

# Hydrolysis, Polycondensation, and Catalytic Properties of Ru(II) Complex of 3-4,5-Dihydroimidazol-1-yl-propyltriethoxysilane

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**ABSTRACT:** The preparation and measurements of some properties of organic–inorganic hybrid materials derived from Ru(II)-3-4,5-dihydroimidazol-1-yl-propyltriethoxysilane inside a polysiloxane network have been achieved. The hydrolysis and polycondensation of Ru(II)-3-4,5-dihydroimidazol-1-yl-propyltriethoxysilane were performed in different experimental conditions, producing a new organic–inorganic silica. The alkoxy-silyl groups available were used for the construction of inorganic backbone by the sol-gel process, and the imidazole group was found suitable for incorporating Ru(II) by coordination. The coordination of metal complex is retained because there is no leaching from the metal complex containing gels. To ensure sufficient catalytic properties, a series of hybrid materials from tetraethoxysilane was prepared. These materials were identified and catalytic activities were tested for cyclization of (*Z*)-3-methylpent-2-en-4-yn-1-ol to 2,3-dimethylfuran. Heterogeneous Ru(II) catalyst can also be recycled and reused without significant loss of selectivity or activity. © 2001 John Wiley & Sons, Inc. *J Appl Polym Sci* 80: 1329–1334, 2001

**Key words:** sol-gel process; organic–inorganic hybrids; polysiloxane network; Ru(II) complex

## INTRODUCTION

It has been shown that the sol-gel route is increasingly extended to prepare organically modified materials by using organically substituted alkoxides of the type  $(\text{RO})_3\text{Si}(\text{CH}_2)_n\text{A}$ . Materials with both interesting physical and chemical properties are obtained if the organic groups are ca-

pable of coordinating metal ions. Groups A that are suitable for coordinating metal ions are, for example,  $\text{NH}_2$ ,  $\text{NHCH}_2\text{CH}_2\text{NH}_2$ ,  $\text{CN}$ , or  $\text{CH}(\text{COMe})_2$ .<sup>1–5</sup> The introduction of metal atoms in a gel is an interesting challenge. The approach of anchoring metal complex moieties to the silicate matrix has been explored for the preparation of supported catalysts.<sup>6</sup> Materials containing ruthenium (II) complexes in a silicate matrix are very interesting materials for the production of new supporting materials of a metal catalyst design. The nature and the structural features of the support may decisively improve activity, selectivity, and lifetime of the catalytic metal species. A more general approach to control dispersion of the metal compound during sol-gel processing is the

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use of chemical links between metal ions and the inorganic component. The silanes  $A(\text{CH}_2)_3\text{Si}(\text{OR})_3$  ( $R = \text{Me, Et; A} = \text{NH}_2$ ),  $\text{NHCH}_2\text{CH}_2\text{NH}_2$ ,  $\text{CN}$ , and  $\text{CH}(\text{COMe})_2$  group have been found to form complexes with transition metal complex.<sup>7–14</sup> However, compounds of the type 3-4,5-dihydroimidazol-1-yl-propyltriethoxysilane (IMEO) meet these requirements as well, because the anchoring group and the spacer are necessary to ensure ideal complex formation during polycondensation. In our investigation 3-4,5-dihydroimidazol-1-yl-propyltriethoxysilane has been chosen to prepare metal/ $\text{SiO}_2$  composites for coordinating the ruthenium complex before polycondensation. We previously reported preliminary results on preparation of ( $\eta$ -arene) ruthenium (II) complexes containing pyridine ligands with pendant vinyl groups<sup>4</sup> and  $\text{Al}(\text{OR})_3$ .<sup>15</sup>

Here, we explored the possibility of employing the 3-4,5-dihydroimidazol-1-yl-propyltriethoxysilane group as a coordinating chelate with ( $\eta$ -arene) ruthenium (II) complexes. In this report we describe the synthesis of  $\text{Ru}(\text{II})$ -3-4,5-dihydroimidazol-1-yl-propyltriethoxysilane (**2**) and hydrolysis of **2** in different experimental conditions leading to new materials. Further goals are catalytic reactions that will be carried out at the transition metal center of the stationary phase.

## EXPERIMENTAL

All reagents were obtained from Aldrich Chemical Company unless stated otherwise. All manipulations were performed under an oxygen-free nitrogen atmosphere using predried solvents,

vacuum line, and Schlenk tube techniques. The reported yields refer to pure isolated materials. Infrared spectra were recorded as KBr pellets in the range  $4000\text{--}400\text{ cm}^{-1}$  on an ATI UNICAM systems 2000 Fourier transform spectrometer.  $^1\text{H}$ -NMR spectra (300.131 MHz)  $^{13}\text{C}$ -NMR spectra (75.5 MHz) on a Bruker AM 300 WB FT spectrometer with  $\delta$  referenced to residual solvent  $\text{CDCl}_3$ . Microanalyses were performed by the TÜBİTAK (Ankara). The identification of  $\text{Si}(\text{OH})_3$  was made using a multichannel Raman spectrometer (DILOR xy) equipped with a CCD detector. The band position was accurate to  $\pm 1\text{ cm}^{-1}$ . All spectra were taken at 20–40 mW on the sample. The specific surface areas, porous volume, and pore size distribution were determined by analyzing the  $\text{N}_2$  adsorption/desorption isotherms according to the BET method (Oregon State University, Corvallis). The standard relative error on specific areas was as high as 5%.

### Preparation of $\text{Ru}(\text{II})$ -3-4,5-dihydroimidazol-1-yl-propyltriethoxysilane (**2**)

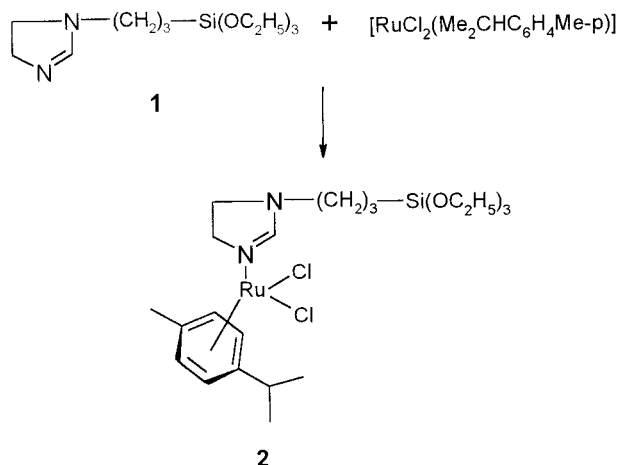
To a solution of 3-4,5-dihydroimidazol-1-yl-propyltriethoxysilane (1.10 g, 4 mmol) in toluene (40 mL) at  $0^\circ\text{C}$  were added dropwise  $[\text{RuCl}_2(p\text{-Me}_2\text{CHC}_6\text{H}_4\text{Me})_2]$  (1.25 g, 2 mmol). The reaction mixture was allowed to stand for 10 h under reflux. Hexane (20 mL) was added to the solution while warm. The solvent was then pumped off and the residue was washed several times with ether. Product **2** was filtered off, washed with *n*-hexane ( $2 \times 20\text{ mL}$ ), and dried in vacuum. Recrystallization from  $\text{CH}_2\text{Cl}_2/\text{Et}_2\text{O}$  at  $-25^\circ\text{C}$  gave orange crystals of **2** (yield 89%, mp  $104\text{--}105^\circ\text{C}$ ).

**Table I** Hydrolysis and Polycondensation of  $\text{Ru}(\text{II})$ -3-4,5-Dihydroimidazol-1-yl-propyltriethoxysilane (**2**)<sup>a</sup>

Entry	Precursor Concentration (mol/L)	Catalyst	Catalyst Concentration (mol eq)	Precipitation Time (h)	Surface Area ( $\text{m}^2/\text{g}$ ) <sup>b</sup>
1	1	—	—	36	148
2	1	NaF	$2 \times 10^{-2}$	5.4	185
3	1	NaF	$10^{-2}$	9.7	222
4	1	NaF	$10^{-3}$	19	148
5	0.5	NaF	$10^{-2}$	28.5	148
6	0.25	NaF	$10^{-3}$	10	185
7	1	$\text{NH}_4\text{F}$	$10^{-2}$	24	174
8	0.25	$\text{NH}_4\text{F}$	$10^{-3}$	12	165

<sup>a</sup> Experimental conditions: solvent ethanol, room temperature, stoichiometric amount of water.

<sup>b</sup> BET surface area from multipoint analysis of  $\text{N}_2$  isotherm.



**Scheme 1** Synthesis of Ru(II)-3-4,5-dihydroimidazol-1-yl-propyltriethoxysilane (**2**).

ANAL. calcd for  $C_{22}H_{40}N_2RuSiO_3Cl_2$ : C: 45.52; H: 6.89; N: 4.83. Found: C: 45.56; H: 6.80; N: 4.75%.  $^1H$ -NMR (300 MHz,  $CDCl_3$ , 297 K,  $\delta$ ) 7.28 (s, 1H, C2-H), 3.49 (t, 2H, C4-H<sub>2</sub>,  $J = 10.3$  Hz), 4.12 (t, 2H, C5-H<sub>2</sub>,  $J = 10.3$  Hz), 3.13 [t, 2H,  $CH_2CH_2CH_2Si(OCH_2CH_3)$ ,  $J = 7.3$  Hz], 1.61 [t., 2H,  $CH_2CH_2CH_2Si(OCH_2CH_3)$ ,  $J = 7.4$  Hz], 0.56 [t, 2H,  $CH_2CH_2CH_2Si(OCH_2CH_3)$ ,  $J = 7.8$  Hz], 3.81 [q, 2H,  $CH_2CH_2CH_2Si(OCH_2CH_3)$ ,  $J = 7.0$  Hz], 1.23 [t, 3H,  $CH_2CH_2CH_2Si(OCH_2CH_3)$ ,  $J = 7.0$  Hz], 5.23 and 5.39 [d, 4H,  $Me_2CHC_6H_4Me-p$ ],  $J = 6.0$  Hz], 3.02 [sept, 1H,  $Me_2CHC_6H_4Me-p$ ],  $J = 6.9$  Hz], 1.31 [d, 6H,  $(CH_3)_2CHC_6H_4Me-p$ ],  $J = 7.0$  Hz], 2.24 [s, 3H,  $Me_2CHC_6H_4(CH_3)-p$ ],  $J = 6.0$  Hz].  $^{13}C$ -NMR (75 MHz,  $CDCl_3$ , 297 K,  $\delta$ ): 161.73 (s, C2-H), 57.9 (s, C4-H<sub>2</sub>),

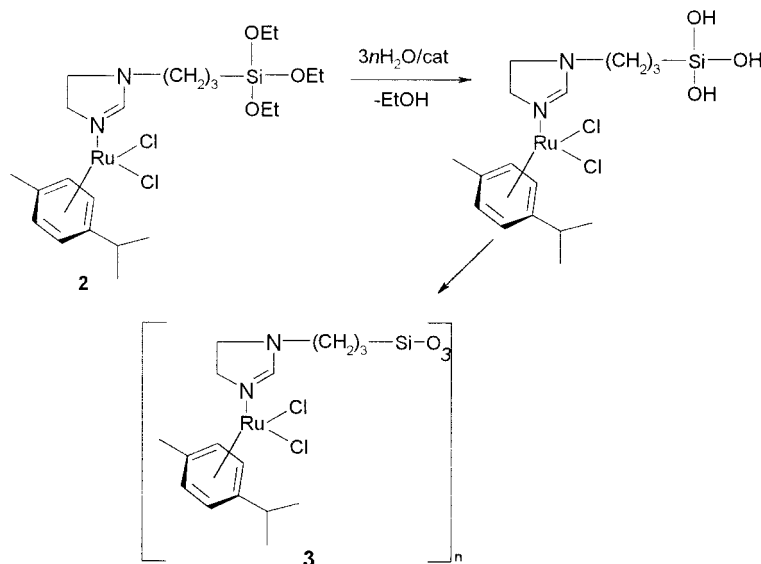
48.97 (s, C5-H<sub>2</sub>), 50.67 [s,  $CH_2CH_2CH_2Si(OCH_2CH_3)$ ], 22.41 [s,  $CH_2CH_2CH_2Si(OCH_2CH_3)$ ], 8.05 [s,  $CH_2CH_2CH_2Si(OCH_2CH_3)$ ], 59.09 [s,  $CH_2CH_2CH_2Si(OCH_2CH_3)$ ], 18.96 [s,  $CH_2CH_2CH_2Si(OCH_2CH_3)$ ], 81.74, 82.35, 97.18, and 102.59 [s,  $Me_2CHC_6H_4Me-p$ ], 31.34 [s,  $Me_2CHC_6H_4Me-p$ ], 22.88 [s,  $(CH_3)_2CHC_6H_4Me-p$ ], 19.30 [s,  $Me_2CHC_6H_4(CH_3)-p$ ],  $J = 6.0$  Hz].

### Hydrolysis and Polycondensation of Ru(II)-3-4,5-dihydroimidazol-1-yl-propyltriethoxysilane (**2**)

The concentration of the reagents and the catalysts are indicated in Table I. Hydrolysis of **2** (Table I, entry 4) is given as an example. To  $5.2 \times 10^{-3}$  mol of **2** in ethanol were added molar equivalents of water and the catalyst. The homogeneous solution was allowed to stand at room temperature. An orange precipitate appeared. The mixture was allowed to stand for 1 week at room temperature; the solid was filtered then washed with water, acetone, and ether. It was dried *in vacuo* at 175°C to yield 1.43 g of a powder (quantitative yield based on  $C_{16}H_{25}RuO_{3/2}(Cl_2Si)$  FTIR (KBr pellet); 3425, 2950, 2854, 1600, 1170, 1089, 1035, 650  $cm^{-1}$ . Elemental analysis found: C, 44.3%; H, 6.70%; Ru, 23%; O, 6.8%; Cl, 17.5%; Si, 7.2%.

### Cocondensation of Ru(II)-3-4,5-dihydroimidazol-1-yl-propyltriethoxysilane (**2**) with $Si(OEt)_4$

The cocondensation of **2** with one equivalent of  $Si(OEt)_4$  (Table I, entry 4) is given as an example.



**Scheme 2** Hydrolysis and polycondensation of Ru(II)-3-4,5-dihydroimidazol-1-yl-propyltriethoxysilane (**2**).

**Table II Hydrolysis and Polycondensation of Ru(II)-3-4,5-Dihydroimidazol-1-yl-propyltriethoxysilane (2) in Different Experimental Conditions**

Entry	Compound	Experimental Conditions	Precursor Concentration (mol/L)	Surface Area (m <sup>2</sup> /g)
9	2	HCl/H <sub>2</sub> O/Δ reflux/24 h	0.1	196
10	2	HCl/MeOH/Δ reflux/14 h	0.1	95
11	2	HCl <sup>a</sup> /H <sub>2</sub> O/Δ reflux/24 h	0.1	54
12	2	HCl/H <sub>2</sub> O <sup>b</sup> /Δ reflux/24 h	0.1	24
13	2	HCl/H <sub>2</sub> O <sup>c</sup> /Δ reflux/RT	0.1	—

<sup>a</sup> HCl concentration 4.5 molar equivalent.

<sup>b</sup> 10 molar equivalent of water in ethanol.

<sup>c</sup> 10 molar equivalent of water at room temperature.

To  $2.87 \times 10^{-3}$  mol of **2** and  $2.87 \times 10^{-3}$  mol of Si(OEt)<sub>4</sub> in THF were added 5 mol eq of water and  $10^{-2}$  mol eq of NaF. The mixture was stirred ultrasonically, then allowed to stand at room temperature. A gel was formed after 19 h. After drying *in vacuo* at 175°C, 1.4 g of an orange-colored powder was obtained (quantative yield based on C<sub>16</sub>H<sub>25</sub>RuO<sub>5/2</sub>Cl<sub>2</sub>Si<sub>2</sub>). The sample was identified by FTIR (KBr): 3422, 2942, 2850, 1600, 1175, 1039 cm<sup>-1</sup>. The X-ray powder diffraction pattern was that of an amorphous material; N<sub>2</sub> BET surface area was 105 m<sup>2</sup>/g. Elemental analysis found: C, 41.2%; H, 6.4%; Ru, 21.2%; O, 8.9%; Cl, 15.1%; Si, 12.4%. The carbon-to-silicon ratio was 7.7 and the ruthenium-to-silicon ratio was 0.47.

### Catalytic Reactions

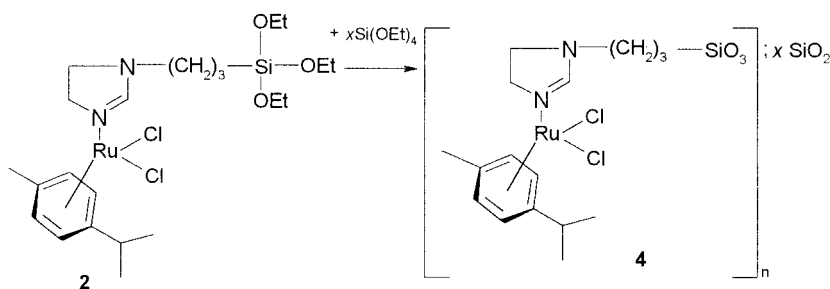
The cyclization experiments were carried out in a 100-mL Schlenk tube equipped with a magnetic stirring bar. The Schlenk tube was evacuated and flushed with argon three times before the reaction mixture was introduced. (*Z*)-3-Methylpent-2-en-4-yn-1-ol (100 mg) in THF and the proper amount

of the matrix were mixed and the mixture was stirred in an oil bath at 80°C. The progress of the reaction was monitored by GC. The product characterized after purification was found to be 2,3-dimethylfuran as evidenced by <sup>1</sup>H- and <sup>13</sup>C-NMR. The analyses were performed quantitatively on a capillary GC equipped with a thermal conductivity detector using a 6-foot column of 10% = V-101 on chromosorp W-HP, 100–120 mesh.

In the recycling tests, when the reaction was complete, the catalyst was removed by filtration from the reaction mixture, washed with 3× toluene (10 mL), dried in a vacuum oven, and used again.

### RESULTS AND DISCUSSION

We first examined the formation of **2** under various conditions (Scheme 1 and Scheme 2). The divalent ruthenium cation is classified as a borderline acid that shows affinity for borderline bases such as pyridine, and thus [(η-arene) RuCl<sub>2</sub>]<sub>2</sub> readily reacts to give **2** with an imidazol



**Scheme 3** Cocondensation of Ru(II)-3-4,5-dihydroimidazol-1-yl-propyltriethoxysilane (**2**) with *x* equivalents of Si(OEt)<sub>4</sub>.

**Table III** Cocondensation of Ru(II)-3-4,5-Dihydroimidazol-1-yl-propyltriethoxysilane (**2**) with  $x$  Equivalents of Si(OEt)<sub>4</sub><sup>a</sup>

Entry	$x$	Catalyst	Catalyst Concentration (mol eq)	Gelation Time (h)	Surface Area (m <sup>2</sup> /g)
14	1	—	—	23	165
15	4	—	—	49	286
16	10	—	—	62	465
17	1	NaF	10 <sup>-2</sup>	19	105
18	4	NaF	10 <sup>-2</sup>	9.7	488
19	10	NaF	10 <sup>-2</sup>	5.4	550

<sup>a</sup> Experimental conditions: solvent ethanol, room temperature, molar concentration, stoichiometric amount of water.

moiety of **1** as shown in Scheme 1. The result of the structure analysis suggests that imidazol of the type (RO)<sub>3</sub>Si(CH<sub>2</sub>) <sub>$n$</sub> A with a functional group in a more distant position would be suitable to form **2**. The compound was identified on the basis of elemental analysis and spectroscopic measurements. The tentative band assignment found in the Raman spectrum was as follows: The band at 430–440 cm<sup>-1</sup> (s) is the Si–O–Si in-plane vibration.<sup>16</sup> The band at 490–500 cm<sup>-1</sup> was attributed to the structures sketched previously.<sup>17</sup> The Si–O–Si symmetric stretching and Si–O–Si asymmetric stretching band appeared at 800 and 1100 cm<sup>-1</sup>, respectively. Internal and surface silanols stretching at 910–920 and 970–985 cm<sup>-1</sup> were also noted.<sup>16–19</sup>

The hydrolysis and polycondensation of **2** were carried out either without catalyst or in the presence of three types of catalysts. In every case, a precipitate formed in 5.4 to 28.5 h.

Aging was performed in a sealed tube at room temperature for 8 days. The solid was then collected, washed, and dried. The results are reported in Tables I and II.

Elemental analysis calculated for C<sub>16</sub>H<sub>25</sub>RuO<sub>3/2</sub>Cl<sub>2</sub>Si gave carbon-to-silicon ratio 8.7 < C/Si < 9 (calculated C/Si = 8) and ruthenium-to-silicon ratio 0.98 (calculated Ru/Si = 1.00). The X-ray powder diffraction pattern showed no crystallinity, irrespective of reaction conditions. The BET surface areas are low in every case.

We examined the possibility of formation of gels by cocondensation of **2** with Si(OEt)<sub>4</sub>, as shown in Scheme 3. The results are reported in Table III. Monolithic gels were obtained in every case. Elemental analysis gave acceptable ruthenium-to-silicon ratios in every case, whereas carbon-to-silicon ratios were higher because of residual ethoxy groups in the material.

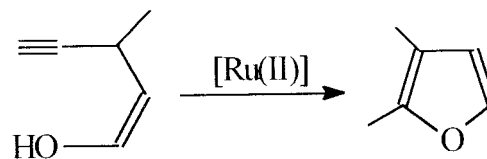
The elemental analysis and the spectra obtained for the solids **3** prepared by hydrolysis and polycondensation of Ru(II)-3-4,5-dihydroimidazol-1-yl-propyltriethoxysilane (**2**) (reported in Table I) are in agreement with the conservation of the ruthenium unit in the silica matrix. The organic moiety is not affected during the polymerization process. The results confirm that the ruthenium unit bound the silica matrix and some ethoxy groups in the materials. In the case of cocondensation with Si(OEt)<sub>4</sub> we can assume that there was no cleavage of the silicon–carbon bond; the structure of ruthenium on imidazoline moiety was retained in the co-gel, as evidenced by spectroscopic results.

The catalytic activity of the Ru(II)-complexed materials was investigated with regard to the cyclization of (*Z*)-3-methylpent-2-en-4-yn-1-ol to 2,3-dimethylfuran reaction (Scheme 4), the results of which are summarized in Table IV.

The catalytic activity is significantly less at room temperature when all complexes were used. Raising the reaction temperature to 80°C increases the catalytic activity. The catalyst was recovered by microporous filtration, washed with distilled hexane, and reused five times without significant loss of activity or selectivity.

## CONCLUSIONS

The use of Ru(II)-3-4,5-dihydroimidazol-1-yl-propyltriethoxysilane (**2**) allows an easy prepara-



**Scheme 4** Synthesis of 2,3-dimethylfuran.

**Table IV** Synthesis of 2,3-Dimethylfuran at 80°C<sup>a</sup>

Entry	Yield <sup>b</sup> (%)	Surface Area (m <sup>2</sup> /g)	Turnover (/h)
1	53	148	17
3	89	222	87
5	45	148	10
7	54	174	96
9	78	196	16
14	92	165	190
15	76	286	64
16	89	465	230
17	88	105	84
18	89	488	200
19	—	550	176

<sup>a</sup> Reaction conditions: to 0.1 g of catalyst 10 mmol of neat (Z)-3-methylpent-2-en-4-yn-1-ol were added. The mixture was stirred in an oil bath at 80°C.

<sup>b</sup> Yields determined by gas chromatography.

tion of mixed organic–inorganic hybrid materials containing a ruthenium atom inside a polysiloxane network. The molecular structure of the organometallic molecule is always retained and novel hybrid solids were obtained. The choice of experimental conditions is very crucial, because in some conditions the carbon–silicon bond is partially cleaved when an excess of water and HCl are used. Monolithic gels are obtained by cocondensation of Ru(II)-3-4,5-dihydroimidazol-1-yl-propyltriethoxysilane (2) with Si(OEt)<sub>4</sub>.

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