

THE INFRARED SPECTRA OF SOME SILYLALKYL ACETATES, GERMYLALKYL ACETATES, AND THEIR COMPLEXES WITH $ZnCl_2$. THE ELECTRONIC EFFECT OF SILYLMETHYL AND GERMYLMETHYL GROUPS IN SILYLMETHYL AND GERMYLMETHYL ACETATES*

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The IR spectra of the acetates CH_3COOR [$R = (CH_2)_nM(CH_3)_2L$ where L is C_6H_5 , C_2H_5 , CH_3 , $CH_2OC(O)CH_3$ and $M = C, Si, Ge; (CH_2)_nCH_3; (CH_2)_nSi(CH_3)_n(C_2H_5O)_{3-n}; Cl_n \cdot H_{3-n}CCH_2$] and their complexes with $ZnCl_2$ are discussed. The nature of the α -effect in α -carbofunctional silicon and germanium compounds is discussed on the basis of the low temperature IR spectrum of trimethylsilylmethyl acetate. The IR spectra of the complexes are interpreted in terms of polarizability of the alkoxy group. It was found that interaction of the compounds $(CH_3)_2 \cdot LMCH_2OC(O)CH_3$ with electrophile ($ZnCl_2$) leads to significant changes in the electronic effect of the $(CH_3)_2LMCH_2$ -groups.

The IR spectra of α -carbofunctional oxygen-containing trimethylsilanes and trimethylgermanes of the type $(CH_3)_3MCH_2OX$ ($M = Si, Ge; X = H, C(O)CH_3, Si(CH_3)_3, alkyl$) were interpreted in terms of the so called α -effect, *i.e.* the shift of electrons from the functional group toward the silicon or germanium^{1,2}. The studies of the basicity^{2,3} and nucleophilicity^{4,5} of the oxygen of these compounds have however shown that both properties are consistent with the electron donor ability of the $(CH_3)_3MCH_2$ -groups. The great difference between the electronic effect of these groups in the ground state of $(CH_3)_3MCH_2OH$ molecules and in interaction of these compounds with electrophilic particles could bear upon the fact that the electron donor character of the $(CH_3)_3MCH_2$ -groups in the compounds $(CH_3)_3 \cdot MCH_2OX$ is the consequence of the electronic requirement of the electrophilic particle attacking the substrate. From this viewpoint it was of interest to find whether and how the interaction of the compounds $(CH_3)_3MCH_2OX$ with electrophiles affects the wavenumbers of $\nu(C-O(X))$ stretching vibrations whose anomalous decrease was ascribed to the α -effect. With this aim we studied in the present work the interaction of the acetates $L_n(CH_3)_{3-n}MCH_2OCOCH_3$ ($L = C_2H_5, C_6H_5, C_2H_5O$;

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M = Si, Ge) and $(\text{CH}_3)_2\text{Si}(\text{CH}_2\text{OCOCH}_3)_2$ with ZnCl_2 . For the sake of comparison the same interaction was examined by IR spectroscopy also for the compounds $(\text{CH}_3)_3\text{M}(\text{CH}_2)_n\text{OCOCH}_3$ ($n > 1$), $\text{CH}_3(\text{CH}_2)_n\text{OCOCH}_3$ ($n = 0-3$), and $\text{Cl}_n\text{H}_{3-n}\text{CCH}_2\text{OCOCH}_3$ ($n = 1-3$).

EXPERIMENTAL

Acetates of the type $\text{Cl}_n\text{H}_{3-n}\text{CCH}_2\text{OCOCH}_3$ were prepared from appropriate alcohols by their reaction with acetyl chloride in the presence of stoichiometric amount of dry pyridine in dry diethyl ether and were obtained in 85–90% yields by rectification. Their purity was verified by gas chromatography and their structure was confirmed by IR and ^1H -NMR spectrometry. $\text{Cl}_n\text{H}_{3-n}\text{CCH}_2\text{OCOCH}_3$ (n , b.p. ($^\circ\text{C}/\text{Torr}$), n_D^{20}): 1, 72/112, 1.4235; 2, 75/105, 1.4430; 3, 88/85, 1.4552).

Bis(acetoxymethyl)dimethylsilane was prepared⁶ by reaction of bis(chloromethyl)dimethylsilane with potassium acetate: b.p. $120^\circ\text{C}/22$ Torr, n_D^{20} 1.4318; ref.⁶ b.p. $124^\circ\text{C}/27$ Torr, n_D^{25} 1.4309. Methyl, ethyl, n-propyl, and n-butyl acetates (all supplied by Lachema, Brno) were rectified before use. The other acetates were reported earlier^{7,8}.

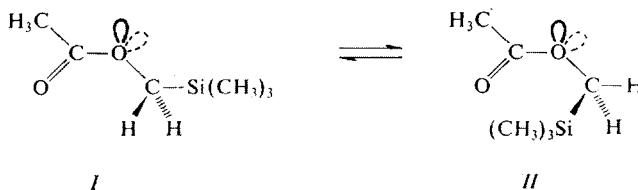
IR spectra in the $400-4000\text{ cm}^{-1}$ region were recorded on a double-beam Zeiss, Jena spectrophotometer, Model UR 20 (KBr, NaCl, and LiF prisms). The frequency scale of the instrument was calibrated in the usual way. The IR spectra of the saturated solution of ZnCl_2 in the acetates were always measured *in substantia*. Measurements were carried out in composed KBr cells. As the polyethylene foil with the sample was inserted between the windows of the cell, the absorption bands of the complexes studied were not read in the region of the polyethylene foil absorption.

The IR spectrum of the solution of $(\text{CH}_3)_3\text{SiCH}_2\text{OCOCH}_3$ in CS_2 was measured at a temperature of liquid nitrogen in the cell with AgCl windows (produced by RIIC) and recorded on a Beckmann spectrophotometer, Model IR-20.

RESULTS AND DISCUSSION

IR Spectra of the Acetates

The assignment of IR absorption bands of all the acetates $\text{L}_n(\text{CH}_3)_{3-n}\text{MCH}_2\text{OCOCH}_3$ ($\text{L} = \text{C}_2\text{H}_5, \text{C}_6\text{H}_5, \text{C}_2\text{H}_5\text{O}$; $\text{M} = \text{C}, \text{Si}, \text{Ge}$), $(\text{CH}_3)_2\text{Si}(\text{CH}_2\text{OCOCH}_3)_2$, $\text{Cl}_n\text{H}_{3-n}\text{C}(\text{CH}_2)_n\text{OCOCH}_3$, and $\text{CH}_3(\text{CH}_2)_n\text{OCOCH}_3$ in the $1800-900\text{ cm}^{-1}$ region was based on the works⁹⁻¹². The stretching vibration $\nu(\text{C}=\text{O})$ of these acetates



occurs in the $1738-1763\text{ cm}^{-1}$ region, the antisymmetrical stretching vibration $\nu(\text{C}-\text{O}(\text{C}))$ in the $1211-1247\text{ cm}^{-1}$ region, and the stretching vibration $\nu(\text{O}-\text{C}_R)$ in the $1022-1053\text{ cm}^{-1}$ region. The wavenumbers of these vibrations are presented in Table I. As a rule, the $\nu(\text{C}-\text{O}(\text{C}))$ absorption bands overlap or are located in the vicinity of other bands ($\delta_{\text{as}}(\text{CH}_3-\text{Si}) \sim 1245-1270\text{ cm}^{-1}$, $\delta_{\text{as}}(\text{CH}_3-\text{Ge}) \sim 1240$ to 1255 cm^{-1} , $\delta(\text{CH}_3-\text{C})$ $1235-1250\text{ cm}^{-1}$, $\gamma(\text{CH}_2) \sim 1290\text{ cm}^{-1}$). The $\nu(\text{O}-\text{C}_R)$ absorption band which, with the exception of the compounds $(\text{CH}_3)_n(\text{C}_2\text{H}_5\text{O})_{3-n}.\text{SiCH}_2\text{OCOCH}_3$, was read as the strongest band in the $1000-1000\text{ cm}^{-1}$ region, was split or strongly deformed for all the acetates but the compounds $(\text{C}_2\text{H}_5\text{O})_n.(\text{CH}_3)_{3-n}.\text{SiCH}_2\text{OCOCH}_3$, $(\text{CH}_3)_2\text{Si}(\text{CH}_2\text{OCOCH}_3)_2$, and $(\text{CH}_3)_3\text{MCH}_2\text{OCOCH}_3$. The absorption bands occurring below 1000 cm^{-1} were due to the $\rho(\text{CH}_3-\text{M})$ ($970-990\text{ cm}^{-1}$) and $\nu(\text{C}-\text{C})$ ($\sim 900-960\text{ cm}^{-1}$) vibrations.

The change of $\nu(\text{C}=\text{O})$ of the esters $\text{R}'\text{C}(\text{O})\text{OR}$ was tentatively assigned to the action of the polar effect of groups R and R' (ref.¹³). The correlation of $\nu(\text{C}=\text{O})$ with σ_R^* constants is not valid generally, and as a rule such correlations are observed with the groups of the esters with the same type of the alkoxy group¹⁴. This shows that the dependence of the wavenumber of $\nu(\text{C}=\text{O})$ of primary alkyl acetates on σ^* constants (Fig. 1; a similar dependence has been found also with the acetates $\text{Cl}_n.\text{H}_{3-n}\text{CCH}_2\text{OCOCH}_3$) must be looked at similarly. From this dependence it follows that the wavenumber of $\nu(\text{C}=\text{O})$ of trimethylsilylmethyl acetate deviates from the correlation. The explanation of this deviation by weakened electron donor effect

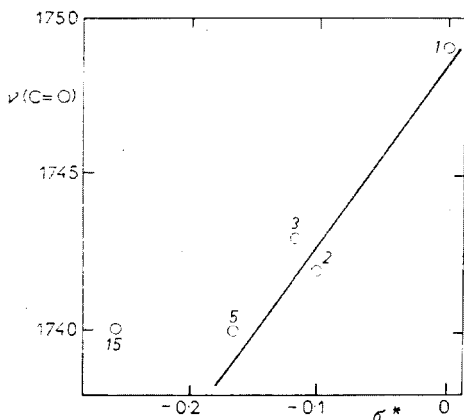


FIG. 1

Dependence of the Wavenumber of $\nu(\text{C}=\text{O})$ (cm^{-1}) of the Compounds ROCOCH_3 (Table I) on σ_R^*

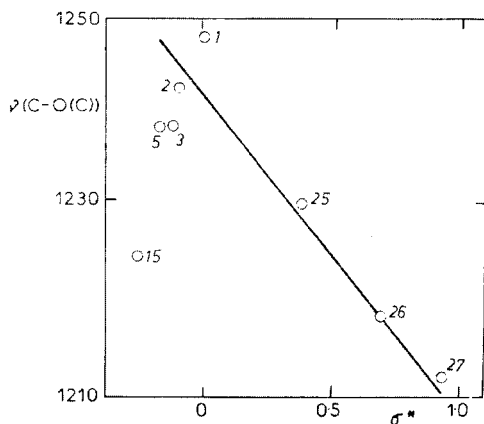


FIG. 2

Dependence of the Wavenumber of $\nu(\text{C}-\text{O}-\text{C})$ (cm^{-1}) of the Compounds ROCOCH_3 (Table I) on σ_R^*

of the $(\text{CH}_3)_3\text{SiCH}_2$ -group can be supported by similar deviation observed in correlating wavenumbers of $\nu(\text{C—O}(\text{C}))$ with σ^* constants (Fig. 2). The fact that the wavenumbers of $\nu(\text{C=O})$ vibration and the less characteristic $\nu(\text{C—O}(\text{C}))$ vibration with substitution in the molecule of $\text{X—C}(\text{O})\text{—Y}$ change in the opposite way and correlate with one another^{15,16} can be explained such that the change of the substituent strengthens one and weakens the other type of the bond. With regard to the value of $\sigma_{(\text{CH}_3)_3\text{SiCH}_2}^*$, the small weakening of the C=O bond in $(\text{CH}_3)_3\text{SiCH}_2\text{OCOCH}_3$ could be connected with the small strengthening of the C—O bond. The general decrease of the wavenumber of $\nu(\text{C—O}(\text{C}))$ of the compounds $\text{L}(\text{CH}_3)_2\text{MCH}_2\text{OCOCH}_3$ ($\text{L} = \text{CH}_3, \text{C}_2\text{H}_5, \text{C}_6\text{H}_5$; $\text{M} = \text{Si, Ge}$) compared to the compounds $(\text{CH}_3)_3\text{M}(\text{CH}_2)_n\text{OCOCH}_3$ ($n > 1$) or $\text{R}(\text{CH}_2)_n\text{OCOCH}_3$ ($\text{R} = (\text{CH}_3)_3\text{C, CH}_3$; $n = 1-4$) is analogous to the decrease of the wavenumber of this vibration in the compounds $\text{Cl}_n\text{H}_{3-n}\text{CCH}_2\text{OCOCH}_3$. The explanation of the decrease of $\nu(\text{C—O}(\text{C}))$ by decreased electron donor effect of the $\text{L}(\text{CH}_3)_2\text{SiCH}_2$ -groups in the ground state of molecules, suggested earlier^{1,2}, seems therefore justified, since mass effect cannot be responsible for this result⁹. The IR spectra of α -carbonyl functional acetates of germanium and silicon are characterized also by the decrease of the $\nu(\text{O—C}_R)$ wavenumbers. Decreases in the wavenumbers of $\nu(\text{C—O}(\text{C}))$ and $\nu(\text{O—C}_R)$ are gradually compensated by progressive substitution of the silicon by ethoxy groups.

A more detailed examination of the nature of the α -effect in the compounds $(\text{CH}_3)_3\text{MCH}_2\text{OCOCH}_3$ ($\text{M} = \text{Si, Ge}$) can be based on the knowledge of changes of their IR spectra with temperature. The IR spectrum of trimethylsilylmethyl acetate shows following changes on cooling the sample from room temperature to -180°C : the intensity of the bands at 703 cm^{-1} , 845 cm^{-1} , 1030 cm^{-1} , and 1250 cm^{-1} decreases, the intensity of the bands around 861 cm^{-1} increases and new bands appear at 995 cm^{-1} and 1050 cm^{-1} . The maximum of the 1224 cm^{-1} band shifts to 1232 cm^{-1} , the intensity of the 1740 cm^{-1} band decreases and its maximum shifts to 1738 cm^{-1} . In view of the fact that the alkyl acetates $\text{CH}_3\text{C}(\text{O})\text{OCH}_2\text{R}$ have planar conformation with periplanar arrangement of the carbonyl group and the $\text{O—CH}_2\text{R}$ (ref.¹⁷) bond and that rotation around the $\text{O—CH}_2\text{R}$ bond leads to formation of conformers, of which that with planar arrangement of heavy atoms is energetically most preferred¹⁸, the above changes in the spectrum of $(\text{CH}_3)_3\text{SiCH}_2\text{OC}(\text{O})\text{CH}_3$ can be accounted for by the shift of conformational equilibrium between conformers *I* and *II*.

The absence of the bands characteristic for the conformer with the lower energy content (for the assignment of conformationally sensitive bands of similar esters see ref.¹⁸) at room temperature indicates that the IR spectrum of trimethylsilylmethyl acetate at this temperature belongs to one conformer. Changes of absorption bands at 1030 cm^{-1} , 1050 cm^{-1} , and 1224 cm^{-1} with temperature show that the α -effect is more pronounced in the conformer with the higher content of energy. This should be conformer *II*, and the α -effect could be then due to $\sigma_{\text{C}(\text{O})-\text{O}} \rightarrow d$

TABLE I

Some Wavenumbers (cm^{-1}) of the Acetates $\text{CH}_3\text{C}(\text{O})\text{OR}$ and Their Shifts on Interaction of the Acetates with ZnCl_2

No	R	$\nu(\text{C}=\text{O})$	$\nu(\text{C}-\text{O}(\text{C}))$	$\nu(\text{O}-\text{C}_R)$	$\Delta\nu(\text{C}-\text{O}(\text{C}))$	Absorption bands in the 1100—900 cm^{-1} region
1	$\text{CH}_3(\text{CH}_2)_n$	$n = 0$	1749	1247	1050	984, 1050
2		1	1742	1242	1050	917, 936, 1050
3		2	1743	1238	1048	966, 1020, 1047
4		3	1744	1243	1046	922, 952, 1034, 1046, 1069
5	$(\text{CH}_3)_3\text{C}(\text{CH}_2)_n$	$n = 1$	1740 ^a	1238 ^a	1046	908, 923, 938, 987, 1028, 1046
6		2	1738 ^a	1240 ^a	1043	920, 972, 1023, 1043, 1089
7		3	1740 ^b	1240	1053	917, 933, 983, 1047, 1053, 1086
8		4	1738 ^b	1239	1049	908, 928, 958, 978, 1040, 1049, 1071
9	$\text{C}_2\text{H}_5(\text{CH}_3)_2\text{SiCH}_2$		1744 ^a	1222 ^a	1029	960, 1009, 1029
10	$\text{C}_6\text{H}_5(\text{CH}_3)_2\text{SiCH}_2$		1740 ^a	1220 ^a	1032	978, 1032
11	$\text{CH}_3\text{COOCH}_2(\text{CH}_3)_2\text{SiCH}_2$		1739	1225	1022	979, 1022
12	$(\text{CH}_3)_{3-n}(\text{C}_2\text{H}_5\text{O})_n\text{SiCH}_2$	$n = 0$	1745 ^c	1229 ^c	1039	952, 1035, 1084
13		1	1745 ^c	1236 ^c	1042	962, 980, 1036, 1084
14		2	1744 ^c	1239 ^c	1046	945, 966, 1038, 1084
15	$(\text{CH}_3)_3\text{Si}(\text{CH}_2)_n$	$n = 1$	1740 ^a	1224 ^a	1030	978, 1030
16		2 ^d	1739 ^a	1239 ^a	1046	905, 938, 973, 1024, 1046
17		3	1740 ^a	1237 ^a	1046	936, 971, 1030, 1046, 1060
18		4	1743 ^a	1237 ^a	1035	942, 968, 986, 1035, 1065
19		5	1746	1245	1043	958, 1043
20		6	1746	1245	1045	919, 950, 970, 1045, 1062
21	$(\text{CH}_3)_3\text{Ge}(\text{CH}_2)_n$	$n = 1$	1738 ^b	1225 ^b	1032	979, 1032
22		2 ^d	1738 ^b	1243 ^b	1053	—
23		3	1739 ^b	1243 ^b	1053	908, 940, 975, 1028, 1053, 1070
24		4	1738 ^b	1240 ^b	1037	940, 974, 1035, 1053, 1070
25	$\text{Cl}_n\text{H}_{3-n}\text{CCH}_2$	$n = 1$	1748	1230	1043	945, 972, 1037, 1070
26		2	1754	1218	1046	—
27		3	1763	1211	1051	923, 1051, 1064, 1090

^a Ref. 1; ^b ref. 2; ^c ref. 8; ^d Interaction of these compounds with ZnCl_2 led to β -decomposition; the IR spectra of solutions of ZnCl_2 in these compounds were composed of the spectra of the compounds $(\text{CH}_3)_3\text{MOC}(\text{O})\text{CH}_3$ and their complexes with ZnCl_2 .¹⁵

or $\sigma_{C(O)-O} \rightarrow \sigma_{(O)C-Si}^*$ interaction. This explanation is somewhat weakened by small shifts of the carbonyl band at 1740 cm^{-1} with temperature. These would be consistent rather with the interpretation of the α -effect suggested for the alcohols $(CH_3)_3MCH_2OH$ ($M = Si, Ge$) (ref.²) and based on the $\sigma_{C(Si)-O} \rightarrow d$ interaction.

IR Spectra of the Complexes of the Acetates with $ZnCl_2$

The IR spectra of the complexes of esters with metal halides show characteristic shifts of the wavenumbers of $\nu(C=O)$ and $\nu(C-O(C))$ (ref.¹⁹⁻²¹). The extent of the interaction of $ZnCl_2$ with the acetates studied in the present work can be therefore estimated from the magnitude of these shifts. The values of $\Delta\nu(C=O)$ for all the acetates are essentially identical and equal to $100 \pm 4\text{ cm}^{-1}$. The more sensitive measure of this interaction is however $\Delta\nu(C-O(C))$ (Table I) which is greatest for the compounds $L(CH_3)_2MCH_2OCOCH_3$ ($M = Si, Ge; \sim 125-129\text{ cm}^{-1}$) and gradually decreases in the order $(CH_3)_2Si(CH_2OCOCH_3)_2$ (117 cm^{-1}), $(C_2H_5O)_n \cdot (CH_3)_{3-n}SiCH_2OCOCH_3$ ($\sim 105-108\text{ cm}^{-1}$), $Cl_nH_{3-n}CCH_2OCOCH_3$ ($n = 1, 2; \sim 100\text{ cm}^{-1}$), $(CH_3)_3M(CH_2)_nOCOCH_3$ ($M = C, Si, Ge; n > 2$), $CH_3(CH_2)_n \cdot OCOCH_3$ ($n > 1; \sim 90-95\text{ cm}^{-1}$), CH_3OCOCH_3 (78 cm^{-1}), and $Cl_3CCH_2 \cdot OCOCH_3$ (64 cm^{-1}). The value of $\Delta\nu(C-O(C))$ for α -carbonyl functional compounds of silicon and germanium is so great that it shifts the wavenumber of the $\nu(C-O(C))$ of the complexes of these compounds with $ZnCl_2$ (further referred as the complexes) above the wavenumber of the complexes formed by the compounds $(CH_3)_3M(CH_2)_n \cdot OCOCH_3$ ($M = C, Si, Ge; n > 2$). Relatively higher value of $\nu(C-O(C))$ of the complexes of the compounds $L(CH_3)_2MCH_2OCOCH_3$ ($M = Si, Ge$) can be accounted for by the greater force constant and thus by the higher C—O(C) bond order due to the electron donor effect of the $L(CH_3)_2MCH_2$ -groups. With regard to the lower wavenumber of the $\nu(C-O(C))$ of the compounds $L(CH_3)_2MCH_2 \cdot OCOCH_3$ in their ground state, the electron donor effect of the $L(CH_3)_2MCH_2$ -groups play a role only on interaction of these compounds with $ZnCl_2$. The lower values of $\Delta\nu(C-O(C))$ for $(CH_3)_2Si(CH_2OCOCH_3)_2$ bears upon the fact that the $(CH_3)_2Si(CH_2)$ -group must compensate the electron deficiency on the two acetoxy groups. Similarly, the value of $\Delta\nu(C-O(C))$ of the compounds $(C_2H_5O)_n(CH_3)_{3-n} \cdot SiCH_2OCOCH_3$ shifts the wavenumber of the $\Delta\nu(C-O(C))$ of their complexes above the wavenumber of the $\nu(C-O(C))$ of the compounds $(CH_3)_3M(CH_2)_n \cdot OCOCH_3$ ($M = C, Si, Ge; n > 2$) and $CH_3(CH_2)_nOCOCH_3$ ($n < 1$) and demonstrates the electron donor effect of the $(C_2H_5O)_n(CH_3)_{3-n}SiCH_2$ -groups in the complexes of these compounds with $ZnCl_2$. The values of $\Delta\nu(C-O(C))$ of the compounds $Cl_nH_{3-n}CCH_2OCOCH_3$ with $n = 1$ and 3 agree with the polar effect of the $Cl_3H_{3-n}CCH_2$ -groups. The wavenumber for the medium member of this series is surprisingly great. When compared to the other alkyl acetates, the lowest value of $\Delta\nu(C-O(C))$ for methyl acetate is consistent with the smallest polarizability of the methyl group^{22,23}.

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