Expedient Synthesis of a Symmetric Cycloheptyne-$\text{Co}_2(\text{CO})_6$ Complex for Orthogonal Huisgen Cycloadditions.

Supplementary Information

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General methods

All commercial materials were used without further purification, unless indicated. $^1$H NMR and $^{13}$C NMR spectra were recorded on a BRUKER AVANCE I 300 Mhz ($^1$H: 300MHz, $^{13}$C: 75.3MHz), spectrometer. The chemical shifts for the NMR spectra are reported in ppm relative to the solvent residual peak. Coupling constants J are reported in hertz (Hz). The following abbreviations are used for the multiplicities: s, singlet; d, doublet; t, triplet; m, multiplet; dd, doublet of doublet; td, triplet of doublet. Yields refer to isolated material determined to be pure by NMR spectroscopy and thin-layer chromatography (TLC), unless specified in the text. Analytical TLCs were performed on Fluka Silica Gel 60 F254.

Procedures

2,5-Dimethylhex-3-yn-2,5-diyldiacetate (5)

\[
\begin{align*}
\text{AcO} & \quad \equiv \\
\end{align*}
\]

The product was prepared by using the modified procedure reported by Nevado et al. $^1$ To a solution of diol 4 (5 mmol, 710 mg, 1 equiv.) in dry dichloromethane under nitrogen (C = 0.1 M), were added Et$_3$N (50 mmol, 6.9 mL, 10 equiv.), DMAP (5 mmol, 610 mg, 1 equiv.) and then Ac$_2$O (25 mmol, 2.5 mL, 5 equiv.) at room temperature. The solution was stirred for 16 h at this temperature. After evaporation of volatiles under vacuum, the crude solution was directly poured into a silica column chromatography (petroleum ether/Et$_2$O 7:3) to afford the compound 5 as a colorless liquid (m = 848 mg, yield = 75%). Analytical data was conformed to the literature.

$^1$H NMR (300 MHz, CDCl$_3$): $\delta$ [ppm] = 2.00 (s, 6 H), 1.62 (s, 12 H).

(2-Methylene propane-1,3-diyl)bis(trimethylsilane) (2c)

\[
\begin{align*}
\text{TMS} & \quad \equiv \\
\end{align*}
\]

The product was prepared by using the modified procedure reported by Yus et al. $^3$ A solution of 3-chloro-2-chloromethylprop-1-ene (8 mmol, 1 mL, 1 equiv.) and TMS-Cl (16 mmol, 2 mL, 2 equiv.) in THF (5 mL) was added under nitrogen to a suspension of freshly cut lithium pieces (56 mmol, 400 mg, 7 equiv.) and naphthalene (0.8 mmol, 100 mg, 0.1 equiv.) in THF (10 mL) at -78 $^\circ$C. The mixture was allowed to reach room temperature, while continuing the stirring for 24 h until a reddish solution appeared. Then, the mixture was quenched with water (10 mL), extracted with Et$_2$O (3 x 20 mL), and the organic phase was dried over anhydrous sodium sulfate. The crude bis-trimethylsilyl derivative 2c was obtained and used without any further purification (m = 1.1 g, crude yield = 68%). Analytical data was conformed to the literature.

$^1$H NMR (300 MHz, CDCl$_3$): $\delta$ [ppm] = 4.39 (s, 2H), 1.49 (s, 4H), 0.05 (s, 18H).

Complex 1

To a solution of 5 (1.2 mmol, 270 mg, 1 equiv.) in dry dichloromethane (C = 0.1 M) under nitrogen was added Co₂(CO)₈ (1.2 mmol, 450 mg, 1 equiv.) at room temperature. The solution was stirred for 4 h at this temperature, and then the solvent was removed under vacuum. The complex was then purified by flash chromatography on silica gel (petroleum ether/Et₂O, 8:2) to give 1 as a red solid (m= 660 mg, yield = 99%). Mp (°C): 86-88. Rf: 0.62 (8:2 cyclohexane/Et₂O).¹H NMR (300 MHz, CDCl₃): δ [ppm] = 2.01 (s, 6H), 1.93 (m, 12H).¹³C NMR (75 MHz, CDCl₃): δ [ppm] = 170.0, 83.6, 28.9, 22.3. HRMS: Not detected after several trials (ESI, EI, FD). FT-IR (v/cm⁻¹, KBr): 2992, 2094, 2053, 2025, 1740, 1453, 1368, 1228, 1135, 1018, 937.

Complex 3a

To a solution of 1 (1.2 mmol, 660 mg, 1 equiv.) in dry dichloromethane (C = 0.1 M) under nitrogen was added the bis-silylenol ether 2a (1.2 mmol, 328 mg, 1 equiv.). The solution was cooled down to -40 °C and BF₃·OEt₂ (2.4 mmol, 0.72 mL, 2 equiv., C = 46.5%) was added. The solution was stirred for 1 h at -40 °C, and the solvent was removed under vacuum. The crude mixture was purified by flash chromatography on silica gel (petroleum ether/Et₂O, 9:1) to give 3a as a red solid (m= 190mg, yield = 30%). Rf: 0.46 (9:1 petroleum ether/Et₂O).¹H NMR (300 MHz, CDCl₃): δ [ppm] = 4.25 – 4.10 (m, 2H), 3.71 (s, 1H), 2.87 (d, J = 10.9 Hz, 1H), 2.63 (d, J = 10.9 Hz, 1H), 2.17 (s, 3H), 1.45 (s, 3H), 1.43 (s, 3H), 1.40 (s, 3H), 1.28 (s, 3H), 1.24 (t, J = 7.1 Hz, 3H).¹³C NMR (75 MHz, CDCl₃): δ [ppm] = 203.9, 167.2, 77.6, 77.2, 76.7, 67.4, 61.1, 59.4, 40.0, 37.8, 33.1, 31.8, 30.7, 27.5, 14.2. HRMS (ESI) C₂₀H₂₀Co₂O₉ calculated for [M+Na]: 544.9663 found 544.9678. FT-IR (v/cm⁻¹, KBr): 2968, 2933, 2091, 2049, 2023, 1751, 1706, 1463, 1323, 1164, 1119.

Complex 3b

To a solution of 1 (1.2 mmol, 660 mg, 1 equiv.) in dry dichloromethane (C = 0.1 M) under nitrogen, was added the bis-silylenol ether 4b (1.2 mmol, 293 mg, 1 equiv.). The solution was cooled down to -40 °C and BF₃·OEt₂ (2.4 mmol, 0.72 mL, 2 equiv., C = 46.5%). The solution was stirred for 1 h at -40 °C, and the solvent was removed under vacuum. The crude mixture was purified by flash chromatography on silica gel (petroleum ether/Et₂O, 9:1) to give 3b as a red solid (m= 176 mg, yield = 30%). Rf: 0.35 (9:1 petroleum ether /Et₂O).¹H NMR (300 MHz, CDCl₃): δ [ppm] = 3.89 (s, 1H), 2.92 (d, J = 10.7 Hz, 1H), 2.66 (d, J = 10.7 Hz, 1H), 2.13 (s, 3H), 1.42 (s, 3H), 1.41 (s, 3H), 1.39 (s, 3H), 1.28 (s, 3H).¹³C NMR (75 MHz, CDCl₃): δ [ppm] = 205.3, 201.0, 74.4, 59.8, 40.6, 38.1, 33.2.

32.3, 31.8, 30.6, 27.4. HRMS (ESI) C_{19}H_{18}Co_{2}O_{8} calculated for [M+Na]: 514.9557 found 514.9563.

**FT-IR (v/cm\(^{-1}\), KBr):** 2986, 2970, 2936, 2090, 2041, 2016, 1731, 1693, 1575, 1453, 1360, 1317, 1158, 1126, 1069, 995.

**Complex 3c**

To a solution of 1 (1.2 mmol, 660 mg, 1 equiv.) in dry dichloromethane (C = 0.1 M) under nitrogen was added the bis-allylsilane 2c (1.2 mmol, 240 mg, 1 equiv.). The solution was cooled down to -40 °C and BF\(_3\)·OEt\(_2\) (2.4 mmol, 0.72 mL, 2 equiv., C= 46.5%). The solution was stirred for 1 h at -40 °C, and the solvent was removed under vacuum. The crude mixture was purified by flash chromatography on silica gel (petroleum ether pure) to give 3c as a red pasty solid (m= 245 mg, yield = 45%). \(R_f\): 0.71 (petroleum ether pure). \(^{1}\)HNMR (300 MHz, CDCl\(_3\)): \(\delta\) [ppm] = 4.96 (s, 2H), 2.28 (s, 4H), 1.27 (s, \(\text{12H}\)). \(^{13}\)C NMR (75 MHz, CDCl\(_3\)): \(\delta\) [ppm] = 143.9, 118.8, 52.6, 37.9, 32.1. HRMS: Not detected after several trials (ESI, EI, FD). \(\text{FT-IR (v/cm}^{-1}\text{, KBr): 2960, 2087, 2045, 2020, 1862, 1453, 1379, 1247, 836.}

**Complex 6**

To a solution of 3c (0.5 mmol, 227 mg, 1 equiv.) in dry THF (C = 0.1 M) under nitrogen was added BH\(_3\)·THF (0.75 mmol, 0.75 mL, 1.5 equiv., C = 1 M in THF) at 0 °C. The solution was stirred at r.t. until full conversion of the starting material (16 h, TLC monitoring), then NaBO\(_2\)·4H\(_2\)O (2.5 mmol, 382 mg, 5 equiv.) in a water suspension (2 mL) was added to the mixture at r.t. The solution was vigorously stirred for 4 h at r.t. (biphasic mixture). Then, the mixture was quenched with water (10 mL), extracted with EtOAc (3 x 10 mL), and the combined organic phases were dried over anhydrous sodium sulfate and evaporated under reduced pressure. The residue was purified by flash chromatography on silica gel (petroleum ether/Et\(_2\)O, 8:2) to give 6 as a red pasty solid (m = 142 mg, yield = 60%). \(R_f\): 0.25 (7:3 cyclohexane/AcOEt). \(^{1}\)HNMR (300 MHz, CDCl\(_3\)): \(\delta\) [ppm] = 3.45 (t, \(J = 6.1\) Hz, 2H), 2.13-1.96 (m, 1H), 1.79 (d, \(J = 13.5\) Hz, 2H), 1.34 (d, \(J = 13.3\) Hz, 2H), 1.29 (s, \(\text{12H}\)). \(^{13}\)C NMR (75 MHz, CDCl\(_3\)): \(\delta\) [ppm] = 69.1, 46.9, 37.9, 35.6, 33.8, 31.9. HRMS: Not detected after several trials (ESI, EI, FD). \(\text{FT-IR (v/cm}^{-1}\text{, KBr): 3371, 2994, 2987, 2086, 2043, 2019, 1591, 1469, 1379, 1362, 1248, 858, 837.}

**Complex 7**
To a solution of 6 (0.3 mmol, 141 mg, 1 equiv.) in dry dichloromethane (C = 0.1 M) under nitrogen, was added 5-hexynoic acyl chloride (0.45 mmol, 59 mg, 1.5 equiv.) and Et$_3$N (0.3 mmol, 41 μL, 1 equiv.) at room temperature. The solution was stirred at r.t. until full conversion of the starting material (1 h, TLC monitoring). After evaporation of volatiles under vacuum, the residue was directly purified by silica column chromatography (petroleum ether/Et$_2$O 8:2) to afford compound 7 as a red pasty solid (m = 86 mg, yield = 51%).

$^1$H NMR (300 MHz, CDCl$_3$): $\delta$ [ppm] = 3.94 (d, $J$ = 6.4 Hz, 2H), 2.50 (t, $J$ = 7.4 Hz, 2H), 2.30 (td, $J$ = 6.9, 2.6 Hz, 2H), 2.00 (t, $J$ = 2.6 Hz, 1H), 1.94 – 1.83 (m, 2H), 1.74 (d, $J$ = 13.0 Hz, 2H), 1.47 – 1.39 (m, 2H), 1.32 (s, 12H).

$^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ [ppm] = 173.0, 109.9, 70.1, 69.2, 46.8, 37.7, 33.6, 32.9, 32.2, 31.7, 23.6, 17.9. HRMS: Not detected after several trials (ESI, EI, FD). FT-IR ($\nu$/cm$^{-1}$, KBr): 3313, 2966, 2937, 2086, 2044, 2019, 1737, 1591, 1469, 1451, 1381, 1363, 1157, 858.

Click-product 8a

![Click-product 8a](image)

To a solution of complex 7 (0.107 mmol, 60 mg, 1 equiv.) and BnN$_3$ (0.22 mmol, 30 mg, 2 equiv.) in CH$_3$CN (C = 0.05 M) under nitrogen was added TMANO (0.55 mmol, 41 mg, 5 equiv.) at 0°C. The solution was stirred at r.t. for 16 h, the mixture becoming black. Then, the mixture was quenched with water (10 mL), extracted with EtOAc (3 x 10 mL), and the combined organic phases were dried over anhydrous sodium sulfate and evaporated under reduced pressure. The crude was purified by flash chromatography on silica gel (petroleum ether/Et$_2$O, 8:2) to give 8a as a colorless oil (m = 18 mg, yield = 41%).

$^1$H NMR (300 MHz, CDCl$_3$): $\delta$ [ppm] = 7.35 - 7.28 (m, 3H), 7.03 - 6.98 (m, 2H), 5.72 (s, 2H), 3.98 - 3.87 (m, 2H), 2.48 (t, $J$ = 7.4 Hz, 1H), 2.27 (td, $J$ = 6.9, 2.7 Hz, 2H), 2.23 - 2.16 (m, 1H), 1.97 (t, $J$ = 2.6 Hz, 1H), 1.91 - 1.79 (m, 2H), 1.74 - 1.63 (m, 2H), 1.61 (s, 3H), 1.58 - 1.45 (m, 2H), 1.34 (s, 3H), 1.28 (s, 3H), 1.03 (s, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ [ppm] = 173.2, 150.9, 138.3, 136.9, 128.9, 127.9, 126.5, 83.3, 69.9, 69.4, 54.5, 49.4, 45.9, 35.0, 33.8, 33.0, 31.9, 31.6, 31.2, 31.1, 27.7, 23.8, 18.0. HRMS (ESI) C$_{25}$H$_{33}$N$_3$O$_2$ calculated for [M+Na]: 430.2464 found 430.2470.

Click-product 8b

![Click-product 8b](image)

To a solution of complex 7 (0.107 mmol, 60 mg, 1 equiv.) and pMeOC$_6$H$_4$CH$_2$N$_3$ (0.22 mmol, 36 mg, 2 equiv.) in CH$_3$CN (C = 0.05 M) under nitrogen was added TMANO (0.55 mmol, 41 mg, 5 equiv.) at 0°C. The solution was stirred at r.t. for 16 h, the mixture becoming black. Then, the mixture was quenched with water (10 mL), extracted with EtOAc (3 x 10 mL), and the combined organic phases
were dried over anhydrous sodium sulfate and evaporated under reduced pressure. The crude was purified by flash chromatography on silica gel (petroleum ether/Et₂O, 8:2) to give 8b as a colorless oil (m= 21 mg, yield = 45%). \(R_f\): 0.29 (8:2 petroleum ether/AcOEt). ¹H NMR (300 MHz, CDCl₃): \(\delta [ppm] = 6.96 (d, J = 8.8 Hz, 2H), 6.84 (d, J = 8.8 Hz, 2H), 5.74-5.58 (m, 2H), 3.92 (dd, J = 6.7, 3.6 Hz, 2H), 3.79 (s, 3H), 2.48 (t, J = 7.4 Hz, 2H), 2.27 (td, J = 6.9, 2.7 Hz, 2H), 2.24-2.17 (m, 1H), 1.97 (t, J = 2.7 Hz, 1H), 1.88-1.81 (m, 2H), 1.73-1.61 (m, 2H), 1.60 (s, 3H), 1.54-1.45 (m, 2H), 1.36 (s, 3H), 1.27 (s, 3H), 1.02 (s, 3H). ¹³C NMR (75 MHz, CDCl₃): \(\delta [ppm] = 173.2, 159.3, 150.9, 138.2, 128.8, 127.9, 83.3, 69.9, 69.4, 55.4, 55.3, 49.4, 45.9, 34.9, 33.8, 33.0, 31.9, 31.6, 31.2, 27.7, 23.8, 18.0, 1.2. HRMS (ESI) C₂₆H₃₅N₃O₃ calculated for [M+Na]: 460.2570 found 460.2574.

**Click-product 9a**

![Click-product 9a](image)

To a solution of 8a (0.022 mmol, 9 mg, 1 equiv.) and pMeOC₆H₄CH₂N₃ (0.026 mmol, 4.3 mg, 1.2 equiv.) in tBuOH/H₂O (3:1, C = 0.05 M) under nitrogen was added CuSO₄ (2.6 µmol, 0.4 mg, 0.1 equiv.) and sodium ascorbate (0.026 mmol, 5.2 mg, 1 equiv.). The solution was stirred at r.t. for 16 h. Then, the mixture was quenched with water (10 mL), extracted with EtOAc (3 x 10 mL), and the combined organic phases were dried over anhydrous sodium sulfate and evaporated under reduced pressure. The crude was purified by flash chromatography on silica gel (dichloromethane/MeOH, 95:5) to give 9a as a colorless oil (m = 8 mg, yield = 64%). \(R_f\): 0.59 (95:5 dichloromethane/MeOH). ¹H NMR (300 MHz, CDCl₃): \(\delta [ppm] = 7.36-7.28 (m, 3H), 7.23-7.13 (m, 3H), 7.03-6.96 (m, 2H), 6.91-6.85 (m, 2H), 5.71 (s, 2H), 5.41 (s, 2H), 3.92-3.87 (m, 2H), 3.80 (s, 3H), 2.72 (t, J = 7.5 Hz, 2H), 2.38 (t, J = 7.5 Hz, 2H), 2.26-2.16 (m, 1H), 2.04-1.91 (m, 2H), 1.67 (d, J = 13.6 Hz, 2H), 1.60 (s, 3H), 1.56-1.45 (m, 2H), 1.32 (s, 3H), 1.26 (s, 3H), 1.01 (s, 3H). ¹³C NMR (75 MHz, CDCl₃): \(\delta [ppm] = 173.4, 160.0, 150.9, 147.6, 138.4, 136.9, 129.7, 128.9, 126.9, 126.5, 120.6, 114.6, 69.8, 55.5, 54.5, 53.8, 49.3, 45.9, 34.9, 33.8, 33.7, 31.9, 31.6, 31.2, 27.7, 25.2, 24.8. HRMS (ESI) C₃₃H₄₂N₆O₃Na calculated for [M+Na]: 593.3210 found 593.3201.

**Click-product 9b**

![Click-product 9b](image)

Via sequence B

To a solution of 8b (0.085 mmol, 37 mg, 1 equiv.) and BnN₃ (0.11 mmol, 15 mg, 1.3 equiv.) in tBuOH/H₂O (3:1, C = 0.05 M) under nitrogen was added CuSO₄ (0.01 mmol, 1.5 mg, 0.1 equiv.) and sodium ascorbate (0.085 mmol, 17 mg, 1 equiv.). The solution was stirred at r.t. for 16 h. Then, the mixture was quenched with water (10 mL), extracted with EtOAc (3 x 10 mL), and the combined organic phases were dried over anhydrous sodium sulfate and evaporated under reduced pressure.
crude was purified by flash chromatography on silica gel (dichloromethane/MeOH, 95:5) to give 9b as a colorless oil (m = 20 mg, yield = 41%).

Via sequence C
To a solution of complex 10 (0.028 mmol, 20 mg, 1 equiv.) and pMeOC₆H₄CH₂N₃ (0.56 mmol, 10 mg, 2 equiv.) in CH₃CN (C = 0.05 M) under nitrogen was added TMANO (0.28 mmol, 21 mg, 10 equiv.) at 0°C. The solution was stirred at r.t. for 16 h, the mixture becoming black. Then, the mixture was quenched with water (10 mL), extracted with EtOAc (3 x 10 mL), and the combined organic phases were dried over anhydrous sodium sulfate and evaporated under reduced pressure. The crude was purified by flash chromatography on silica gel (dichloromethane/MeOH, 95:5) to give 9b as a colorless oil (m = 10 mg, yield = 63%).

\[ R_f: 0.61 \text{(95:5 dichloromethane /MeOH)} \]

\[ ^1H \text{ NMR (300 MHz, CDCl}_3\): } \delta \text{ [ppm] } = 7.39-7.33 (m, 3H), 7.25-7.21 (m, 2H), 7.21 (s, 1H), 6.96 (d, \text{ J }= 8.8 \text{ Hz, 2H}), 6.84 (d, \text{ J }= 8.8 \text{ Hz, 2H}), 5.73-5.56 (m, 2H), 5.49 (s, 2H), 3.96-3.85 (m, 2H), 3.78 (s, 3H), 2.74 (t, \text{ J }= 7.6 \text{ Hz, 2H}), 2.39 (t, \text{ J }= 7.5 \text{ Hz, 2H}), 2.25-2.15 (m, 1H), 2.02-1.94 (m, 2H), 1.67 (d, \text{ J }= 13.5 \text{ Hz, 2H}), 1.59 (s, 3H), 1.49 (d, \text{ J }= 13.6 \text{ Hz, 2H}), 1.35 (s, 3H), 1.25 (s, 3H), 1.00 (s, 3H).

\[ ^{13}C \text{ NMR (75 MHz, CDCl}_3\): } \delta \text{ [ppm] } = 173.3, 159.2, 150.6, 143.4, 141.4, 138.1, 134.8, 129.1, 128.7, 128.0, 127.7, 120.7, 114.2, 69.7, 55.3, 54.1, 53.9, 49.2, 45.7, 34.9, 33.7, 33.6, 31.8, 31.5, 31.1, 31.0, 27.6, 25.1, 24.7. \text{ HRMS (ESI) } C_{33}H_{42}N_6O_3 \text{ calculated for [M+Na]: 593.3210 found 593.3214.}

Complex 10

To a solution of complex 7 (0.089 mmol, 50 mg, 1 equiv.) and BnN₃ (0.11 mmol, 15 mg, 1.3 equiv.) in tBuOH/H₂O (3:1, C = 0.05 M) under nitrogen was added the CuSO₄ (0.009 mmol, 1.5 mg, 0.1 equiv.) and sodium ascorbate (0.089 mmol, 17 mg, 1 equiv.). The solution was stirred at r.t. for 16 h. Then, the mixture was quenched with water (10 mL), extracted with EtOAc (3 x 10 mL), and the combined organic phases were dried over anhydrous sodium sulfate and evaporated under reduced pressure. The crude was purified by flash chromatography on silica gel (petroleum ether/Et₂O, 8:2) to give 10 as a red pasty solid (m= 32 mg, yield = 54%).

\[ R_f: 0.5 \text{(6:4 petroleum ether/EtOAc)} \]

\[ ^1H \text{ NMR (300 MHz, CDCl}_3\): } \delta \text{ [ppm] } = 7.42-7.34 (m, 3H), 7.31-7.23 (m, 2H), 7.21 (s, 1H), 5.49 (s, 2H), 3.88 (d, \text{ J }= 6.4 \text{ Hz, 2H}), 2.75 (t, \text{ J }= 7.6 \text{ Hz, 2H}), 2.39 (t, \text{ J }= 7.4 \text{ Hz, 2H}), 2.25-2.11 (m, 1H), 2.07-1.92 (m, 2H), 1.70 (d, \text{ J }= 13.7 \text{ Hz, 2H}), 1.39 (d, \text{ J }= 13.6 \text{ Hz, 2H}), 1.27 (s, 12H).

\[ ^{13}C \text{ NMR (75 MHz, CDCl}_3\): } \delta \text{ [ppm] } = 173.4, 147.8, 134.9, 129.2, 128.8, 128.2, 120.8, 110.0, 70.2, 54.2, 46.9, 37.8, 33.7, 33.6, 32.3, 31.9, 25.2, 24.7. \text{ HRMS (ESI) } C_{33}H_{30}Co₃N₂O₈ \text{ calculated for [M+]: 693.0932 found 693.0935. } \text{ FT-IR (v/cm}\^3\text{, KBr): } 2965, 2935, 2086, 2043, 2018, 1734, 1591, 1455, 1380, 1362, 1176, 1050, 858.
$^1$H, $^{13}$C NMR and IR spectra

$^1$H, 300 MHz, CDCl$_3$

$^{13}$C, 75 MHz, CDCl$_3$
$^1$H, 300 MHz, CDCl$_3$

$^{13}$C, 75 MHz, CDCl$_3$
$^1$H, 300 MHz, CDCl$_3$

$^{13}$C, 75 MHz, CDCl$_3$
$^1H$, 300 MHz, CDCl$_3$

$^{13}C$, 75 MHz, CDCl$_3$
$^1$H, 300 MHz, CDCl$_3$

$m/z 358 p$
1H NMR CDCl$_3$ 2(0.1), CSH EF 17

$^1$H, 300 MHz, CDCl$_3$

$^{13}$C, 75 MHz, CDCl$_3$

$m/z 358 p 3x$
13C NMR CDCl$_3$ 2(0.1), CSH EF 8

$^{13}$C, 75 MHz, CDCl$_3$
$^1$H, 300 MHz, CDCl$_3$

$^{13}$C, 75 MHz, CDCl$_3$
$^{1}H$, 300 MHz, CDCl$_3$

$^{13}C$, 75 MHz, CDCl$_3$
$^1$H, 300 MHz, CDCl$_3$
$^1$H, 300 MHz, CDCl$_3$

$^{13}$C, 75 MHz, CDCl$_3$
IR (KBr)