Supporting Information

for

A carbohydrate approach for the formal total synthesis of (−)-aspergillide C

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Experimental details and analytical data

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Experimental

General Methods. $^1$H NMR and $^{13}$C NMR spectra were recorded in CDCl$_3$ as solvent on 300 MHz or 500 MHz spectrometer at ambient temperature. The chemical shifts are reported in ppm downfield from TMS as internal standard and signal patterns are indicated as follows: s = singlet, d = doublet, t = triplet, q = quartet, sext = sextet, m = multiplet, br = broad. The coupling constants $J$ are given in Hz. FTIR spectra were recorded on KBr pellets and reported in wave number (cm$^{-1}$). Optical rotations were measured on digital polarimeter using a 1 mL cell with 1 dm path length. For low (MS) and High (HRMS) resolution, $m/z$ ratios are reported as values in atomic mass units. Mass analysis was done in ESI mode. All reagents were reagent grade and used without further purification unless specified otherwise. Solvents for reactions were distilled prior to use: THF, and diethyl ether were distilled from Na and benzophenone ketyl; CH$_2$Cl$_2$ from CaH$_2$. All air- or moisture-sensitive reactions were conducted under a nitrogen or argon atmosphere in flame-dried or oven-dried glassware with magnetic stirring. Column chromatography was carried out using silica gel (100–200 mesh) packed in glass columns. Technical grade ethyl acetate and petroleum ether used for column chromatography were distilled prior to use.

(R)-7-(Trimethylsilyl)hept-6-yn-2-yl benzoate (7). To a magnetically stirred solution of mono-silylated alcohol 10 (6.7 g, 36.4 mmol) in CH$_2$Cl$_2$ (60 mL), pyridine (5.9 mL, 72.8 mmol) was added at 0 °C followed by C$_6$H$_5$COCl (5.5 mL, 54.6 mmol) at the same temperature. The alcohol 10 was consumed within 2 h, and the reaction was then quenched with 1 N HCl (110 mL, 110
mmol) at 0 °C and extracted with CH₂Cl₂ (3 x 20 mL). The organic layer was washed with water (30 mL) and sat. aq. NaHCO₃ (20 mL), and dried with anhydrous Na₂SO₄. Evaporation of CH₂Cl₂ \textit{in vacuo} gave the crude product, which was purified by column chromatography (hexane/EtOAc 9.7:0.3) to afford pure compound 7 as a pale yellow oil (10.3 g, 98%); \( R_f = 0.5 \) (hexane/EtOAc 9.5:0.5); \(^1\)H NMR (300 MHz, CDCl₃): \( \delta 8.04 \) (d, \( J = 6.8 \) Hz, 2H), 7.60-7.52 (m, 1H), 7.48-7.40 (m, 2H), 5.31-5.12 (m, 1H), 2.27 (t, \( J = 6.8 \) Hz, 2H), 2.00-1.60 (m, 4H), 1.36 (dd, \( J = 6.8, 2.3 \) Hz, 3H), 0.15 (s, 9H). ppm; \(^{13}\)C NMR (75 MHz, CDCl₃): \( \delta 166.1, 132.7, 130.7, 129.5, 128.3, 106.8, 84.9, 71.1, 35.1, 24.5, 20.1, 19.7, 0.1 \) ppm.

**\((R)-7-((2S,5R,6R)-5-Acetoxy-6-(acetoxy)methyl)-5,6-dihydro-2\text{-}H\text{-}pyran-2-yl)hept-6-yn-2-yl benzoate (11).** To a magnetically stirred solution of tri-O-acetyl-D-galactal (2.24 g, 8.24 mmol) and 7 (2.49 g, 8.65 mmol) in CH₂Cl₂ (40 mL), SnCl₄ (1 M in heptane, 4.2 mL, 0.52 mmol) was added at 0 °C. The ice bath was then removed and stirring was continued at room temp. Within 1 h, all the starting materials were consumed (reaction monitored by TLC) and the reaction was quenched with sat. aq. NaHCO₃ (20 mL) at 0 °C, diluted with water (20 mL), stirred at room temp. for 1 h, then extracted with CH₂Cl₂ (3 x 10 mL). The organic layer was washed with brine (20 mL), and dried with anhydrous Na₂SO₄. Evaporation of CH₂Cl₂ \textit{in vacuo} gave the crude product, which was subjected to purification on silica gel column chromatography (hexane/EtOAc, 9:1) to give the desired product 11 as light-yellow oil (3.57 g, 85%). \( R_t = 0.5 \) (hexane/EtOAc, 7:3); \([\alpha]_D^{24} = -47 \) (c 0.3, CHCl₃); IR (KBr): 2939, 2216, 1744, 1715, 1451, 1373, 1276, 1230, 715 cm\(^{-1}\); \(^1\)H NMR (300
MHz, CDCl$_3$): $\delta$ 8.08-8.00 (m, 2H), 7.60-7.52 (m, 1H), 7.48-7.39 (m, 2H), 6.04 (dd, $J = 10.0, 6.4$ Hz, 1H), 5.96 (ddd, $J = 10.0, 5.1, 1.5$ Hz, 1H), 5.25-5.13 (m, 1H), 5.05 (dd, $J = 5.1, 2.3$ Hz, 1H), 5.04-4.99 (m, 1H), 4.33 (ddd, $J = 7.4, 5.3, 2.5$ Hz, 1H), 4.29-4.08 (m, 2H), 2.29 (td, $J = 8.9, 7.0, 1.9$ Hz, 2H), 2.08 (s, 3H), 2.06 (s, 3H), 1.90-1.73 (m, 2H), 1.73-1.55 (m, 2H), 1.36 (d, $J = 6.2$ Hz, 3H) ppm; $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 170.3, 170.4, 160.1, 132.8, 132.6, 130.6, 129.4, 128.3, 121.8, 87.2, 75.8, 70.9, 69.3, 64.0, 63.3, 62.8, 35.0, 24.3, 20.8, 20.7, 20.0, 18.6 ppm; HRMS calcd for C$_{24}$H$_{28}$O$_7$Na [M + Na]$^+$ 451.1727, found 451.1724.

(R,E)-7-((2R,5R,6R)-5-Acetoxy-6-(acetoxymethyl)-5,6-dihydro-2H-pyran-2-yl)hept-6-en-6-triethoxysilyl-2-yl benzoate (12). To a vigorously stirring solution of alkyne 11 (3.0 g, 5.88 mmol) and (EtO)$_3$SiH (4.42 mL, 23.53 mmol) in CH$_2$Cl$_2$ (60 mL), [Cp*(MeCN)$_3$Ru]PF$_6$ (30 mg, 0.059 mmol) was added at 0 °C under an argon atmosphere, and the ice bath was then removed. Within 1 h, alkyne 11 was completely consumed (reaction monitored by TLC). Evaporation of CH$_2$Cl$_2$ in vacuo gave the crude vinyl siloxane 12, which was subjected to silica gel column chromatography (hexane/EtOAc, 9:1) to give the desired product 12 (3.95 g, 95%) as light yellow oil. $R_f$ = 0.45 (hexane/EtOAc, 8:2); $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 8.04-8.00 (m, 2H), 7.55-7.51 (m, 1H), 7.44-7.39 (m, 2H), 6.19-6.16 (m, 1H), 5.98 (dd, $J = 10.2, 3.2$ Hz, 1H), 5.92 (ddd, $J = 10.2, 5.0, 2.0$ Hz, 1H), 5.25-5.21 (m, 1H), 5.19-5.13 (m, 1H), 5.06 (dd, $J = 4.9, 2.6$ Hz, 1H), 4.20 (dd, $J = 5.0, 2.8$ Hz, 1H), 4.19-4.16 (m, 2H), 3.79 (dq, $J = 14.0, 7.0, 0.9$ Hz, 6H), 2.19-2.13 (m, 2H), 2.07 (s, 3H), 2.02 (s, 3H), 1.79-1.69 (m, 2H), 1.65-1.45 (m, 2H), 1.33 (d, $J = 6.3$ Hz, 3H) ppm; $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 170.3, 170.4, 160.1, 132.8, 132.6, 130.6, 129.4, 128.3, 121.8, 87.2, 75.8, 70.9, 69.3, 64.0, 63.3, 62.8, 35.0, 24.3, 20.8, 20.7, 20.0, 18.6 ppm; HRMS calcd for C$_{24}$H$_{28}$O$_7$Na [M + Na]$^+$ 451.1727, found 451.1724.
1.19 (t, J = 7.0 Hz, 9H) ppm; $^{13}$CNMR (75 MHz, CDCl$_3$): δ 170.7, 170.6, 166.1, 140.8, 136.4, 134.9, 132.7, 130.8, 129.4, 128.2, 121.1, 72.1, 71.4, 68.1, 63.5, 62.8, 58.4, 37.2, 35.7, 29.6, 20.9, 20.7, 20.0, 18.1 ppm.

(R,E)-7-((2R,5R,6R)-5-Acetoxy-6-(acetoxymethyl)-5,6-dihydro-2H-pyran-2-yl)hept-6-en-2-yl benzoate (5). To a vigorously stirring solution of vinyl siloxane 12 (3.0 g, 5.06 mmol) in THF (40 mL), pyridine (4.8 mL, 58.8 mmol) followed by HF/pyridine (70:30 solution, 0.82 mL, 29.4 mmol) were added dropwise at 0 °C. After all vinyl siloxane was consumed (~5 min; reaction monitored by TLC), the reaction was quenched with water (20 mL), neutralized with 0.5 N HCl (35 mL), and extracted with EtOAc (3 x 30 mL). The organic layers were washed with water (60 mL) and brine (40 mL), and dried with anhydrous Na$_2$SO$_4$. Evaporation of solvent in vacuo gave the crude product, which was subjected to silica gel column chromatography (hexane/EtOAc, 8.5:1.5) to give the desired product 5 as a light-yellow oil (1.96 g, 90%). $R_f$ = 0.5 (hexane/EtOAc, 3:2); [$\alpha$]$_D^{24}$ = -14 (c 0.3, CHCl$_3$); IR (KBr): 2937, 1745, 1715, 1624, 1451, 1371, 1276, 1229, 1113, 716 cm$^{-1}$; $^1$H NMR (300 MHz, CDCl$_3$): δ 8.07-8.00 (m, 2H), 7.60-7.52 (m, 1H), 7.48-7.39 (m, 2H), 6.11-5.97 (m, 2H), 5.75-5.60 (m, 1H), 5.51 (dd, $J$ = 15.8, 5.1 Hz, 1H), 5.24-5.11 (m, 1H), 5.03 (dd, $J$ = 4.7, 2.5 Hz, 1H), 4.81-4.74 (m, 1H), 4.27-4.10 (m, 2H), 4.06 (ddd, $J$ = 8.7, 6.4, 2.5 Hz, 1H), 2.20-2.10 (m, 2H), 2.09 (s, 3H), 2.03 (s, 3H), 1.83-1.40 (m, 4H), 1.35 (d, $J$ = 6.4 Hz, 3H) ppm; $^{13}$CNMR (75 MHz, CDCl$_3$): δ 170.6, 170.5, 166.1, 134.6, 133.9, 132.7, 130.7, 129.4, 128.2, 126.3, 122.3, 72.6, 71.3, 67.9, 63.8, 63.1, 35.4, 32.1, 24.8, 20.9, 20.7, 20.0 ppm; HRMS calcd for C$_{24}$H$_{30}$O$_7$Na [M + Na]$^+$ 453.1884, found 453.1883.
(R,E)-7-((2R,5R,6R)-5-Hydroxy-6-(hydroxymethyl)-5,6-dihydro-2H-pyran-2-yl)hept-6-en-2-yl benzoate (13). To the magnetically stirring solution of 5 (1.1 g, 2.15 mmol) in dry methanol (20 mL), acetyl chloride (0.03 mL, 0.43 mmol) was added at 0 °C and stirring was continued while allowing the reaction mixture to attain room temperature. After complete consumption of starting material (ca. 4 h, as monitored by TLC) solvent was removed under reduced pressure to give the crude mass which was subjected to column chromatography (EtOAc/Hexane 3:7) to give the diol 13 as colorless oil. \( R_f = 0.5 \) (hexane/EtOAc, 3:7); \( [\alpha]_D^{24} = -31 \) (c 0.25, CHCl\(_3\)); IR (KBr): 3407, 2934, 1713, 1452, 1380, 1229, 1112, 714 cm\(^{-1}\); \(^1\)H NMR (500 MHz, CDCl\(_3\)): \( \delta \) 8.08-7.99 (m, 2H), 7.60-7.51 (m, 1H), 7.49-7.39 (m, 2H), 6.05 (ddd, \( J = 10.0, 5.3, 1.7 \) Hz, 1H), 5.94 (dd, \( J = 10.2, 3.4 \) Hz, 1H), 5.72-5.48 (m, 2H), 5.23-5.09 (m, 1H), 4.75-4.68 (m, 1H), 3.94-3.72 (m, 4H), 2.66-2.41 (bs, 1H), 2.40-2.20 (bs, 1H), 2.20-2.02 (m, 2H), 1.83-1.40 (m, 4H), 1.34 (d, \( J = 6.2 \) Hz, 3H), ppm; \(^{13}\)C NMR (75 MHz, CDCl\(_3\)): \( \delta \) 166.2, 134.6, 132.8, 132.0, 130.7, 129.5, 128.3, 126.9, 126.6, 73.0, 71.6, 71.4, 63.0, 62.9, 35.4, 32.1, 24.8, 20.0 ppm; HRMS calcd for C\(_{20}\)H\(_{26}\)O\(_5\)Na [M + Na]\(^+\) 369.1673, found 369.1672.

(R,E)-7-((2R,5R,6R)-5-((tert-Butyldimethylsilyl)oxy)-6-(hydroxymethyl)-5,6-dihydro-2H-pyran-2-yl)hept-6-en-2-yl benzoate (14). To a magnetically stirring solution of compound 13 (1 g, 2.34 mmol) in CH\(_2\)Cl\(_2\) (15 mL), imidazole (0.95 g, 1.4 mmol), TBSCl (1.06 g, 7 mmol), and DMAP (0.15 g, 1.2 mmol) were added at 0 °C under nitrogen atmosphere and stirring was continued until complete consumption of starting material was observed (ca. 12 h). The reaction mixture was quenched with sat. aq. NH\(_4\)Cl (10 mL), diluted
with water (10 mL), and extracted with CH₂Cl₂ (3 x 10 mL). The combined
organic layer was washed with brine (10 mL) and dried over anhydrous
Na₂SO₄. Evaporation of CH₂Cl₂ in vacuo gave the crude product, which was
subjected to silica gel column chromatography to give the desired product 14a
as a colorless liquid.

To the magnetically stirring solution of 14a (7.0 g, 16.8 mmol) in THF (90 mL)
in a teflon vial, HF/pyridine (7:3 complex, 4.4 mL, 0.15 mmol) was added at 0
°C and stirring continued at the same temperature while monitoring the
reaction progress by TLC analysis. After complete consumption of starting
material (ca. 12 h) the reaction mixture was neutralized with solid NaHCO₃
and the filtrate was concentrated to give the crude mass, which upon column
purification (EtOAc/Hexane 1.5:8.5) gave the mono TBS ether 14 as colorless
oil (4.6 g, 90%).

14a: (1.30 g, 98%); Rₜ = 0.5 (hexane/EtOAc, 9.5:0.5); [α]D24 = -8 (c 0.25,
CHCl₃); IR (KBr): 2954, 2930, 2857, 1718, 1636, 1466, 1360, 1275, 1254,
1112, 836, 775 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): δ 8.10-8.04 (m, 2H), 7.62-
7.54 (m, 1H), 7.51-7.42 (m, 2H), 5.96-5.82 (m, 2H), 5.76-5.62 (m, 1H), 5.61
(dd, J = 15.7, 5.3 Hz, 1H), 5.26-5.12 (m, 1H), 4.74-4.68 (m, 1H), 4.05-3.99 (m,
1H), 3.89-3.68 (m, 2H), 3.29-3.23 (m, 1H), 2.21-2.06 (m, 2H), 1.85-1.41 (m,
4H), 1.36 (d, J = 6.2 Hz, 3H), 0.92 (s, 9H), 0.91 (s, 9H), 0.11 (s, 6H), 0.08 (s,
6H) ppm; ¹³CNMR (75 MHz, CDCl₃): δ 166.1, 133.0, 132.7, 131.0, 130.8,
129.5, 128.2, 127.6, 126.9, 73.6, 72.2, 71.4, 63.0, 62.5, 35.6, 32.2, 25.9, 25.7,
24.9, 20.1, 18.3, 18.2, -4.1, -4.6, -5.2, -5.3 ppm; HRMS calcd for
C₉₂H₅₄O₅NaSi₂ [M + Na]+ 597.3402, found 597.3396.
14: $R_i = 0.5$ (hexane/EtOAc, 7:3); $[\alpha]_D^{25} = -9$ (c 0.25, CHCl$_3$); IR (KBr): 3496, 2932, 2858, 1715, 1625, 1454, 1356, 1277, 1113, 838, 777 cm$^{-1}$; $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 8.05-8.00 (m, 2H), 7.58-7.50 (m, 1H), 7.47-7.38 (m, 2H), 5.89-5.78 (m, 2H), 5.69-5.56 (m, 1H), 5.48 (dd, $J = 15.9, 5.3$ Hz, 1H), 5.21-5.09 (m, 1H), 4.71-4.64 (m, 1H), 4.09-4.01 (m, 1H), 3.91-3.77 (m, 2H), 3.72 (dd, $J = 9.8, 3.0$ Hz, 1H), 2.43-2.21 (bs, 1H), 2.14-2.01 (m, 2H), 1.81-1.38 (m, 4H), 1.32 (d, $J = 6.0$ Hz, 3H), 0.88 (s, 9H), 0.08 (s, 6H) ppm; $^{13}$CNMR (75 MHz, CDCl$_3$): $\delta$ 166.2, 134.1, 132.7, 131.0, 130.8, 129.5, 128.3, 127.7, 126.7, 72.7, 72.0, 71.4, 64.3, 62.7, 35.5, 32.1, 25.8, 24.9, 20.0, 18.1, -4.2, -4.7 ppm; HRMS calcd for C$_{26}$H$_{40}$O$_5$NaSi [M + Na]$^+$ 483.2537, found 483.2538.

$(R,E)$-7-((2$R,5R,6R$)-5-((tert-Butyldimethylysilyl)oxy)-6-(cyanomethyl)-5,6-dihydro-2H-pyran-2-yl)hept-6-en-2-yl benzoate (15). To the stirring solution of alcohol 14 (0.27 g, 0.6 mmol) in CH$_2$Cl$_2$ (5 mL), 2,6-lutidine (0.21 mL, 1.8 mmol) followed by Tf$_2$O (0.11 mL, 0.66 mmol) were added at 0 °C. Within 5 min entire starting material was consumed (reaction monitored by TLC) and the reaction mixture was quenched with sat. aq. NH$_4$Cl (5 mL) and extracted with CH$_2$Cl$_2$ (2 x 10 mL). The combined organic layers were dried over anhydrous Na$_2$SO$_4$, filtered, concentrated under reduced pressure to give the triflate. To the triflate dissolved in DMF (5 mL), 18-crown-6 (31 mg, 0.12 mmol) followed by NaCN (0.15 g, 3.0 mmol) were added and the mixture was heated to 90 °C. After the entire starting material was consumed (~30 min) the reaction mixture was diluted with ice cold water (20 mL) and extracted with Et$_2$O (3 x 10 mL), the combined extracts were washed with brine (10 mL), dried over anhydrous Na$_2$SO$_4$, filtered and concentrated under reduced
pressure to give crude product which upon column purification (Hexane/EtOAc 9/1) gave nitrile 15 (0.245 g, 88%) as colour less oil. $R_f = 0.5$ (hexane/EtOAc, 3:7); [$\alpha$]$_D^{24} = -122$ (c 0.25, CHCl$_3$); IR (KBr): 2932, 2858, 2251, 1715, 1456, 1359, 1275, 1113, 837, 775 cm$^{-1}$; $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 8.06-8.02 (m, 2H), 7.55 (tt, $J = 1$ Hz), 7.46-7.42 (m, 2H), 5.88-5.82 (m, 2H), 5.68 (ddddd, 1H), 5.48 (qt, $J = 1$ Hz), 5.20-5.13 (m, 1H), 4.69-4.64 (m, 1H), 4.10-4.00 (m, 2H), 2.66-2.62 (m, 2H), 2.15-2.06 (m, 2H), 1.80-1.70 (m, 1H), 1.69-1.59 (m, 1H), 1.58-1.40 (m, 2H), 1.35 (d, $J = 6.4$ Hz, 3H), 0.90 (s, 9H), 0.12 (s, 3H), 0.10 (s, 3H) ppm; $^{13}$CNMR (75 MHz, CDCl$_3$): $\delta$ 166.1, 134.5, 132.7, 130.7, 129.4, 128.2, 126.9, 126.1, 117.9, 72.1, 71.3, 69.2, 63.3, 35.4, 32.0, 25.7, 24.7, 20.0, 19.0, 18.1, -4.1, -4.7 ppm; HRMS calcd for C$_{27}$H$_{39}$O$_4$NNaSi [M + Na]$^+$ 492.2541, found 492.2541.

2-((2R,3R,6R)-3-((tert-Butyldimethylsilyl)oxy)-6-((R,E)-6-hydroxyhept-1-en-1-yl)-3,6-dihydro-2H-pyran-2-yl)acetic acid (4). Aqueous 8 N NaOH solution was added to a solution of cyanide 15 (0.1 g, 0.21 mmol) in EtOH and refluxed for 3 h. The reaction was neutralized using 3 N HCl solution after cooling to rt. The mixture was diluted with water and extracted with ethyl acetate (3 x 20 mL). The combined organic layers were washed with brine, dried over anhydrous Na$_2$SO$_4$, concentrated in vacuo, and purified by column chromatography to give 4 (0.06 g, 75%) as a pale yellow oil. $R_f = 0.3$ (hexane/EtOAc, 1:1); [$\alpha$]$_D^{24} = -145.0$ (c 0.27, CHCl$_3$); IR (KBr): 3422, 3037, 2999, 1699, 1050, 830 cm$^{-1}$; $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 5.91-5.84 (m, 2H), 5.77-5.60 (m, 1H), 5.51 (dd, $J = 15.8$, 6.0 Hz, 2H), 4.68 (d, $J = 5.3$ Hz, 1H), 4.18 (dt, $J = 9.0$, 3.2 Hz, 1H), 3.93-3.98 (m, 1H), 3.82 (sex, $J = 6.0$ Hz, 1H).
2.71 (dd, $J = 15.9, 9.1$ Hz, 1H), 2.53 (dd, $J = 15.9, 3.8$ Hz, 1H), 1.96-2.20 (m, 2H), 1.63-1.38 (m, 2H), 1.25 (s, 1H, OH), 1.18 (d, $J = 6.0$ Hz, 3H), 0.9 (s, 9H), 0.10-0.08 (m, 6H). ppm; $^{13}$CNMR (75 MHz, CDCl$_3$): $\delta$ 175.0, 134.6, 130.7, 127.5, 126.7, 72.4, 69.6, 68.1, 64.4, 37.8, 35.9, 31.7, 25.9, 24.5, 22.9, 18.2, -4.1, -4.6 ppm. HRMS calcd for C$_{20}$H$_{37}$O$_5$Si [M + Na]$^+$ 385.2366, found 385.2384.
$^1$H and $^{13}$C NMR spectra of key compounds:

$^1$H NMR Spectrum of 7 (300 MHz, CDCl$_3$)

$^{13}$C NMR Spectrum of 7 (75 MHz, CDCl$_3$)
$^1$H NMR Spectrum of 11 (300 MHz, CDCl$_3$)

$^{13}$C NMR Spectrum of 11 (75 MHz, CDCl$_3$)
$^1$H NMR Spectrum of 12 (500 MHz, CDCl$_3$)

$^{13}$C NMR Spectrum of 12 (125 MHz, CDCl$_3$)
$^1$H NMR Spectrum of 5 (300 MHz, CDCl$_3$)

$^{13}$C NMR Spectrum of 5 (75 MHz, CDCl$_3$)
$^1$H NMR Spectrum of 13 (300 MHz, CDCl$_3$)

$^{13}$C NMR Spectrum of 13 (75 MHz, CDCl$_3$)
**H NMR Spectrum of 14a** (300 MHz, CDCl$_3$)

**C NMR Spectrum of 14a** (75 MHz, CDCl$_3$)
$^1$H NMR Spectrum of 14 (300 MHz, CDCl$_3$)

$^{13}$C NMR Spectrum of 14 (75 MHz, CDCl$_3$)
$\text{H NMR Spectrum of 15 (500 MHz, CDCl}_3\text{)}$

$\text{C NMR Spectrum of 15 (75 MHz, CDCl}_3\text{)}$
$^1$H NMR Spectrum of 4 (300 MHz, CDCl$_3$)

$^{13}$C NMR Spectrum of 4 (75 MHz, CDCl$_3$)