Supporting Information
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Supporting Information for:

An Inverse Electron Demand Diels-Alder-Based Total Synthesis of Urolithin

M7

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1. Experimental Procedures and Characterization Data

General Experimental Procedure

All reactions were performed under an atmosphere of N₂ unless otherwise noted. Except where stated, commercial reagents and all solvents were used as received. THF was distilled immediately prior to use from sodium/benzophenone. Melting points (mp) were measured with a Fisher-Johns melting point apparatus and are uncorrected.

Thin layer chromatography (TLC) was performed using Macherey-Nagel Polygram® SIL G/UV₂₅₄ precoated silica plates. TLC plates were visualized using a short wave (254 nm) UV lamp or a PMA solution. Flash chromatography was carried out using Silica Gel 60 (E. Merck, 230-400 mesh) with the mobile phase indicated in the experimental sections.

¹H NMR spectra were obtained from CDCl₃ solutions, except when noted, using a General Electric 300 NB instrument operating at 300.1 MHz or a Bruker Avance 500 instrument operating at 500.1 MHz. Chemical shifts are relative to internal Me₄Si standard. Coupling constants are reported in Hz. Reported multiplicities are apparent. ¹³C NMR spectra are recorded at 75.47 and 125.8 MHz. Chemical shifts are reported relative to the solvent (δ 77.0 for CDCl₃, δ 39.5 for DMSO-d₆) and the number of attached protons, as determined by an attached proton experiment are given in parentheses. Assignments, where given, were established using HMQC, HMBC, and COSY experiments.

Infrared (IR) spectra (cm⁻¹) were recorded on a Mattson Polaris FT-IR spectrophotometer using neat samples or as Nujol mulls in NaCl cells. Low resolution mass spectrometric (MS) data were obtained on a V.G. Micromass 7070HS instrument operating at 70 eV. High-resolution mass spectrometric data were performed by the University of Ottawa Mass
Spectrometry Centre. Combustion analyses were obtained at the MicroAnalytical Service Laboratory at the University of Alberta and Canadian Microanalytical Service, Ltd. UV-VIS spectra were obtained on a Cary 5e spectrophotometer.

**Experimental Procedures**

**Methyl (E)-3-(7-methoxy-2-oxo-2H-chromen-3-yl) acrylate (10) and**

**3-methoxycarboxyl-2-methoxycarboxylmethyl-7-methoxy-2H-chromene (13)**

![Chemical structures](image)

To a magnetically stirred solution of 2-hydroxy-4-methoxybenzaldehyde 11 (5.00 g, 32.9 mmol) and 12 (4.86 mL, 34.5 mmol) in benzene (100 mL) was added piperidine (1.63 mL, 16.5 mmol) dropwise and the resulting mixture was heated at reflux with azeotropic removal of water for 3 h. The reaction mixture was cooled to room temperature, whereupon a white precipitate formed. The mixture was further cooled in an ice/water bath and the precipitate was isolated by suction filtration and the filter cake was washed with cool benzene (50 mL) to afford 10 (6.69 g, 78%) as a white solid. The filtrate was concentrated under reduced pressure and the residue was subjected to flash chromatography on silica gel (5% ethyl acetate/CH₂Cl₂) to afford 13 (2.13 g, 22%) as a yellow oil.

**10**: mp 207–208 °C; IR (nujol) ν = 1719 (s), 1604 (s), 1190 (s) cm⁻¹; ¹H NMR (CDCl₃, 500 MHz) δ = 7.81 (s, 1H, C4-H), 7.55 (d, J = 16.2 Hz, 1H, C1'-H), 7.44 (d, J = 7.8 Hz, 1H, C5-H), 7.05 (d, J = 15.5 Hz, 1H, C2'-H), 6.88 (dd, J = 9.0, 2.5 Hz, 1H, C6-H), 6.83 (d, J = 1.6 Hz, 1H, C8-H), 3.90 (s, 3H, C5'-H), 3.80 (s, 3H, C4'-H); ¹³C NMR (CDCl₃, 125.8 MHz) δ = 167.6 (0,
C3'), 163.9 (0, C7), 159.3 (0, C2), 155.6 (0, C8a), 143.9 (1, C4), 138.6 (1, C1'), 129.6 (1, C5), 121.8 (1, C2'), 118.8 (0, C3), 113.4 (1, C6), 112.7 (0, C4a), 100.5 (1, C8), 55.9 (3, C5'), 51.8 (3, C4'); EI-MS m/z (%) 260 (M+, 47), 229 (24), 201 (100), 173 (6), 102 (10); Anal. calcd for C14H12O5: C, 64.61; H, 4.65. Found C, 64.23; H, 4.78.

13: IR (neat) ν = 3003 (m), 2953 (s), 2842 (m), 1738 (s), 1706 (s), 1612 (s) cm⁻¹; ¹H NMR (CDCl₃, 500 MHz) δ = 7.47 (s, 1H, C4-H), 7.07 (d, J = 8.2 Hz, 1H, C5-H), 6.51 (dd, J = 8.3, 2.7 Hz, 1H, C6-H), 6.42 (d, J = 2.6 Hz, 1H, C8-H), 5.71 (dd, J = 10.1, 3.3 Hz, 1H, C1-H), 3.81 (s, 3H, C2'-H), 3.79 (s, 3H, C6'-H), 3.72 (s, 3H, C5'-H), 2.80 (dd, J = 15.2, 9.9 Hz, 1H, C3'-H), 2.57 (dd, J = 15.7, 3.2 Hz, 1H, C3'-H); ¹³C NMR (CDCl₃, 125.8 MHz) δ = 170.3 (0, C4'), 165.0 (0, C1'), 163.4 (0, C7), 154.4 (0, C8a), 133.6 (1, C4), 129.9 (1, C5), 120.7 (0, C3), 113.2 (0, C4a), 108.7 (1, C6), 102.3 (1, C8), 71.0 (1, C2), 55.5 (3, C6'), 52.8 (3, C2' + C5'), 39.6 (2, C3'); EI-MS m/z (%) 292 (M⁺, 12), 232 (21), 219 (100), 174 (11), 126 (12), 99 (15); HRMS (El) calcd for C₁₅H₁₆O₆: 292.0946, found 292.0958.

4-Methoxy-9,10-dihydro-9-oxaphenanthren-10-one-2-carboxylic acid methyl ester (14)

A solution of dimethoxyacetaldehyde (60 wt. % solution in water) (3.27 mL, 21.7 mmol) and pyrrolidine (1.82 mL, 21.8 mmol) in benzene (20 mL) was heated at reflux with azeotropic removal of water for 1 h. The resulting mixture was allowed to cool for 10 min and solid 3 (0.50 g, 2.17 mmol) was added in one portion. The resulting mixture was heated at reflux for 1 d. The reaction mixture was cooled to room temperature then concentrated under reduced pressure. The
residue was taken up in CH$_2$Cl$_2$ (20 mL) and washed with aqueous 1 M HCl solution (5 × 10 mL), dried over MgSO$_4$, gravity filtered and, concentrated under reduced pressure to afford 14 (0.62 g, 100%) as a colorless solid: mp 204–205 °C; IR (powder) $\nu$ = 1715 (s), 1601 (m) cm$^{-1}$; $^1$H NMR (CDCl$_3$, 500 MHz) $\delta$ = 8.86 (dd, $J$ = 8.3, 1.5 Hz, 1H), 8.70 (d, $J$= 1.7 Hz), 7.94 (d, $J$=1.7 Hz, 1H), 7.51-7.48 (m, 1H), 7.36–7.30 (m, 2H), 4.13 (s, 3H), 3.98 (s, 3H); $^{13}$C NMR (CDCl$_3$, 500 MHz) $\delta$ 165.6, 160.6, 157.4, 151.2, 130.8, 130.6, 129.0, 127.0, 127.9, 124.0, 123.1, 117.3, 117.1, 116.6, 56.3, 52.6; APCI(+)-MS $m/z$ (%) 285 ([M+1]$^+$, 100), 286 (20); HRMS (EI) calcd for C$_{16}$H$_{12}$O$_5$: 284.0685, found 284.0688.

2’-Hydroxy-2-(hydroxymethyl)-6-methoxybiphenyl-4-carboxylic acid methyl ester (15)

![Structure of 2’-Hydroxy-2-(hydroxymethyl)-6-methoxybiphenyl-4-carboxylic acid methyl ester (15)](structure.png)

To a mixture of 14 (52 mg, 0.18 mmol), NaBH$_4$ (34 mg, 0.23 mmol) and DME (0.63 mL) was added MeOH (0.31 mL) slowly over a period of 1 h and the resulting mixture was heated at 70 °C for 2 h. After cooling to room temperature 1 M aqueous HCl solution (2.0 mL) was added and the resulting mixture was extracted with ethyl acetate (3 × 5 mL). The combined organic layers were dried over MgSO$_4$, gravity filtered and concentrated under reduced pressure. The residue was subjected to flash chromatography on silica gel (50% ethyl acetate / CH$_2$Cl$_2$) to afford 15 (33 mg, 64%) as a white solid: mp 148–150 °C; IR (powder) $\nu$ = 3411 (m), 3360 (s), 1703 (s) cm$^{-1}$; $^1$H NMR (CDCl$_3$, 500 MHz) $\delta$ = 7.90 (d, $J$ = 1.5 Hz, 1H), 7.62 (d, $J$ = 1.5 Hz, 1H), 7.32–7.29 (m, 1H), 7.06–7.04 (m, 1H), 7.02–6.99 (m, 2H), 4.45 and 4.43 (AB system, $J$ = 12.7 Hz, 2H), 3.95 (s, 3H), 3.80 (s, 3H); $^{13}$C NMR (CDCl$_3$, 500 MHz) $\delta$ = 166.7, 157.4, 152.9,
141.7, 131.4, 130.8, 129.8, 129.2, 122.32, 122.29, 120.9, 116.8, 111.3, 63.2, 56.2, 52.4; APCI(–)
)-MS m/z (%) 287 ([M–1]$^+$, 100), 288 (20); 225 (10); HRMS (EI) calcd for C$_{16}$H$_{16}$O$_5$: 288.0998, found 288.1001.

3,10-Dimethoxy-6$H$-dibenzo[b,d]pyran-6-one-8-carboxylic acid methyl ester (9)

A solution of dimethoxyacetaldehyde (60 wt. % solution in water) (19.6 mL, 0.130 mol) and pyrrolidine (9.76 mL, 0.117 mol) in benzene (150 mL) was heated at reflux with azeotropic removal of water for 1 h. The resulting mixture was allowed to cool for 10 min and solid 10 (3.38 g, 13.0 mmol) was added in one portion. The resulting mixture was heated at reflux for 7 d. The reaction mixture was cooled to room temperature then concentrated under reduced pressure. The residue was taken up in CH$_2$Cl$_2$ (150 mL) and washed with aqueous 1 M HCl solution (5 × 50 mL), dried over MgSO$_4$, gravity filtered and concentrated under reduced pressure to afford 9 (4.08 g, 100%) as a tan solid: mp 195–196 °C; IR (nujol) ν = 1735 (s), 1715 (s), 1607 (m), 1121 (s) cm$^{-1}$; $^1$H NMR (CDCl$_3$, 500 MHz) δ = 8.76 (d, J = 9.3 Hz, 1H, C5-H), 8.57 (d, J = 1.1 Hz, 1H, C1-H), 7.80 (d, J = 1.0 Hz, 1H, C3-H), 6.81 (dd, J = 9.6, 3.0 Hz, 1H, C6-H), 6.76 (d, J = 2.6 Hz, 1H, C8-H), 4.08 (s, 3H, C2'-H), 3.96 (s, 3H, C4'-H), 3.87 (s, 3H, C1'-H); $^{13}$C NMR (CDCl$_3$, 125.8 MHz) δ = 165.6 (0, C3'), 161.3 (0, C7), 160.7 (0, C10), 156.4 (0, C4), 152.7 (0, C8a), 130.0 (1, C5), 129.1 (0), 128.0 (0, C8c), 124.0 (1, C1), 121.3 (0), 116.1 (1, C3), 111.7 (1, C6), 110.0 (0, C8b), 101.3 (1, C8), 56.1 (3, C2'), 55.3 (3, C1'), 52.4 (3, C4'); EI-
To a 0 °C slurry of LiAlH₄ (1.77 g, 46.6 mmol) in THF (10 mL) was added a solution of 9 (2.32 g, 7.37 mmol) in THF (150 mL) dropwise. The ice/water bath was then removed and the resulting mixture was heated at reflux for 5 h. After cooling to room temperature water (1.7 mL) was added carefully and stirring was continued for 10 min. Aqueous 15% NaOH solution (1.7 mL) was then added and stirring was continued for 10 min. Another portion of water (5.1 mL) was added and stirring was continued for 30 min. The mixture was diluted with aqueous 1 M HCl solution (100 mL) and extracted with EtOAc (3 × 50 mL). The combined organic layers were dried over MgSO₄, gravity filtered and concentrated under reduced pressure to afford 16 (2.01 g, 94%) as a white solid: mp 147.5–148 °C; IR (nujol) ν = 3408 (brs), 1611 (m) cm⁻¹; ¹H NMR (CDCl₃, 500 MHz) δ = 9.08 (br s, 1H, C2'-OH), 7.11 (s, 1H, C3-H), 6.85 (s, 1H, C5-H), 6.78 (d, J = 8.3 Hz, 1H, C6'-H), 6.44 (d, J = 2.5 Hz, 1H, C3'-H), 6.39 (dd, J = 8.3, 2.4 Hz, 1H, C5'-H), 5.15 (t, J = 5.7 Hz, 1H, C8-OH), 4.85 (br s, 1H, C7-OH), 4.52 (d, J = 5.3 Hz, 2H, C8-H), 4.19 (d, J = 13.5 Hz, 1H, C7-H), 4.10 (d, J = 14.3 Hz, 1H, C7-H), 3.72 (s, 3H, C8'-H), 3.61 (s, 3H, C10-H); ¹³C NMR (CDCl₃, 125.8 MHz) δ = 159.3 (0, C4'), 156.6 (0, C6), 155.6 (0, C2'), 142.02 (0), 142.00 (0), 131.8 (1, C6'), 123.2 (0, C1), 116.3 (1, C3), 115.6 (0, C1'), 107.4 (1, C5), 104.2 (1, C5'), 101.2 (1, C3'), 63.3 (2, C8), 60.7 (2, C7), 55.3 (3), 54.8 (3); EI-MS m/z (%) 290
(M+, 29), 272 (100), 257 (50), 241 (21), 227 (5), 128 (8); HRMS (EI) calcd for C_{16}H_{18}O_{5}: 290.1153, found 290.1152.

3,10-Dimethoxy-6H-dibenzo[b,d]pyran-6-one-8-carbaldehyde (17)

To a magnetically stirred solution of 16 (241 mg, 0.831 mmol) in benzene (50 mL) was added freshly made Fétizon reagent (8.55 g, 12.5 mmol) in one portion and the resulting mixture was heated at reflux for 24 h. The mixture was cooled to room temperature then suction filtered to remove the excess Fétizon reagent. The filtrate was concentrated under reduced pressure and the residue was subjected to flash chromatography on silica gel (4% ethyl acetate / CH$_2$Cl$_2$) to afford 17 (0.234 g, 99%) as a white solid: mp 225–227 °C; IR (nujol) 1731 (s), 1699 (s), 1622 (s) cm$^{-1}$; $^1$H NMR (CDCl$_3$, 500 MHz) $\delta$ = 10.07 (s, 1H, C1''-H), 8.93 (d, J = 8.8 Hz, 1H, C1-H), 8.51 (d, J = 1.7 Hz, 1H, C7-H), 7.78 (d, J = 1.6 Hz, 1H, C9-H), 6.93–6.89 (m, 2H, C2-H + C4-H), 4.14 (s, 3H, C1'''-H), 3.91 (s, 3H, C1'-H); $^{13}$C NMR (CDCl$_3$, 125.8 MHz) δ 190.5 (1, C1'''), 161.9 (0, C3), 160.7 (0, C6), 157.3 (0, C10), 153.3 (0, C4a), 135.3 (0), 130.5 (1, C1), 130.2 (0, C10a), 127.4 (1, C7), 121.9 (0), 112.9 (0), 112.4 (1, C9), 112.2 (1, C2), 101.5 (1, C4), 56.3 (3, C1'''), 55.7 (3, C1'); EI-MS m/z (%) 284 (M$^+$, 100), 269 (56), 254 (7), 198 (6), 142 (9), 114 (6); HRMS (EI) calcd for C$_{16}$H$_{12}$O$_5$: 284.0684, found 284.0685.
3,10-Dimethoxy-6H-dibenzo[b,d]pyran-6-one-8-carboxylic acid (19)

A solution of 9 (1.22 g, 3.89 mmol) in 9:1 1 M NaOH/EtOH (100 mL) was heated at reflux for 1 h. The heat source was removed and the pH of the reaction was immediately made acidic using aqueous concentrated HCl while the mixture was still hot. Once acidic, a white precipitate formed. The mixture was cooled to room temperature and the precipitate was isolated by suction filtration to afford 19 (1.17 g, 100 %) as a white solid: mp > 300 °C; IR (nujol) ν = 3158–2465 (br s), 1737 (s), 1690 (s), 1610 (s) cm⁻¹; ¹H NMR (DMSO-d₆, 500 MHz) δ = 13.44 (br s, 1H, CO₂H), 8.77 (d, J = 8.5 Hz, 1H, C1-H), 8.32 (d, J = 1.5 Hz, 1H, C7-H), 7.83 (d, J = 1.7 Hz, 1H, C9-H), 6.95 (d, J = 2.5 Hz, 1H, C4-H), 6.92 (dd, J = 9.6, 2.8 Hz, 1H, C2-H), 4.08 (s, 3H, C1'''-H), 3.86 (s, 3H, C1′-H); ¹³C NMR (DMSO-d₆, 125.8 MHz) δ = 165.9 (0, C1''), 161.0 (0, C3), 159.9 (0, C6), 156.3 (0, C10), 152.3 (0, C4a), 130.2 (0), 129.6 (1, C1), 126.9 (0, C10a), 122.7 (1, C7), 120.9 (0), 116.4 (1, C9), 111.8 (0, C2), 109.5 (0, C10b), 101.3 (1, C4), 56.3 (3, C1'''), 55.7 (3, C1'); EI-MS m/z (%) 300 (M⁺, 100), 285 (57), 214 (6), 150 (4), 113 (4); Anal. calcd for C₁₆H₁₂O₆: C, 64.00; H, 4.03. Found C, 63.88; H, 4.00.

8-Acetyl-3,10-dimethoxy-6H-dibenzo[b,d]pyran-6-one (20)
To a slurry of 19 (2.00 g, 6.66 mmol) in THF (80 mL) at 0 °C (ice/water bath) was added NaH (60% dispersion in mineral oil) (0.296 g, 7.33 mmol) slowly and the resulting mixture was allowed to stir and warm to room temperature for 1 h. The mixture was concentrated under reduced pressure and freshly distilled oxalyl chloride (70 mL) was added to the residue. The resulting mixture was then heated at reflux for 12 h, cooled to room temperature and concentrated under reduced pressure. THF (80 mL) was added to the residue and the mixture was cooled to 0 °C (ice/water bath). A 2 M solution of Zn(CH₃)₂ in hexanes (1.3 mL, 20 mmol) was added dropwise followed by Pd(PPh₃)₄ (80 mg, 0.15 mmol) in one portion and the mixture was stirred at room temperature for 4 d. The reaction was quenched with aqueous saturated ammonium chloride solution (50 mL) and stirred at room temperature for 30 min. The THF was removed under reduced pressure. To the remaining aqueous mixture was added CH₂Cl₂ (500 mL) and the layers were separated. The aqueous layer was extracted with CH₂Cl₂ (2 × 50 mL) and the combined organic layers were dried over MgSO₄, gravity filtered and concentrated under reduced pressure. The residue was then subjected to flash chromatography on silica gel (5% ethyl acetate / CH₂Cl₂) to afford 20 (1.67 g, 84%) as a white solid: mp 249–250 °C; IR (nujol) ν = 1731 (s), 1676 (s), 1629 (m) cm⁻¹; ¹H NMR (CDCl₃, 500 MHz) δ = 8.91 (d, J = 9.0 Hz, 1H, C1-H), 8.58 (d, J = 1.3 Hz, 1H, C7-H), 7.91 (d, J = 1.8 Hz, 1H, C9-H), 6.92–6.88 (m, 2H, C2-H + C4-H), 4.12 (s, 3H, C1‴-H), 3.90 (s, 3H, C1’-H), 2.71 (s, 3H, C2”-H); ¹³C NMR (CDCl₃, 125.8 MHz) δ = 196.5 (0, C1’), 161.7 (0, C3), 161.0 (0, C6), 157.0 (0, C10), 153.0 (C4a), 136.0 (0, C8), 130.3 (1, C1), 128.9 (0, C10a), 123.8 (1, C7), 121.5 (0, C6a), 114.1 (1, C9), 112.1 (1, C2), 110.2 (0, C10b), 101.5 (1, C4), 56.2 (3, C1‴′′), 55.7 (3, C1′′′), 26.5 (3, C2’’); GC-MS m/z (%) 298 (M⁺, 100), 283 (98), 255 (16), 212 (16), 141 (9), 113 (9); HRMS (EI) calcd for C₁₇H₁₄O₅: 298.0840, found 298.0862.
8-Acetoxy-3,10-dimethoxy-6H-dibenzo[b,d]pyran-6-one (21)

To a stirred solution of 20 (114 mg, 0.382 mmol) and m-CPBA (0.503 g, 1.91 mmol) in CH₂Cl₂ (5 mL) was added trifluoroacetic acid (0.03 mL, 0.4 mmol) dropwise and the resulting mixture was stirred for 4 d at room temperature. The reaction was diluted with CH₂Cl₂ (25 mL) and then concentrated under reduced pressure onto silica gel. The residue was immediately subjected to flash chromatography on silica gel (4% ethyl acetate / CH₂Cl₂) to afford 21 (88.9 mg, 74%) as a white solid: mp 207–208 °C; IR (nujol) ν = 1769 (s), 1726 (s), 1621 (s) cm⁻¹; ¹H NMR (CDCl₃, 500 MHz) δ = 8.80 (d, J = 9.6 Hz, 1H, C1-H), 7.70 (d, J = 3.1 Hz, 1H, C7-H), 7.08 (d, J = 2.8 Hz, 1H, C9-H), 6.88–6.86 (m, 2H, C2-H + C4-H), 4.04 (s, 3H, C1'''-H), 3.88 (s, 3H, C1'-H), 2.36 (s, 3H, C2''-H); ¹³C NMR (CDCl₃, 125.8 MHz) δ = 169.0 (0, C1'''), 160.8 (0, C6), 160.5 (0, C3), 157.6 (0, C10), 152.0 (0, C4a), 149.9 (0, C8), 129.1 (1, C1), 122.7 (0, C10a), 122.3 (0, C6a), 114.7 (1, C7), 111.8 (1, C2), 111.1 (1, C9), 110.4 (0, C10b), 101.4 (1, C4), 56.2 (3, C1''''), 55.5 (3, C1'), 21.1 (3, C2''); GC-MS m/z (%) 314 (M⁺, 16), 272 (100), 257 (43), 228 (8), 185 (6), 115 (3); HRMS (EI) calcd for C₁₇H₁₄O₆: 314.0789, found 314.0773.

3,8,10-Trihydroxy-6H-dibenzo[b,d]pyran-6-one (2)
To 21 (20.1 mg, 0.0639 mmol) was added aqueous 65% HI solution (5 mL) and the resulting mixture was heated at reflux for 30 min. The reaction was cooled to room temperature and the solid was isolated by suction filtration using a sintered glass funnel. The solid was washed with cold water (5 mL) and allowed to dry under reduced pressure in a dessicator for 24 h to afford 2 (15.3 mg, 98%) as a white solid: mp >300 °C (lit. mp >300 °C), IR (nujol) ν = 3418 (m), 1700 (s), 1617 (m) cm⁻¹; ¹H NMR (DMSO-d₆, 500 MHz) δ = 10.76 (s, 1H), 10.01 (s, 1H), 9.97 (s, 1H), 8.74 (d, J = 9.0 Hz, 1H), 7.12 (d, J = 2.8 Hz, 1H); 6.85 (d, J = 2.0 Hz, 1H), 6.69 (dd, J = 8.8, 2.5 Hz, 1H); ¹³C NMR (DMSO-d₆, 125.8 MHz) δ = 160.7, 157.2, 157.0, 156.0, 150.2, 127.9, 121.6, 114.7, 112.3, 109.91, 109.86, 105.9, 102.6; EI-MS m/z (%) 244 (M⁺, 3).
2. $^1\text{H}$ and $^{13}\text{C}$ NMR spectra.