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Cyclo- and carbomagnesiation of 1,2-dienes catalyzed by Zr complexes

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Abstract—Cyclo- and carbomagnesiation of 1,2-dienes with EtMgR′ (R′=Et, Br) in the presence of Cp_2ZrCl_2 catalyst lead to alkylidenemagnesiocyclopentanes. Deuterolysis provides insights into the reaction pathways.

1. Introduction

Introduction of Zr-containing catalysts into the application of organomagnesium reagents stimulated further investigations directed toward the development of novel synthetic methods to produce 'nonGrignard' organomagnesium compounds.

Carbomagnesiation of olefins with RMgR′ (R′=alkyl, Hal) in the presence of Cp_2ZrCl_2 complex was first carried out[1] in 1983 and successfully developed since then,[2–4] and is now widely used for the selective construction of C–C and C–M bonds.[5–13] In 1989, these investigations[1–4] resulted in the discovery of the cyclomagnesiation reaction[14] of olefins which allowed the formation of magnesiumcyclopentanes and (or) 1,4-dimagnesium compounds[15–21] in high regio- and stereoselectivity.

In accordance with the reported data,[8,18,20] the cyclo- and carbomagnesiation reactions of olefins catalyzed by Cp_2ZrCl_2 are envisioned to proceed via zirconacyclopentane intermediates responsible for the formation of magnesiumcyclopentanes (and/or acyclic 1,4-dimagnesium compounds). The chemoselectivity of the reactions was found to depend upon the solvent nature, temperature and the ratio of starting compounds.[8,20]

The reactions of organomagnesium compounds (OMC) were studied predominantly with substrates such as α-olefins, norbornenes[1–4] and α,ω-dienes.[7,18] Conjugated 1,3-dienes are essentially inert toward cyclo- and carbomagnesiation.

We now report on the results of our further investigations in the field of cyclo- and carbomagnesiation of 1,2-dienes specifically alkyl-, aryl- and cycloalkenylsubstituted allenes, in the presence of RMgR′ and Zr-containing catalysts.[†]

2. Results and discussion

Two different observations are characteristic for the interaction of 1,2-alkadienes and organomagnesium reagents. Thus, the reaction of allenes with Et_2Mg (twofold excess) in diethyl ether (Et_2O) effected by catalytic amounts of Cp_2ZrCl_2 (5 mol%) under the optimized conditions (Et_2O, ~0 °C, 8 h) was found to produce a mixture of regioisomeric magnesiocyclopentanes **1**, **2**, **3** (and/or 1,4-dimagnesium compounds) and the minor products of carbomagnesiation **4**, **5** and **6**, established by deuterolysis (Scheme 1). The reaction mixture upon deuterolysis afforded di- (**7**/**8**/**9**=6:3:1) and monodeuterated olefins (**10**/**11**/**12**=6:3:1) in a ratio of approximately 6:1 (in accord with mass spectral analysis) in a combined yield of 80%. This process did not proceed in the presence of Cp_2TiCl_2.

In contrast to the previous reaction, the interaction between Et_2Mg and allenes in tetrahydrofuran (THF) in the presence of Cp_2ZrCl_2 catalyst at ambient temperature was found to generate predominantly the carbomagnesiation adducts **4**, **5** and **6**, which on deuterolysis afforded a mixture of mono-

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Keywords: Catalysis; Zr complexes; Organomagnesium compounds; Cyclomagnesiation; Carbomagnesiation; 1,2-Dienes; Deuterolysis.

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[†] For comparison, in certain experiments Ti complexes were used as catalysts to gain insight into the factors influencing the carbo- and cyclomagnesiation reactions.

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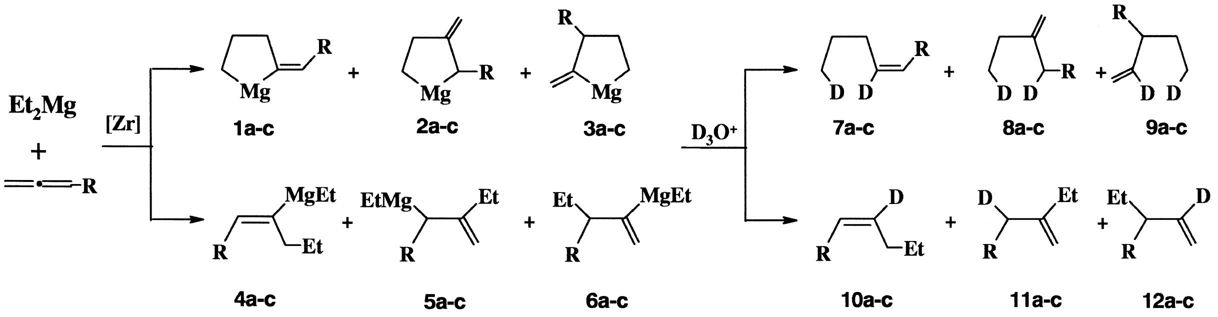
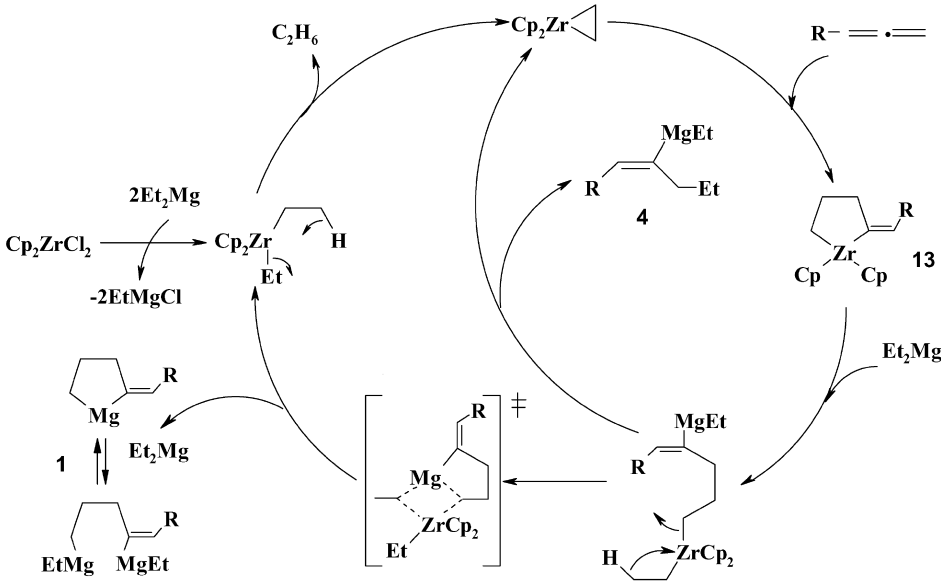


a: R = n-C$_5$H$_{11}$; b: R = n-C$_7$H$_{15}$; c: R = n-CH$_2$Ph

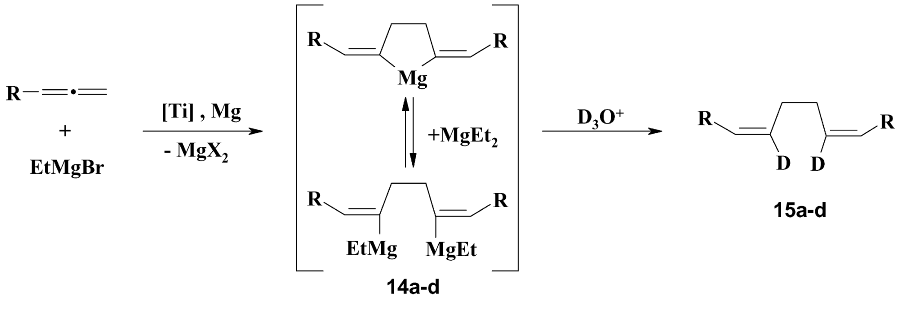
Scheme 1.

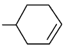


Scheme 2.

(**10**/**11**/**12**=1:2:2) and dideuterated olefins (**7**/**8**/**9**=1:2:2) in a ratio of 8:1 in a combined yield of 76%. In accordance with the reported data,[6,8,18,20] we have represented the formation of cyclic- and acyclic OMC as a sequence of chemical steps combined into a catalytic cycle (Scheme 2), that involves the generation of zirconacyclopentane complexes **13** as the key intermediates. The consecutive transformations of the latter species depending on the reaction conditions affected by Et$_2$Mg led to adducts of cyclo-(**1**) or carbomagnesiation **4**.

Analogous to the synthesis of aluminacyclopentanes described in literature[22] we have succeeded in the synthesis of magnesiocyclopentanes from RMgHal and Mg (as an acceptor of halogen ions) in the presence of Ti-containing catalysts. Thus, the interaction of 1,2-alkadienes with a twofold excess of EtMgBr in the presence of chemically activated Mg[23] and effected by catalytic amounts of Cp$_2$TiCl$_2$ (5 mol%) under the optimum conditions (Et$_2$O, rt, 8 h) led to 2,5-dialkylidenemagnesiocyclopentanes **14a** and **14b** (and/or 1,4-dimagnesium compounds), which on



[Ti] = Cp $_2$TiCl$_2$; X = Cl, Br ; a: R = n-C$_5$H$_{11}$; b: R = n-C$_7$H$_{15}$; c: R = (CH$_2$)$_2$–  ; d: R = CH$_2$Ph

Scheme 3.

deuterolysis afforded 1,6-dialkyl-2,5-dideuteriohexadienes **15a** and **15b** in a combined yield of more than 90% (Scheme 3).

The replacement of THF by Et_2O did not change product yield and composition of the OMC. However, the use of Cp_2ZrCl_2 as a catalyst instead of Cp_2TiCl_2 decreased the cyclomagnesiation selectivity leading to a regioisomeric mixture of unsaturated OMC. It should be noted that our attempts to effect cyclomagnesiation of allenes using inactivated Mg were unsuccessful.

Similarly, the interaction of ethylmagnesium bromide and 5-(cyclohex-3-enyl)-1,2-pentadiene or 4-phenyl-1,2-butadiene in the presence of Cp_2TiCl_2 catalyst was found to afford **14c** and **14d**.

The selectivity of the cyclomagnesiation of allenes with RMgHal assisted by Cp_2TiCl_2 was established to depend essentially upon the structure of the initial 1,2-diene. Thus, the interaction of phenylallene with ethylmagnesium bromide in the presence of Cp_2TiCl_2 and activated Mg led to unsaturated OMC, which upon deuterolysis afforded a complicated regioisomeric mixture of dideuteriodienes containing di- and trisubstituted double bonds.

3. Conclusion

The interaction between Et_2Mg and 1,2-dienes catalyzed by Cp_2ZrCl_2 was found to afford the products of cyclo- and carbomagnesiation. The reaction of 1,2-dienes with EtMgBr in the presence of activated Mg led to the formation of 2,5-dialkylidenemagnesiumcyclopentanes (and/or unsaturated 1,4-dimagnesium compounds) if Cp_2TiCl_2 was used as a catalyst. These reactions reveal a considerable synthetic potential and are under further investigation in our laboratory.

4. Experimental

4.1. General

All solvents were dried (hexane over $LiAlH_4$, Et_2O and THF over Na) and freshly distilled prior to use. Dialkyl Mg derivatives, prepared from solid Mg alkylate according to a literature method,[24] were used as ethereal solutions. All reactions were carried out under a dry argon atmosphere. The reaction products were analyzed using chromatography on a 'Chrom-5' instrument (2 m×3 mm column packed with 5% of SE-30 and 3 m×3 mm column packed with 5% PEG-6000 on Chromaton N-AW, carrier gas—He). Preparative separation was performed on a 'Carlo Erba Fractovap Mod.GW' instrument (4 m×6 mm column, 5% SE-30 on Chromaton N-AW, helium as a carrier gas, 300 mL min^{-1}). Infrared spectra (IR) were recorded on an IR-75 instrument (thin film). Mass spectral measurements were performed on a MX-1306 spectrometer at 70 eV and working temperature 200 °C. The ^1H and ^{13}C NMR spectra were recorded in $CDCl_3$ on a 'Bruker AM-300' spectrometer (75.46 MHz for ^{13}C and 300 MHz for ^1H). The chemical shifts are reported as δ values in ppm relative to internal standard Me_4Si. NMR ^{13}C spectra were edited by *J*-modulation (JMOD) on CH constants.

4.2. Cyclomagnesiation of 1,2-dienes with Et_2Mg catalyzed by Cp_2ZrCl_2 (method A)

A 50 mL glass reactor was charged with Et_2Mg (1.5 M solution in diethyl ether, 25 mmol), 1,2-diene (10 mmol) and Cp_2ZrCl_2 (0.5 mmol) under a dried argon atmosphere at 0 °C and stirred for 10 h. The reaction mixture was allowed to warm to room temperature and quenched with an 8–10% aqueous solution of HCl (or DCl, 10–12% solution in D_2O). The crude products were extracted with Et_2O or hexane. After the solvent was removed, the residue was distilled in vacuo, and pure adducts were separated by the preparative GLC.

4.3. Carbomagnesiation of 1,2-dienes with Et_2Mg catalyzed by Cp_2ZrCl_2 (method B)

A 50 mL glass reactor was charged with Et_2Mg (1.5 M solution in THF, 25 mmol), 1,2-diene (10 mmol) and Cp_2ZrCl_2 (0.5 mmol) under a dried argon atmosphere at 0 °C. The resulting solution was allowed to warm to rt (ca. 20 °C) and stirred for 10 h. The reaction mixture quenched with an 8–10% aqueous solution of HCl (or DCl, 10–12% solution in D_2O). The crude products were extracted with Et_2O or hexane. After the solvent was removed, the residue was distilled in vacuo, and pure adducts were separated by the preparative GLC.

4.3.1. 1,4-Dideuteriodec-4*Z*-ene (7a). ^1H NMR ($CDCl_3$): δ 0.89 (t, 5H, J=6.0 Hz, CH_2D and CH_3); 1.32 (m, 8H, CH_2), 2.00 (m, 4H, $CH_2-{=}-$), 5.38 (t, H, J=6.0 Hz, $C\mathit{H}{=}CD$); ^{13}C NMR ($CDCl_3$): δ 13.47 (t, J_{CD}=19.0 Hz), 14.06, 22.66, 22.82, 29.30, 31.00, 31.62, 32.00, 130.21; IR (thin film): 2920, 2910, 2830, 2160, 1620, 1450, 1370, 1100, 880, 720 cm^{-1}; MS, *m/z*: 142 (M^+). Anal. calcd for $C_{10}H_{18}D_2$: C, 84.43; H, 12.76; D, 2.81. Found: C, 84.23; H, 15.34.

4.3.2. 1,4-Dideuteriododec-4*Z*-ene (7b). ^1H NMR ($CDCl_3$): δ 0.89 (t, 5H, J=6.0 Hz, CH_2D and CH_3), 1.32 (m, 10H, CH_2), 2.00 (m, 4H, $CH_2-{=}-$), 5.38 (t, H, J=6.0 Hz, $C\mathit{H}{=}CD$); ^{13}C NMR ($CDCl_3$): δ 12.75 (t, J_{CD}=19.0 Hz), 14.15, 22.86, 23.06, 28.95, 29.50, 29.66, 31.05, 31.64, 32.08, 130.21, 130.84 (t, J_{CD}=23.5 Hz); MS, *m/z*: 170 (M^+). Anal. calcd for $C_{12}H_{22}D_2$: C, 84.63; H, 13.02; D, 2.35. Found: C, 84.51; H, 15.16.

4.3.3. (3,6-Dideuteriohex-2*Z*-ene-1-yl)benzene (7c). ^1H NMR ($CDCl_3$): δ 0.91 (t, 2H, J=6.0 Hz, CH_2D), 1.38 (m, 2H, CH_2), 2.15 (m, H, $CH_2-{=}-$), 3.41 (d, 2H, Ph–$C\mathit{H_2}-{=}$), 5.50 (m, 2H, $C\mathit{H}{=}C\mathit{H}$), 7.25 (m, 5H, Ph); ^{13}C NMR ($CDCl_3$): δ 13.53 (t, J_{CD}=19.5 Hz), 22.75, 29.17, 39.07, 125.90, 128.18, 128.45, 128.97, 141.37; MS, *m/z*: 162 (M^+). Anal. calcd for $C_{12}H_{14}D_2$: C, 88.83; H, 8.69; D, 2.46. Found: C, 88.62; H, 10.97.

4.3.4. 2-(2-Deuterioethane-1-yl)-3-deuteriooct-1-ene (8a). ^1H NMR ($CDCl_3$): δ 0.89 (t, 5H, J=6.0 Hz, CH_2D and CH_3), 1.32 (m, 8H, CH_2), 2.00 (m, 3H, $C\mathit{H_2}-(C{=}CH_2)-C\mathit{H}D$), 4.83 (s, 2H, $\mathit{H_2}C{=}$); ^{13}C NMR

($CDCl_3$): δ 12.41 (t, J_{CD}=19.5 Hz), 14.06, 22.81, 27.21, 29.75, 31.43, 31.96, 36.02 (t, J_{CD}=19.0 Hz), 107.82, 151.82; MS, *m/z*: 142 (M^+). Anal. calcd for $C_{10}H_{18}D_2$: C, 84.43; H, 12.76; D, 2.81. Found: C, 84.25; H, 15.37.

4.3.5. 2-(2-Deuterioethane-1-yl)-3-deuteriodec-1-ene (8b). ^1H NMR ($CDCl_3$): δ 0.89 (t, 5H, *J*=6.0 Hz, CH_2D and CH_3), 1.32 (m, 12H, CH_2), 2.00 (m, 3H, CH_2–($C{=}CH_2$)–CHD), 4.85 (s, 2H, $H_2C{=}$); ^{13}C NMR ($CDCl_3$): δ 12.41 (t, J_{CD}=19.5 Hz), 14.06, 22.84, 27.23, 29.50, 29.75, 28.95, 31.44, 32.03, 36.08 (t, J_{CD}=19.0 Hz), 107.85, 151.84; MS, *m/z*: 170 (M^+). Anal. calcd for $C_{12}H_{22}D_2$: C, 84.63; H, 13.02; D, 2.35. Found: C, 84.44; H, 15.11.

4.3.6. 2-(2-Deuterioethane-1-yl)-3-deuterio-3-benzylprop-1-ene (8c). ^1H NMR ($CDCl_3$): δ 0.91 (t, 2H, *J*=6.0 Hz, CH_2D), 2.20 (m, 3H, CHD–$C{=}CH_2$–CH_2), 3.41 (d, 2H, Ph–CH_2), 4.92 (s, 2H, $H_2C{=}$), 7.20 (m, 5H, Ph); ^{13}C NMR ($CDCl_3$): δ 11.58 (t, J_{CD}=19.5 Hz), 33.43, 34.41, 38.76 (t, J_{CD}=19.0 Hz), 108.21, 125.90, 128.45, 128.97, 141.37, 148.52; MS, *m/z*: 162 (M^+). Anal. calcd for $C_{12}H_{14}D_2$: C, 88.83; H, 8.69; D, 2.46. Found: C, 88.59; H, 10.94.

4.3.7. 2-Deuterio-3-(2-deuterioethane-1-yl)oct-1-ene (9a). ^1H NMR ($CDCl_3$): δ 0.91 (t, 5H, *J*=6.0 Hz, CH_2D and CH_3), 1.32 (m, 10H, CH_2), 1.95 (m, H, CH–$C{=}C$), 4.69 (s, 2H, $H_2C{=}CD$); ^{13}C NMR ($CDCl_3$): δ 13.89 (t, J_{CD}=20.5 Hz), 14.22, 22.80, 27.29, 27.74, 29.56, 34.77, 45.82, 113.96; MS (*m/z*, %): 142 (4, M^+), 113 (2), 112 (15), 85 (7), 71 (100), 57 (38), 43 (64), 29 (38). Anal. calcd for $C_{10}H_{18}D_2$: C, 84.43; H, 12.76; D, 2.81. Found: C, 84.29; H, 15.42.

4.3.8. 2-Deuterio-3-(2-deuterioethane-1-yl)dec-1-ene (9b). ^1H NMR ($CDCl_3$): δ 0.91 (t, 5H, *J*=6.0 Hz, CH_2D and CH_3), 1.32 (m, 14H, CH_2), 1.95 (m, H, CH–$C{=}C$), 4.82 (s, 2H, $H_2C{=}CD$); ^{13}C NMR ($CDCl_3$): δ 13.78 (t, J_{CD}=20.5 Hz), 14.25, 22.84, 27.74, 27.94, 29.52, 29.80, 32.10, 34.90, 45.96, 115.04; MS, *m/z*: 170 (M^+). Anal. calcd for $C_{12}H_{22}D_2$: C, 84.63; H, 13.02; D, 2.35. Found: C, 84.41; H, 15.13.

4.3.9. 2,5-Dideuterio-3-benzylpent-1-ene (9c). ^1H NMR ($CDCl_3$): δ 0.91 (t, 2H, *J*=6.0 Hz, CH_2D), 1.33 (m, 2H, CH_2), 2.20 (m, H, CH–$CD{=}CH_2$), 3.44 (d, 2H, Ph–CH_2), 4.85 (s, 2H, $CH_2{=}CD$), 7.10 (m, 5H, Ph); ^{13}C NMR ($CDCl_3$): δ 12.33 (t, J_{CD}=19.5 Hz), 28.91, 41.48, 41.52, 114.57, 125.90, 128.45, 128.97, 141.37; MS, *m/z*: 162 (M^+). Anal. calcd for $C_{12}H_{14}D_2$: C, 88.83; H, 8.69; D, 2.46. Found: C, 88.65; H, 11.01.

4.4. Reaction of 1,2-dienes with EtMgBr catalyzed by Cp_2TiCl_2 in the presence of activated Mg (method C)

A 50 mL glass reactor was charged with chemically activated Mg (12 mmol), EtMgBr (2 M solution in diethyl ether, 22 mmol), Cp_2TiCl_2 (0.5 mmol) and 1,2-diene (10 mmol) under a dried argon atmosphere at 0 °C. The resulting solution was allowed to warm to rt and stirred for 10 h. The reaction mixture was quenched with an 8–10% aqueous solution of HCl (or DCl, 10–12% solution in D_2O). The crude products were extracted with Et_2O or hexane and purified by distillation in vacuo.

4.4.1. 7,10-Dideuteriohexadeca-6*Z*,10*Z*-diene (15a). Bp 114–115 °C (2 Torr). ^1H NMR ($CDCl_3$): δ 0.89 (t, 6H, *J*=6.0 Hz, CH_3), 1.28 (m, 12H, CH_2), 2.06 (m, 8H, CH_2–=), 5.38 (t, 2H, *J*=5.0 Hz, $HC{=}CD$); ^{13}C NMR ($CDCl_3$): δ 14.05, 22.61, 27.23, 27.41, 29.45, 31.56, 128.58 (t, J_{CD}=23.5 Hz), 130.02; MS (*m/z*, %): 224 (4, M^+), 167 (0.6), 153 (9), 140 (9), 112 (4.6), 98 (6.6), 84 (13), 71 (7), 70 (100), 57 (14.6), 43 (19), 29 (40). Anal. calcd for $C_{16}H_{28}D_2$: C, 85.64; H, 12.58; D, 1.78. Found: C, 85.89; H, 14.21. Yield 94%.

4.4.2. 9,12-Dideuterioeicosa-8*Z*,12*Z*-diene (15b). Bp 155–156 °C (2 Torr). ^1H NMR ($CDCl_3$): δ 0.89 (t, 6H, *J*=6.0 Hz, CH_3), 1.21 (m, 20H, CH_2), 1.98 (m, 8H, CH_2–=), 5.30 (t, 2H, *J*=5.0 Hz, $HC{=}CD$); ^{13}C NMR ($CDCl_3$): δ 14.12, 22.72, 27.34, 27.35, 29.27, 29.29, 29.82, 31.97, 128.96 (t, J_{CD}=23.5 Hz), 130.40. Anal. calcd for $C_{20}H_{36}D_2$: C, 85.63; H, 12.94; D, 1.43. Found: C, 85.88; H, 14.19. Yield 92%.

4.4.3. 4,7-Dideuterio-1,10-(dicyclohex-3-ene-1-yl)deca-3*Z*,7*Z*-diene (15c). Bp 190–192 °C (1 Torr). ^1H NMR ($CDCl_3$): δ 1.10–2.45 (m, 26H, CH and CH_2), 5.30–5.91 (m, 6H, $HC{=}CD$ and $HC{=}CH$ cyclic); ^{13}C NMR ($CDCl_3$): δ 25.23, 27.38, 28.81, 31.81, 31.90, 33.04, 36.62, 126.63, 127.08, 129.17 (t, J_{CD}=22.5 Hz), 130.14. Anal. calcd for $C_{22}H_{32}D_2$: C, 87.93; H, 10.73; D, 1.33. Found: C, 88.19; H, 11.85. Yield 90%.

4.4.4. 3,6-Dideuterio-1,8-diphenylocta-2*Z*,6*Z*-diene (15d). Bp 178–179 °C (1 Torr). ^1H NMR ($CDCl_3$): δ 2.37 (M, 4H, –CH_2–=), 3.36 (d, 4H, *J*=7.0 Hz, Ph–CH_2–), 5.52 (t, 2H, *J*=5.0 Hz, $CH{=}$), 7.15 (m, 10H, Ph); ^{13}C NMR ($CDCl_3$): δ 27.18, 33.47, 125.94, 128.44, 128.64, 128.91, 129.65 (t, J_{CD}=22.5 Hz), 141.18. Anal. calcd for $C_{20}H_{20}D_2$: C, 90.86; H, 7.62; D, 1.51. Found: C, 91.13; H, 8.98. Yield 75%.

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