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Review

SILYLATION OF HYDROXYL-CONTAINING COMPOUNDS WITH ARYL- AND HETEROARYL-HYDROSILANES IN THE PRESENCE OF AMINES *

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Introduction

The dehydrocondensation of hydrosilanes with alcohols and other hydroxyl-containing compounds, a well-known reaction in organosilicon chemistry, is generally carried out in the presence of catalysts [1,2]. Surprisingly, amines, being readily available and sometimes effective as catalysts, have been only rarely applied [3–15]. At the same time, such popular catalysts as alkali, alkali metal alkoxides, metal halides, acids and complexes of Group VIII metals are not suitable for the dehydrocondensation of furyl- and thienyl-silanes because of the side reactions [16,17].

We have shown that the dehydrocondensation of aryl-, furyl- and thienyl-silanes with alcohols [17–25], as well as with phenols [26,27], oximes [28,29] and carboxylic acids [17,28,30], can be carried out satisfactorily in the presence of amines even when only a single Si–H bond is present in the silane. The reactions with amino alcohols are autocatalytic [17,19–24,31,32].

Alcohols

We have studied the dehydrocondensation of aryl- and heteroaryl-silanes with alcohols in the presence of amines [17,25]:

$$RR'_2SiH + HOR'' \xrightarrow[-H_2]{amine} RR'_2SiOR''$$

where R = alkyl, aryl, thienyl, furyl; R′ = aryl, thienyl, furyl, naphthyl.

Dimethylaryl(heteroaryl)silanes are unreactive towards ethanol at room temperature and when heating to 100°C, but the yields of di- and tri-heteroarylalkoxysilanes and triphenylethoxysilane in this reaction amount to 80%. Side reactions, such as cleavage of the furyl or thienyl ring from the hydrosilane molecule (either in the presence of alkali or of alkali metal alkoxides) and hydrosilylation of the multiple bonds in unsaturated alcohols (when hexachloroplatinic acid is present), are not observed in this case. The only product resulting from the reaction of methyldi-2-

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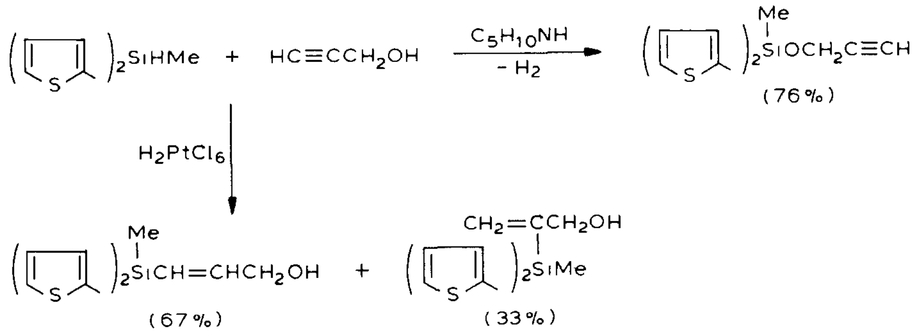
* Dedicated to Prof. M. Kumada.

thienylsilane with propargyl alcohol in the presence of piperidine is methyldi-2-thienyl propargyloxysilane. When chloroplatinic acid is present, the products of hydrosilylation of the multiple bond in the propargyl alcohol are formed as well, apart from the dehydrocondensation product [17]:



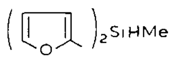
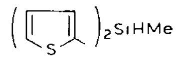
Catalysts and solvents [25]

No reaction of methyldi-2-furyl-, methyldi-2-thienyl- and triethoxy-silane with alcohols was observed in the absence of catalysts, either in dimethylformamide or in the absence of solvent. Equally, dehydrocondensation failed to occur in pyridine or *N,N*-diethylaniline (pK_a 5.23 and 6.61, respectively). Hydrogen evolution was very slow in the reaction carried out in *N*-allylmorpholine (pK_a 7.02). A further rise in amine basicity increased the reaction rate (Table 1).

Piperidine is the most efficient catalyst. However, 2-methyl-, *N*-methyl- and 2,6-dimethyl-piperidine are significantly weaker catalysts than piperidine ($10^2k(33°)$ in the reaction with methyldi-2-thienyl silane being 0.1, 0.042 and 0.017 l mol^{-1} min^{-1}, respectively). If we assume that the role of amine in the reaction is to facilitate hydrogen elimination due to OH ··· N bonding, it would be expected that a stronger proton acceptor would also be a stronger catalyst (Table 1), whereas a reduction in reaction rate in the last three cases is explained by the steric hindrance during intermediate formation.

TABLE 1

DEPENDENCE OF THE RATE OF DEHYDROCONDENSATION OF HYDROSILANES AND ETHANOL AT 33° C ON THE NATURE OF CATALYST

No.	Amine	pK_a	10^2k l mol^{-1} min^{-1}	
				
1	*N*-Propargylpiperidine	8.11	0.015	0.009
2	*N*-Allylpiperidine	9.50	0.069	0.027
3	*N*-Methylpiperidine	9.94	0.078	0.042
4	Triethylamine	10.76	0.300	0.11
5	Piperidine	11.21	5.26	1.98

A study of the dehydrocondensation of methyldi-2-thienylsilane with ethanol in various solvents (with 10 mol% added piperidine) demonstrated an enhanced reaction rate in polar solvents with good solvation properties (e.g. dimethylformamide, pyridine).

Influence of alcohol structure [25]

Examination of the influence of the alcohol structure on the rate of dehydrocondensation has shown that the reaction rate grows with increasing −*I*-effect of the substituents in the alcohol molecule. The logarithms of the rate constants plotted against the Taft's constants of the substituents give two straight lines, one for electron-withdrawing and the other for electron-donating substituents, (Fig. 1), which are described by the Taft's equation (Table 2).

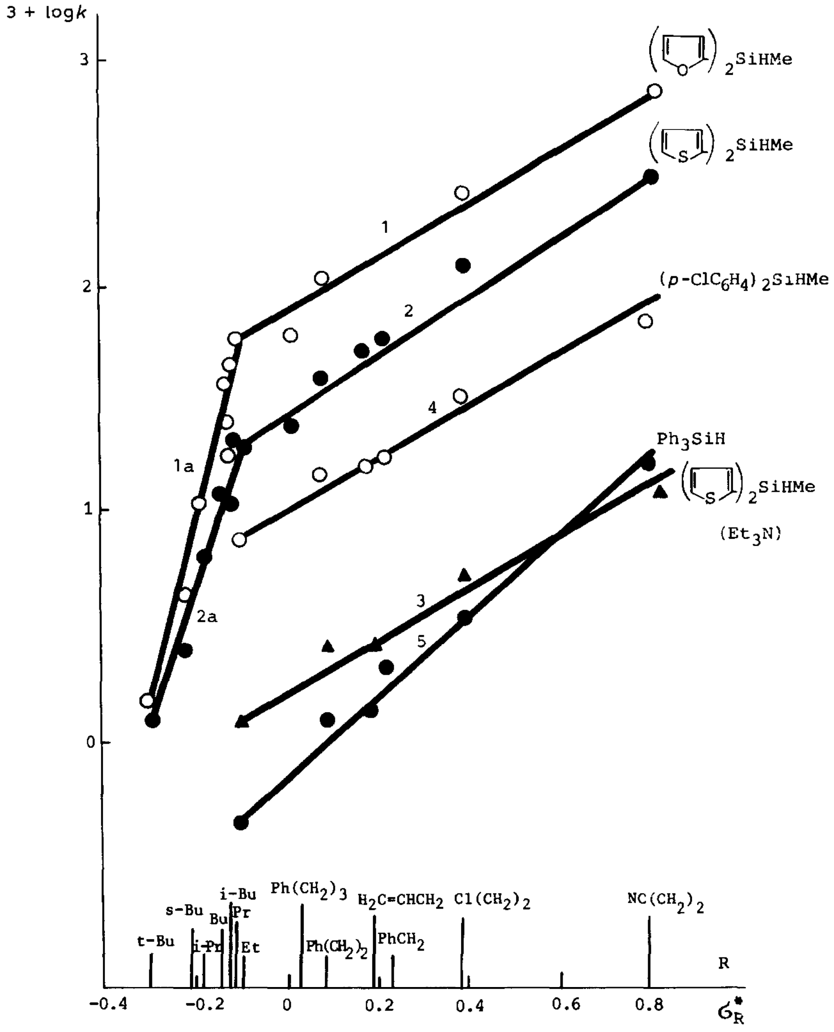
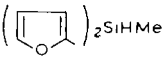
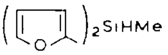
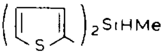
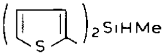
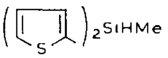


Fig. 1. Dependence of the rate of dehydrocondensation of hydrosilanes and alcohols in the presence of piperidine on R in the alcohols ROH.

TABLE 2

CORRELATION PARAMETERS ESTIMATED FROM THE EQUATION $\log k = \log k_0 + \rho\sigma^*$ (rate constants estimated at 33°C)

Silane	Line [a]	Equation terms				
		$\log k_0$	ρ	r	s	n [b]
	1	−1.19	1.32	0.979	0.007	6
	1a	−0.5	8.09	0.981	0.01	7
	2	−1.55	1.33	0.964	0.01	7
	2a	−1.06	6.53	0.980	0.017	7
	3	−2.77	1.07	0.983	0.005	5
$(p\text{-ClC}_6H_4)_2SiHMe$	4	−1.97	1.02	0.995	0.001	6
Ph_3SiH	5	−3.13	1.73	0.991	0.005	6

[a] In Fig. 1. [b] Points used in plot.

A two-factor correlation was undertaken, describing both the inductive and steric effects of the electron-donating substituents:

$$\log k = -0.61 + 5.72\,\sigma^* + 0.29\,E_s^0;\ r = 0.989,\ s = 0.1;$$
(line 1a)

$$\log k = -1.14 + 4.82\,\sigma^* + 0.21\,E_s^0;\ r = 0.987,\ s = 0.1.$$
(line 2a)

Amino alcohols

We have found that the dehydrocondensation of aryl- and heteroaryl-silanes with amino alcohols proceeds in the absence of catalyst, and moreover, the aminoalkoxysilanes are formed at 70% yield [17]:

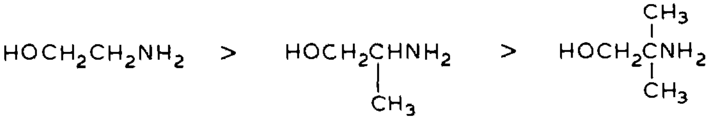
$$\geqslant SiH + HOCH_2CH_2NR_2 \xrightarrow[-H_2]{} \geqslant SiOCH_2CH_2NR_2$$

A survey of the accumulated experimental evidence on the kinetics of the reaction of hydrosilanes with amino alcohols [32] indicates that if the amino group is assumed to withdraw the proton from the hydroxyl group, O–H ··· N, then the reaction rate as a function of amino alcohol is determined by a set of mutually related factors: (1) the −*I*-effect of the amino group enhancing the reaction due to electron withdrawal from the O–H bond; (2) the proton-acceptor properties of the nitrogen atom, determined by the electronic and steric effects of the substituents bonded to the nitrogen; (3) the −*I*-effect of the hydroxyl group retarding the reaction due to reduced proton-acceptor properties of the nitrogen atom; (4) the stability of intra- and inter-molecular hydrogen bonds; (5) the steric effects preventing solvation and the formation of an intermediate complex with the silane.

The rate of dehydrocondensation of $HO(CH_2)_nNH_2$ amino alcohols with methyldi-2-thienylsilane rises with increasing distance between the amino and hydroxyl groups, $10^2k(33°)$ being equal to 17.7, 20.5 and 31.2 l mol^{-1} min^{-1} at $n = 2$, 3 and 4, respectively.

The basicity of amino alcohols and the stability of the intramolecular hydrogen bonds O–H ··· N increase in the same direction.

With substitution of the hydrogen atoms of the methylene group α to the nitrogen atom with methyl groups, the rate of reaction with methyldi-2-thienylsilane decreases in the order:



$$HOCH_2CH_2NH_2 > HOCH_2CH(CH_3)NH_2 > HOCH_2C(CH_3)_2NH_2$$

17.7 12.4 3.8 ($10^2k(33°)$ (lmol^{-1} min^{-1}))

This is accounted for by the steric influence of the methyl groups prevailing over the increased proton-acceptor properties of the nitrogen atom.

The rate of dehydrocondensation with methyldi-2-thienylsilane drops from alcohols with a primary amino group to those with a secondary or a tertiary one: $HOCH_2CH_2NH_2 > HOCH_2CH_2NHR > HOCH_2CH_2NR_2$ (R = CH_3, C_2H_5, C_4H_9).

In this case, a crucial role may be attributed also to steric factors preventing the formation of an intermediate complex with the silane.

Dehydrocondensation of hydrosilanes with amino alcohols is facilitated when the dielectric constant and the dipole moment of the solvent are increased (e.g. by using dimethylformamide or pyridine).

Phenols

Aryl- and heteroaryl-silanes readily undergo dehydrocondensation with phenols in the presence of amines to give aroxysilanes [17]:

$$\geq SiH + HOPh \xrightarrow[-H_2]{C_5H_{10}NH} \geq SiOPh$$

In contrast to ethanol, phenol reacts not only with methyldi-2-thienyl-, but also with dimethyl-2-thienyl-silane (in 63 and 42% yields, respectively).

A study of the relationship between the phenol structure and the rate of reaction has revealed that the rate rises with decreasing +*I*-effect of the substituents (Fig. 2), and there is a linear correlation between the logarithms of the rate constants and the inductive constants of the substituents: $\log k = 0.086 + 2.57\ \sigma_p$; $r = 0.981$, $s = 0.1$.

The *p*-nitrophenol reacting at a slower rate is an exception to the rule. It is a stronger acid than the other phenols and it behaves similarly to carboxylic acids when the silicon atom is attacked by the anion whose reactivity is hindered by the electron-withdrawing nitro group.

Phenol reacts faster than phenyl-substituted alcohols, and an increase of the distance of the phenyl group from the hydroxyl leads to a reduction in the reaction rate in the series:

$$PhOH > PhCH_2OH > Ph(CH_2)_2OH > Ph(CH_2)_3OH$$

12.2 5.4 3.6 2.3 ($10^2k(33°)$ (lmol^{-1} min^{-1}))

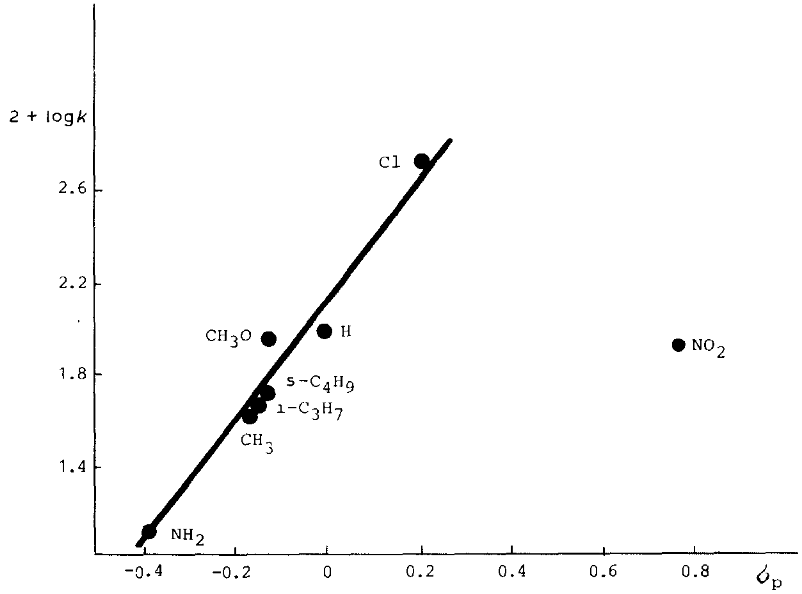


Fig. 2. Dependence on R of the rate of reaction of methyldi-2-thienylsilane with phenols p-RC_6H_4OH in the presence of piperidine.

Oximes

We have found that piperidine is useful for the silylation of aldoximes and ketoximes with aryl- and heteroaryl-silanes [28,29], the reaction proceeding without

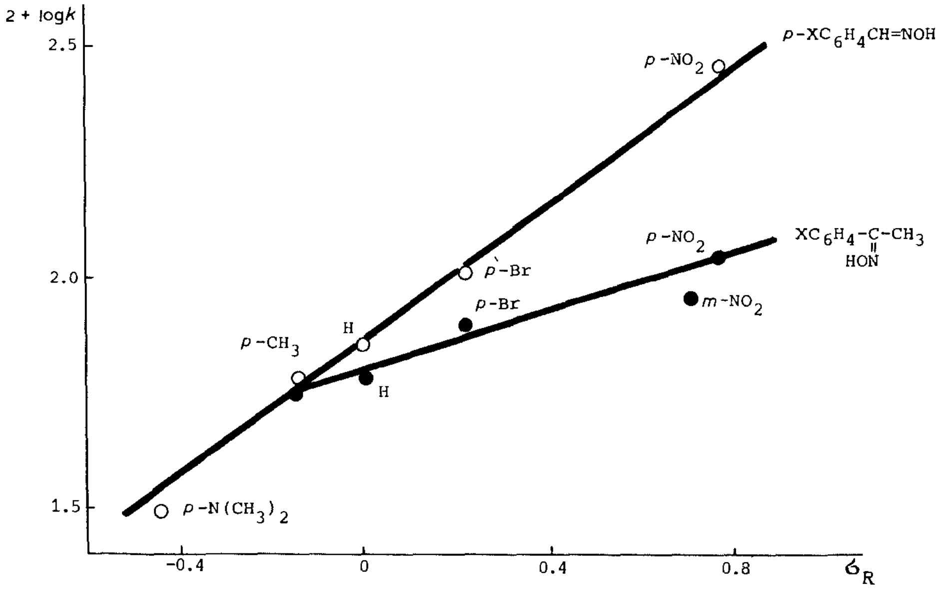


Fig. 3. Dependence on their structure of the rate of dehydrocondensation of oximes with methyldi-2-thienylsilane in the presence of piperidine.

the side effects of reduction, accompanied by the cleavage of the N–O bond in oximes, which occurs in the presence of hexachloroplatinic acid or zinc chloride [33–34]:

$$\geqslant\text{SiH} + \text{HON=CRR}' \xrightarrow[-\text{H}_2]{\text{C}_5\text{H}_{10}\text{NH}} \geqslant\text{SiON=CRR}'$$

In the reaction of dimethyl-2-thienyl-, methyldi-2-thienyl- and tri-2-thienyl-silanes with acetone oxime in the presence of piperidine the yields are 41, 63 and 69%, respectively [29].

The rate of dehydrocondensation of benzaldehyde oxime, and of its derivatives, with methyldi-2-thienylsilane rises with the introduction of electron-acceptor substituents into the benzene ring (Fig. 3) [29]: $\log k = 1.83 + 0.83\ \sigma_p$; $r = 0.995$, $s = 0.3$.

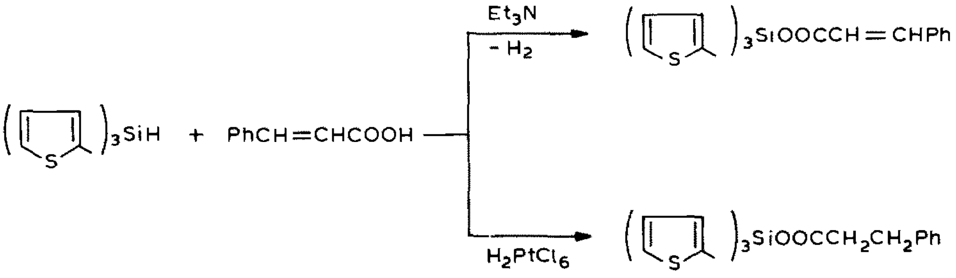
It has been noted that the dehydrocondensation of the oximes of acetophenone derivatives (substituted in the benzene ring) with methyldi-2-thienylsilane is also facilitated by electron-withdrawing substituents: $\log k = 1.82 + 0.25\ \sigma_p$; $r = 0.951$, $s = 0.13$.

The oximes of benzaldehyde derivatives are more sensitive to substituent effects and are more reactive than the oximes of acetophenone derivatives ($\rho = 0.83$ and 0.25, respectively).

Carboxylic acids

Amines catalyse the reaction of aryl- and heteroaryl-silanes with carboxylic acids, leading to acyloxysilanes [17,28,30]. The yields of acetoxysilanes in the reaction of R_nSiHMe_{3-n} silanes (R = 2-thienyl, $n = 1$–3) with acetic acid in the presence of triethylamine are 53, 66 and 79%, respectively, with n increasing from 1 to 3 [17].

In the presence of triethylamine, tri-2-thienylsilane reacts with cinnamic acid to afford only the dehydrocondensation product, while with hexachloroplatinic acid present the multiple bonds in the cinnamic acid undergo hydrogenation [17]:



A study of the kinetics of dehydrocondensation of methyldi-2-thienylsilane with a number of benzoic acids has shown that, in contrast to alcohols, phenols and oximes undergoing dehydrocondensation, the reaction rate falls with increasing $-I$-effect of the substituents in the acid molecule [30]. The plot of the logarithms of rate constants against σ_p-constants shows a linear correlation (Fig. 4): $\log k = -1.14 - 0.68\ \sigma_p$; $r = 0.975$, $s = 0.3$.

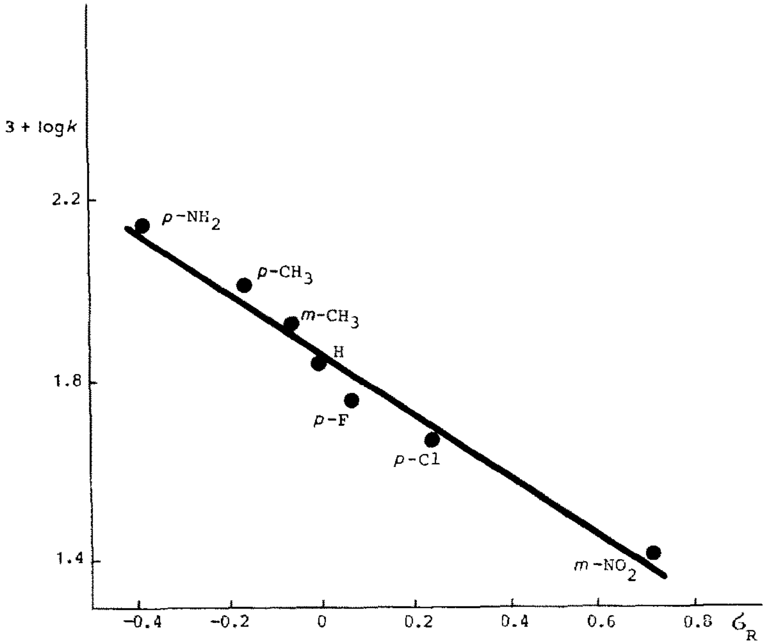


Fig. 4. Dependence on R of the rate of dehydrocondensation of methyldi-2-thienylsilane with the benzoic acids RC_6H_4COOH in the presence of piperidine.

The influence of silane structure

The rate of hydrosilane dehydrocondensation with alcohols and amino alcohols, being a function of the radical at the silicon atom, decreases in the order: 2-furyl > 2-thienyl > phenyl > α-naphthyl > 2-(4,5-dihydrofuryl) > 3-furyl [35–37]. The reaction rate and the product yield increase with increase in the number of aryl and heteroaryl groups in the silane molecule.

The elevated reactivity of hydrosilanes in the reaction with propargyl alcohol and 3-aminopropanol are only qualitatively related to the increase in stretching frequencies ν(Si–H) and in some cases also to shifts of ^{1}H(Si) NMR signals to lower fields and ^{29}Si signals to higher fields.

The ^1J(Si–H) spin–spin coupling constants are more sensitive to changes in hydrosilane structure than the ^{1}H(Si) chemical shifts and correlate with the sum of the inductive constants of the substituents at the silicon atom: $^1J(\text{Si–H}) = 184.4 + 7.9\ \Sigma\sigma_R^\star$; $r = 0.967$, $s = 0.1$ (Table 3, compounds 1–3, 5–14).

The logarithms of the rate constants for the reaction of aryl- and heteroaryl-silanes with propargyl alcohol correlate fairly well with ^1J(Si–H) values [35]: $\log k = -33.5 + 0.17\ ^1J(\text{Si–H})$; $r = 0.975$, $s = 0.1$.

Some deviation is possible due to the increase in the contribution of the steric factor in response to the growing size of the alkyl group.

As ^1J(Si–H) shows a correlation with the sum of Taft's inductive constants the rate of alcoholysis of aryl- and heteroaryl-silanes appears to rise with the increasing $-I$-effect of the substituents at the silicon atom, the effect of p_π–d_π interaction being absent in this case. This is also supported by the linear relationship between

TABLE 3

THE RATE OF REACTION OF HYDROSILANES WITH PROPARGYL ALCOHOL AND WITH 3-AMINOPROPANOL AT 33° C

No.	Silane[a]	$\Sigma\sigma_R^*$	ν(SiH) (cm^{-1})	δ(H–Si) (ppm)	^1J(Si–H), (Hz)	δ(^{29}Si) (ppm)	$10^2 k$ (l mol^{-1} min^{-1})	
							$HC{\equiv}CCH_2OH$[b]	$NH_2(CH_2)_3OH$
1	Ph_2SiHMe	1.2	2123	5.08	194.3	−17.56	0.075	0.18
2	$(2\text{-}C_4H_3S)SiHMe_2$	1.31	2132	4.59	194.3	−23.19	0.18	0.1
3	$(2\text{-}C_4H_3O)SiHMe_2$	1.5[c]	2142	4.41	195.0	−28.49	0.136	0.116
4	$(3\text{-}C_4H_3O)_2SiHMe$	–	2150	4.84	198.6	−36.80	0.43	0.44
5	Ph_3SiH	1.8	2126	5.53	199.5	−18.01	0.55	4.23
6	$(2\text{-}C_4H_3S)SiHPhMe$	1.91	2145	5.10	200.7	−29.56	1.24	1.53
7	$(2\text{-}C_4H_3S)SiHNpMe$[d]	2.05	2145	5.50	200.7	−18.03	0.91	2.41
8	$(2\text{-}C_4H_3O)SiHPhMe$	2.1	2150	–	201.3	−25.6	1.75	1.42
9	$(2\text{-}C_4H_3S)_2SiHMe$	2.62	2150	5.20	203.1	−31.29	11.3	20.5
10	$(2\text{-}C_4H_3S)SiHPh_2$	2.51	2145	5.55	205.7	−25.79	7.06	22.6
11	$(2\text{-}C_4H_3O)_2SiHBu$	2.87	2160	4.88	205.7	−39.39	8.83	–
12	$(2\text{-}C_4H_3O)_2SiHEt$	2.9	2160	4.86	206	−37.25	12.4	–
13	$(2\text{-}C_4H_3O)_2SiHMe$	3.0	2165	4.94	208.9	−42.23	28.4	49.3
14	$(2\text{-}C_4H_3O)(2\text{-}C_4H_3S)$-SiHMe	2.81	2160	5.07	209	−36.74	22.9	38.4

[a] C_4H_3S = thienyl, C_4H_3O = furyl. [b] In the presence of piperidine. [c] The value of σ^* for 2-furyl was taken from ref. 41. [d] Np = 1-naphthyl.

the logarithm of the reaction rate and the sum of the Taft's constants of the substituents at the silicon atom in hydrosilanes: $\log k = -2.72 + 1.37\ \Sigma\sigma_R^*$; $r = 0.982$, $s = 0.05$ (Table 3, compounds 1–14).

In the case of 3-aminopropanol the correlation between the reaction rate and the sum of inductive constants is not so good [36]: $\log k = -2.81 + 1.55\ \Sigma\sigma_R^*$; $r = 0.957$, $s = 0.35$ (Table 3, compounds 1–10, 13, 14).

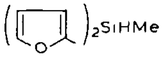
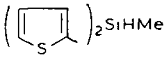
Certain differences in the hydrosilane reactivity observed in their dehydrocondensation with alcohols and amino alcohols, and a poorer correlation between the logarithms of the rate constants of amino alkanols and the sum of the Taft's constants of the substitutients at the silicon atom, suggest different structures for the intermediate complexes. This can be explained by the fact that in the case of amino alcohols both intra- and inter-molecular OH ··· N bonding may occur, as well as the possibililty that the aminoalkoxysilane obtained in the reaction may act as a catalyst.

It must be noted that in the presence of amines not only aryl- and heteroaryl-silanes, but also alkoxysilanes, hydrosiloxanes and even alkylsilanes, if they contain sufficiently strong electron-acceptor substituents (e.g. CF_3), undergo dehydrocondensation with alcohols [37–40]. The silylation pattern of alcohols with these silanes is the same as in the case of aryl- and heteroaryl-silanes. The reactivity of hydrosilanes in dehydrocondensation with phenol, acetophenone oxime and acetic acid (in the presence of piperidine) increases with the rise in the electron-acceptor properties of substituents at the silicon atom (Table 4), which corresponds to increased values of Si–H stretching frequencies and ^1J(Si–H) spin–spin coupling constants [37].

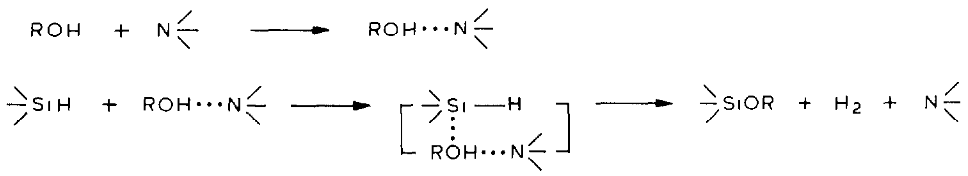
Our findings permit the assumption that the dehydrocondensation of aryl-, furyl- and thienyl-silanes with alcohols, phenols and oximes in the presence of amines, and

TABLE 4

REACTIVITY OF HYDROSILANES IN THE REACTIONS WITH PHENOL, ACETOPHENONE OXIME AND ACETIC ACID AT 33 °C IN THE PRESENCE OF PIPERIDINE

Silane	$\Sigma\sigma_R^*$	ν(Si–H) (cm^{-1})	^1J(Si–H) (Hz)	$10^3 k$ (l mol^{-1} min^{-1})		
				C_6H_5OH	$C_6H_5-\underset{\underset{HON}{\parallel}}{C}-CH_3$	CH_3COOH
	3.0	2165	208.9	–	460	53.2
	2.62	2150	203.1	121.7	440	47.6
(p-$ClC_6H_4)_2SiHMe$	1.74	2145	197.5	14.8	27.5	7.1
Ph_2SiHMe	1.2	2123	194.3	2.2	3.7	0.98

with amino alcohols (amino alcohols themselves acts as catalysts), proceeds via a molecular mechanism with an intermediate complex being formed which contains a five-coordinated silicon atom:



The rate-determining formation of the intermediate is facilitated by the electron-withdrawing substituents in the silane molecule which, by increasing the positive charge on the silicon atom, contribute to the Si ··· O coordination outweighing the p_π–d_π interaction of the aryl and heteroaryl substituents with the silicon atom.

This mechanism for dehydrocondensation is also corroborated by the kinetic isotope effects in the reaction of methyldi-2-thienylsilane with ethanol (k(SiH)/k(SiD) = 1.65; k(OH)/k(OD) = 1.35).

Electron-acceptor substituents in the molecules of alcohol, phenol and oxime, and the increase in the proton-acceptor properties of the amine, facilitate the elimination of proton from the hydroxyl group, leading to the formation of a molecular complex, ROH ··· N, between the amine and alcohol, phenol or oxime. In the case of carboxylic acids and p-nitrophenol the ionic complex RO^- ··· $^+HN\langle$ is formed, the silicon atom undergoing nucleophilic attack by the anion whose reactivity is reduced by the electron-acceptor substituents in the acid molecule.

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