Intermolecular Cycloalumination of Cyclic and Acyclic Alkynes with Et_nAlCl_{3-n} in the Presence of Cp_2ZrCl_2

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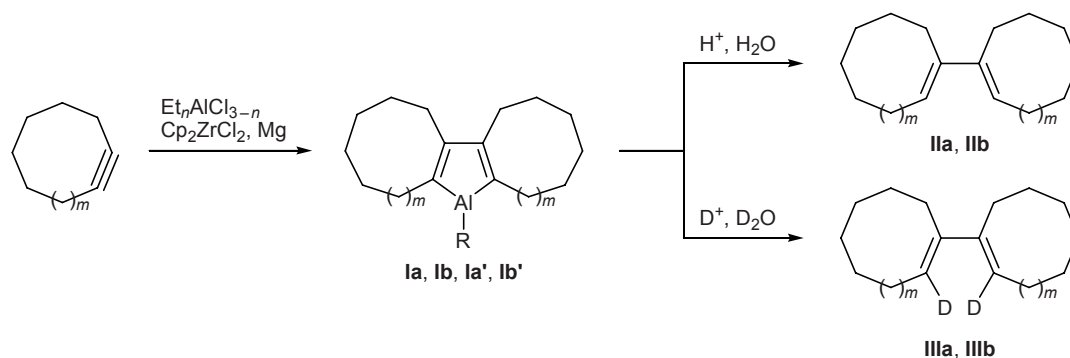*e-mail: ink@anrb.ru*

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Abstract—Intramolecular cycloalumination of cyclic and acyclic alkynes with Et_nAlCl_{3-n} ($n = 0, 1$) in the presence of Cp_2ZrCl_2 gave previously unknown unsaturated bi- and tricyclic organoaluminum compounds in up to 80% yield.

It is known [1], that internal alkynes react with $EtAlCl_2$ in the presence of Cp_2ZrCl_2 as catalyst to give the corresponding cycloalumination products. From 1,2-diaryl-, 1-alkyl-2-aryl-, and 1-aryl-2-allylacetylenes we thus synthesized 2,3,4,5-tetrasubstituted aluminacyclopenta-2,4-dienes. However, the behavior of cyclic alkynes under catalytic cycloalumination conditions remained unclear. With a view to elucidate whether cyclic alkynes can be involved in cycloalumination and obtain new organoaluminum compounds, di- and tricyclic aluminacyclopenta-2,4-dienes, we examined reactions of cyclic alkynes with Et_nAlCl_{3-n} ($n = 0, 1$) in the presence of Cp_2ZrCl_2 which is widely used as catalyst in cycloalumination of unsaturated compounds (Dzhemilev reaction) [2–10]. As substrates we used cyclooctyne, cyclododecyne, and acyclic internal alkynes. The reactions were carried out with commercially available $AlCl_3$, $EtAlCl_2$, and Cp_2ZrCl_2.

Cyclooctyne reacted with $EtAlCl_2$ in the presence of Cp_2ZrCl_2 and magnesium powder (molar ratio cyclooctyne–$EtAlCl_2$–Mg–Cp_2ZrCl_2 $10:7.5:7.5:1$) under the conditions developed previously [1] (THF, 20°C, 6 h) to give tricyclic organoaluminum compound **Ia** in ~78% yield. The structure of **Ia** was determined by analysis of the ^{13}C NMR spectra of its hydrolysis and deuterolysis products **IIa** and **IIIa** (Scheme 1). The ^{13}C NMR spectrum of **IIa** contained two downfield signals at δ_C 126.33 and 140.23 ppm, which were assigned to double-bonded carbon atoms, and six signals in the region δ_C 25–31 ppm due to sp^3-hybridized carbon atoms in the cyclic fragments. These data indicated symmetric structure of molecule **IIa**. In the ^{13}C NMR spectrum of **IIIa**, the signal from the double-bonded carbon atom was split into a triplet due to coupling with deuterium ($J_{CD} = 23.4$ Hz), and the upfield α-isotope shift of the triplet center was $\Delta\delta_C = 0.31$ ppm

Scheme 1.

$n = 0, 1$; $m = 1$ (**a**), 5 (**b**); **I**, R = Et; **I′**, R = Cl.

(relative to the corresponding signal of **IIa**). The signals were assigned using the additivity scheme and increments for cyclooctene given in [11]. The molecular weights of **IIa** and **IIIa** (218 and 220, respectively) were determined by mass spectrometry.

On the basis of the ^{1}H and ^{13}C NMR and mass spectra, the hydrolysis and deuterolysis products were identified as 1,1′-bi(cyclooctene) (**IIa**) and 2,2′-dideutero-1,1′-bi(cyclooctene) (**IIIa**). The formation of 1,4-dideutero derivative **IIIa** from compound **Ia** suggests that the aluminum atom in the latter is linked to C^9 and C^{11}. Therefore, compound **Ia** was assigned the structure of 10-ethyl-10-aluminatricyclo[6.9.0.0^{2,9}]-heptadeca-1(11),2(9)-diene.
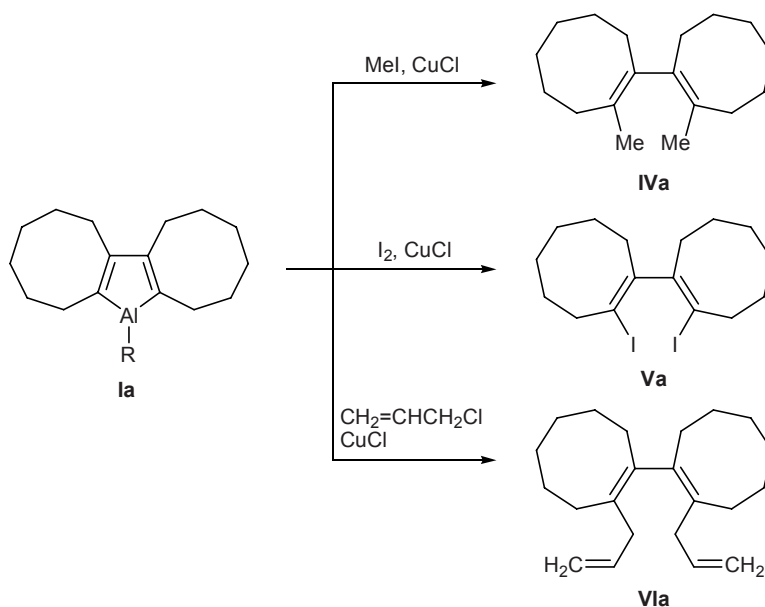
Like cyclooctyne, the Cp_2ZrCl_2-catalyzed reaction of cyclododecyne with $EtAlCl_2$ under analogous conditions led to the formation of 14-ethyl-14-alyuminatricyclo[10.13.0.0^{2,13}]pentacosa-1(15),2(13)-diene (**Ib**) in 64% yield. No appreciable effect on the product yield was observed when $AlCl_3$ was used instead of $EtAlCl_2$ in the intermolecular cycloalumination of cycloalkynes. After acid hydrolysis of organoaluminum compounds **Ia′** and **Ib′**, hydrocarbons **IIa** and **IIb** were isolated from the reaction mixtures in 76 and 65% yield, respectively.

Aluminacyclopenta-2,4-diene **Ia** was brought into reactions with iodine, methyl iodide, and allyl chloride in the presence of a catalytic amount of copper(I) chloride (10 mol %). Compound **Ia** showed high reactivity in these processes, and the corresponding prod-
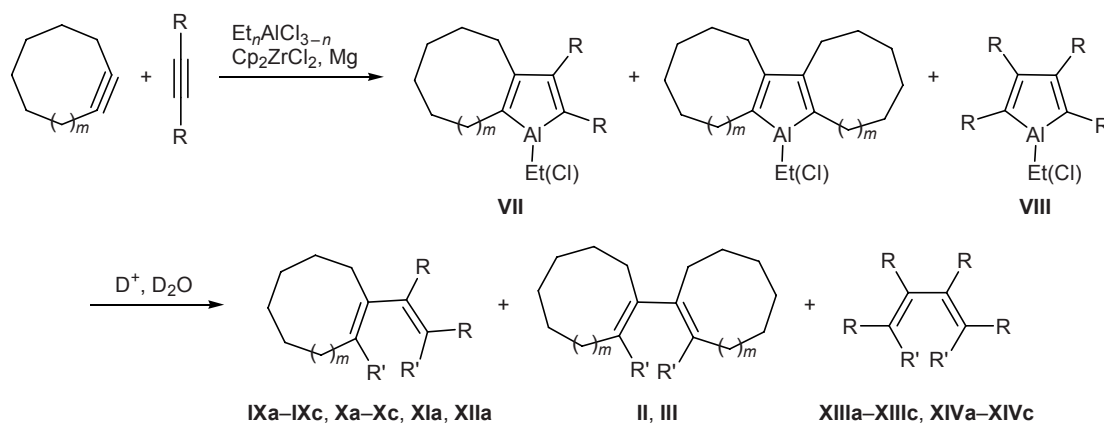
ucts, 2,2′-diiodo-1,1′-bi(cyclooctene) (**IVa**), 2,2′-dimethyl-1,1′-bi(cyclooctene) (**V**), and 2,2′-diallyl-1,1′-bi(cyclooctene) (**VI**) were isolated in 70–85% yield (Scheme 2).

We also made an attempt to perform joint cycloalumination of cycloalkynes with linear terminal alkynes with a view to synthesize new organoaluminum compounds, bicyclic aluminacyclopenta-2,4-dienes. By reaction of equimolar amounts of cyclooctyne and hex-3-yne as model compounds we obtained a mixture of aluminacyclopenta-2,4-dienes **VII**, **I**, and **VIII** at a ratio of 1.5 : 1 : 1 (according to the GLC data for the hydrolysis products) in an overall yield of ~91% (Scheme 3). The reaction conditions were optimized so that to maximize the yield of bicyclic compound **VII**. Variation of the reactant ratio, solvent nature, temperature, and reaction time allowed us to find conditions ensuring formation of **VII** in 75% yield (ratio cyclooctyne–hex-3-yne–$EtAlCl_2$–Mg–Cp_2ZrCl_2 10 : 12 : 16.5 : 16.5 : 2.2, THF, 20°C, 6 h); in this case, the ratio of aluminacyclopenta-2,4-dienes **VII**, **I**, and **VIII** was 8 : 1 : 2, and the overall yield reached 94%. In the reaction with $AlCl_3$, other conditions being equal, the product ratio and the overall yield were almost the same as in the reaction with $EtAlCl_2$. Minor products **VIIIa**–**VIIIc** were identified by comparing physical properties and spectral data of the corresponding hydrolysis products **XIa**–**XIc** with those of authentic samples [1]. Bicyclic compound **VII** was assigned the structure of 9,10,11-triethyl-9-aluminabicyclo[6.3.0]undeca-1(8),10-diene on the basis of the ^{1}H and ^{13}C NMR and

Scheme 2.

Scheme 3.

$n = 0, 1$; **IX**, **X**, $m = 1$; **XI**, **XII**, $m = 5$; **IX**, **XI**, R′ = H; **X**, **XII**, R′ = D; R = Et (**a**), Pr (**b**), Bu (**c**).

mass spectra of hydrolysis and deuterolysis products **IX** and **X**.

Our subsequent experiments showed that analogous pattern is typical of reactions of cyclooctyne with oct-4-yne and dec-5-yne, as well as of reactions of cyclododecyne with hex-3-yne. However, the overall yield decreased from 94 to 67% in going from hex-3-yne to acetylenes with longer alkyl groups (oct-4-yne and dec-5-yne) and from cyclooctyne to cyclododecyne having a larger ring.

Thus we were the first to perform Cp_2ZrCl_2-catalyzed intermolecular cycloalumination of cycloalkynes and joint cycloalumination of cycloalkynes and linear internal alkynes with Et_nAlCl_{3-n} and obtain new bi- and tricyclic unsaturated organoaluminum compounds. The products possess a broad synthetic potential and attract interest as new co-catalysts for Ziegler–Natta systems and promising organoaluminum reagents for organic and organometallic synthesis.

EXPERIMENTAL

All reactions with organometallic compounds were carried out in a stream of dry argon. Tetrahydrofuran and diethyl ether were dehydrated by boiling over metallic sodium. Dichloroethylaluminum (86%) and trichloroaluminum (99.8%) were commercial products. Gas–liquid chromatography was performed on a Chrom-5 instrument (1200×3-mm column packed with 5% of SE-30 or 15% of PEG-6000 on Chromaton N-AW; carrier gas helium). The elemental compositions were determined on a Carlo Erba Model 1106 analyzer. The mass spectra (electron impact, 70 eV) were recorded on an MKh-1306 spectrometer (ion source temperature 200°C). The ^{1}H and ^{13}C NMR

spectra were measured from solutions in $CDCl_3$ on a JEOL FX-90Q spectrometer at 89.55 (^{1}H) and 22.5 MHz (^{13}C). The yields of organoaluminum compounds were determined by GLC analysis of the hydrolysis products using *n*-hexadecane as internal standard. The complex Cp_2ZrCl_2 was synthesized from $ZrCl_4$ according to the procedure described in [12]. Initial cycloalkynes were prepared as reported in [13]. Compounds **XIIIa–XIIIc** and **XIVa–XIVc** were identified by comparison with authentic samples [1].

Reaction of cycloalkynes with Et_nAlCl_{3-n} in the presence of Cp_2ZrCl_2 (*general procedure*). A glass reactor was filled with dry argon and charged at ~0°C under stirring with 1.0 mmol of Cp_2ZrCl_2, 7.5 mmol of magnesium powder, 10 mmol of cyclooctyne or cyclododecyne, 10 ml of THF, and 7.5 mmol of $EtAlCl_2$ or $AlCl_3$. The mixture was allowed to warm up to room temperature (20–22°C), stirred for 6 h, treated with 7–10% hydrochloric acid (or a 7–10% solution of DCl in D_2O), and extracted with hexane. The extract was dried over $MgSO_4$, and the products were isolated by vacuum distillation.

1,1′-Bi(cyclooctene) (IIa). Yield 78%, bp 121–123°C (1 mm). ^{1}H NMR spectrum, δ, ppm: 1.10–1.55 m (16H, CH_2), 2.16–2.38 m (8H, **C**H_2CH=), 5.29–5.38 m (2H, CH=). ^{13}C NMR spectrum, δ$_C$, ppm: 25.32, 26.17, 27.41, 27.54, 29.36, 30.60, 126.33, 140.23. Found, %: C 87.79; H 11.92. *M* 218. $C_{16}H_{26}$. Calculated, %: C 88.00; H 12.00.

(2,2′-^{2}H$_2$)-1,1′-Bi(cyclooctene) (IIIa). ^{1}H NMR spectrum, δ, ppm: 1.12–1.58 m (16H, CH_2), 2.14–2.36 m (8H, CH_2C=). ^{13}C NMR spectrum, δ$_C$, ppm: 25.34, 26.12, 27.45, 27.61, 29.45, 30.63, 126.02 t (J_{CD} = 23.4 Hz), 140.24. Found, %: C 87.05;

H+D 12.76. M 220. $C_{16}H_{24}D_2$. Calculated, %: C 87.20; H 10.98; D 1.82.

1,1′-Bi(cyclododecene) (IIb). Yield 64%, bp 210–212°C (1 mm). ^{1}H NMR spectrum, δ, ppm: 1.05–1.49 m (32H, CH$_2$), 2.11–2.36 m (8H, –C**H$_2$**CH=), 5.31–5.41 m (2H, CH=). ^{13}C NMR spectrum, δ_C, ppm: 26.07, 26.37, 26.56, 26.82, 27.05, 28.09, 28.61, 29.82, 31.38, 32.13, 121.64, 136.72. Found, %: C 87.12; H 12.75. M 330. $C_{24}H_{42}$. Calculated, %: C 87.19; H 12.81.

(2,2′-^{2}H$_2$)-1,1′-Bi(cyclododecene) (IIIb). ^{1}H NMR spectrum, δ, ppm: 1.08–1.51 m (32H, CH$_2$), 2.14–2.39 m (8H, CH$_2$C=). ^{13}C NMR spectrum, δ_C, ppm: 26.08, 26.37, 26.54, 26.86, 27.11, 28.21, 28.68, 29.81, 31.39, 32.23, 121.34 t (J_{CD} = 23.5 Hz), 137.15. Found, %: C 86.55; H+D 13.21. M 332. $C_{24}H_{40}D_2$. Calculated, %: C 86.67; H 12.12; D 1.21.

2,2′-Diiodo-1,1′-bi(cyclooctene) (IVa). A glass reactor was filled with dry argon and charged at ~0°C under stirring with 1.0 mmol of Cp$_2$ZrCl$_2$, 7.5 mmol of magnesium powder, 10 mmol of cyclooctyne or cyclo-dodecyne, 10 ml of THF, and 7.5 mmol of EtAlCl$_2$ or AlCl$_3$. The mixture was allowed to warm up to room temperature (20–22°C), stirred for 6 h, and cooled to –20°C, 1 mmol of CuCl–2 Ph$_3$P was added, and a solution of 12.5 mmol of iodine in 8 ml of THF was slowly added dropwise. The mixture was allowed to warm up to room temperature, stirred for 3 h, treated with 5% hydrochloric acid, and extracted with diethyl ether. The extract was dried over MgSO$_4$, and the product was isolated by column chromatography on silica gel L (180–250 μm) using hexane–ethyl acetate (10:1) as eluent. Yield 67%, R_f 0.42. ^{1}H NMR spectrum, δ, ppm: 1.10–1.55 m (16H, CH$_2$), 2.36–2.98 m (8H, C**H$_2$**CH=). ^{13}C NMR spectrum, δ_C, ppm: 25.42, 26.82, 28.09, 31.38, 33.69, 40.34, 101.72, 152.77. Found, %: C 40.48; H 5.09. $C_{16}H_{24}I_2$. Calculated, %: C 40.87; H 5.15.

Reactions of 10-ethyl-10-aluminatricyclo-[6.9.0.0^{2,9}]heptadeca-1(11),2(9)-diene with methyl iodide and allyl chloride (*general procedure*). A glass reactor was filled with dry argon and charged at ~0°C under stirring with 1.0 mmol of Cp$_2$ZrCl$_2$, 7.5 mmol of magnesium powder, 10 mmol of cyclooctyne or cyclo-dodecyne, 10 ml of THF, and 7.5 mmol of EtAlCl$_2$ or AlCl$_3$. The mixture was allowed to warm up to room temperature (20–22°C), stirred for 6 h, and cooled to –20°C, 1 mmol of CuCl–2 Ph$_3$P was added, and 12.5 mmol of methyl iodide or allyl chloride was slowly added dropwise. The mixture was allowed to warm up to room temperature, stirred for 6 h, treated with 5% hydrochloric acid, and extracted with diethyl ether. The solvent was distilled off, and the residue was subjected to vacuum distillation.

2,2′-Dimethyl-1,1′-bi(cyclooctene) (Va). Yield 61%, bp 132–133°C (1 mm). ^{1}H NMR spectrum, δ, ppm: 1.11–1.52 m (16H, CH$_2$), 1.63 s (6H, CH$_3$), 2.17–2.34 m (8H, CH$_2$C=). ^{13}C NMR spectrum, δ_C, ppm: 20.14, 25.12, 26.67, 27.51, 28.24, 29.45, 31.67, 136.33, 143.15. Found, %: C 87.42; H 12.01. M 246. $C_{18}H_{30}$. Calculated, %: C 87.73; H 12.27.

2,2′-Diallyl-1,1′-bi(cyclooctene) (VIa). Yield 55%, bp 171–173°C (1 mm). ^{1}H NMR spectrum, δ, ppm: 1.08–1.54 m (16H, CH$_2$), 2.15–2.32 m (8H, CH$_2$C=), 2.52–2.74 d (4H, CH$_2$CH=), 4.81–4.89 m (4H, H$_2$C=), 5.57–5.84 m (2H, H$_2$C=CH). ^{13}C NMR spectrum, δ_C, ppm: 25.12, 26.31, 26.79, 27.28, 29.37, 32.23, 42.86, 114.35, 136.62, 137.50, 140.72. Found, %: C 87.34; H 11.27. M 298. $C_{22}H_{34}$. Calculated, %: C 88.52; H 11.48.

Joint cycloalumination of cycloalkynes and linear internal alkynes with Et$_n$AlCl$_{3−n}$ in the presence of Cp$_2$ZrCl$_2$. A glass reactor was filled with dry argon and charged at ~0°C uner stirring with 2.2 mmol of Cp$_2$ZrCl$_2$, 16.5 mmol of magnesium powder, 10 mmol of cyclooctyne or cyclododecyne, 12 mmol of hex-3-yne, oct-4-yne, or dec-5-yne, 15 ml of THF, and 16.5 mmol of EtAlCl$_2$ or AlCl$_3$. The mixture was allowed to warm up to room temperature (20–22°C), stirred for 6 h, treated with 7–10% hydrochloric acid or a 7–10% solution of DCl in D$_2$O, and extracted with hexane. The extract was dried over MgSO$_4$, and the products were isolated by vacuum distillation.

1-[(1*E*)-1-Ethylbut-1-en-1-yl]cyclooctene (IXa). Yield 75%, bp 116–117°C (5 mm). ^{1}H NMR spectrum, δ, ppm: 0.85–0.98 m (6H, CH$_3$), 1.20–1.54 m (8H, CH$_2$), 1.68–2.31 m (8H, CH$_2$C=), 5.34–5.68 m (2H, CH=). ^{13}C NMR spectrum, δ_C, ppm: 14.06, 14.17, 20.96, 23.31, 23.36, 25.32, 26.23, 27.41, 27.54, 30.61, 125.32, 126.98, 140.24, 144.73. Found, %: C 87.35; H 12.49. M 192. $C_{14}H_{24}$. Calculated, %: C 87.42; H 12.58.

1-[(1*E*)-1-Ethyl(2-^{2}H)but-1-en-1-yl](2-^{2}H)cyclo-octene (Xa). ^{1}H NMR spectrum, δ, ppm: 0.84–0.99 m (6H, CH$_3$), 1.21–1.53 m (8H, CH$_2$), 1.68–2.30 m (8H, CH$_2$C=). ^{13}C NMR spectrum, δ_C, ppm: 14.07, 14.17, 20.94, 23.28, 23.46, 25.34, 26.24, 27.42, 27.57, 30.63, 125.03 t (J_{CD} = 23.4 Hz), 126.67 t (J_{CD} = 24.0 Hz), 140.26, 144.70. Found, %: C 86.45; H+D 13.29.

M 194. $C_{14}H_{22}D_2$. Calculated, %: C 86.52; H 11.41; D 2.07.

1-[(1E)-1-Propylpent-1-en-1-yl]cyclooctene (IXb). Yield 70%, bp 128–130°C (2 mm). ^{1}H NMR spectrum, δ, ppm: 0.83–0.96 m (6H, CH$_3$), 1.21–1.56 m (12H, CH$_2$), 1.67–2.32 m (8H, CH$_2$C=), 5.31–5.66 m (2H, CH=). ^{13}C NMR spectrum, δ$_C$, ppm: 13.87, 14.62, 22.03, 23.28, 23.35, 25.32, 26.21, 27.35, 27.54, 29.94, 30.31, 30.62, 125.21, 126.65, 140.32, 144.68. Found, %: C 87.05; H 12.76. M 220. $C_{16}H_{28}$. Calculated, %: C 87.19; H 12.81.

1-[(1E)-1-Propyl(2-^{2}H)pent-1-en-1-yl](2-^{2}H)-cyclooctene (Xb). ^{1}H NMR spectrum, δ, ppm: 0.83–0.95 m (6H, CH$_3$), 1.22–1.58 m (12H, CH$_2$), 1.67–2.31 m (8H, CH$_2$C=). ^{13}C NMR spectrum, δ$_C$, ppm: 13.79, 14.05, 22.06, 23.19, 23.35, 25.31, 26.24, 27.36, 27.51, 29.98, 30.28, 30.61, 124.91 t (J_{CD} = 23.5 Hz), 126.31 t (J_{CD} = 24.0 Hz), 141.02, 143.89. Found, %: C 86.37; H+D 13.48. M 222. $C_{16}H_{26}D_2$. Calculated, %: C 86.41; H 11.78; D 1.81.

1-[(1E)-1-Butylhex-1-en-1-yl]cyclooctene (IXc). Yield 58%, bp 135–137°C (1 mm). ^{1}H NMR spectrum, δ, ppm: 0.82–0.97 m (6H, CH$_3$), 1.22–1.56 m (16H, CH$_2$), 1.65–2.31 m (8H, CH$_2$C=), 5.32–5.64 m (2H, CH=). ^{13}C NMR spectrum, δ$_C$, ppm: 14.05, 14.32, 22.05, 23.26, 23.31, 25.38, 26.25, 27.34, 27.55, 27.64, 27.83, 29.98, 30.32, 30.61, 125.42, 126.64, 140.30, 144.69. Found, %: C 86.95; H 12.86. M 248. $C_{18}H_{32}$. Calculated, %: C 87.02; H 12.98.

1-[(1E)-1-Butyl(2-^{2}H)hex-1-en-1-yl](2-^{2}H)cyclo-octene (Xc). ^{1}H NMR spectrum, δ, ppm: 0.82–0.95 m (6H, CH$_3$), 1.22–1.57 m (16H, CH$_2$), 1.65–2.32 m (8H, CH$_2$C=). ^{13}C NMR spectrum, δ$_C$, ppm: 14.02, 14.28, 22.07, 23.18, 23.35, 25.32, 26.26, 27.36, 27.52, 27.59, 27.82, 29.91, 30.21, 30.66, 125.12 t (J_{CD} = 23.5 Hz), 126.38 t (J_{CD} = 24.5 Hz), 141.02, 144.89. Found, %: C 86.27; H+D 13.46. M 250. $C_{18}H_{30}D_2$. Calculated, %: C 86.32; H 12.07; D 1.61.

1-[(1E)-1-Ethylbut-1-en-1-yl]cyclododecene (XIa). Yield 68%, bp 156–158°C (10 mm). ^{1}H NMR spectrum, δ, ppm: 0.79–0.95 m (6H, CH$_3$), 1.13–1.52 m (16H, CH$_2$), 1.68–2.16 m (8H, CH$_2$C=), 5.24–5.36 m (2H, CH=). ^{13}C NMR spectrum, δ$_C$, ppm: 13.70, 13.86, 21.77, 22.43, 22.62, 23.11, 24.05, 24.38, 24.48, 24.83, 24.93, 27.44, 30.11, 32.13, 126.59, 126.95, 141.79, 141.99. Found, %: C 86.94; H 12.86. M 248. $C_{18}H_{32}$. Calculated, %: C 87.02; H 12.98.

1-[(1E)-1-Ethyl(2-^{2}H)but-1-en-1-yl](2-^{2}H)cyclo-dodecene (XIIa). ^{1}H NMR spectrum, δ, ppm: 0.78–0.95 m (6H, CH$_3$), 1.11–1.53 m (16H, CH$_2$), 1.67–2.14 m (8H, CH$_2$C=). ^{13}C NMR spectrum, δ$_C$, ppm: 13.72, 13.85, 21.78, 22.43, 22.64, 23.15, 24.16, 24.37, 24.41, 24.83, 24.89, 27.44, 30.12, 32.13, 126.28 t (J_{CD} = 23.4 Hz), 126.37 t (J_{CD} = 23.4 Hz), 141.78, 141.87. Found, %: C 86.27; H+D 13.49. M 250. $C_{18}H_{30}D_2$. Calculated, %: C 86.32; H 12.07; D 1.61.

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