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Hydrosilylation in the Presence of Platinum(II) and Rhodium(I) Complexes with Chloride and Trichlorostannate(II) Anions

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Abstract—Hydrosilylation of allyl butyl ether and acetophenone with 1,1,3,3-tetramethyldisiloxane in the presence of [Pt(LL')XY] and $[Rh(Ph_3P)_3X]$ (L, L' = cod, dmso, Py, Bn_2S, Ph_3P; X, *y* = Cl, $SnCl_3$) was studied.

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The modern trend in the development of the hydrosilylation reaction is characterized by a search for comparatively inexpensive and simple catalytic systems capable of yielding the target product in a short time with high selectivity. Recently, a large number of reports have been devoted to use of compounds of base metals for this purpose. However, complexes of platinum and rhodium still remain the most efficient catalysts and, therefore, are in the highest demand [1].

$SnCl_3^-$ is efficient in hydroformylation [2–4], alkoxycarbonylation [5], and hydrogenation and dehydrogenation [6–9] it exerts a high *trans*-influence and *trans*-effect, easily dissociates, and promotes coordination of olefins and stabilization of pentacoordinated transition states [5, 10–13]. At the same time, there are no unambiguous data on the effect of tin on the hydrosilylation process. For example, it is known the acetylenes are hydrosilylated in the presence of tin-containing cobalt carbonyls [14], and in situ addition of tin(II) chlorides to H_2PtCl_6 inhibits hydrosilylation [15].

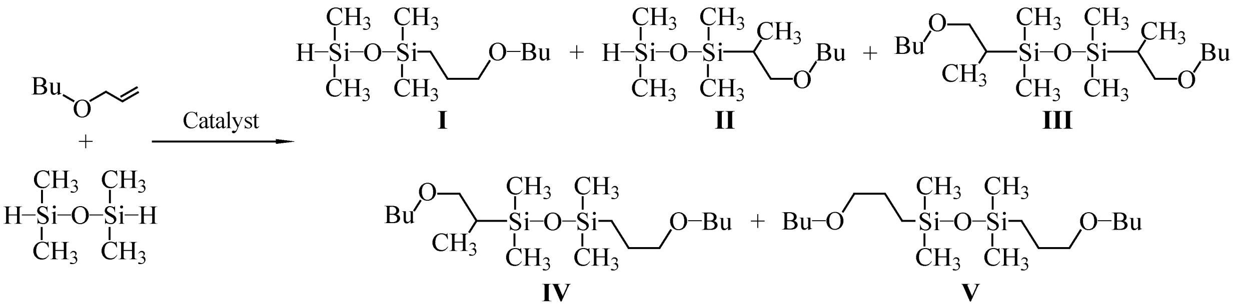
Therefore, the goal of our study was to examine the effect of the trichlorostannate(II) anion on the rate and selectivity of hydrosilylation of acetophenone and allyl butyl ether (AllOBu).

As catalysts were chosen cyclooctadiene, dimethylsulfoxide, dibenzyl sulfide, and triphenylphosphine chloride complexes of platinum(II) and Wilkinson' catalyst $[Rh(Ph_3P)_3Cl]$, which is due to the good understanding of their catalytic properties in hydrosilylation of a wide variety of substrates [1, 14, 16–21], and their trichlorostannate(II) anion derivatives. As the hydrosilylating agent was chosen 1,1,3,3-tetramethyldisiloxane (TMDS), which, on the one hand, is a model siloxane caoutchouc with terminal SiH groups and, on the other, possesses the most favorable combination of properties (stability in air, low boiling point, simple NMR spectrum), which enables a qualitative and quantitative analysis of the reaction.

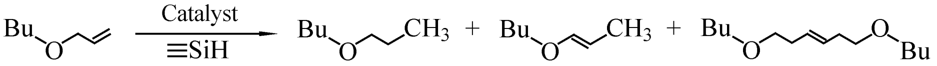
Allyl butyl ether. The hydrosilylation of allyl butyl ether with TMDS occurs in accordance with Farmer's and Markovnikov's rules (compounds **I** and **II**, respectively), with silicon predominantly added to the terminal carbon atom, in good agreement with published data on the hydrosilylation of allyl ethers [1, 22, 23] (Scheme 1).

The presence of two SiH groups in TMDS predetermines formation of double-addition products (compounds **III–V**). The maximum overall yield of siloxanes **III–V** is observed in the presence of cyclooctadiene complexes of platinum(II) (18 %); with *cis*-$[Pt(Bn_2S)_2Cl_2]$ or $[Pd(dmso)_2Cl_2]$, it reaches a value of 15%. The minimum yield of double-addition products is observed in the case of hydrosilylation in the presence of $[Rh(Ph_3P)_3SnCl_3]$ (1%).

Scheme 1.



Scheme 2.



Together with the hydrosilylation, there occur reduction, isomerization, and dimerization of AllOBu, and dehydrocondensation and disproportionation of TMDS (Scheme 2).

The main by-products of TMDS conversion are 1,1,3,3,5,5-hexamethyltrisiloxane (HMTS) and 1,1,1,3,3-pentamethyldisiloxane (transformations of TMDS are described in more detail in [21, 24]). In turn, HMTS enters into a reaction with AllOBu to give the corresponding products of addition in accordance with Farmer's and Markovnikov's rules, whose maximum amount is observed in the presence of [Pt(cod)X_2], *cis*-[Pt(Bn_2S)$_2$$Cl_2$], and *cis*-[Pt(dmso)$_2$$Cl_2$] (about 4%). It should be noted that no cyclosiloxanes are formed in hydrosilylation of AllOBu, in contrast to the case of hydrosilylation of vinylsiloxanes and α-methylstyrene [18, 21, 24].

The side processes accompanying the hydrosilylation depend both on the nature of a catalyst and on the reaction conditions. Isomerization of AllOBu occurs to a greater extent on [Pt(cod)Cl_2] and all dimethylsulfoxide and dibenzyl sulfide platinum catalysts (5–6% yield of propenyl butyl ether), whereas in the case of a trichlorostannate cyclooctadiene complex of platinum(II) or a trichlorostannate phosphine complex of rhodium(I), the yield of propenyl butyl ether is the lowest (0.3–0.5%). Propyl butyl ether is formed in the presence of [Pt(cod)Cl_2] (0.7% yield), whereas in the presence of both the chloride and trichlorostannate phosphine complexes of platinum(II) or rhodium(I), its yield is less than 0.1%.

A large amount of by-products of TMDS conversion is formed with all pyridine or phosphine complexes of platinum(II): in the early stage of the reaction (2–3 days), only 30–40% of the total conversion of TMDS is expended for the target reaction. In further course of the reaction, this parameter increases to 70–80% of the total conversion of TMDS, i.e., the disproportionation and dehydrocondensation of TMDS in the presence of pyridine or phosphine complexes of platinum(II) dominate over hydrosilylation from the very beginning. For the rest of the catalysts, the fraction of the target-related conversion of TMDS is originally larger (in 2–3 days, it is 75–85% of the total conversion of TMDS), but starts to decrease as the 85–90% conversion of AllOBu is reached (hydrosilylation comes to its end). This suggests that the hydrosilylation predominates over the rate of the side conversion of TMDS in the presence of cyclooctadiene, dimethylsulfoxide, and dibenzyl sulfide complexes of platinum(II) and phosphine complexes of rhodium(I).

The TMDS-induced competing hydrosilylation of propenyl butyl ether formed via isomerization of AllOBu must for the most part yield siloxane (II), which, in turn, can raise the total amount of siloxane (II). However, the hydrosilylation of internal C=C bonds occurs at a substantially slower rate, compared with that of terminal bonds [25–27]. and, in addition, there is no correlation between the yields of siloxane (II) and propenyl butyl ether. This suggests that the contribution of a parallel reaction, hydrosilylation of propenyl butyl ether, to the process of interaction between AllOBu and

TMDS is insignificant.

The ^1H NMR spectrum of the reaction mixture contain singlets and triplets of methyl groups at silicon atoms (δ_H 0.03–0.19 ppm) and of methyl groups unbound to silicon (δ_H 0.84–1.23 ppm); signals of $SiCH_2$ (δ_H 0.40–0.56 ppm), methylene protons (δ_H 1.29–1.67, 3.30–3.93, and 5.14–5.27 ppm), and methine protons (δ_H 5.85–5.95 ppm); and a characteristic multiplet of the SiH group (δ_H 4.7 ppm). However, application of the NMR technique to monitoring of the reaction course is exceedingly inconvenient, because most of signals of the starting compounds and the compounds being formed are superimposed. Only the following signals can be rather clearly distinguished (δ_H, ppm; *J*, Hz): $–CH=CH_2$ fragment of the starting compound [5.17 d (1H, CH_2, *J* 37.6), 5.22 d (1H, CH2, *J* 45.0), 5.89 dq (1/2H, CH, *J* 17.0, 5.6), and 5.92 dq (1/2H, CH, *J* 17.0, 5.8)], part of methylene protons of propenyl butyl ether [3.62 t (2H, OCH_2, *J* 6.6], and some groups of propyl butyl ether [1.22 dt (6H, CH_3, *J* 6.3, 1.3), 3.67 t (2H, OCH_2, *J* 6.2), and 3.71 t (2H, OCH_2, *J* 5.9)].

In the case under consideration, IR spectroscopy is inefficient because it gives no way of clearly determining the way in which silicon hydride groups are consumed and the amount of the products being formed.

The most convenient monitoring technique is gas chromatography. A typical chromatogram contains the following peaks (retention times, RT, min): PrOBu (3.3); TMDS (4.7); $HMe_2SiOSiMe_3$ (5.7); toluene (9.1, internal standard); AllOBu (9.8); $HMe_2SiOSi(Me_2)OSi(Me)_2H$ (10.3); propenyl butyl ether (11.1); $BuO(CH_2)_2–CH=CH–(CH_2)_2OBu$ (22.6); siloxanes: **II** (24.2), **I** (25.4), **IV** (39.4), **V** (38.7), and, in some cases, product **III** (38.7); and products of α- and β-addition of HMTS (32.9 and 33.3, respectively). In addition, the chromatogram shows unidentified peaks with retention times of 11.0, 13.1, 15.5, 22.1, 29.7, 29.8, and 46.9 min, which appear as a result of side transformations of TMDS (these peaks are observed in chromatograms of products formed in interaction between TMDS and catalysts without allyl butyl ether and are absent in chromatograms of products of interaction between allyl butyl ether and catalysts without TMDS).

The highest activity in the interaction of allyl butyl ether with TMDS is exhibited by $[Pt(cod)Cl_2]$; however, replacement of the chloride anion with the trichlorostannate anion markedly diminishes the catalytic activity of the complex (Table 1).

The catalytic activity of *cis*-$[Pt(Bn_2S)_2Cl_2]$ and *cis*-$[Pt(dmso)_2Cl_2]$ is lower than that of $[Pt(cod)Cl_2]$. Replacement of Cl with $SnCl_3^-$ in the dibenzyl sulfide catalyst makes it activity lower, but not so strongly as it occurs in hydrosilylation in the presence of a cyclooctadiene complex of platinum(II). At the same time, presence of $SnCl_3^-$ in the dimethylsulfoxide catalyst exerts a strong negative influence on its activity (Table 1).

The rate of hydrosilylation in the presence of *cis*-$[Pt(Py)_2Cl_2]$ and *cis*-$[Pt(Ph_3P)_2Cl_2]$ is rather low, with a lower activity exhibited by *cis*-$[Pt(Py)_2Cl_2]$. The activity of the pyridine catalyst can be raised by introduction of the trichlorostannate(II) anion, but this procedure is inefficient in the case of phosphine-type and other platinum catalysts (Table 1).

Thus, the activity of chloride platinum catalysts in the interaction of allyl butyl ether with TMDS decreases in the following order of ligands: cod > dmso > Bn_2S > Ph_3P > Py; for trichlorostannate complexes, the order is somewhat modified: cod > dmso > Bn_2S > Py > Ph_3P.

Wilkinson's catalyst exhibits a substantially higher activity in the interaction of allyl butyl ether with TMDS, compared with the phosphine complex of platinum(II); nevertheless, the activity of $[Rh(Ph_3P)Cl]$ is lower than that of the most active platinum catalyst, $[Pt(cod)Cl_2]$. $[Rh(Ph_3P)_3(SnCl_3)]$ is inferior to $[Rh(Ph_3P)_3Cl]$ in catalytic activity; however, the yield of the target product **I** was the highest (90.5%) in the presence of the former.

The hydrosilylation selectivity, i.e., the share of the hydrosilylation reaction in the observed processes of AllOBu conversion is, on average, 84–95% (Table 1). For *cis*-$[Pt(Py)_2Cl_2]$ and the phosphine complex of platinum(II) with the trichlorostannate(II) anion, the selectivity was fund to be below this range. The highest selectivity is observed when the reaction is performed in the presence of Wilkinson's catalyst or its trichlorostannate analog.

It is the most convenient to evaluate the selectivity of addition of silicon hydrides (TMDS and HMTS) to the C=C bond by the α-/β-addition ratio (Table 1). For almost all of the catalysts under consideration, the selectivity of β-addition is 96.5–97.5% and only slightly depends on the catalyst structure. The best result was obtained in the presence of a phosphine complex of rhodium(I) with the trichlorostannate anion (98%).

Table 1. Characteristics of allyl butyl ether hydrosilylation with 1,1,3,3-tetramethyldisiloxane. Temperature 20°C, All–O–Bu : TMDS = 1 : 1.5

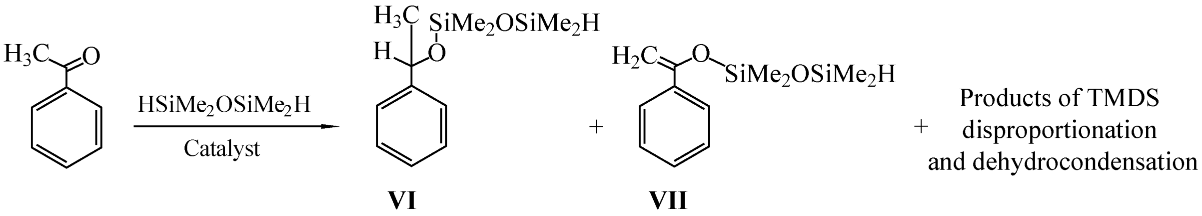
Catalyst	X	Time, days	AllOBu conversion, %	Yield, %			Selectivity,* %	α-/β- ratio
				I	**II**	**V**		
$[Rh(Ph_3P)_3X]$	Cl	4	97	89.0	2.2	1.0	95	3.3/96.7
		7	100	88.2	2.6	2.7	95	3.7/96.3
	$SnCl_3$	4	10	5.5	0	0.7	71	–
		7	98	90.5	1.7	0.7	95	2.0/98.0
$[Pt(Ph_3P)_2(Cl)X]$	Cl	2	19	13.5	0	2.0	82	–
		7	51	36.6	0.7	4.4	83	2.3/97.6
		10	70	56.2	1.2	5.2	91	2.7/97.2
	$SnCl_3$	2	15	12.3	0.6	0.2	88	3.9/96.1
		7	19	13.7	0.8	0.5	82	3.8/96.2
		10	25	14.4	1.1	2.0	76	4.1/95.9
$[Pt(dmso)_2(Cl)X]$	Cl	2	90	66.0	1.9	10.5	90	3.0/97.0
		7	99	65.9	2.0	11.3	85	3.2/96.8
		10	100	65.9	2.0	11.5	84	3.2/96.8
	$SnCl_3$	2	29	21.7	0.6	0.7	88	3.5/96.5
		7	88	69.9	1.9	9.0	93	3.4/96.6
		10	91	70.5	1.9	10.0	93	3.1/96.9
$[Pt(Bn_2S)_2(Cl)X]$	Cl	2	87	59.9	2.5	13.6	89	3.5/96.5
		15	98	64.5	2.0	17.5	87	2.8/97.2
	$SnCl_3$	2	74	54.7	0.8	7.9	86	2.3/97.7
$[Pt(Py)_2(Cl)X]$	Cl	2	2	0.3	0	0.8	59	–
		9	14	6.8	0.2	0.1	52	2.7/97.3
	$SnCl_3$	2	17	14.8	0.2	0.3	90	3.5/96.5
		10	56	45.9	1.2	3.7	92	3.4/96.6
$[Pt(cod)(Cl)X]$	Cl	2	97	68.9	1.9	12.3	89	3.1/96.9
		7	100	64.0	1.9	14.4	85	3.2/96.8
	$SnCl_3$	2	67	55.0	1.3	4.2	92	2.8/97.2
		7	100	74.4	1.8	12.1	93	3.2/96.8

[a] The selectivity is defined as the sum of hydrosilylation products divided by the sum of al products formed from the allyl ether.

Acetophenone. Interaction of acetophenone with TMDS in the presence of the catalysts considered above yields silyl ethers of 1-phenylethanol (**VI**) and enol (**VII**). Together with ethers **VI** and **VII**, the reaction mixture contains products of TMDS disproportionation and dehydrocondensation, similar to those produced in hydrosilylation of AllOBu, and, additionally, 1–3% octamethylcyclotetrasiloxane. This means that, in the given case, TMDS is partly converted to cyclosiloxanes, primarily because of the change in the temperature mode of the process (80°C instead of 20°C) (Scheme 3).

In the case under consideration, ^{1}H NMR more clearly reflects the conversion of the acetophenone reaction mixture, compared with that containing AllOBu, and, for a reaction performed in an excess of TMDS, enables easy determination of the conversion and the reaction selectivity [with or without toluene as internal standard (δ_H 2.36 ppm)]. In the course of the reaction, the area of the singlet associated with the methyl group of acetophenone (δ_H 2.60 ppm) decreases and there appear a characteristic doublet of the methyl group of product **VI** (δ_H 1.49 ppm, *J* 6.3 Hz) and two characteristic doublets of the methylene group of enol **VII** (δH 4.47 and 4.97 ppm, *J* 1.2 Hz). However, the

Scheme 3.



conversion and reaction selectivity calculated from the ^{1}H NMR data nearly always differ by 2–5% from the results of the chromatographic analysis, which is due to the higher sensitivity of the latter method.

A typical chromatogram of the reaction mixture contains the following peaks (RT, min): TMDS (2.67); HMTS (2.98); toluene (3.03); octamethylcyclotetrasiloxane (3.57); acetophenone (4.12); ether **VI** (5.33); enol **VII** (6.05); silyl ethers of 1-phenylethanol and enol, formed as a result of hydrosilylation of acetophenone with HMTS (5.57 and 6.76, respectively). The chromatogram also shows peaks we could not identify, with retention times of 4.43, 7.76, 8.82, 9.12, and 10.24 min.

The highest activity in the reaction under consideration was exhibited by Wilkinson's catalyst (Table 2), in good agreement with the known high activity of rhodium catalysts in hydrosilylation of ketones [1, 14, 26]. Replacement of the chloride anion with a trichlorostannate anion leads to a decrease in the reaction rate, similarly to the case in which AllOBu is hydrosilylated.

Table 2. Characteristics of acetophenone hydrosilylation with 1,1,3,3-tetramethyldisiloxane. Time 6 h, temperature 80°C, acetophenone : TMDS = 1 : 1.5

Catalyst	Acetophenone conversion	Addition selectivity
	%	
$[Pt(dmso)_2Cl_2]$	54	88
$[Pt(dmso)_2(SnCl_3)Cl]$	9	~100
$[Pt(Bn_2S)_2Cl_2]$	80	81
$[Pt(Bn_2S)_2(SnCl_3)Cl]$	56	~100
$[PtPy_2Cl_2]$	55	~100
$[PtPy_2(SnCl_3)Cl]$	10	94
$[Pt(cod)Cl_2]$	58	94
$[Pt(cod)(SnCl_3)Cl]$	20	~100
$[Pt(Ph_3P)_2Cl_2]$	4	~100
$[Pt(Ph_3P)_2(SnCl_3)Cl]$	3	~100
$[Rh(Ph_3P)_3Cl]$	87	98
$[Rh(Ph_3P)_3(SnCl_3)]$	72	97

Platinum catalysts are less active than$[Rh(Ph_3P)_3Cl]$. The best performance among these catalysts is exhibited by the dibenzyl sulfide complex of platinum(II) with a chloride anion (80%), and the minimum conversion of acetophenone is observed for phosphine complexes of platinum(II) (Table 2). The latter is presumably due to the long induction period (incomplete during the time interval under consideration) whose existence is characteristic of hydrosilylation in the presence of phosphine complexes of platinum(II) [16, 19].

The catalytic activities of $[Pt(cod)Cl_2]$, *cis*-$[Pt(Py)_2Cl_2]$, and *cis*-$[Pt(dmso)_2Cl_2$ are lower than that of *cis*-[Pt(Bn2S)2Cl2] (the corresponding conversions are 58, 55, 54, and 80%, respectively, Table 2). Presence of the trichlorostannate anion in the catalysts under consideration makes the hydrosilylation rate lower, which is especially strongly pronounced for the dimethylsulfoxide and pyridine complexes. It should be noted that the behavior of pyridine complexes of platinum(II) strongly depends on the nature of reactants: they exhibit zero activity in siloxane systems [16, 18, 28], are the most active in hydrosilylation of α-methylstyrene [21], and their activity additionally depends on the nature of the anionic ligand in the case of acetophenone and AllOBu.

Thus, the activity of the platinum catalysts in hydrosilylation of acetophenone with TMDS differs from that in the reaction of allyl butyl ether with TMDS and decreases in the following order of ligands: $Bn_2S >> cod > Py \approx dmso > Ph_3P; Cl > SnCl_3^-$.

The selectivity of hydrosilylation in the presence of the catalysts under consideration was found to be sufficiently high, which is primarily due to the chosen conditions: aerobic hydrosilylation without any special pretreatment of solvents and reactants, rather than to the structure of the catalysts. In this case, we have rather readily occurring hydrolysis of enol silyl ether, i.e., regeneration of acetophenone [1, 29, 30], which positively affects the selectivity of the process. It can, however, be easily noticed that the selectivity of hydrosilylation in the presence of chloride complexes exceeds that for catalysts with the trichlorostannate(II) anion (Table 2). The only exception is the pyridine complex of platinum(II), for which the “anomalous” behavior remains an empirical factor so far.

EXPERIMENTAL

^{1}H NMR spectra were recorded on a Bruker WM-400 instrument with a storage unit at a working frequency of 400 (^{1}H) Hz in $CDCl_3$ with internal deuterium lock and no additional references.

The reaction mixture was subjected to a gas-chromatographic analysis on an Agilent 7890 A chromatograph with a katharometer: DB-1 column (30 m × 0.32 mm); carrier-gas, helium (pressure 283.93 Pa [5.93 psi]); evaporator temperature 250°C, detector temperature 255°C; sample volume 1 μl; flow split ratio 250 : 1. In the case of AllOBu, the column was kept at a temperature of 70°C for 5 min, heated to 200°C at a rate of 5 deg min^{-1} and then to 225°C at 25 deg min^{-1}, and kept at 225°C for 16 min; in the case of acetophenone, it was heated from 150 to 200°C at 15 deg min^{-1} and kept at 225°C for 35 min. Calculations and peak assignment were made relative to signals of individual compounds and toluene.

The hydrosilylation was performed in sealed ampules at a catalyst concentration of $(1.0–1.5) \times 10^{-6}$ M. The first to be poured-in was the less volatile component of the mixture, a catalyst dissolved in methylene chloride was added at the end. The AllOBu (acetophenone) : TMDS : toluene molar ratio was 1 : 1.5 : 0.25. The reaction course was monitored by ^{1}H NMR spectroscopy (acetophenone) and gas chromatography (AllOBu and acetophenone). The conversion of AllOBu and acetophenone was determined relative to toluene. The reaction selectivity was found as the sum of hydrosilylation products divided by the sum of all products formed from allyl butyl ether for AllOBu, and as the ratio between ethers **VI** and **VII** for acetophenone.

In our experiments, we used TMDS purchased from Acros and allyl butyl ether from Aldrich; acetophenone, methylene chloride, and toluene were of chemically pure grade. The chloride platinum and rhodium catalysts were synthesized by methods described in [31, 32], and the platinum(II) and rhodium(I) complexes with the trichlorostannate anion, by those reported in [33, 34].

CONCLUSIONS

(1) The highest activity in the interaction between allyl butyl ether and 1,1,3,3-tetramethyldisiloxane is exhibited by the $[Pt(cod)Cl_2]$ catalyst; however, the maximum yield of the target product is observed in the presence of $[Rh(Ph_3P)_3(SnCl_3)]$.

(2) The hydrosilylation of AllOBu is accompanied by intense disproportionation and dehydrocondensation of TMDS and reduction, isomerization, and dimerization of AllOBu; in the case of hydrosilylation of acetophenone, the highest activity is exhibited by$[Rh(Ph_3P)_3Cl]$.

(3) In most cases, the rate of hydrosilylation in the presence of platinum(II) and rhodium(I) complexes with the trichlorostannate anion is lower than that on chloride catalysts, i.e., the increase in the *trans*-effect and *trans*-influence adversely affects the hydrosilylation, presumably via stabilization of transition states.

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