h under nitrogen atmosphere. The reaction mixture was distilled under reduced pressure to remove extra triethoxysilane to give 139 mg of dark brown–yellow oil. Toluene (4 ml) and fumed silica (100 mg) were added to the above resulting oil, and the mixture was stirred at  $80^\circ\text{C}$  for 8 h under nitrogen atmosphere, and then an additional 5 h after addition of 0.5 ml of distilled water. The

cooled mixture was filtered by suction, washed with acetone and dichloromethane, and dried at  $90^\circ\text{C}/26.7\text{ Pa}$  under argon for 3 h. Silica-bound (undecenylseleno)ferrocene platinum complex (**7**) was obtained as a grey powder. Anal. Found: Pt, 0.79; Fe, 0.28%.

#### 2.7. Hydrosilylation of olefins

Hydrosilylation was carried out in a 5 ml plane-bottomed flask equipped with a magnetic stirrer and a reflux condenser to the upper of which a drying system was attached. Olefin and platinum complex were stirred at the reaction temperature for 0.5 h before triethoxysilane was added. The structure and yield of hydrosilylation products were determined based on a standard sample and a standard curve by GLC at regular intervals. Typical reaction conditions are as follows: olefin 5.0 mmol, triethoxysilane 5.0 mmol, platinum complex  $10^{-3}\text{--}10^{-4}$  mmol Pt.

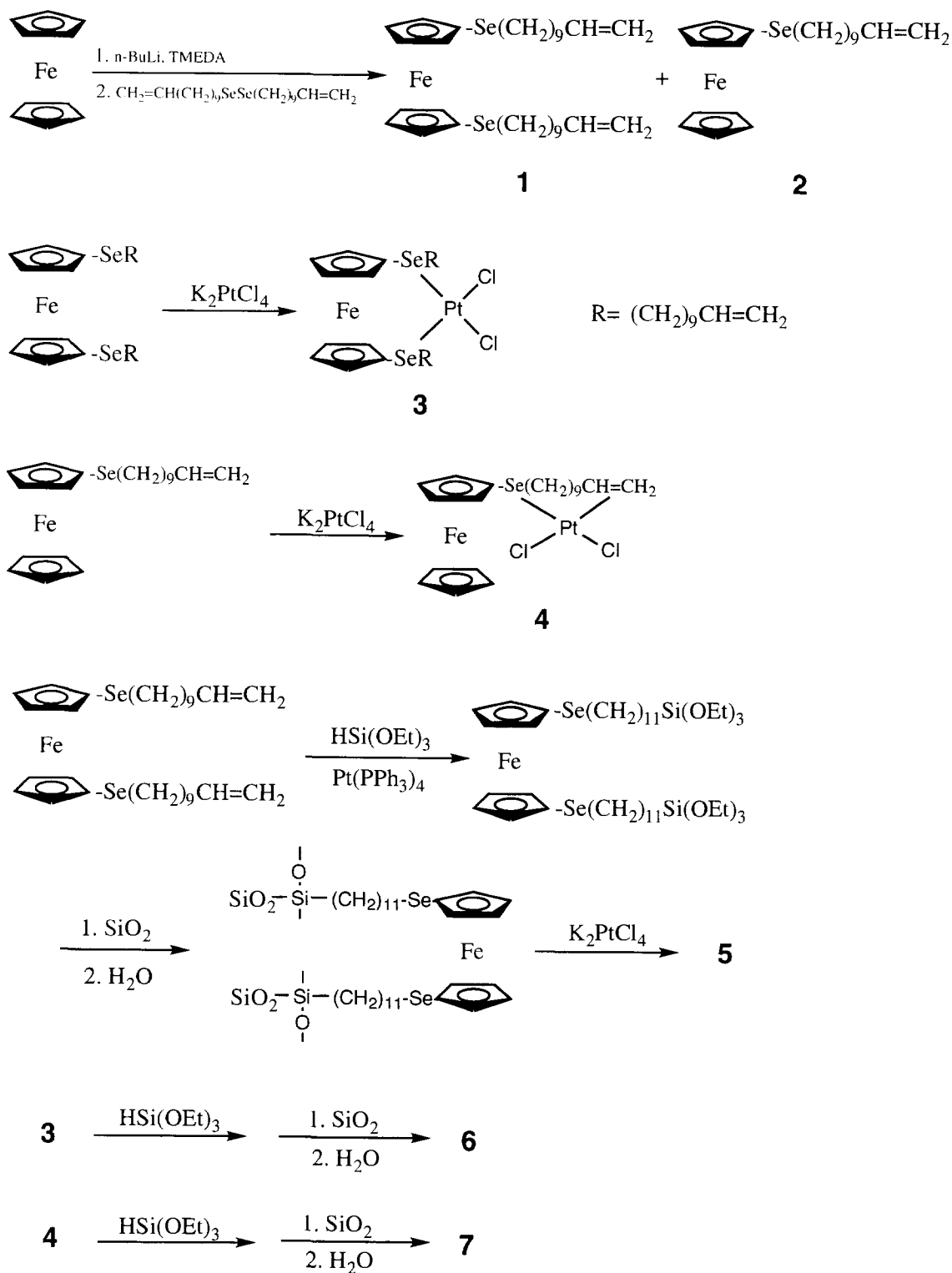
### 3. Results and discussion

#### 3.1. Preparation and characterization of metal complexes

The following synthetic route is adopted: (a) preparation of 1,1'-bis(undecenylseleno)ferrocene and 1-mono(undecenylseleno)ferrocene; (b) hydrosilylation of undecenylseleno- or 1,1'-bis(undecenylseleno)ferrocene with triethoxysilane; (c) immobilization on fumed silica and then reaction with potassium chloro-platinite. Fumed silica-bound (undecenylseleno)- or 1,1'-bis(undecenylseleno)ferrocene platinum complexes were also synthesized from triethoxysilane and (undecenylseleno)- or 1,1'-bis(undecenylseleno)ferrocene platinum complex directly without addition of another platinum catalyst. The synthetic scheme is shown in Scheme 1.

The results of elemental analysis of **3** and **4** indicated that **1** or **2** coordinates with platinum in the molar ratio 1:1. The IR spectrum of **4** showed no bands assignable to  $\nu(\text{C}=\text{C})$  in the region around  $1640\text{ cm}^{-1}$ , indicating that the double bond had participated in coordination. On the other hand, the IR spectrum of **3** showed an IR band at  $1640\text{ cm}^{-1}$  assignable to  $\nu(\text{C}=\text{C})$  which is close to that observed for **1**. These data indicate that the double bond had not participated in coordination. The  $^1\text{H}$  NMR spectra of the above mentioned compounds confirmed these results. The chemical shifts for the vinyl protons at 5.80 (m), 5.01 (m), 4.92 (m) ppm for the platinum complex **3** are almost identical to those of **1**. However, the chemical shifts for the vinyl protons changed from 5.80 (m), 5.01 (m), 4.92 (m) ppm for **2** to 4.87–5.29 ppm (broad multiplet) for **4**.

The new complexes were also characterized by XPS. Table 1 lists the XPS data of **1**, **3**, **5** and **6**. A compari-



Scheme 1.

son of the binding energy of the elements in ligand and platinum complexes showed that the binding energy of  $\text{Pt}_{4f7/2}$  is 0.30–0.4 eV less than that in  $\text{K}_2\text{PtCl}_4$ , but 1.4–1.5 eV larger than that in Pt foil; the  $\text{Se}_{3d}$  binding energy increased by 0.3–0.5 eV after coordination.

These results indicate that complex formation caused the electron pair transfer from selenium to platinum. The difference of  $\text{Cl}_{2p}$  binding energy can also be explained as the result of the formation of platinum complex, or coordination of selenium to platinum atom.

Table 1  
The binding energy of platinum complexes

Sample	Binding energy (eV)		
	Pt <sub>4f7/2</sub>	Sc <sub>3d</sub>	Cl <sub>2p</sub>
Pt foil	71.3		
K <sub>2</sub> PtCl <sub>4</sub>	73.1		198.9
<b>1</b>		56.0	
<b>3</b>	72.8	56.4	198.6
<b>5</b>	72.7	56.5	198.4
<b>6</b>	72.7	56.3	198.5

### 3.2. The catalytic behavior of **3**

Allyl phenyl ether, 1-decene, and 1-dodecene were used as substrates to test the catalytic activity and selectivity of **3**. Decene, allyl phenyl ether and dodecene were hydrosilylated with HSi(OEt)<sub>3</sub> to give the saturated products in 87%, 83%, and 76% respectively. Speier's catalyst was not so effective when HSi(OEt)<sub>3</sub> was used. It was reported that the yield of decyltriethoxysilane was only 40% in the case of using H<sub>2</sub>PtCl<sub>6</sub> as catalyst [22].

The catalytic activity of the platinum complex at different temperatures was also investigated using the hydrosilylation of 1-decene with triethoxysilane as the model reaction. The experimental results show that no remarkable induction period was observed, and the reac-

tion rate became faster with the increase of the temperature. In the range 70 to 110 °C, the catalytic reaction finished within 40 min.

### 3.3. The catalytic behavior of **5** and **6**

The hydrosilylation of 1-decene with triethoxysilane was examined to investigate the catalytic activity of **5** and **6** at different temperatures. For **5**, the yield of decyltriethoxysilane varied little in the reaction temperature range from 90 to 110 °C. At 70 °C, the supported complex had a long induction period (about 60 min) and the yield of decyltriethoxysilane was low. For **6**, the reaction rate became faster; the induction period became shorter; the yield of decyltriethoxysilane became higher with the increase of temperature. The catalytic activity of **6** prepared by the method of immobilization after coordination was higher than that of **5** prepared by the method of coordination after immobilization. The yield of decyltriethoxysilane catalyzed by **6** at 110 °C was similar to that by **3** at 90 °C. The results are shown in Fig. 1.

The hydrosilylation of 1-decene with triethoxysilane was examined to evaluate the reusable property of **5**. It was demonstrated that the supported platinum complex could be recovered and reused several times. However, under identical conditions the yield of decyltriethoxysi-

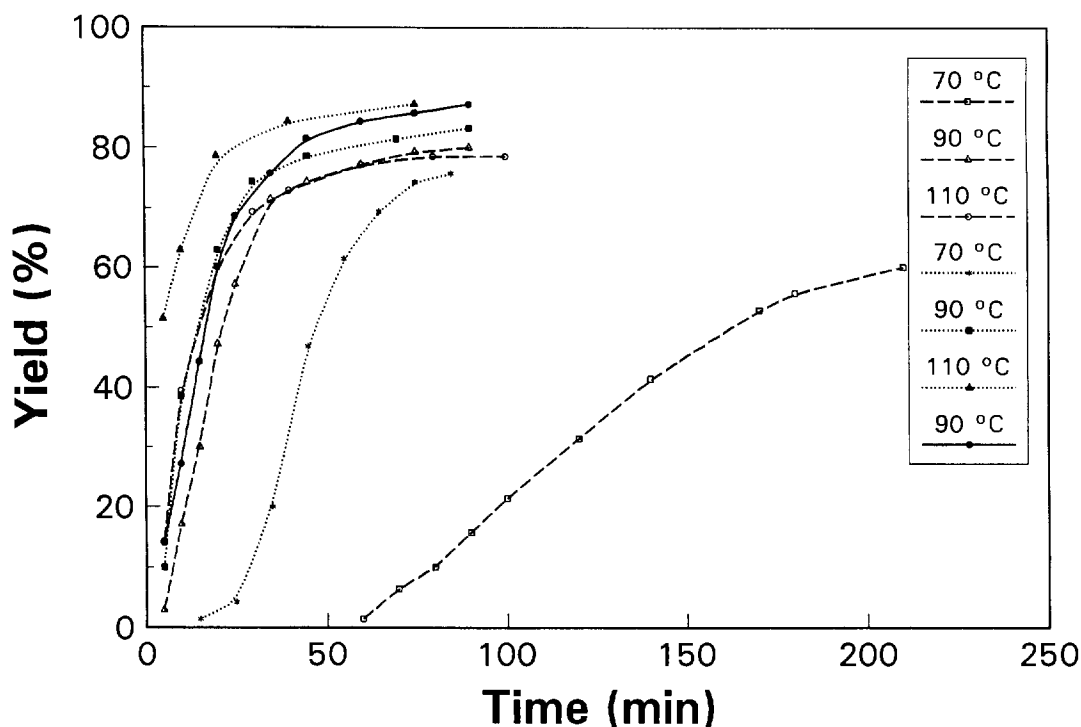


Fig. 1. Plot of the yield of decyltriethoxysilane vs. time with **5** (---), **6** (···) and **3** (---) as catalysts. Conditions: 1-decene 5.0 mmol, HSi(OEt)<sub>3</sub> 5.0 mmol, catalyst  $5.0 \times 10^{-4}$  mmol Pt.

lane decreased with increasing runs. The yields of decyltriethoxysilane from the first to the 50th run were 80%, 74%, 67%, 55% and 42%. The most likely reason for the reduction of activity is the leaching of platinum from the support. The supported platinum complex **6** had better reusable properties than **5**. The yields of decyltriethoxysilane from the first to the 50th run were 82%, 79%, 75%, 78% and 76%. The better reusable properties of **6** may be due to the fact that Pt is chelated with Se in **6**, whereas the structure of **5** is less defined, and probably some of them are not chelated.

### 3.4. The catalytic behavior of **4** and **7**

Triethoxysilane was respectively added to allyl phenyl ether, 1-decene, and 1-dodecene to explore the catalytic activity and selectivity of **4**. The yields of the adducts of allyl phenyl ether, decene and dodecene were 83%, 77%, and 76% respectively. The catalytic activity of **4** was similar to that of **3**, but the yield of decyltriethoxysilane catalyzed by **4** was 77% and that by **3** was 87%.

1-Decene was used as a probe to investigate the influence of temperature on catalytic activity of the monosubstituted ferrocenyl selenoether platinum complex **4** and its supported product **7**. The catalytic activity was characterized by the reaction rate of 1-decene with triethoxysilane and the yield of decyltriethoxysilane. For **4**, the reaction rate increased with the increase of temperature, but the final yields of decyltriethoxysilane were similar within the temperature range from 80 to 100°C, the final yields were 84%. For **7**, the reaction rates were distinctly different at 70 and 90°C: at 70°C, an induction period of about 25 min was observed, the final yield was 72%; at 90°C, no obvious induction period was observed, the final yield was 83%. The catalytic activity of **7** was similar to that of **4** when the reaction temperature was 90°C. Increasing the temperature, the yield as well as the reaction rate of decyltriethoxysilane increased. At 110°C the yield was 86%.

As expected, the reusable property of **7** was not good, since the platinum is coordinated with only one Se. The yields of decyltriethoxysilane from the first to the 50th run were 84%, 70%, 58%, 41% and 32%.

## Acknowledgements

The authors gratefully thank Dr. Guochen Jia for kindly revising the manuscript and giving valuable suggestions. This work was supported by the National Natural Science Foundation of China.

## References

- [1] F.R. Hartley and P.N. Vezey, *Adv. Organomet. Chem.*, **15** (1977) 189.
- [2] F.R. Hartley, in R. Ugo and B.R. James (eds.), *Supported Metal Complexes*, Reidel, Dordrecht, 1985.
- [3] K.G. Allure, R.D. Hancock, I.V. Howell, T.E. Lester, S. McKenzie, R.C. Pitkethly and P.J. Robinson, *J. Organomet. Chem.*, **107** (1976) 393.
- [4] K.G. Allum, R.D. Hancock, I.V. Howell, S. McKenzie, R.C. Pitkethly and P.J. Robinson, *J. Organomet. Chem.*, **87** (1975) 203.
- [5] J.P. Collman, J.A. Belmont and J.I. Brauman, *J. Am. Chem. Soc.*, **105** (1983) 7288.
- [6] M. Berglund, C. Andersson and R. Larsson, *J. Organomet. Chem.*, **258** (1983) 195.
- [7] D.K. Liu, M.S. Wrighton, D.R. McKay and G.E. Maciel, *Inorg. Chem.*, **23** (1984) 212.
- [8] R.D. Sanner, R.G. Austin, M.S. Wrighton, W.D. Honnick and C.U. Pittman, Jr., *Inorg. Chem.*, **18** (1979) 928.
- [9] M. Czaková and M. Capka, *J. Mol. Catal.*, **11** (1981) 313.
- [10] S.I. Woo and C.G. Hill, *J. Mol. Catal.*, **15** (1982) 309.
- [11] C.-P. Lau, B.-H. Chang, R.H. Grubbs and C.H. Brubaker, Jr., *J. Organomet. Chem.*, **214** (1981) 325.
- [12] D.C. Bailey and S.H. Langer, *Chem. Rev.*, **81** (1981) 109.
- [13] Y.I. Yermakov, B.N. Kuznetsov and V.A. Zakharov, *Catalysis by Supported Complexes*, Elsevier Scientific, Amsterdam, 1981.
- [14] J. Lieto, D. Milstein, R.L. Albright, J.V. Minkiewitz and B.C. Gates, *Chem. Tech.*, **13** (1983) 46.
- [15] C.U. Pittman, Jr. and R.F. Felis, *J. Organomet. Chem.*, **72** (1974) 389.
- [16] M.S. Ichikawa, *J. Chem. Soc., Chem. Commun.*, (1976) 26.
- [17] R.V. Honeychuck, M.O. Okoroafor, L.-H. Shen and C.H. Brubaker, Jr., *Organometallics*, **5** (1986) 482.
- [18] A.A. Naini, C.-K. Lai, D.L. Ward and C.H. Brubaker, Jr., *J. Organomet. Chem.*, **390** (1990) 73.
- [19] J.-Z. Yao, B.-S. Tian and Y.-Y. Chen, *Chin. Chem. Lett.*, **4** (1993) 601.
- [20] I.R. Butler, W.R. Cullen, N.F. Han, F.G. Herring, N.R. Jagannathan and J.-J. Ni, *Appl. Organomet. Chem.*, **2** (1988) 263.
- [21] L. Syper and J. Mlochowski, *Synthesis*, (1984) 439.
- [22] M. Capka and J. Hetflejš, *Collect. Czech. Chem. Commun.*, **39** (1974) 154.