

## VARIABLE ELECTRONIC EFFECT OF SILYL GROUPS ATTACHED TO OXYGEN\*

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The oxygen basicity in the  $R_1R_2R_3SiOR$  compounds with  $R_1$ ,  $R_2$ , and  $R_3$  substituents being both electron donors and electron acceptors and  $R = C_2H_5$  and  $Cl_3CCH_2$  was determined from the IR spectra of hydrogen bonds of phenol interacting with the  $R_1R_2R_3SiOR$  and an approximate  $\sigma^*$  constants of  $R_1R_2R_3Si$  groups in  $R_1R_2R_3SiOC_2H_5$  were estimated from the  $\Delta\nu(OH)$  vs  $\sigma^*(R)$  relationship valid for  $R-O-C_2H_5$  compounds. The  $R_1R_2R_3Si$  groups in  $R_1R_2R_3SiOCH_2CCl_3$  have been revealed to be forced by the  $Cl_3CCH_2$  group to donate electrons to the oxygen, and their enormous polarizability was attributed to mutual polarizability effect of the  $R_1$ ,  $R_2$ , and  $R_3$  substituents.

The electronic substituent parameter, very originally thought to be dependent solely on a substituent's nature and character of its bonding, is now becoming considered to be affected also by nature of a remote molecular part or by a substitution in it, of which the necessity of introduction of  $\sigma^n$  (or  $\sigma^0$ ),  $\sigma^+$  and  $\sigma^-$  is the best evidence<sup>1-3</sup>. The electronic effect of a substituent is now suggested<sup>4-6</sup> to be a blend of polar and polarizability effect and the latter component can be presumed (ref.<sup>7</sup> and refs therein) to be responsible for the overall electronic effect variation during activation process up to the transition state, and also in the ground state of molecule, or in conjugate acid or base, thanks to its ability to answer the electronic demand of a nearby center.

Among the organosilicon molecules the trimethylsilyl (*e.g.* refs<sup>8-15</sup>), triphenylsilyl<sup>16</sup>, and poly(methylsilyl)<sup>17</sup> groups attached to an aromatic system were reported to change their electronic effect upon substitution in the rest of molecule. Variable electronic effect was also established for the vinyl group when bonded to silicon<sup>18</sup>. The  $(CH_3)_3SiCH_2$  group in  $(CH_3)_3SiCH_2OY$  compounds having  $Y = H$ ,  $(CH_3)_3Si$ ,  $CO(CH_3)$  and alkyl can be regarded<sup>19</sup> to have its electronic effect dependent of reaction and the anchimeric assistance of some silyl groups was inferred from the observed dependence of the relative basicity of silyl ethers on the strength of the interacting acid<sup>20</sup>. In our previous works an electron accepting character of the  $(CH_3)_3Si$  group in  $(CH_3)_3SiOR$  compounds has been found to be determined by the nature of the group  $R$  (refs<sup>21,22</sup>), the  $ClCH_2$  group bonded to silicon in the compounds  $ClCH_2SiXYZ$  was ascertained<sup>23</sup> to increase its electron withdrawing effect with increasing electron-withdrawing ability of  $X$ ,  $Y$ , and  $Z$  groups, and  $Cl_n(CH_3)_{3-n}Si$  groups displaying electron accepting behaviour in the  $Cl_n(CH_3)_{3-n}SiOC_2H_5$  compounds were revealed to act as electron donors when attached to the oxygen of the  $OCH_2CCl_3$  moiety<sup>24</sup>.

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This paper, intended to extend the last work, presents a study of the electronic effect of other silyl groups as  $(C_6H_5)_n(CH_3)_{3-n}Si$  with  $n = 1-3$ ,  $R(CH_3)_2Si$  with  $R = C_6H_5CH_2$ ,  $CH_2=CH$ ,  $ClCH_2$ , and  $BrCH_2$ ,  $Br_3Si$ , and  $RCl_2Si$  with  $R = Cl_2CH$  and 2-thienyl, all bonded to the oxygen atom of both the  $OC_2H_5$  and  $OCH_2CCl_3$  moieties. Relative basicity of the oxygen in ethoxy- and 2,2,2-trichloroethoxysilanes determined from the IR spectra of hydrogen bonds of phenol interacting with these compounds is analysed by means of Taft equation with a view to estimate the electronic effect of silyl groups attached to the oxygen of the above moieties.

## EXPERIMENTAL

*Model compounds.* All the monoalkoxysubstituted silanes  $(C_6H_5)_n(CH_3)_{3-n}SiOR$  with  $n = 1-3$ ,  $R'(CH_3)_2SiOR$  with  $R' = C_6H_5CH_2$ ,  $CH_2=CH$ ,  $ClCH_2$ , and  $BrCH_2$ ,  $R'Cl_2SiOR$  with  $R' = Cl_2CH$  and 2-thienyl, and  $Br_3SiOR$ , R group of those designates both the ethyl and 2,2,2-trichloroethyl moiety, were prepared from the corresponding chlorosilanes  $(C_6H_5)_n \cdot (CH_3)_{3-n}SiCl$ ,  $R'(CH_3)_2SiCl$ , and  $R'SiCl_3$  or from  $SiBr_4$ . The first two series of compounds were prepared by a treatment of a stoichiometric amount of chlorosilane with equal amounts of dry pyridine and alcohol  $C_2H_5OH$  or  $Cl_3CCH_2OH$  in diethyl ether. Monoalkoxydichlorosilanes  $R'Cl_2SiOR$  were obtained by the reaction of little excess of corresponding chlorosilanes  $R'SiCl_3$  with the above alcohols in light petroleum. Monoalkoxytribromosilanes were prepared in the same manner. All the procedures are usual and deserve no particular comment. All the monoalkoxysilanes were obtained by rectification. All operations were carried out under nitro-

TABLE I  
Physical Properties and Yields of New Monoalkoxysubstituted Silanes

Compound	B.p., °C/Torr	$n_D^{20}$	Yield, %
$C_6H_5(CH_3)_2SiOCH_2CCl_3$	167/27	1.5150	66
$(C_6H_5)_2CH_3SiOCH_2CCl_3^a$	158/0.5	—	77
$(C_6H_5)_3SiOCH_2CCl_3^b$	202/0.6	—	72
$CH_2=CH(CH_3)_2SiOCH_2CCl_3$	103/40	1.4578	84
$C_6H_5CH_2(CH_3)_2SiOCH_2CCl_3$	147/9.5	1.5159	67
$ClCH_2(CH_3)_2SiOCH_2CCl_3$	111/21	1.4724	80
$BrCH_2(CH_3)_2SiOCH_2CCl_3$	121/19	1.4900	81
$Cl_2(Cl_2CH)SiOCH_2CCl_3$	122/15	1.4927	40
$Cl_2(C_4H_3S)SiOCH_2CCl_3^c$	102/0.6	1.5333	38
$Br_3SiOCH_2CCl_3^d$	187/0.6	—	39
$Cl_2(Cl_2CH)SiOC_2H_5$	175	1.4525	44
$Cl_2(C_4H_3S)SiOC_2H_5$	104.5/20	1.5070	49

<sup>a</sup> M.p. 63°C. <sup>b</sup> M.p. 82°C. <sup>c</sup>  $C_4H_3S$  denotes 2-thienyl. <sup>d</sup> Slightly impure.

gen. The products purity was checked by gas-liquid chromatography, their identity confirmed by NMR spectroscopy and in the case of the up to now unreported compounds also by elemental analysis. The physical constants of those compounds described previously were in agreement with literature data<sup>25,26</sup>. Physical constants of new compounds first prepared in this study are given together with the reaction yields in Table I.

Relative basicity of the oxygen was determined from the IR spectra of hydrogen bonds of phenol interacting with alkoxysilanes in  $\text{CCl}_4$  as described<sup>27</sup>.

## RESULTS AND DISCUSSION

Relative basicity of the oxygen in species like alcohols<sup>28,29</sup>, ethers<sup>28,30</sup>, siloxanes<sup>31</sup> and alkoxysilanes<sup>31,32</sup> defined as  $\Delta\nu(\text{OH})$  of phenol (or  $\Delta\nu(\text{XH})$  of any other proton donor) interacting with the species can be satisfactorily predicted by means of Taft equation (1).

$$\Delta\nu(\text{OH}) = \rho^* \cdot \sum \sigma^* + C \quad (1)$$

TABLE II

Wavenumber Shift,  $\Delta\nu(\text{OH})$  in  $\text{cm}^{-1}$ , of Phenol Due to Its Interaction with  $\text{R}_1\text{R}_2\text{R}_3\text{SiOR}$  in  $\text{CCl}_4$  and Approximate Values of  $\sigma^*$  of  $\text{R}_1\text{R}_2\text{R}_3\text{Si}$ - Groups in  $\text{R}_1\text{R}_2\text{R}_3\text{SiOR}$

$\text{R}_1\text{R}_2\text{R}_3\text{Si}$	$\Delta\nu(\text{OH})^a$		$\Delta \Delta\nu(\text{OH})$	$\sigma^*^d$
	$\text{R}=\text{C}_2\text{H}_5^b$	$\text{R}=\text{CH}_2\text{CCl}_3^c$		
$(\text{CH}_3)_3\text{Si}^e$	280 <sup>f</sup>	135	145	$-0.15 \pm 0.04$
$\text{CH}_2=\text{CH}(\text{CH}_3)_2\text{Si}$	276	123	153	$-0.10 \pm 0.04$
$\text{C}_6\text{H}_5(\text{CH}_3)_2\text{Si}$	267 (55)	135 (48)	132	$-0.02 \pm 0.03$
$\text{C}_6\text{H}_5\text{CH}_2(\text{CH}_3)_2\text{Si}$	259	131	128	$0.06 \pm 0.03$
$(\text{C}_6\text{H}_5)_2\text{CH}_3\text{Si}$	246 (53)	136 (47)	110	$0.19 \pm 0.04$
$\text{ClCH}_2(\text{CH}_3)_2\text{Si}$	241 <sup>f</sup>	123	118	$0.23 \pm 0.05$
$\text{BrCH}_2(\text{CH}_3)_2\text{Si}$	231	123	108	$0.33 \pm 0.06$
$(\text{C}_6\text{H}_5)_3\text{Si}$	219 (51) <sup>f</sup>	138 (48)	81	$0.45 \pm 0.06$
$\text{Cl}(\text{CH}_3)_2\text{Si}^e$	207	115	92	$0.57 \pm 0.07$
$\text{Cl}_2(\text{CH}_3)\text{Si}^e$	136	120	16	$1.26 \pm 0.11$
$\text{Cl}_2(\text{C}_4\text{H}_9\text{S})\text{Si}^g$	124	121	3	$1.38 \pm 0.12$
$\text{Br}_3\text{Si}$	123	117	6	$1.39 \pm 0.02$
$\text{Cl}_3\text{Si}$	84	130	-46	$1.77 \pm 0.15$
$(\text{Cl}_2\text{CH})\text{Cl}_2\text{Si}$	- <sup>h</sup>	- <sup>i</sup>	-	-

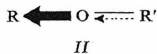
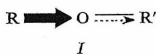
<sup>a</sup> Values in parentheses relate to  $\text{C}_6\text{H}_5$  group. <sup>b</sup>  $\pm 3 \text{ cm}^{-1}$ . <sup>c</sup>  $\pm 5 \text{ cm}^{-1}$ . <sup>d</sup> In  $\text{R}_1\text{R}_2\text{R}_3\text{SiOC}_2\text{H}_5$ . <sup>e</sup> Ref.<sup>24</sup>. <sup>f</sup> In agreement with ref.<sup>38</sup>. <sup>g</sup> 2-Thienyl. <sup>h</sup> Very flat and weak absorption band  $\nu(\text{OH})_{\text{assoc}}$ . <sup>i</sup>  $\nu(\text{OH})_{\text{free}}$  quickly disappears.

However, the  $\Delta\nu(\text{OH})$  vs  $\sum\sigma^*$  correlations reported<sup>28,30</sup> for alcohols and ethers reflect the authors' effort to place all the  $\Delta\nu(\text{OH})$  values on one correlation line.\* The electronic effect, that is a blend of polar and polarizability effect, of a substituent bonded to the oxygen can then be suggested constant irrespective of a moiety to which the substituent is attached.

We have previously reported<sup>7</sup> that relative oxygen basicity in ethers ROR' is controlled by a blend of polar and polarizability effect, this conclusion being arisen from the different  $\rho^*$  constants of  $\Delta\nu(\text{OH})$  vs  $\sigma^*(\text{R})$  plot for the individual sets ROR' with R' = alkyl, H, ClCH<sub>2</sub>, Ph and the like. The order of relative polarizabilities of some organic and silyl groups based on the  $\rho^*$  constants can be written as follows<sup>7,24</sup>:

$$\begin{aligned} \text{R}(\rho^*): \text{t-C}_4\text{H}_9 (127.7 \pm 12.9) &< \text{alkyl} (118.5 - 95.8) < \\ &< (\text{CH}_3)_3\text{Si} (95.0 \pm 3.6, \text{ref.}^{33}) < \text{Cl}(\text{CH}_3)_2\text{Si} (87.9 \pm 4.6) < \\ &< \text{Cl}_2\text{CH}_3\text{Si} (40.1 \pm 2.8) < \text{ClCH}_2 (33.2 \pm 7.45). \end{aligned}$$

Provided that polarizability effect of R' in ROR' operates in a direction to favor the electronically rich or deficient oxygen by either electron-withdrawing action (R is electron-donating, structure I), or electron-donating action (R is electron-withdrawing, drawing, structure II), the overall electronic effect of R' can be suggested<sup>7</sup> to be modified by variation in R. This polarizability effect diminishes the slope of the  $\Delta\nu(\text{OH}) - \sigma_{(\text{R})}^*$  relationship, which implicates that the lower  $\rho^*$  of the equation (I) is acquired for ROR', the higher is the polarizability effect of R' group.

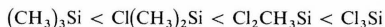


The oxygen basicity data for ethoxysilanes given in Table II respond the variation in silyl group nature quite considerably and approximate  $\sigma^*$  constants of R<sub>1</sub>R<sub>2</sub>R<sub>3</sub>Si groups in R<sub>1</sub>R<sub>2</sub>R<sub>3</sub>SiOC<sub>2</sub>H<sub>5</sub> can be obtained (Table II) from the  $\Delta\nu(\text{OH})$  vs  $\sigma_{(\text{R})}^*$  relationship for ROC<sub>2</sub>H<sub>5</sub> compounds (Eq. (I)) established earlier<sup>7</sup>. They show the silyl groups in the first three entries of Table II to have electron-donating ability, while the other silyl groups to behave as electron acceptors.

The  $\Delta\nu(\text{OH})$  data for 2,2,2-trichloroethoxysilanes are very unusual, since they are very close one to another irrespective of the nature of silyl group. The  $\Delta\nu(\text{OH})$  vs  $\sigma_{\text{silyl}}^*$  plot for Cl<sub>3</sub>CCH<sub>2</sub>OSiR<sub>1</sub>R<sub>2</sub>R<sub>3</sub> compounds is therefore represented by almost zero

\* Different plots  $\Delta\nu(\text{OH})$  vs  $\sum\sigma^*$  for the ethers ROR' with  $sp^3$  and  $sp^2$  hybridized carbon bonded to oxygen were reported without emphasising their different slopes in paper<sup>30</sup>.

slope. We suppose strongly electron-withdrawing  $\text{Cl}_3\text{CCH}_2$  group to develop electron deficiency on the oxygen atom, which is significantly diminished by silyl groups as a consequence of their enormous polarizability. (A greater electron donating effect of  $(\text{C}_6\text{H}_5)_n(\text{CH}_3)_{3-n}\text{Si}$  groups in 2,2,2-trichloroethoxysilanes with respect to that in ethoxysilanes is consistent with a lower proton acceptor ability of the phenyl rings in these compounds). Regardless of their nature, all the silyl groups therefore operate as electron-donating substituents in  $\text{R}_1\text{R}_2\text{R}_3\text{SiOCH}_2\text{CCl}_3$  compounds. A difference between  $\Delta\nu(\text{OH})$  of corresponding  $\text{R}_1\text{R}_2\text{R}_3\text{SiOR}$  compounds with  $\text{R} = \text{C}_2\text{H}_5$  and those with  $\text{R} = \text{CH}_2\text{CCl}_3$ ,  $\Delta \Delta\nu(\text{OH})$  in Table II, has then to reflect the silyl group polarizability; the less  $\Delta \Delta\nu(\text{OH})$ , the higher silyl group polarizability. A perusal of Table II reveals that polarization effect of silyl group in  $\text{R}_1\text{R}_2\text{R}_3\text{SiOCH}_2\text{CCl}_3$  is of the opposite order to the order of the oxygen basicities in  $\text{R}_1\text{R}_2\text{R}_3\text{SiOC}_2\text{H}_5$ . Thus *e.g.* successive substitution of the trimethylsilyl group by chlorine or by the phenyl group leads to the orderings of group polarizabilities



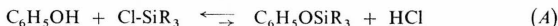
and



the former one being in harmony with that established from the slope  $\Delta\nu(\text{OH}) - \sigma_{(\text{R})}^*$  plots for  $\text{Cl}_n(\text{CH}_3)_{3-n}\text{SiOR}$  (see before, ref.<sup>24</sup>).

It seems fair to state that no relationship exists between the silyl group polarizability and an extent of back-bonding (ref.<sup>34,35</sup>) within silyl group. The  $\text{Cl}_3\text{CCH}_2$  group actually levels the relative basicity of  $\text{R}_1\text{R}_2\text{R}_3\text{SiOCH}_2\text{CCl}_3$  and consequently makes an electron-donating effect of  $\text{R}_1\text{R}_2\text{R}_3\text{Si}$  groups in these compounds almost the same, regardless whether the  $\text{R}_1$ ,  $\text{R}_2$ , and  $\text{R}_3$  substituents are able to be involved in back-bonding (phenyl, vinyl) or not (Br). We suppose mutual polarizability effect of electronegative substituents bonded to silicon to operate and be chiefly responsible for tremendous polarizability of silyl groups in  $\text{R}_1\text{R}_2\text{R}_3\text{SiOCH}_2\text{CCl}_3$ . An operation of this effect is consonant with our results on the oxygen basicity data of other alkoxy-silanes dealt with in our next paper<sup>36</sup>, but can be supported already here by the  $\Delta\nu(\text{OH})$  data for  $(\text{Cl}_2\text{CH})\text{Cl}_2\text{SiOR}$  (Table II). Relative oxygen basicity in  $(\text{Cl}_2\text{CH})\text{Cl}_2\text{SiOC}_2\text{H}_5$  is less than that in trichloroethoxysilane and hence the dichloromethyl group in ethoxysilane acts as stronger electron withdrawing group than chloride. This fails, certainly, to agree with the known  $\sigma^*$  constants of the  $\text{Cl}_2\text{CH}$  and  $\text{Cl}$  substituents and reflects thus the chlorine to be involved in a mutual polarizability effect of to the silicon attached substituents more than the dichloromethyl group. Additionally, the basicity of chlorine in chlorosilanes is directly measurable by IR spectroscopy, similarly as that of oxygen in alkoxy-silanes, thanks to the fact that the

reaction equilibrium (A) between phenol and chlorosilane is dominantly shifted



to the left in  $\text{CCl}_4$  (ref.<sup>37</sup>). This is apparently also so with  $(\text{Cl}_2\text{CH})\text{Cl}_2\text{SiOC}_2\text{H}_5$ , since very weak  $\nu_{(\text{OH})}^{\text{assoc}}$  absorption band is observed in the IR spectra. However,  $(\text{Cl}_2\text{CH})\text{Cl}_2\text{SiOCH}_2\text{CCl}_3$  represents the unique example of chlorosilane reacting under given conditions with phenol. The interaction of phenol with chlorosilane can be suggested to occur *via* the attack of the phenol oxygen to silicon and/or *via* the coordination of chlorine to the hydrogen of phenol O—H bond. The lack of chlorosilane-phenol reaction with  $(\text{Cl}_2\text{CH})\text{Cl}_2\text{SiOC}_2\text{H}_5$  can be then obviously brought about by an unsuitably high electron density on the silicon and/or reduced basicity of chlorine, that is by the features resulting from mutual polarizability effect of the Si—Cl chlorines. On the other hand, the fact that the reaction proceeds with  $(\text{Cl}_2\text{CH})\text{Cl}_2\text{SiOCH}_2\text{CCl}_3$  demonstrates that both the driving forces of the reaction are with this compounds facilitated and mutual polarizability effect has to be diminished.

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