Organometallic Chemistry

Synthesis and transformations of metallocycles
19.* Synthesis of 3-alkylalumacyclopentanes and 2-alkyl-1,4-dialuminiobutanes on Zr-containing complex catalysts

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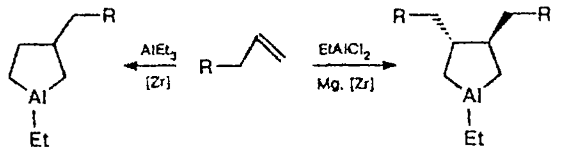
A regioselective method for the synthesis of 3-alkylalumacyclopentanes and 2-alkyl-1,4-dialuminiobutanes by the reactions of α-olefins with $EtAlCl_2$, $ROAlCl_2$, R_2NAlCl_2, or Et_2AlCl and the Mg—$ClCH_2CH_2Cl$—Cp_2ZrCl_2 ($ZrCl_4$) system was developed.

Key words: organoaluminum compounds, zirconium-containing catalysts, cycloalumination, olefins, magnesium, alkyl-, alkoxy-, and dialkylaminodihaloalanes.

We have previously developed selective methods for the synthesis of di-[2,3] or trisubstituted[4,5] alumacyclopentanes (ACPs 1 and 2) from α-olefins, $AlEt_3$ or $EtAlCl_2$, and Zr complexes (Scheme 1).

Scheme 1



1 2

High regio- and stereoselectivities of the preparation of compounds 2 based[4,5] on the use of accessible fire- and explosion-safe reagents ($RAlCl_2$) made this method promising in synthetic practice. However, no formation of monoalkyl-substituted ACPs (1) is observed in experiments with $EtAlCl_2$ or $RAlCl_2$.

For the purpose of developing an efficient catalytic method for the synthesis of 3-substituted ACPs with different structures on the basis of the aforementioned approach to ACPs from $RAlCl_2$ and α-olefins, we studied the Cp_2ZrCl_2-catalyzed reactions of $RAlCl_2$ (R = Alk, OR, NR_2) with α-olefins and ethylene, assuming that the reaction can thus be directed toward the formation of compounds 1 (Scheme 2).

However, the reaction of $EtAlCl_2$ with 1-hexene and ethylene gives a mixture of cyclic organoaluminum compounds (OACs) (1—3), whose ratio depends on the concentration of gaseous ethylene in the reaction mixture.

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* For Part 18, see Ref. 1.

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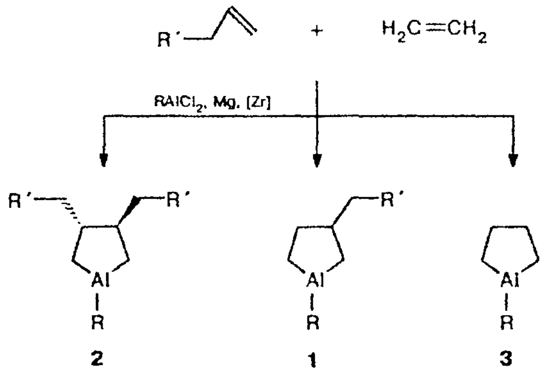
The attempts to direct the reaction toward the selective formation of ACPs **1** by changing the ratio of the starting olefins and the catalyst concentration failed. A mixture of mono-, di-, and trisubstituted ACPs (**1**—**3**) was obtained in all experiments.

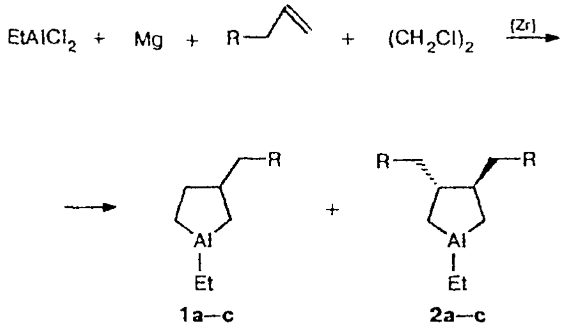
Scheme 2



Since it is difficult to precisely dose gaseous ethylene in experiments under atmospheric pressure, we assumed that the generation of ethylene directly under cycloalumination conditions from 1,2-dichloroethane by highly reactive magnesium, which is used in this reaction as the acceptor of chlorine ions, according to the method described previously,[6] might increase the yield of ACPs **1**.

In fact, the reaction of $EtAlCl_2$ with equimolar amounts of 1-hexene and 1,2-dichloroethane in the presence of excess Mg and catalytic amounts of Cp_2ZrCl_2 or $ZrCl_4$ (5 mol.%) in THF results (Scheme 3) in 1-ethyl-3-(*n*-butyl)alumacyclopentane (**1a**) in ~70% yield and 1-ethyl-*trans*-3,4-di(*n*-butyl)alumacyclopentane (**2a**) (<10%). The position of alkyl substituents and their configurations in compounds **2** were established by ^{13}C NMR spectroscopy.[4,5,7]

Scheme 3

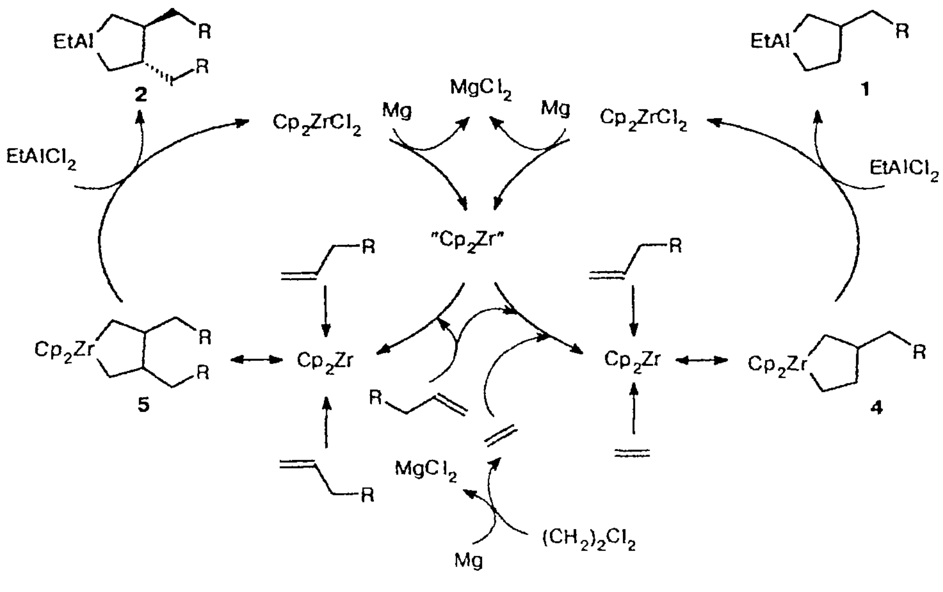


a: R = Pr^n; b: R = n-C_5H_{11}; c: R = n-C_7H_{15}

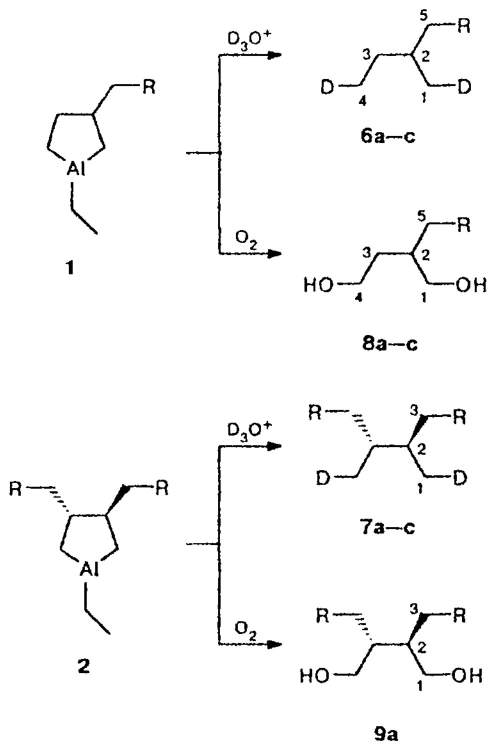
The ratio of ACPs **1** and **2** depends on the method of introduction of the starting α-olefins and 1,2-dichloroethane into the reaction mixture. The highest yields of **1** were obtained when equimolar amounts of α-olefin and 1,2-dichloroethane in THF are introduced slowly. For example, when a mixture of 1-hexene and 1,2-dichloroethane (1 : 1) in THF is added to the reaction mixture over 5 min followed by stirring for 10 h, compounds **1a** and **2a** are formed in a ratio of ~1 : 1 in an overall yield of ~80%. When the starting reagents are introduced over 4 and 6 h, the **1a** : **2a** ratios are 4 : 1 and 7 : 1, respectively, and the yield after 10 h amounts to 80—85%.

Based on the available data,[4,5,7—11] one can assume that the formation of ACPs **1** and **2** occurs through the

Scheme 4

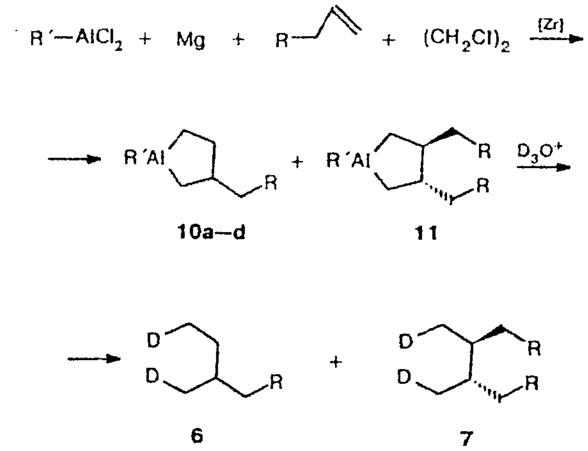


Scheme 5



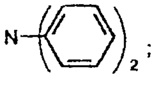
a: R = Pr^n; b: R = n-C_5H_{11}; c: R = n-C_8H_{17}

Scheme 6

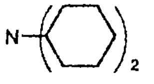


a: R = n-C_5H_{11}, R′ = OBu^n; b: R = n-C_5H_{11}, R′ = NEt_2;

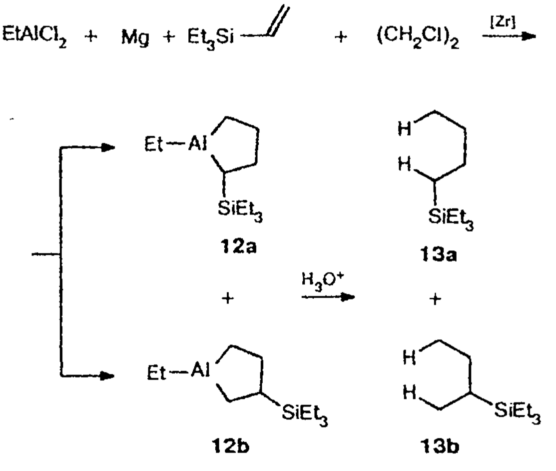
c: R = Bu^n, R′ =



d: R = Bu^n, R′ =



Scheme 7



stage of generation of "Cp_2Zr," which coordinates the starting olefins and transforms into 3- and 3,4-substituted zirconacyclopentanes (Scheme 4). Transmetallation of zirconacyclopentanes (**4** and **5**) with excess $EtAlCl_2$ results in ACPs **1** and **2**.

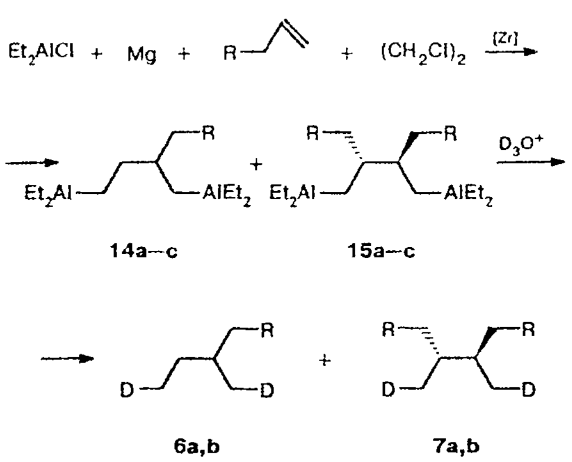
The structure and stoichiometry of substituted ACPs **1** and **2** were confirmed by their transformation into the corresponding substituted 1,4-dideuteriobutanes (**6** and **7**) and butane-1,4-diols (**8** and **9**) (Scheme 5). Stereochemistry of the compounds obtained was proved unambiguously by ^{13}C NMR spectroscopy by analogy with the data presented previously.[4,5,7]

In addition to $EtAlCl_2$, such compounds as $ROAlCl_2$ and R_2NAlCl_2 can be involved in this reaction, and the corresponding mono- (**10**) and disubstituted ACPs (**11**) are formed in a ratio of ~6 : 1 (Scheme 6).

Unlike α-olefins,[2] cycloalumination of vinyltriethylsilane occurs with the formation of 2- and 3-silyl-substituted ACPs (**12a,b**), whose hydrolysis results in (n-butyl)triethylsilane (**13a**) and (butan-2-yl)triethylsilane (**13b**) in a ratio of ~2 : 3 (Scheme 7).

When $RAlCl_2$ is replaced by R_2AlCl, transmetallation of 3-substituted zirconacyclopentane intermediates (**4**) affords 3-alkyl-substituted 1,4-dialuminiobutanes — this is a new method for their synthesis. For example, the reaction of 1-octene with a twofold excess of Et_2AlCl in the presence of 1,2-dichloroethane, magnesium, and 5 mol.% Cp_2ZrCl_2 or $ZrCl_4$ gave a mixture of 2-(n-hexyl)-1,4-bis(diethylaluminio)butane (**14b**) and *threo*-2,3-di(n-hexyl)-1,4-bis(diethylaluminio)butane (**15b**) in a ratio of ~6 : 1 in an overall yield of ~75% (Scheme 8). The introduction of 1-octene and 1,2-dichloroethane in THF into the reaction mixture over 4—6 h is pre-

Scheme 8



a: R = Pr^n; **b**: R = C_5H_{11}; **c**: R = C_7H_{15}

requisite for the predominant formation of monosubstituted 1,4-dialuminiobutane (**14b**). The structure and configuration of alkyl substituents in compounds **7** and **15** were established by ^{13}C NMR spectroscopy.[7,12] Numeration of atoms in molecules of OACs studied and their hydrolysis and oxidation products is presented in Fig. 1, and the data of their ^{13}C NMR spectra are given in Table 1. In the spectra of 1,4-dialuminiobutanes **14** and **15**, the signals for the C atoms of the ethyl groups are more intense than the corresponding signals in the spectra of alumacyclopentanes **1** and **2**.

No formation of OACs **14** was observed when the reaction was carried out in the absence of 1,2-dichloroethane; only *threo*-2,3-disubstituted 1,4-dialuminiobutanes (**15**) were obtained in this case.[4,5,12]

Thus, we have developed a new efficient approach to the synthesis of 3-alkylalumacyclopentanes and 2-alkyl-*threo*-1,4-dialuminiobutanes by the reaction of aluminum mono- and dihalides with a mixture of equimolar amounts of α-olefin and 1,2-dichloroethane in the presence of Mg and catalytic amounts of Cp_2ZrCl_2 or $ZrCl_4$.

Experimental

Reactions with organometallic compounds were performed in a flow of dry argon. THF was distilled from $LiAlH_4$ prior to use. Hydrolysis or oxidation products were analyzed by GLC on a Chrom-5 chromatograph with He as the carrier gas using a column 1200×3 mm with 5% SE-30 or 15% PEG-6000 on Chromaton N-AW. IR spectra were recorded on a UR-20 spectrometer (film), and mass spectra were recorded on an MKh-1306 spectrometer (70 eV, 200 °C). ^{1}H NMR spectra were recorded in $CDCl_3$ on a Tesla BS-567 spectrometer (100 MHz) with Me_4Si as the internal standard. ^{13}C NMR spectra were obtained on JEOL FX-90Q (22.5 MHz) and Bruker AM-300 spectrometers (75.46 MHz) with a wide-band and off-resonance proton decoupling. ^{13}C NMR spectra of OACs were obtained on a JEOL FX-90Q spectrometer (22.5 MHz) with complete and partial proton decoupling. Dilute solutions of OACs were used with addition of C_6D_6 (C_6D_{12}, C_7D_8) for internal stabilization of the field. Solutions of OACs were placed in sealed ampoules in a dry argon atmosphere. Me_4Si was used as the internal standard. The yields of OACs were determined by GLC analysis of the hydrolysis products.

Reactions of alkenes with organoaluminum compounds. *A.* Cp_2ZrCl_2 (0.5 mmol), Mg powder (30 mmol), THF (15 mL), and $EtAlCl_2$ (12 mmol) or Et_2AlCl (22 mmol) were stirred in a glass reactor in a dry argon atmosphere at 0 °C. The solution was warmed to 22—23 °C, and a mixture of α-olefin (10 mmol) and 1,2-dichloroethane (12 mmol) in THF (10 mL) was added dropwise over 6 h followed by stirring for 4 h. Hydrolysis of the reaction mixture with 15% DCl in D_2O results in 2-alkyl-1,4-dideuteriobutanes (**6**) and *threo*-2,3-dialkyl-1,4-dideuteriobutanes (**7**) in a (**6**—**7**) : 1 ratio in an overall yield of 65—85% (in the case of Et_2AlCl, the yield is 70—75%). Oxidation of ACPs **1** and **2** with dried atmospheric oxygen results in the corresponding 2-alkylbutane-1,4-diols (**8**) and *threo*-2,3-dialkylbutane-1,4-diols (**9**) in the same ratio.

B. Under conditions similar to those presented for method *A*, a mixture of $ZrCl_4$ (0.5 mmol), Mg powder (30 mmol), THF (15 mL), and 1-(*n*-butoxy)aluminum dichloride (12 mmol) (1-(diphenylamino)-, 1-(dicyclohexylamino)-, 1-(diethylamino)aluminum dichlorides) in THF (3 mL) was treated with a mixture of α-olefin (10 mmol) and 1,2-dichloroethane (15 mmol) in THF (10 mL). The reaction mixture was treated with 15% DCl in D_2O, and the target products were extracted with ether, dried with $MgSO_4$, and isolated by distillation. Compounds **6** and **7** were obtained in a (5—7) : 1 ratio. The hydrolysis and oxidation products were identified by spectral methods.

1-Deuterio-3-deuteriomethylheptane (6a), b.p. 120—121 °C (760 Torr), n_D^{24} 1.3985. IR, ν/cm^{-1}: 2935, 2860, 2175 (C—D); 1440, 1380, 720. ^{1}H NMR ($(CD_3)_2CO$), δ: 0.85 (m, 7 H, CH_2D, CH_3); 1.25 (m, 9 H, CH, CH_2). MS, *m/z*: 116 $[M]^+$. Found (%): C, 82.61; H, 16.96. $C_8H_{16}D_2$. Calculated (%): C, 82.76; H, 13.79; D, 3.45.

1-Deuterio-3-deuteriomethylnonane (6b), b.p. 65—66 °C (22 Torr), n_D^{22} 1.4126. IR, ν/cm^{-1}: 2930, 2860, 2180 (C—D); 1472, 1380, 725. ^{1}H NMR (CCl_4), δ: 0.80 (m, 7 H, CH_2D, CH_3); 1.20 (m, 13 H, CH, CH_2). MS, *m/z*: 144 $[M]^+$. Found (%): C, 83.18; H, 16.48. $C_{10}H_{20}D_2$. Calculated (%): C, 83.33; H, 13.89; D, 2.78.

1-Deuterio-3-deuteriomethyldodecane (6c), b.p. 72—73 °C (3 Torr), n_D^{22} 1.4220. IR, ν/cm^{-1}: 2940, 2870, 2195 (C—D); 1475, 1385, 735. ^{1}H NMR ($(CD_3)_2CO$), δ: 0.79 (m, 7 H, CH_2D, CH_3); 1.25 (m, 19 H, CH, CH_2). MS, *m/z*: 186 $[M]^+$. Found (%): C, 83.74; H, 16.02. $C_{13}H_{26}D_2$. Calculated (%): C, 83.87; H, 13.98; D, 2.15.

5,6-Bis(deuteriomethyl)decane (7a), b.p. 65—67 °C (3 Torr). IR, ν/cm^{-1}: 2975, 2949, 2872, 2165, 1478, 1385, 1070. ^{1}H NMR, δ: 0.65—1.0 (m, 10 H, CH_3, CH_2D); 1.08—1.60 (m, 14 H, CH, CH_2). MS, *m/z*: 172 $[M]^+$. Found (%): C, 83.68; H, 16.19. $C_{12}H_{24}D_2$. Calculated (%): C, 83.72; H, 13.95; D, 2.33.

7,8-Bis(deuteriomethyl)tetradecane (7b), b.p. 102—103 °C (2 Torr). IR, ν/cm^{-1}: 2910, 2220, 1450, 1400, 1290. ^{1}H NMR, δ: 0.70—1.0 (m, 10 H, CH_3, CH_2D); 1.00—1.50 (m, 22 H, CH, CH_2). MS, *m/z*: 228 $[M]^+$. Found (%): C, 84.06; H, 15.69. $C_{16}H_{32}D_2$. Calculated (%): C, 84.21; H, 14.03; D, 1.75.

10,11-Bis(deuteriomethyl)eicosane (7c), b.p. 162—163 °C (2 Torr). IR, ν/cm^{-1}: 2938, 2857, 1185, 1473, 1382, 1075, 750. ^{1}H NMR, δ: 0.62—0.93 (m, 10 H, CH_3CH_2D); 1.15—1.48 (m, 34 H, CH, CH_2). MS, *m/z*: 312 $[M]^+$. Found (%):

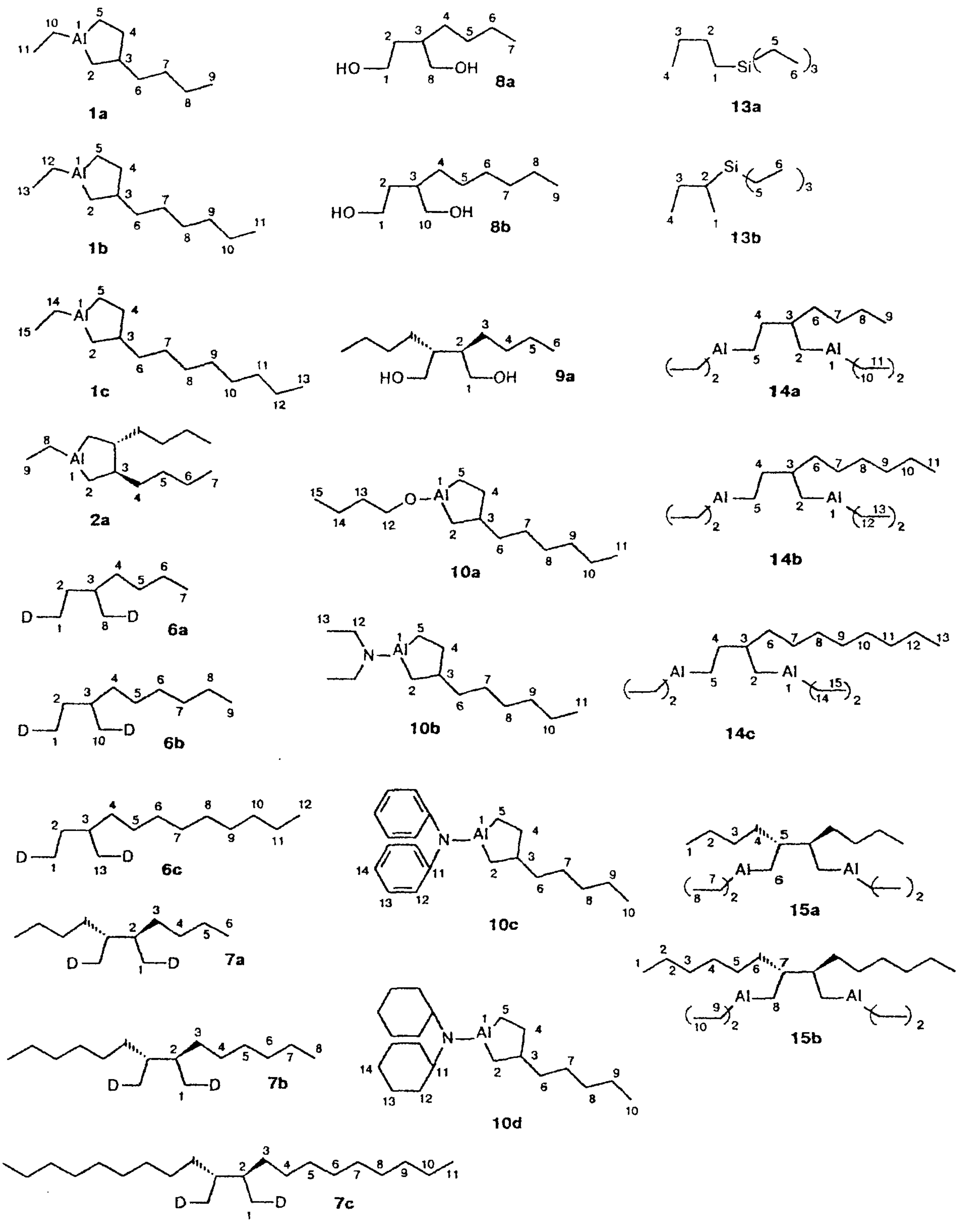


Fig. 1. Numeration of atoms in molecules of OAC and their hydrolysis and oxidation products **1a—c**, **2a**, **6a—c**, **7a—c**, **8a,b**, **9a**, **10a—d**, **13a,b**, **14a—c**, and **15a,b** studied by ^{13}C NMR spectroscopy.

Table 1. ^{13}C NMR spectra of OAC and their hydrolysis and oxidation products **1a—c**, **2a**, **6a—c**, **7a—c**, **8a,b**, **9a**, **10a—d**, **13a,b**, **14a—c**, and **15a,b**

Compound	Solvent	d ($^1J_{13C,D}$/Hz)														
		C-1	C-2	C-3	C-4	C-5	C-6	C-7	C-8	C-9	C-10	C-11	C-12	C-13	C-14	C-15
1a	C_7D_8	—	18.36 br.t	43.10 d	34.29 t	8.74 br.t	30.70 t	29.16 t	23.16 t	14.46 q	0.38 br.t	8.74 q				
1b	C_6D_{12}	—	18.30 br.t	41.39 d	34.01 t	8.94 br.t	32.64 t	28.51 t	34.40 t	32.80 t	23.49 t	14.53 q	0.94 br.t	9.26 q		
1c	C_7D_8	—	18.30 br.t	42.49 d	36.18 t	9.26 br.t	32.34 t	28.70 t	29.87 t	30.33 t	29.87 t	32.34 t	23.04 t	14.19 q	1.02 br.t	9.78 q
2a	C_6D_6	—	11.68 br.t	43.97 d	37.68 t	30.25 t	23.58 t	14.35 q	0.72 br.t	8.90 q						
6a	$CDCl_3$	11.25 t (19.4)	29.64 t	34.52 d	36.54 t	29.62 t	23.25 t	14.22 q	19.03 t (19.5)							
6b	$CDCl_3$	11.23 t (19.2)	29.50 t	35.03 d	36.65 t	27.81 t	29.82 t	32.10 t	22.80 t	14.15 q	19.10 t (19.5)					
6c	$CDCl_3$	11.44 t (19.2)	30.82 t	35.10 d	37.38 t	27.82 t	30.43 t	30.54 t	30.52 t	30.15 t	32.65 t	23.32 t	14.45 q	19.24 t (19.5)		
7a	$CDCl_3$	14.25 t (19.5)	36.73 d	34.85 t	30.24 t	22.63 t	14.28 q									
7b	$CDCl_3$	14.10 t (19.4)	36.50 d	34.97 t	27.69 t	29.70 t	31.85 t	22.70 t	14.05 q							
7c	$CDCl_3$	14.97 t (19.2)	36.48 d	34.90 t	27.68 t	29.97 t	29.33 t	29.62 t	29.58 t	31.80 t	22.63 t	14.97 q				
8a	$CDCl_3$	60.79 t	35.89 t	39.23 d	31.78 t	29.67 t	23.45 t	14.23 q	65.98 t							
8b	$CDCl_3$	60.68 t	35.88 t	39.13 d	32.22 t	27.53 t	30.58 t	32.46 t	23.23 t	14.28 q	65.88 t					
9a	$CDCl_3$	60.88 t	42.50 d	29.40 t	30.10 t	23.12 t	14.19 q									
10a	C_6D_6	—	19.14 br.t	34.75 d	34.12 t	9.06 br.t	32.08 t	26.62 t	29.15 t	32.08 t	22.98 t	14.13 q	70.77 t	29.28 t	19.01 t	14.13 q
10b	C_6D_6	—	14.39 br.t	33.20 d	34.16 t	9.51 br.t	32.80 t	26.62 t	29.22 t	32.15 t	22.98 t	14.06 q	44.11 t	14.06 q		
10c	C_6D_6	—	17.49 br.t	33.45 d	32.41 t	9.45 br.t	33.97 t	27.60 t	32.41 t	23.04 t	14.19 q	143.93 s	117.46 d	129.3 d	120.33 d	
10d	C_6D_6	—	18.60 br.t	34.20 d	32.58 t	9.81 br.t	32.19 t	26.79 t	32.19 t	23.02 t	14.11 q	53.26 d	34.79 t	25.49 t	26.79 t	
13a	C_6D_6	22.90 t	31.97 t	22.93 t	13.70 q	2.58 t	7.78 q									
13b	C_6D_6	14.15 q	25.14 d	19.22 t	13.89 q	2.58 t	7.78 q									
14a	C_6D_6	—	18.42 br.t	44.70 d	25.64 t	8.90 br.t	33.90 t	31.56 t	22.72 t	14.00 q	1.06 br.t	9.00 q				
14b	C_6D_6	—	18.16 br.t	44.24 d	25.26 t	8.99 br.t	34.03 t	25.26 t	29.09 t	32.15 t	22.85 t	14.20 q	0.74 br.t	8.99 q		
14c	C_7D_8	—	18.15 br.t	44.55 d	29.34 t	9.76 br.t	34.15 t	25.63 t	29.86 t	29.53 t	29.86 t	32.26 t	23.03 t	14.18 q	0.72 br.t	8.85 q
15a	C_6D_6	14.00 q	22.72 t	31.56 t	33.90 t	44.70 d	18.42 br.t	1.06 br.t	9.00 q							
15b	C_6D_6	14.20 q	22.85 t	32.15 t	29.09 t	25.26 t	34.03 t	44.24 d	18.16 br.t	0.74 t	8.99 q					

C, 84.53; H, 15.26. $C_{22}H_{44}D_2$. Calculated (%): C, 84.62; H, 14.10; D, 1.28.

2-(*n*-Butyl)butane-1,4-diol (8a), b.p. 99—100 °C (2 Torr), n_D^{22} 1.4543. IR, ν/cm^{-1}: 3343, 2948, 2868, 1470, 1385, 1055, 740. ^{1}H NMR (CCl_4), δ: 0.85 (t, 3 H, CH_3); 1.20—1.76 (m, 9 H, CH, CH_2); 3.18—3.76 (m, 4 H, CH_2O); 4.85 (s, 2 H, OH). Found (%): C, 65.62; H, 12.27. $C_8H_{18}O_2$. Calculated (%): C, 65.75; H, 12.33.

2-(*n*-Hexyl)butane-1,4-diol (8b), b.p. 129—130 °C (2 Torr), n_D^{24} 1.4532. IR, ν/cm^{-1}: 3350, 2935, 2880, 1470, 1380, 1060, 745. ^{1}H NMR (CCl_4), δ: 0.85 (t, 3 H, CH_3); 1.20—1.79 (m, 13 H, CH, CH_2); 3.19—3.80 (m, 4 H, CH_2O); 5.00 (s, 2 H, OH). Found (%): C, 68.85; H, 12.56. $C_{10}H_{22}O_2$. Calculated (%): C, 68.97; H, 12.64.

2-(*n*-Nonyl)butane-1,4-diol (8c), b.p. 130 °C (2 Torr), n_D^{24} 1.4602. IR, ν/cm^{-1}: 3335, 2935, 2868, 1475, 1390, 1070, 735. ^{1}H NMR (CCl_4), δ: 0.90 (t, 3 H, CH_3, $J_{H,H}$ = 7 Hz); 1.20—1.85 (m, 19 H, CH, CH_2); 3.25—3.80 (m, 4 H, CH_2O); 4.90 (s, 2 H, OH). Found (%): C, 72.09; H, 12.88. $C_{13}H_{28}O_2$. Calculated (%): C, 72.22; H, 12.96.

2,3-Di(*n*-butyl)butane-1,4-diol (9a), R_f 0.55 (hexane—acetone, 10 : 3). IR, ν/cm^{-1}: 3335, 2943, 2874, 1470, 1394, 1062, 735. ^{1}H NMR, δ: 0.70—1.05 (m, 6 H, CH_3); 1.10—1.75 (m, 14 H, CH, CH_2), 3.25—3.60 (m, 4 H, CH_2O), 4.75 (br.s, 2 H, OH). Found (%): C, 71.17; H, 12.78. $C_{12}H_{26}O_2$. Calculated (%): C, 71.29; H, 12.87.

(*n*-Butyl)triethylsilane (13a), ^{1}H NMR, δ: 0.42—0.75 (m, 8 H, CH_2Si); 1.17—1.71 (m, 4 H, CH_2). Found (%): C, 69.71; H, 13.87. $C_{10}H_{24}Si$. Calculated (%): C, 69.77; H, 13.95.

(Butan-2-yl)triethylsilane (13b), ^{1}H NMR, δ: 0.80—1.02 (m, 9 H, CH_3); 0.45—0.64 (m, 6 H, CH_2Si); 0.85—1.05 (m, 6 H, CH_3); 1.16—1.50 (m, 3 H, CH_2, CHSi). Found (%): C, 69.85; H, 13.89. $C_{10}H_{24}Si$. Calculated (%): C, 69.77; H, 13.95.

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