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Homogeneous catalytic hydrosilylation of the C=C double bond in the presence of transition metal catalysts

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Abstract

Hydrosilylation of unsaturated esters and other vinyl- and vinylidene type olefins has been carried out with $PtCl_2(PhCN)_2$, $RhCl(PPh_3)_3$ and platinum-phosphine catalysts. With $PtCl_2(PhCN)_2$ and platinum(0)-phosphine complexes as catalysts the hydrosilylation of methyl methacrylate gave the linear product **2a**, whereas in the $RhCl(PPh_3)$-catalyzed reaction the silyl ketene acetal derivative **4a** was formed selectively. In the reactions of other esters and unsaturated hydrocarbons the main product is highly dependent on the structure of the substrate, but the catalyst used is also very important. With unsaturated hydrocarbons the selectivity is rather poor, but in the case of 1-vinyl-2-pyrrolidinone the linear- (**9e**) and the branched hydrosilylated derivative (**10e**) are formed in high yields depending on the catalyst used. The platinum(II)-phosphine complexes are inactive in the absence of air, but some platinum(0)-phosphine catalysts are also effective under argon in the hydrosilylation of methyl methacrylate.

Introduction

We previously described the hydrosilylation of methyl methacrylate with Et_3SiH and Ph_3SiH in the presence of platinum catalysts [1]. The activities of these catalysts were found to depend strongly on the metal/ligand ratio. $PtCl_2(PPh_3)_2$ and complexes of chelating phosphines were inactive.

We present here the results of a study of the hydrosilylation of methyl methacrylate with various hydrosilanes (Et_3SiH, Ph_3SiH, Me_2ClSiH, Cl_3SiH) in the presence of platinum and rhodium catalysts. Some other olefins were also investigated, and the effects on the catalytic activity and regioselectivity of substituents in the olefins determined.

Results and discussion

Hydrosilylation of methyl methacrylate with various hydrosilanes

The hydrosilylation of methyl methacrylate catalysed by $PtCl_2(PhCN)_2$ with various monohydrosilanes gave several products (Scheme 1, Table 1). The main product was the linear derivative **2a** but the hydrogenated compound **6a** was also

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Table 1

Hydrosilylation of methyl methacrylate with various hydrosilanes

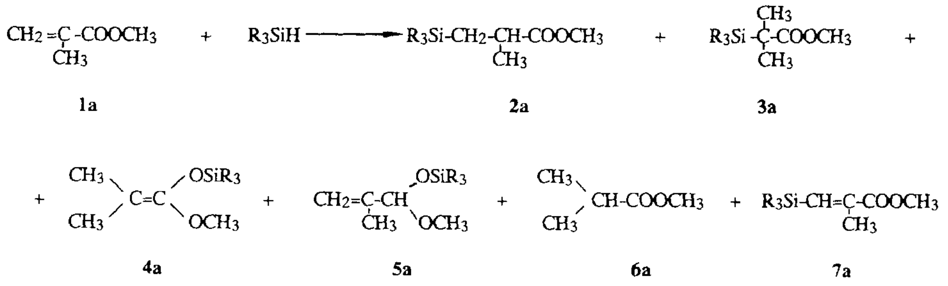
Silane	Catalyst	Reaction temp. (°C)	Reaction time (h)	Conv. [c] (%)	Product distribution (%) [d]					
					2a	**3a**	**4a**	**5a**	**6a**	**7a**
Et_3SiH	$PtCl_2(PhCN)_2$ [a]	25	24	89	93	–	–	–	6	1
	$RhCl(PPh_3)_3$ [b]	25	24	97	–	–	92	7	1	–
Ph_3SiH	$PtCl_2(PhCN)_2$ [a]	25	96	95	99	–	–	–	1	–
	$RhCl(PPh_3)_3$ [b]	25	96	99	–	–	100	–	–	–
Me_2ClSiH	$PtCl_2(PhCN)_2$ [a]	25	24	54	45 [e]	19 [e]	17 [e]	–	19	–
	$RhCl(PPh_3)_3$ [b]	110	10	45	–	–	70	–	30	–
Cl_3SiH	$PtCl_2(PhCN)_2$ [a]	60	38	57	75	–	–	–	25	–
	$RhCl(PPh_3)_3$ [b]	60	38	–	–	–	–	–	–	–

[a] Reaction conditions: 0.0036 mmol catalyst, 1.4 mmol substrate, 1.6 mmol hydrosilane, solvent: $CHCl_3$, under argon atmosphere. [b] Reaction conditions: 0.0017 mmol catalyst, 1.4 mmol substrate, 1.6 mmol hydrosilane, solvent: toluene, under argon atmosphere. [c] Mol reacted substrate/mol initial substrate × 100. [d] Mol product/mol reacted substrate × 100. [e] Dimethyl-hydrosilyl derivative.

formed in each case and a small amount of the unsaturated product **7a** was observed with Et_3SiH. As it is well-known [2], in the presence of $RhCl(PPh_3)_3$ 1,4- and 1,2-addition of Et_3SiH takes place. In the case of Ph_3SiH and Me_2ClSiH, the 1,4-addition product **4a** was mainly formed, but with the latter hydride the hydrogenated derivative **6a** was also detected. The fact that Me_2ClSiH gives not only the dimethyl-chlorosilyl- but also the dimethyl-hydrosilyl derivative brings to mind the similar outcome of Ni-catalyzed hydrosilylation reactions with this hydride [3]. The results can be explained in terms of the disproportionation of Me_2ClSiH to Me_2SiCl_2 and Me_2SiH_2 in the coordination sphere of the metal; the Me_2SiCl_2 formed during the reaction was detected by GLC-MS. The selectivity in this case is rather poor, but neither change of solvent nor increase in the temperature changes it very much (Table 2).

It should be mentioned that the 1,4-addition product **4a** is rather unstable, and in the presence of air it decomposes within a few days.

When Ph_2SiH_2 was used, mainly $Ph_2HSiSiHPh_2$ and other organosilanes were formed, and only traces of hydrosilylation products were detected.



Scheme 1

Table 2

Hydrosilylation of methyl methacrylate with Me_2ClSiH [a]

Solvent	Reaction temp. (°C)	Reaction time (h)	Conv. [b] (%)	Product distribution (%) [c]			
				2a [d]	**3a** [d]	**4a** [d]	**6a**
Chloroform	25	24	54	45	19	17	19
Chloroform	65	3	51	48	21	12	19
Benzene	65	3	51	52	10	38	–
Toluene	65	3	51	51	9	40	–

[a] Reaction conditions: 0.0036 mmol catalyst, 1.4 mmol substrate, 1.6 mmol Me_2ClSiH under argon atmosphere. [b] Mol reacted substrate/mol initial substrate × 100. [c] Mol product/mol reacted substrate × 100. [d] Dimethyl-hydrosilyl derivative.

Hydrosilylation of methyl methacrylate with Et_3SiH in the presence of platinum-phosphine catalysts

No conversion was observed with the Pt^{II}-phosphine catalysts ($PtCl_2(PPh_3)_2$, $PtCl_2$(diop), and $PtCl_2$(dppb)) in the hydrosilylation of methyl methacrylate when the reaction was carried out in argon atmosphere. In the presence of a small amount of oxygen reaction began, but stopped after some hours, and additional oxygen was needed to restart it. When the reaction was carried out with exposure to air the conversion was about 50% after 7 hours, but some polysiloxanes were also formed and no Et_3SiH remained in the reaction mixture.

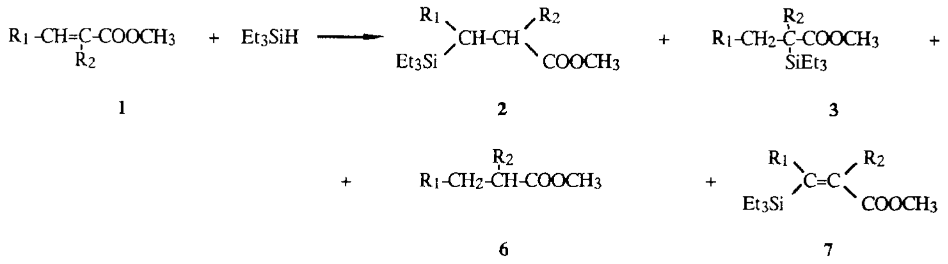
We monitored the reaction by ^{31}P-NMR spectroscopy and observed the formation of trans-$PtHCl(PPh_3)_2$ in the reaction of $PtCl_2(PPh_3)_2$ and Et_3SiH under argon at 110 °C after a few minutes. There was no further change in the composition of the mixture after several hours heating. In the presence of oxygen immediate formation of $OPPh_3$ but no other phosphine-containing platinum complexes except for $PtHCl(PPh_3)_2$ could be detected. This observation and the fact that in the absence of silane the decomposition of the catalyst and precipitation of Pt metal was observed, seems to support the view that the active catalyst contains no phosphine ligands.

Table 3

Hydrosilylation of methyl methacrylate with Et_3SiH in the presence of platinum(0)-phosphine catalysts [a]

Catalyst	Reaction temp. (°C)	Reaction time (h)	Conv. [b] (%)	Product distribution (%) [c]		
				2a	**4a**	**6a**
$Pt(PPh_3)_4$	75	16	48	89	–	11
$Pt(dppb)_2$	90	22	62	88	–	12
$Pt(dppb)_2$ [d]	90	16	60	81	–	19
$Pt(O_2)(dppb)$	90	16	64	84	–	16
$Pt(diop)_2$	90	16	–	–	–	–
$Pt(diop)_2$ [d]	90	16	30	69	18	13

[a] Reaction conditions: 0.0036 mmol catalyst, 1.4 mmol substrate, 1.6 mmol Et_3SiH in toluene. [b] Mol reacted substrate/mol initial substrate. [c] Mol product/mol reacted substrate. [d] 0.04 mmol oxygen was added.



a: R_1=H R_2=CH_3
b: R_1=R_2=H
c: R_1=CH_3 R_2=H
d: R_1=H R_2=CH_2COOCH_3

Scheme 2

Although some Pt^0-phosphine complexes are active catalysts (Table 3) even when oxygen is absent, its presence generally accelerates the hydrosilylation, and with DIOP as ligand its presence is necessary for the reaction. No optical induction was observed in this latter case. The selectivity is similar to that for the $PtCl_2(PhCN)_2$ catalyzed reactions, but the amount of the hydrogenated derivative **6a** is higher when phosphines are present.

Hydrosilylation of other unsaturated esters in the presence of $PtCl_2(PhCN)_2$

Hydrosilylation of unsaturated esters with rhodium catalysts is well documented [2,4]. In almost every case the 1,4- and 1,2-addition products were obtained. In the present study we investigated the platinum-catalyzed hydrosilylation of methyl acrylate, dimethyl itaconate, and methyl crotonate, and compared the results with those for methyl methacrylate (Scheme 2, Table 4).

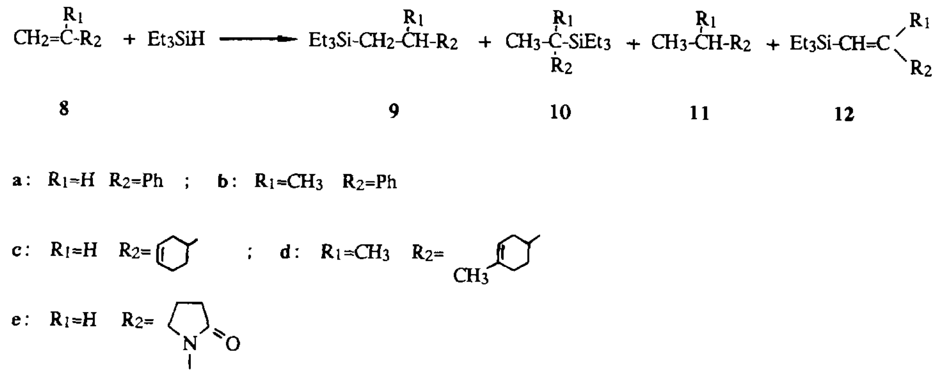
Using dimethyl itaconate **1d** as substrate the selectivity is similar to that for methyl methacrylate **1a** but the reaction is faster. At lower temperature the branched isomer **3b** is favoured, but at higher temperature the linear hydrosilylation product

Table 4

Hydrosilylation of unsaturated esters with Et_3SiH in the presence of $PtCl_2(PhCN)_2$ [a]

Substrate	Reaction temp. (°C)	Reaction time (h)	Conv. [b] (%)	Product distribution (%) [c]			
				2	**3**	**6**	**7**
1a	65	8	42	92	–	8	–
1b	30	2	85	7	80	–	13
	65	2	85	27	57	4	12
	90	2	82	54	26	8	12
1c	65	10	–	–	–	–	–
1d	65	8	73	90	–	10	–

[a] Reaction conditions: 0.0036 mmol catalyst, 1.4 mmol substrate, 1.6 mmol Et_3SiH in toluene under argon atmosphere. [b] Mol reacted substrate/mol initial substrate. [c] Mol product/mol reacted substrate.



Scheme 3

2b predominates in hydrosilylation of the methyl acrylate **1b**; the increase in the temperature also results in formation of the hydrogenated product **6b**.

No reaction occurred with methyl crotonate **1c** even when the reaction mixture was kept at 65 °C for 10 h.

Hydrosilylation of vinyl- and vinylidene-type olefins

Hydrosilylation of styrene and α-methyl-styrene in the presence of $PtCl_2$ [1,5] and rhodium catalysts [6] has been described. Here we report our results of a study

Table 5

Hydrosilylation of olefins with Et_3SiH in the presence of $PtCl_2(PhCN)_2$, $PtCl_2$ and $RhCl(PPh_3)_3$

Substrate	Catalyst	Reaction time (h)	Conv. [d] (%)	Product distribution (%) [a]			
				9	**10**	**11**	**12**
8a	$PtCl_2(PhCN)_2$ [a]	16	10	69	3	6	22
	$PtCl_2$ [b]	4	98	76	2	11	11
	$RhCl(PPh_3)_3$ [c]	2	71	23	10	40	27
8b	$PtCl_2(PhCN)_2$ [a]	16	–	–	–	–	–
	$PtCl_2$ [b]	96	4	100	–	–	–
	$RhCl(PPh_3)_3$ [c]	16	–	–	–	–	–
8c	$PtCl_2(PhCN)_2$ [a]	3	52	40	–	60	–
	$PtCl_2$ [b]	4	75	75	–	25	–
	$RhCl(PPh_3)_3$ [c]	3	44	19	–	42	39
8d	$PtCl_2(PhCN)_2$ [a]	16	–	–	–	–	–
	$PtCl_2$ [b]	96	–	–	–	–	–
8e	$PtCl_2(PhCN)_2$ [a]	10	31	100	–	–	–
	$PtCl_2$ [b]	24	3	100	–	–	–
	$RhCl(PPh_3)_3$ [c]	10	97	8	92	–	–

[a] 0.0036 mmol catalyst, 1.4 mmol substrate, 1.6 mmol Et_3SiH, in toluene at 65 °C under argon. [b] 0.01 mmol catalyst, 5 mmol substrate, 5.8 mmol Et_3SiH, without solvent at room temperature under argon. [c] 0.0017 mmol catalyst, 1.4 mmol substrate, 1.6 mmol Et_3SiH, in toluene at 65 °C under argon. [d] Mol reacted substrate/mol initial substrate. [e] Mol product/mol reacted substrate.

on hydrosilylation of some vinyl- and vinylidene-type olefins with $PtCl_2$, $PtCl_2(PhCN)_2$ and $RhCl(PPh_3)_3$ catalysts (Scheme 3, Table 5).

The vinylidene-type olefins: α-methyl-styrene **8b** and limonene **8d** virtually undergo no hydrosilylation under these conditions. The high reactivity of methyl methacrylate (Table 1,4 [1]) may be due to the effect of the carboxyl group. The selectivity is also poorer with these substrates except for 1-vinyl-2-pyrrolidinone **8e**, with which $PtCl_2(PhCN)_2$ gives the linear **9e** whereas $RhCl(PPh_3)_3$ gives the branched hydrosilylated derivative **10e**. With $PtCl_2$ the degree of conversion is very low, even though the precursor dissolves readily in 1-vinyl-2-pyrrolidinone (as in styrene [5]); probably coordination of the amido-group to the metal gives a relatively stable complex of low reactivity.

Experimental

Reagents

$PtCl_2(PhCN)_2$ and $Pt(PPh_3)_4$ were prepared as described previously [7,8]. $Pt(dppb)_2$ (dppb: 1,4-bis-diphenylphosphino-butane) and $Pt(diop)_2$ (diop: isopropylidene-2,3-dihydroxy-1,4-bis-diphenylphosphinobutane) were prepared by the method described for $Pt[CH_3C(CH_2PPh_2)_3]PR_3$ [9]. $RhCl(PPh_3)_3$ was purchased from Aldrich. $Pt(O_2)(dppb)$ was made by dissolving $Pt(dppb)_2$ in benzene in the presence of oxygen and filtering the white powder obtained.

Solvents were dried and distillled under argon, substrates were freshly distilled before use.

The ^1H-NMR spectra were recorded in $CDCl_3$ with TMS as internal standard on a Tesla BS 487C spectrometer at 80 mHz, ^{13}C-NMR spectra at 20.1 MHz and ^{31}P-NMR spectra at 32.2 MHz on a Varian CFT-20 spectrometer. Mass spectra were recorded with a JEOLCO MS 01 SG-2 spectrometer at 75 eV. GLC analyses were carried out on a Hewlett-Packard 5830A gas chromatograph fitted with a column coated with OV-1.

A typical hydrosilylation experiment

The catalyst was dissolved in the solvent (or in the absence of solvent in the substrate) under argon in a Schlenk-tube. The substrate, the hydrosilane, and if necessary oxygen (10 molar proportions relative to the catalyst) were added. The mixture was heated at the chosen temperature and the progress of the reaction monitored by GC.

Preparation of samples for ^{31}P-NMR

A mixture of 47.2 mg (0.1 mmol) $PtCl_2(PhCN)_2$ and 52.4 mg (0.2 mmol) of PPh_3 in 10 ml of toluene was stirred under argon in a Schlenk-tube until a white precipitate separated. Then 0.4 mmol of methyl methacrylate and 0.8 mmol of Et_3SiH were added and the mixture was heated at 110 °C. The complex dissolved within a few min. The mixture was then heated under argon atmosphere or in presence of added oxygen. After 2 h the mixture was concentrated in vacuum to ca. 0.5 ml and 1 ml of toluene-d_8 was added.

Characterization of the products

*1-Methoxy-2-methyl-1-triphenylsiloxy-prop-1-ene (**4a**, R = Ph)*

^{1}H NMR ($CDCl_3$): 1.46 (s, 6H, (CH_3)$_2$C=); 3.05 (s, 3H, OCH_3); 7.35 (m, 15H, Si(C_6H_5)$_3$).

*Methyl 2-methyl-3-triphenylsilyl-propionate (**2a**, R = Ph)*

^{1}H NMR ($CDCl_3$): 1.03 (d, J = 7.2 Hz, 3H, CH_3CH); 1.37 (dd, J = 6.8, 15 Hz, 1H, SiCH_aH$_b$CH); 1.83 (dd, J = 7, 15 Hz, 1H, SiCH$_a$$H_b$CH); 2.62 (m, 1H, CH); 3.17 (s, 3H, OCH_3); 7.35 (m, 15H, Si(C_6H_5)$_3$).

*Methyl 2-methyl-3-dimethylhydrosilyl-propionate (**2a**, R_3 = Me_2H)*

^{1}H NMR ($CDCl_3$): 0.4 (m, 6H, Si(CH_3)$_2$); 1.05 (m, 2H, SiCH_2CH); 1.17 (d, J = 7 Hz, 3H, CH_3); 2.67 (sx, J = 7 Hz, 1H, CH); 3.55 (m, 1H, SiH), 3.6 (s, 3H, OCH_3). ^{13}C NMR ($CDCl_3$): 2.34 (Si(CH_3)$_2$); 20.39 (SiCH$_2$CH); 23.82 (CH_3); 35.04 (CH); 51.66 (OCH_3); 177.13 (COOCH$_3$). MS: m/z (rel. intensity): 145 (80); 129 (110); 89 (1000); 75 (320); 70 (950); 59 (550); 42 (320).

*Methyl 2-methyl-2-dimethylhydrosilyl-propionate (**3a**, R_3 = Me_2H)*

^{1}H NMR ($CDCl_3$): 0.4 (m, 6H, Si(CH_3)$_2$); 1.16 (s, 6H, (CH_3)$_2$C); 3.55 (m, 1H, SiH); 3.57 (s, 3H, OCH_3). ^{13}C NMR ($CDCl_3$): 2.34 (Si(CH_3)$_2$); 18.96 (SiCCO); 21.16 (C(CH$_3$)$_2$); 51.40 (OCH_3); 177.10 (COOCH$_3$).

*1-Methoxy-2-methyl-1-dimethylhydrosiloxy-prop-1-ene (**4a**, R_3 = Me_2H)*

^{1}H NMR ($CDCl_3$): 0.42 (m, 6H, Si(CH_3)$_2$); 1.15 (s, 3H, CH_3C=); 1.22 (s, 3H, CH_3C=); 3.55 (m, 1H, SiH); 3.60 (s, 3H, OCH_3). ^{13}C NMR ($CDCl_3$): 2.34 (Si(CH_3)$_2$); 16.07 (CH$_3$C=); 16.77 (CH$_3$C=); 57.32 (OCH_3); 92.90 ((CH_3)$_2$$C$=C); 148.46 (=$C$(OCH$_3$)OSi).

*1-Methoxy-2-methyl-1-dimethylchlorosiloxy-prop-1-ene (**4a**, R_3 = Me_2Cl)*

^{1}H NMR ($CDCl_3$): 0.55 (s, 6H, Si(CH_3)$_2$); 1.48 (s, 3H, CH_3C=); 1.58 (s, 3H, CH_3C=); 3.45 (s, 3H, OCH_3). ^{13}C NMR ($CDCl_3$): 2.36 (Si(CH_3)$_2$); 16.08 (CH$_3$C=); 16.78 (CH$_3$C=); 57.34 (OCH_3); 92.90 ((CH_3)$_2$$C$=C); 148.52 (=$C$(OCH$_3$)OSi). MS: m/z (rel. intensity): 194 (200); 109 (100); 93 (450); 70 (1000); 55 (180).

*Methyl 2-methyl-3-trichlorosilyl-propionate (**2a**, R = Cl)*

^{1}H NMR ($CDCl_3$): 1.30 (d, J = 7 Hz, 3H, CH_3CH); 1.53 (dd, J = 7.2, 16 Hz, 1H, SiCH_aH$_b$CH); 1.95 (dd, J = 7, 16 Hz, 1H, SiCH$_a$$H_b$CH); 2.82 (m, 1H, CH); 3.65 (s, 3H, OCH_3). ^{13}C NMR ($CDCl_3$): 19.55 (CH$_3$CH); 28.49 (SiCH$_2$CH); 34.36 (CH$_3$$C$H); 52.07 ($OCH_3$); 175.48 ($C$OOCH$_3$).

*Methyl 3-triethylsilyl-propionate (**2b**)*

^{1}H NMR ($CDCl_3$): 0.65 (m, 6H, Si(CH_2CH$_3$)$_3$; 0.95 (m, 9H, Si(CH$_2$CH_3)$_3$); 1.25 (t, J = 7.5 Hz, 2H, SiCH_2CH$_2$); 2.32 (t, J = 7.5 Hz, 2H, SiCH$_2$CH_2); 3.70 (s, 3H, OCH_3). MS: m/z (rel. intensity): 173 (1000); 117 (780); 89 (400).

*Methyl 2-triethylsilyl-propionate (**3b**)*

^{1}H NMR (CDCl$_3$): 0.65 (m, 6H, $\mathrm{Si(C}H_2\mathrm{CH_3)_3}$); 0.95 (m, 9H, $\mathrm{Si(CH_2C}H_3\mathrm{)_3}$); 1.0 (m, 1H, CH); 1.51 (d, $J = 6.8$ Hz, 3H, $\mathrm{C}H_3\mathrm{CH}$); 3.57 (s, 3H, OCH$_3$). MS: m/z (rel. intensity): 202 (180); 173 (230); 159 (820); 117 (830); 103 (1000); 89 (530); 75 (710).

*Methyl 3-triethylsilyl-acrylate (**7b**)*

MS: m/z (rel. intensity): 171 (950); 143 (1000); 115 (230); 89 (100); 59 (160).

*Dimethyl (triethylsilylmethyl)-succinate (**2d**)*

^{1}H NMR (CDCl$_3$): 0.53 (m, 6H, $\mathrm{Si(C}H_2\mathrm{CH_3)_3}$); 0.90 (m, 9H, $\mathrm{Si(CH_2C}H_3\mathrm{)_3}$); 1.2 (m, 2H, $\mathrm{SiC}H_2\mathrm{CH}$); 2.68 (m, 3H, $\mathrm{CH_2C}H\mathrm{C}H_2$); 3.68 (s, 3H, OCH$_3$); 3.75 (s, 3H, OCH$_3$). ^{13}C NMR (CDCl$_3$): 3.35 ($\mathrm{Si(}C\mathrm{H_2CH_3)_3}$); 7.27 ($\mathrm{Si(CH_2}C\mathrm{H_3)_3}$); 14.76 ($\mathrm{Si}C\mathrm{H_2CH}$); 37.26 (CH); 39.26 ($\mathrm{CH}C\mathrm{H_2CO}$); 51.63 (OCH$_3$); 172.21 ($C\mathrm{OOCH_3}$); 176.35 ($C\mathrm{OOCH_3}$). MS: m/z (rel. intensity): 245 (900); 215 (150); 149 (390); 117 (1000); 89 (530); 87 (250); 69 (210); 59 (300).

*1-(Cyclohex-3-enyl)-2-triethylsilyl-ethane (**9c**)*

^{1}H NMR (CDCl$_3$): 0.57 (m, 6H, $\mathrm{Si(C}H_2\mathrm{CH_3)_3}$); 0.95 (m, 9H, $\mathrm{Si(CH_2C}H_3\mathrm{)_3}$); 1.1–2.5 (m, 11H, $(\mathrm{C}H_2)_5$, $\mathrm{C}H$); 5.70 (br. s, 2H, CH=CH). MS: m/z (rel. intensity): 195 (1000); 167 (750); 115 (350); 87 (920); 59 (310).

*1-(Cyclohex-3-enyl)-2-triethylsilyl-ethylene (**12c**)*

MS: m/z (rel. intensity) 193 (1000); 167 (420); 137 (190); 87 (250); 59 (220).

*1-(Pyrrolidin-2-on-1-yl)-2-triethylsilyl-ethane (**9e**)*

^{1}H NMR (CDCl$_3$): 0.6 (m, 6H, $\mathrm{Si(C}H_2\mathrm{CH_3)_3}$); 0.85 (m, 11H, $\mathrm{C}H_2\mathrm{Si(CH_2C}H_3\mathrm{)_3}$); 2.05 (m, 4H, $\mathrm{COC}H_2\mathrm{C}H_2$); 3.25 (m, 4H, $\mathrm{C}H_2\mathrm{NC}H_2$). ^{13}C NMR (CDCl$_3$): 1.0 ($\mathrm{Si(}C\mathrm{H_2CH_3)_3}$); 5.02 ($\mathrm{Si(CH_2}C\mathrm{H_3)_3}$); 7.98 (CH$_2$Si); 15.56 ($\mathrm{CH_2}C\mathrm{H_2CH_2}$); 28.91 ($\mathrm{CO}C\mathrm{H_2}$); 35.98 ($\mathrm{N}C\mathrm{H_2CH_2Si}$); 43.75 ($\mathrm{N}C\mathrm{H_2CH_2CH_2}$); 171.26 (CO).

*1-(Pyrrolidin-2-on-1-yl)-1-triethylsilyl-ethane (**10e**)*

^{1}H NMR (CDCl$_3$): 0.6 (m, 6H, $\mathrm{Si(C}H_2\mathrm{CH_3)_3}$); 0.85 (m, 9H, $\mathrm{Si(CH_2C}H_3\mathrm{)_3}$; 1.12 (d, $J = 7.2$ Hz, 3H, CH$_3$); 1.95 (m, 2H, $\mathrm{CH_2C}H_2\mathrm{CH_2}$); 2.22 (m, 2H, COCH$_2$); 3.27 (m, 2H, $\mathrm{NC}H_2\mathrm{CH_2}$); 3.78 (q, $J = 7.2$ Hz, 1H, CH).

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