Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information of 'Electron and Steric Effects on The Three-fold Scholl-Type Cycloheptatriene Ring Formation Around A Tribenzotriquinacene Core'

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1. Synthesis

General: All reagents were purchased from commercial suppliers and used without further purification. Dichloromethane was freshly distilled from calcium hydride. All reactions were carried out under nitrogen atmosphere unless otherwise stated. All reactions were monitored by thin layer chromatographic analysis on pre-coated silica gel plates, which were visualized by UV lamp at 254 or 365 nm and/or stained using 5% (w/v) dodecamolybdophosphoric acid in ethanol followed by heating. Flash column chromatography was performed on glass column of silica gel (230–400 mesh) and solvent ratios were expressed in volume to volume. ^{1}H, ^{13}C, COSY NMR spectra for structural characterization were recorded either on a Bruker Avance DPX 400 spectrometer or a Bruker Avance III 400 spectrometer (^{1}H: 400 MHz; ^{13}C: 100 MHz) or a Bruker Avance III 700 spectrometer (^{1}H: 700 MHz; ^{13}C: 176 MHz) as specified. Unless otherwise stated, all NMR measurements were conducted in $CDCl_3$ at 22 °C. The residual signals of the deuterated solvents or TMS were used as the internal standards. Chemical shifts were reported as parts per million in δ scale using the solvent residual peak as internal standard for ^{1}H and ^{13}C NMR. Coupling constants (*J*) were reported in Hertz (Hz). All mass spectra were obtained on a ThermoFinnigan MAT 95 XL double focusing sector mass spectrometer or on a Bruker SolariX 9.4 T FT-ICR mass spectrometer with electron spray ionization (ESI) technique, or on a Bruker Daltonics Autoflex MALDI-TOF mass spectrometer using 2,5-dihydroxybenzoic acid (DHB) as matrices, or on a Fisons VG Autospec X double-focusing mass spectrometer (EI, 70 eV). The reported molecular masses, given as *m*/*z* values, were monoisotopic mass unless otherwise stated. Melting points were measured on an Electrothermal® 9100 digital melting point apparatus or a Stuart® automatic melting point apparatus SMP40 and were uncorrected.

Compound 6a: Compound **5** (100 mg, 0.136 mmol), palladium(II) acetate (21.3 mg, 0.095 mmol), 2-dicyclohexylphosphino-2′,6′-dimethoxybiphenyl (56 mg, 0.136 mmol), cesium fluoride (372 mg, 2.448 mmol), phenylboronic acid (300 mg, 2.448 mmol) were mixed in dioxane (4 mL) in a Schlenk tube and degassed. The reaction mixture was stirred at 100 °C for 5 d. The mixture was filtered through Celite, concentrated under reduced pressure and the residue was purified by flash column chromatography (hexane/EtOAc = 80/1) to afford compound **6a** (68 mg, 0.130 mmol, 97%) as a colorless solid. M.p.: 298–300 °C; R_f: 0.23 (hexane/EtOAc = 80/1); ^{1}H NMR (400 MHz): 7.66 (d, J = 7.2 Hz, 2 H, ArH), 7.61–7.37 (m, 13 H, ArH), 7.31–7.29 (m, 2 H, ArH), 7.01 (d, J = 7.6 Hz, 1 H, ArH), 6.81 (t, J = 7.6 Hz, 1 H, ArH), 6.58–6.53 (m, 2 H, ArH), 5.80 (d, J = 8 Hz, 1 H, ArH), 5.65–5.59 (m, 2 H, ArH), 5.24 (s, 1 H, CH), 5.09 (s, 1 H, CH), 4.96 (s, 1 H, CH), 1.82 (s, 3 H, CH_3); ^{13}C NMR (100 MHz): 146.0, 145.9, 144.7, 144.1, 143.4, 143.3, 142.9, 142.8, 142.7, 139.1, 138.84, 138.82, 130.13, 130.10, 129.4, 129.2, 129.1, 129.0, 127.6, 127.5, 127.4, 127.3, 126.85, 126.83, 125.3, 125.0, 124.9, 62.6, 62.4, 61.8, 59.6, 29.0; MS (EI): *m*/*z* (%) 522.3 (100, $[M]^{+\bullet}$), 507.2 (15, $[M - CH_3]^+$), 406.2 (27); MS (ESI): *m*/*z* (%) 545.2 (100, $[M + Na]^+$). Accurate mass ($[M + Na]^+$, ESI) calcd for $C_{41}H_{30}Na^+$: 545.2240, found: 545.2246.

Compound 6b: The synthetic procedure was similar to that yielding compound **6a**, using 4-*tert*-butylphenylboronic acid instead of phenylboronic acid. The crude product was purified by flash column chromatography (hexane/EtOAc = 20/1) to afford compound **6b** (84 mg, 0.122 mmol, 89%) as a colorless solid. M.p.: 226–229 °C; R_f: 0.17 (hexane/EtOAc = 20/1); ^{1}H NMR (400 MHz): 7.58 (s, 4 H, ArH), 7.55–7.47 (m, 6 H, ArH), 7.37 (d, J = 8.3 Hz, 2 H, ArH), 7.31–7.27 (m, 2 H, ArH), 6.99 (d, J = 7.4 Hz, 1 H, ArH), 6.75 (t, J = 7.7 Hz, 1 H, ArH), 6.51–6.47 (m, 2 H, ArH), 5.64

(d, J = 7.9 Hz, 1 H, ArH), 5.47–5.46 (m, 2 H, ArH), 5.21 (s, 1 H, CH), 5.05 (s, 1 H, CH), 4.92 (s, 1 H, CH), 1.79 (s, 3 H, CH_3), 1.43 (s, 9 H, $C(CH_3)_3$), 1.40 (s, 9 H, $C(CH_3)_3$), 1.38 (s, 9 H, $C(CH_3)_3$); ^{13}C NMR (100 MHz): 150.64, 150.55, 150.4, 146.0, 145.9, 144.8, 144.1, 143.6, 143.4, 139.9, 139.81, 139.76, 139.1, 138.6, 129.9, 129.8, 129.11, 129.07, 128.9, 127.4, 126.6, 126.1, 126.0, 125.8, 125.4, 125.1, 124.9, 62.58, 62.57, 61.9, 59.6, 34.82, 34.79, 34.7, 31.64, 31.61, 31.59, 29.0; MS (EI): m/z (%) 690.4 (100, $[M]^{+\bullet}$), 675.4 (9, $[M - CH_3]^+$), 633.4 (22, $[M - CH_3 - C_3H_6]^+$), 574.4 (13), 337.7 (6, $[M - CH_3]^{2+(\bullet)}$), 330.2 (30, $[M - 2\ CH_3]^{2+}$), 57.1 (74, $C_4H_9^+$); MS (ESI): m/z (%) 713.4 (100, $[M + Na]^+$). Accurate mass ($[M + Na]^+$, ESI) calcd for $C_{53}H_{54}Na^+$: 713.4118, found: 713.4138.

Compound 6c: The synthetic procedure was similar to that yielding compound **6a**, using 2-methoxyphenylboronic acid instead of phenylboronic acid. The crude product was purified by flash column chromatography (hexane/EtOAc = 9/1) to afford compound **6c** (68 mg, 0.112 mmol, 82%) as a colorless solid. M.p.: 243–245 °C (dec.); R_f: 0.25 (hexane/EtOAc = 9/1); ^{1}H NMR (400 MHz, 1,1,2,2-tetrachloroethane-d_2, 60 °C): 7.54–7.41 (m, 5 H, ArH), 7.33 (d, J = 7.3 Hz, 1 H, ArH), 7.27 (s, 2 H, ArH), 7.25–7.06 (m, 6 H, ArH), 7.02 (d, J = 7.4 Hz, 1 H, ArH), 6.87 (t, J = 7.6 Hz, 1 H, ArH), 6.65–6.64 (m, 2 H, ArH), 5.93 (d, J = 7.7 Hz, 1 H, ArH), 5.81–5.80 (m, 2 H, ArH), 4.94 (s, 1 H, CH), 4.81 (s, 1 H, CH), 4.67 (s, 1 H, CH), 3.93 (s, 3 H, OCH_3), 3.88 (s, 3 H, OCH_3), 3.81 (s, 3 H, OCH_3), 1.77 (s, 3 H, CH_3); ^{13}C NMR (100 MHz, 1,1,2,2-tetrachloroethane-d_2, 60 °C): 156.5, 146.3, 145.7, 145.3, 144.9, 135.3, 134.8, 131.8, 131.6, 131.4, 131.3, 129.7, 129.1, 128.8, 128.75, 128.66, 126.6, 126.13, 126.06, 125.2, 125.0, 124.6, 121.0, 120.9, 120.8, 111.8, 111.5, 63.4, 63.1, 62.5, 59.4, 55.5, 55.4, 55.3, 28.2; MS (MALDI): m/z (%) 635.3 (100, $[M + Na]^+$). Accurate mass ($[M + Na]^+$, MALDI) calcd for $C_{44}H_{36}NaO_3^+$: 635.2562, found: 635.2556.

Compound 6d: The synthetic procedure was similar to that yielding compound **6a**, using 3-methoxyphenylboronic acid instead of phenylboronic acid. The crude product was purified by flash column chromatography (hexane/EtOAc = 20/1) to afford compound **6d** (76 mg, 0.124 mmol, 91%) as a colorless solid. M.p.: 130–133 °C; R_f: 0.33 (hexane/EtOAc = 10/1); ^{1}H NMR (400 MHz): 7.52–7.44 (m, 2 H, ArH), 7.40 (t, J = 8 Hz, 1 H, ArH), 7.32–7.27 (m, 3 H, ArH), 7.23–7.22 (m, 2 H, ArH), 7.18–7.15 (m, 1 H, ArH), 7.11 (d, J = 7.6 Hz, 1 H, ArH), 7.07–6.96 (m, 5 H, ArH), 6.88 (t, J = 7.6, 1 H, ArH), 6.67–6.63 (m, 2 H, ArH), 5.95 (d, J = 7.8 Hz, 1 H, ArH), 5.81–5.76 (m, 2 H, ArH), 5.26 (s, 1 H, CH), 5.12 (s, 1 H, CH), 5.00 (s, 1 H, CH), 3.92 (s, 3 H, OCH_3), 3.89 (s, 3 H, OCH_3), 3.86 (s, 3 H, OCH_3), 1.84 (s, 3 H, CH_3); ^{13}C NMR (100 MHz): 160.3, 160.2, 160.1, 146.0, 145.9, 144.7, 144.14, 144.11, 144.08, 144.0, 143.4, 143.3, 138.9, 138.8, 138.7, 130.25, 130.16, 130.01, 129.99, 129.9, 129.1, 127.6, 126.9, 125.4, 125.1, 121.9, 121.8, 115.2, 115.1, 112.9, 112.7, 62.6, 62.5, 61.9, 59.6, 55.53, 55.52, 55.48, 29.0; MS (EI): *m*/*z* (%) 612.3 (100, $[M]^{+\bullet}$), 597.3 (13, $[M - CH_3]^+$), 496.2 (33), 390.2 (7), 306.1 (12, $[M]^{2+}$); MS (MALDI): *m*/*z* (%) 635.3 (100, $[M + Na]^+$). Accurate mass ($[M + Na]^+$, MALDI) calcd for $C_{44}H_{36}NaO_3^+$: 635.2562, found: 635.2554.

Compound 6e: The synthetic procedure was similar to that yielding compound **6a**, using 4-methoxyphenylboronic acid instead of phenylboronic acid. The crude product was purified by flash column chromatography (hexane/EtOAc = 9/1) to afford compound **6e** (77 mg, 0.126 mmol, 93%) as a colorless solid. M.p.: 165–168 °C; R_f: 0.24 (hexane/EtOAc = 9/1); ^{1}H NMR (400 MHz): 7.56 (d, J = 8.4 Hz, 2H, ArH), 7.50 (d, J = 8.8 Hz, 2 H, ArH), 7.38 (d, J = 8.4 Hz, 2 H, ArH), 7.23 (A of AB system, d, J = 7.8 Hz, 1 H, ArH), 7.22 (B of AB system, d, J = 7.8 Hz, 1 H, ArH), 7.08 (d, J = 8.7 Hz, 2 H, ArH), 7.05 (d, J = 8.7 Hz, 2 H, ArH), 7.00 (d, J = 8.7 Hz, 2 H, ArH), 6.97 (d, J =

7.2 Hz, 1 H, ArH), 6.81 (t, *J* =7.6 Hz, 1 H, ArH), 6.62–6.57 (m, 2 H, ArH), 5.89 (d, *J* = 7.6 Hz, 1 H, ArH), 5.78–5.72 (m, 2 H, ArH), 5.19 (s, 1 H, CH), 5.05 (s, 1 H, CH), 4.94 (s, 1 H, CH), 3.91 (s, 3 H, OCH_3), 3.89 (s, 3 H, OCH_3), 3.87 (s, 3 H, OCH_3), 1.80 (s, 3 H, CH_3); ^{13}C NMR (100 MHz): 159.1, 159.0, 158.9, 146.02, 146.98, 144.8, 144.0, 143.4, 138.7, 138.12, 138.08, 135.3, 135.25, 135.17, 130.4, 130.12, 130.09, 129.1, 127.7, 126.9, 125.3, 125.0, 124.6, 114.6, 114.5, 114.4, 62.7, 62.4, 61.8, 59.4, 55.52, 55.47, 55.4, 29.0; MS (EI): *m*/*z* (%) 612.3 (100, $[M]^{+\bullet}$), 597.2 (11, $[M - CH_3]^+$), 496.2 (18), 390.2 (7), 306.1 (12, $[M]^{2+}$); MS (MALDI): *m*/*z* (%) 635.3 (100, $[M + Na]^+$). Accurate mass ($[M + Na]^+$, MALDI) calcd for $C_{44}H_{36}NaO_3^+$: 635.2562, found: 635.2562.

Compound 6g: The synthetic procedure was similar to that yielding compound **6a**, using 2,4-dimethoxyphenylboronic acid instead of phenylboronic acid. The crude product was purified by flash column chromatography (hexane/EtOAc = 5/1) to afford compound **6g** (95 mg, 0.135 mmol, 99%) as a colorless solid. M.p.: 203–205 °C (dec.); R_f: 0.23 (hexane/EtOAc = 5/1); ^1H NMR (400 MHz, 1,1,2,2-tetrachloroethane-d_2, 60 °C): 7.44 (d, *J* = 8 Hz, 1 H, ArH), 7.38 (d, *J* = 8.1 Hz, 1 H, ArH), 7.27–7.23 (m, 3 H, ArH), 7.02 (d, *J* = 7.4 Hz, 1 H, ArH), 6.91 (t, *J* = 7.6 Hz, 1 H, ArH), 6.79–6.69 (m, 8 H, ArH), 6.05 (d, *J* = 7.6 Hz, 1 H, ArH), 5.96–5.95 (m, 2 H, ArH), 4.94 (s, 1 H, CH), 4.81 (s, 1 H, CH), 4.68 (s, 1 H, CH), 3.99 (s, 3 H, OCH_3), 3.96 (s, 3 H, OCH_3), 3.94 (s, 3 H, OCH_3), 3.93 (s, 3 H, OCH_3), 3.89 (s, 3 H, OCH_3), 3.83 (s, 3 H, OCH_3), 1.78 (s, 3 H, CH_3); ^{13}C NMR (100 MHz, 1,1,2,2-tetrachloroethane-d_2, 60 °C): 160.7, 160.6, 160.5, 157.43, 157.38, 157.2, 146.4, 145.7, 145.4, 145.1, 144.9, 144.2, 135.1, 134.4, 134.3, 132.2, 132.1, 130.0, 129.4, 126.6, 126.2, 126.1, 125.3, 125.1, 124.5, 124.4, 124.33, 124.27, 105.4, 105.3, 105.2, 99.5, 99.3, 63.4, 63.1, 62.5, 59.2, 55.51, 55.47, 55.3, 28.2; MS (MALDI): *m*/*z*

(%) 741.3 (100, $[M + K]^+$). Accurate mass ($[M + K]^+$, MALDI) calcd for $C_{47}H_{42}KO_6^+$: 741.2613, found: 741.2602.

Compound **6h**: The synthetic procedure was similar to that yielding compound **6a**, using 2,5-dimethoxyphenylboronic acid instead of phenylboronic acid. The crude product was purified by flash column chromatography (hexane/EtOAc = 5/1) to afford compound **6h** (78 mg, 0.112 mmol, 82%) as a colorless solid. M.p.: 182-185 °C; R_f: 0.33 (hexane/EtOAc = 3/1); ^{1}H NMR (400 MHz, 1,1,2,2-tetrachloroethane-d_2, 60 °C): 7.29–7.25 (m, 2 H, ArH), 7.12–7.11 (m, 1 H, ArH), 7.08–7.03 (m, 6 H, ArH), 7.01–6.98 (m, 2 H, ArH), 6.93–6.88 (m, 2 H, ArH), 6.71–6.66 (m, 2 H, ArH), 6.02 (s, 1 H, ArH), 5.89–5.87 (m, 2 H, ArH), 4.95 (s, 1 H, CH), 4.82 (s, 1 H, CH), 4.70 (s, 1 H, CH), 3.90 (s, 3 H, OCH_3), 3.864 (s, 3 H, OCH_3), 3.855 (s, 3 H, OCH_3), 3.83 (s, 3 H, OCH_3), 3.81 (s, 3 H, OCH_3), 3.75 (s, 3 H, OCH_3), 1.75 (s, 3 H, CH_3); ^{13}C NMR (100 MHz, 1,1,2,2-tetrachloroethane-d_2, 60 °C): 154.1, 154.0, 153.9, 151.0, 150.8, 146.2, 145.7, 145.3, 144.8, 135.2, 134.9, 134.8, 132.4, 132.20, 132.18, 129.7, 129.1, 126.6, 126.2, 126.1, 125.4, 125.2, 124.9, 117.8, 117.7, 114.3, 114.2, 113.2, 113.0, 63.3, 63.1, 62.5, 59.5, 56.2, 56.1, 56.10, 56.04, 28.2; MS (EI): *m*/*z* (%) 702.3 (100, $[M]^{+\bullet}$), 687.3 (7, $[M - CH_3]^+$), 671.3 (28, $[M - OCH_3]^+$), 351.1 (10, $[M]^{2+}$), 343.6 (7, $[M - CH_3]^{2+(\bullet)}$); MS (ESI): *m*/*z* (%) 725.3 (100, $[M + Na]^+$). Accurate mass ($[M + Na]^+$, ESI) calcd for $C_{47}H_{42}NaO_6^+$: 725.2874, found: 725.2886.

Compound 6j: The synthetic procedure was similar to that yielding compound **6a**, using 3,5-dimethoxyphenylboronic acid instead of phenylboronic acid. The crude product was purified by flash column chromatography (hexane/EtOAc = 7/1) to afford compound **6j** (92 mg, 0.131 mmol, 96%) as a colorless solid. M.p.: 260 °C (dec.); R_f: 0.43 (hexane/EtOAc = 4/1); ^{1}H NMR (400 MHz): 7.27 (A of AB system, d,

J = 7.8 Hz, 1 H, ArH), 7.26 (B of AB system, d, *J* = 7.8 Hz, 1 H, ArH), 7.03 (d, *J* = 7.2 Hz, 1 H, ArH), 6.88 (t, *J* = 7.6 Hz, 1 H, ArH), 6.79 (d, *J* = 2.4 Hz, 2 H, ArH), 6.73 (d, *J* = 2.4 Hz, 2 H, ArH), 6.68–6.64 (m, 2 H, ArH), 6.63 (d, *J* = 2 Hz, 2 H, ArH), 6.57 (t, *J* = 2.4 Hz, 1 H, ArH), 6.54 (t, *J* = 2 Hz, 1 H, ArH), 6.51 (t, *J* = 2 Hz, 1 H, ArH), 6.04 (d, *J* = 8 Hz, 1 H, ArH), 5.92–5.87 (m, 2 H, ArH), 5.19 (s, 1 H, CH), 5.06 (s, 1 H, CH), 4.96 (s, 1 H, CH), 3.87 (s, 6 H, OCH_3), 3.84 (s, 6 H, OCH_3), 3.81 (s, 6 H, OCH_3), 1.78 (s, 3 H, CH_3); ^{13}C NMR (100 MHz): 161.5, 161.4, 161.2, 146.0, 145.8, 144.68, 144.66, 144.5, 144.0, 143.3, 143.2, 139.0, 138.9, 129.8, 129.7, 128.9, 127.6, 126.9, 125.6, 125.4, 125.3, 107.7, 107.6, 99.5, 99.3, 62.7, 62.5, 62.0, 59.6, 55.68, 55.67, 55.6, 29.0; MS (EI): *m*/*z* (%) 702.3 (100, $[M]^{+\bullet}$), 687.3 (12, $[M - CH_3]^+$), 671.3 (9, $[M - OCH_3]^+$), 586.2 (19), 351.1 (20, $[M]^{2+}$); MS (MALDI): *m*/*z* (%) 725.3 (100, $[M + Na]^+$). Accurate mass ($[M + Na]^+$, MALDI) calcd for $C_{47}H_{42}NaO_6^+$: 725.2874, found: 725.2876.

Compound 6k: The synthetic procedure was similar to that yielding compound **6a**, using 2,3,4-trimethoxyphenylboronic acid instead of phenylboronic acid. The crude product was purified by flash column chromatography (hexane/EtOAc = 2/1) to afford compound **6k** (106 mg, 0.134 mmol, 99%) as a colorless solid. M.p.: 114–116 °C; R_f: 0.41 (hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz, 1,1,2,2-tetrachloroethane-d_2, 60 °C): 7.27–7.25 (m, 2 H, ArH), 7.22 (d, *J* = 8.4 Hz, 1 H, ArH), 7.16 (d, *J* = 8.4 Hz, 1 H, ArH), 7.03 (s, 1 H, ArH), 7.01 (s, 1 H, ArH), 6.92 (d, *J* = 8.4 Hz, 1 H, ArH), 6.89–6.86 (m, 2 H, ArH), 6.81 (d, *J* = 8.8 Hz, 1 H, ArH), 6.69–6.64 (m, 2 H, ArH), 6.01 (s, 1 H, ArH), 5.90–5.88 (m, 1 H, ArH), 5.85–5.83 (m, 1 H, ArH), 5.03 (s, 1 H, CH), 4.92 (s, 1 H, CH), 4.80 (s, 1 H, CH), 4.02 (s, 6 H, OCH_3), 3.98 (s, 3 H, OCH_3), 3.97 (s, 3 H, OCH_3), 3.96 (s, 3 H, OCH_3), 3.94 (s, 3 H, OCH_3), 3.68 (s, 3 H, OCH_3), 3.64 (s, 3 H, OCH_3), 3.56 (s, 3 H, OCH_3), 1.76 (s, 3 H, CH_3); ^{13}C NMR (100 MHz,

1,1,2,2-tetrachloroethane-d_2, 60 °C): 153.42, 153.38, 153.3, 151.43, 151.38, 151.3, 146.1, 145.9, 145.36, 143.3, 143.1, 135.0, 134.6, 134.53, 134.49, 129.79, 129.75, 129.3, 129.2, 129.1, 129.0, 126.4, 126.1, 125.5, 125.3, 125.2, 124.7, 108.43, 108.39, 108.36, 108.2, 63.2, 62.9, 62.4, 61.04, 61.01, 60.9, 60.8, 60.6, 59.4, 56.44, 56.38, 28.5; MS (ESI): *m*/*z* (%) 815.3 (100, $[M + Na]^+$). Accurate mass ($[M + Na]^+$, ESI) calcd for $C_{50}H_{48}NaO_9^+$: 815.3191, found: 815.3193.

Compound 6l: The synthetic procedure was similar to that yielding compound **6a**, using 3,4,5-trimethoxyphenylboronic acid instead of phenylboronic acid. The crude product was purified by flash column chromatography (hexane/EtOAc = 2/1) to afford compound **6l** (106 mg, 0.134 mmol, 99%) as a colorless solid. M.p.: 253–255 °C; R_f: 0.41 (hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz): 7.29 (A of AB system, d, *J* = 8 Hz, 1 H, ArH), 7.29 (B of AB system, d, *J* = 8 Hz, 1 H, ArH), 7.05 (d, *J* = 7.6 Hz, 1 H, ArH), 6.89 (t, *J* = 8 Hz, 1 H, ArH), 6.84 (s, 2 H, ArH), 6.78 (s, 2 H, ArH), 6.68–6.66 (m, 4 H, ArH), 5.93 (d, *J* = 7.6 Hz, 1 H, ArH), 5.79–5.74 (m, 2 H, ArH), 5.17 (s, 1 H, CH), 5.04 (s, 1 H, CH), 4.92 (s, 1 H, CH), 3.97 (s, 3 H, OCH_3), 3.95 (s, 3 H, OCH_3), 3.93 (s, 9 H, OCH_3), 3.89 (s, 6 H, OCH_3), 3.85 (s, 6 H, OCH_3), 1.82 (s, 3 H, CH_3); ^{13}C NMR (100 MHz): 154.0, 153.9, 153.7, 145.8, 145.5, 144.5, 143.9, 143.3, 142.9, 139.2, 139.0, 138.1, 137.97, 137.93, 137.8, 129.8, 129.7, 129.0, 127.6, 127.04, 127.00, 125.7, 125.4, 125.3, 106.7, 62.8, 62.4, 61.38, 61.36, 61.3, 59.6, 56.5, 56.50, 56.46, 29.1; MS (ESI): *m*/*z* (%) 815.3 (100, $[M + Na]^+$). Accurate mass ($[M + Na]^+$, ESI) calcd for $C_{50}H_{48}NaO_9^+$: 815.3191, found: 815.3184.

Compound 7a: Compound **6a** (68 mg, 0.130 mmol) was dissolved in anhydrous CH_2Cl_2 (10 mL) and cooled to 0 °C. 2,3-Dichloro-5,6-dicyano-*p*-benzoquinone (148 mg, 0.65 mmol) was added in one portion and the mixture was stirred for 5 min.

Trifluoromethanesulfonic acid (0.2 mL) was added and the mixture was stirred for 1 h. The reaction mixture was quenched by washing with saturated aqueous sodium carbonate (20 mL) and then concentrated under reduced pressure. The residue was purified by flash column chromatography (hexane/EtOAc = 6/1) to afford compound **7a** (24 mg, 0.046 mmol, 35%) as a colorless solid. M.p.: >300 °C; R_f: 0.19 (hexane/EtOAc = 6/1); ^{1}H NMR (400 MHz): 7.90–7.86 (m, 2 H, ArH), 7.60–7.52 (m, 12 H, ArH), 7.50–7.45 (m, 2 H, ArH), 7.35 (d, *J* = 7.8 Hz, 2 H, ArH), 6.59–6.55 (m, 2 H, ArH), 5.59–5.55 (m, 2 H, ArH), 5.22 (s, 2 H, CH), 2.12 (s, 1 H, OH), 1.76 (s, 3 H, CH_3); ^{13}C NMR (100 MHz): 147.6, 144.7, 142.0, 140.3, 139.0, 136.5, 133.8, 131.0, 130.0, 129.5, 129.2, 129.1, 128.0, 127.7, 127.2, 125.6, 89.6, 64.3, 62.8, 22.9; MS (ESI): *m*/*z* (%) 559.2 (34, $[M + Na]^+$), 519.2 (100, $[M + H - H_2O]^+$). Accurate mass ($[M + Na]^+$, ESI) calcd for $C_{41}H_{28}NaO^+$: 559.2032, found: 559.2057.

Compound 7b: The synthetic procedure was similar to that yielding compound **7a**, using **6b** (84 mg, 0.122 mmol) as the starting material. The crude product was purified by flash column chromatography (hexane/EtOAc = 20/1) to afford compound **7b** (31 mg, 0.044 mmol, 36%) as a colorless solid. M.p.: >300 °C; R_f: 0.17 (hexane/EtOAc = 20/1); ^{1}H NMR (400 MHz): 7.87 (d, *J* = 1.6 Hz, 1 H, ArH), 7.79 (d, *J* = 8 Hz, 1 H, ArH), 7.57–7.53 (m, 7 H, ArH), 7.49–7.47 (m, 4 H, ArH), 7.34 (d, *J* = 5.6 Hz, 1 H, ArH), 7.32 (d, *J* = 5.6 Hz, 1 H, ArH), 6.53–6.49 (m, 2 H, ArH), 5.46–5.42 (m, 2 H, ArH), 5.19 (s, 2 H, CH), 2.19 (s, 1 H, OH), 1.74 (s, 3 H, CH_3), 1.45 (s, 9 H, $C(CH_3)_3$), 1.41 (s, 18 H, $C(CH_3)_3$); ^{13}C NMR (100 MHz): 150.72, 150.70, 150.66, 147.41, 147.36, 144.6, 140.14, 140.12, 138.9, 138.7, 138.5, 135.9, 133.9, 133.5, 133.3, 130.49, 130.47, 129.4, 129.0, 128.9, 128.7, 126.9, 126.8, 125.9, 125.5, 125.0, 89.4, 64.0, 62.7, 34.8, 34.7, 31.5, 31.4, 22.8; MS (ESI): *m*/*z* (%) 727.4 (100, $[M + Na]^+$). Accurate mass ($[M + Na]^+$, ESI) calcd for $C_{53}H_{52}NaO^+$: 727.3910, found: 727.3915.

Compound 10: 2,3-Dichloro-5,6-dicyano-*p*-benzoquinone (37 mg, 0.163 mmol) was added in one portion to a solution of compound **6e** (100 mg, 0.163 mmol) in anhydrous CH_2Cl_2 (10 mL) at 0 °C. After 5 min, trifluoromethanesulfonic acid (0.2 mL) was added and the mixture was stirred for 1 h. It was then quenched by washing with saturated sodium carbonate solution (20 mL) and concentrated under reduced pressure. The residue was purified by flash column chromatography (hexane/EtOAc = 9/1) to afford a mixture containing compound **10** and compound **11e** in 0.87:1 ratio. Compound **10 (**7 mg, 0.011 mmol, 7%) was further purified by crystallization from a hexane/EtOAc mixture as colorless solid. M.p.: >300 °C; R_f: 0.51 (hexane/EtOAc = 7/1); ^{1}H NMR (700 MHz): 7.71 (d, J = 9.1 Hz, 1 H, ArH), 7.49–7.46 (m, 5 H, ArH), 7.42 (d, J = 7.7 Hz, 1 H, ArH), 7.29 (s, 1 H, ArH), 7.23 (d, J = 7.7 Hz, 1 H, ArH), 7.21 (d, J =7.7 Hz, 1 H, ArH), 7.06–7.05 (m, 5 H, ArH), 6.62–6.59 (m, 2 H, ArH), 5.72–5.70 (m, 2 H, ArH), 5.14 (s, 2 H, CH), 4.17 (s, 1 H, CH), 3.94 (s, 3 H, OCH_3), 3.90 (s, 6 H, OCH_3), 1.78 (s, 3 H, CH_3); ^{13}C NMR (176 MHz): 159.1, 159.0, 158.7, 147.5, 147.0, 145.3, 145.2, 140.7, 140.5, 139.1, 138.6, 137.8, 134.7, 134.6, 133.7, 133.6, 131.1, 130.8, 130.3, 129.4, 127.7, 127.4, 126.84, 126.81, 125.3, 114.5, 114.4, 114.3, 113.6, 63.8, 63.7, 60.8, 59.2, 55.5, 55.4, 27.9; MS (MALDI): *m*/*z* (%) 610.3 (100, $[M]^{+\bullet}$). Accurate mass ($[M]^{+\bullet}$, MALDI) calcd for $C_{44}H_{34}O_3^{+\bullet}$: 610.2502, found: 610.2505.

Compound 11c: The synthetic procedure was similar to that yielding compound **10**, using **6c** (56 mg, 0.091 mmol) as the starting material. The crude product was purified by flash column chromatography (hexane/EtOAc = 9/1) to afford compound **11c (**2.2 mg, 0.004 mmol, 4%) as a colorless solid. M.p.: 201–203 °C; R_f: 0.33 (hexane/EtOAc = 9/1); ^{1}H NMR (400 MHz, 1,1,2,2-tetrachloroethane-d_2, 60 °C): 7.52–7.37 (m, 4 H,

ArH), 7.36–7.24 (m, 4 H, ArH), 7.23–7.00 (m, 6 H, ArH), 6.97 (t, J = 7.2 Hz, 2 H, ArH), 6.92 (d, J = 7.8 Hz, 2 H, ArH), 6.82 (t, J = 7.4 Hz, 1 H, ArH), 6.06 (s, 1 H, ArH), 5.83 (d, J = 7.6 Hz, 1 H, ArH), 4.95 (s, 1 H, CH), 4.91 (s, 1 H, CH), 4.34 (s, 1 H, CH), 3.90 (s, 3 H, OCH_3), 3.90–3.68 (m, 6 H, OCH_3), 1.78 (s, 3 H, CH_3); ^{13}C NMR (176 MHz, 1,1,2,2-tetrachloroethane-d_2, 60 °C): 155.9, 146.2, 145.0, 144.6, 136.9, 135.1, 132.2, 131.12, 131.06, 131.0, 130.1, 129.8, 129.0, 128.6, 128.0, 126.9, 126.8, 126.1, 123.8, 122.9, 120.9, 120.3, 120.2, 111.4, 110.6, 99.4, 62.5, 60.3, 60.1, 59.4, 55.4, 55.2, 55.1, 28.0; MS (MALDI): m/z (%) 635.3 (100, $[M + Na]^+$). Accurate mass ($[M + Na]^+$, MALDI) calcd for $C_{44}H_{36}NaO_3^+$: 635.2562, found: 635.2559.

Compound 11d: The synthetic procedure was similar to that yielding compound **10**, using **6d** (134 mg, 0.219 mmol) as the starting material. The crude product was purified by flash column chromatography (hexane/EtOAc = 12/1) to afford compound **11d (**14 mg, 0.023 mmol, 11%) as a colorless solid. M.p.: 198–199 °C; R_f: 0.4 (hexane/EtOAc = 6/1); ^1H NMR (700 MHz, CD_2Cl_2): 7.49–7.45 (m, 2 H, ArH), 7.39 (d, J = 7.7 Hz, 1 H, ArH), 7.35 (d, J = 7.0 Hz, 1 H, ArH), 7.30–7.27 (m, 4 H, ArH), 7.25 (t, J = 8.4 Hz, 1 H, ArH), 7.22 (d, J =7.7 Hz, 1 H, ArH), 7.16 (s, 1 H, ArH), 7.13 (s, 1 H, ArH), 7.06 (t, J = 7.7 Hz, 1 H, ArH), 7.02 (d, J = 8.4 Hz, 1 H, ArH), 6.98 (d, J = 7.7 Hz, 1 H, ArH), 6.82 (d, J = 8.4 Hz, 1 H, ArH), 6.77–6.75 (m, 2 H, ArH), 6.72 (d, J = 7.7 Hz, 1 H, ArH), 6.16 (s, 1 H, ArH), 5.80 (d, J = 7.7 Hz, 1 H, ArH), 5.24 (s, 1 H, CH), 5.19 (s, 1 H, CH), 4.37 (s, 1 H, CH), 3.88 (s, 3 H, OCH_3), 3.83 (s, 3 H, OCH_3), 3.75 (s, 3 H, OCH_3), 1.8 (s, 3 H, CH_3); ^{13}C NMR (176 MHz, CD_2Cl_2): 160.8, 160.6, 160.1, 146.5, 146.0, 145.7, 144.9, 144.3, 144.14, 144.05, 143.9, 142.8, 140.2, 139.13, 139.08, 130.7, 130.4, 130.170, 130.167, 129.6, 127.7, 127.2, 126.8, 126.0, 124.8, 124.4, 124.2, 122.0, 121.7, 119.9, 115.33, 115.26, 113.24, 113.21, 113.15, 112.2, 62.91, 62.85, 62.7, 60.7, 55.8, 55.6, 55.5, 28.5; MS (EI): m/z (%) 612.3 (100, $[M]^{+\bullet}$),

597.3 (15, $[M - CH_3]^+$), 496.2 (8), 390.2 (9), 306.1 (10, $[M]^{2+}$). Accurate mass ($[M]^{+\bullet}$, EI): calcd for $C_{44}H_{36}O_3^{+\bullet}$: 612.2659, found: 612.2654.

Compound 11e: R_f: 0.51 (hexane/EtOAc = 7/1); ^{1}H NMR (400 MHz): 7.37–7.33 (m, 2 H, ArH), 6.86 (d, J = 8.7 Hz, 2 H, ArH), 6.77 (t, J = 7.6 Hz, 1 H, ArH), 6.09 (s, 1 H, ArH), 5.84 (d, J = 7.8 Hz, 1 H, ArH), 5.21 (s, 1 H, CH), 5.17 (s, 1 H, CH), 4.35 (s, 1 H, CH), 3.92 (s, 3 H, OCH_3), 3.89 (s, 3 H, OCH_3), 3.84 (s, 3 H, OCH_3), 1.79 (s, 3 H, CH_3)

Note: Some signals of compound **11e** were overlapped by those of compound **10,** making the full characterization of compound **11e** impossible.

Compound 16: The synthetic procedure was similar to that yielding compound **10**, using **6k** (80 mg, 0.100 mmol) as the starting material. The crude product was purified by flash column chromatography (hexane/EtOAc = 2/1) to afford compound **16 (**60 mg, 0.075 mmol, 75%) as a colorless solid. M.p.: 150–152 °C; R_f: 0.4 (hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz, 1,1,2,2-tetrachloroethane-d_2, 60 °C): 7.64 (d, J = 8 Hz, 1 H, ArH), 7.51 (d, J = 8 Hz, 1 H, ArH), 7.28 (d, J = 7.8 Hz, 1 H, ArH), 7.24 (d, J = 7.9 Hz, 1 H, ArH), 7.14–7.12 (m, 3 H, ArH), 6.86 (d, J = 8.5 Hz, 2 H, ArH), 6.68–6.64 (m, 2 H, ArH), 5.74–5.70 (m, 2 H, ArH), 5.04 (s, 1 H, CH), 5.01 (s, 1 H, CH), 4.13 (s, 1 H, CH), 4.05 (s, 6 H, OCH_3), 3.98 (s, 12 H, OCH_3), 3.86 (s, 3 H, OCH_3), 3.62 (s, 6 H, OCH_3), 1.81 (s, 3 H, CH_3); ^{13}C NMR (100 MHz, 1,1,2,2-tetrachloroethane-d_2, 60 °C): 153.5, 153.4, 152.2, 151.8, 151.5, 151.4, 147.9, 147.8, 145.4, 143.20, 143.18, 141.9, 141.5, 140.8, 134.6, 134.3, 134.0, 133.4, 129.8, 129.1, 128.7, 128.42, 128.38, 126.4, 126.14, 126.10, 125.6, 125.50, 125.46, 124.9, 109.4, 108.3, 108.2, 64.8, 64.6, 61.0, 60.8, 60.7, 60.6, 60.3, 59.1, 56.38, 56.36, 56.15, 27.65; MS (ESI): m/z (%) 813.3 (100, $[M + Na]^+$). Accurate mass ($[M + Na]^+$, ESI)

calcd for $C_{50}H_{46}NaO_9^+$: 813.3034, found: 813.3033.

Compound 17: The synthetic procedure was similar to that yielding compound **10**, using **6l** (50 mg, 0.063 mmol) as the starting material. The crude product was purified by flash column chromatography (hexane/EtOAc = 2/1) to afford compound **17 (**20 mg, 0.025 mmol, 40%) as a colorless solid. M.p.: 205–207 °C; R_f: 0.27 (hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz): 7.58 (d, *J* = 7.9 Hz, 1 H, ArH), 7.43 (d, *J* = 7.8 Hz, 1 H, ArH), 7.22 (d, *J* = 7.8 Hz, 1 H, ArH), 7.16 (d, *J* = 8 Hz, 1 H, ArH), 7.02 (s, 1 H, ArH), 6.76–6.74 (m, 4 H, ArH), 6.67–6.63 (m, 2 H, ArH), 5.71–5.65 (m, 2 H, ArH), 5.08 (s, 1 H, CH), 5.05 (s, 1 H, CH), 4.13 (s, 1 H, CH), 4.01 (s, 3 H, OCH_3), 4.00 (s, 3 H, OCH_3), 3.96 (s, 3 H, OCH_3), 3.95 (s, 3 H, OCH_3), 3.87 (s, 12 H, OCH_3), 3.81 (s, 3 H, OCH_3), 1.79 (s, 3 H, CH_3); ^{13}C NMR (100 MHz): 153.8, 153.7, 152.5, 151.9, 148.4, 148.3, 145.0, 144.9, 141.9, 140.1, 139.5, 138.8, 138.2, 138.1, 137.9, 137.8, 137.7, 134.4, 134.2, 130.1, 129.2, 128.9, 128.1, 127.4, 127.0, 126.9, 125.8, 125.6, 124.8, 108.8, 106.6, 64.4, 64.3, 61.3, 61.2, 61.1, 60.4, 59.6, 56.4, 56.2, 28.0; MS (ESI): *m*/*z* (%) 813.3 (100, $[M + Na]^+$). Accurate mass ($[M + Na]^+$, ESI) calcd for $C_{50}H_{46}NaO_9^+$: 813.3034, found: 813.3028.

Compound 18: Compound **16** (60 mg, 0.075 mmol) was dissolved in anhydrous CH_2Cl_2 (10 mL) and cooled to 0 °C. 2,3-Dichloro-5,6-dicyano-*p*-benzoquinone (34 mg, 0.152 mmol) was added in one portion and the mixture was stirred for 5 min. Trifluoromethanesulfonic acid (0.2 mL) was added and the resulting mixture was stirred for 1 h. The reaction mixture was quenched and washed with saturated sodium carbonate solution (20 mL), concentrated under reduced pressure and the residue was purified by flash column chromatography (hexane/EtOAc = 2/1) to afford compound **18 (**34 mg, 0.043 mmol, 57%) as a colorless solid. M.p.: 253 °C (dec.); R_f: 0.4

(hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz): 7.43 (d, *J* = 8 Hz, 1 H, ArH), 7.391 (A of AB system, d, *J* = 8.5 Hz, 1 H, ArH), 7.386 (B of AB system, d, *J* = 8.3 Hz, 1 H, ArH), 7.33–7.29 (m, 2 H, ArH), 7.24 (d, *J* = 8 Hz, 1 H, ArH), 7.04 (s, 1 H, ArH), 7.02 (s, 2 H, ArH), 4.443 (s, 1 H, CH), 4.438 (s, 1 H, CH), 4.422 (s, 1 H, CH), 4.01 (s, 12 H, OCH_3), 3.98 (s, 6 H, OCH_3), 3.89 (s, 3 H, OCH_3), 3.87 (s, 3 H, OCH_3), 3.86 (s, 3 H, OCH_3), 1.84 (s, 3 H, CH_3); ^{13}C NMR (100 MHz): 152.5, 152.42, 152.39, 151.93, 151.87, 151.8, 145.3, 145.1, 145.0, 144.92, 144.85, 144.6, 141.9, 141.8, 141.7, 134.41, 134.37, 134.13, 134.09, 134.06, 133.9, 130.4, 129.5, 129.4, 129.2, 129.1, 128.8, 127.8, 126.9, 124.8, 124.6, 124.5, 108.4, 108.2, 62.7, 62.63, 62.55, 61.4, 61.3, 61.2, 58.9, 56.2, 26.1; MS (ESI): *m*/*z* (%) 809.3 (100, $[M + Na]^+$). Accurate mass ($[M + Na]^+$, ESI) calcd for $C_{50}H_{42}NaO_9^+$: 809.2721, found: 809.2726.

2. X-ray Crystallography

Crystal data were collected on a Bruker D8 venture diffractometer or Bruker Kappa ApexII Duo diffractometer with Mo-Kα radiation (λ = 0.71073 Å). The crystal structures were solved by direct methods using SHELXS-97[1] and all the non-hydrogen atoms were anisotropically refined by full-matrix least-squares on F^2 using the SHELXL-97 program.[2] The highly disordered co-crystallized solvent molecules were removed by the standard SQUEEZE[3] protocol incorporated in PLATON.[4]

X-ray crystal structure of compound 7a

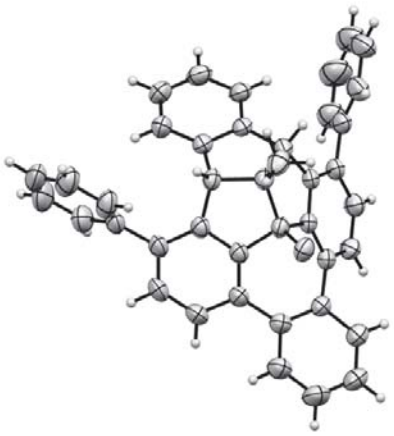


Figure S1. X-ray crystal structure of compound **7a**.

A single crystal of **7a** was obtained from a chloroform solution by slow evaporation.

X-ray crystal data for **7a**: $C_{41}H_{27}O$; M = 535.62; monoclinic; a = 23.6823(14), b = 14.1831(8), c = 19.4393(10) Å; β = 101.9212(17); V = 6388.6(6) Å^3 ; space group $C2/c$; Z = 8; ρ_{calcd} = 1.114 Mg m^{-3} ; T = 296(2) K; λ (MoKα) = 1.54178 Å; 63194 reflections collected; 5781 independent reflections; R_{int} = 0.0613; observed data with I

$\geq 2\sigma(I) = 4070$; $R_1 = 0.0864$, $wR_2 = 0.1598$ [$I \geq 2\sigma(I)$]. CCDC-1478751 contains the supplementary crystallographic data for **7a**.

3. References

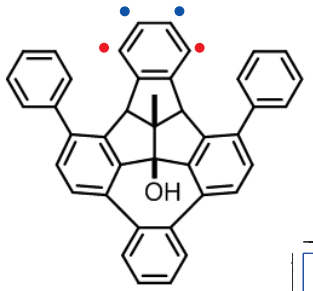
(1) G. M. Sheldrick, *SHELXS-97, Program for Crystal Structure Solution*, University of Göttingen, Göttingen, Germany, 1997.

(2) G. M. Sheldrick, *SHELXL-97, Program for the Refinement of Crystal Structures from Diffraction Data*, University of Göttingen, Göttingen, Germany, 1997.

(3) P. A. van der Sluis, A. L. Spek, *Acta Crystallogr. A* **1990**, *A46*, 194.

(4) A. L. Spek, *PLATON, A Multipurpose Crystallographic Tool*, Utrecht University, Utrecht, The Netherlands, 2000.

4. 2D COSY Spectra of 7a, 7b, 10, 16 and 17



7a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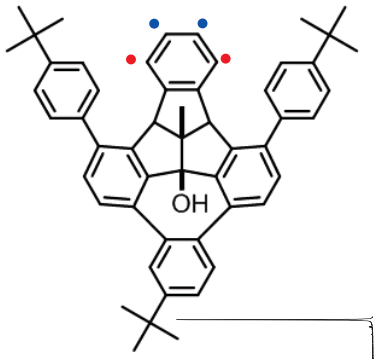
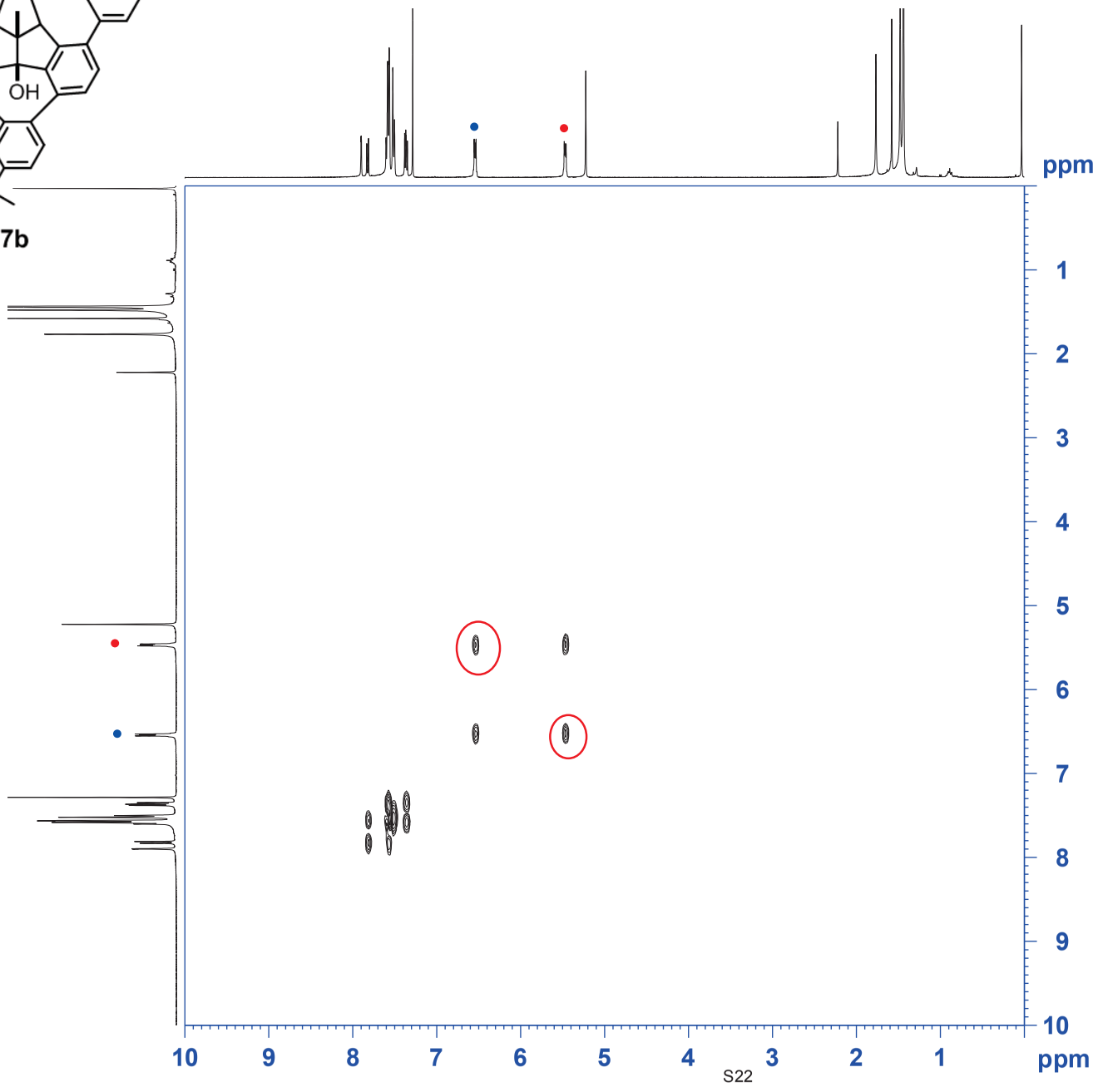


7b

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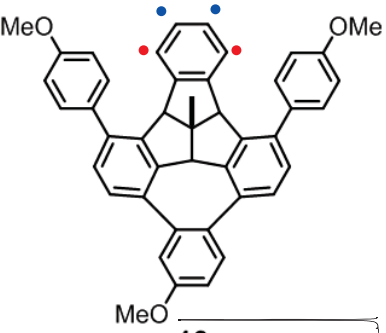
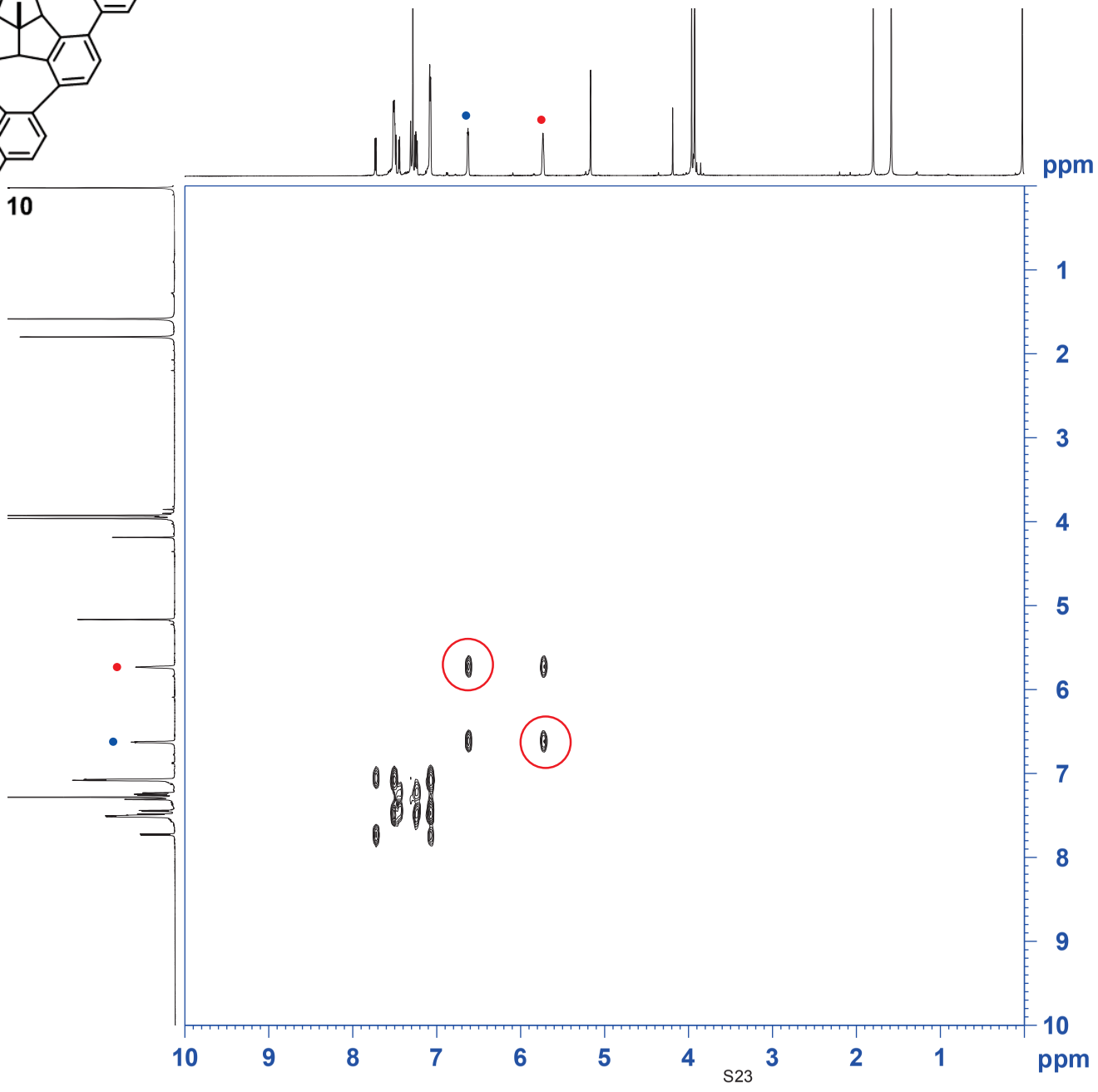
LB        0 Hz
GB        0
PC                  1.40

F1 - Processing parameters
SI                  1024
MC2                   QF
SF           400.2300000 MHz
WDW                 SINE
SSB       0
LB        0 Hz
GB        0
```



10




```
Current Data Parameters
NAME      Eric_9a_4_OMe_data
EXPNO                  222
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160126
Time              17.02
INSTRUM           spect
PROBHD   5 mm CPTCI 1H-
PULPROG      cosygpmfqf
TD                2048
SOLVENT           CDCl3
NS                   4
DS                  16
SWH           14097.744 Hz
FIDRES         6.883664 Hz
AQ            0.0726357 sec
RG                 203
DW                35.467 usec
DE                10.00 usec
TE                298.0 K
D0           0.00000300 sec
D1           2.00000000 sec
D13          0.00000400 sec
D16          0.00020000 sec
IN0          0.00007140 sec

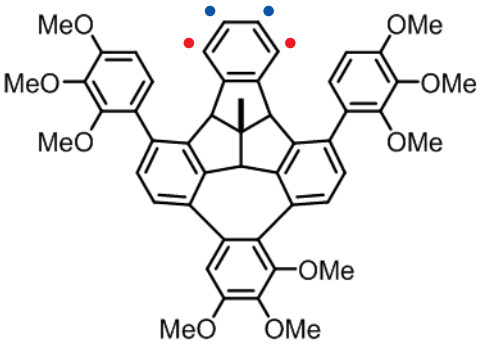
======== CHANNEL f1 ========
SFO1        700.1642010 MHz
NUC1                1H
P1                 8.45 usec
PLW1         7.00000000 W

====== GRADIENT CHANNEL =====
GPNAM[1]     SMSQ10.100
GPNAM[2]     SMSQ10.100
GPNAM[3]     SMSQ10.100
GPZ1              16.00 %
GPZ2              12.00 %
GPZ3              40.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                 128
SFO1          700.1642 MHz
FIDRES       109.418770 Hz
SW            20.003 ppm
FnMODE              QF

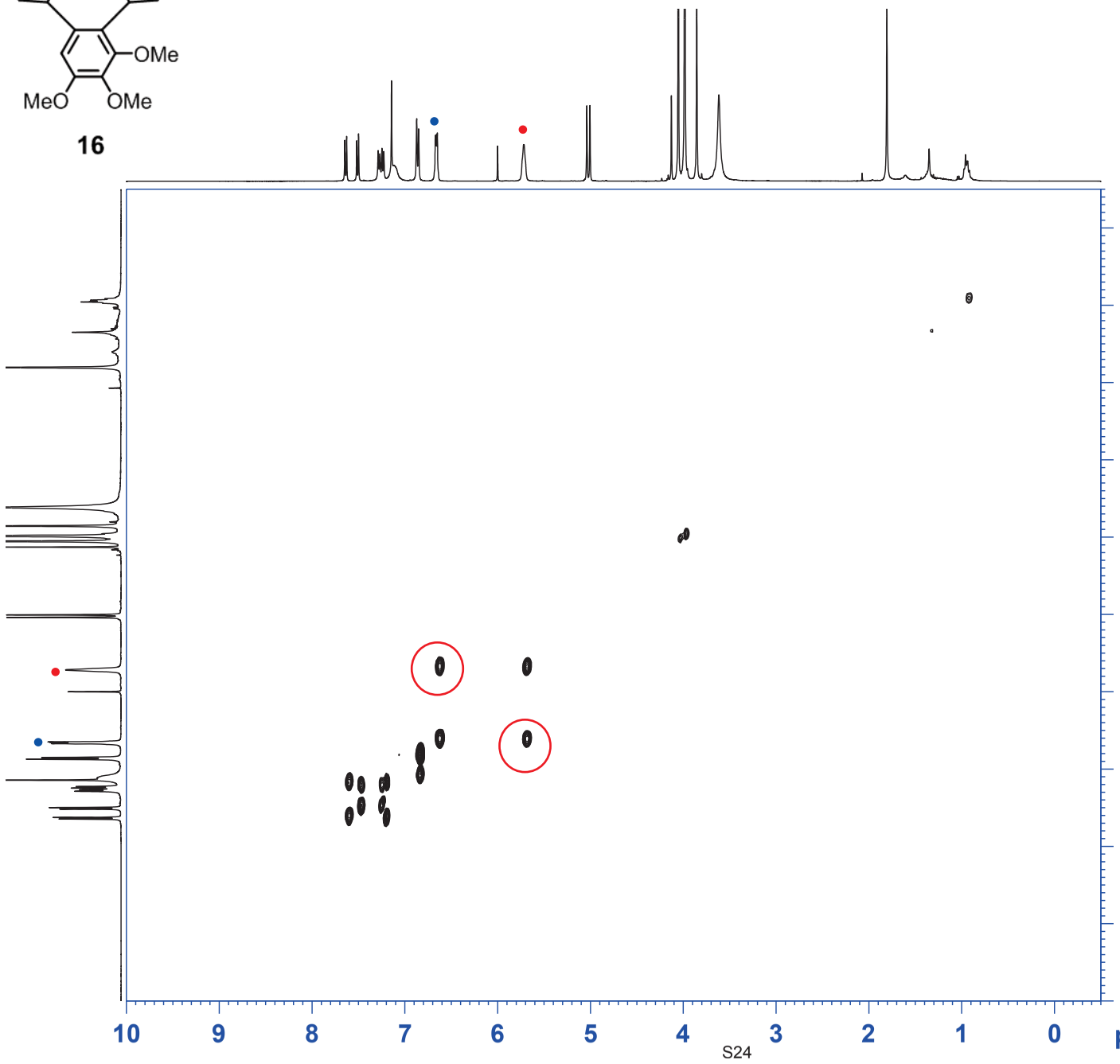
F2 - Processing parameters
SI                1024
SF          700.1600000 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
PC                1.40

F1 - Processing parameters
SI                1024
MC2                 QF
SF          700.1600000 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
```



16

60 °C



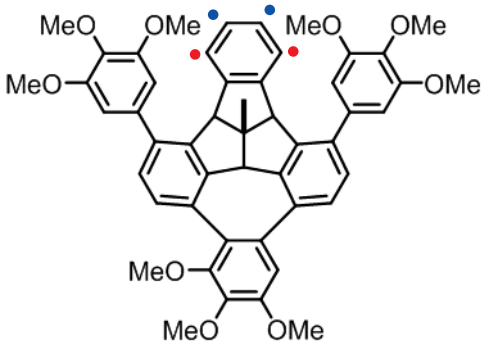

```
Current Data Parameters
NAME      Eric_9a_2_3_4_OMe_mono_data
EXPNO                 333
PROCNO                  1

F2 - Acquisition Parameters
Date_           20170126
Time              15.37
INSTRUM           spect
PROBHD   Z824601_0021 (
PULPROG      cosygpmfqf
TD                2048
SOLVENT           CDCl3
NS                   4
DS                  16
SWH            8012.820 Hz
FIDRES         3.912510 Hz
AQ             0.1277952 sec
RG                 203
DW                62.400 usec
DE                 6.50 usec
TE                333.2 K
D0           0.00000300 sec
D1           2.00000000 sec
d13          0.00000400 sec
D16          0.00020000 sec
in0       0 sec
ST1CNT               0
d0orig       0.00000300 sec
ph1loop              0
t1loop               0
SFO1        400.1324008 MHz
NUC1               1H
P1                15.00 usec
PLW1           8.31000042 W
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPZ1              16.00 %
GPZ2              12.00 %
GPZ3              40.00 %
P16             1000.00 usec

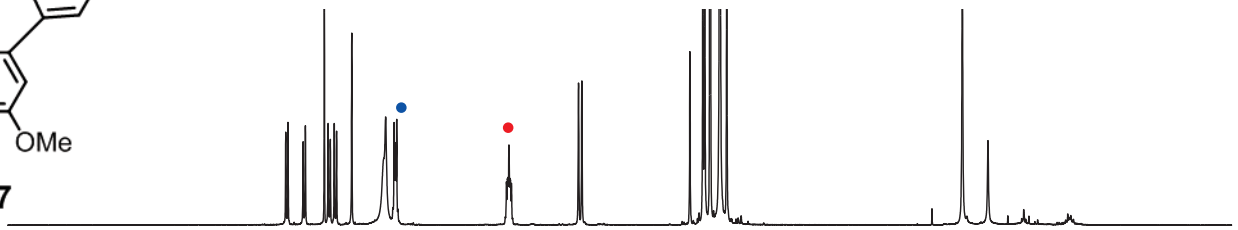
F1 - Acquisition parameters
TD                 128
SFO1          400.1324 MHz
FIDRES        62.500000 Hz
SW               19.993 ppm
FnMODE              QF

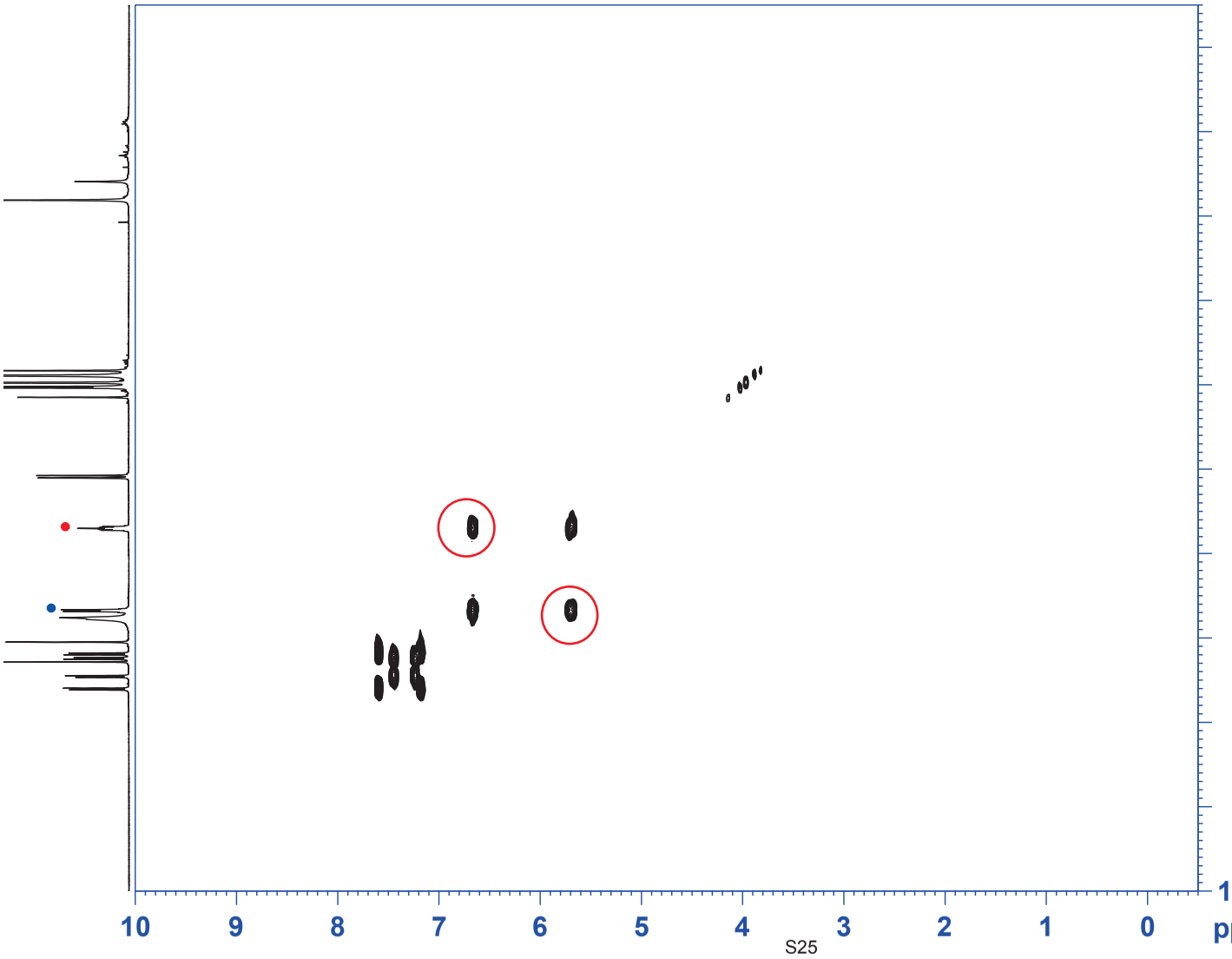
F2 - Processing parameters
SI                1024
SF          400.1305184 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
PC                1.40

F1 - Processing parameters
SI                1024
MC2                 QF
SF          400.1305228 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
```



17






```
Current Data Parameters
NAME      Eric_8a_3_4_5_OMe_mono_data
EXPNO                 222
PROCNO                  1

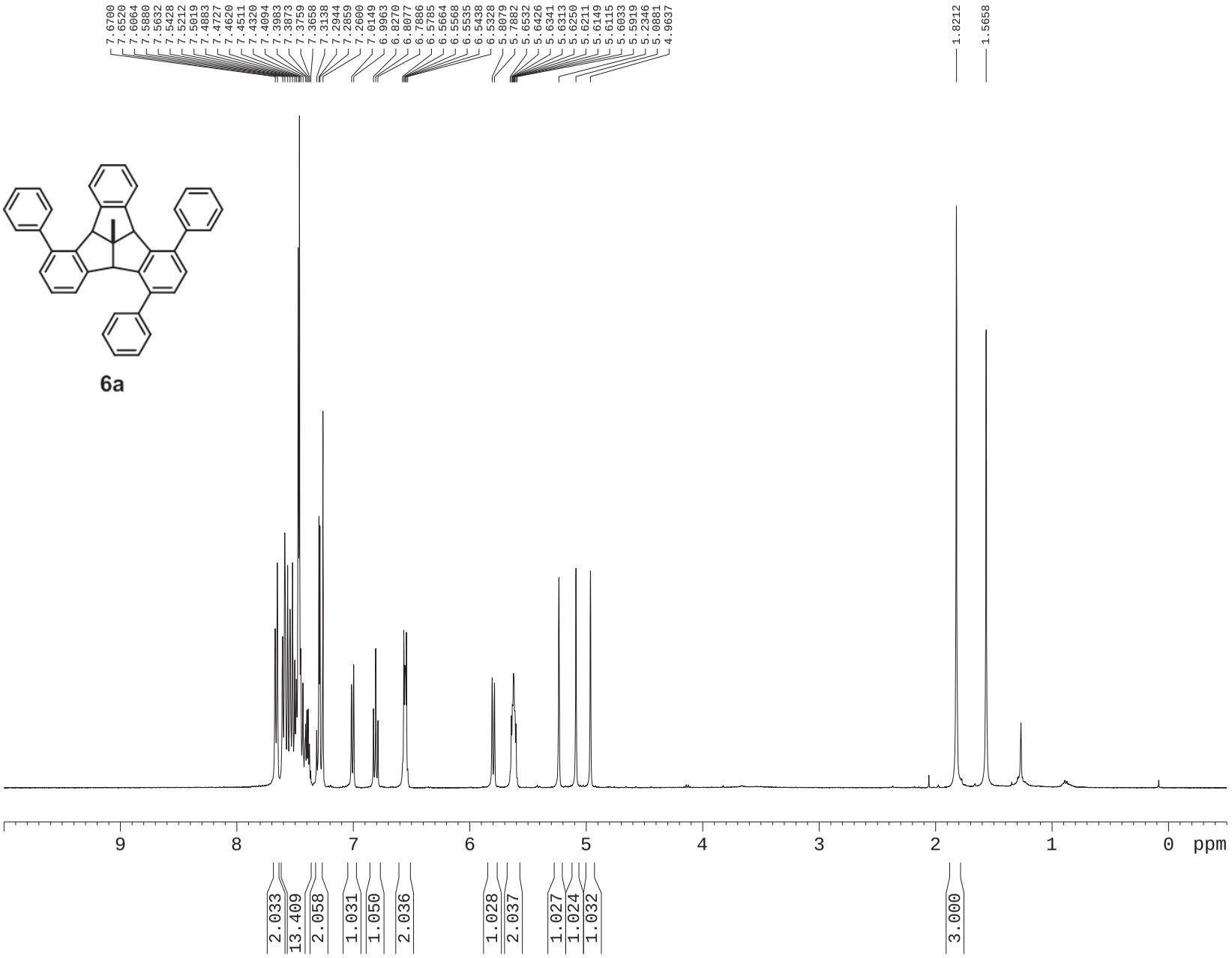
F2 - Acquisition Parameters
Date_           20170118
Time              18.17
INSTRUM           spect
PROBHD   Z824601_0021 (
PULPROG      cosygpmfqf
TD                2048
SOLVENT           CDCl3
NS                   4
DS                  16
SWH            8012.820 Hz
FIDRES         3.912510 Hz
AQ             0.1277952 sec
RG                 203
DW                62.400 usec
DE                 6.50 usec
TE                295.1 K
D0           0.00000300 sec
D1           2.00000000 sec
d13          0.00000400 sec
D16          0.00020000 sec
in0       0 sec
ST1CNT               0
d0orig       0.00000300 sec
ph1loop              0
t1loop               0
SFO1         400.1324008 MHz
NUC1                1H
P1                15.00 usec
PLW1          8.31000042 W
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPZ1              16.00 %
GPZ2              12.00 %
GPZ3              40.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                 128
SFO1           400.1324 MHz
FIDRES         62.500000 Hz
SW                19.993 ppm
FnMODE              QF

F2 - Processing parameters
SI                1024
SF           400.1300000 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
PC                1.40

F1 - Processing parameters
SI                1024
MC2                 QF
SF           400.1300000 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
```

5. List of NMR and mass spectra



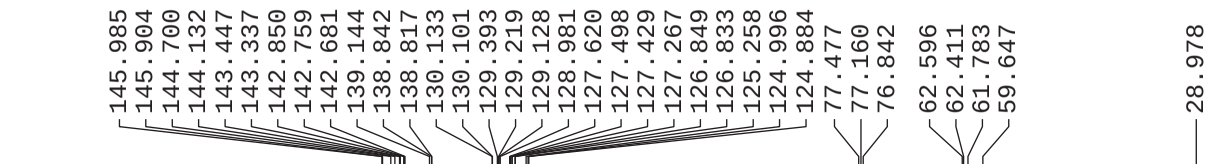
AV400Q NMR

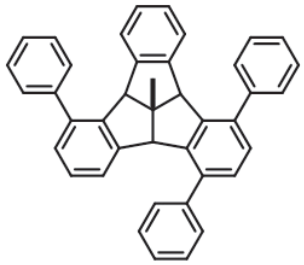
BRUKER

Current Data Parameters
NAME Eric_8a_un_data
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160203
Time 10.42 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 17
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447233 sec
RG 114
DW 62.400 usec
DE 6.50 usec
TE 294.9 K
D1 1.00000000 sec
TD0 1
SFO1 400.2324714 MHz
NUC1 1H
P1 12.80 usec
PLW1 13.56000042 W

F2 - Processing parameters
SI 65536
SF 400.2300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





6a


```
Current Data Parameters
NAME      Eric_8a_un_data
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160112
Time               13.19 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   162
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                296.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          100.6228298 MHz
NUC1                 13C
P1                 9.50 usec
PLW1          41.25000000 W
SFO2          400.1316005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2           8.31000042 W
PLW12          0.23083000 W
PLW13          0.11611000 W

F2 - Processing parameters
SI                 32768
SF            100.6127581 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

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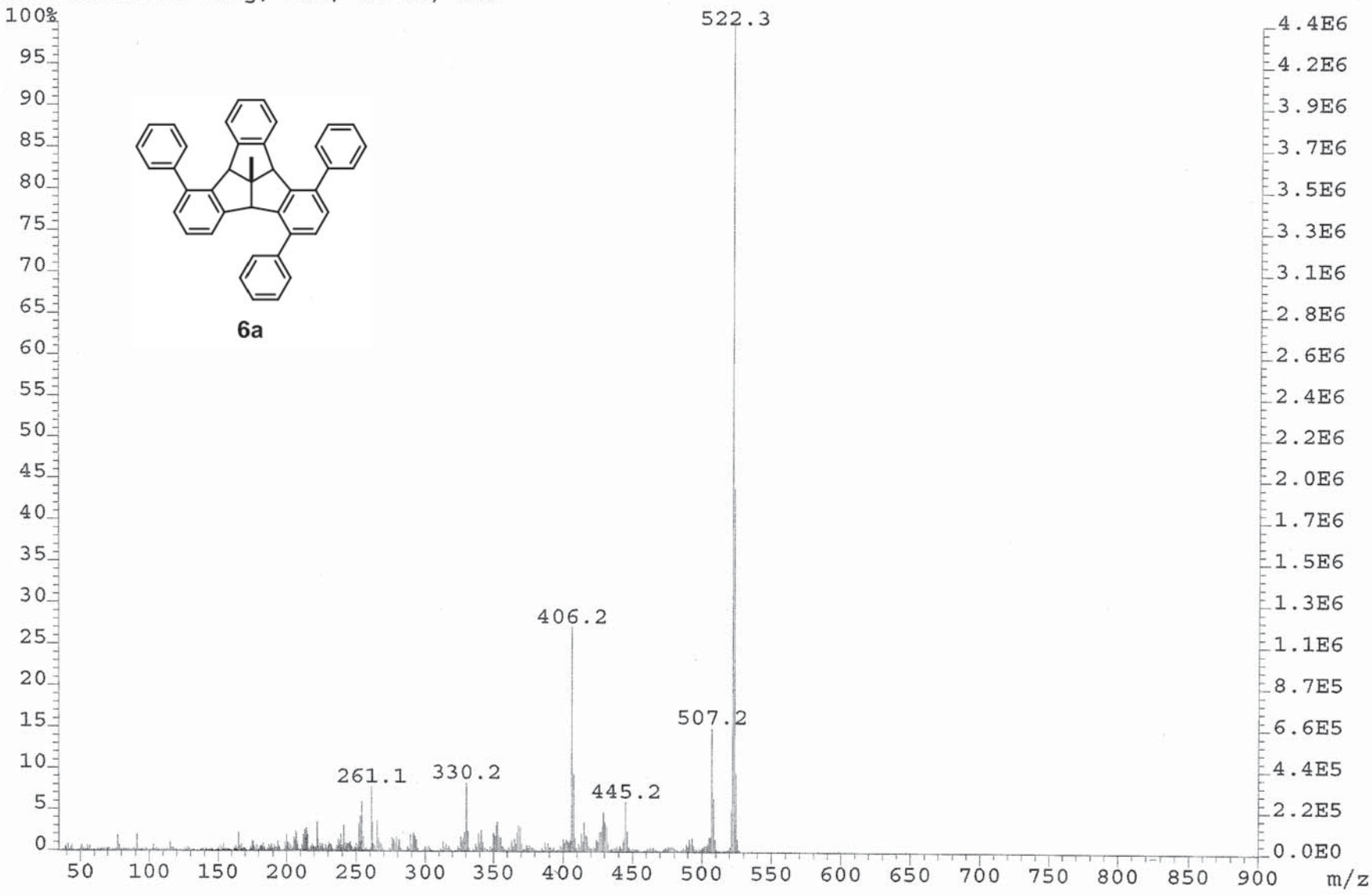
File Text:Er. Wang, OC1, Er-07, HWP



6a

Bruker 9.4T FTICR MS Analysis Report

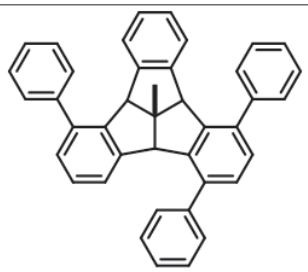
Faculty of Science, The Chinese University of Hong Kong

Analysis Info

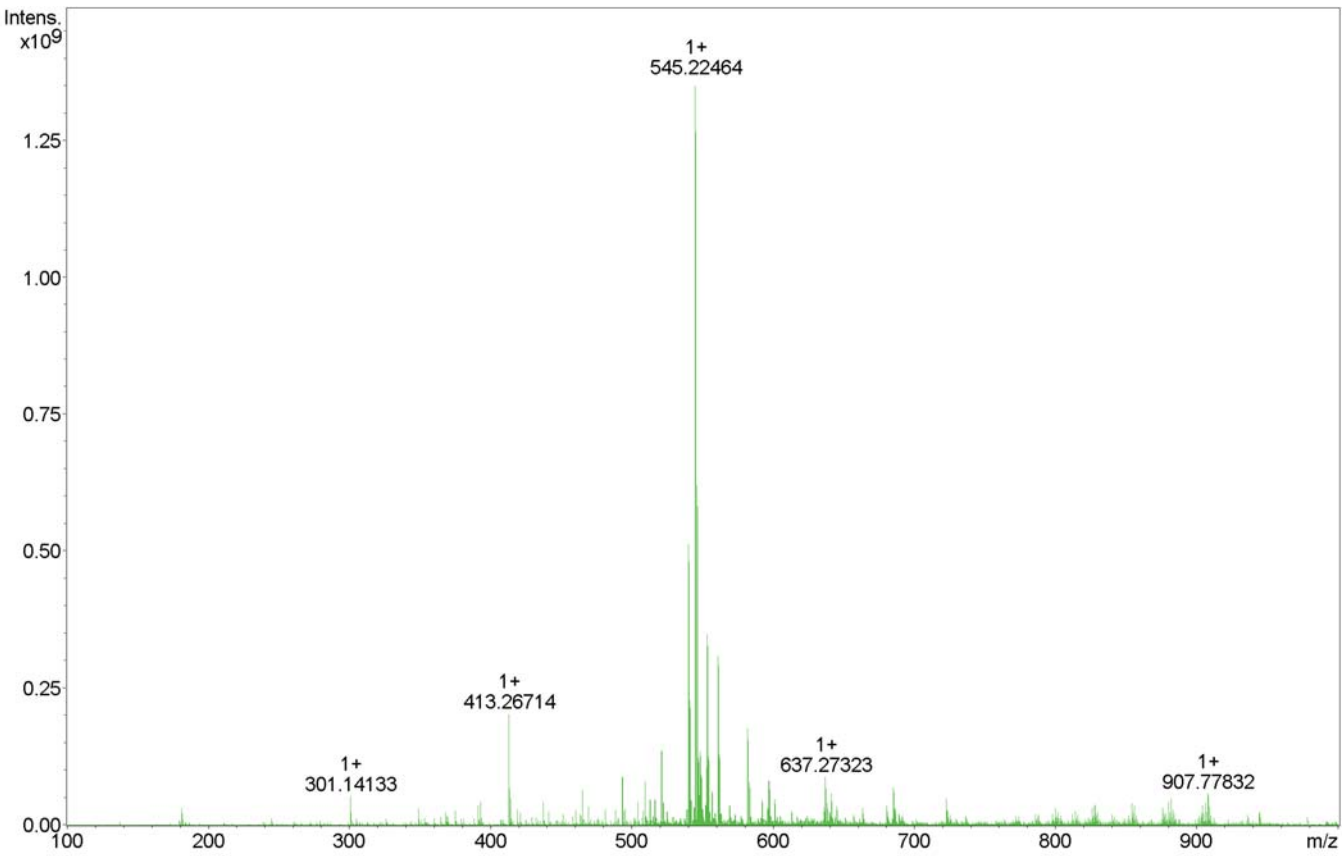
Sample Name :	Eric_8a_un	Reference No. :	xhfc042
Applicant Name :	Ip Ho Wang	Analysis Date :	4/2/2016 11:18:37
Analysis Path :	xhfc042_000001.d		
Instrument :	solariX	Polarity	Positive
Method	4_17_mass_range_pos_7T	Acquired Scans	11
Comment :	4.4kV, 120ml/hr, 1.0 nebulizer gas		

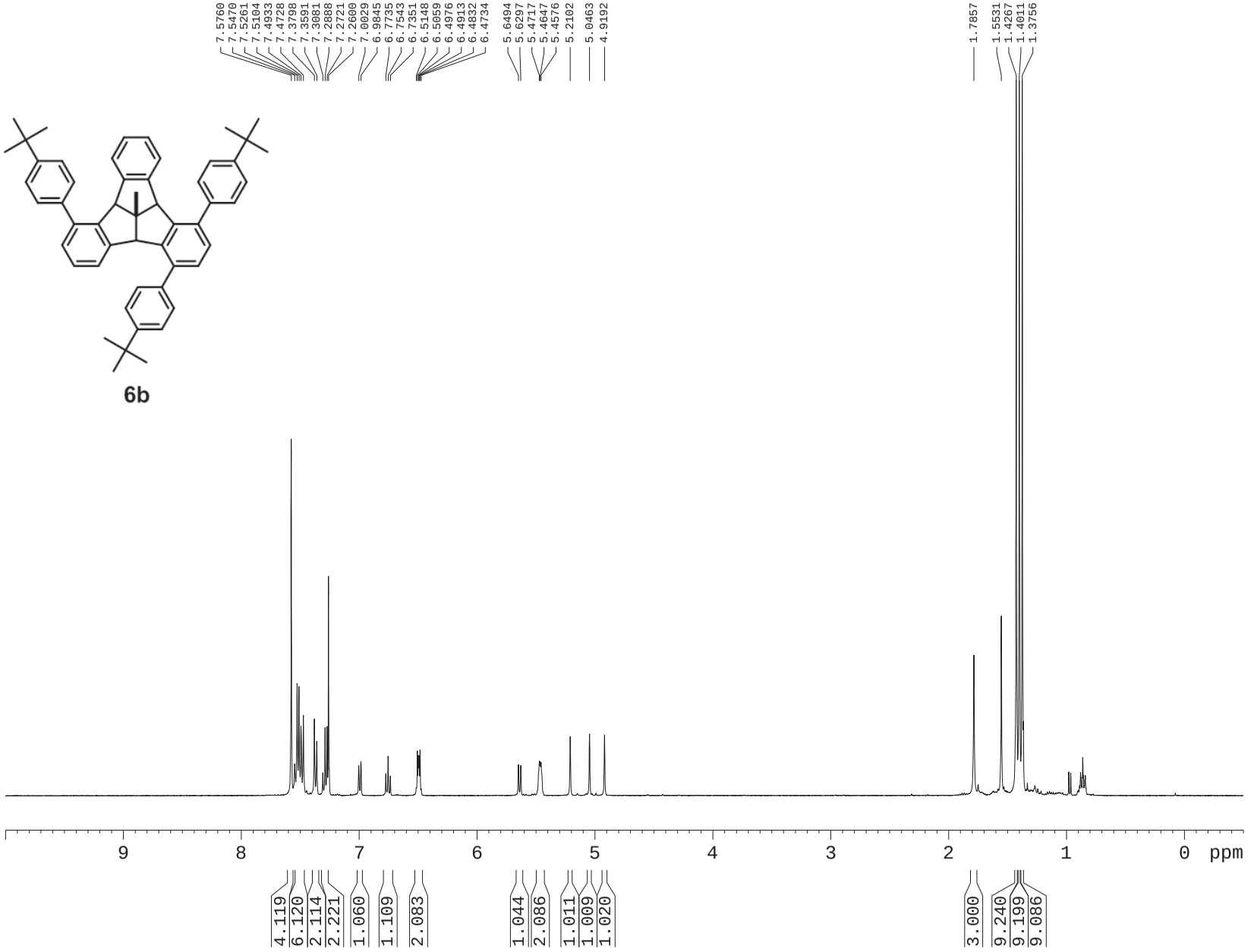
Accurate Mass Measurement

Molecular formula :	C41H30
Abundant Isotopic (theoretical) [M+Na]+ :	545.223972
Monoisotopic (theoretical) [M+Na]+ :	545.223972
(experimental) [M+Na]+ :	545.22464
error (ppm) :	1.2



6a



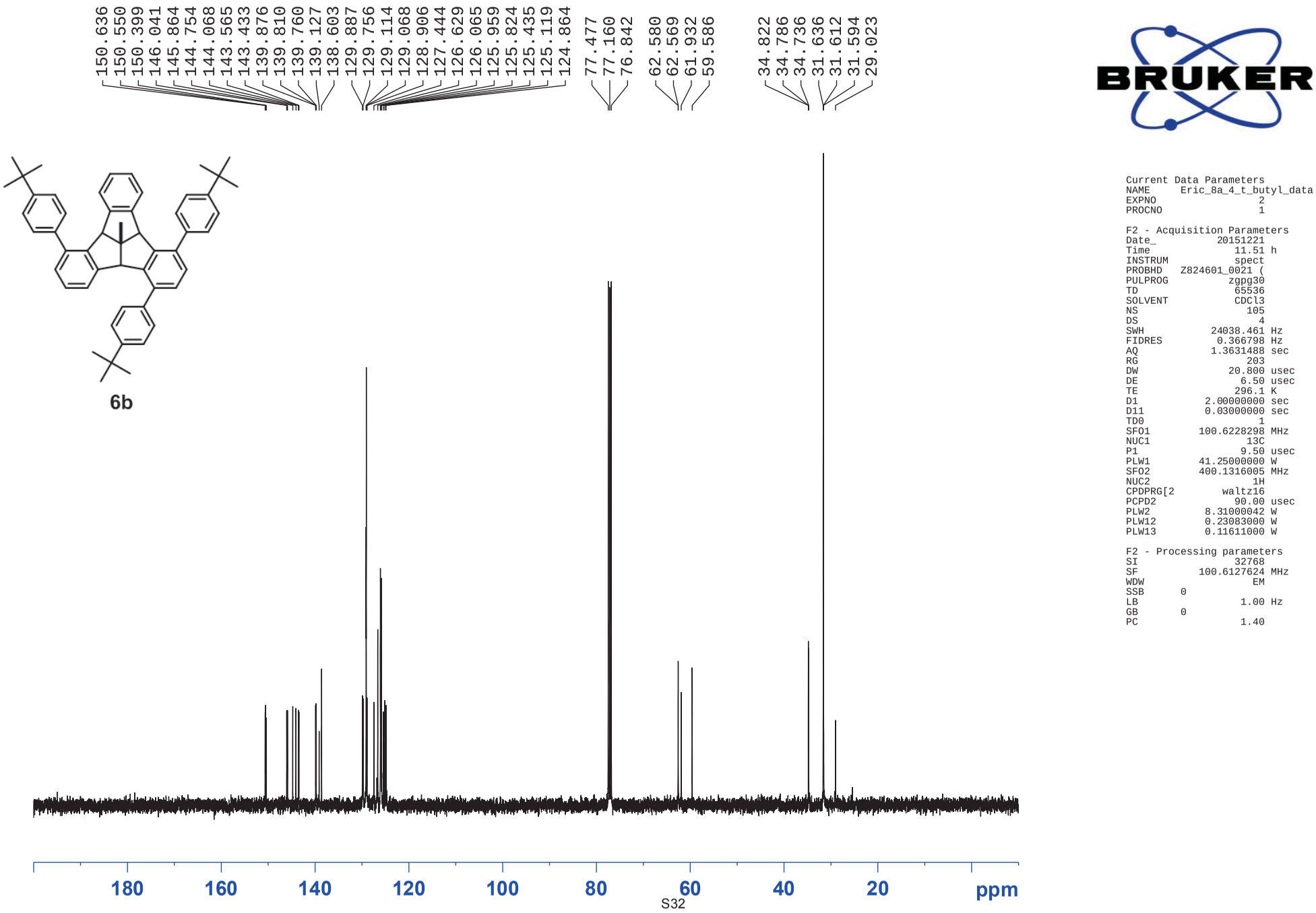


AV400Q NMR

Current Data Parameters
NAME Eric_8a_4_t_butyl_data
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151221
Time 11.33 h
INSTRUM spect
PROBHD Z824601_0021 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 28
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447233 sec
RG 203
DW 62.400 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.31000042 W

F2 - Processing parameters
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




```
Current Data Parameters
NAME      Eric_8a_4_t_butyl_data
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151221
Time              11.51 h
INSTRUM           spect
PROBHD   Z824601_0021 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  105
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                  203
DW               20.800 usec
DE                6.50 usec
TE               296.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                 9.50 usec
PLW1        41.25000000 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         8.31000042 W
PLW12        0.23083000 W
PLW13        0.11611000 W

F2 - Processing parameters
SI                32768
SF          100.6127624 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

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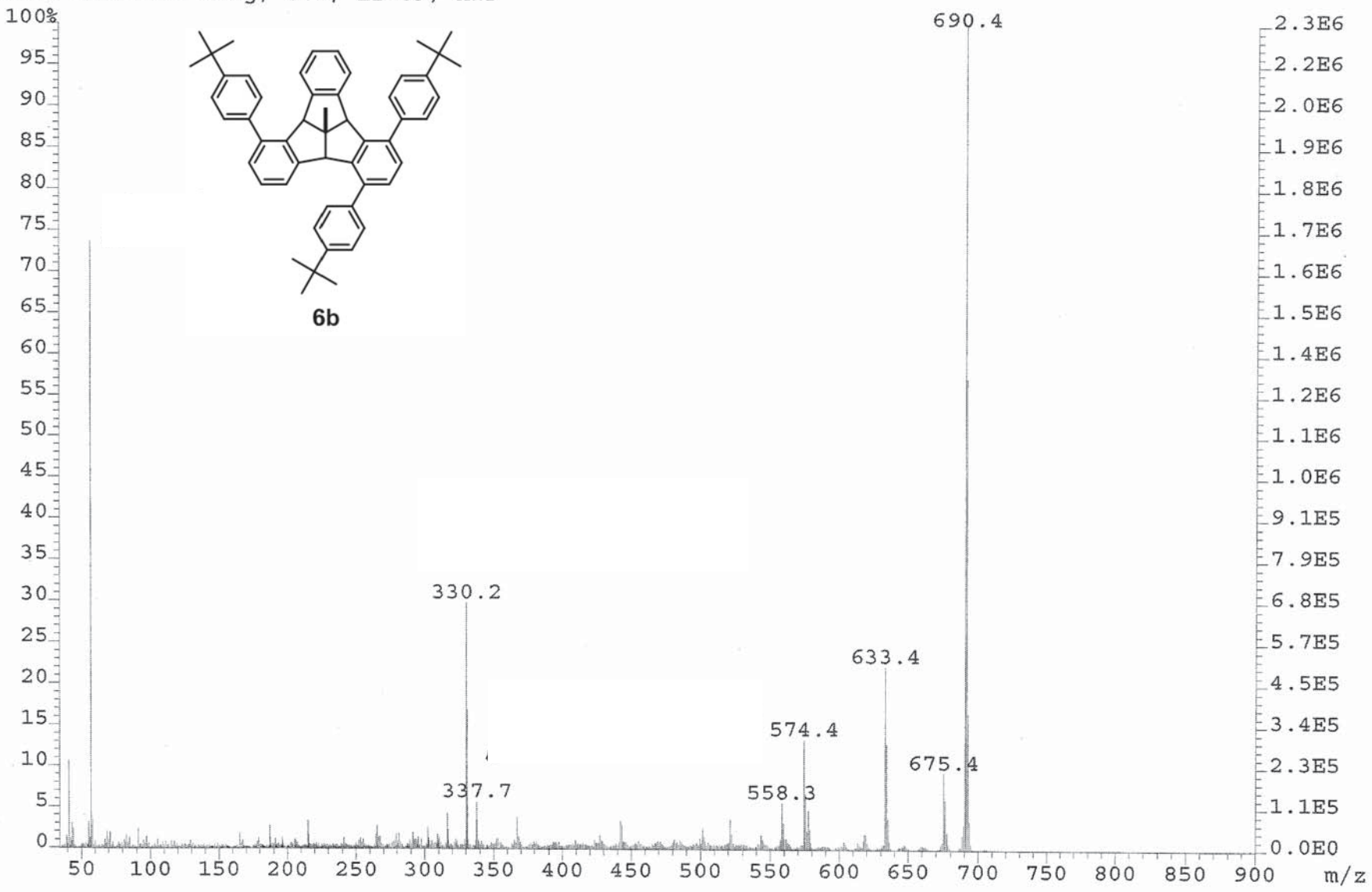
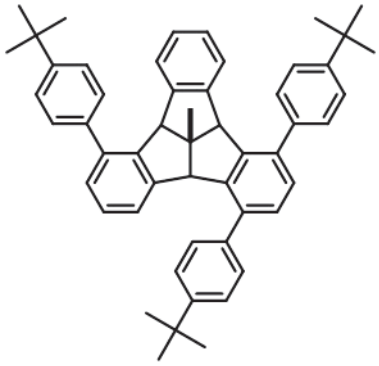
File Text:Er. Wang, OC1, Er-09, HWP



9.4T FTICR MS Analysis Report

Faculty of Science, The Chinese University of Hong Kong

Sample No.	Eric02
Reference No.	CHF0024
Applicant name	Ip Ho Wang
Analysis Path	\\192.168.0.1\Data\Service\MSonly\20160509\Eric02_000004.d
Instrument	solariX
Polarity	Positive
Acquired Scans	4
Operator	Winnie
Analysis Date	5/9/2016 3:25:05 PM

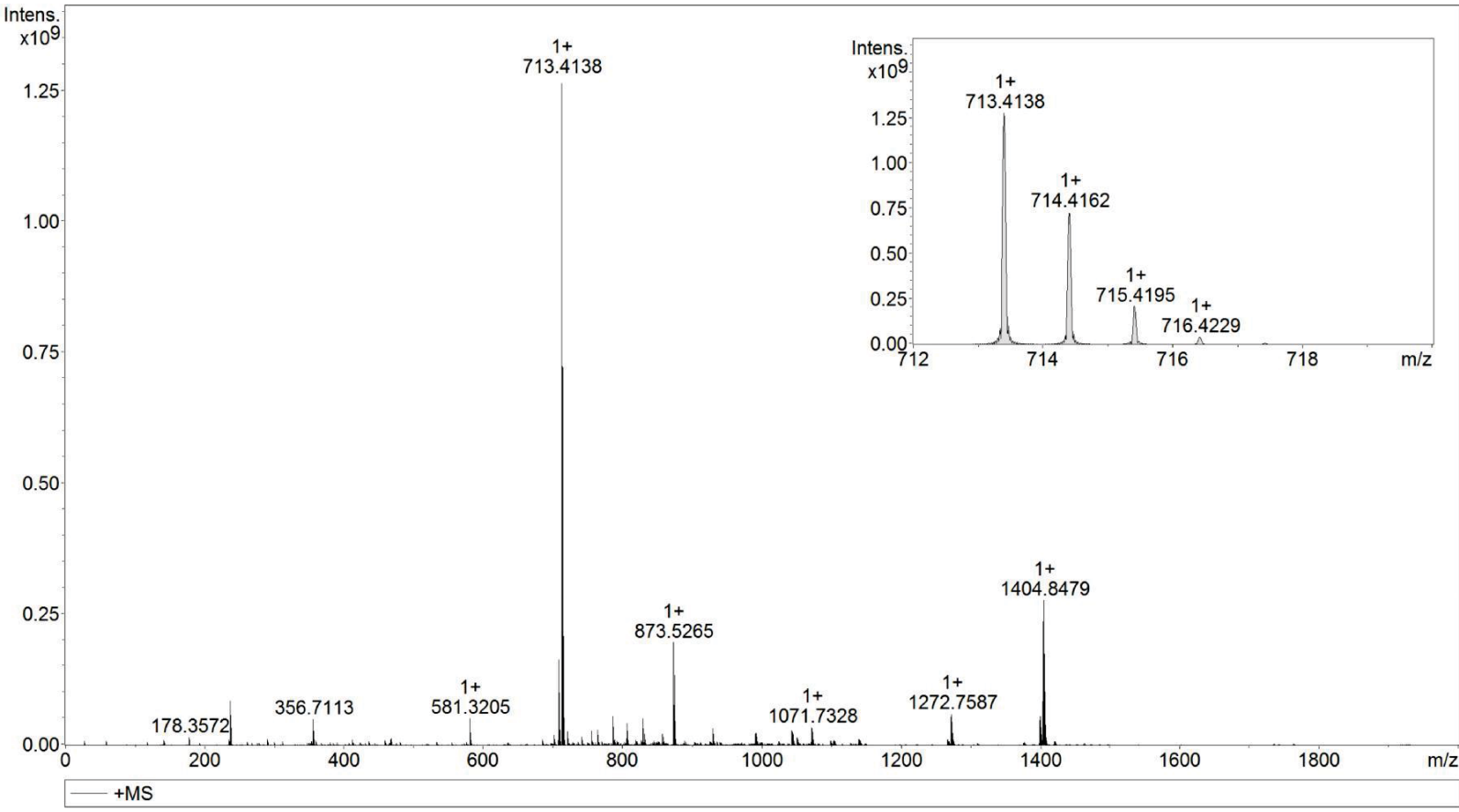


6b

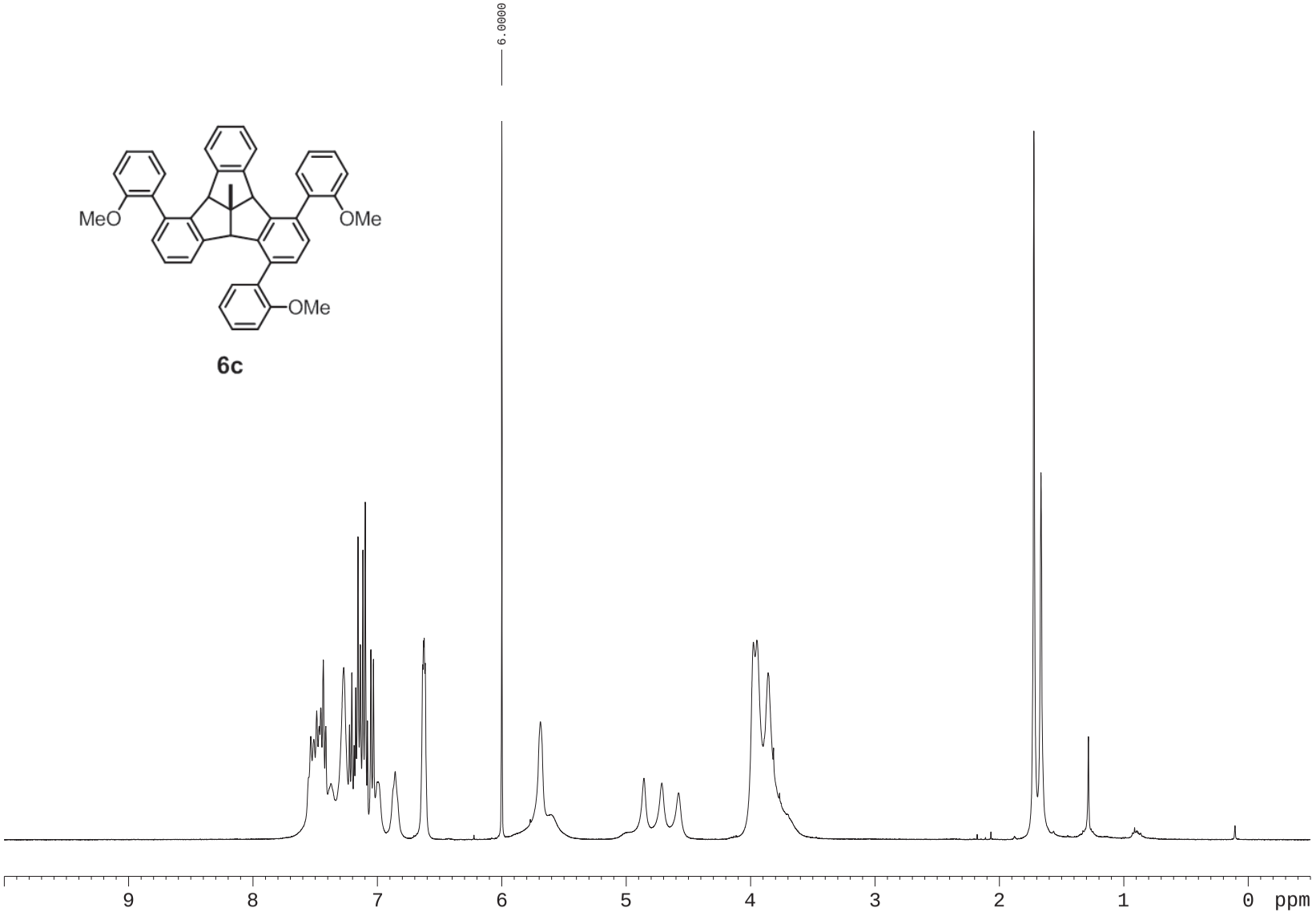
Molecular Formula: C53H54

Abundant Isotopic (theoretical)[M+Na]+ : 713.4118

Monoisotopic (theoretical)[M+Na]+ : 713.4118 (experimental)[M+Na]+: 713.4138 error(ppm): 2.80



22 °C



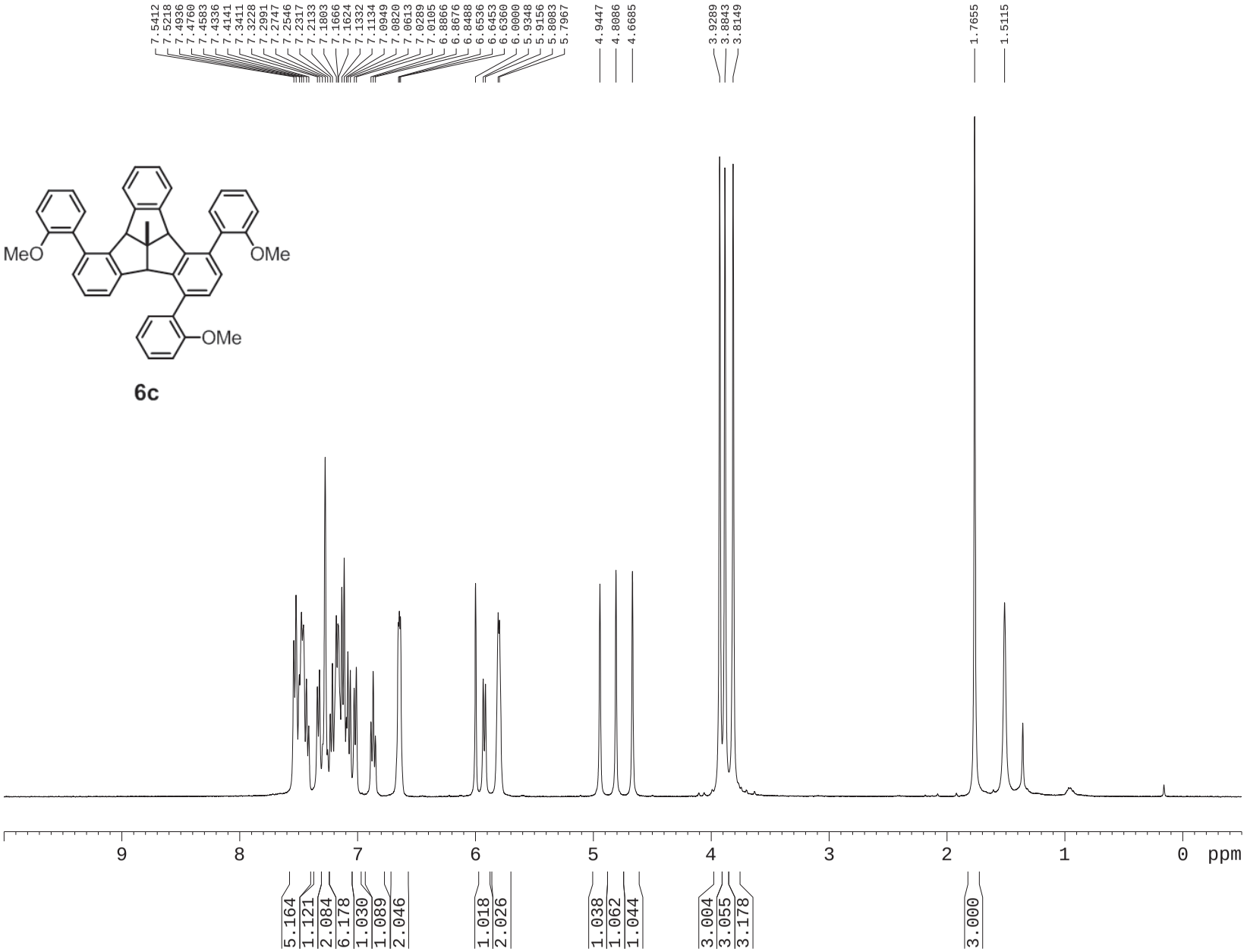
AV400Q NMR

```
Current Data Parameters
NAME      Eric_8a_2_OMe_rb
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_              20160418
Time                  9.07 h
INSTRUM               spect
PROBHD   Z824601_0021 (
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       17
DS                        2
SWH               8012.820 Hz
FIDRES               0.244532 Hz
AQ                2.0447233 sec
RG                      181
DW                   62.400 usec
DE                    6.50 usec
TE                   294.9 K
D1                1.00000000 sec
TD0                       1
SFO1            400.1324708 MHz
NUC1                     1H
P1                    15.00 usec
PLW1             8.31000042 W

F2 - Processing parameters
SI                    65536
SF              400.1305146 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```

60 °C



AV400Q NMR

BRUKER

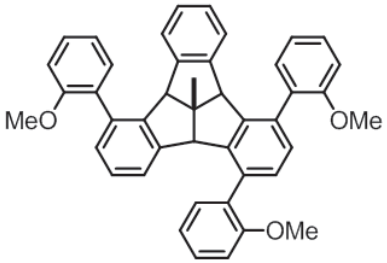
```
Current Data Parameters
NAME      Eric_8a_2_OMe_rb
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20160415
Time                16.10 h
INSTRUM              spect
PROBHD   Z824601_0021 (
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      21
DS                       2
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ               2.0447233 sec
RG                     203
DW                62.400 usec
DE                 6.50 usec
TE                 334.5 K
D1              1.00000000 sec
TD0                      1
SFO1           400.1324708 MHz
NUC1                   1H
P1                  15.00 usec
PLW1            8.31000042 W

F2 - Processing parameters
SI                   65536
SF             400.1305124 MHz
WDW                     EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

60 °C

156.542
146.274
145.659
145.282
144.896
135.266
134.826
131.773
131.583
131.412
131.333
129.746
129.127
128.817
128.746
128.664
126.572
126.127
126.061
125.219
125.014
124.629
120.976
120.906
120.752
111.762
111.538
74.055
73.780
73.505
63.380
63.081
62.511
59.411
55.483
55.419
55.279
28.182



6c


```
Current Data Parameters
NAME      Eric_8a_2_OMe_rb
EXPNO                    2
PROCNO                   1

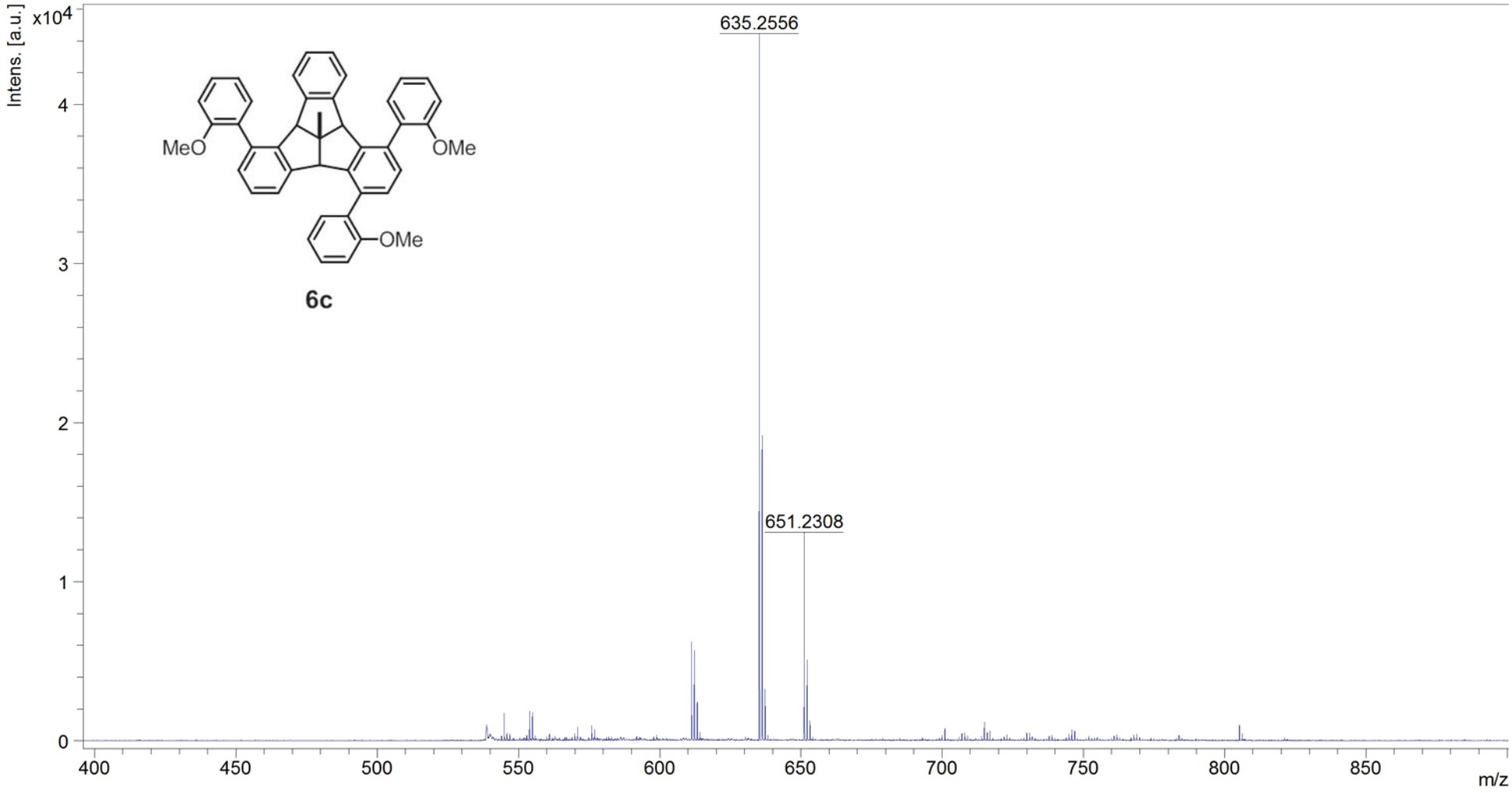
F2 - Acquisition Parameters
Date_           20160415
Time              16.15 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    551
DS                      4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                    203
DW                20.800 usec
DE                 6.50 usec
TE                333.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1
SFO1          100.6228298 MHz
NUC1                  13C
P1                  9.50 usec
PLW1          41.25000000 W
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2           8.31000042 W
PLW12          0.23083000 W
PLW13          0.11611000 W

F2 - Processing parameters
SI                  32768
SF            100.6129235 MHz
WDW                    EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

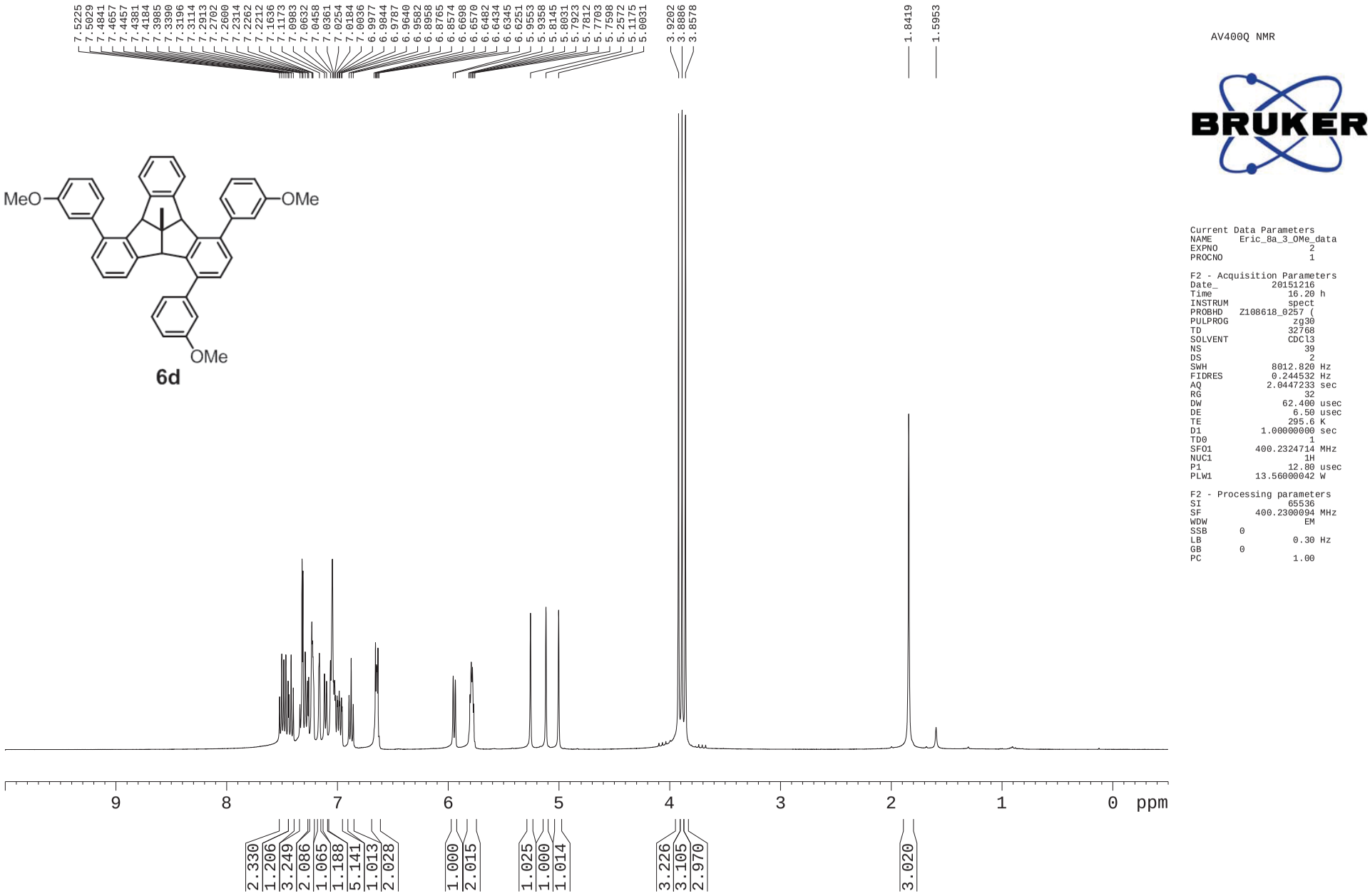
G:\MS Angew\9.Eric_8a_2_OMe(C9)\0_C9\1\1SRef

Comment 1

Comment 2



Bruker Daltonics flexAnalysis printed: 5/11/2016 6:58:20 PM



AV400Q NMR

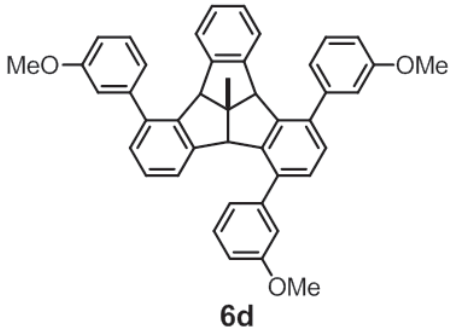
BRUKER

Current Data Parameters
NAME Eric_8a_3_OMe_data
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151216
Time 16.20 h
INSTRUM spect
PROBHD Z108618_0257 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 39
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447233 sec
RG 32
DW 62.400 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
SFO1 400.2324714 MHz
NUC1 1H
P1 12.80 usec
PLW1 13.56000042 W

F2 - Processing parameters
SI 65536
SF 400.2300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

160.297
160.210
160.086
145.981
145.882
144.669
144.143
144.108
144.080
143.991
143.393
143.270
138.946
138.763
138.744
130.249
130.157
130.008
129.994
129.938
129.055
127.618
126.887
125.404
125.137
121.882
121.845
115.156
115.083
112.906
112.654
77.477
77.160
76.842
62.620
62.464
61.859
59.603
55.532
55.521
55.482
28.970



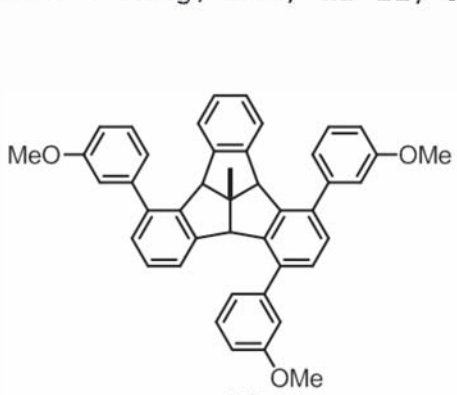
6d


```
Current Data Parameters
NAME      Eric_8a_3_OMe_data
EXPNO                      3
PROCNO                     1

F2 - Acquisition Parameters
Date_           20151216
Time              16.52 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    98
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                   181
DW                20.800 usec
DE                 6.50 usec
TE                295.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          100.6479773 MHz
NUC1                  13C
P1                  9.50 usec
PLW1          55.34000015 W
SFO2          400.2316009 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          13.56000042 W
PLW12          0.27428001 W
PLW13          0.13796000 W

F2 - Processing parameters
SI                 32768
SF            100.6379059 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

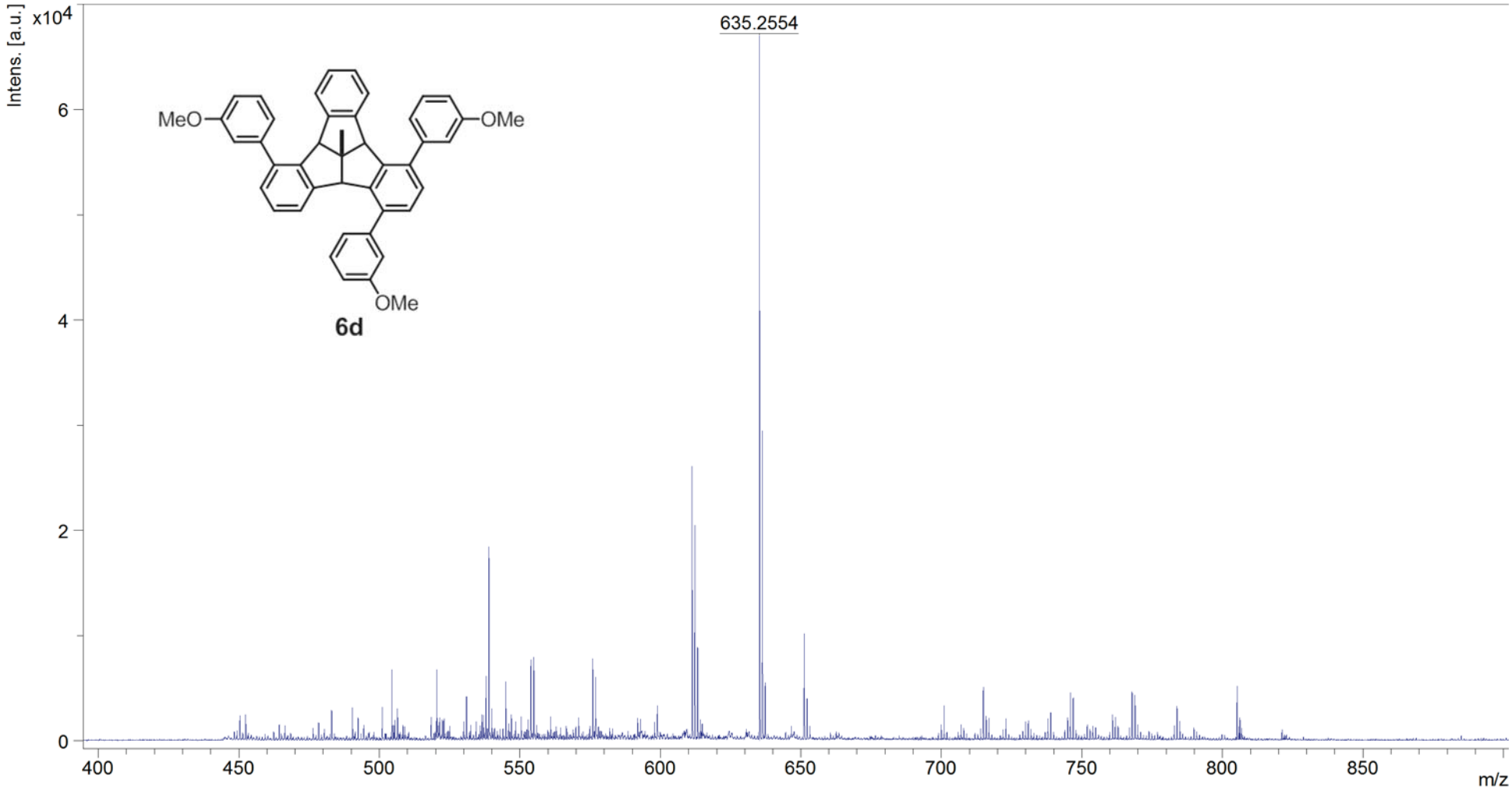
File:EI2016_072 Ident:118_129 Win 500PPM Acq:15-FEB-2016 21:55:50 +12:48 Cal:EI_POS_CAL_900
AutoSpec EI+ Magnet BpM:612 BpI:1061781 TIC:10480833 Flags:HALL
File Text:Er. Wang, OC1, Er-11, HWP

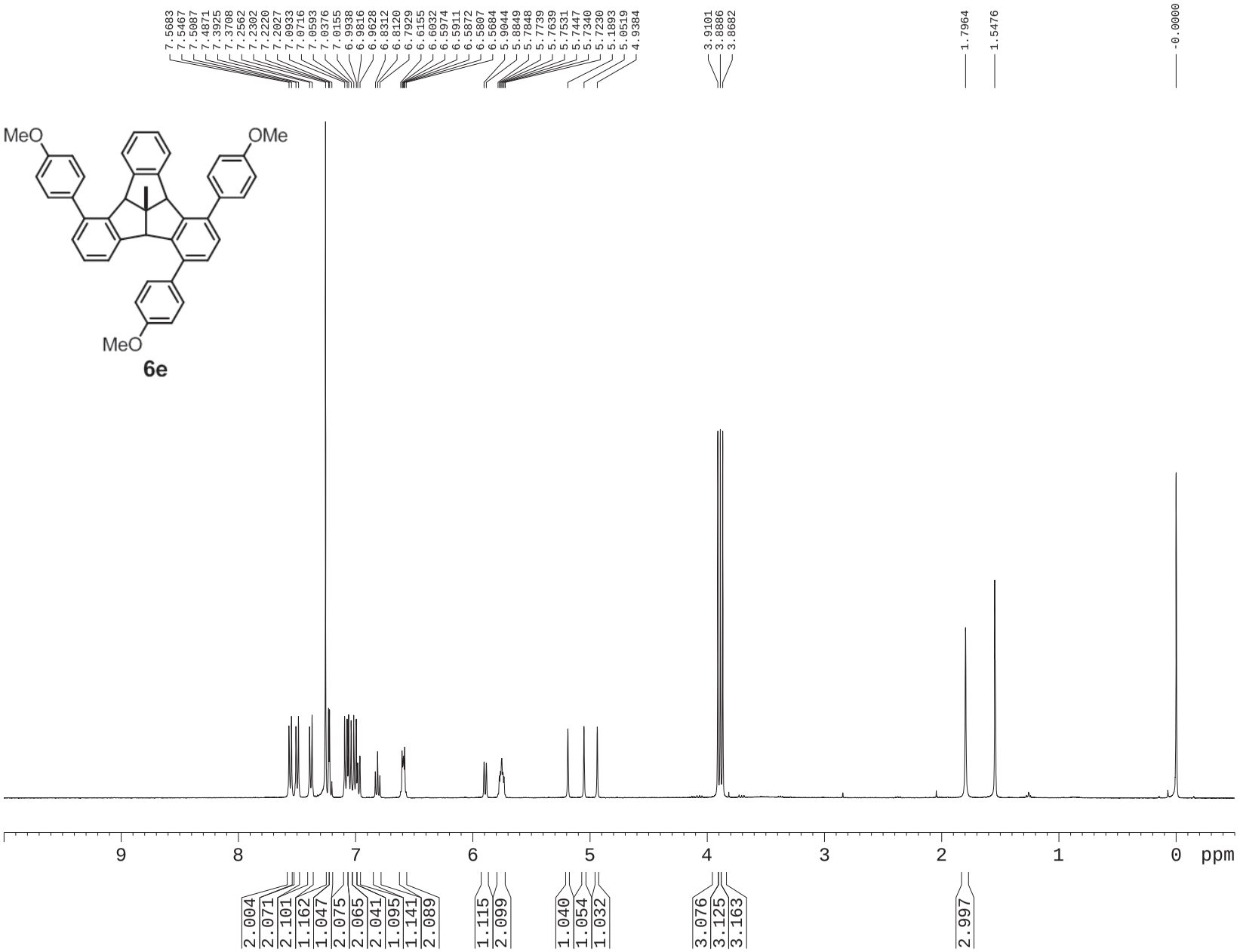


6d

Comment 1

Comment 2





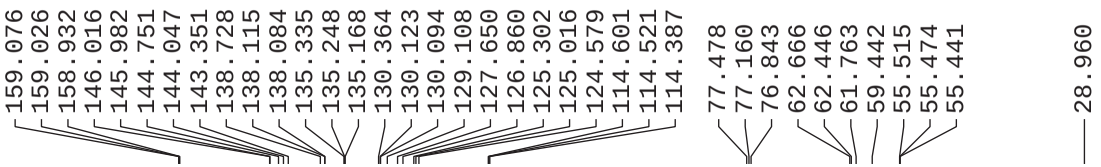
AV400Q NMR

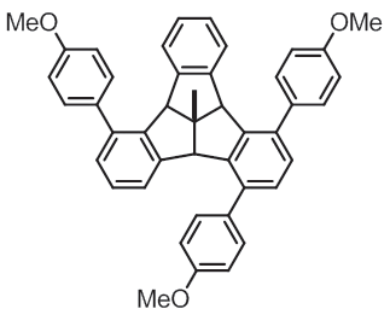
BRUKER

```
Current Data Parameters
NAME      Eric_8a_4_OMe_data
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160115
Time              11.53 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG             zg30
TD                 32768
SOLVENT             CDCl3
NS                    26
DS                     2
SWH            8012.820 Hz
FIDRES          0.244532 Hz
AQ             2.0447233 sec
RG                   161
DW                62.400 usec
DE                 6.50 usec
TE                295.8 K
D1             1.00000000 sec
TD0                    1
SFO1         400.2324714 MHz
NUC1                  1H
P1                 12.80 usec
PLW1        13.56000042 W

F2 - Processing parameters
SI                 65536
SF           400.2300120 MHz
WDW                   EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```





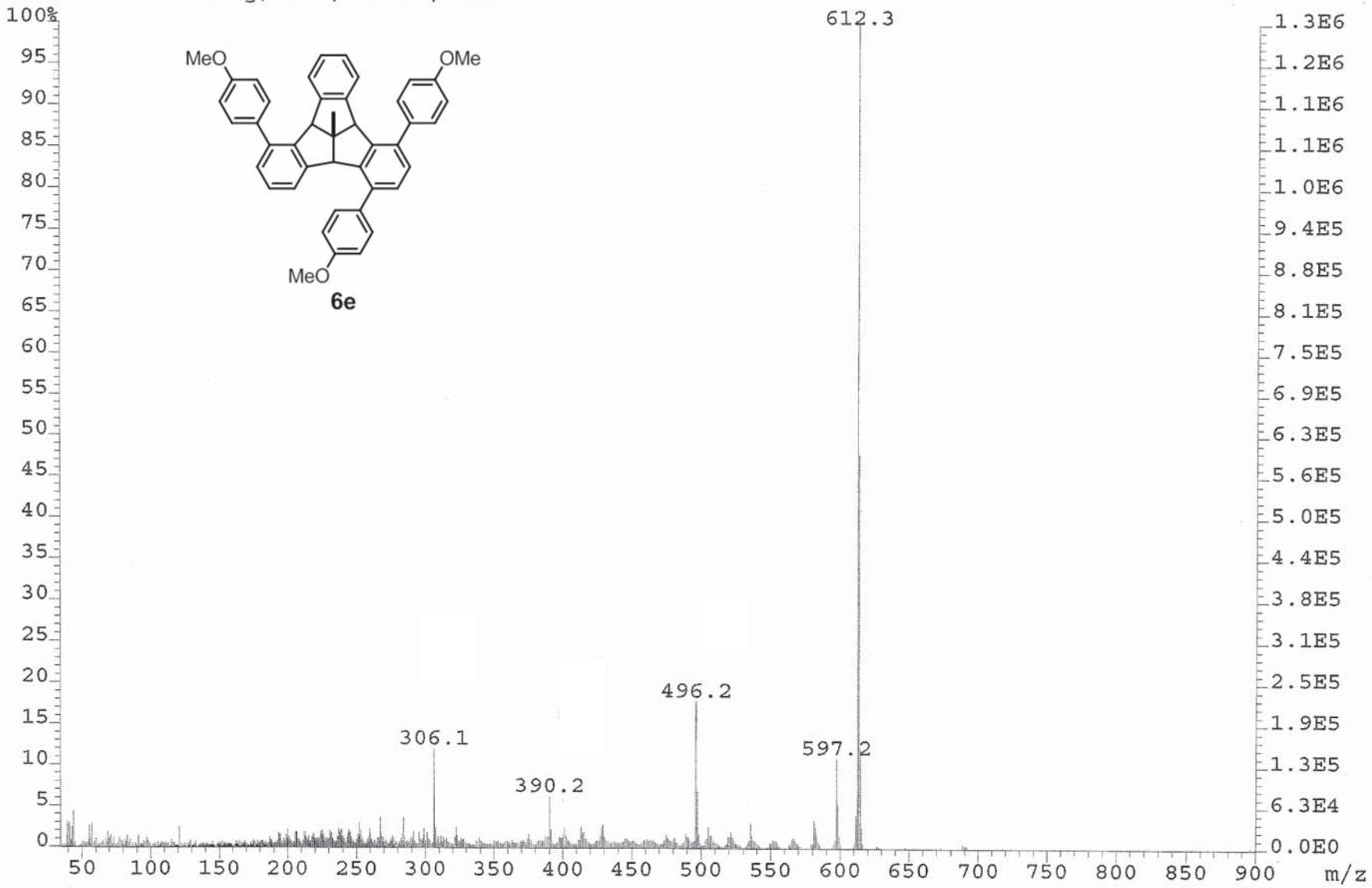
6e


```
Current Data Parameters
NAME      Eric_8a_4_OMe_data
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_           20160115
Time              12.21 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   104
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                  90.5
DW                20.800 usec
DE                 6.50 usec
TE                295.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          100.6479773 MHz
NUC1                 13C
P1                  9.50 usec
PLW1          55.34000015 W
SFO2          400.2316009 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          13.56000042 W
PLW12          0.27428001 W
PLW13          0.13796000 W

F2 - Processing parameters
SI                 32768
SF            100.6379114 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

File:EI2016_075 Ident:165_210 Win 500PPM Acq:17-FEB-2016 20:39:52 +19:23 Cal:EI_POS_CAL_900
AutoSpec EI+ Magnet BpM:612 BpI:1252736 TIC:10374387 Flags:HALL
File Text:Er. Wang, OC1, Er-13, HWP

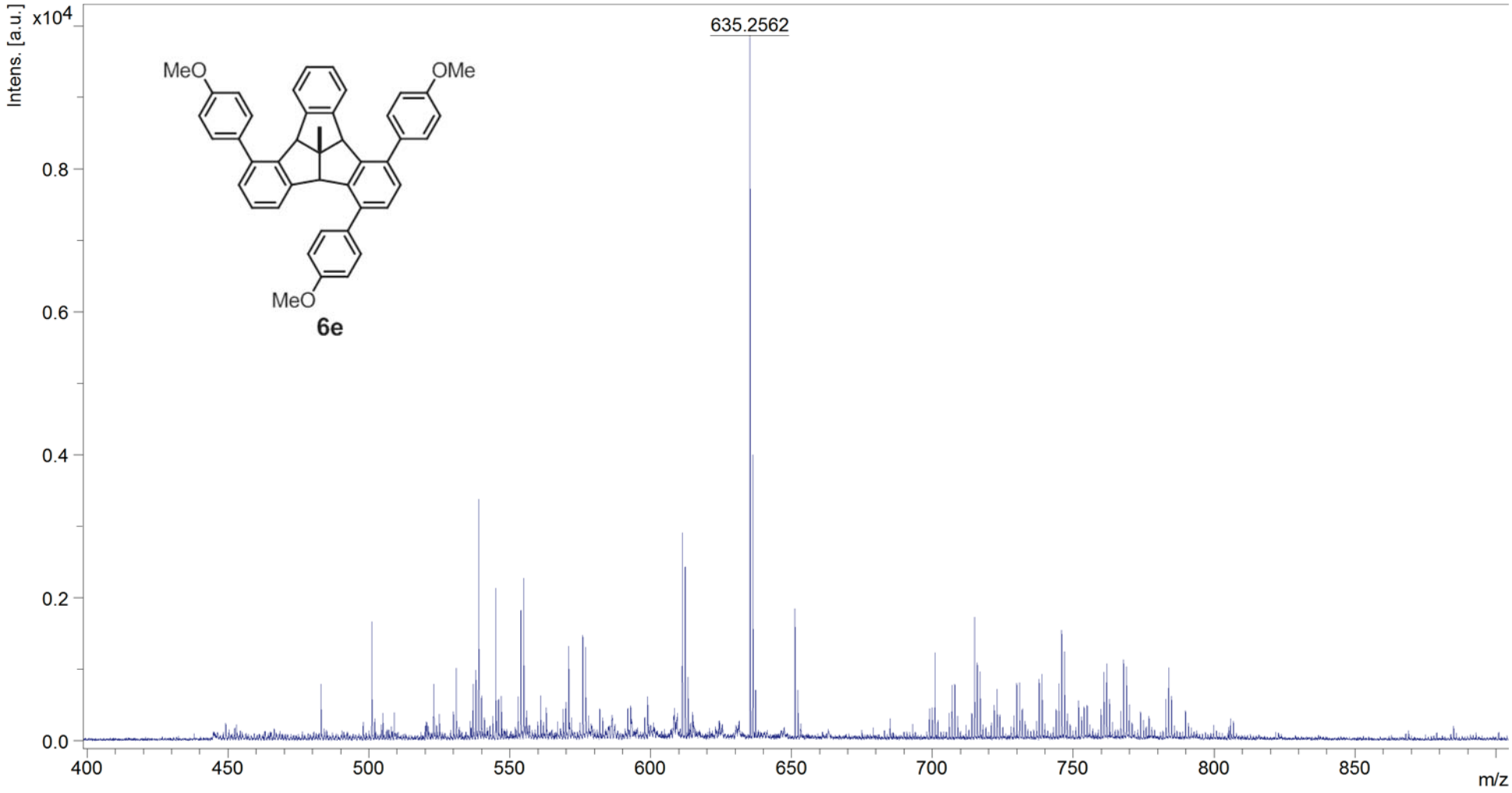


6e

G:\MS Angew\11.Eric_8a_4_OMe(A9)\0_A9\1\1SRef

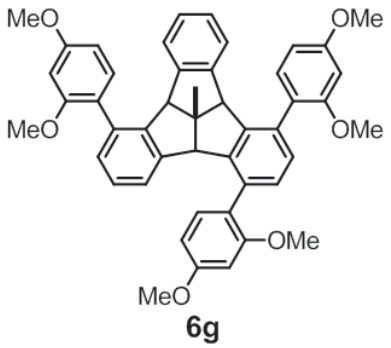
Comment 1

Comment 2



Bruker Daltonics flexAnalysis printed: 5/11/2016 7:02:39 PM

22 °C



6g

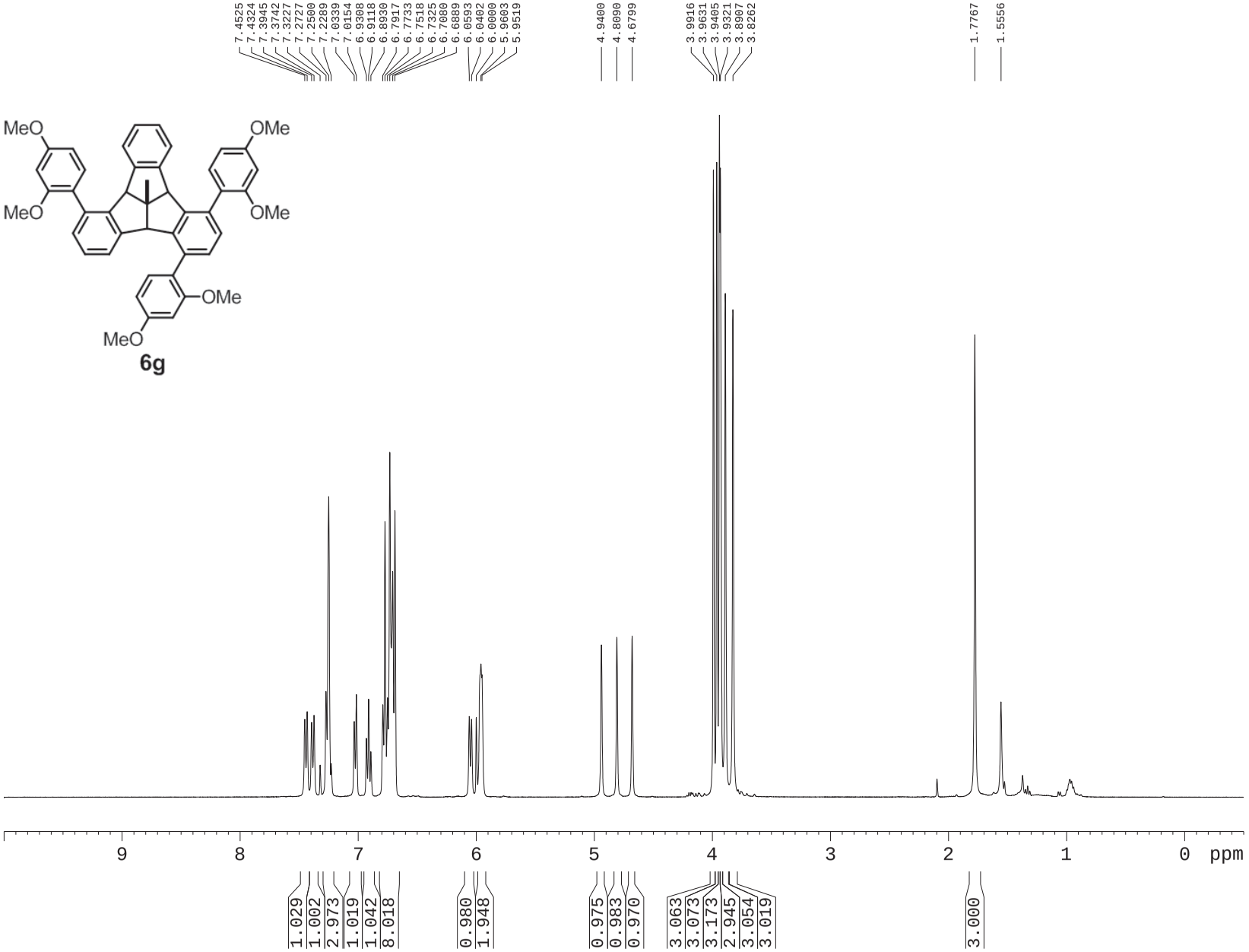
AV400Q NMR

```
Current Data Parameters
NAME      Eric_8a_2_4_OMe_data_25
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_              20160413
Time                 18.08 h
INSTRUM              spect
PROBHD    Z824601_0021 (
PULPROG              zg30
TD                  32768
SOLVENT              CDCl3
NS                      15
DS                       2
SWH               8012.820 Hz
FIDRES               0.244532 Hz
AQ                2.0447233 sec
RG                      64
DW                   62.400 usec
DE                    6.50 usec
TE                   295.2 K
D1             1.00000000 sec
TD0                      1
SFO1          400.1324708 MHz
NUC1                    1H
P1                   15.00 usec
PLW1           8.31000042 W

F2 - Processing parameters
SI                    65536
SF            400.1305140 MHz
WDW                     EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```

60 °C


7.4525
7.4324
7.3945
7.3742
7.3227
7.2727
7.2500
7.2289
7.0339
7.0154
6.9308
6.9118
6.8930
6.7917
6.7733
6.7518
6.7325
6.7080
6.6889
6.0593
6.0402
6.0000
5.9603
5.9519
4.9400
4.8090
4.6799
3.9916
3.9631
3.9405
3.9321
3.8907
3.8262
1.7767
1.5556
MeO
MeO
OMe
OMe
OMe
MeO
6g
AV400Q NMR
BRUKER
Current Data Parameters
NAME Eric_8a_2_4_OMe_data_60
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160414
Time 15.48 h
INSTRUM spect
PROBHD Z824601_0021 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 25
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447233 sec
RG 80.6
DW 62.400 usec
DE 6.50 usec
TE 333.3 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.31000042 W
F2 - Processing parameters
SI 65536
SF 400.1305132 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.029
1.002
2.973
1.019
1.042
8.018
0.980
1.948
0.975
0.983
0.970
3.063
3.073
3.173
2.945
3.054
3.019
3.000

60 °C

160.681
160.599
160.547
157.433
157.380
157.223
146.401
145.718
145.382
145.075
144.891
144.239
135.052
134.443
134.345
132.164
132.084
129.990
129.353
126.583
126.159
126.078
125.291
125.068
124.472
124.414
124.334
124.268
105.417
105.298
105.155
99.468
99.330
74.055
73.780
73.504
63.433
63.124
62.510
59.206
55.514
55.471
55.282
28.197

6g


```
Current Data Parameters
NAME      Eric_8a_2_4_OMe_data_60
EXPNO                           3
PROCNO                          1

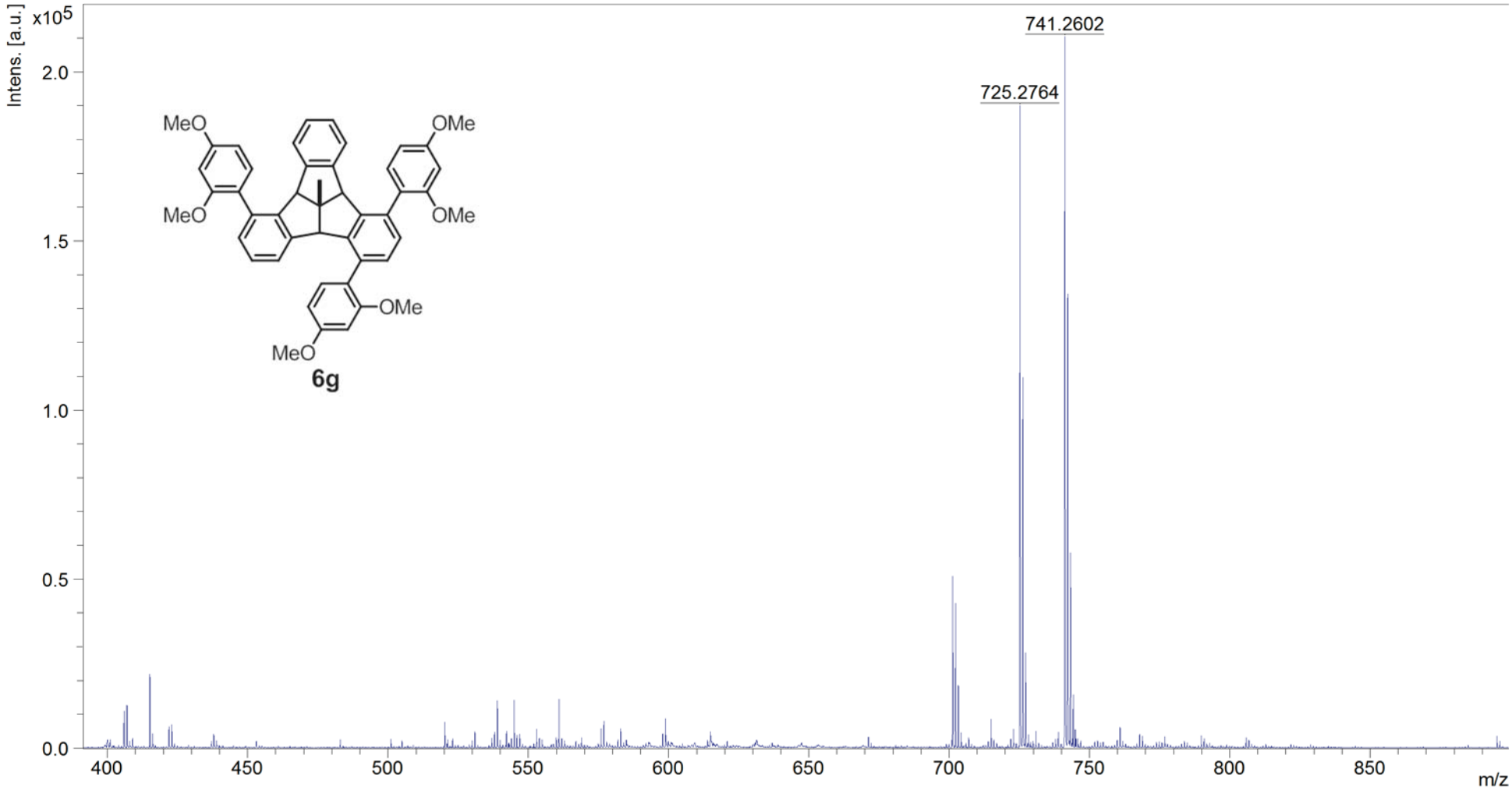
F2 - Acquisition Parameters
Date_           20160418
Time              10.09 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   978
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                333.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1         100.6228298 MHz
NUC1                 13C
P1                 9.50 usec
PLW1        41.25000000 W
SFO2        400.1316005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         8.31000042 W
PLW12        0.23083000 W
PLW13        0.11611000 W

F2 - Processing parameters
SI                 32768
SF          100.6129258 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

G:\MS Angew\13.Eric_8a_2_4_OMe(C24) good\0_C24\1\1SRef

Comment 1

Comment 2



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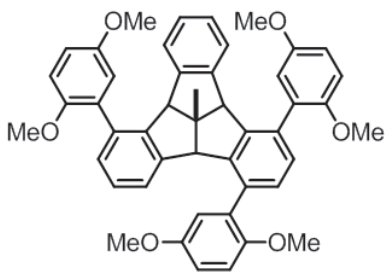
22 °C



6h

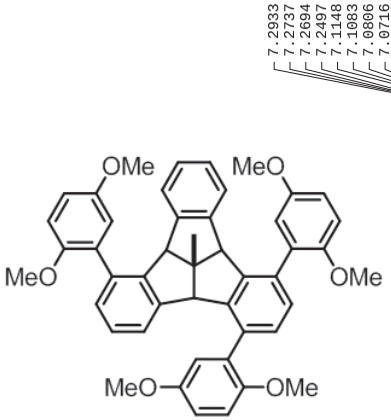
AV400Q NMR

```
Current Data Parameters
NAME      Eric_8a_2_5_OMe_data_25
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_              20160413
Time                17.47 h
INSTRUM              spect
PROBHD    Z824601_0021 (
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     21
DS                      2
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ               2.0447233 sec
RG                    203
DW                 62.400 usec
DE                  6.50 usec
TE                 295.0 K
D1            1.00000000 sec
TD0                    1
SFO1         400.1324708 MHz
NUC1                  1H
P1                 15.00 usec
PLW1          8.31000042 W

F2 - Processing parameters
SI                  65536
SF           400.1305141 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

60 °C



6h

7.2933 7.2737 7.2694 7.2497 7.1148 7.1083 7.0806 7.0716 7.0643 7.0510 7.0395 7.0324 7.0259 6.9925 6.9871 6.9272 6.9174 6.8979 6.8890 6.8789 6.7107 6.6977 6.6866 6.6755 6.6626 6.6231 6.0000 5.8906 5.8696

4.9519 4.8244 4.6956

3.9023 3.8642 3.8551 3.8272 3.8137 3.7465

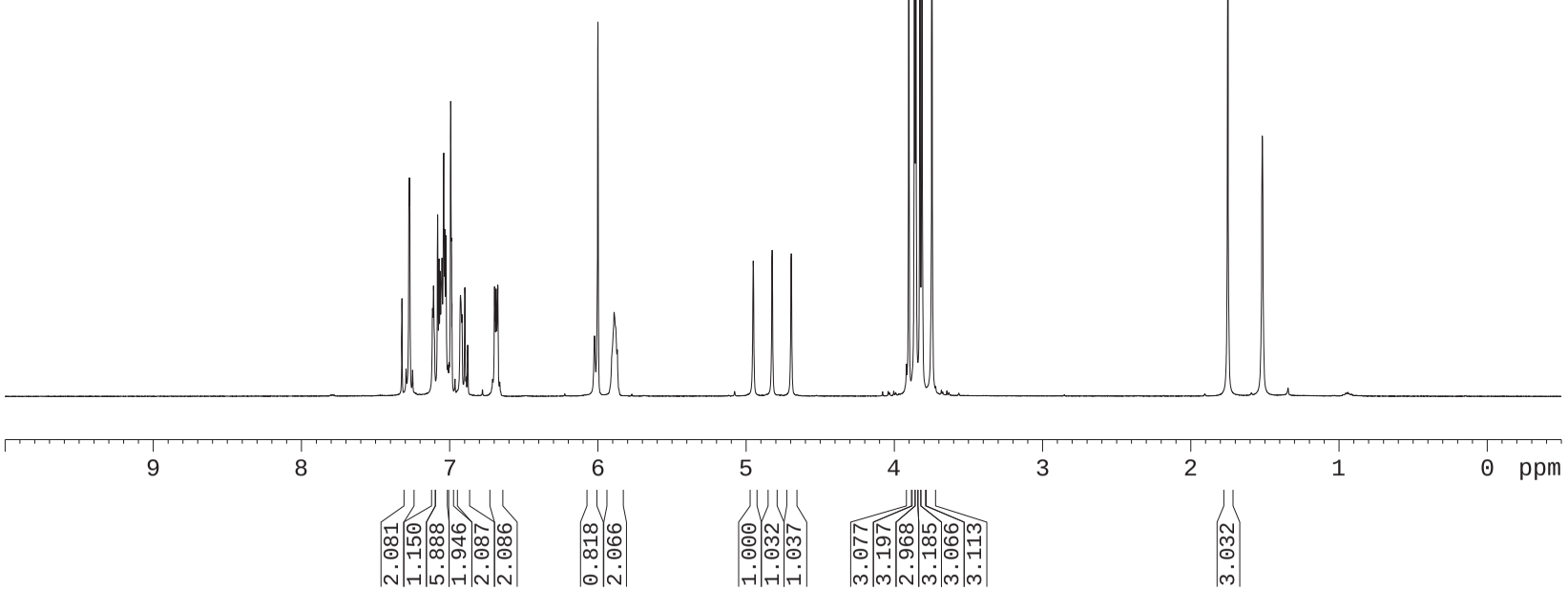
1.7500 1.5170

AV400Q NMR

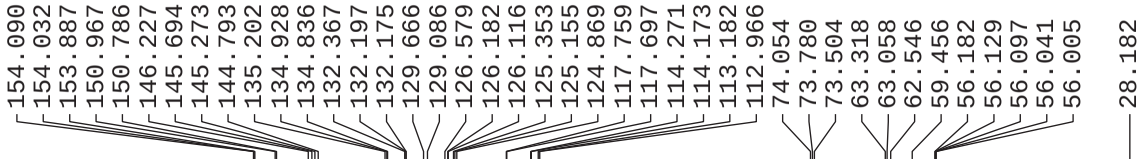

```
Current Data Parameters
NAME      Eric_8a_2_5_OMe_data_60
EXPNO                  1
PROCNO                 1

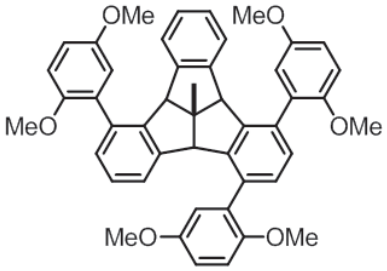
F2 - Acquisition Parameters
Date_              20160122
Time                 16.20 h
INSTRUM              spect
PROBHD    Z824601_0021 (
PULPROG              zg30
TD                  32768
SOLVENT              CDCl3
NS                     57
DS                      2
SWH               8012.820 Hz
FIDRES               0.244532 Hz
AQ                2.0447233 sec
RG                    203
DW                  62.400 usec
DE                   6.50 usec
TE                  333.6 K
D1             1.00000000 sec
TD0                     1
SFO1          400.1324708 MHz
NUC1                   1H
P1                   15.00 usec
PLW1           8.31000042 W

F2 - Processing parameters
SI                    65536
SF            400.1305125 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```



60 °C





6h


```
Current Data Parameters
NAME      Eric_8a_2_5_OMe_data_60
EXPNO                          2
PROCNO                         1

F2 - Acquisition Parameters
Date_           20160122
Time              16.34 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   478
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                333.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1        100.6228298 MHz
NUC1                 13C
P1                 9.50 usec
PLW1        41.25000000 W
SFO2        400.1316005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2         8.31000042 W
PLW12        0.23083000 W
PLW13        0.11611000 W

F2 - Processing parameters
SI                 32768
SF          100.6129242 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

File:EI2016_080 Ident:178_239 Win 500PPM Acq:18-FEB-2016 01:06:21 +21:33 Cal:EI_POS_CAL_900
AutoSpec EI+ Magnet BpM:702 BpI:684480 TIC:8581441 Flags:HALL
File Text:Er. Wang, OC1, Er-18, HWP

6h

Bruker 9.4T FTICR MS Analysis Report

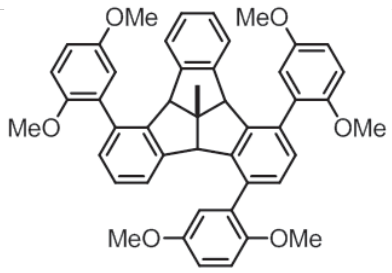
Faculty of Science, The Chinese University of Hong Kong

Analysis Info

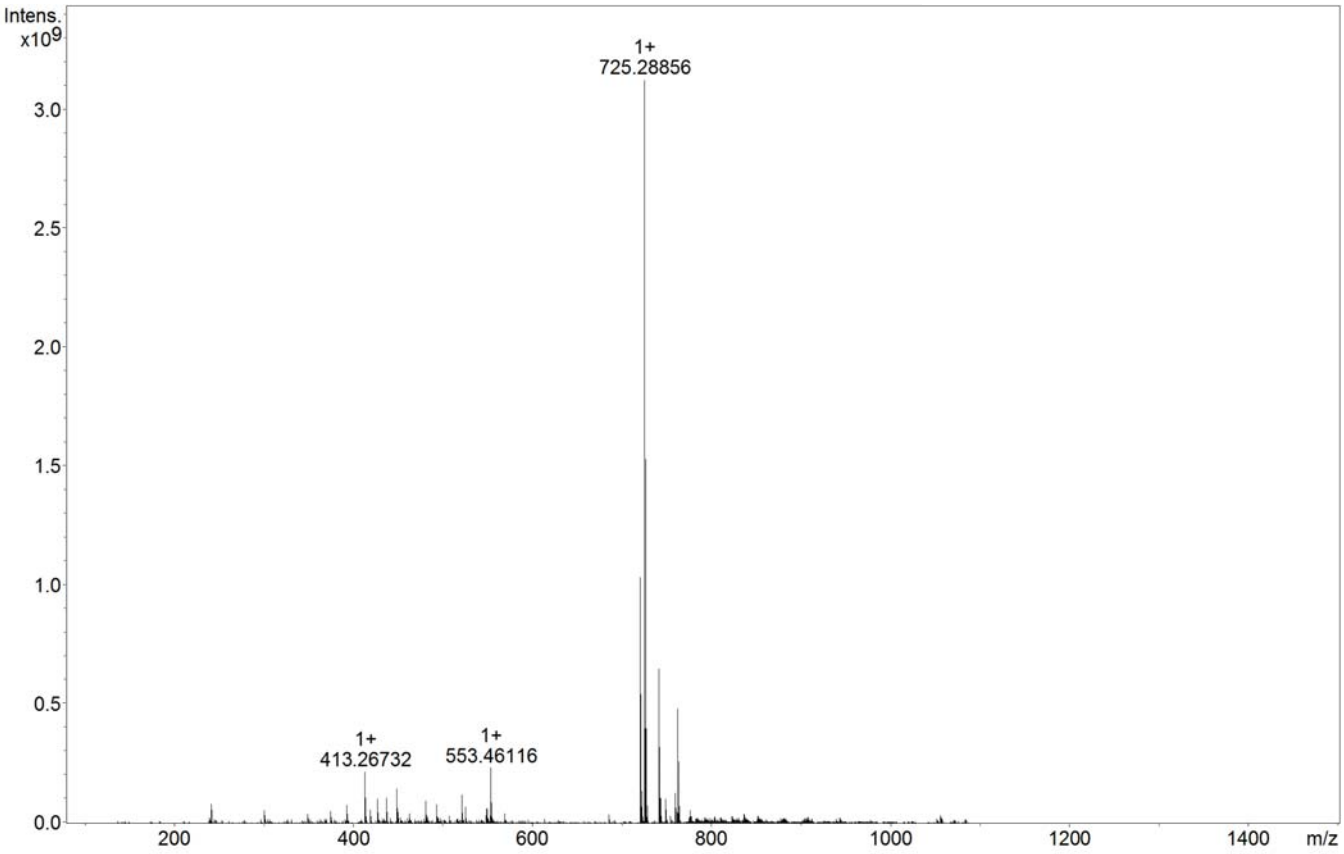
Sample Name :	Eric_8a_2_5_OMe	Reference No. :	xhfc044
Applicant Name :	Ip Ho Wang	Analysis Date :	4/2/2016 11:45:24
Analysis Path :	xhfc044_000001.d		
Instrument :	solariX	Polarity	Positive
Method	4_17_mass_range_pos_7T	Acquired Scans	9
Comment :	4.4kV, 120ml/hr, 1.0 nebulizer gas		

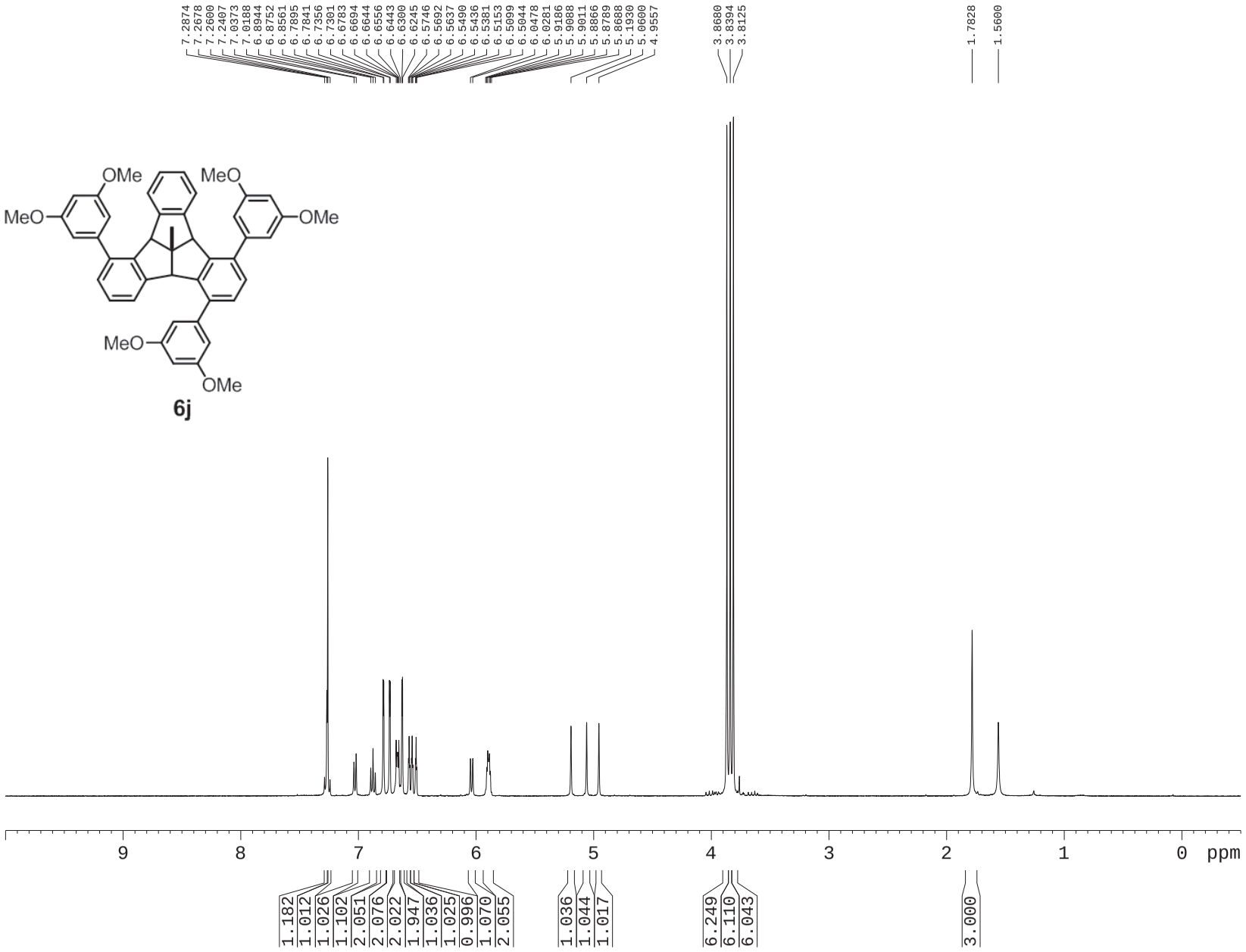
Accurate Mass Measurement

Molecular formula :	C47H42O6
Abundant Isotopic (theoretical) [M+Na]+ :	725.287360
Monoisotopic (theoretical) [M+Na]+ :	725.287360
(experimental) [M+Na]+ :	725.28856
error (ppm) :	1.6



6h





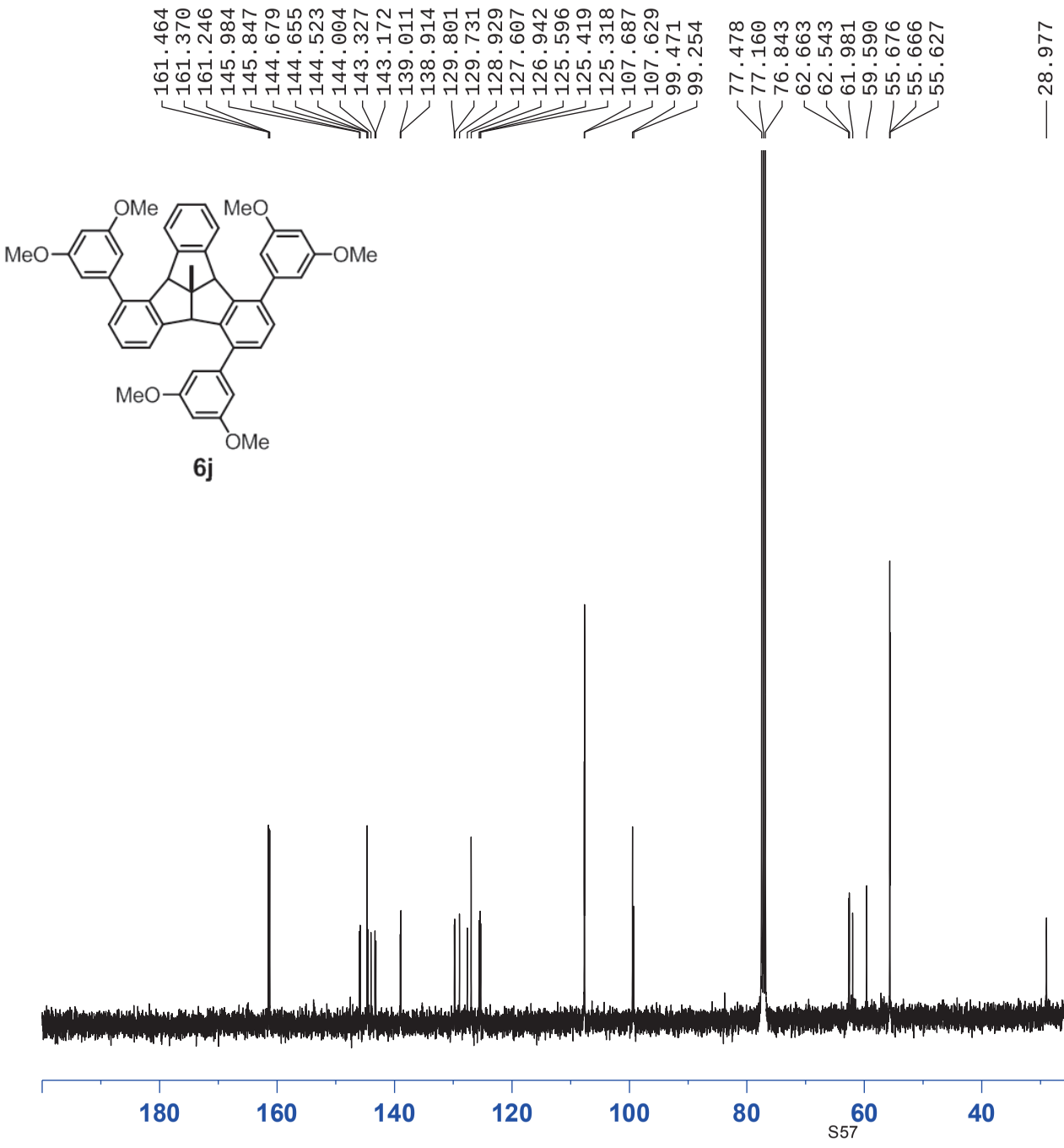
AV400Q NMR

BRUKER

```
Current Data Parameters
NAME      Eric_8a_3_5_OMe_data
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_              20151216
Time                  16.15 h
INSTRUM                spect
PROBHD    Z108618_0257 (
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                        12
DS                         2
SWH                 8012.820 Hz
FIDRES                0.244532 Hz
AQ                 2.0447233 sec
RG                       144
DW                    62.400 usec
DE                     6.50 usec
TE                    295.3 K
D1                 1.00000000 sec
TD0                        1
SFO1              400.2324714 MHz
NUC1                      1H
P1                    12.80 usec
PLW1              13.56000042 W

F2 - Processing parameters
SI                     65536
SF                400.2300097 MHz
WDW                       EM
SSB       0
LB                     0.30 Hz
GB        0
PC                     1.00
```

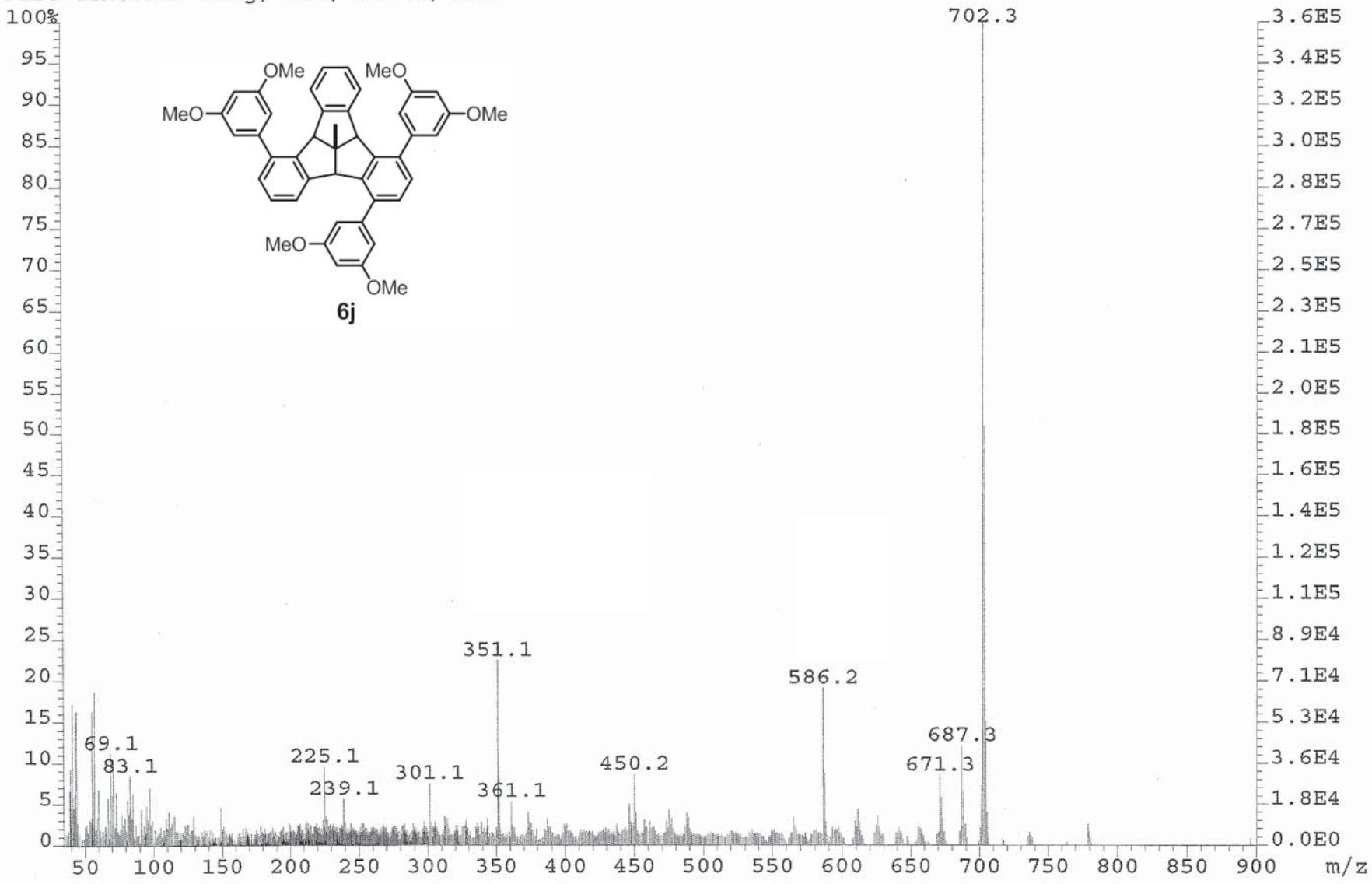



```
Current Data Parameters
NAME      Eric_8a_3_5_OMe_data
EXPNO                        3
PROCNO                       1

F2 - Acquisition Parameters
Date_              20151216
Time                  16.33 h
INSTRUM                spect
PROBHD    Z108618_0257 (
PULPROG               zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       238
DS                         4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                1.3631488 sec
RG                       181
DW                  20.800 usec
DE                   6.50 usec
TE                   296.3 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1
SFO1            100.6479773 MHz
NUC1                     13C
P1                    9.50 usec
PLW1            55.34000015 W
SFO2            400.2316009 MHz
NUC2                     1H
CPDPRG[2             waltz16
PCPD2                90.00 usec
PLW2            13.56000042 W
PLW12            0.27428001 W
PLW13            0.13796000 W

F2 - Processing parameters
SI                       32768
SF              100.6379016 MHz
WDW                         EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

File:EI2016_081 Ident:152_191 Win 500PPM Acq:18-FEB-2016 01:56:59 +17:45 Cal:EI_POS_CAL_900
AutoSpec EI+ Magnet BpM:702 BpI:355994 TIC:6511747 Flags:HALL
File Text:Er. Wang, OC1, Er-19, HWP

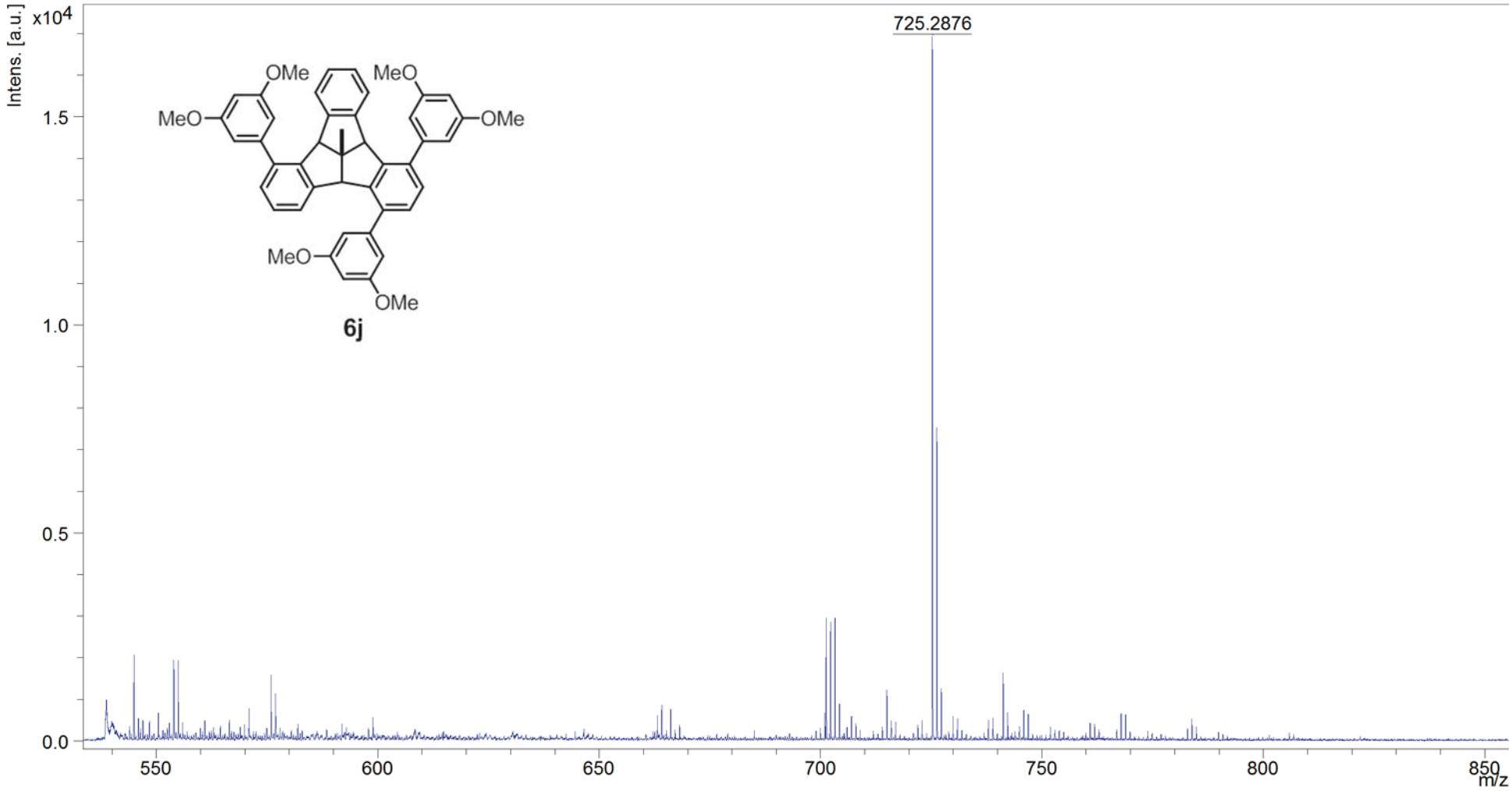


6j

G:\MS Angew\16.Eric_8a_3_5_OMe(C7)\0_C7\1\1SRef

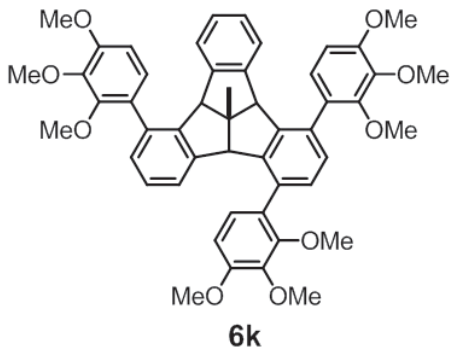
Comment 1

Comment 2



Bruker Daltonics flexAnalysis printed: 5/12/2016 2:24:14 PM

22 °C



6k

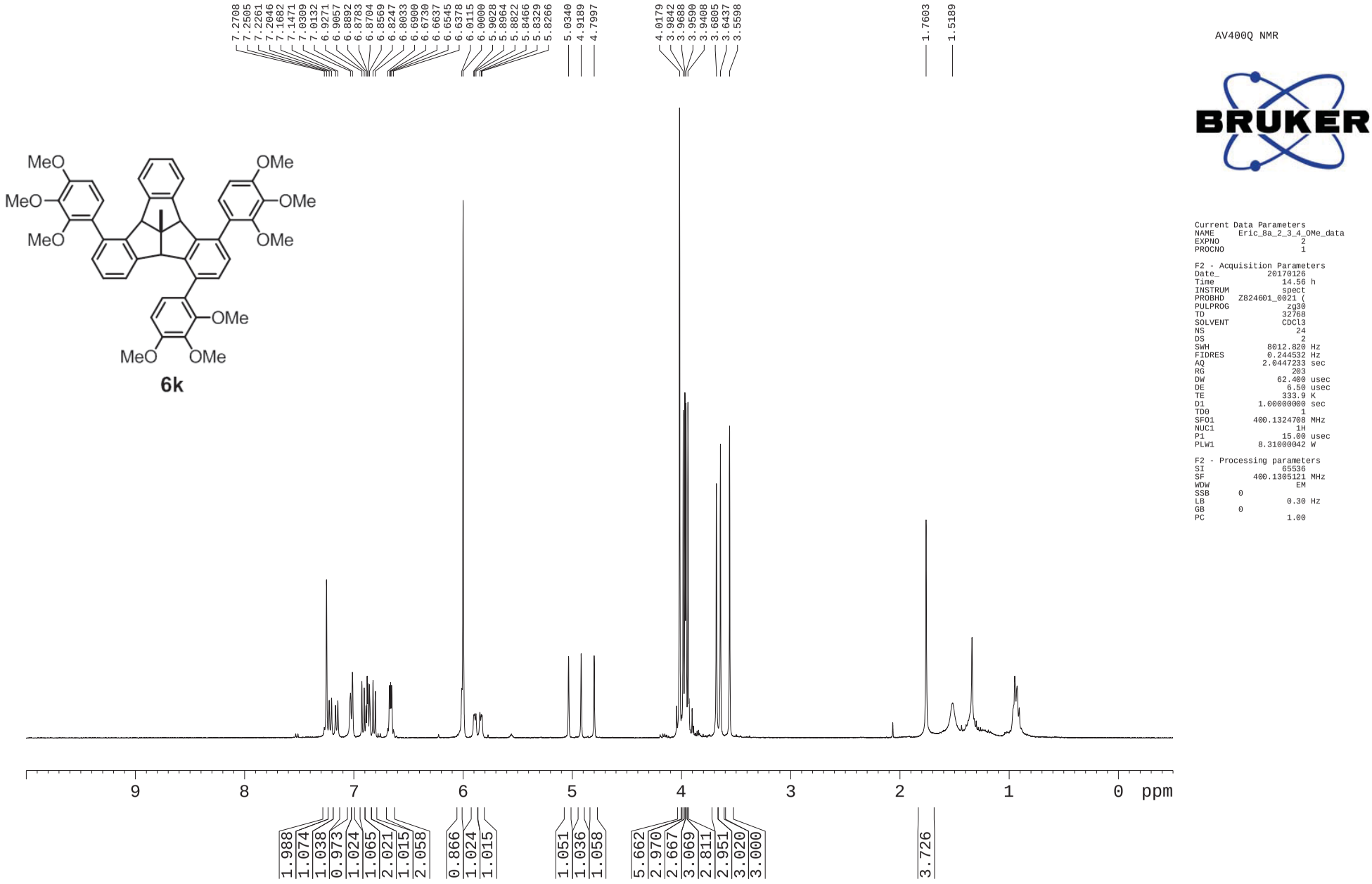
AV400Q NMR


```
Current Data Parameters
NAME      Eric_8a_2_3_4_OMe_data
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_              20170126
Time                  14.47 h
INSTRUM                spect
PROBHD    Z824601_0021 (
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       22
DS                        2
SWH                8012.820 Hz
FIDRES                0.244532 Hz
AQ                 2.0447233 sec
RG                      203
DW                   62.400 usec
DE                    6.50 usec
TE                   295.2 K
D1                 1.00000000 sec
TD0                       1
SFO1              400.1324708 MHz
NUC1                     1H
P1                   15.00 usec
PLW1               8.31000042 W

F2 - Processing parameters
SI                    65536
SF                400.1305137 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```

60 °C



AV400Q NMR


```
Current Data Parameters
NAME      Eric_8a_2_3_4_OMe_data
EXPNO                          2
PROCNO                         1

F2 - Acquisition Parameters
Date_              20170126
Time                  14.56 h
INSTRUM                spect
PROBHD    Z824601_0021 (
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       24
DS                        2
SWH                8012.820 Hz
FIDRES                0.244532 Hz
AQ                 2.0447233 sec
RG                      203
DW                     62.400 usec
DE                      6.50 usec
TE                     333.9 K
D1                 1.00000000 sec
TD0                       1
SFO1              400.1324708 MHz
NUC1                      1H
P1                     15.00 usec
PLW1               8.31000042 W

F2 - Processing parameters
SI                        65536
SF                400.1305121 MHz
WDW                       EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```

60 °C

153.422
153.376
153.286
151.429
151.383
151.263
146.137
145.897
145.358
143.259
143.135
134.972
134.555
134.527
134.487
129.790
129.751
129.287
129.205
129.075
129.027
126.443
126.076
125.481
125.339
125.174
124.696
108.428
108.388
108.356
108.172
74.056
73.780
73.504
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61.005
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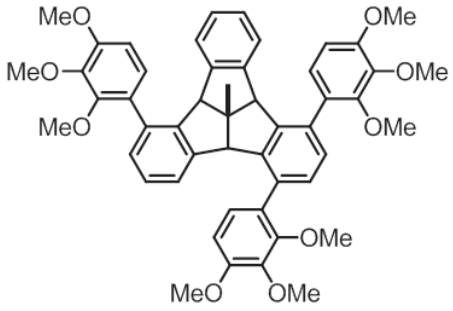
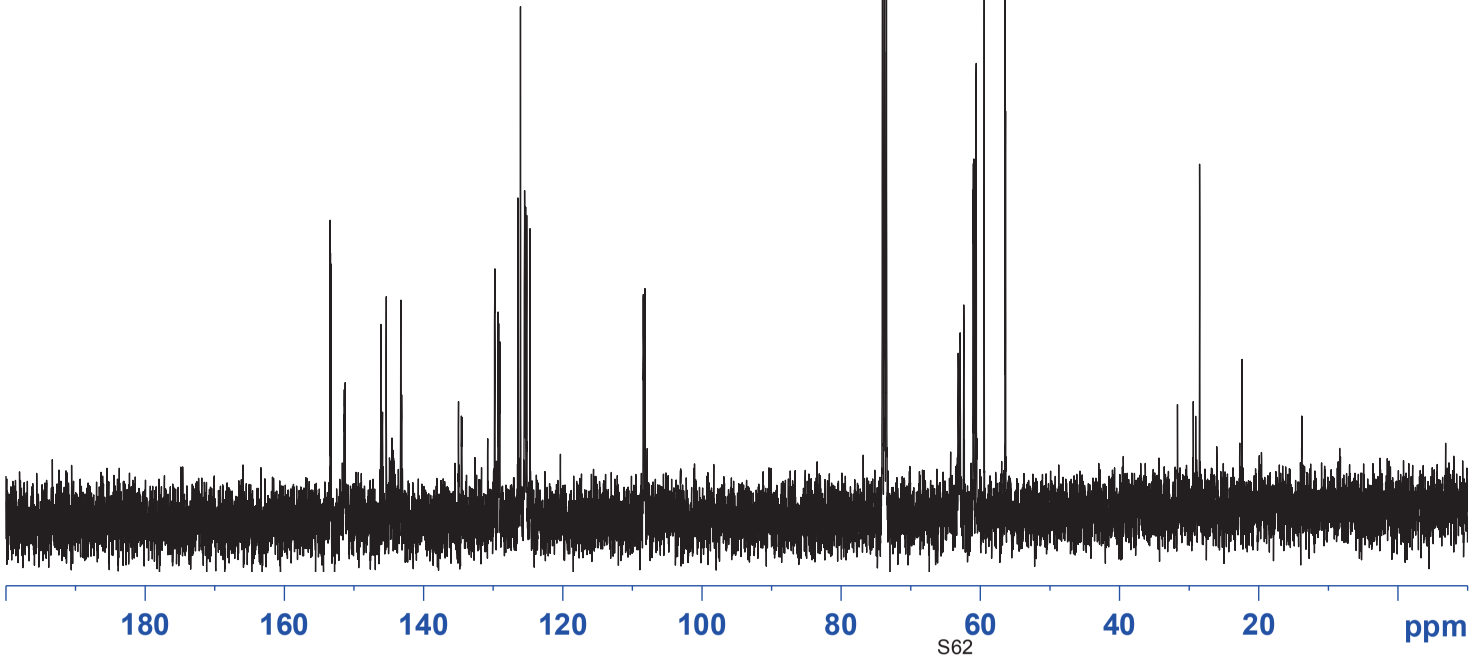
28.495



6k



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NAME Eric_8a_2_3_4_OMe_data
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170126
Time 15.03 h
INSTRUM spect
PROBHD Z824601_0021 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 342
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 333.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 9.50 usec
PLW1 41.25000000 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 8.31000042 W
PLW12 0.23083000 W
PLW13 0.11611000 W

F2 - Processing parameters
SI 32768
SF 100.6129230 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

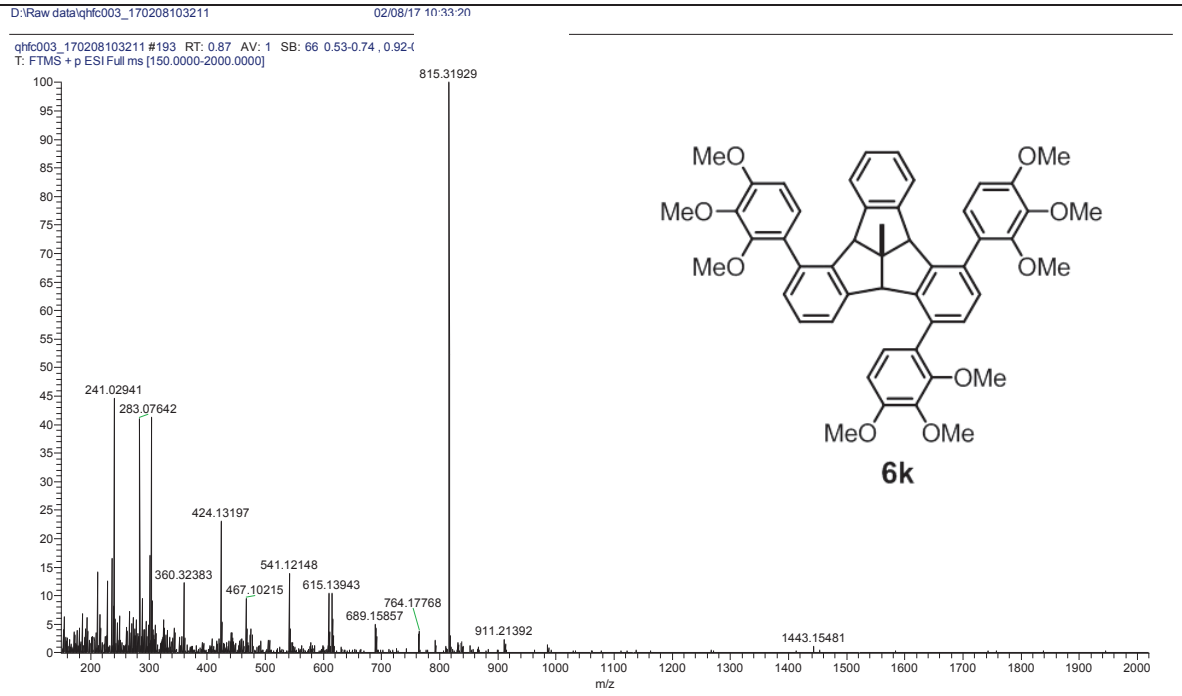
Thermo QEFMS Analysis Report

Analysis Info

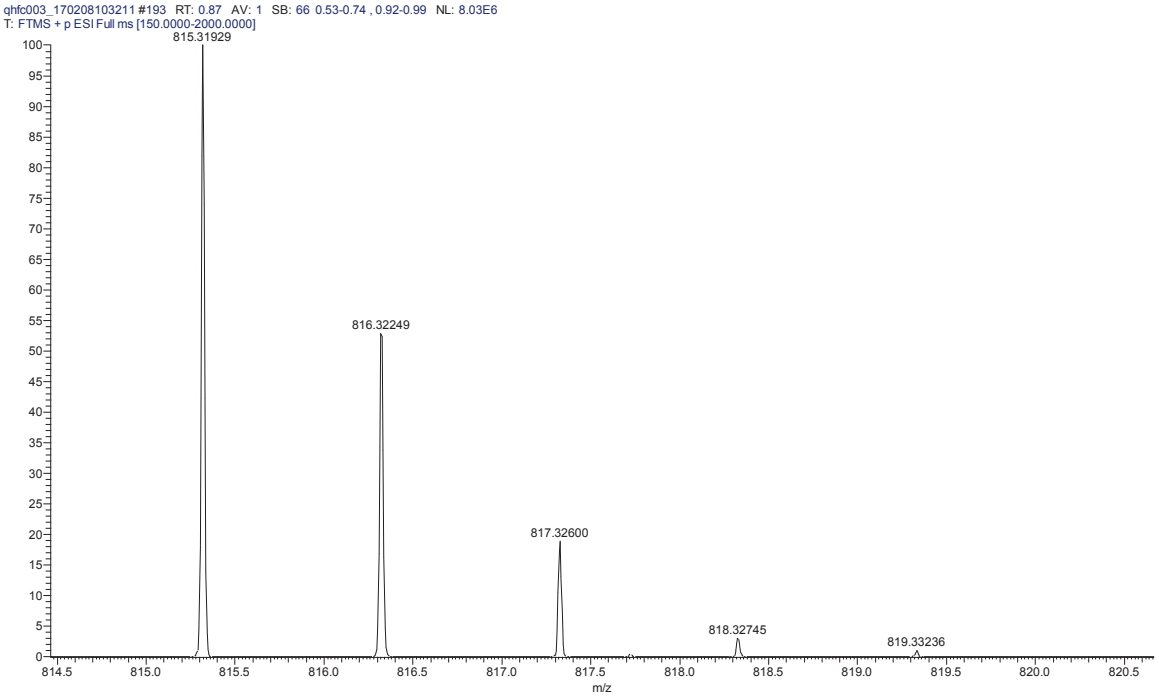
Sample Name :	Eric_26	Reference No.:	Qhfc003
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

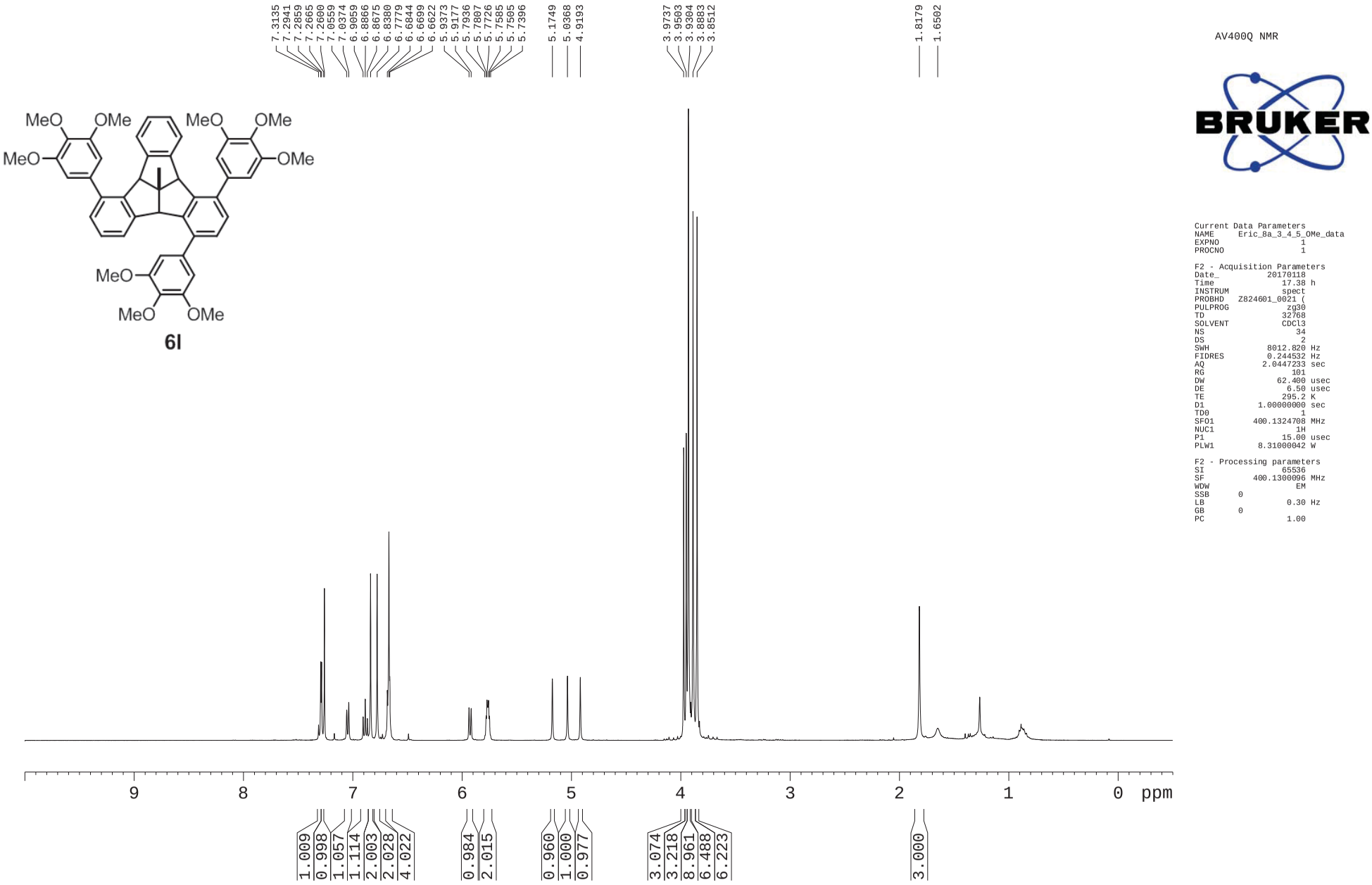
Accurate Mass Measurement

Molecular formula :	$C_{50}H_{48}O_9$
Experimental Mass $[M+Na]^+$:	815.31929
Theoretical Mass $[M+Na]^+$:	815.31905
Error (ppm) :	0.2



6k

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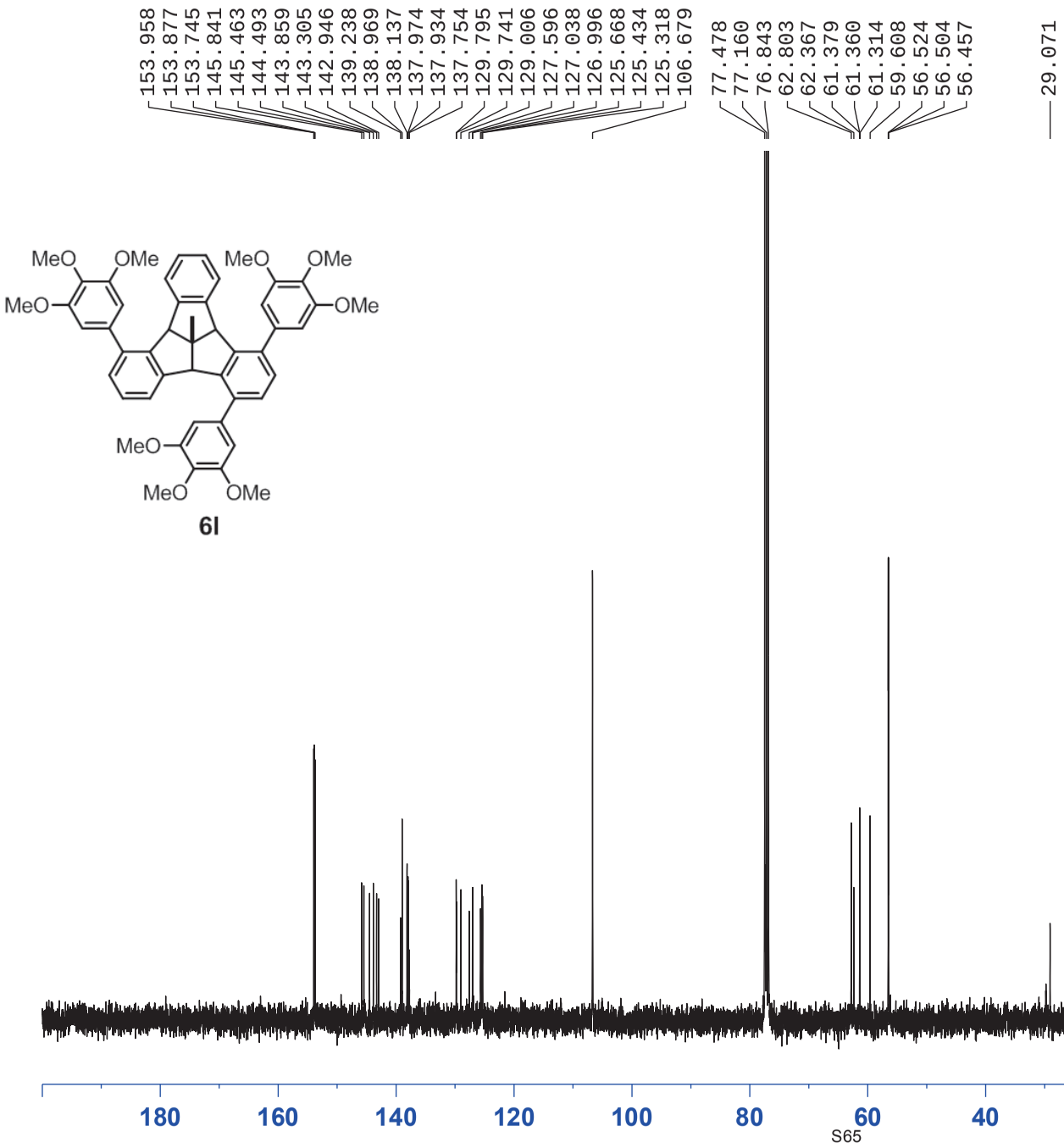
6.8866
6.8675
6.8380
6.7779
6.6844
6.6699
6.6622
5.9373
5.9177
5.7936
5.7807
5.7726
5.7585
5.7505
5.7396
5.1749
5.0368
4.9193
3.9737
3.9503
3.9304
3.8883
3.8512
1.8179
1.6502
MeO
OMe
MeO
OMe
MeO
OMe
MeO
OMe
MeO
OMe
6l
AV400Q NMR
BRUKER
Current Data Parameters
NAME Eric_8a_3_4_5_OMe_data
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170118
Time 17.38 h
INSTRUM spect
PROBHD Z824601_0021 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 34
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447233 sec
RG 101
DW 62.400 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.31000042 W
F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.009
0.998
1.057
1.114
2.003
2.028
4.022
0.984
2.015
0.960
1.000
0.977
3.074
3.218
8.961
6.488
6.223
3.000




```
Current Data Parameters
NAME      Eric_8a_3_4_5_OMe_data
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170118
Time              17.43 h
INSTRUM           spect
PROBHD   Z824601_0021 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  230
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                  203
DW               20.800 usec
DE                6.50 usec
TE               295.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6228298 MHz
NUC1                13C
P1                 9.50 usec
PLW1         41.25000000 W
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2          8.31000042 W
PLW12         0.23083000 W
PLW13         0.11611000 W

F2 - Processing parameters
SI                32768
SF           100.6127591 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

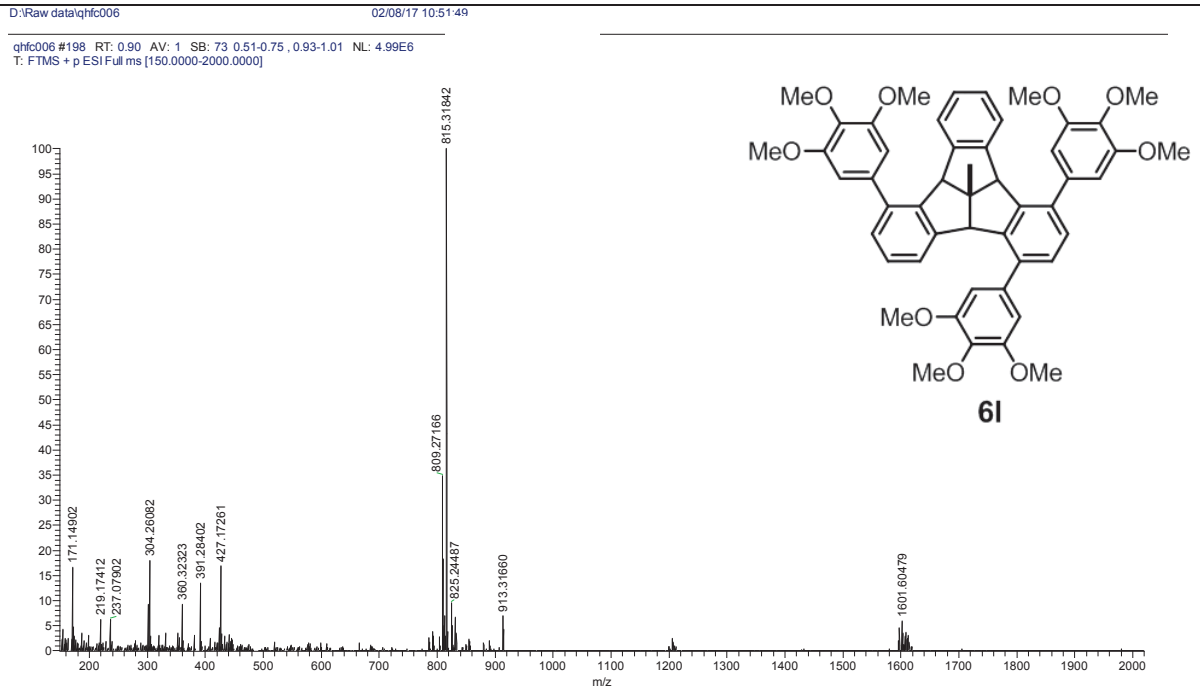
Thermo QEFMS Analysis Report

Analysis Info

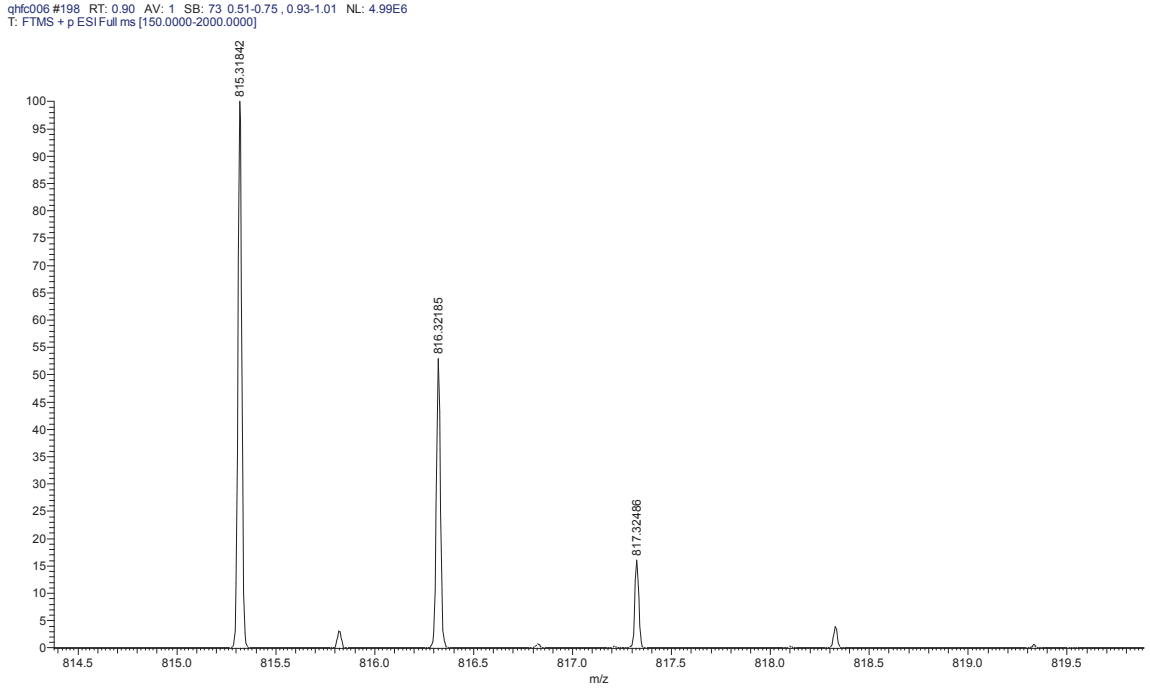
Sample Name :	Eric_29	Reference No.:	Qhfc006
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

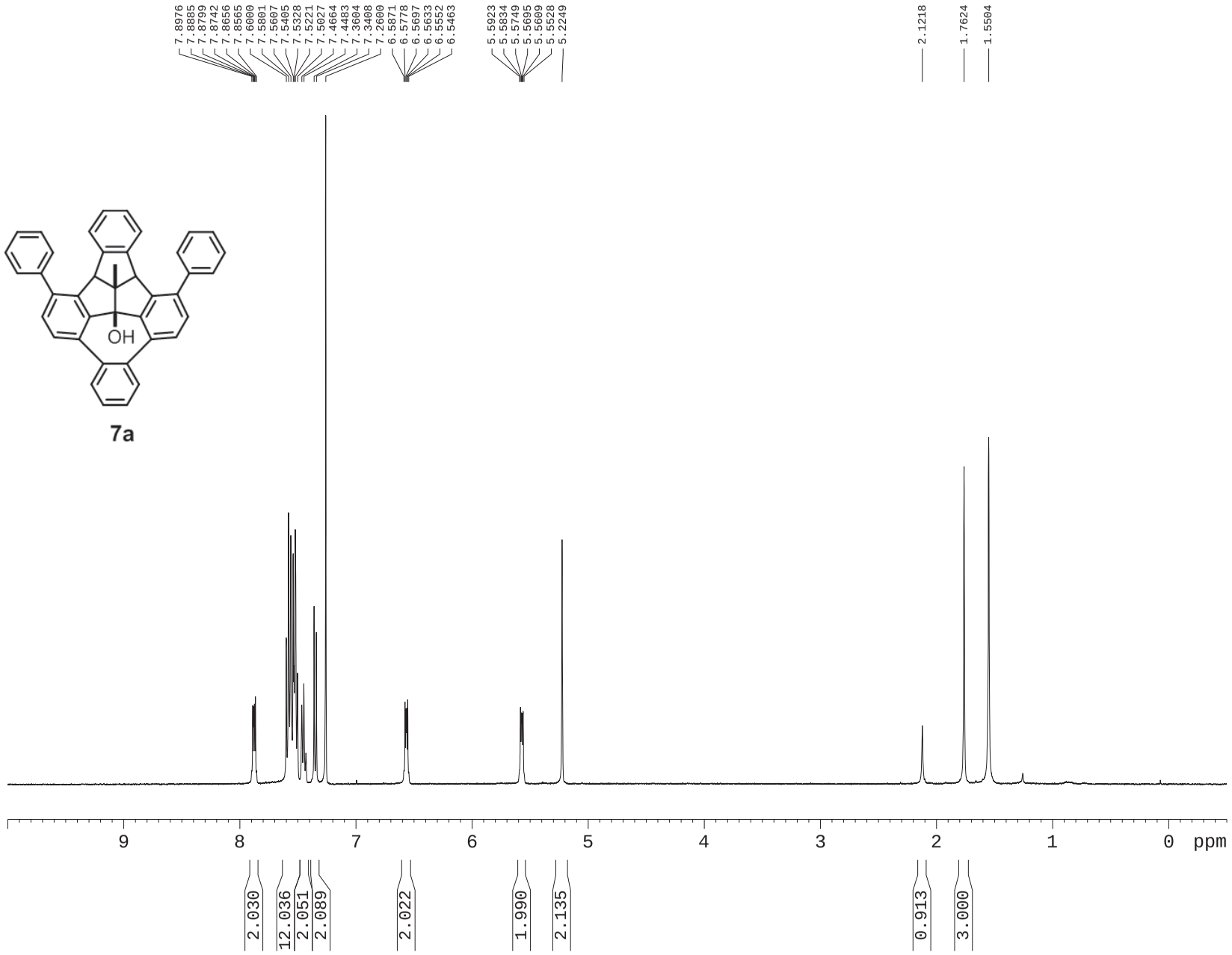
Accurate Mass Measurement

Molecular formula :	$C_{50}H_{48}O_9$
Experimental Mass $[M+Na]^+$:	815.31842
Theoretical Mass $[M+Na]^+$:	815.31905
Error (ppm) :	0.7



6l





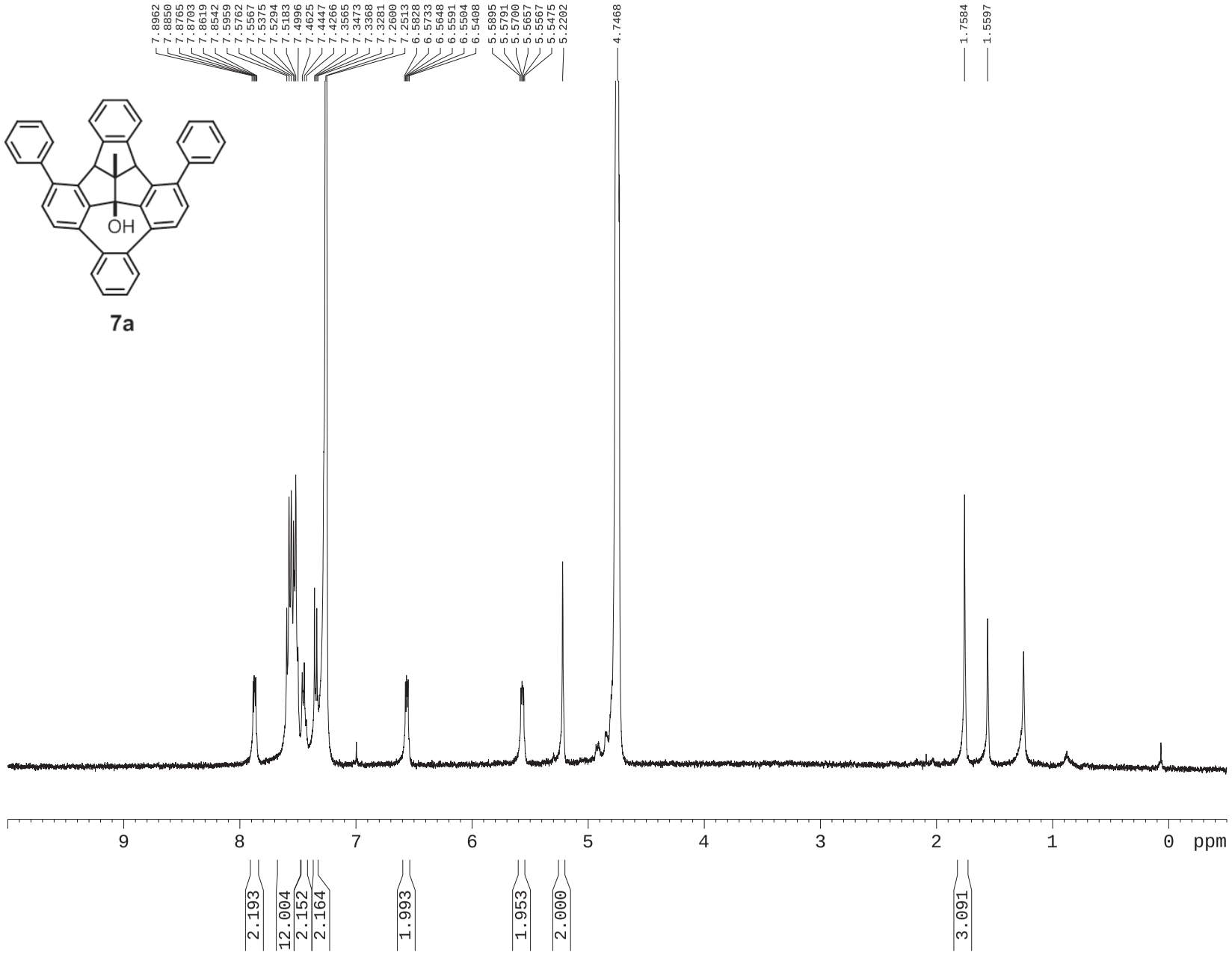
AV400Q NMR

Current Data Parameters
NAME Eric_8a_un_mono_OH_data
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151216
Time 12.41 h
INSTRUM spect
PROBHD Z824601_0021 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 19
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447233 sec
RG 203
DW 62.400 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.31000042 W

F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

$CDCl_3/D_2O$ = 50/1

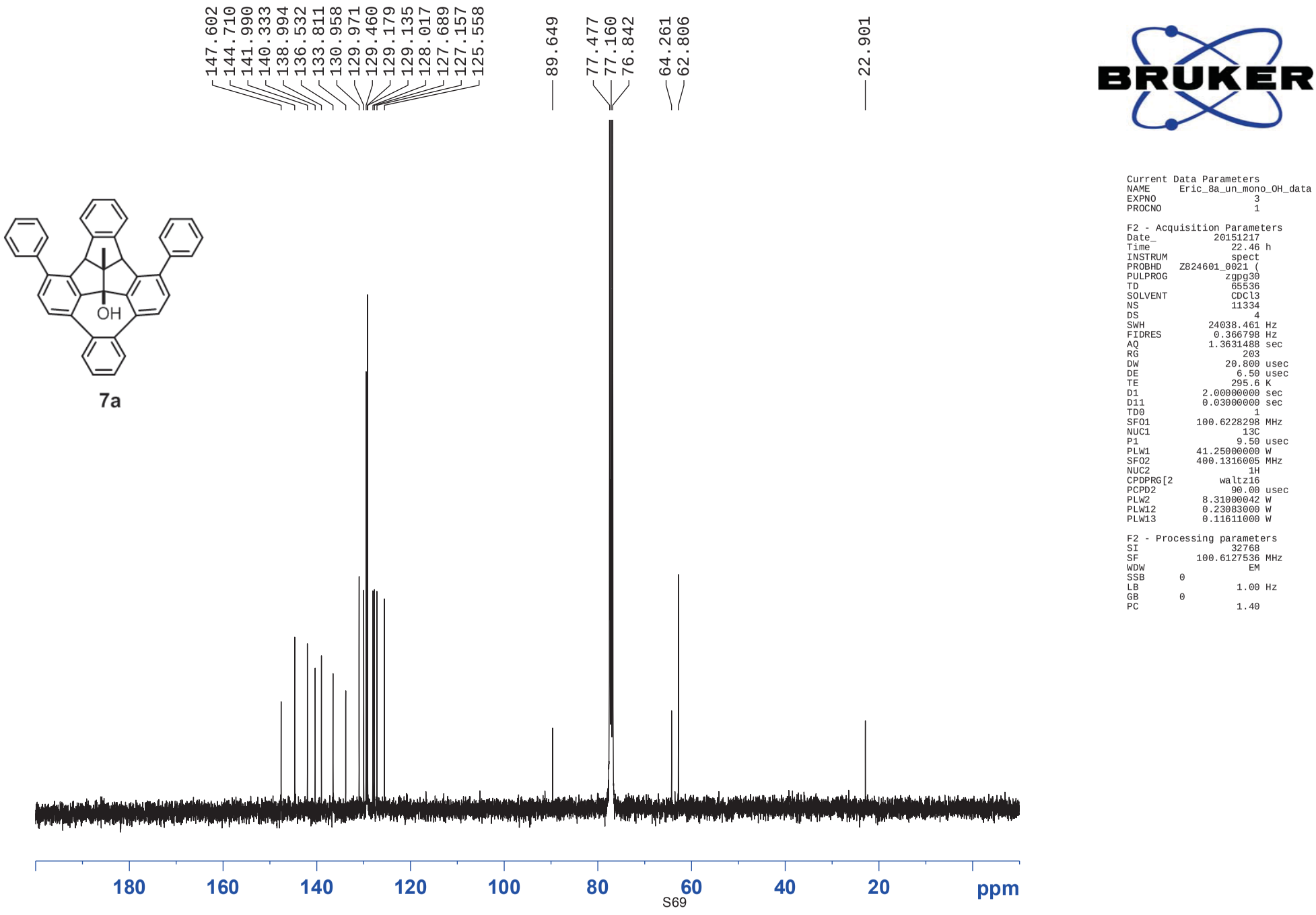


AV400Q NMR


```
Current Data Parameters
NAME      Eric_9a_un_mono_OH_data_D2O
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20170206
Time              18.40 h
INSTRUM             spect
PROBHD     Z108618_0257 (
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    80
DS                     2
SWH             8012.820 Hz
FIDRES             0.244532 Hz
AQ              2.0447233 sec
RG                   203
DW                62.400 usec
DE                 6.50 usec
TE                295.2 K
D1             1.00000000 sec
TD0                    1
SFO1           400.2324714 MHz
NUC1                  1H
P1                 12.80 usec
PLW1           13.56000042 W

F2 - Processing parameters
SI                 65536
SF             400.2300109 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



BRUKER

```
Current Data Parameters
NAME      Eric_8a_un_mono_OH_data
EXPNO                           3
PROCNO                          1

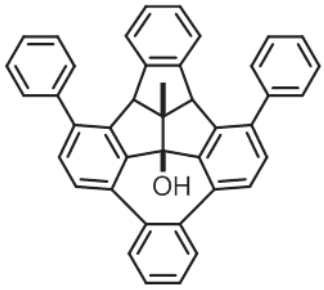
F2 - Acquisition Parameters
Date_           20151217
Time              22.46 h
INSTRUM             spect
PROBHD   Z824601_0021 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                 11334
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                295.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1         100.6228298 MHz
NUC1                 13C
P1                 9.50 usec
PLW1        41.25000000 W
SFO2        400.1316005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2         8.31000042 W
PLW12        0.23083000 W
PLW13        0.11611000 W

F2 - Processing parameters
SI                 32768
SF           100.6127536 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

9.4T FTICR MS Analysis Report

Faculty of Science, The Chinese University of Hong Kong

Sample No.	Eric12
Reference No.	CHF0025
Applicant name	Ip Ho Wang
Analysis Path	\\192.168.0.1\Data\Service\MSonly\20160509\Eric12_000002.d
Instrument	solariX
Polarity	Positive
Acquired Scans	4
Operator	Winnie
Analysis Date	5/9/2016 3:32:19 PM

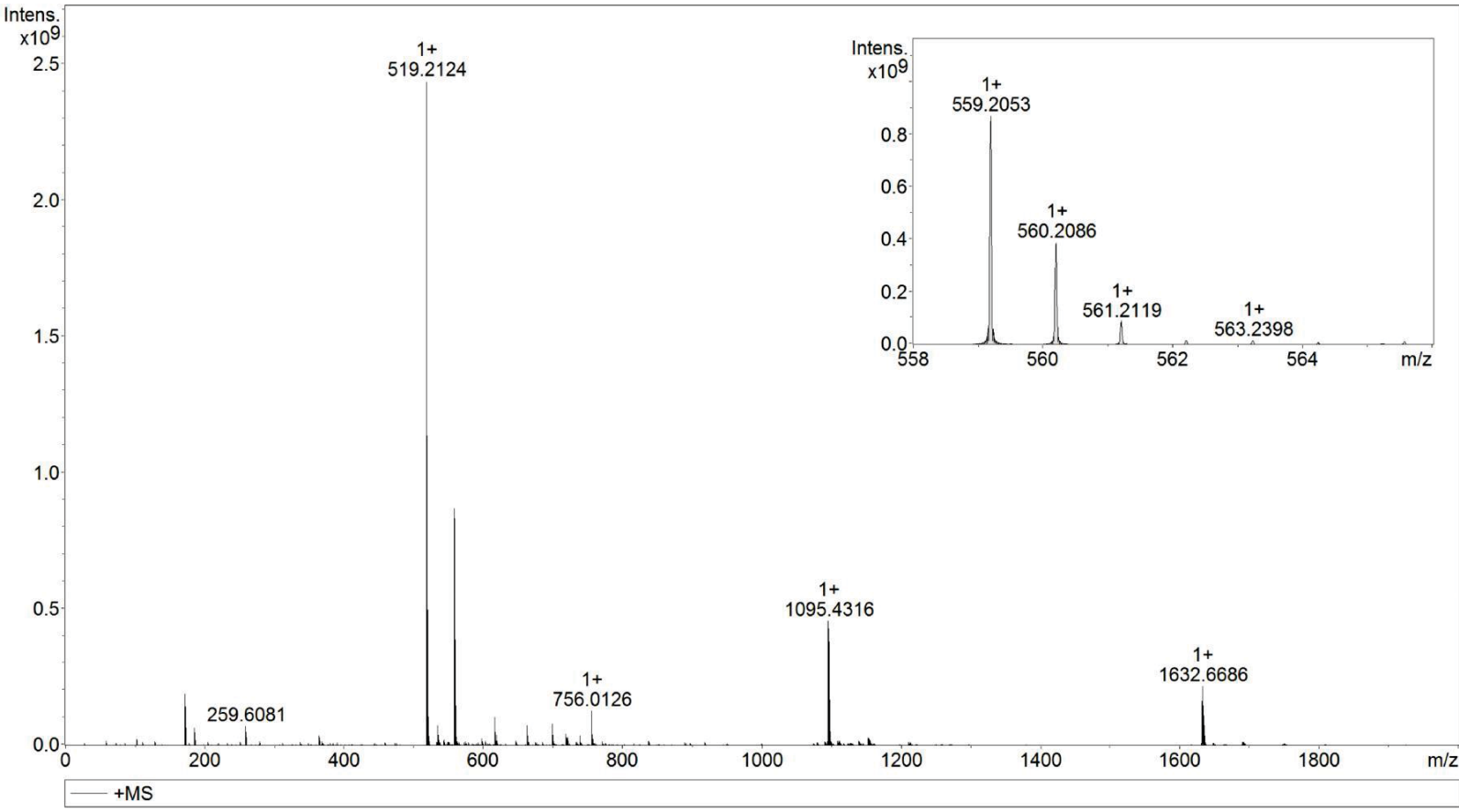


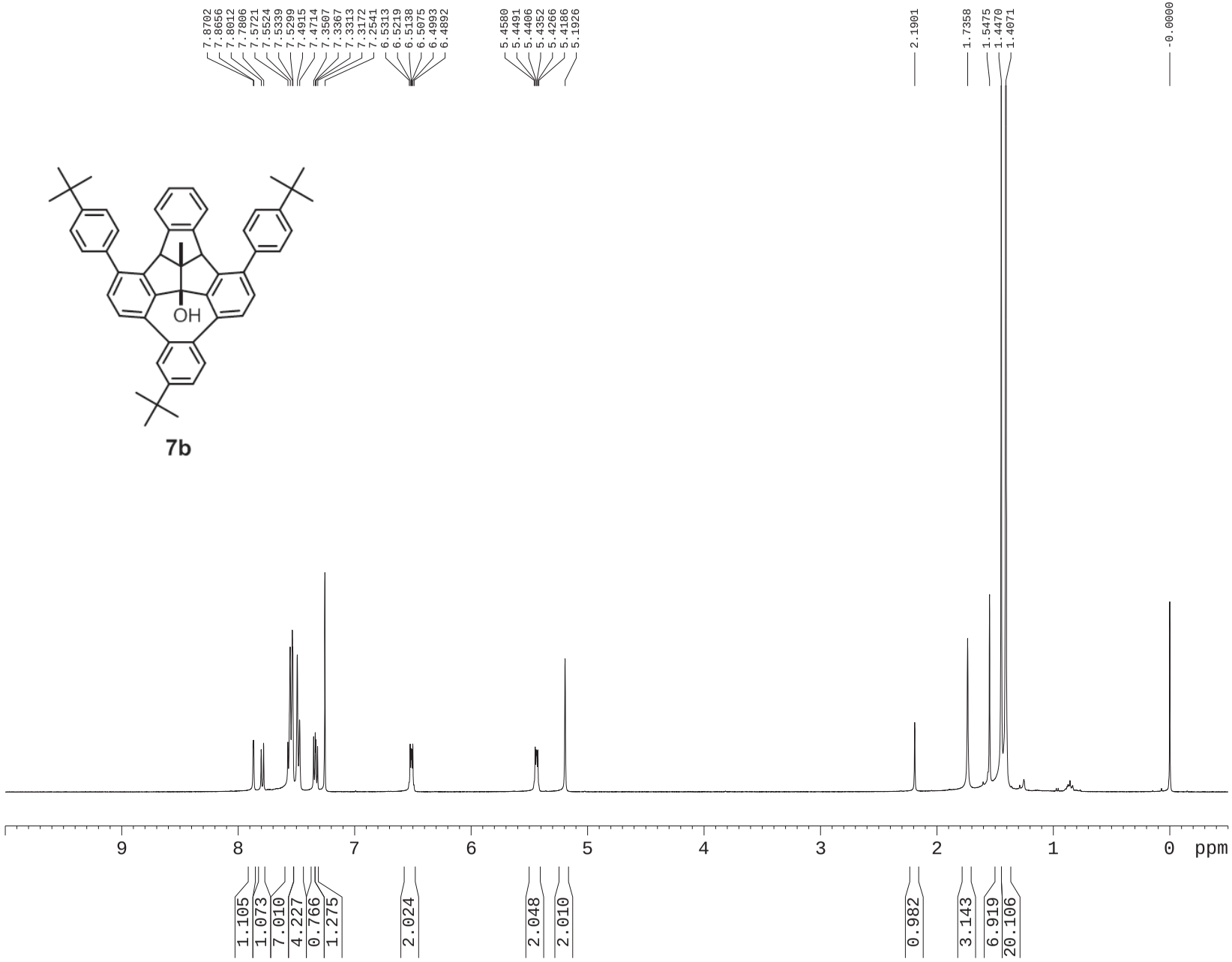
7a

Molecular Formula: C41H28O

Abundant Isotopic (theoretical)[M+Na]+ : 559.2032

Monoisotopic (theoretical)[M+Na]+ : 559.2032 (experimental)[M+Na]+: 559.2057 error(ppm): 4.47





AV400Q NMR

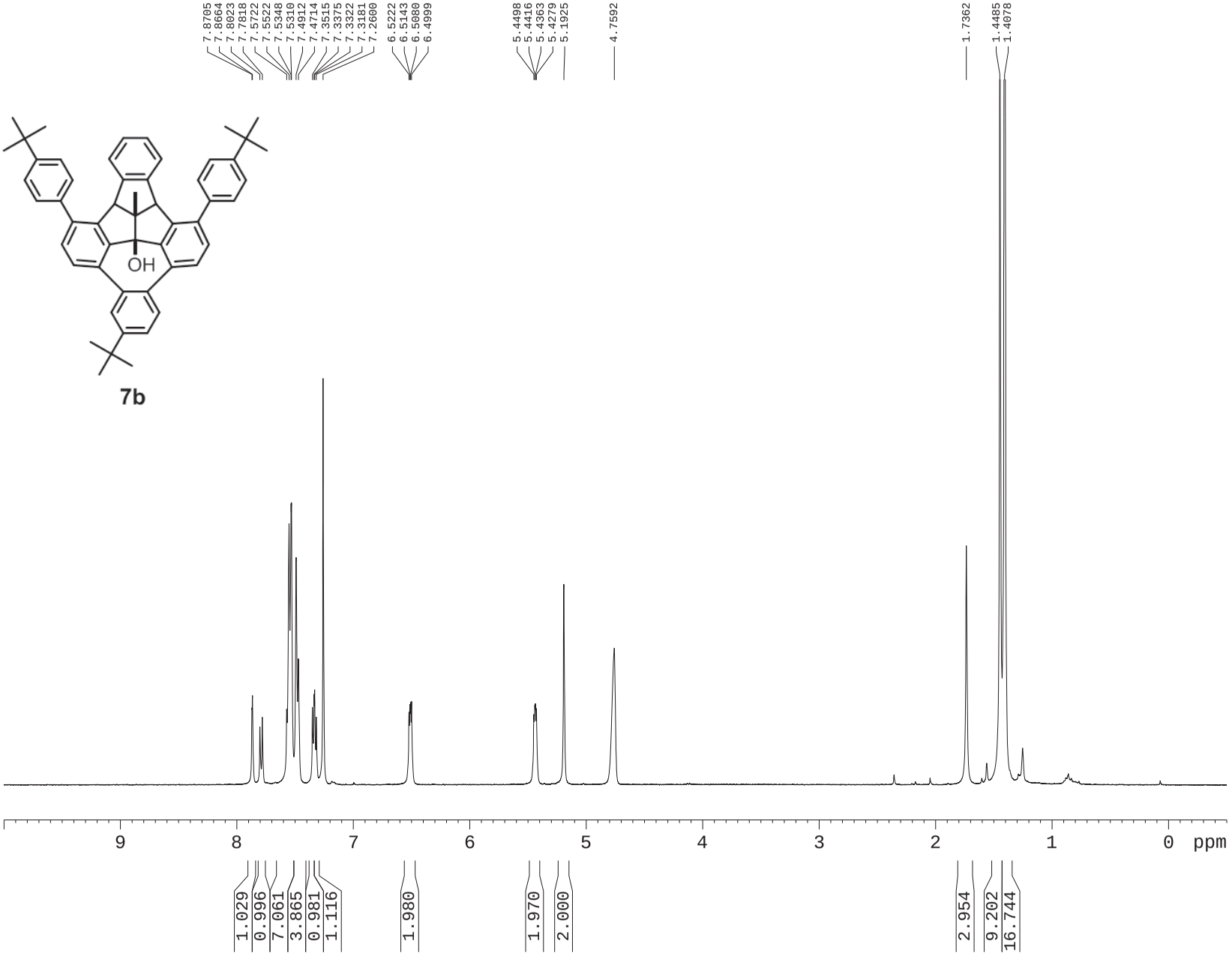
BRUKER

```
Current Data Parameters
NAME      Eric_9a_4_t_butyl_mono_OH_data_2
EXPNO                 111
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160212
Time              11.31 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG             zg30
TD                 32768
SOLVENT             CDCl3
NS                     27
DS                      2
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ               2.0447233 sec
RG                    128
DW                 62.400 usec
DE                  6.50 usec
TE                294.5 K
D1             1.00000000 sec
TD0                     1
SFO1         400.2324714 MHz
NUC1                   1H
P1                 12.80 usec
PLW1         13.56000042 W

F2 - Processing parameters
SI                  65536
SF           400.2300124 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

$CDCl_3/D_2O$ = 50/1



AV400Q NMR


```
Current Data Parameters
NAME      Eric_9a_4_tBu_mono_OH_data_D2O
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20170206
Time              18.47 h
INSTRUM             spect
PROBHD   Z108618_0257 (
PULPROG             zg30
TD                 32768
SOLVENT             CDCl3
NS                    49
DS                     2
SWH             8012.820 Hz
FIDRES             0.244532 Hz
AQ              2.0447233 sec
RG                   161
DW                  62.400 usec
DE                   6.50 usec
TE                 295.0 K
D1              1.00000000 sec
TD0                    1
SFO1          400.2324714 MHz
NUC1                  1H
P1                   12.00 usec
PLW1          13.56000042 W

F2 - Processing parameters
SI                 65536
SF            400.2300117 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
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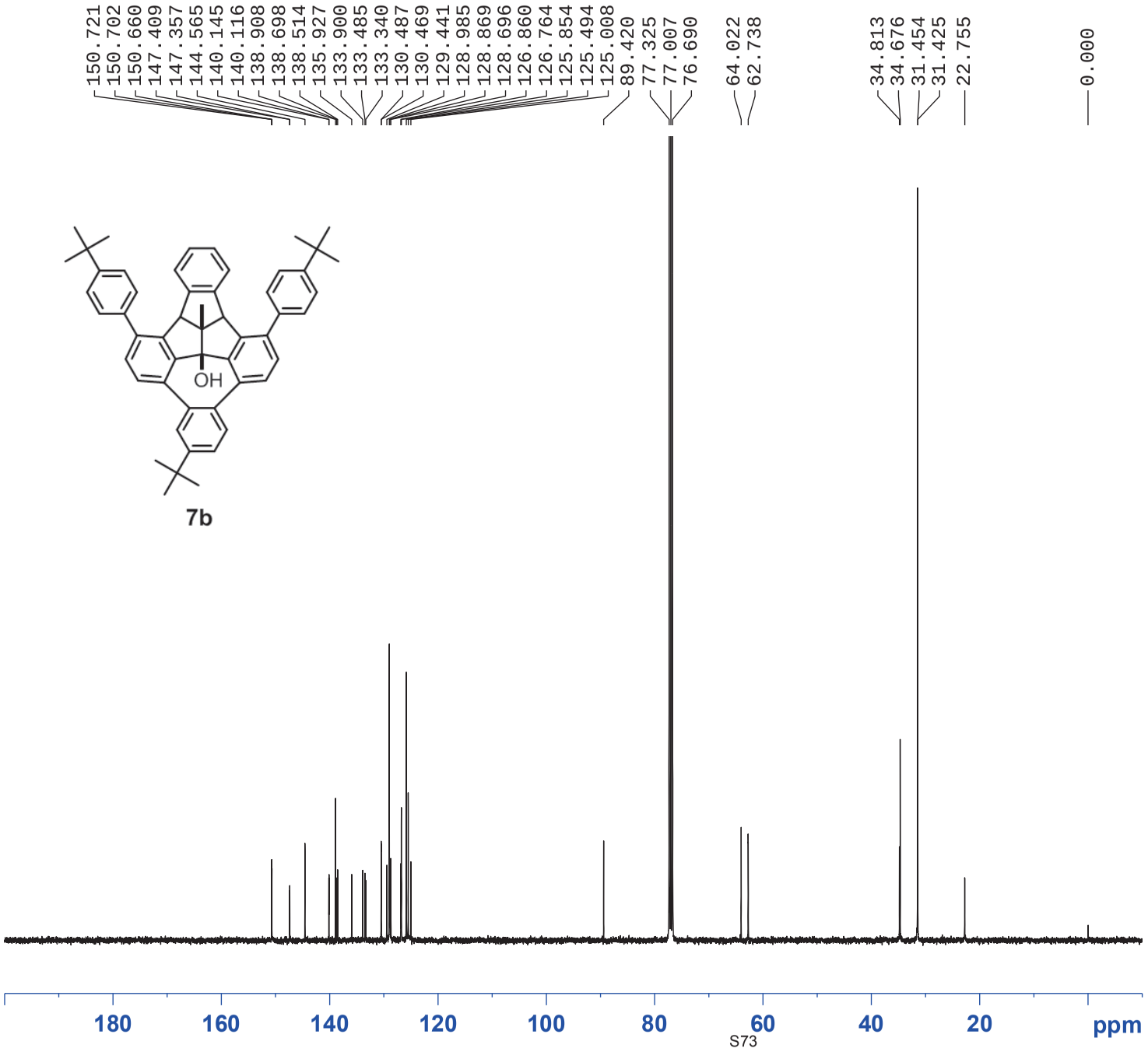
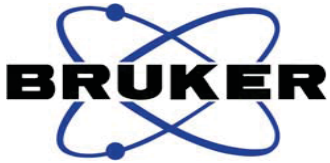


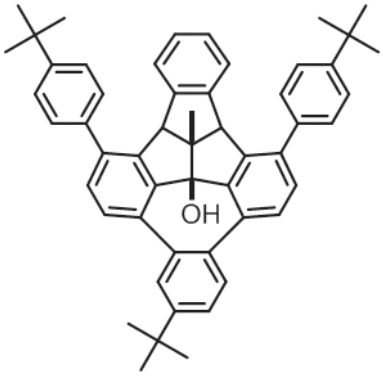



```
Current Data Parameters
NAME      Eric_9a_4_t_butyl_mono_OH_data_2
EXPNO                 222
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160212
Time              13.15 h
INSTRUM             spect
PROBHD    Z108618_0257 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1366
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                  161
DW               20.800 usec
DE                6.50 usec
TE               295.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1          100.6479773 MHz
NUC1                13C
P1                 9.50 usec
PLW1          55.34000015 W
SFO2          400.2316009 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2          13.56000042 W
PLW12          0.27428001 W
PLW13          0.13796000 W

F2 - Processing parameters
SI                32768
SF            100.6379179 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

9.4T FTICR MS Analysis Report

Faculty of Science, The Chinese University of Hong Kong

Sample No.	Eric03
Reference No.	CHF0026
Applicant name	Ip Ho Wang
Analysis Path	D:\Data\Service\MSonly\20160516\CHF\Eric03_000004.d
Instrument	solariX
Polarity	Positive
Acquired Scans	12
Operator	Winnie
Analysis Date	5/16/2016 2:42:22 PM

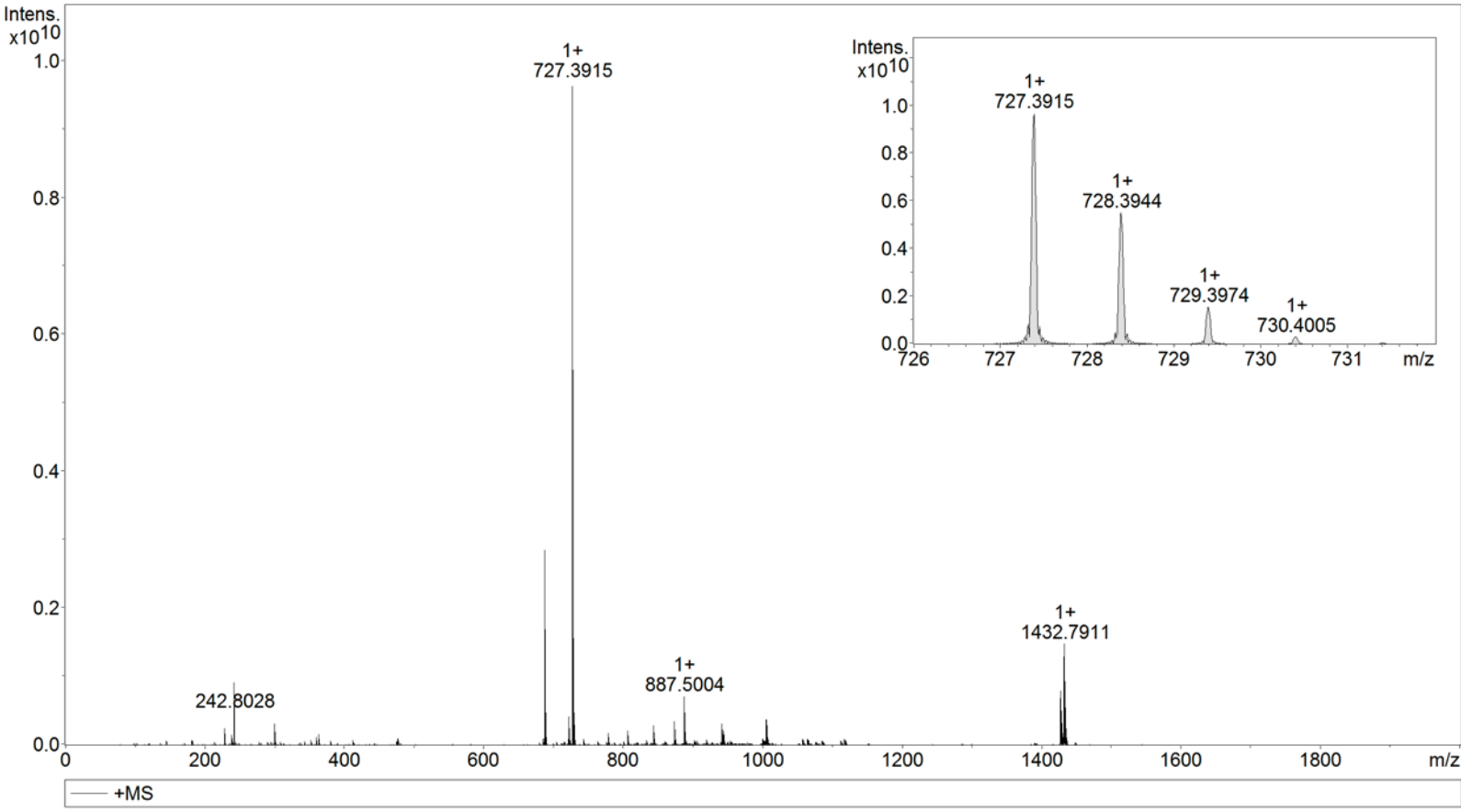


7b

Molecular Formula: C53H52O

Abundant Isotopic (theoretical)[M+Na]+ : 727.3910 (experimental)[M+Na]+: 727.3915 error(ppm): 0.69

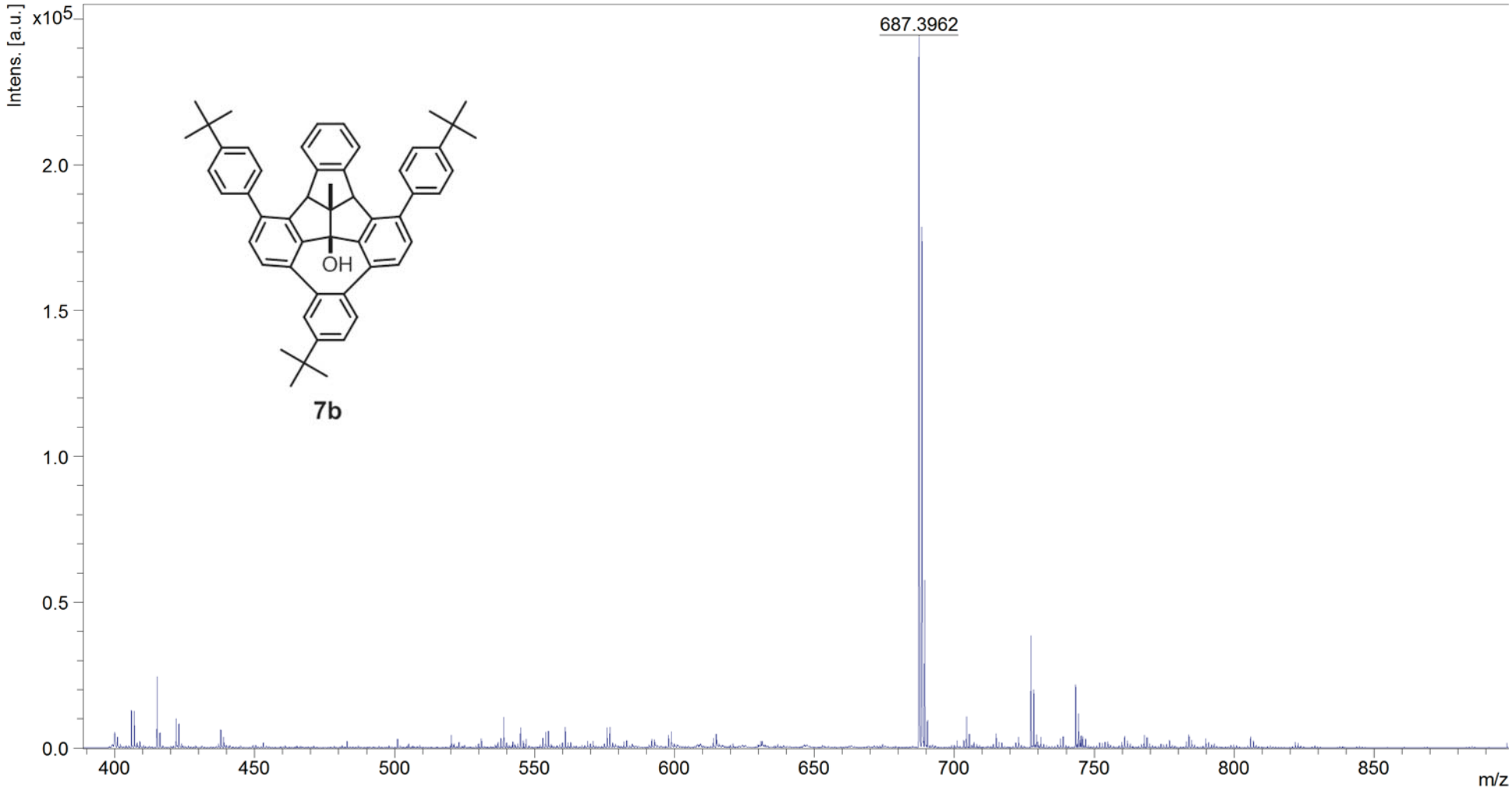
Monoisotopic (theoretical)[M+Na]+ : 727.3910



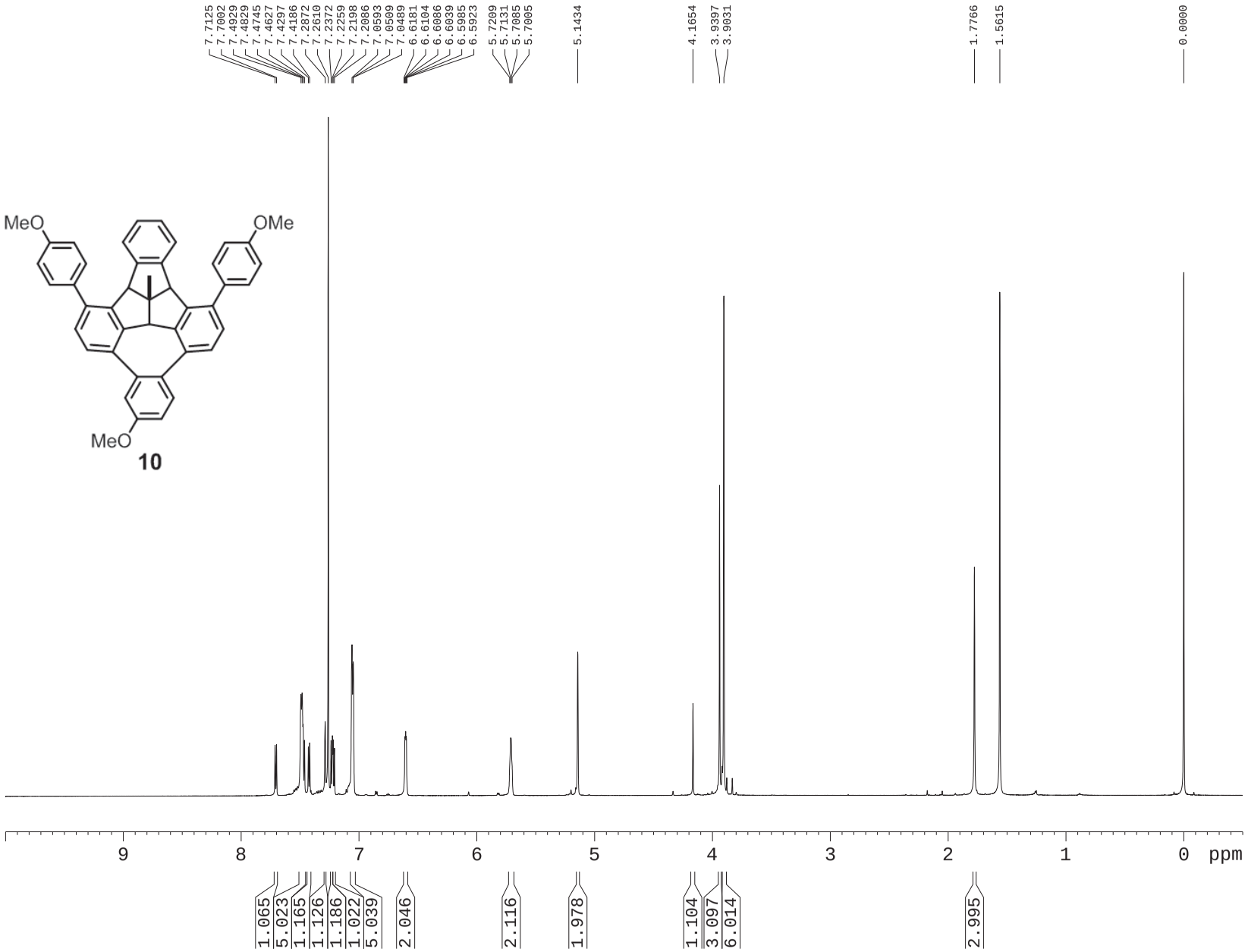
G:\MS Angew\18.Eric_9a_4tBu_mono_OH(A20) good\0_A22\1\1SRef

Comment 1

Comment 2



Bruker Daltonics flexAnalysis printed: 5/12/2016 2:25:46 PM



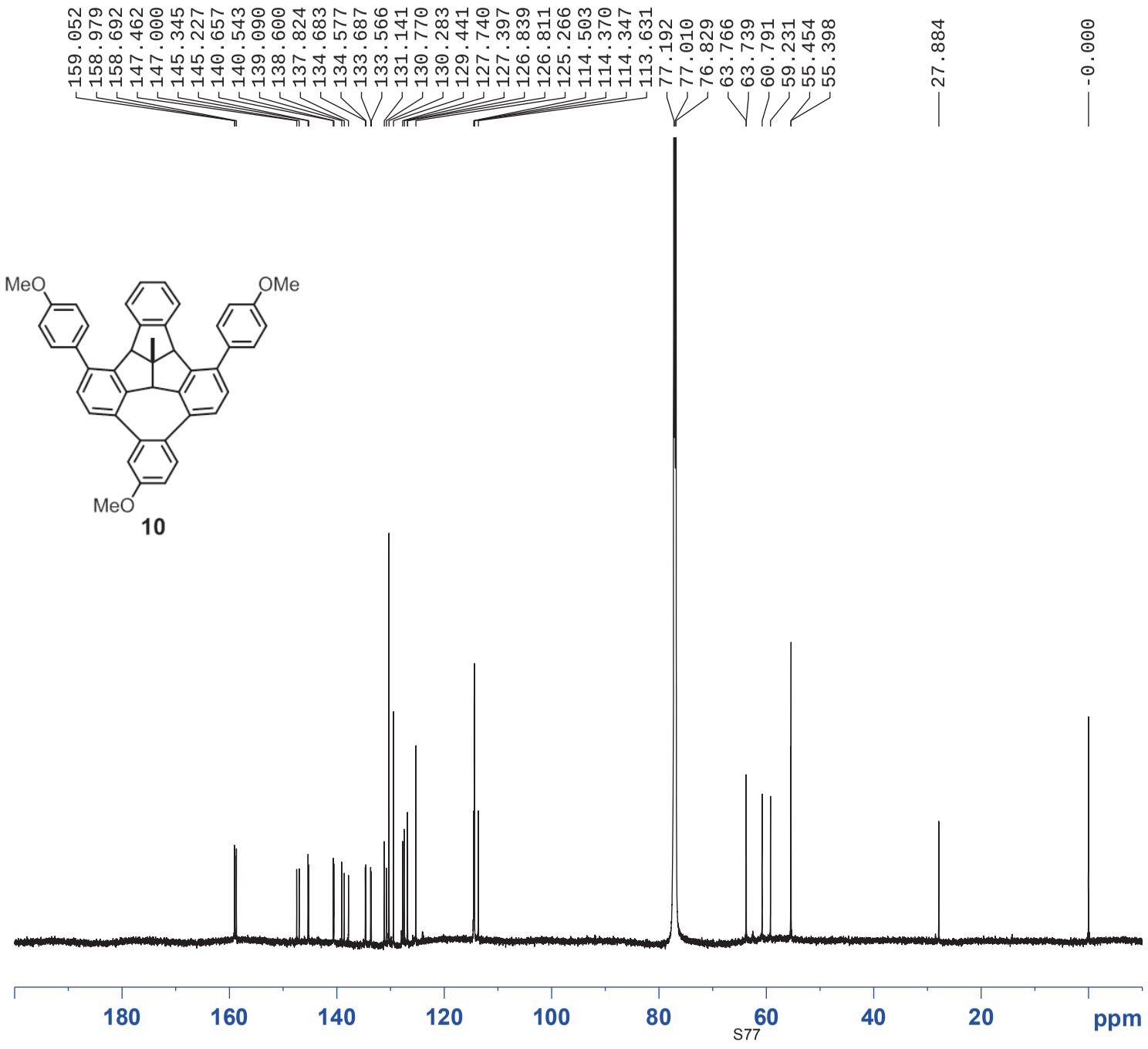
AV400Q NMR

Current Data Parameters
NAME Eric_9a_4_OMe_data
EXPNO 111
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160126
Time 16.56
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 41
DS 2
SWH 14097.744 Hz
FIDRES 0.430229 Hz
AQ 1.1621717 sec
RG 9
DW 35.467 usec
DE 10.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 700.1643238 MHz
NUC1 1H
P1 8.45 usec
PLW1 7.00000000 W

F2 - Processing parameters
SI 65536
SF 700.1600158 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

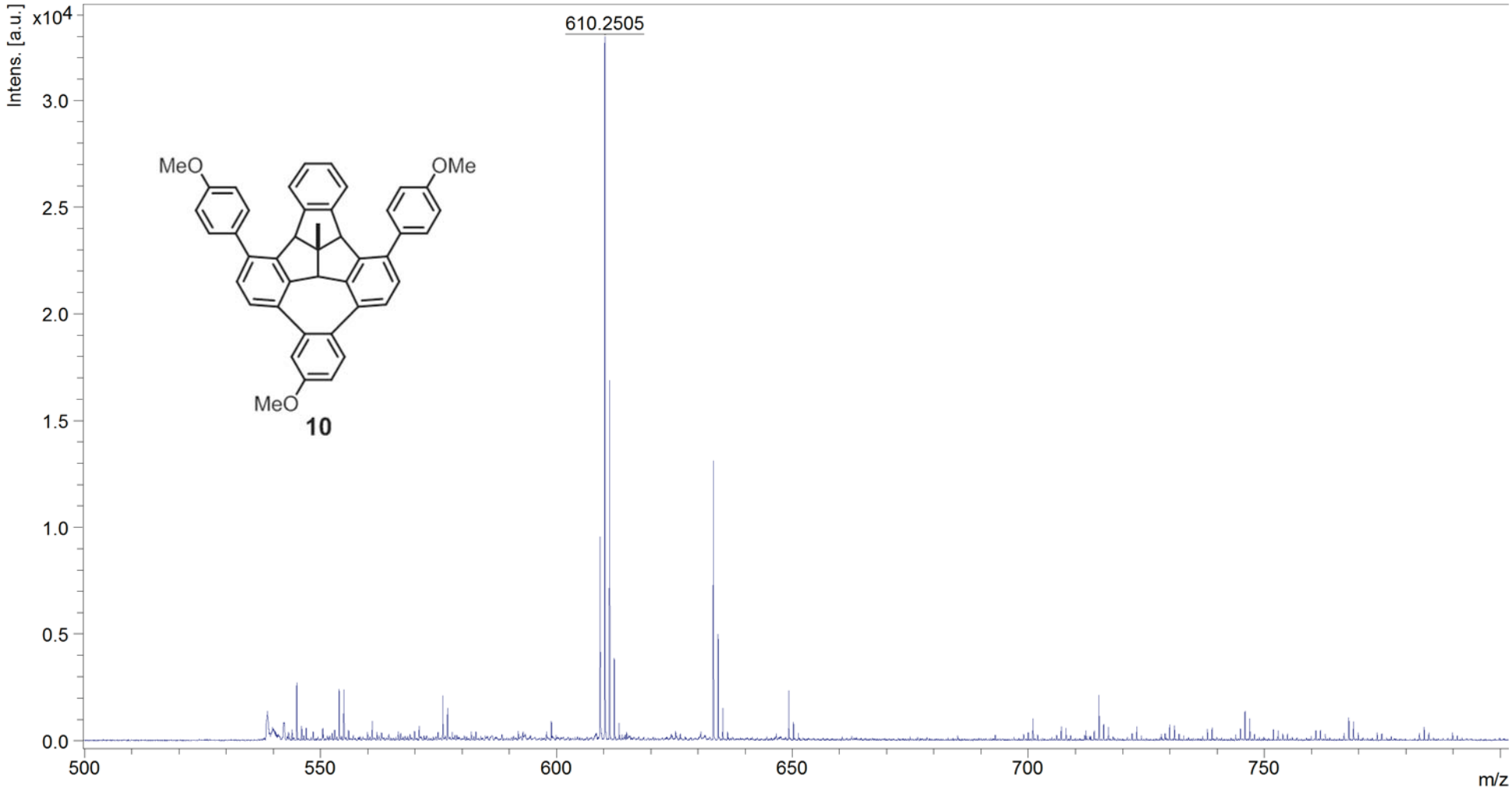



```
Current Data Parameters
NAME      Eric_9a_4_OMe_data
EXPNO                   3333
PROCNO                     1

F2 - Acquisition Parameters
Date_           20160126
Time              17.23
INSTRUM           spect
PROBHD   5 mm CPTCI 1H-
PULPROG          zgpg30
TD                65400
SOLVENT           CDCl3
NS                21782
DS                    4
SWH           41666.668 Hz
FIDRES         0.637105 Hz
AQ             0.7848000 sec
RG                  203
DW               12.000 usec
DE                18.00 usec
TE                298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        176.0729767 MHz
NUC1               13C
P1               13.00 usec
PLW1        88.00000000 W

======== CHANNEL f2 ========
SFO2        700.1628006 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2              65.00 usec
PLW2          7.00000000 W
PLW12         0.11830000 W
PLW13         0.04998200 W

F2 - Processing parameters
SI                 32768
SF          176.0553767 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

G:\MS Angew\19.Eric_8a_4_OMe_mono(C5)\0_C5\1\1SRef

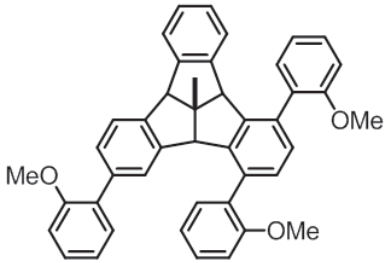
Comment 1

Comment 2



Bruker Daltonics flexAnalysis printed: 5/12/2016 2:26:59 PM

22 °C

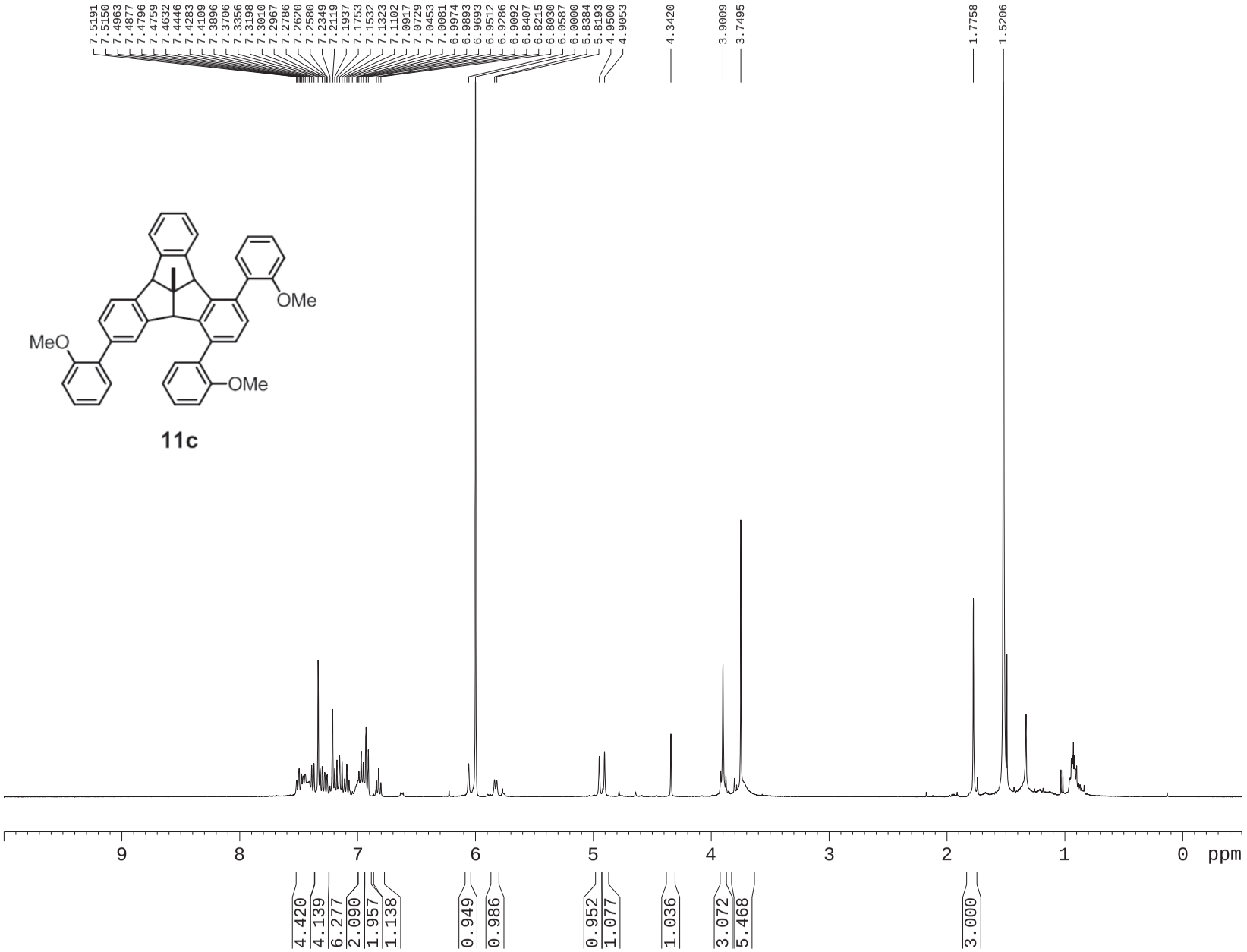


11c

AV400Q NMR

```
Current Data Parameters
NAME      Eric_9a_2_OMe_data
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20160419
Time                  12.35 h
INSTRUM               spect
PROBHD   Z824601_0021 (
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       51
DS                        2
SWH                8012.820 Hz
FIDRES             0.244532 Hz
AQ                 2.0447233 sec
RG                      203
DW                   62.400 usec
DE                    6.50 usec
TE                   294.8 K
D1                 1.00000000 sec
TD0                       1
SFO1             400.1324708 MHz
NUC1                     1H
P1                    15.00 usec
PLW1              8.31000042 W

F2 - Processing parameters
SI                    65536
SF               400.1305137 MHz
WDW                      EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```

60 °C



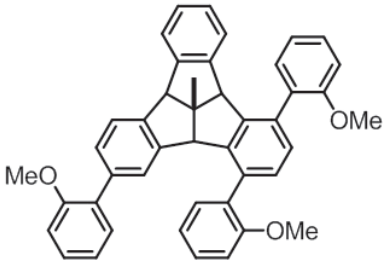
AV400Q NMR


```
Current Data Parameters
NAME      Eric_9a_2_OMe_data
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20160419
Time                 13.03 h
INSTRUM              spect
PROBHD   Z824601_0021 (
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                     128
DS                       2
SWH               8012.820 Hz
FIDRES               0.244532 Hz
AQ                2.0447233 sec
RG                     203
DW                  62.400 usec
DE                   6.50 usec
TE                  333.3 K
D1               1.00000000 sec
TD0                      1
SFO1            400.1324708 MHz
NUC1                    1H
P1                   15.00 usec
PLW1             8.31000042 W

F2 - Processing parameters
SI                   65536
SF              400.1305124 MHz
WDW                     EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```

60 °C

155.875
146.219
145.037
144.576
136.879
135.052
132.215
131.122
131.063
130.967
130.101
129.837
128.951
128.620
127.992
126.920
126.756
126.080
123.796
122.903
120.915
120.250
120.198
111.358
110.558
99.427
73.938
73.780
73.623
62.498
60.260
60.145
59.359
55.376
55.216
55.117

28.046



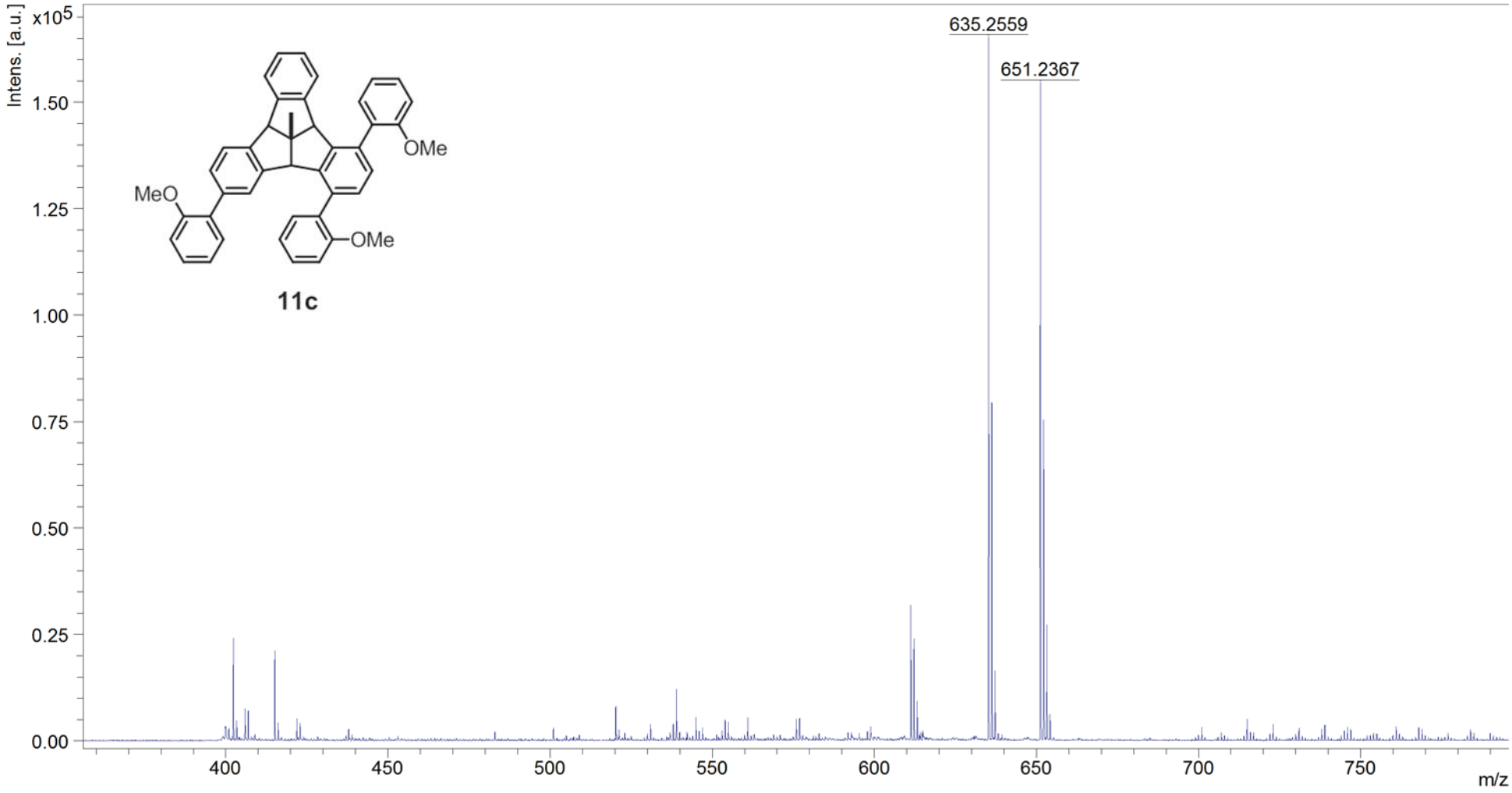
11c


```
Current Data Parameters
NAME      Eric_9a_2_OMe_700
EXPNO                 3333
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160512
Time              12.24
INSTRUM             spect
PROBHD   5 mm CPTCI 1H-
PULPROG           zgpg30
TD                65400
SOLVENT             CDCl3
NS                25635
DS                    4
SWH           41666.668 Hz
FIDRES         0.637105 Hz
AQ             0.7848000 sec
RG                  203
DW                12.000 usec
DE                18.00 usec
TE                333.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         176.0729767 MHz
NUC1                13C
P1                13.00 usec
PLW1         88.00000000 W

======== CHANNEL f2 ========
SFO2         700.1628006 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             65.00 usec
PLW2          7.00000000 W
PLW12         0.11830000 W
PLW13         0.04998200 W

F2 - Processing parameters
SI                32768
SF           176.0556367 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

G:\MS Angew\20.Eric_9a_2_OMe_rearrange(A24)\0_A24\1\1SRef

Comment 1

Comment 2



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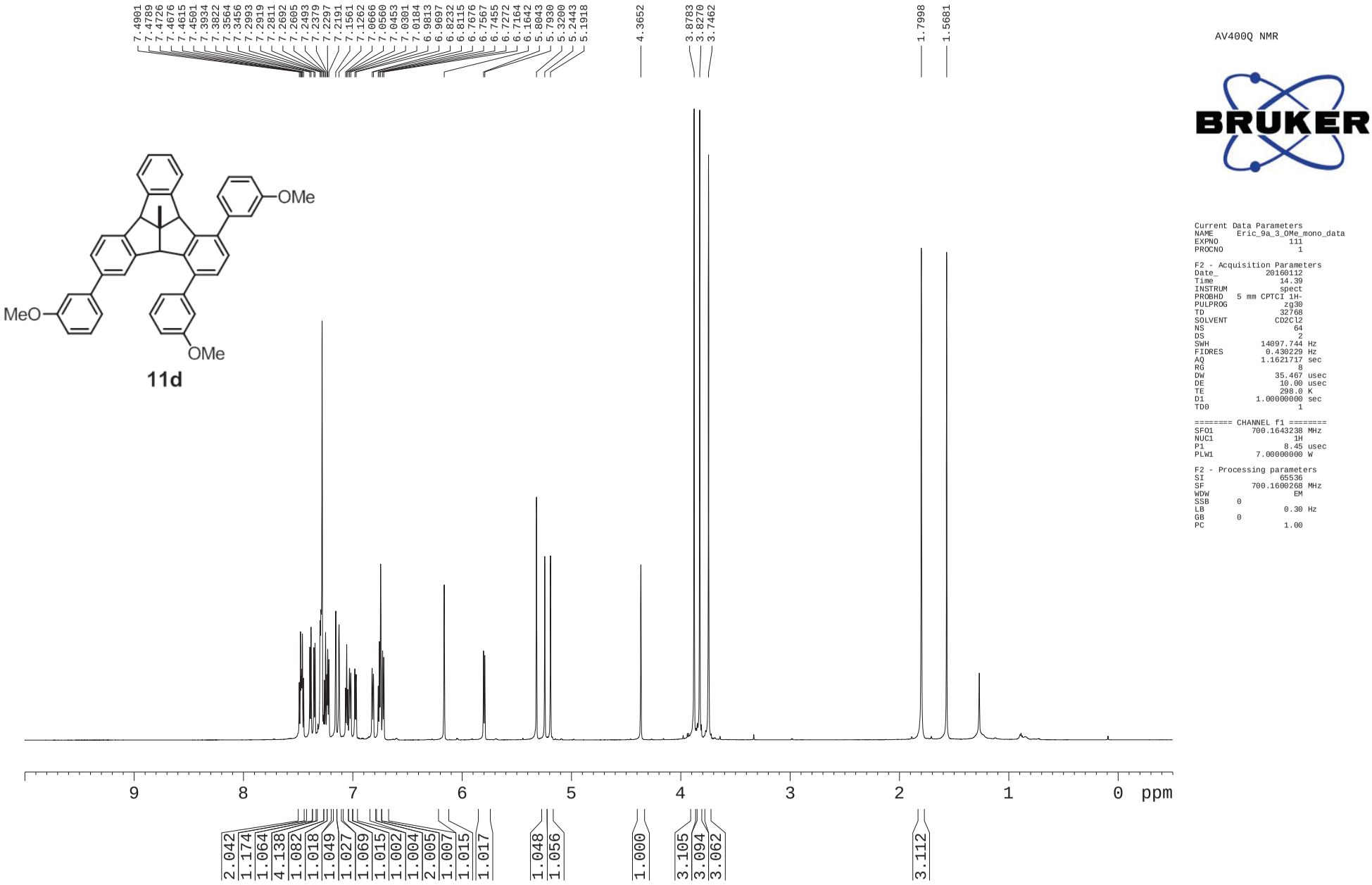
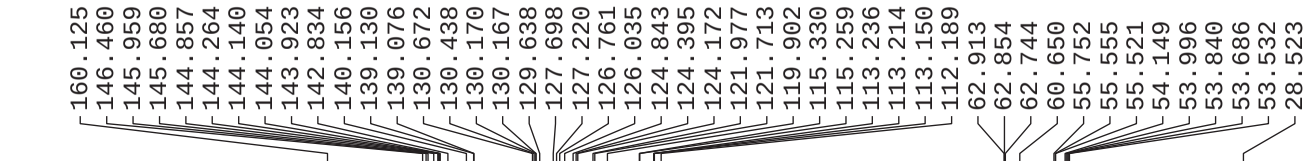
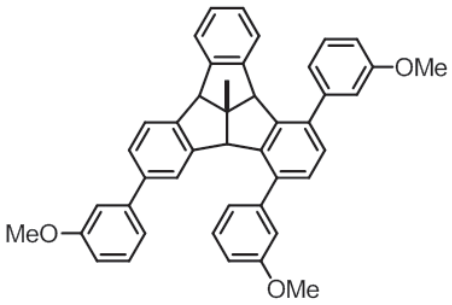


AV400Q NMR

BRUKER

```
Current Data Parameters
NAME      Eric_9a_3_OMe_mono_data
EXPNO                 111
PROCNO                  1

F2 - Acquisition Parameters
Date_              20160112
Time                 14.39
INSTRUM              spect
PROBHD    5 mm CPTCI 1H-
PULPROG              zg30
TD                  32768
SOLVENT            CD2Cl2
NS                     64
DS                      2
SWH             14097.744 Hz
FIDRES              0.430229 Hz
AQ                1.1621717 sec
RG                      8
DW                 35.467 usec
DE                 10.00 usec
TE                 298.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1           700.1643238 MHz
NUC1                   1H
P1                  8.45 usec
PLW1           7.00000000 W

F2 - Processing parameters
SI                  65536
SF             700.1600268 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```





11d


```
Current Data Parameters
NAME      Eric_9a_3_OMe_mono_data
EXPNO                3333
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160112
Time               14.53
INSTRUM            spect
PROBHD   5 mm CPTCI 1H-
PULPROG          zgpg30
TD                65536
SOLVENT           CD2Cl2
NS                 2000
DS                    4
SWH           41666.668 Hz
FIDRES         0.635783 Hz
AQ            0.7864320 sec
RG                  203
DW               12.000 usec
DE               18.00 usec
TE               298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         176.0729767 MHz
NUC1                13C
P1                13.00 usec
PLW1        88.00000000 W

======== CHANNEL f2 ========
SFO2         700.1628006 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             65.00 usec
PLW2          7.00000000 W
PLW12         0.11830000 W
PLW13         0.04998200 W

F2 - Processing parameters
SI                 32768
SF           176.0553087 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Universität Bielefeld

Fakultät für Chemie - Organische Chemie I
Abteilung Massenspektrometrie F02-217

Universitätsstraße 25
33501 Bielefeld
☎ 0521 - 106-2108

Accurate Mass Measurement

Sample Name :	Er-12	**Group :**	OC1
Sample Supplier :	Er. Wang		
Sample Filename :	MFB2016_026.		

Instrument :	Autospec
Ionisation Method :	EI
Matching Method :	HR with calibration PFK
Resolution :	> 7000
Substance Inlet :	EI Schubstange

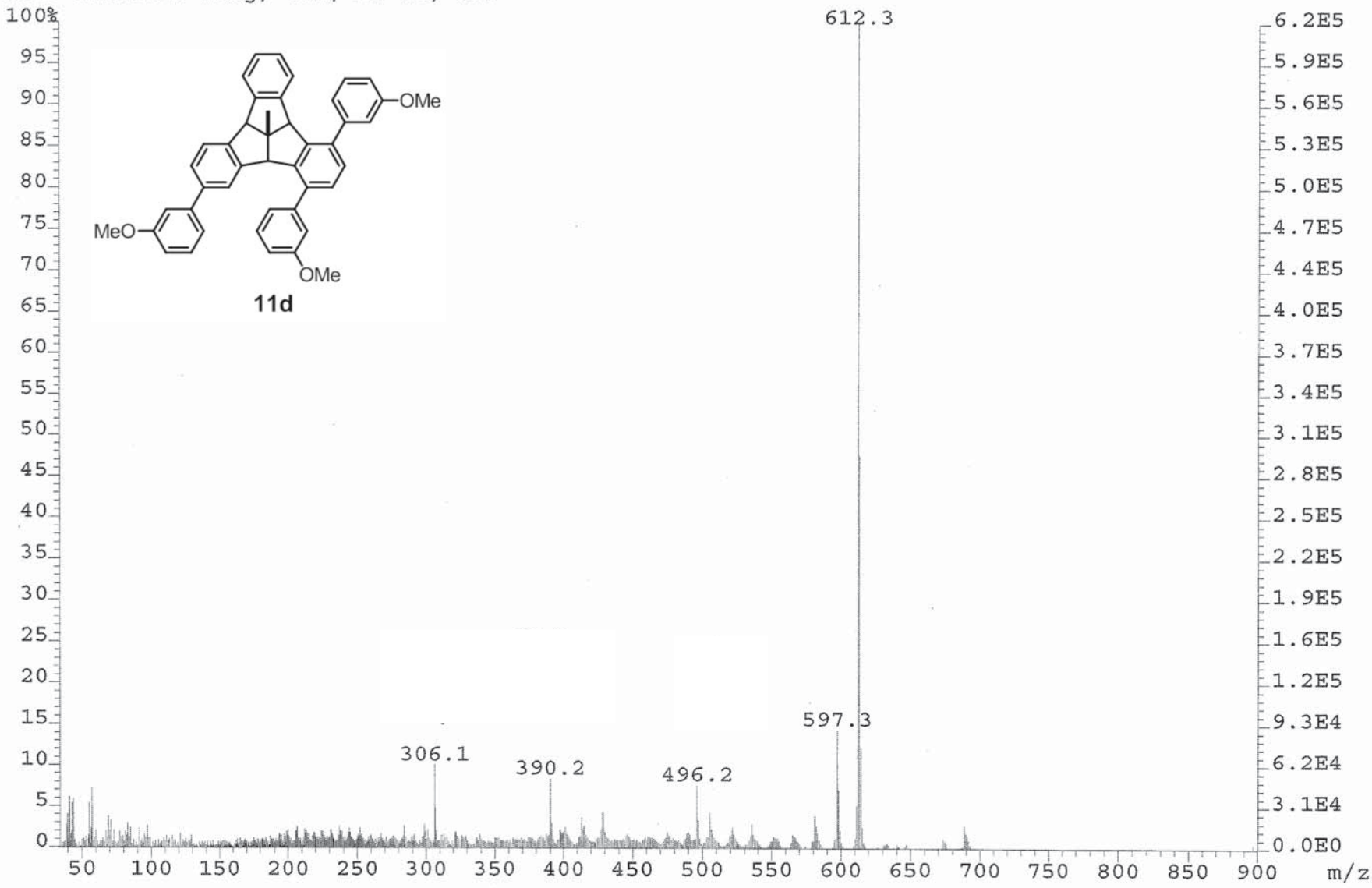
11d

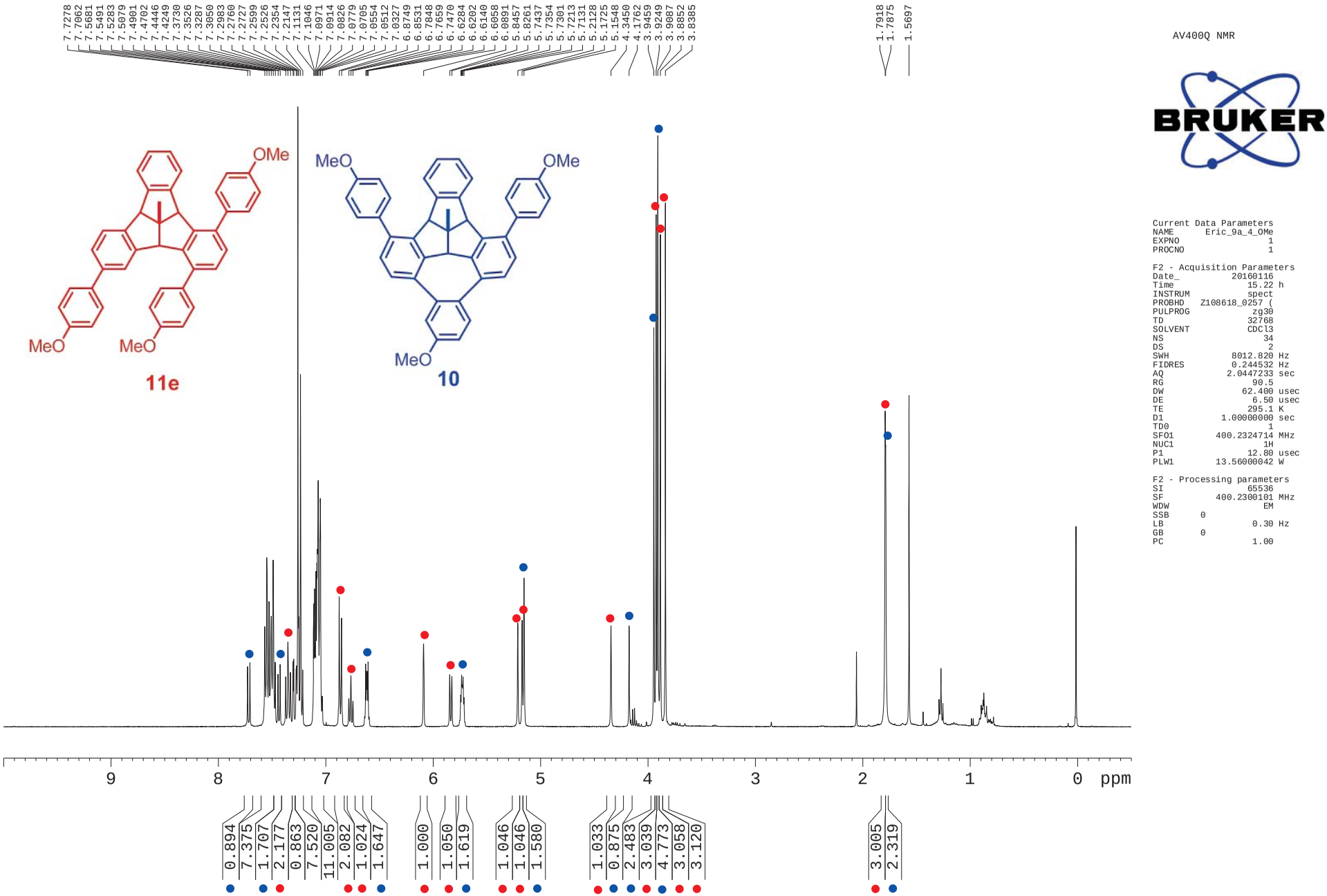
Measured PFK Mass(es) :	616,96327	**Deviation [mmu] :**	0,49 [mmu]
Measured Ion Mass(es) :	612,26541	**Deviation [ppm] :**	0,80 [ppm]
Calculated Ion Mass(es) :	612,26590		
Potential Molecular Formula :	C44H36O3+		

Comment : Measured aud calculated masses are true ion masses, taking into account the mass of lost (or added) electrons.

Bielefeld, 17.02.2016

File:EI2016_074 Ident:159_181 Win 500PPM Acq:15-FEB-2016 22:50:05 +17:35 Cal:EI_POS_CAL_900
AutoSpec EI+ Magnet BpM:612 BpI:622353 TIC:6185087 Flags:HALL
File Text:Er. Wang, OC1, Er-12, HWP



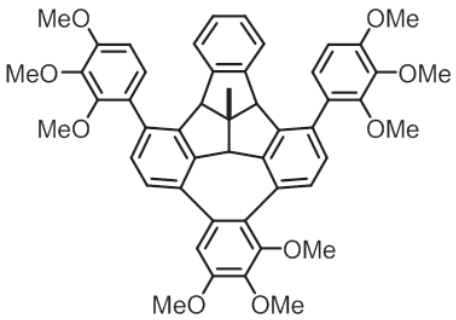


AV400Q NMR

BRUKER

```
Current Data Parameters
NAME        Eric_9a_4_OMe
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160116
Time                15.22 h
INSTRUM             spect
PROBHD    Z108618_0257 (
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     34
DS                      2
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ               2.0447233 sec
RG                   90.5
DW                 62.400 usec
DE                  6.50 usec
TE                 295.1 K
D1              1.00000000 sec
TD0                     1
SFO1           400.2324714 MHz
NUC1                   1H
P1                  12.80 usec
PLW1           13.56000042 W

F2 - Processing parameters
SI                  65536
SF             400.2300101 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```

22 °C

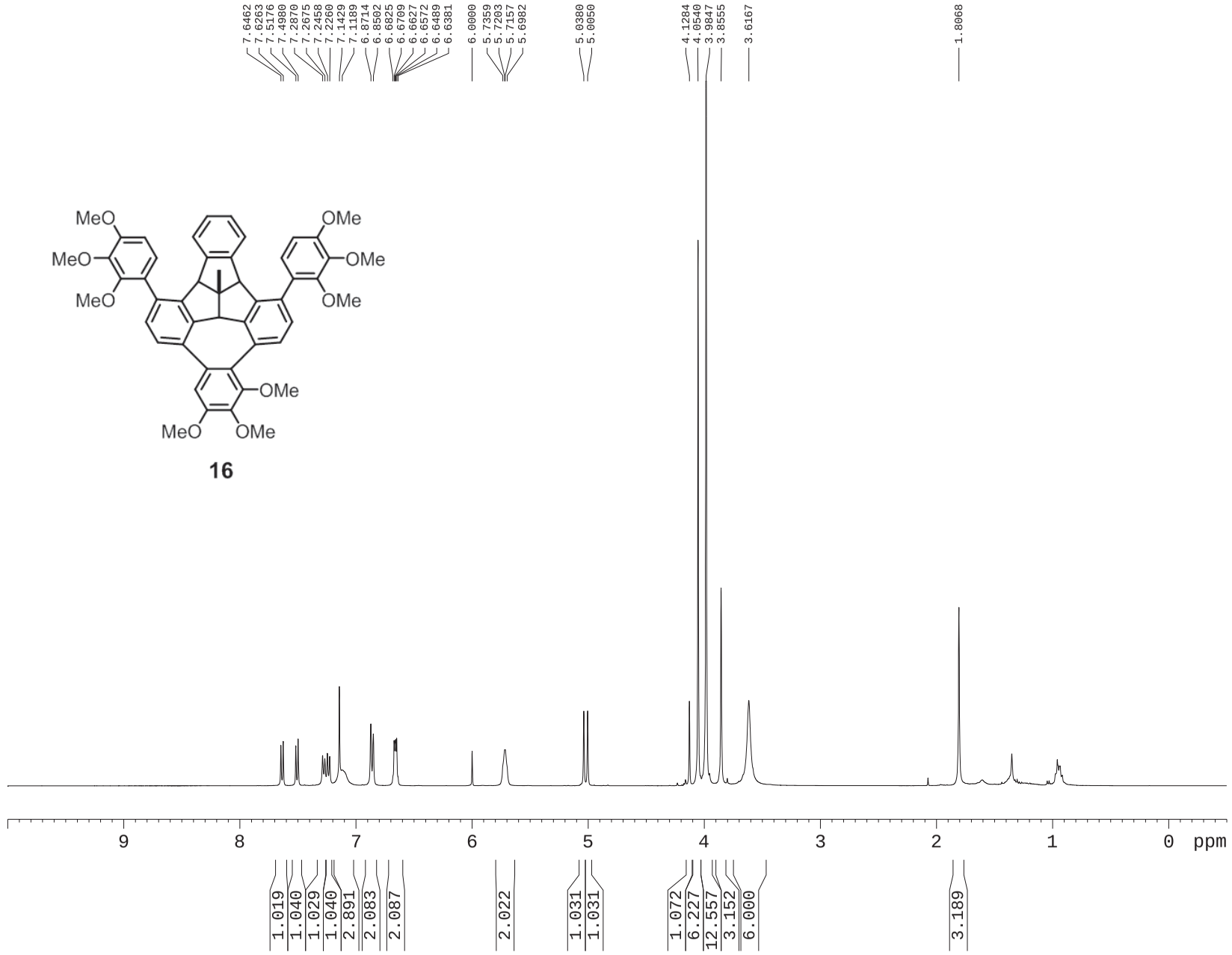


16

AV400Q NMR

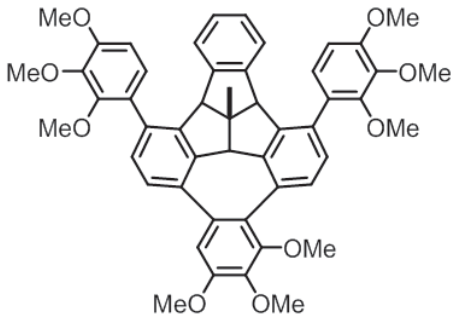
```
Current Data Parameters
NAME      Eric_9a_2_3_4_OMe_mono_data
EXPNO                 111
PROCNO                  1

F2 - Acquisition Parameters
Date_           20170126
Time              14.41 h
INSTRUM             spect
PROBHD     Z824601_0021 (
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    19
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              2.0447233 sec
RG                  90.5
DW                62.400 usec
DE                 6.50 usec
TE                295.3 K
D1           1.00000000 sec
TD0                    1
SFO1         400.1324708 MHz
NUC1                 1H
P1                15.00 usec
PLW1          8.31000042 W

F2 - Processing parameters
SI                 65536
SF           400.1305134 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

60 °C

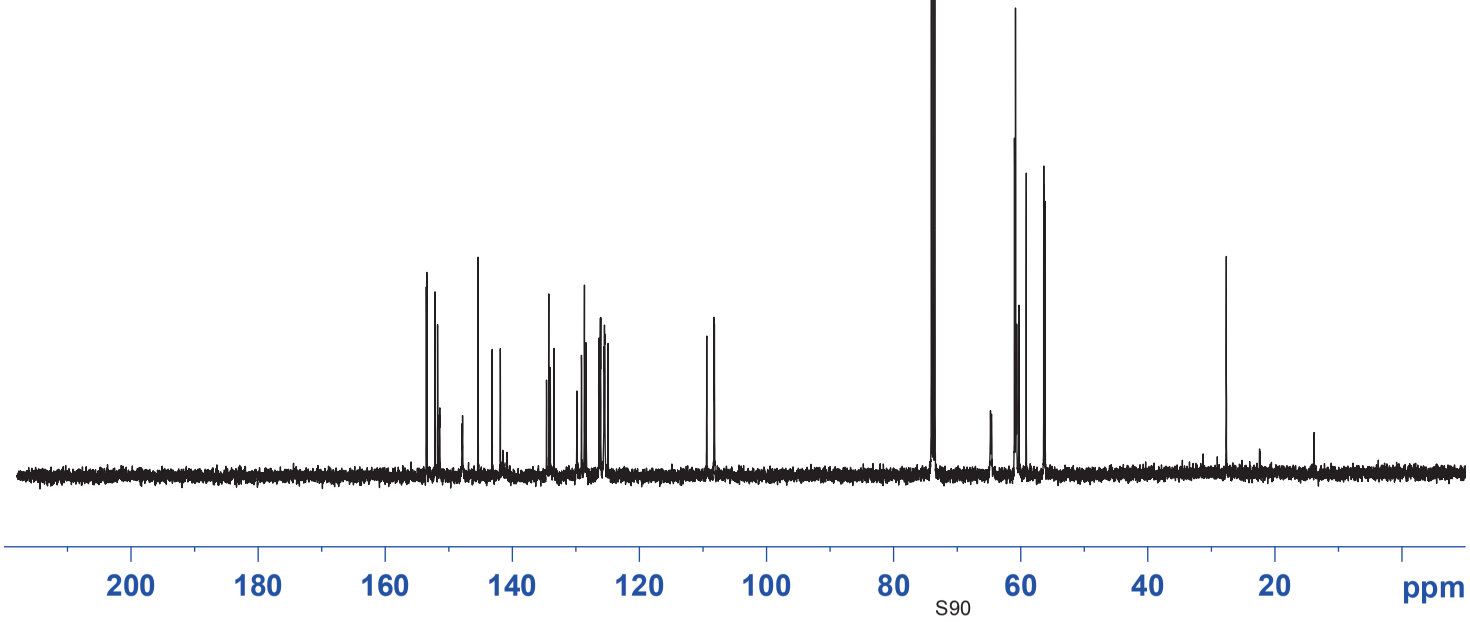

7.6462
7.6263
7.5176
7.4980
7.2870
7.2675
7.2458
7.2260
7.1429
7.1189
6.8714
6.8502
6.8825
6.6709
6.6627
6.6572
6.6489
6.6381
6.0000
5.7359
5.7203
5.7157
5.6982
5.0380
5.0050
4.1284
4.0540
3.9847
3.8555
3.6167
1.8068
AV400Q NMR
BRUKER
MeO
MeO
MeO
OMe
OMe
OMe
OMe
OMe
MeO
OMe
16
Current Data Parameters
NAME Eric_9a_2_3_4_OMe_mono_data
EXPNO 555
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170126
Time 16.40 h
INSTRUM spect
PROBHD Z824601_0021 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447233 sec
RG 64
DW 62.400 usec
DE 6.50 usec
TE 333.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.31000042 W
F2 - Processing parameters
SI 65536
SF 400.1305128 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.019
1.040
1.029
1.040
2.891
2.083
2.087
2.022
1.031
1.031
1.072
6.227
12.557
3.152
6.000
3.189

60 °C

153.533
153.439
152.166
151.766
151.489
151.423
147.917
147.833
145.397
143.204
143.182
141.887
141.498
140.832
134.620
134.271
134.028
133.433
129.828
129.116
128.655
128.418
128.381
126.359
126.144
126.099
125.579
125.504
125.458
124.939
109.410
108.262
108.201
74.055
73.780
73.504
64.757
64.593
60.977
60.812
60.718
60.630
60.281
59.127
56.378
56.355
56.153
27.645



16



Current Data Parameters
NAME Eric_9a_2_3_4_OMe_mono_data
EXPNO 444
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170126
Time 16.02 h
INSTRUM spect
PROBHD Z824601_0021 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 600
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 333.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 9.50 usec
PLW1 41.25000000 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 8.31000042 W
PLW12 0.23083000 W
PLW13 0.11611000 W

F2 - Processing parameters
SI 32768
SF 100.6129249 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

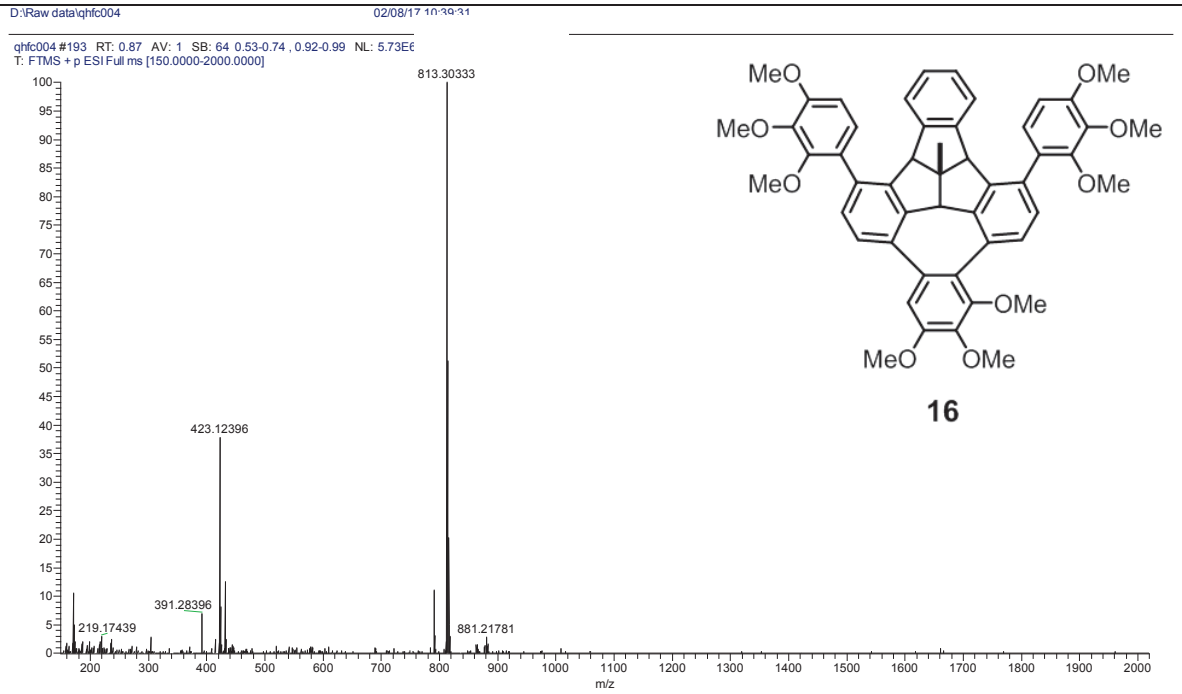
Thermo QEFMS Analysis Report

Analysis Info

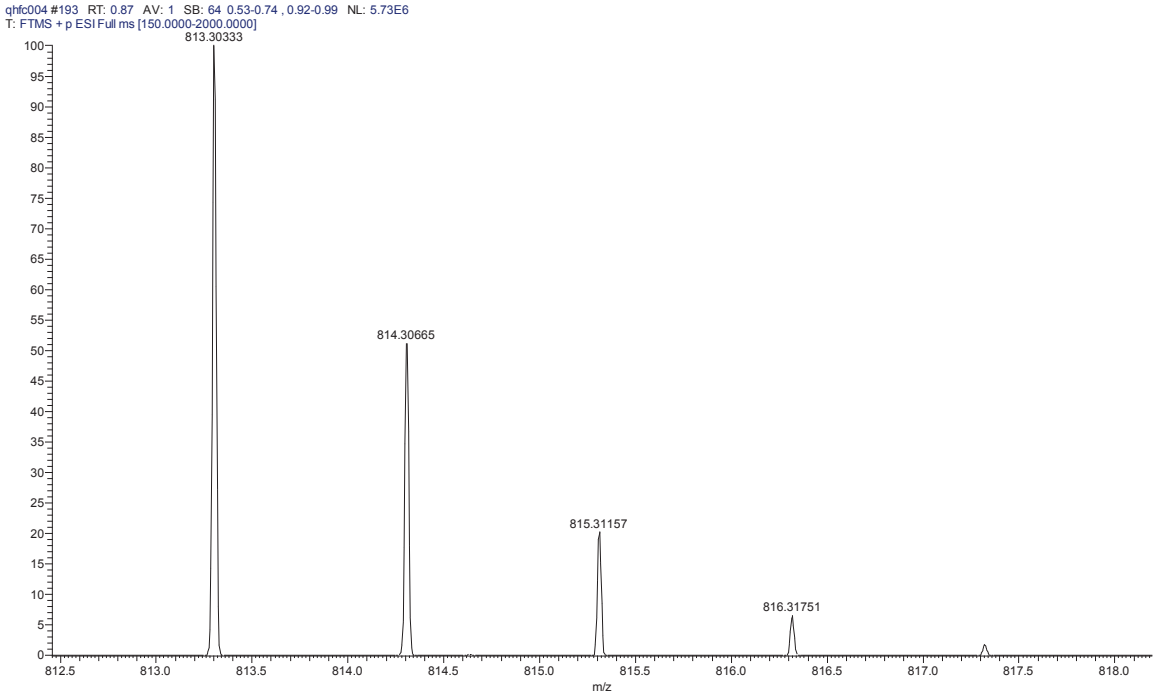
Sample Name :	Eric_27	Reference No.:	Qhfc004
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

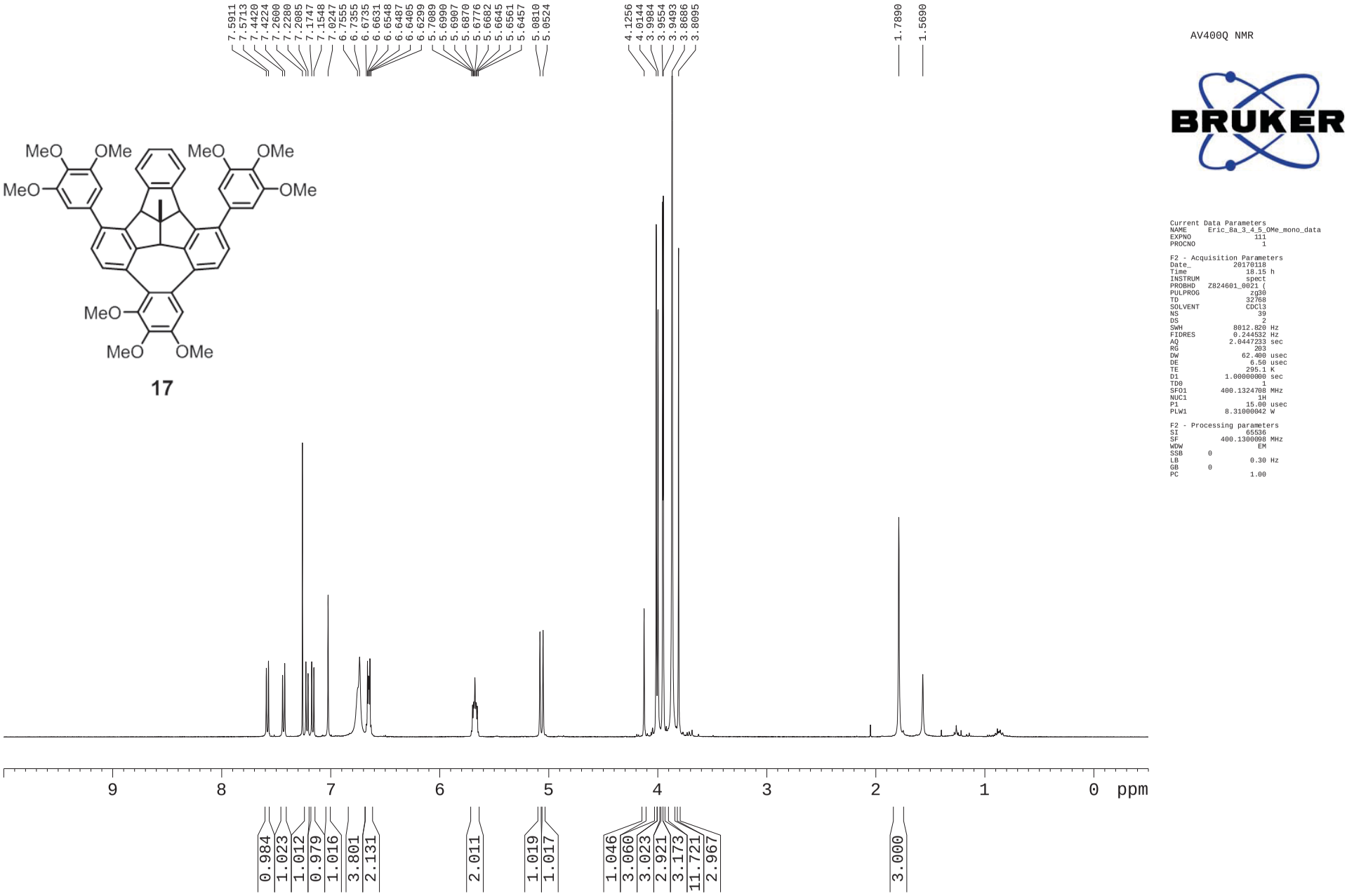
Accurate Mass Measurement

Molecular formula :	$C_{50}H_{46}O_9$
Experimental Mass $[M+Na]^+$:	813.30333
Theoretical Mass $[M+Na]^+$:	813.30340
Error (ppm) :	0.0



16





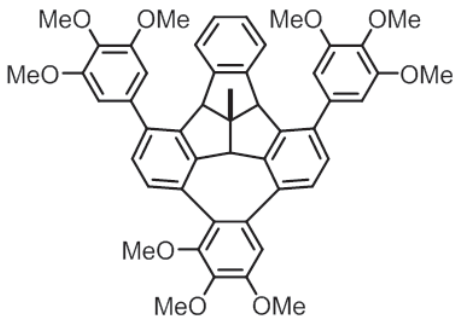
AV400Q NMR

Current Data Parameters
NAME Eric_8a_3_4_5_OMe_mono_data
EXPNO 111
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170118
Time 18.15 h
INSTRUM spect
PROBHD Z824601_0021 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 39
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447233 sec
RG 203
DW 62.400 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.31000042 W

F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

153.775
153.732
152.484
151.909
148.439
148.329
145.011
144.946
141.880
140.149
139.500
138.831
138.218
138.082
137.882
137.785
137.735
134.434
134.219
130.132
129.224
128.852
128.132
127.350
126.971
126.876
125.778
125.612
124.800
108.790
106.621
77.479
77.160
76.842
64.432
64.310
61.308
61.194
61.148
60.391
59.576
56.426
56.163
27.988



17


```
Current Data Parameters
NAME      Eric_8a_3_4_5_OMe_mono_data
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_           20170120
Time              17.07 h
INSTRUM             spect
PROBHD    Z824601_0021 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  979
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                  203
DW               20.800 usec
DE                6.50 usec
TE                295.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                9.50 usec
PLW1        41.25000000 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         8.31000042 W
PLW12        0.23083000 W
PLW13        0.11611000 W

F2 - Processing parameters
SI                32768
SF          100.6127671 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

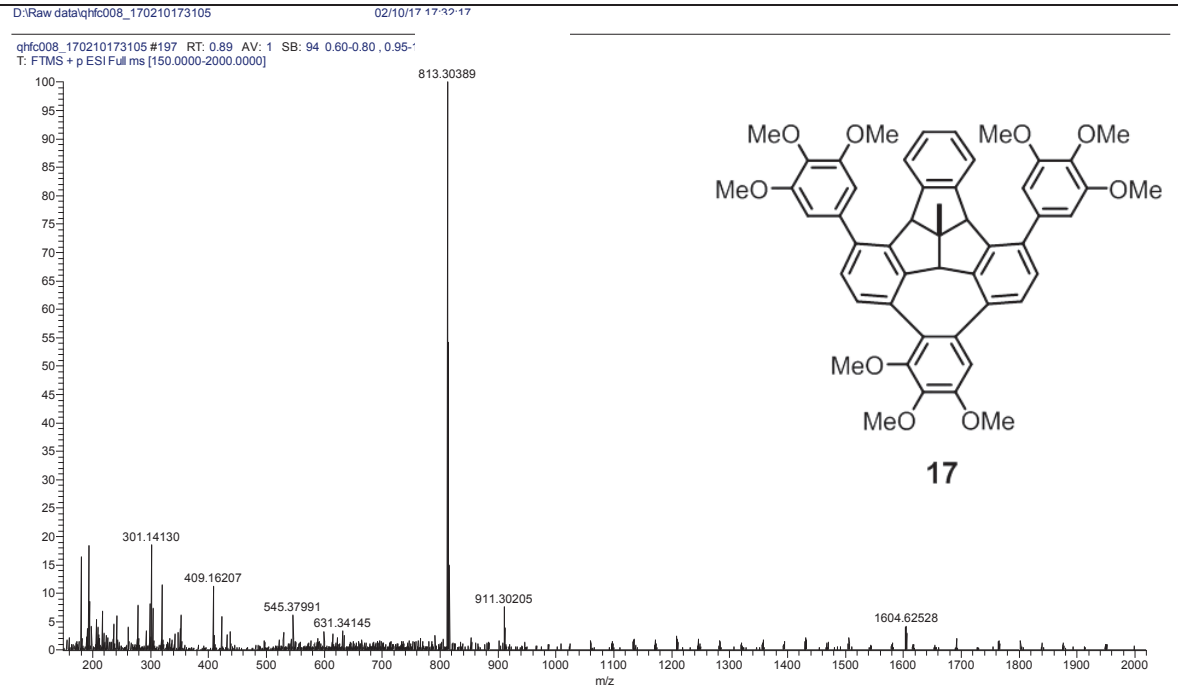
Thermo QEFMS Analysis Report

Analysis Info

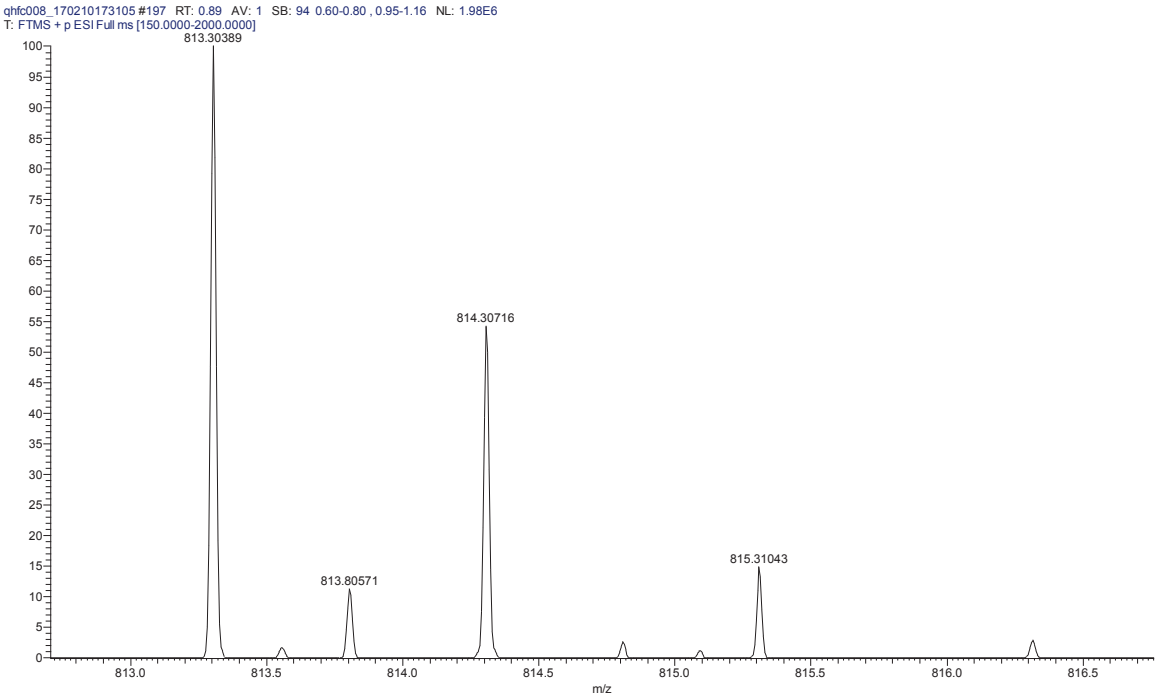
Sample Name :	Eric_30	Reference No.:	Qhfc008
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.0kV, by LC, with sheath gas		

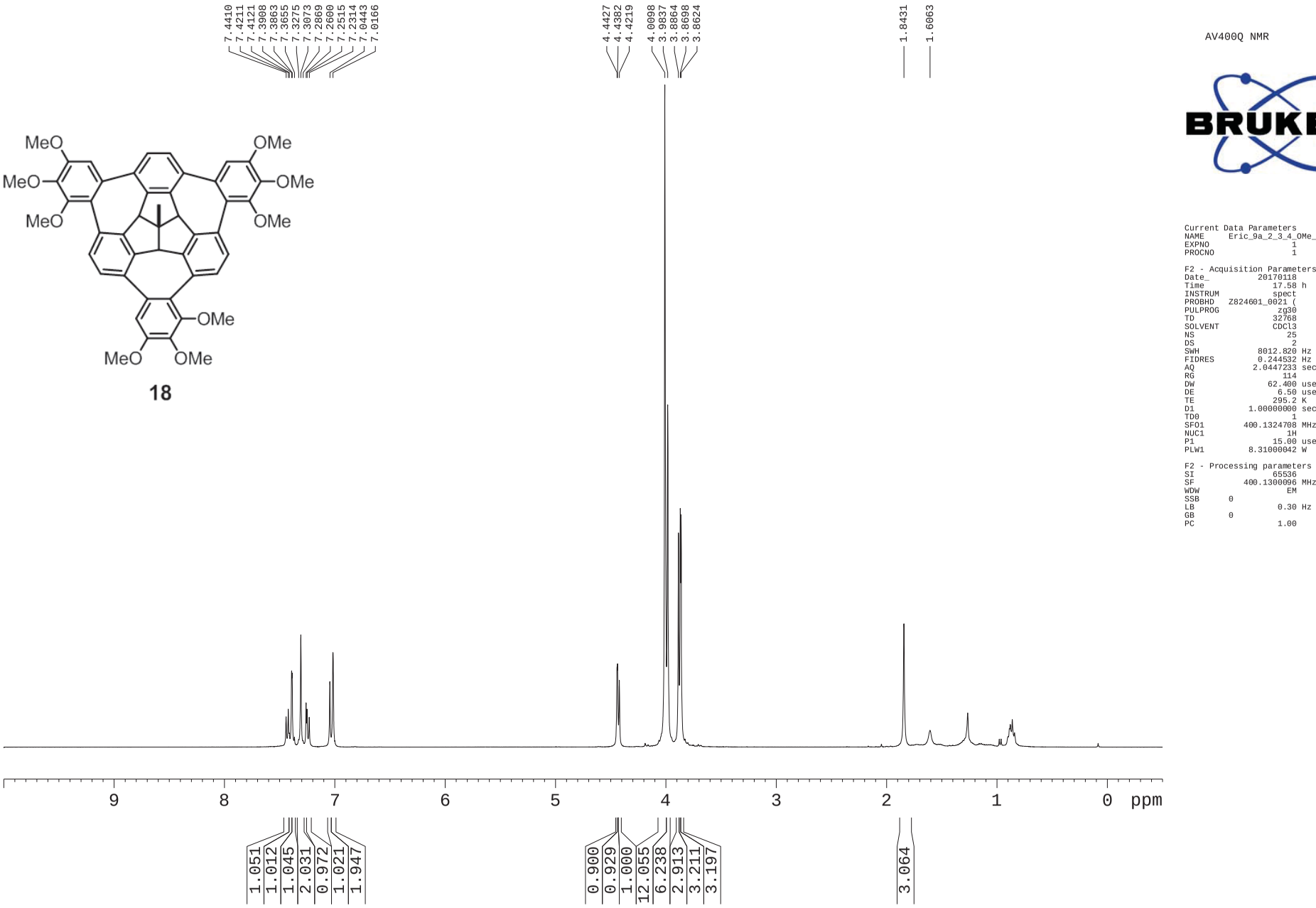
Accurate Mass Measurement

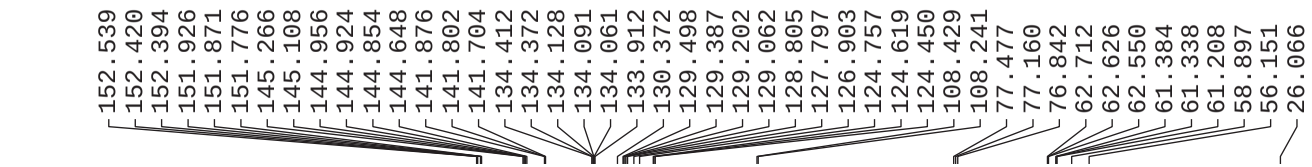
Molecular formula :	$C_{50}H_{46}O_9$
Experimental Mass $[M+Na]^+$:	813.30389
Theoretical Mass $[M+Na]^+$:	813.30340
Error (ppm) :	0.6

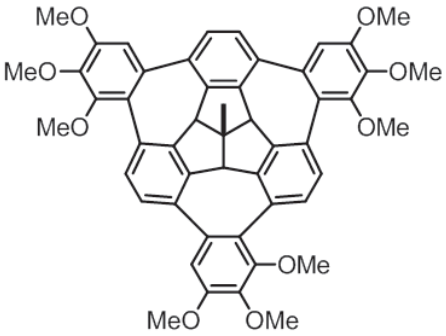


17




7.4410
7.4211
7.4121
7.3908
7.3863
7.3655
7.3275
7.3073
7.2869
7.2600
7.2515
7.2314
7.0443
7.0166
4.4427
4.4382
4.4219
4.0098
3.9837
3.8864
3.8698
3.8624
1.8431
1.6063
MeO
MeO
MeO
OMe
OMe
OMe
OMe
MeO
OMe
18
AV400Q NMR
BRUKER
Current Data Parameters
NAME Eric_9a_2_3_4_OMe_data
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170118
Time 17.58 h
INSTRUM spect
PROBHD Z824601_0021 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 25
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447233 sec
RG 114
DW 62.400 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.31000042 W
F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.051
1.012
1.045
2.031
0.972
1.021
1.947
0.900
0.929
1.000
12.055
6.238
2.913
3.211
3.197
3.064





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```
Current Data Parameters
NAME      Eric_9a_2_3_4_OMe_data
EXPNO                          2
PROCNO                         1

F2 - Acquisition Parameters
Date_               20170118
Time                   18.03 h
INSTRUM                spect
PROBHD    Z824601_0021 (
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       185
DS                         4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631488 sec
RG                       203
DW                    20.800 usec
DE                     6.50 usec
TE                    295.5 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1
SFO1              100.6228298 MHz
NUC1                     13C
P1                     9.50 usec
PLW1              41.25000000 W
SFO2              400.1316005 MHz
NUC2                     1H
CPDPRG[2              waltz16
PCPD2                 90.00 usec
PLW2               8.31000042 W
PLW12              0.23083000 W
PLW13              0.11611000 W

F2 - Processing parameters
SI                     32768
SF                100.6127581 MHz
WDW                       EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```

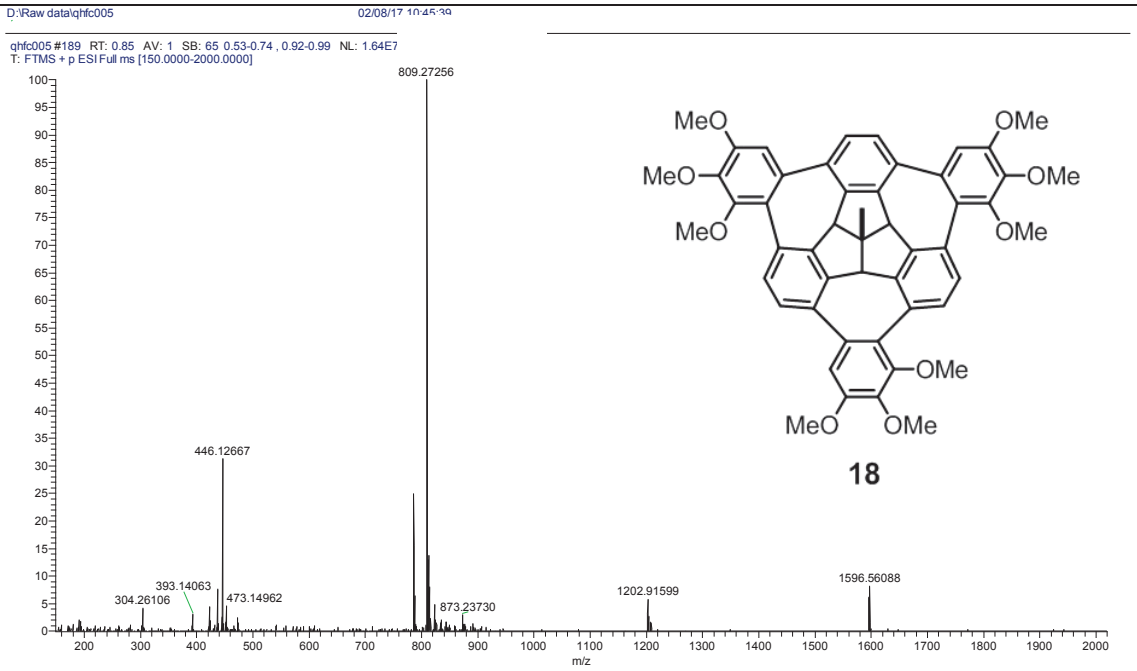
Thermo QEFMS Analysis Report

Analysis Info

Sample Name :	Eric_28	Reference No.:	Qhfc005
Instrument :	Q Exactive Focus Orbitrap		
Source :	HESI II	Polarity :	Positive
Comment :	ESI pos, 3.2kV, by LC, with sheath gas		

Accurate Mass Measurement

Molecular formula :	$C_{50}H_{42}O_9$
Experimental Mass $[M+Na]^+$:	809.27256
Theoretical Mass $[M+Na]^+$:	809.27210
Error (ppm) :	0.5



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