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To cite this article: Ali Z Al-Rubaie *et al* 2021 *J. Phys.: Conf. Ser.* **2063** 012022

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# Synthesis of some new Rhodium complexes containing diaryl chalcogenide ligands and their uses as catalysts in hydrosilylation of alkenes

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**Abstract.** Diaryl Chalcogenides (*i.e.* Ar<sub>2</sub>E where Ar = 4-CH<sub>3</sub>C<sub>6</sub>H<sub>4</sub>, 4-BrC<sub>6</sub>H<sub>4</sub>, E= S, Se and Te) were reacted with [RhCl(CO)<sub>2</sub>]<sub>2</sub> and rhodium(III) chloride trihydrate to give compounds of type [RhCl(CO)<sub>2</sub>(Ar<sub>2</sub>E)] and [RhCl<sub>3</sub>(Ar<sub>2</sub>E)<sub>3</sub>], respectively. All compounds were characterized by IR, NMR, and mass spectroscopic data. Attempts to prepare hydroxyapatite (HAp) supported rhodium catalyst by using different methods were unsuccessful. Complexes [RhCl(CO)<sub>2</sub>((4-R-C<sub>6</sub>H<sub>4</sub>)<sub>2</sub>S)], [RhCl(CO)<sub>2</sub>((4-R-C<sub>6</sub>H<sub>4</sub>)<sub>2</sub>Se)] and [RhCl(CO)<sub>2</sub>((4-R-C<sub>6</sub>H<sub>4</sub>)<sub>2</sub>Te), where R= CH<sub>3</sub> or Br], were evaluated as catalysts for hydrosilylation of allyl phenyl ether and 1-decene. They showed good catalytic activities for hydrosilylation of alkenes with triethoxysilane.

## 1. Introduction

In recent years, due to their exceptional properties calcium apatites, Ca<sub>10</sub>(PO<sub>4</sub>)<sub>6</sub>(OH)<sub>2</sub> (HAp), has wide importance in the fields of biomedicine, dental implants, catalysis, and drug delivery [1-8]. Several papers described the preparation of heterogeneous catalysts from transition metal ions and HAp. It was found that Pd and Ru supported HAp are the suitable heterogeneous catalyst for oxidation, aldol reaction, and Diels-Alder reactions [9-11]. On the other hand, metal-organic coordination compounds have drawn great research interest in recent years because of their potential applications in catalysis [12]. It is worth noting that our previous work described the synthesis of some rhodium-tellurium heterocycle complexes [13]. Rhodium complexes containing organotellurium compounds, namely diphenyl telluride, and dimethyl telluride, as donors have also been previously reported [14-16]. Thus, it is therefore of interest to study rhodium(I) carbonyl and rhodium (III) complexes containing monodentate chalcogenide ligands since such complexes are very rare in literature and not used as catalysts, as far as we are aware. Rhodium(I) carbonyl complexes containing monodentate phosphine chalcogenides were reported and their catalytic activity for carbonylation of methanol was studied [17,18]. The present work describes the preparation of some new rhodium-chalcogenide complexes. Our recent work [19] described the catalytic activity of platinum(II) complexes of hexenyl aryl tellurides in the hydrosilylation of olefins promoted us to support these new complexes on hydroxyapatite to investigate their catalytic activity. Furthermore, the catalytic activity of these new complexes in the hydrosilylation of allyl phenyl ether and 1-decene will be examined at different temperatures.

## 2. Experimental

All the preparations were performed in a dry nitrogen atmosphere. All chemicals were reagent grade and used without further purification. Bis(4-methylphenyl) sulfide, bis(4-methylphenyl) selenide, bis(4-bromophenyl) sulfide and bis(4-bromophenyl) selenide were prepared by reported methods [19-21]. Bis(4-methylphenyl) telluride and bis(4-bromophenyl) telluride were prepared according to a literature



method [20–21].  $[\text{Rh}_2(\mu\text{-Cl})_2(\text{CO})_4]$  was prepared according to a literature method [22] by bubbling carbon monoxide through a refluxing ethanolic solution of rhodium trichloride trihydrate. All diaryl Chalcogenides (*i.e.*,  $\text{Ar}_2\text{E}$  where  $\text{Ar} = 4\text{-CH}_3\text{C}_6\text{H}_4$ ,  $4\text{-BrC}_6\text{H}_4$ ,  $\text{E} = \text{S}$ ,  $\text{Se}$ , and  $\text{Te}$ ) were characterized by spectroscopic methods and CHN analysis and are not reported.

### 2.1. Synthesis of $[\text{Rh}(\text{CO})_2\text{CIL}]$ complexes

Complexes **1–6** (*i.e.*  $[\text{Rh}(\text{CO})_2\text{CIL}]$ ,  $\text{L} = (4\text{-CH}_3\text{C}_6\text{H}_4)_2\text{E}$  and  $(4\text{-BrC}_6\text{H}_4)_2\text{E}$ , where  $\text{E} = \text{S}$ ,  $\text{Se}$  and  $\text{Te}$ ) were prepared by the following general method:

To a solution of  $[\text{Rh}(\text{CO})_2\text{Cl}]_2$  (150 mg, 0.65 mmol) in 30 mL of THF was added a solution of the respective ligand (1.35 mmol) in 20 mL of THF. The reaction mixture was stirred for 6 h at room temperature and the solvent was removed under vacuum by a rotatory evaporator. The remaining solid was washed with pentane (3 x 10 mL) and dried under vacuum over silica gel in a desiccator.

All these new complexes along with their melting points and elemental analyses, which were prepared by the above general route, are listed in Table 1.

#### 2.1.1. Synthesis of $[\text{RhCl}_3(\text{Ar}_2\text{E})_3]$ complexes

Complexes **7–12** (*i.e.*  $[\text{RhCl}_3(\text{Ar}_2\text{E})_3]$ , where  $\text{Ar} = (4\text{-CH}_3\text{C}_6\text{H}_4)_2\text{E}$  and  $(4\text{-BrC}_6\text{H}_4)_2\text{E}$ ,  $\text{E} = \text{S}$ ,  $\text{Se}$  and  $\text{Te}$ ) were prepared by the following method: Rhodium trichloride trihydrate (0.79 g, 3.0 mmol) in dry ethanol (50 mL) was added to an ethanolic solution of diaryl chalcogenide (9.2 mmol). The mixture was stirred at room temperature for 6 h. A brownish-red solid was obtained, collected by filtration, washed with ethanol, and dried *in vacua*. The rhodium(III)-diaryl chalcogenide complexes which were prepared by the above general route (along with their melting points and elemental analyses) are listed in Table 2.

#### 2.1.2. Attempts to prepare the Rh-complex/HAp catalysts.

The following methods were used to prepare Rh-complex/HAp catalysts:

a) To 0.20 mmol of the corresponding rhodium complex (*i.e.*  $[\text{Rh}(\text{CO})_2\text{CIL}]$ ) in 100 mL of acetone was added to 2.5 g of HAp. The light-yellow mixture was stirred at room temperature for 12 h and the liquid became colorless. The mixture was filtered. The solid product was collected by filtration and washed with several amounts of acetone and water, respectively. The product was dried under a vacuum. The obtained solid was grid into powders and divided into two parts which were then calcined in a muffle oven under static air at 300 and 500°C respectively, for 12 h.

b) Three rhodium concentration (10 mM, 20 mM, and 50 mM) solutions were prepared by dissolving  $[\text{Rh}(\text{CO})_2\text{CIL}]$  in ethanol. To each of the above solutions (40 mL), and 2.0 g of HAp was added respectively. The solutions were stirred at 70°C. The solid was collected by filtration. The product was washed several times with a mixture of ethanol and water. The dried samples were kept in an oven maintained at 100°C for 12 h. Finally, the dried product was divided into two parts and calcined in the air at 300°C and 500°C for 12 h. The above methods were also unsuccessful to support  $[\text{RhCl}_3(\text{Ar}_2\text{E})_3]$  complexes on hydroxyapatite (HAp).

All samples prepared by the above methods showed no presence of rhodium, sulfur, selenium, or tellurium by the powder X-ray diffraction analysis. In general, no significant variation is observed in the diffraction pattern of samples prepared with different rhodium ligands and no characteristic peaks of sulfur, selenium, tellurium, or rhodium were observed. The peaks obtained were compared with standard references [23].

## 2.2. Catalytic activity

### 2.2.1 Hydrosilylation of olefins

Hydrosilylation was carried out in a round-bottomed flask equipped with a magnetic stirrer and reflux condenser. A drying system was attached to the upper of the condenser. Olefin and platinum complex were stirred at the reaction temperature (50, 70, and 90°C) for 60 min before triethoxysilane was added. The structure and the yield of hydrosilylation products were determined by GLC using a standard sample and a standard curve at regular intervals.

Typical conditions were by using 10 mmol of  $\text{HSi}(\text{OEt})_3$ , 10 mmol of olefin, and  $10^{-3} - 10^{-4}$  mmol of rhodium complex.

### 2.3. Physical measurements

IR spectra were recorded as CsI discs in the range  $4000-200\text{cm}^{-1}$  using a Pye-Unicam SP3-300s spectrophotometer.  $^1\text{H}$  NMR spectra were recorded on a Bruker AVANCE 300 (300.13 MHz) or a Bruker 400(400.13 MHz) spectrophotometer.  $^{13}\text{C}$  NMR ( $^1\text{H}$  decoupled) spectra were recorded on a Bruker AVANCE 300 (75.47 MHz) or Bruker DRX 400 (100.63 MHz) spectrometer. Spectra were recorded in deuterated chloroform ( $\text{CDCl}_3$ ) or  $\text{CD}_2\text{Cl}_2$  unless otherwise stated and chemical shifts are reported in parts per million downfield from tetramethylsilane (TMS) for  $^1\text{H}$  NMR spectra and relative to the central signal of deuterated solvent for  $^{13}\text{C}$  NMR spectra. Coupling constants are given in Hertz. Microanalysis of carbon, hydrogen, and nitrogen was carried out by a Carlo Erba EA 1108-Elemental Analyzer instrument. Melting points were determined on a Gallenkamp melting point apparatus and are uncorrected. GLC determinations were carried out on a Hewlett Packard-5840A chromatograph equipped with a 6mX1/8 column of OV-17 on chromosorb w. Mass spectra were measured with an MAT 1125 Finnigan mass spectrometer.

### 3. Results and Discussion

The dimeric precursor  $[\text{Rh}(\text{CO})_2\text{Cl}]_2$  reacts with various diaryl chalcogenide ( $\text{Ar}_2\text{E}$ ) ligands in 1 : 1 mole ratio afforded the new *cis*-dicarbonyl complexes of the type  $[\text{Rh}(\text{CO})_2\text{ClL}]$  (**1-6**) (where  $\text{L} = \text{Ar}_2\text{E}$ ;  $\text{Ar} = 4\text{-CH}_3\text{C}_6\text{H}_4$ ,  $4\text{-BrC}_6\text{H}_4$ ,  $\text{E} = \text{S}$ ,  $\text{Se}$  and  $\text{Te}$ ), Scheme 1. Reaction of rhodium (III) chloride trihydrate with an excess amount of  $\text{Ar}_2\text{E}$  gave new complexes of the type  $[\text{RhCl}_3(\text{Ar}_2\text{E})_3]$  (*i.e.*, compounds **7-12**), Scheme 1. All these complexes (**1 - 12**) gave a satisfactory CHN analysis, Tables 1 and 2.

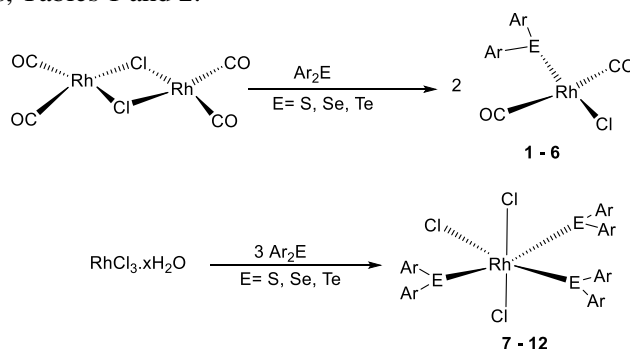


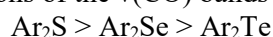
Table 1. Physical properties of  $[\text{RhCl}(\text{CO})_2(\text{Ar}_2\text{E})]$  complexes

$\text{Ar}_2\text{E}$	M.p. $^\circ\text{C}$	Colour	Elemental analyses Found(calc.)	
			C	H
$(4\text{-CH}_3\text{C}_6\text{H}_4)_2\text{S}$ ( <b>1</b> )	172	deep brown	46.86(47.02)	3.52(3.45)
$(4\text{-CH}_3\text{C}_6\text{H}_4)_2\text{Se}$ ( <b>2</b> )	156	red-brown	42.03(42.18)	2.95(3.10)
$(4\text{-CH}_3\text{C}_6\text{H}_4)_2\text{Te}$ ( <b>3</b> )	145	dark red	37.86(38.11)	2.76(2.80)
$(4\text{-BrC}_6\text{H}_5)_2\text{S}$ ( <b>4</b> )	180	red-brown	30.85(31.23)	1.24(1.50)
$(4\text{-BrC}_6\text{H}_5)_2\text{Se}$ ( <b>5</b> )	160	dark red	28.45(28.73)	1.23(1.38)
$(4\text{-BrC}_6\text{H}_5)_2\text{Te}$ ( <b>6</b> )	149	deep red	26.48(26.52)	1.21(1.27)

Table 2. Physical properties of  $[\text{RhCl}_3(\text{Ar}_2\text{E})_3]$  complexes

Ar <sub>2</sub> E	M.p.°C	Colour	Elemental analyses Found(calc)	
			C	H
(4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub> ) <sub>2</sub> S ( <b>7</b> )	150(d)	red–brown	58.76 (59.19)	5.05 (4.97)
(4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub> ) <sub>2</sub> Se ( <b>8</b> )	120(d)	red–brown	50.91 (50.81)	4.02 (4.26)
(4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub> ) <sub>2</sub> Te ( <b>9</b> )	68	red–brown	43.86 (44.30)	3.58 (3.72)
(4-BrC <sub>6</sub> H <sub>5</sub> ) <sub>2</sub> S ( <b>10</b> )	76	dark brown	35.04 (34.83)	2.03 (1.95)
(4-BrC <sub>6</sub> H <sub>5</sub> ) <sub>2</sub> Se ( <b>11</b> )	110	dark brown	31.07 (31.28)	1.66 (1.75)
(4-BrC <sub>6</sub> H <sub>5</sub> ) <sub>2</sub> Te ( <b>12</b> )	121	dark brown	27.86 (28.30)	1.52 (1.58)

The IR spectra of the complexes **1-6** showed two almost equally intense terminal  $\nu(\text{CO})$  bands in the range 2003-2093  $\text{cm}^{-1}$  indicating two carbonyl groups are mutually cis to one another [24,25], Table 3. The positions of the  $\nu(\text{CO})$  bands follow the following order:



This may be explained in terms of ‘hard-soft’ interactions between the metal and the chalcogenide donor [26]. The IR spectra of complexes **7-12** show some characteristic bands between 320 and 385  $\text{cm}^{-1}$  due to the terminal  $\nu(\text{Rh-Cl})$ [13,27], Table 3. The appearance of a medium band near 345  $\text{cm}^{-1}$  typical of asymmetric stretching vibration of a linear Cl-Rh-Cl group, indicates a *trans* arrangement of chlorine atoms[28-30]. Thus, based on the above evidence, square planar and octahedral structures can be assigned for complexes **1 – 12**, Scheme 1.

Table 3. Characteristic IR frequencies ( $\text{cm}^{-1}$ ) of complexes **1 – 12**.

Complex	$\nu(\text{C-H})$ Phenyl ring	$\nu(\text{CO})$	$\nu(\text{Rh-Cl})$	Asy. $\nu(\text{Cl-Rh-Cl})$
<b>1</b>	3056	2062, 1995	295, 285	
<b>2</b>	3060	2055, 1945	290, 282	
<b>3</b>	3057	2058, 1938	302, 275	
<b>4</b>	3053	2068, 2001	284, 270	
<b>5</b>	3056	2058, 1985	296, 280	
<b>6</b>	3056	2047, 1956	300, 276	
<b>7</b>	3063		380, 322	345
<b>8</b>	3065		385, 322	337
<b>9</b>	3058		380, 322	345
<b>10</b>	3061		385, 322	342
<b>11</b>	3060		370, 318	345
<b>12</b>	3058		385, 320	343

<sup>1</sup>H and <sup>13</sup>C spectra for all complexes (**1-12**) agree well with their structure (Table 4). The <sup>1</sup>H NMR spectra of **1-6** exhibit two doublet resonances in the range  $\delta$  7.32-8.83 ppm for the disubstituted benzene rings. The <sup>13</sup>C NMR spectra of **1-6** show characteristic resonances of terminal carbonyl groups in the range  $\delta$  182-188 ppm and four signals in the region  $\delta$  112-140 ppm for carbon atoms of a benzene ring, Table 4.

Table 4.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectroscopic data

Complex	$^1\text{H}$ NMR ( $\delta$ , 300 MHz, $\text{CDCl}_3$ )	$^{13}\text{C}$ ( $\delta$ , 75 MHz, $\text{CDCl}_3$ )
1	7.35 (d, $J = 7.8$ Hz, 4H, H-Ar), 4H), 7.06 (d, $J = 8.1$ Hz, 4H, H-Ar),), 2.14 (s, 6H, $\text{CH}_3$ ).	186.7, 182, 139.00, 132.50, 131.66, 130.29, 21.19
2	7.47–7.44 (m, 4H, H-Ar), 7.35 (d, $J =$ 8.3 Hz, 4H, H-Ar), 2.38 (s, 6H, $\text{CH}_3$ ).	188.1, 185, 137.4, 137.1, 129.4, 128.7, 21.6.
3	7.48 – 7.36 (m, 4H, H-Ar), 7.15 (d, $J =$ 8.1 Hz, 4H), 2.38 (s, 6H, $\text{CH}_3$ ).	186.2, 182.6, 137.4, 137.1, 129.4, 109.3, 21.5
4	7.77 – 7.67 (m, 4H, H-Ar), 7.54 – 7.43 (m, 4H, H-Ar).	187.1, 184.3, 134.5, 132.6, 130.7, 124.6.
5	7.55 (d, $J = 7.8$ Hz, 4H, H-Ar), 7.25 – 7.15 (m, 4H).	187.6, 183.8, 139.4, 132.60, 128.8, 122.5.
6	7.44 (d, $J = 8.3$ Hz, 4H, H-Ar), 7.34 (d, $J = 7.9$ Hz, 4H, H-Ar).	186.5, 182.5, 139.4, 132.6, 122.5, 112.4.
7	7.41 (d, $J = 7.8$ Hz, 4H, H-Ar), 4H), 7.22 (d, $J = 8.1$ Hz, 4H, H-Ar),), 2.34 (s, 6H, $\text{CH}_3$ ).	138.3, 132.7, 131.5, 130.3, 22.4
12	7.35 (d, $J = 8.1$ Hz, 4H, H-Ar), 7.27.15 (d, $J = 8.3$ Hz, 4H, H-Ar).	138.5, 133.1, 131.2, 119.3

Attempts to support these new complexes (1–12) on hydroxyapatite were unsuccessful. The IR spectra of all products exhibited only the characteristic absorption peaks of hydroxyapatite [28,29], Fig. 1.

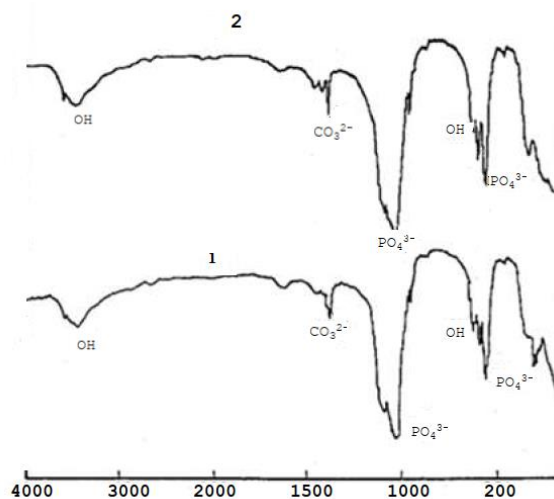


Figure 1. IR spectra of Hap and Rh complexes at 1) 300°C and 2) 500°C.

All samples obtained for hydroxyapatite-rhodium complexes by different methods showed no presence of rhodium, sulphur, selenium, or tellurium by the powder X-ray diffraction analysis, Fig. 2 and no significant variation was observed in the diffraction pattern of samples prepared with different rhodium ligands. Furthermore, no characteristic peaks of sulphur, selenium, tellurium or rhodium were observed. The peaks obtained were compared with a standard reference [23].

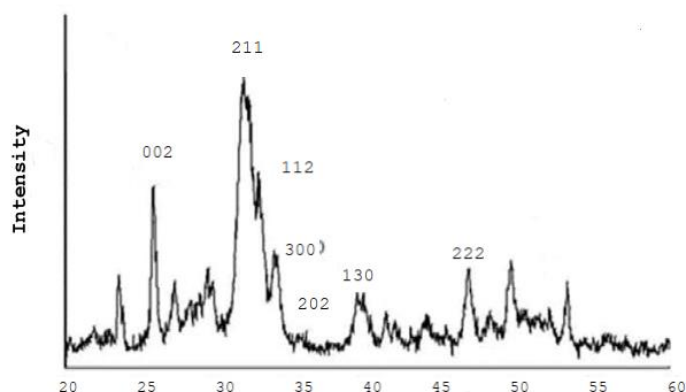


Figure 2. XRD of HAp and Rh complexes

The unsuccessful attempts to support the rhodium (I) or rhodium (III) complexes on hydroxyapatite, prompted us to investigate the catalytic activities of complexes **1-6** on hydrosilylation of allyl phenyl ether and 1-decene. It is worth noting that our previous work[31] described the hydrosilylation of olefins using platinum (II) complexes containing hexenyl aryl telluride ligands.

The catalytic activity of complexes **1**, **2**, and **3** were examined between 50 and 90°C using 1-decene and triethoxysilane as a model reaction, Fig. 3. The experimental results indicated that no remarkable induction period was observed and the reaction rate became faster. In the range 50 to 90°C, the catalytic reaction finished within 50 min, see Fig. 3.

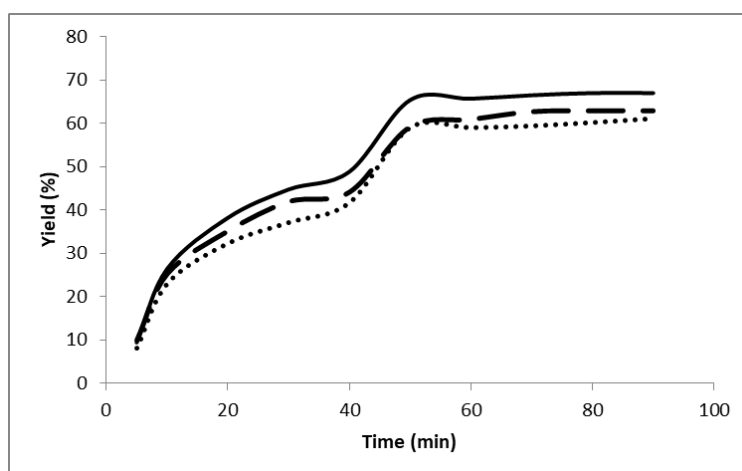


Figure 3. The catalytic activity of complexes **1**(—), **2**(- -) and **3**(.....) between 50 and 90°C using 1-decene and triethoxysilane as a model reaction

Thus, the catalytic behavior of complexes **1**, **2**, and **3** were examined by using allyl phenyl ether and 1-decene as substrates. The substrates were hydrosilylated with  $\text{HSi}(\text{OEt})_3$  by stirring the olefin and rhodium(I) complexes for 60 min followed by the addition of triethoxysilane to give the saturated products between 59- 65% yields, Table 5. The catalytic activity of complexes **4**, **5**, and **6** were also examined with allyl phenyl ether and 1-decene by the same procedure and gave the saturated products in the range 48 - 60%, Table 5.

The efficacy of complexes **1**, **2**, and **3** towards the hydrosilylation was found to follow the order  $\mathbf{1} > \mathbf{2} > \mathbf{3}$ . This may be due to the difference in bond lengths of Rh-S, Rh-Se, and Rh-Te. In general, complexes **1**, **2**, and **3** showed moderate catalytic activity. On other hand, Complexes **7**, **8**, and **9** showed no catalytic activity in comparison with Rh(I) complexes.

#### 4. Conclusion

In conclusion, some new Rh(I) and Rh(III) complexes were prepared and characterized by various spectroscopic techniques. Attempts to support Rh(I) and Rh(III) complexes on hydroxyapatite by different methods were unsuccessful. Complexes  $[\text{RhCl}(\text{CO})_2((4\text{-CH}_3\text{C}_6\text{H}_4)_2\text{S})]$ ,  $[\text{RhCl}(\text{CO})_2((4\text{-CH}_3\text{C}_6\text{H}_4)_2\text{Se})]$  and  $[\text{RhCl}(\text{CO})_2((4\text{-CH}_3\text{C}_6\text{H}_4)_2\text{Te})]$  showed moderate to good catalytic activity for the hydrosilylation. Complexes of type  $[\text{RhCl}_3(\text{Ar}_2\text{E})_3]$  (*i.e.* compounds 7, 8, and 9) showed no catalytic activity in comparison with Rh(I) complexes.

#### Acknowledgements

The financial support of this work by the Ministry of Higher Education and Scientific Research/ Research and Development is hereby gratefully acknowledged.

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