Supporting Information

3-Step Catalytic Asymmetric Total Syntheses of 13-methyltetrahydroprotoberberine Alkaloids

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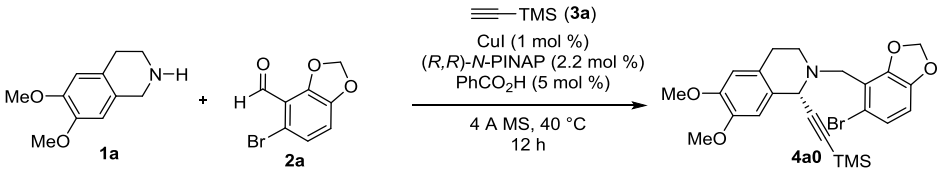
1. General Information

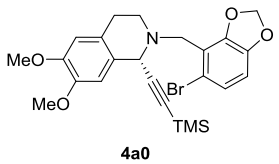
Reactions were carried out in oven or flame-dried glassware under a nitrogen atmosphere, unless otherwise noted. Tetrahydrofuran (THF) was freshly distilled before use from sodium using benzophenone as indicator. Dichloromethane was freshly distilled before use from calcium hydride (CaH_2). All other solvents were dried over 3Å or 4Å molecular sieves. Solvents used in workup, extraction and column chromatography were used as received from commercial suppliers without prior purification. Reactions were magnetically stirred and monitored by thin layer chromatography (TLC, 0.25 mm) on Merck pre-coated silica gel plates. Flash chromatography was performed with silica gel 60 (particle size 0.040 – 0.062 mm) supplied by Grace. Infrared spectra were collected on a Bruker model TENSOR27 spectrophotometer. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AV-400 spectrometer (400 MHz for ^{1}H, 100 MHz for ^{13}C). Chemical shifts are reported in parts per million (ppm) as values relative to the internal chloroform (7.26 ppm for ^{1}H and 77.0 ppm for ^{13}C) or DMSO-d_6 (2.50 ppm for ^{1}H and 39.50 ppm for ^{13}C). Abbreviations for signal coupling are as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. Optical rotations were measured on a JASCO Perkin-Elmer model P-2000 polarimeter. High resolution mass spectra were measured at the Hong Kong University of Science and Technology Mass Spectrometry Service Center on an Agilent GC/MS 5975C system.

2. Experimental Procedures and Characterization Data

2.1. Redox-A^3 Reaction of Tetrahydroisoquinolines, Aldehydes, and Terminal Alkynes

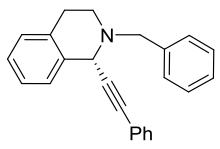


Gneral Procedure A (redox-A^3): To a flame-dried Schlenk tube were sequentially added (*R*,*R*)-*N*-PINAP (12.8 mg, 0.022 mmol), CuI (2.0 mg, 0.01 mmol), 4 Å molecular sieves (300 mg), and toluene (2 mL) under the argon atmosphere. The reaction mixture was then stirred at rt for 30 min and then $PhCO_2H$ (6.1 mg, 0.05 mmol), **1a** (270 mg, 1.4 mmol)/toluene (1 mL), **2a** (320.0 mg, 1.4 mmol, 98%)/toluene (1 mL), and **3a** (98 mg, 1.0 mmol)/toluene (2 mL) were sequentially added under the argon atmosphere. The Schlenk tube was placed in a pre-heated oil bath at 40 °C and the reaction mixture was stirred at 40 °C for 12 h. After cooling to room temperature, the crude reaction mixture was filtrated through a short pad of silica gel washed with Et_2O (50 mL). After solvent evaporation, the residue was purified by chromatography on silica gel to afford compound **4a0**.



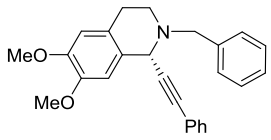
4a0

4a0 (401 mg, 80% yield) as pale yellow solid: m.p. 157-159 °C (hexane/dichloromethane); 50% *ee* (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 214 nm, t_R(major) = 11.9 min, t_R(minor) = 5.7 min); $[\alpha]^{25}_D$ = -64.0 (*c* = 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.04 (d, *J* = 8.0 Hz, 1H), 6.76 (s, 1H), 6.63 (d, *J* = 8.0 Hz, 1H), 6.54 (s, 1H), 6.03–5.96 (m, 2H), 4.65 (s, 1H), 3.96 (d, *J* = 12.0 Hz, 1H), 3.86 (s, 3H), 3.85–3.78 (m, 4H), 3.05–2.91 (m, 1H), 2.86–2.70 (m, 2H), 2.70–2.59 (m, 1H), 0.18 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 148.1, 148.0, 147.2, 146.7, 127.1, 126.1, 125.3, 120.3, 117.4, 111.2, 110.5, 108.5, 104.3, 101.5, 90.3, 56.0, 55.8, 55.5, 53.0, 45.0, 28.6, 0.16. IR (KBr) 2954.7, 2906.2, 2836.3, 2154.9, 1610.2, 1514.7, 1454.3, 1350.6, 1252.5, 1130.3, 1047.8, 921.7, 848.3, 795.4, 741.8, 590.8 cm^{-1}; HRMS (CI$^+$) (m/z) calcd. for $C_{24}H_{28}BrNO_4Si$ $[M]^+$ 502.1049; found 502.0986.



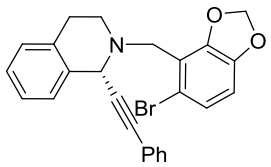
4a1

4a1 (253 mg) was obtained by using **General Procedure A** from THIQ **1a'** (186.2 mg, 1.4 mmol), aldehyde **2a'** (148.4 mg, 1.4 mmol), alkyne **3a'** (102.1 mg, 1.0 mmol) as pale yellow oil in 78% yield as pale yellow oil: 95% *ee* (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 100/1, 1.0 mL/min, λ = 214 nm, t_R(minor) = 5.0 min, t_R(major) = 7.7 min); $[\alpha]^{25}_D$ = -145.0 (*c* = 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.52–7.42 (m, 4H), 7.40–7.33 (m, 2H), 7.33–7.24 (m, 5H), 7.22–7.10 (m, 3H), 4.81 (s, 1H), 4.00–3.90 (m, 2H), 3.20–3.05 (m, 2H), 2.90–2.70 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 138.3, 135.5, 134.1, 131.8, 129.2 (×2), 129.0, 128.3 (×2), 128.2, 128.0, 127.8, 127.1, 126.9, 125.8, 123.2, 87.5, 86.8, 59.6, 54.3, 45.7, 29.0. IR (KBr) 3063.2, 2923.6, 2856.5, 1709.2, 1641.4, 1488.0, 1450.6, 1263.0, 794.2, 747.4, 705.5, 652.3 cm^{-1}; HRMS (CI$^+$) (m/z) calcd. for $C_{24}H_{21}N$ $[M+H]^+$ 324.1752; found 324.1726.



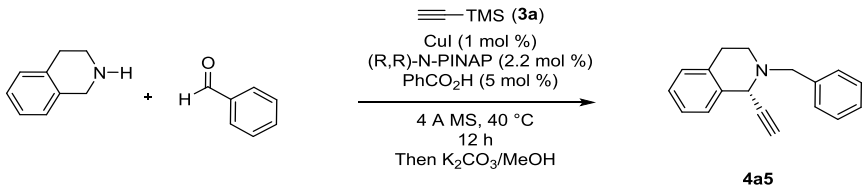
4a2

4a2 (318 mg) was obtained by using **General Procedure A** from THIQ **1a** (270.2 mg, 1.4 mmol), aldehyde **2a'** (148.4 mg, 1.4 mmol), alkyne **3a'** (102.1 mg, 1.0 mmol) as pale yellow oil in 83% yield as pale yellow oil: 86% *ee* (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R(major) = 8.2 min, t_R(minor) = 10.7 min); $[\alpha]^{25}_D$ = -103.0 (*c* = 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.54–7.46 (m, 4H), 7.40–7.20 (m, 6H), 6.77 (s, 1H), 6.63 (s, 1H), 4.73 (s, 1H), 4.0–3.9 (m, 2H), 3.87 (s, 3H), 3.86 (s, 3H), 3.16–3.05 (m, 1H), 3.05–2.94 (m, 1H), 2.90–2.80 (m, 1H), 2.79–2.69 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 148.1, 147.3, 138.3, 131.7, 129.3, 128.4, 128.24, 128.16, 128.0, 127.3, 127.1, 126.0, 111.4, 110.5, 87.5, 86.7, 59.5, 55.9, 55.81, 53.82, 45.9, 28.6. IR (KBr) 3060.8, 2918.0, 2827.8, 1710.4, 1645.9, 1604.8, 1514.7, 1454.7, 1352.0, 1265.1, 1225.9, 1128.3, 1019.6, 854.5, 746.8, 698.7; 573.2 cm^{-1}; HRMS (CI^+) (m/z) calcd. for $C_{26}H_{25}NO_2$ $[M]^+$ 383.1885; found 383.1870.



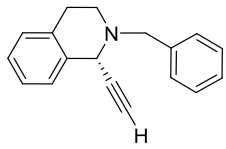
4a3

4a3 (334 mg) was obtained by using **General Procedure A** from THIQ **1a'** (186.2 mg, 1.4 mmol), aldehyde **2a** (320.6 mg, 1.4 mmol), alkyne **3a'** (102.1 mg, 1.0 mmol) in 75% yield as pale yellow oil: 73% *ee* (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 500/1, 1.0 mL/min, λ = 214 nm, t_R(major) =20.6 min, t_R(minor) = 28.3 min); $[\alpha]^{25}_D$ = -88.0 (*c* = 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.53–7.43 (m, 2H), 7.42–7.27 (m, 4H), 7.25–7.15 (m, 2H), 7.15–7.05 (m, 2H), 6.67 (d, *J* = 8.4 Hz, 1H), 6.03–5.97 (m, 2H), 5.02 (s, 1H), 4.02 (d, *J* = 12.8 Hz, 1H), 3.98 (d, *J* = 12.8 Hz, 1H), 3.22–3.12 (m, 1H), 3.04–2.84 (m, 2H), 2.84–2.74 (m 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 148.0, 146.6, 135.5, 134.0, 131.7, 128.9, 128.1, 127.9, 127.7, 126.8, 125.7, 125.3, 123.3, 120.3, 117.3, 108.5, 101.5, 87.9, 86.4, 55.4, 53.1, 45.0, 29.0. IR (KBr) 3062.3, 2899.4, 1596.8, 1492.2, 1452.4, 1244.8, 1048.5, 933.6, 852.20, 797.7, 745.31, 696.13 cm^{-1}; HRMS (CI^+) (m/z) calcd. for $C_{25}H_{20}BrNO_2$ $[M + H]^+$ 446.0756; found 446.0673.



4a5

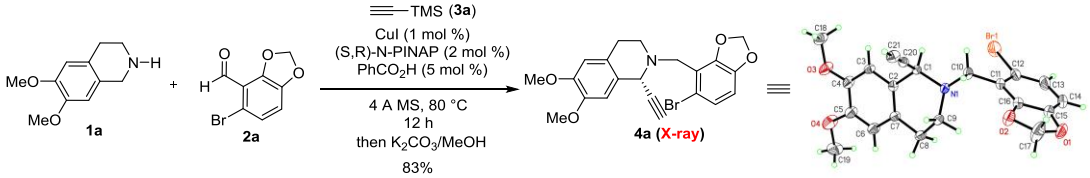
The crude TMS protected product obtained by redox-A^3 using **General Procedure A** from THIQ **1a'** (186.2 mg, 1.4 mmol), aldehyde **2a'** (148.4 mg, 1.4 mmol), alkyne **3a** (98.2 mg, 1.0 mmol). Then the crude product was directly subjected to desilylation as described below. After evaporation of the volatile solvents, the crude product was dissolved in MeOH (10 mL) and treated with K_2CO_3 (27 mg, 0.2 mmol). After TLC analysis indicated completion of the desilylation, the reaction mixture was concentrated using rotary evaporator. The crude mixture was added DCM (10 mL) and then washed with brine, dried over anhydrous Na_2SO_4, and concentrated in *vacuo*. The residue was purified by flash column chromatography on silica gel using eluents (hexane/EtOAc = 30:1) to afford the corresponding redox-A^3 reaction product **4a5** (180 mg) as pale yellow oil in 73% yield.



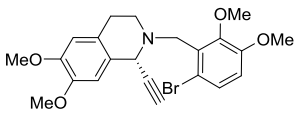
4a5

4a5 (180 mg, 73% yield) as pale yellow oil: 95% *ee* (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 500/1, 1.0 mL/min, λ = 214 nm, t_R(major) =11.3 min, t_R(minor) = 16.2 min); $[\alpha]^{25}_D$ = -67.0 (*c* = 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.57–7.50 (m, 2H), 7.49–7.39 (m, 2H), 7.39–7.32 (m, 1H), 7.30–7.15 (m, 4H), 4.68 (s, 1H), 3.95 (dd, *J* = 16.0 Hz, 12.0 Hz, 2H), 3.16–3.02 (m, 2H), 2.95–2.84 (m, 1H), 2.87–2.76 (m, 1H), 2.54 (d, *J* = 2.4, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 138.2, 135.0, 133.9, 129.1, 129.0, 128.3, 127.5, 127.1, 127.0, 125.8, 81.6, 74.5, 59.3, 53.6, 45.3, 28.9. IR (KBr) 3287.9, 3028.3, 2914.8, 2823.7, 1719.6, 1593.2, 1492.9, 1454.0, 1356.2, 1263.4, 1199.1, 1132.1, 1087.8, 948.2, 742.9, 698.9, 647.8 cm^{-1}; HRMS (CI$^+$) (m/z) calcd. for $C_{18}H_{17}N$ $[M]^+$ 247.1361; found 247.1325.

2.2. Catalytic Asymmetric Redox-A^3 reaction

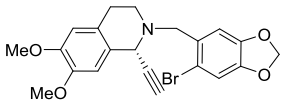


General Procedure B (catalytic asymmetric redox-A^3): To a flame-dried bottom-rounded flask (25 mL) were added CuI (1.2 mg, 0.01 mmol), (*S, R*)-*N*-PINAP (11 mg, 0.02 mmol), and newly activated 4 Å molecular sieves (300 mg). To the reaction flask under nitrogen atmosphere were sequentially added toluene (10 mL), aldehyde **2a** (229 mg, 1.0 mmol), THIQ **1a** (212 mg, 1.1 mmol), and trimethyl silyl acetylene **3a** (196 mg, 2.0 mmol), $PhCO_2H$ (6.1 mg, 0.05 mmol). The reaction mixture was heated with an oil bath at 40 ºC for 12 h. After TLC analysis indicated the completion of the reaction, the reaction mixture was passed through a short column of silica gel and washed with hexane/ethyl acetate (10/1–2/1). The combined filtrate was concentrated in vacuum. To the crude product in MeOH (10 mL) was added K_2CO_3 (27 mg, 0.2 mmol) and the reaction mixture was stirred at room temperature for 2 h. After TLC analysis indicated the completion of the desilylation, the reaction mixture was concentrated and then diluted with DCM (10 mL) and washed with brine, dried over anhydrous Na_2SO_4. The concentrated residue was purified by flash column chromatography on silica gel using eluents (hexane/EtOAc = 10:1) to afford the corresponding redox-A^3 reaction product **4a** (357 mg, 83%) as white solid: m.p. 144-146 ºC (hexane/dichloromethane); 95.5% *ee* (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 214 nm, t_R(major) =20.4 min, t_R(minor) = 10.5 min); $[\alpha]^{25}_D$ = –69.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.05 (d, *J* = 8.4 Hz, 1H), 6.72 (s, 1H), 6.64 (d, *J* = 8.4 Hz, 1H), 6.56 (s, 1H), 6.00 (d, *J* = 2.0 Hz, 2H), 4.69 (s, 1H), 3.91–3.84 (m, 5H), 3.83 (s, 3H), 3.09–2.96 (m, 1H), 2.92–2.77 (m, 2H), 2.65–2.58 (m, 1H), 2.47 (d, *J* = 2.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 148.2, 148.0, 147.4, 146.7, 127.0, 126.0, 125.4, 120.3, 117.4, 111.4, 110.1, 108.5, 101.6, 82.2, 74.2, 56.0, 55.9, 54.3, 52.9, 44.8, 28.6. IR (KBr) 3288.8, 2906.7, 2833.0, 2249.4, 1518.9, 1453.9, 1265.0, 1224.6, 1129.0, 1047.2, 731.1 cm^{-1}; HRMS (CI$^+$) (*m/z*) calcd. for $C_{21}H_{21}BrNO_4$ $[M + H]^+$ 430.0654; found 430.0643. For the X-ray data of **4a** (CCDC 1524047), see page S-26.



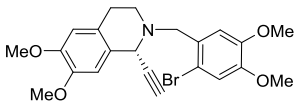
4b

4b (378 mg) was obtained by using **General Procedure B** from THIQ **1a** (212 mg, 1.1 mmol), aldehyde **2b** (245.0 mg, 1.0 mmol), alkyne **3a** (196 mg, 2.0 mmol) in 75% yield as yellow oil: 95% *ee* (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R(major) =34.8 min, t_R(minor) = 20.7 min); $[\alpha]^{25}_D$ = –88.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.27 (d, *J* = 8.8 Hz, 1H), 6.74 (d, *J* = 8.8 Hz, 1H), 6.72 (s, 1H), 6.54 (s, 1H), 4.71 (s, 1H), 4.00–3.88 (m, 2H), 3.86 (s, 3H), 3.85 (s, 6H), 3.81 (s, 3H), 3.11–2.99 (m, 1H), 2.76–2.92 (m, 2H), 2.53–2.62 (m, 1H), 2.47 (d, *J* = 2.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 152.3, 149.3, 148.1, 147.3, 132.8, 127.8, 127.2, 126.0, 116.9, 112.5, 111.3, 110.1, 82.6, 73.7, 61.5, 55.9, 55.83, 55.79, 54.0, 53.1, 44.8, 28.7. IR (KBr) 3288.3, 2935.7, 2834.4, 2251.9, 1611.9, 1518.9, 1472.0, 1224.5, 1129.4, 1038.5, 730.7 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{22}H_{25}BrNO_4$ $[M + H]^+$ 446.0967; found 446.0974.



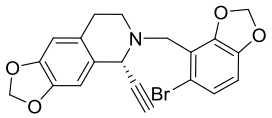
4c

4c (317 mg) was obtained by using **General Procedure B** from THIQ **1a** (212 mg, 1.1 mmol), aldehyde **2c** (229.0 mg, 1.0 mmol), alkyne **3a** (196 mg, 2.0 mmol) in 74% yield as yellow oil: 97% *ee* (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 214 nm, t_R(major) =14.7 min, t_R(minor) = 23.5 min); $[\alpha]^{25}_D$ = –81.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.09 (s, 1H), 7.01 (s, 1H), 6.70 (s, 1H), 6.59 (s, 1H), 5.96 (s, 2H), 4.56 (s, 1H), 3.854 (s, 3H), 3.85–3.82 (m, 5H), 3.07–2.86 (m, 2H), 2.85–2.70 (m, 1H), 2.68–2.60 (m, 1H), 2.47 (d, *J* = 2.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 148.3, 147.41, 147.36, 147.3, 130.9, 126.8, 126.0, 114.8, 112.7, 111.4, 110.3, 110.1, 101.6, 82.0, 74.4, 58.5, 56.0, 55.9, 53.5, 45.4, 28.6. IR (KBr): 3452.7, 2072.9, 1636.7, 1518.5, 1478.0, 1262.1, 1227.3, 1128.7, 1036.9, 730.3 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{21}H_{21}BrNO_4$ $[M + H]^+$ 430.0654; found 430.0653.



4d

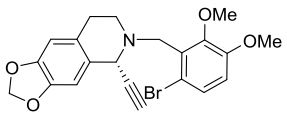
4d (357 mg) was obtained by using **General Procedure B** from THIQ **1a** (212 mg, 1.1 mmol), aldehyde **2d** (245.0 mg, 1.0 mmol), alkyne **3a** (196 mg, 2.0 mmol) in 80% yield as yellow oil: 97.5% *ee* (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 214 nm, t_R(major) =17.3 min, t_R(minor) = 10.5 min); $[\alpha]^{25}_D$ = –83.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.13 (s, 1H), 7.02 (s, 1H), 6.71 (s, 1H), 6.59 (s, 1H), 4.57 (s, 1H), 4.07–3.73 (m, 2H), 3.86 (s, 3H), 3.85 (s, 3H), 3.84 (s, 6H), 3.06–2.82 (m, 2H), 2.78–2.70 (m, 1H), 2.66 (m, 1H), 2.48 (d, *J* = 2.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 148.5, 148.3, 148.2, 147.4, 129.6, 126.8, 126.0, 115.4, 114.5, 113.1, 111.4, 110.1, 82.0, 74.3, 58.2, 56.11, 56.05, 55.9, 55.8, 53.7, 45.1, 28.6. IR (KBr) 3287.5, 2934.6, 2834.6, 2254.1, 1611.0, 1505.0, 1463.7, 1377.6, 1281.2, 1157.6, 1129.8, 1031.1, 731.1 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{22}H_{25}BrNO_4$ $[M + H]^+$ 446.0967; found 446.0948.



4e

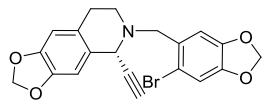
4e (293 mg) was obtained by using **General Procedure B** from THIQ **1b** (195 mg, 1.1 mmol), aldehyde **2a** (229.0 mg, 1.0 mmol), alkyne **3a** (196 mg, 2.0 mmol) in 71% yield as white solid: m.p. 130-132 °C (hexane/dichloromethane); 91% *ee* (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 99/1+0.5% Et_2NH, 1.0 mL/min, λ = 210 nm, t_R(major) =30.8 min, t_R(minor) = 25.7 min); $[\alpha]^{25}_D$ = –89.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.05 (d, *J* = 8.4 Hz, 1H), 6.70 (s, 1H), 6.64 (d, *J* = 8.4 Hz, 1H), 6.53 (s, 1H), 6.01–5.99 (m, 2H), 5.90–5.87 (m, 2H), 4.67 (s, 1H), 3.90–3.40 (m, 2H), 3.09–2.90 (m, 1H), 2.90–2.73 (m, 2H), 2.63–2.51

(m, 1H), 2.47 (d, *J* = 2.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 148.0, 146.8, 146.7, 145.8, 128.0, 127.2, 125.4, 120.2, 117.3, 108.6, 108.5, 107.3, 101.6, 100.8, 82.0, 74.2, 54.7, 52.9, 44.6, 28.9. IR (KBr) 3290.6, 2898.3, 2821.7, 2245.2, 1602.2, 1504.2, 1483.1, 1454.4, 1257.9, 1224.9, 1040.4, 930.8, 733.2 cm^{-1}; HRMS (CI$^+$) (*m/z*) calcd. for $C_{20}H_{16}BrNO_4$ $[M + H]^+$ 414.0341; found 414.0320.



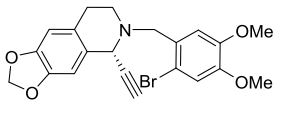
4f

4f (391 mg) was obtained by using **General Procedure B** from THIQ **1b** (195 mg, 1.1 mmol), aldehyde **2b** (245.0 mg, 1.0 mmol), alkyne **3a** (196 mg, 2.0 mmol) in 91% yield as white solid: m.p. 138-140 °C (hexane/dichloromethane); 94% *ee* (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 100/1, 1.0 mL/min, λ = 214 nm, t_R(major) =46.6 min, t_R(minor) = 26.9 min); $[\alpha]^{25}_D$ = –108.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.27 (d, *J* = 8.8 Hz, 1H), 6.74 (d, *J* = 8.8 Hz, 1H), 6.70 (s, 1H), 6.51 (s, 1H), 5.88 (d, *J* = 1.9 Hz, 2H), 4.70 (s, 1H), 3.88–3.96 (m, 2H), 3.85 (s, 6H), 3.12–2.94 (m, 1H), 2.89–2.71 (m, 2H), 2.59–2.50 (m, 1H), 2.47 (d, *J* = 2.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 152.3, 149.4, 146.7, 145.8, 132.7, 128.3, 127.8, 127.4, 116.9, 112.6, 108.5, 107.3, 100.7, 82.4, 73.8, 61.5, 55.9, 54.5, 53.1, 44.5, 29.1. IR (KBr) 3286.2, 2935.7, 2903.4, 2836.0, 2248.3, 1693.0, 1573.9, 1504.1, 1472.1, 1285.7, 1264.1, 1224.3, 1039.0, 1009.8, 804.0 cm^{-1}; HRMS (CI$^+$) (*m/z*) calcd. for $C_{21}H_{21}BrNO_4$ $[M + H]^+$ 430.0654; found 430.0651.



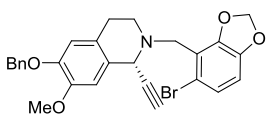
4g

4g (310 mg) was obtained by using **General Procedure B** from THIQ **1b** (195 mg, 1.1 mmol), aldehyde **2c** (229.0 mg, 1.0 mmol), alkyne **3a** (196 mg, 2.0 mmol) in 75% yield as yellow oil: 94% *ee* (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 100/1 + 0.5%Et_2NH, 1.0 mL/min, λ = 214 nm, t_R(major) =32.7 min, t_R(minor) = 24.5 min); $[\alpha]^{25}_D$ = –112.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.07 (s, 1H), 7.01 (s, 1H), 6.68 (s, 1H), 6.56 (s, 1H), 5.96 (s, 2H), 5.90 (d, *J* = 2.4 Hz, 2H), 4.54 (s, 1H), 3.83 (d, *J* = 3.6 Hz , 2H), 3.06–2.80 (m, 2H), 2.80–2.69 (m, 1H), 2.65–2.55 (m, 1H), 2.47 (d, *J* = 2.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 147.38, 147.34, 146.8, 145.9, 130.8, 127.8, 127.2, 114.8, 112.7, 110.3, 108.5, 107.3, 101.6, 100.8, 81.8, 74.4, 58.4, 53.9, 45.2, 29.0. IR (KBr) 3442.6, 1641.6, 1502.3, 1479.1, 1236.7, 1114.6, 1038.5, 732.2 cm^{-1}; HRMS (CI$^+$) (*m/z*) calcd. for $C_{20}H_{16}BrNO_4$ $[M + H]^+$ 414.0341; found 414.0357.



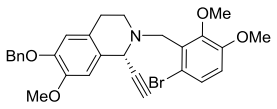
4h

4h (301 mg) was obtained by using **General Procedure B** from THIQ **1b** (195 mg, 1.1 mmol), aldehyde **2d** (245.0 mg, 1.0 mmol), alkyne **3a** (196 mg, 2.0 mmol) in 70% yield as yellow oil: 96% *ee* (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 100/1 + 0.5%Et_2NH, 1.0 mL/min, λ = 230 nm, t_R(major) =33.2 min, t_R(minor) = 27.4 min); $[\alpha]^{25}_D$ = –120.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.11 (s, 1H), 7.02 (s, 1H), 6.69 (s, 1H), 6.56 (s, 1H), , 5.90 (d, *J* = 2.8 Hz, 2H), 4.55 (s, 1H), 3.90 (d, *J* = 14.0 Hz, 1H), 3.87 (s, *3*H), 3.84 (s, 3H), 3.80 (d, *J* = 14.0 Hz, 1H), 3.03–2.81 (m, 2H), 2.81–2.69 (m, 1H), 2.69–2.57 (m, 1H), 2.48 (d, *J* = 2.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 148.5, 148.4, 146.8, 145.9, 129.5, 127.9, 127.3, 115.5, 114.5, 113.1, 108.5, 107.3, 100.8, 81.9, 74.4, 58.2, 56.14, 56.08, 54.1, 45.0, 29.0. IR (KBr) 3285.8, 2902.5, 2838.3, 2254.2, 1602.2, 1504.6, 1483.1, 1255.3, 1221.5, 1158.9, 1009.8, 731.7 cm^{-1}; HRMS (CI$^+$) (*m/z*) calcd. for $C_{21}H_{21}BrNO_4$ $[M + H]^+$ 430.0654; found 430.0653.



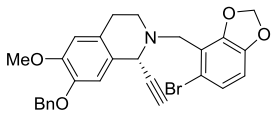
4i

4i (424 mg) was obtained by using **General Procedure B** from THIQ **1c** (296 mg, 1.1 mmol), aldehyde **2a** (229.0 mg, 1.0 mmol), alkyne **3a** (196 mg, 2.0 mmol) in 84% yield as yellow oil: 96% *ee* (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R(major) =31.1 min, t_R(minor) = 20.8 min); $[\alpha]^{25}_D$ = –125.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.47–7.39 (m, 2H), 7.39–7.32 (m, 2H), 7.32–7.27 (m, 1H), 7.05 (d, *J* = 8.0 Hz, 1H), 6.75 (s, 1H), 6.64 (d, *J* = 8.0 Hz, 1H), 6.59 (s, 1H), 6.01–5.98 (m, 2H), 5.10 (s, 2H), 4.69 (s, 1H), 3.92–3.80 (m, 5H), 3.06–2.88 (m, 1H), 2.87–2.68 (m, 2H), 2.60–2.50 (m, 1H), 2.46 (d, *J* = 2.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 147.98, 147.97, 147.4, 146.6, 137.2, 128.5, 127.7, 127.5, 127.2, 126.0, 125.3, 120.2, 117.3, 114.0, 110.7, 108.5, 101.5, 82.1, 74.2, 70.9, 56.1, 54.3, 52.9, 44.7, 28.4. IR (KBr) 3445.5, 2086.8, 1644.6, 1467.4, 1284.9, 1239.5, 1080.4, 702.6 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{27}H_{25}BrNO_4$ $[M + H]^+$ 506.0967; found 506.0979.



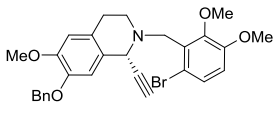
4j

4j (444 mg) was obtained by using **General Procedure B** from THIQ **1c** (296 mg, 1.1 mmol), aldehyde **2b** (245.0 mg, 1.0 mmol), alkyne **3a** (196 mg, 2.0 mmol) in 85% yield as yellow oil: 97% *ee* (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R(major) =17.2 min, t_R(minor) = 23.7 min); $[\alpha]^{25}_D$ = –93.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.41 (d, *J* = 7.6 Hz, 2H), 7.40–7.30 (m, 2H), 7.30–7.22 (m. 2H), 6.74 (d, *J* = 4.0 Hz, 2H), 6.57 (s, 1H), 5.08 (s, 2H), 4.71 (s, 1H), 3.99–3.87 (m, 2H), 3.85 (s, 9H), 3.03–3.00 (m, 1H), 2.93–2.65 (m, 2H), 2.54–2.44 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 152.3, 149.3, 148.0, 147.4, 137.3, 132.8, 128.5, 127.9, 127.7, 127.5, 127.2, 126.1, 117.0, 114.1, 112.5, 110.8, 82.6, 73.7, 71.0, 61.5, 56.1, 55.9, 54.1, 53.1, 44.8, 28.6. IR (KBr) 3450.8, 2096.7, 1625.8, 1518.5, 1473.8, 1269.7, 1212.3, 1069.9, 1012.7, 736.5 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{28}H_{29}BrNO_4$ $[M + H]^+$ 522.1280; found 522.1263.



4k

4k (410 mg) was obtained by using **General Procedure B** from THIQ **1d** (296 mg, 1.1 mmol), aldehyde **2a** (229.0 mg, 1.0 mmol), alkyne **3a** (196 mg, 2.0 mmol) in 81% yield **4k** as yellow oil: 98% *ee* (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 214 nm, t_R(major) =17.3 min, t_R(minor) = 13.7 min); $[\alpha]^{25}_D$ = –88.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.45 (d, *J* = 7.2 Hz, 2H), 7.36 (t, *J* = 7.2 Hz, 2H), 7.30 (d, *J* = 7.2 Hz, 1H), 7.04 (d, *J* = 8.8 Hz, 1H), 6.78 (s, 1H), 6.63 (d, *J* = 8.8 Hz, 1H), 6.58 (s, 1H), 5.98 (d, *J* = 2.4 Hz, 2H), 5.11 (s, 2H), 4.63 (s, 1H), 3.95–3.78 (m, 5H), 3.05–2.91 (m, 1H), 2.9 –2.69 (m, 2H), 2.67–2.51 (m, 1H), 2.42 (d, *J* = 2.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 149.0, 148.0, 146.7, 146.6, 137.2, 128.5, 127.8, 127.4, 127.0, 126.7, 125.4, 120.2, 117.3, 113.1, 112.0, 108.5, 101.6, 82.1, 74.1, 71.3, 56.0, 54.3, 52.9, 44.8, 28.6. IR (KBr) 3442.6, 2084.9, 1641.6, 1453.4, 1264.9, 1229.3, 1089.5, 697.7 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{27}H_{25}BrNO_4$ $[M + H]^+$ 506.0967; found 506.0989.

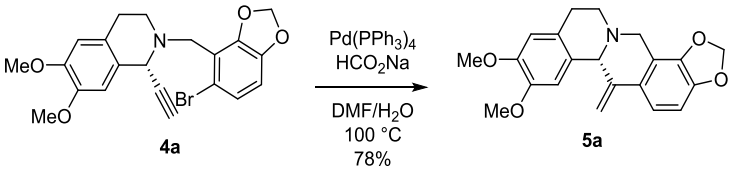


4l

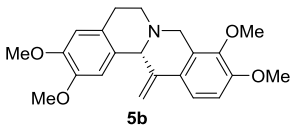
4l (454 mg) was obtained by using **General Procedure B** from THIQ **1d** (296 mg, 1.1 mmol), aldehyde **2b** (245.0 mg, 1.0 mmol), alkyne **3a** (196 mg, 2.0 mmol) in 87% yield as yellow oil: 98% *ee* (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 87.5/12.5, 1.0 mL/min, λ = 214 nm, t_R(major) =30.6 min, t_R(minor) = 36.2 min); $[\alpha]^{25}_D$ = –85.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.45 (d, *J* = 8.4 Hz, 2H), 7.36 (t, *J* = 8.4 Hz, 2H), 7.33–7.25 (m, 2H), 6.79 (s, 1H), 6.75 (d, *J* = 8.8

Hz, 1H), 6.57 (s, 1H), 5.11 (s, 2H), 4.67 (s, 1H), 3.96–3.88 (m, 2H), 3.85 (s, 3H), 3.84 (s, 3H), 3.82 (s, 3H), 3.10–2.96 (m, 1H), 2.89–2.73 (m, 2H), 2.63–2.49 (m, 1H), 2.42 (d, J = 2.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 152.3, 149.3, 148.8, 146.5, 137.2, 132.8, 128.4, 127.8, 127.7, 127.5, 127.3, 126.8, 116.9, 113.0, 112.5, 112.0, 82.5, 73.7, 71.2, 61.5, 56.0, 55.8, 54.1, 53.1, 44.7, 28.7. IR (KBr): 3442.9, 2095.0, 1634.0, 1517.5, 1471.4, 1268.4, 1222.3, 1074.8, 1010.9, 732.4 cm^{-1}; HRMS (CI$^+$) (*m/z*) calcd. for $C_{28}H_{29}BrNO_4$ $[M + H]^+$ 522.1280; found 522.1293.

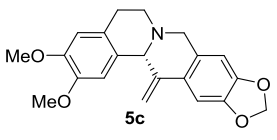
2.3. Pd-catalyzed Reductive Heck *exo*-Carbocyclization



General Procedure C (Reductive Heck): To a solution of **4a** (215 mg, 0.5 mmol) in DMF/H_2O (12 mL/4 mL) was added HCO_2Na (68 mg, 1 mmol). The solution was bubbled with a stream of dry nitrogen gas for 15 min before addition of $Pd(PPh_3)_4$ (29 mg, 0.025 mmol). The reaction mixture was heated at 100 ^{o}C in an oil bath and stirred for 2 h under the nitrogen atmosphere. The reaction mixture was diluted with CH_2Cl_2 (10 mL). The organic phase was collected and the aqueous phase was extracted with CH_2Cl_2 (3 x 10 mL). The combined organic fractions were washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel using eluents (hexane/EtOAc = 1:1) to provide the cyclization product **5a** (137 mg, 78% yield) as yellow oil. $[\alpha]^{25}_D$ = +103 (c = 0.3, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.15 (d, J = 8.0 Hz, 1H), 6.72 (d, J = 8.0 Hz, 1H), 6.64 (s, 1H), 6.63 (s, 1H), 5.97 (d, J = 8.8 Hz, 2H), 5.60 (s, 1H), 4.68 (s, 1H), 4.51 (s, 1H), 4.19 (d, J = 16.8 Hz, 1H), 3.98 (d, J = 16.8 Hz, 1H), 3.86 (s, 3H), 3.82 (s, 3H), 3.20–3.05 (m, 1H), 2.93–2.79 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 147.9, 146.7, 146.6, 143.6, 141.9, 128.6, 127.0, 126.4, 117.8, 116.1, 111.7, 111.6, 110.8, 107.0, 101.4, 62.0, 56.0, 55.8, 50.5, 47.6, 27.9. IR (KBr) 3442.6, 1639.5, 1518.7, 1465.7, 1360.3, 1262.5, 1127.1, 1044.8, 729.2 cm^{-1}; HRMS (CI$^+$) (*m/z*) calcd. for $C_{21}H_{22}NO_4$ $[M + H]^+$ 352.1549; found 352.1544.

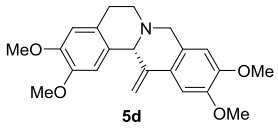


5b (134 mg) was obtained by using **General Procedure C** from **4b** (223 mg, 0.5 mmol) in 73% yield as yellow oil: $[\alpha]^{25}_D$ = +93.3 (c = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.36 (d, J = 8.4 Hz, 1H), 6.84 (d, J = 8.4 Hz, 1H), 6.65 (s, 1H), 6.64 (s, 1H), 5.59 (s, 1H), 4.70 (s, 1H), 4.46 (s, 1H), 4.21 (d, J = 17.2 Hz, 1H), 4.14 (d, J = 17.2 Hz, 1H), 3.88 (s, 3H), 3.87 (s, 3H), 3.84 (s, 3H), 3.83 (s, 3H), 3.20–3.09 (m, 1H), 2.97–2.80 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 151.9, 147.8, 146.6, 145.0, 142.3, 128.2, 127.8, 127.3, 126.5, 120.1, 111.8, 111.6, 110.9, 109.9, 62.0, 60.1, 56.0, 55.84, 55.77, 51.9, 47.9, 28.2. IR (KBr) 3419.0, 2918.5, 1697.9, 1558.7, 1518.7, 1285.5, 1116.6, 1021.6 cm^{-1}; HRMS (CI$^+$) (*m/z*) calcd. for $C_{22}H_{26}NO_4$ $[M + H]^+$ 368.1862; found 368.1867.



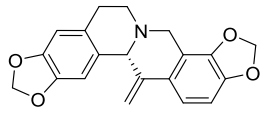
5c (128 mg) was obtained by using **General Procedure C** from **4c** (215 mg, 0.5 mmol) in 73% yield as yellow oil: $[\alpha]^{25}_D$ = +121.0 (c = 0.2, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.07 (s, 1H), 6.64 (s, 1H), 6.63 (s, 1H), 6.52 (s, 1H), 5.94 (s, 1H), 5.92 (s, 1H), 5.54 (s, 1H), 4.69 (s, 1H), 4.52 (s, 1H), 4.27 (d, J = 16.4 Hz, 1H), 3.87 (d, J = 16.4 Hz, 1H), 3.86 (s, 3H), 3.83 (s, 3H), 3.17–3.06 (m, 1H), 2.92–

2.80 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 147.9, 147.7, 146.7, 146.6, 142.1, 127.7, 127.2, 126.9, 126.5, 111.8, 111.6, 110.5, 105.9, 104.1, 100.9, 62.0, 56.1, 56.0, 55.8, 47.3, 27.9. IR (KBr) 3443.1, 1635.8, 1518.8, 1455.9, 1356.3, 1262.5, 1107.2, 1054.6, 725.5 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{21}H_{22}NO_4$ $[M + H]^+$ 352.1549; found 352.1558.



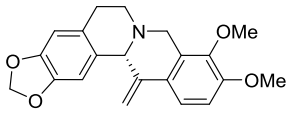
5d

5d (143 mg) was obtained by using **General Procedure C** from **4d** (223 mg, 0.5 mmol) in 78% yield as yellow oil: $[\alpha]^{25}_D$ = +124.0 (*c* = 0.2, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.09 (s, 1H), 6.65 (s, 2H), 6.54 (s, 1H), 5.58 (s, 1H), 4.69 (s, 1H), 4.55 (s, 1H), 4.35 (d, *J* = 16.0 Hz, 1H), 3.91 (s, 4H), 3.87 (s, 6H), 3.84 (s, 3H), 3.15 (s, 1H), 2.97–2.80 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 149.5, 147.8 (×2), 146.5, 142.1, 127.0, 126.7, 126.4, 126.0, 111.8, 111.6, 110.1, 108.5, 106.9, 61.9, 55.99, 55.95 (×2), 55.86, 55.83, 47.2, 28.1. IR (KBr) 3442.6, 1635.8, 1518.3, 1465.9, 1259.0, 1217.2, 1023.1, 668.6 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{22}H_{26}NO_4$ $[M + H]^+$ 368.1862; found 368.1866.



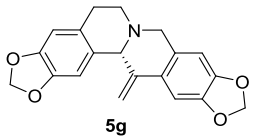
5e

5e (119 mg) was obtained by using **General Procedure C** from **4e** (207 mg, 0.5 mmol) in 71% yield as yellow oil: $[\alpha]^{25}_D$ = + 90.0 (*c* = 0.2, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.16 (d, *J* = 8.4 Hz, 1H), 6.73 (d, *J* = 8.4 Hz, 1H), 6.62 (s, 1H), 6.60 (s, 1H), 5.97 (d, *J* = 7.6 Hz, 2H), 5.91 (d, *J* = 2.0 Hz, 2H), 5.64 (s, 1H), 4.69 (s, 1H), 4.51 (s, 1H), 4.18 (d, *J* = 16.4 Hz, 1H), 3.95 (d, *J* = 16.4 Hz, 1H), 3.09–3.19 (m, 1H), 2.96–2.81 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 146.7, 146.5, 145.3, 143.6, 141.5, 128.4, 128.1, 127.6, 117.8, 116.0, 111.1, 108.8, 108.6, 107.0, 101.4, 100.7, 62.3, 50.0, 47.4, 28.1. IR (KBr) 3442.7, 1636.3, 1481.9, 1262.7, 1035.9, 799.12, 731.3 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{20}H_{18}NO_4$ $[M + H]^+$ 336.1236; found 336.1231.



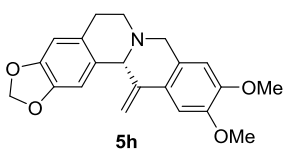
5f

5f (126 mg) was obtained by using **General Procedure C** from **4f** (215 mg, 0.5 mmol) in 72% yield as yellow oil: $[\alpha]^{25}_D$ = + 91.5 (*c* = 0.2, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.37 (d, *J* = 8.4 Hz, 1H), 6.83 (d, *J* = 8.8 Hz, 1H), 6.62 (d, *J* = 2.0 Hz, 2H), 5.91 (d, *J* = 2.4 Hz, 2H), 5.63 (s, 1H), 4.71 (s, 1H), 4.46 (s, 1H), 4.18 (d, *J* = 15.2 Hz, 1H), 4.09 (d, *J* = 15.2 Hz, 1H), 3.87 (s, 3H), 3.83 (s, 3H), 3.19–3.08 (m, 1H), 2.92–2.80 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 151.9, 146.4, 145.3, 145.0, 141.6, 128.1, 127.8, 127.4, 127.3, 120.2, 111.0, 110.5, 108.8, 108.6, 100.7, 62.1, 60.1, 55.8, 51.1, 47.6, 28.1. IR (KBr) 3443.1, 2078.5, 1636.3, 1485.2, 1285.4, 1279.5, 1033.7, 728.5 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{21}H_{22}NO_4$ $[M + H]^+$ 352.1549; found 352.1538.



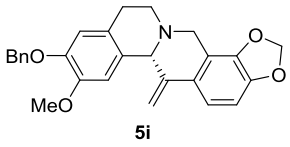
5g

5g (132 mg) was obtained by using **General Procedure C** from **4g** (207 mg, 0.5 mmol) in 79% yield as yellow oil: $[\alpha]^{25}_D$ = + 95.0 (*c* = 0.2, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.08 (s, 1H), 6.61 (s, 1H), 6.60 (s, 1H), 6.50 (s, 1H), 5.94-5,89 (m, 4H), 5.57 (s, 1H), 4.70 (s, 1H), 4.45 (s, 1H), 4.24 (d, *J* = 16.0 Hz, 1H), 3.85 (d, *J* = 16.0 Hz, 1H), 3.19–3.00 (m, 1H), 2.92–2.84 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 147.7, 146.7, 146.4, 145.2, 141.7, 128.0, 127.7 (×2), 127.0, 110.8, 108.8, 108.7, 105.9, 104.1, 100.9, 100.7, 62.2, 55.6, 47.0, 28.1. IR (KBr) 3442.7, 2075.7, 1635.5, 1485.2, 1242.6, 1035.1, 727.5 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{20}H_{18}NO_4$ $[M + H]^+$ 336.1236; found 336.1240.



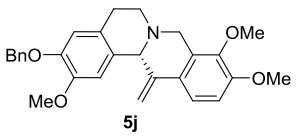
5h

5h (133 mg) was obtained by using **General Procedure C** from **4h** (215 mg, 0.5 mmol) in 76% yield as yellow oil: $[\alpha]^{25}_{D}$ = + 103.5 (*c* = 0.2, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.09 (s, 1H), 6.62 (s, 2H), 6.53 (s, 1H), 5.91 (d, *J* = 2.4 Hz, 2H), 5.62 (s, 1H), 4.71 (s, 1H), 4.54 (s, 1H), 4.30 (d, *J* = 16.2 Hz, 1H), 3.91 (s, 3H), 3.90–3.84 (m, 4H), 3.20–3.07 (m, 1H), 2.94–2.82 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 149.6, 147.9, 146.5, 145.2, 141.5, 127.9, 127.6, 126.1, 125.5, 110.6, 108.8, 108.7, 108.5, 106.9, 100.7, 62.2, 56.0, 55.9, 55.2, 46.9, 28.1. IR (KBr) 3442.7, 1636.7, 1516.9, 1258.7, 1034.8, 723.0 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{21}H_{22}NO_4$ $[M + H]^+$ 352.1549; found 352.1574.



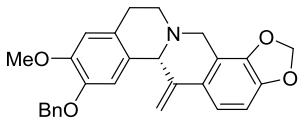
5i

5i (149 mg) was obtained by using **General Procedure C** from **4i** (253 mg, 0.5 mmol) in 70% yield as yellow oil: $[\alpha]^{25}_{D}$ = + 162.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.45 (d, *J* = 7.2 Hz, 2H), 7.37 (t, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 1H), 7.16 (d, *J* = 8.4 Hz, 1H), 6.73 (d, *J* = 8.4 Hz, 1H), 6.68 (s, 1H), 6.66 (s, 1H), 5.99 (s, 1H), 5.96 (s, 1H), 5.61 (s, 1H), 5.13 (s, 2H), 4.69 (s, 1H), 4.51 (s, 1H), 4.16 (d, *J* = 16.8 Hz, 1H), 3.97 (d, *J* = 16.8 Hz, 1H), 3.83 (s, 3H), 3.20–3.00 (m, 1H), 2.96–2.70 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 147.3, 147.1, 146.6, 143.6, 141.8, 137.2, 128.6, 128.5, 127.8, 127.3, 127.1, 127.0, 117.7, 116.1, 114.4, 112.4, 110.9, 107.0, 101.4, 71.0, 62.0, 56.2, 50.5, 47.6, 27.8. IR (KBr) 3445.5, 1629.4, 1536.5, 1445.7, 1324.3, 1258.6, 1122.1, 1037.6, 733.1; HRMS (CI^+) (*m/z*) calcd. for $C_{27}H_{25}NO_4$ $[M + H]^+$ 428.1862; found 428.1852.



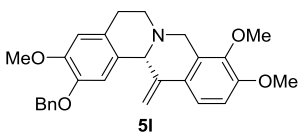
5j

5j (170 mg) was obtained by using **General Procedure C** from **4j** (261 mg, 0.5 mmol) in 77% yield as yellow oil: $[\alpha]^{25}_{D}$ = + 153.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.45 (d, *J* = 7.6 Hz, 2H), 7.41–7.33 (m, 3H), 7.30 (d, *J* = 7.3 Hz, 1H), 6.86 (d, *J* = 8.8 Hz, 1H) , 6.68 (s, 1H), 6.67 (s, 1H), 5.60 (s, 1H), 5.13 (s, 2H), 4.71 (s, 1H), 4.47 (s, 1H), 4.16 (s, 1H), 4.13 (s, 1H), 3.88 (s, 3H), 3.83 (s, 6H), 3.15–3.10 (m, 1H), 2.92–2.75 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 151.9, 147.3, 147.1, 145.0, 142.1, 137.3, 128.5, 128.1, 127.8, 127.7, 127.3, 127.19, 127.16, 120.1, 114.4, 112.5, 110.9, 110.1, 71.1, 61.9, 60.1, 56.2, 55.8, 51.7, 47.8, 28.0. IR (KBr) 3445.5, 1629.4, 1536.5, 1445.7, 1324.3, 1258.6, 1122.1, 1037.6, 733.1; HRMS (CI^+) (*m/z*) calcd. for $C_{28}H_{29}NO_4$ $[M + H]^+$ 443.2097; found 443.2045.



5k

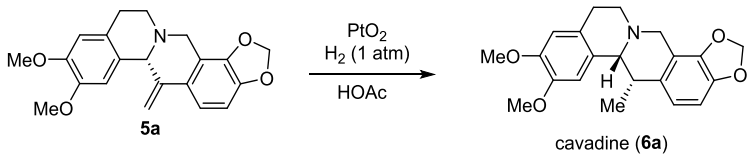
5k (158 mg) was obtained by using **General Procedure C** from **4k** (253 mg, 0.5 mmol) in 74% yield as yellow oil: $[\alpha]^{25}_{D}$ = + 100.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.40 (d, *J* = 8.0 Hz, 2H), 7.34 (t, *J* = 8.0 Hz, 2H), 7.31 – 7.27 (m, 1H), 7.12 (d, *J* = 8.0 Hz, 1H), 6.72 (d, *J* = 8.0 Hz, 1H), 6.66 (s, 1H), 6.64 (s, 1H), 5.97 (s, 1H), 5.96 (s, 1H), 5.51 (s, 1H), 5.11 (s, 2H), 4.53 (s, 1H), 4.46 (s, 1H), 4.12 (d, *J* = 16.0 Hz, 1H), 3.96 (d, *J* = 16.0 Hz, 1H), 3.88 (s, 3H), 3.19–3.02 (m, 1H), 2.92–2.74 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 148.7, 146.6, 145.5, 143.5, 141.6, 137.2, 128.53, 128.45, 127.7, 127.4 (×2), 126.4, 117.8, 116.1, 115.0, 112.2, 110.7, 107.0, 101.4, 71.2, 61.9, 56.0, 50.4, 47.7, 27.9. IR (KBr) 3453.5, 1634.3, 1527.5, 1448.6, 1353.3, 1256.6, 1134.1, 731.1 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{27}H_{25}NO_4$ $[M + H]^+$ 428.1862; found 428.1850.



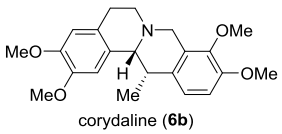
5l

5l (170 mg) was obtained by using **General Procedure C** from **4l** (261 mg, 0.5 mmol) in 77% yield as yellow oil: $[\alpha]^{25}_{D}$ = + 112.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.41 (d, *J* = 7.3 Hz, 2H), 7.38–7.30 (m, 3H), 7.28 (d, *J* = 7.3 Hz, 1H), 6.82 (d, *J* = 8.4 Hz, 1H), 6.67 (s, 1H), 6.65 (s, 1H), 5.49 (s, 1H), 5.11 (s, 2H), 4.52 (s, 1H), 4.39 (s, 1H), 4.20–4.10 (m, 2H), 3.88 (s, 6H), 3.82 (s, 3H), 3.20–2.99 (m, 1H), 2.96–2.72 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 151.8, 148.6, 145.5, 145.0, 141.9, 137.3, 128.4, 128.0, 127.9, 127.7, 127.6, 127.4, 126.4, 120.1, 115.1, 112.2, 110.9, 109.9, 71.2, 61.8, 60.1, 56.0, 55.8, 51.7, 47.9, 28.1. IR (KBr) 3679.4, 3622.5, 2937.0, 1580.2, 1494.9, 1283.3, 1025.1 cm^{-1}; HRMS (CI$^+$) (*m/z*) calcd. for $C_{28}H_{29}NO_4$ $[M]^+$ 443.2097; found 443.2003.

2.4. PtO_2-catalyzed Hydrogenation



General Procedure D: To a solution of compound **5a** (35 mg, 0.1 mmol) in acetic acid (2.0 mL) was added PtO_2 (2.3 mg, 0.01 mmol) and the reaction suspension was stirred under an atmosphere of hydrogen (1 atm) for 12 h. The solvent (acetic acid) was removed by rotary evaporation under reduced pressure. The residue was dissolved in DCM (5 mL) and washed with saturated aqueous sodium carbonate (2 x 5 mL). The organic layer was collected, dried over $MgSO_4$, and concentrated. The residue was purified by flash column chromatography on silica gel using eluents (hexane/EtOAc = 5:1) to provide cavidine[1] (**6a**, 33.9 mg, 96% yield) as white solid: m.p. 180-182 °C (hexane/dichloromethane); 94% *ee* (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R(major) =9.8 min, t_R(minor) = 18.4 min); $[\alpha]^{25}_{D}$ = +242.0 (*c* = 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.72 (d, *J* = 8.0 Hz, 1H), 6.69 (s, 1H), 6.67 (d, *J* = 8.0 Hz, 1H), 6.61 (s, 1H), 5.96 (s, 1H), 5.92 (s, 1H), 4.07 (d, *J* = 16.0 Hz, 1H), 3.90 (s, 2H), 3.73 (d, *J* = 13.6 Hz, 1H), 3.50 (d, *J* = 16.0 Hz, 1H), 3.25 (dt, *J* = 24.4, 12.4 Hz, 1H), 3.19–3.00 (m, 1H), 2.63–2.57 (m, 2H), 0.94 (d, *J* = 6.8 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 147.7, 147.2, 144.7, 143.0, 135.9, 128.4, 128.3, 121.2, 116.8, 111.2, 108.6, 106.7, 101.0, 63.1, 56.1, 55.8, 53.3, 51.3, 38.6, 29.3, 18.4. IR (KBr) 2927.2, 2788.6, 1610.7, 1515.5, 1256.8, 1043.2, 808.6 cm^{-1}; HRMS (CI$^+$) (m/z) calcd. for $C_{21}H_{24}NO_4$ $[M + H]^+$ 354.1705; found 354.1722.



corydaline (**6b**)

Corydaline[2] **6b** (33.5 mg) was obtained by using **General Procedure D** from **5b** (36.7 mg, 0.1 mmol) in 91% yield as white solid: m.p. 138-140 °C (hexane/dichloromethane); 93% *ee* (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R(major) =10.0 min, t_R(minor) = 18.9 min); $[\alpha]^{25}_{D}$ = +249.0 (*c* = 0.5, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.91 (d, *J* = 8.8 Hz, 1H), 6.82 (d, *J* = 8.8 Hz, 1H), 6.68 (s, 1H), 6.61 (s, 1H), 4.20 (d, *J* = 16.0 Hz, 1H), 3.88 (s, 6H), 3.862 (s, 3H), 3.858 (s, 3H), 3.69 (s, 1H), 3.50 (d, *J* = 16.0 Hz, 1H), 3.29–3.02 (m, 3H), 2.71–2.50 (m, 2H), 0.94 (d, *J* = 8.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 150.0, 147.6, 147.1, 144.8, 134.9, 128.5, 128.45, 128.35, 124.0, 111.1, 110.9, 108.7, 63.0, 60.1, 56.1, 55.9, 55.8, 54.4, 51.4, 38.3, 29.3, 18.3. IR (KBr) 2933.6, 2806.1, 2755.3, 1622.5, 1531.2,1462.1, 1259.8, 1064.0, 1028.3 cm^{-1}; HRMS (CI$^+$) (m/z) calcd. for $C_{22}H_{28}NO_4$ $[M + H]^+$ 370.2018; found 370.2055.

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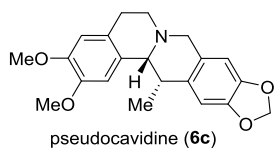
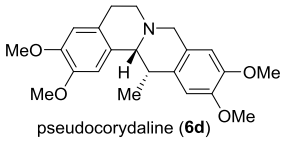
[1] Kitsiou, C.; Unsworth, W. P.; Coulthard, G.; Taylor, R. J. *Tetrahedron*, **2014**, 70, 7172–7180.

[2] Yu, L. L.; Li, R. T.; Ai, Y. B.; Liu, W.; Deng, Z. S.; Zou, Z. M. *Molecules* **2014**, *19*, 13332–13341.



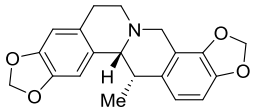
pseudocavidine (**6c**)

Pseudocavidine[3,4] **6c** (33.2 mg) was obtained by using **General Procedure D** from **5c** (35.1 mg, 0.1 mmol) in 94% yield as white solid: m.p. 183-185 °C (hexane/dichloromethane); 94% *ee* (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 214 nm, t_R(major) = 10.0 min, t_R(minor) = 18.4 min); $[\alpha]^{25}_D$ = +270.0 (*c* = 0.5, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.68 (s, 1H), 6.65 (s, 1H), 6.60 (s, 1H), 6.53 (s, 1H), 5.90 (s, 2H), 3.92 (d, *J* = 14.8 Hz, 1H), 3.879 (s, 3H), 3.872 (s, 3H), 3.70 (s, 1H), 3.59 (d, *J* = 14.8 Hz, 1H), 3.20–3.00 (m, 3H), 2.60–2.50 (m, 2H), 0.94 (d, *J* = 6.8 Hz, 3H) ^{13}C NMR (100 MHz, $CDCl_3$) δ = 147.7, 147.2, 146.0, 145.7, 134.4, 128.34, 128.27, 127.0, 111.2, 108.63, 108.56, 105.8, 100.6, 63.2, 58.9, 56.1, 55.8, 51.2, 38.8, 29.2, 18.1. IR (KBr) 2933.5, 2800.6, 1635.9, 1512.8, 1483.5, 1257.0, 1037.6, 730.1 cm^{-1}; HRMS (CI^+) (m/z) calcd. for $C_{21}H_{24}NO_4$ $[M + H]^+$ 354.1705; found 354.1735.



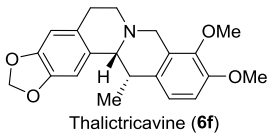
pseudocorydaline (**6d**)

Pseudocorydaline 6d (35.4 mg) was obtained by using **General Procedure D** from **5d** (36.7 mg, 0.1 mmol) in 96% yield as white solid: m.p. 143-145 °C (hexane/dichloromethane); 93.5% *ee* (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 97.5/2.5, 1.0 mL/min, λ = 214 nm, t_R(major) = 28.2 min, t_R(minor) = 36.9 min); $[\alpha]^{25}_D$ = +259.0 (*c* = 0.2, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.70 (s, 1H), 6.68 (s, 1H), 6.61 (s, 1H), 6.56 (s, 1H), 3.94 (d, *J* = 14.4 Hz, 2H), 3.88 (s, 9H), 3.85 (s, 3H), 3.73 (s, 1H), 3.62 (d, *J* = 14.4 Hz, 1H), 3.16–3.25 (m, 1H), 3.03–3.16 (m, 2H), 2.51–2.63 (m, 2H), 0.96 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 147.62, 147.56, 147.3, 147.1, 133.3, 128.5, 128.3, 126.0, 111.5, 111.1, 108.8, 108.6, 63.3, 58.6, 56.0, 55.9 (×2), 55.8, 51.3, 38.4, 29.2, 18.0. IR (KBr) 2929.5, 1610.5, 1511.2, 1227.0, 1028.4, 749.6 cm^{-1}; HRMS (CI^+) (m/z) calcd. for $C_{22}H_{28}NO_4$ $[M + H]^+$ 370.2018; found 370.2028.



tetrahydrocorysamine (**6e**)

Tetrahydrocorysamine[5] **6e** (31.3 mg) was obtained by using **General Procedure D** from **5e** (33.5 mg, 0.1 mmol) in 93% yield as white solid: m.p. 200-202 °C (hexane/dichloromethane); 91% *ee* (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, t_R(major) = 12.0 min, t_R(minor) = 20.7 min); $[\alpha]^{25}_D$ = +226.0 (*c* = 0.2, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.71 (d, *J* = 8.0 Hz, 1H), 6.69 (s, 1H), 6.66 (d, *J* = 8.0 Hz, 1H), 6.58 (s, 1H), 5.98–5.90 (m, 4H), 4.06 (d, *J* = 15.2 Hz, 1H), 3.70 (s, 1H), 3.48 (d, *J* = 15.2 Hz, 1H), 3.25–3.16 (m, 1H), 3.16–2.97 (m, 2H), 2.59 (dd, *J* = 15.2, 7.6 Hz, 2H), 0.95 (d, *J* = 8.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 146.3, 145.6, 144.7, 143.0, 135.9, 129.6, 129.3, 121.3, 116.7, 108.3, 106.8, 105.6, 101.0, 100.7, 63.5, 53.3, 51.2, 38.7, 29.8, 18.4. IR (KBr) 3459.5, 2933.5, 2866.7, 1637.3, 1484.7, 1265.7, 1040.9, 637.5 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{20}H_{20}NO_4$ $[M + H]^+$ 338.1392; found 338.1385.

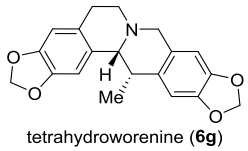


Thalictricavine (**6f**)

Thalictricavine[6,7] **6f** (34.5 mg) was obtained by using **General Procedure D** from **5f** (35.1 mg, 0.1 mmol) in 98% yield as white solid: m.p. 204-206 °C (hexane/dichloromethane); 93% *ee* (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 250/1 + 0.5% Et_2NH, 1.0 mL/min, λ = 230 nm,

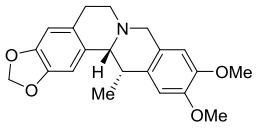
[3] Shamma, M.; Jones, C. D.; Weiss, J. A. *Tetrahedron* **1969**, *25*, 4347–4355.
[4] Pai, B. R.; Nagarajan, K.; Suguna, H.; Natarajan, S. *Heterocycles* **1978**, *9*, 1287–1288.
[5] Hanaoka, M.; Hirasawa, T.; Cho, W. J.; Yasuda, S. *Chem. Pharm. Bull.* **2000**, *48*, 399–404.
[6] Hanaoka, M.; Yoshida, S.; Mukai, C. *Chem. Pharm. Bull.* **1989**, *37*, 3264–3267.
[7] Takao, N.; Iwasa, K.; Kamigauhi M.; Sugiura, M. *Chem. Pharm. Bull.* **1977**, 25, 1426–1435.

t_R(major) = 25.2 min, t_R(minor) = 21.8 min); $[\alpha]^{25}_D$ = +261 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.89 (d, *J* = 8.4 Hz, 1H), 6.82 (d, *J* = 8.4 Hz, 1H), 6.68 (s, 1H), 6.58 (s, 1H), 5.92 (d, *J* = 7.2 Hz, 2H), 4.19 (d, *J* = 16.0 Hz, 1H), 3.86 (s, 6H), 3.66 (s, 1H), 3.48 (d, *J* = 16.0 Hz, 1H), 3.12-3.25 (m, 2H), 3.00-3.12 (m, 1H), 2.58 (d, *J* = 14.4 Hz, 2H), 0.96 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 150.0, 146.3, 145.6, 144.8, 134.9, 129.7, 129.3, 128.4, 124.0, 111.0, 108.3, 105.6, 100.7, 63.4, 60.1, 55.9, 54.4, 51.3, 38.4, 29.8, 18.2. IR (KBr) 3445.7, 2926.1, 2745.9, 1683.8, 1457.1, 1220.8, 1039.0, 796.3 cm^{-1}; HRMS(CI^+) (*m/z*) calcd. for $C_{21}H_{24}NO_4$ $[M + H]^+$ 354.1705; found 354.1710.



tetrahydroworenine (**6g**)

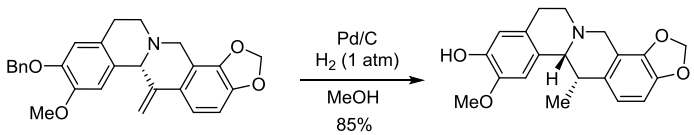
Tetrahydroworenine [8] **6g** (31.0 mg) was obtained by using **General Procedure D** from **5g** (33.5 mg, 0.1 mmol) in 92% yield as white solid: m.p. 208-209 ºC (hexane/dichloromethane); 94% *ee* (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 100/1 + 0.5% Et_2NH, 1.0 mL/min, λ = 214 nm, t_R(major) = 20.2 min, t_R(minor) = 17.7 min); $[\alpha]^{25}_D$ = +240.0 (*c* = 0.2, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.68 (s, 1H), 6.63 (s, 1H), 6.58 (s, 1H), 6.52 (s, 1H), 5.90 (s, 2H), 5.92 (s, 2H), 3.91 (d, *J* = 14.8 Hz, 1H), 3.68 (s, 1H), 3.58 (d, *J* = 14.8 Hz, 1H), 3.17–2.94 (m, 3H), 2.58–2.48 (m, 2H), 0.95 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 146.3, 146.0, 145.7, 145.6, 134.4, 129.5, 129.3, 126.9, 108.6, 108.3, 105.8, 105.5, 100.7, 100.6, 63.6, 58.9, 51.1, 38.9, 29.7, 18.1. IR (KBr) 3444.3, 2956.5, 2876.6, 1635.3, 1384.6, 1260.9, 1033.9, 669.5 cm^{-1}; HRMS (CI^+) (m/z) calcd. for $C_{20}H_{20}NO_4$ $[M + H]^+$ 338.1392; found 338.1385.



pseudothalictricavine (**6h**)

Pseudothalictricavine [9] **6h** (33.5 mg) was obtained by using **General Procedure D** from **5h** (35.1 mg, 0.1 mmol) in 95% yield as white solid: m.p. 168-169 ºC (hexane/dichloromethane); 95.5% *ee* (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 100/1 + 0.5% Et_2NH, 1.0 mL/min, λ = 230 nm, t_R(major) = 25.2 min, t_R(minor) = 21.8 min); $[\alpha]^{25}_D$ = +246.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.69 (s, 1H), 6.66 (s, 1H), 6.58 (s, 1H), 6.55 (s, 1H), 5.93 (d, *J* = 9.2 Hz, 2H), 3.93 (d, *J* = 16.0 Hz, 1H), 3.88 (s, 3H), 3.85 (s, 3H), 3.71 (s, 1H), 3.60 (d, *J* = 16.0 Hz, 1H), 3.00–3.20 (m, 2H), 2.51–2.61 (m, 1H), 0.98 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 147.6, 147.3, 146.3, 145.6, 133.3, 129.7, 129.3, 125.9, 111.6, 108.8, 108.3, 105.6, 100.7, 63.7, 58.5, 55.9 (×2), 51.2, 38.5, 29.7, 18.0. IR (KBr) 3414.4, 2929.3, 1611.1, 1388.3, 1037.5, 750.1cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{21}H_{24}NO_4$ $[M + H]^+$ 354.1705; found 354.1721.

2.5. Pd-catalyzed Hydrogenation and Debenzylation:



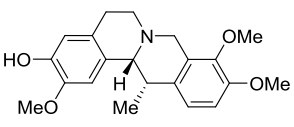
5i isoapocavidine (**6i**)

General Procedure E: To a solution of **5i** (43 mg, 0.1 mmol) in MeOH (2.0 mL) was added Pd/C (10.6 mg, 0.01 mmol, 10 wt%). The suspension was stirred under an atmosphere of hydrogen (1 atm) for 12 h. After completion of the reaction, CH_2Cl_2 (4 mL) was added to the solution and the reaction mixture was washed with saturated aqueous brine. The organic layer was collected and the aqueous layer was extracted with CH_2Cl_2 (3 x 4 mL). The combined fractions were dried over anhydrous magnesium sulfate

[8] Govindachari, T. R.; Nagarajan, K.; Natarajan, S.; Pai, B. R. *Indian J. Chem.* **1971**, *9*, 1313–1315.

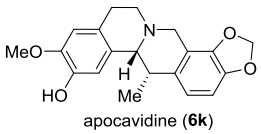
[9] Cushman, M.; Gentry, J.; Dekow, F. W. *J. Org. Chem.* **1977**, *42*, 1111–1116.

and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel using eluents (hexane/EtOAc = 5:1) to provide isoapocavidine (**6i**, 28.7 mg, 85% yield) as colorless oil: 95.5% *ee* (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R(major) = 16.1 min, t_R(minor) = 20.6 min); $[\alpha]^{25}_D$ = +265.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.71 (d, *J* = 8.0 Hz, 1H), 6.68 (s, 1H), 6.67 (d, *J* = 8.0 Hz, 1H), 6.66 (s, 1H), 5.96 (s, 1H), 5.93 (s, 1H), 4.06 (d, *J* = 16.0 Hz , 1H), 3.88 (s, 3H), 3.74–3.68 (m, 1H), 3.49 (d, *J* = 16.0 Hz, 1H), 3.22 (qt, *J* = 16.4, 8.2 Hz, 1H), 3.16–3.08 (m, 1H), 3.08–2.95 (m, 1H), 2.64–2.49 (m, 2H), 0.93 (d, *J* = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 145.4, 144.7, 143.6, 143.0, 135.9, 129.1, 127.9, 121.2, 116.8, 114.0, 107.8, 106.7, 101.0, 63.2, 56.1, 53.4, 51.2, 38.7, 29.1, 18.4. IR (KBr) 2933.2, 2764.6, 1620.5, 1462.3, 1358.7, 1256.8, 1143.2, 864.6 cm^{-1}; HRMS (CI$^+$) (m/z) calcd. for $C_{20}H_{21}NO_4$ $[M]^+$ 339.1471; found 339.1474.



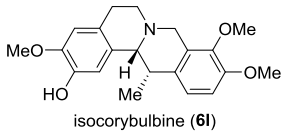
corybulbine (**6j**)

Corybulbine[10] **6j** (32 mg) was obtained by using **General Procedure E** from **5j** (44.3 mg, 0.1 mmol) in 90% yield as colorless oil: 96% *ee* (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 97.5/2.5, 1.0 mL/min, λ = 214 nm, t_R(major) = 24.1 min, t_R(minor) = 34.1 min); $[\alpha]^{25}_D$ = +277.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.90 (d, *J* = 8.4 Hz, 1H), 6.82 (d, *J* = 8.4 Hz, 1H), 6.68 (s, 1H), 6.66 (s, 1H), 5.50 (s, 1H), 4.19 (d, *J* = 11.6 Hz, 1H), 3.88 (s, 3H), 3.86 (s, 6H), 3.68 (s, 1H), 3.49 (d, *J* = 11.6 Hz, 1H), 3.24–3.10 (m, 2H), 3.05 (t, *J* = 13.9 Hz, 1H), 2.63–2.47 (m, 2H), 0.95 (d, *J* = 8.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 150.0, 145.4, 144.9, 143.6, 134.9, 129.2, 128.5, 128.0, 123.9, 114.0, 110.9, 107.9, 63.1, 60.1, 56.1, 55.9, 54.5, 51.4, 38.4, 29.1, 18.3. IR (KBr) 2944.3, 1637.9, 1532.2, 1247.0, 1068.4, 755.7 cm^{-1}; HRMS (CI$^+$) (m/z) calcd. for $C_{21}H_{25}NO_4$ $[M]^+$ 355.1784; found 355.1779.



apocavidine (**6k**)

Apocavidine[11] **6k** (28.8 mg) was obtained by using **General Procedure E** from **5k** (42.7 mg, 0.1 mmol) in 85% yield as colorless oil: 95.5% *ee* (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R(major) = 13.9 min, t_R(minor) = 18.0 min); $[\alpha]^{25}_D$ = +211.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.77 (s, 1H), 6.71 (d, *J* = 8.0 Hz, 1H), 6.66 (d, *J* = 8.0 Hz, 1H), 6.59 (s, 1H), 5.94 (d, *J* = 12.2 Hz, 2H), 4.06 (d, *J* = 16.0 Hz, 1H), 3.89 (s, 3H), 3.70(s, 1 H), 3.48 (d, *J* = 16.0 Hz, 1H), 3.59–3.39 (m, 1H), 3.39–3.00 (m, 1 H), 2.71–2.41 (m, 2H), 0.94 (d, *J* = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 144.7, 144.6, 144.1, 143.0, 136.0, 129.2, 127.5, 121.3, 116.7, 111.4, 110.4, 106.7, 101.0, 63.0, 55.8, 53.3, 51.4, 38.3, 29.3, 18.5. IR (KBr) 2929.5, 2778.3, 1643.7, 1535.5, 1235.7, 1144.3, 1067.9, 877.6 cm^{-1}; HRMS (CI$^+$) (m/z) calcd. for $C_{20}H_{22}NO_4$ $[M + H]^+$ 340.1549; found 340.1552.



isocorybulbine (**6l**)

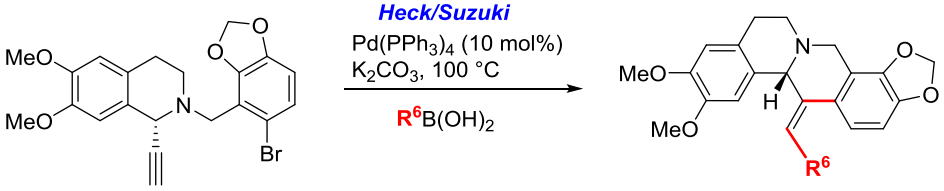
Isocorybulbine 6l (32.3 mg) was obtained by using **General Procedure E** from **5l** (44.3 mg, 0.1 mmol) in 91% as colorless oil: 94.5% *ee* (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 214 nm, t_R(major) = 10.4 min, t_R(minor) = 17.5 min); $[\alpha]^{25}_D$ = +225.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.89 (d, *J* = 8.4 Hz, 1H), 6.82 (d, *J* = 8.4 Hz, 1H), 6.77 (s, 1H), 6.59 (s, 1H), 4.19 (d, *J* = 15.6 Hz, 1H), 3.88 (s, 3H), 3.86 (s, 6H), 3.65 (s, 1H), 3.49 (d, *J* = 15.6

[10] Saito, S. Y.; Tanaka, M.; Matsunaga, K.; Li, Y.; Ohizumi, Y. *Biol. Pharm. Bull.* **2004**, 27, 1270-1274.

[11] Yu, C. K.,; MacLean, D. B.; Rodrigo, R. G. A.; Manske, R. H. F. *Can. J. Chem. Eng.* **1970**, *48*, 3673–3678.

Hz, 2H), 3.25–3.12 (m, 2H), 3.10–3.00 (m, 1H), 2.59 (dd, *J* = 12.8, 6.8 Hz, 2H), 0.95 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 150.0, 144.8, 144.7, 144.0, 135.0, 129.3, 128.4, 127.6, 124.1, 111.4, 110.9, 110.4, 62.9, 60.1, 55.8 (×2), 54.4, 51.5, 38.1, 29.4, 18.3. IR (KBr) 2935.3, 1622.9, 1544.4, 1253.0, 1124.5, 1069.8, 743.6 cm^{-1}; HRMS (CI$^+$) (m/z) calcd. for $C_{21}H_{25}NO_4$ $[M]^+$ 355.1784; found 355.1786.

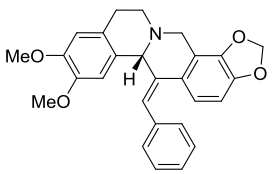
2.6. Preparation of Compounds 9a–9c and 10 via Cascade Heck-Suzuki Reaction



4a (94% ee)

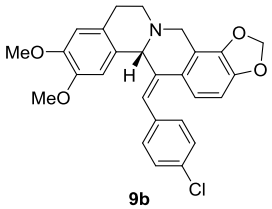
9a: R^6 = Ph, **92%**
9b: R^6= 4-Cl-Ph, **95%**
9c: R^6= 3-CF_3-Ph, **93%**
9d: R^6= C_4H_9, **0%**

General Procedure F (Heck-Suzuki): To a solution of compound **4a** (86 mg, 0.2 mmol) and boronic acid (0.3 mmol) in DMF/H_2O (6.0 mL/2.0 mL) was added K_2CO_3 (55.2 mg, 0.4 mmol). The solution was bubbled with a stream of dry nitrogen gas for 15 min before the addition of $Pd(PPh_3)_4$ (11.5 mg, 0.01 mmol). The reaction mixture was stirred at 100 °C for 2 h and then the mixture was diluted with EtOAc, washed with water and brine. The organic layers were dried with Na_2SO_4 and concentrated. The residue was purified by column chromatography on silica gel to afford the corresponding compounds **9a–9c** and **9k**.



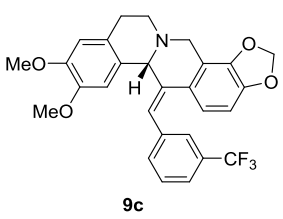
9a

9a (78.5 mg) was obtained by using **General Procedure F** from **4a** (86 mg, 0.2 mmol) in 92% as colorless oil: $[\alpha]^{25}_D$ = +121.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.20–7.16 (m, 4H), 7.16–7.10 (m, 1H), 6.72 (d, *J* = 8.0 Hz, 1H), 6.70 (s, 1H), 6.63 (s, 1H), 6.43 (d, *J* = 8.0, 1H), 6.03 (s, 1H), 5.97 (s, 2H), 4.50 (s, 1H), 4.31 (d, *J* = 16.0 Hz, 1H), 4.02 (q, *J* = 16.0 Hz, 1H), 3.88 (s, 3H), 3.87 (s, 3H), 3.41–3.30 (m, 1H), 3.00–2.88 (m, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ = 148.0, 146.9, 146.1, 143.3, 138.0, 135.7, 129.1, 128.1, 127.8, 127.6, 126.8, 126.6, 126.2, 122.6, 117.8, 111.6, 111.5, 106.0, 101.3, 63.4, 56.1, 55.8, 50.9, 48.0, 28.0. IR (KBr) 2922.8, 1645.1, 1607.4, 1512.2, 1463.1, 1363.1, 1258.7, 1128.8, 1043.8, 914.9, 811.6, 731.4, 646.5, 582.4, 476.8 cm^{-1}. HRMS (CI$^+$) (m/z) calcd. for $C_{27}H_{25}NO_4$ $[M+H]^+$ 428.1862; found 428.1825.



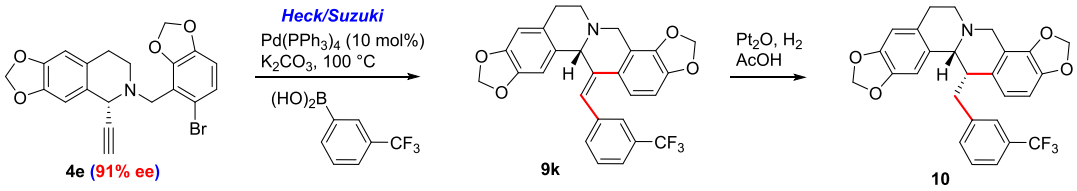
9b

9b (87.0 mg) was obtained by using **General Procedure F** from **4a** (86 mg, 0.2 mmol) in 95% as colorless oil: $[\alpha]^{25}_D$ = +137.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.16–7.08 (m, 4H), 6.69 (d, *J* = 8.0 Hz, 1H), 6.67 (s, 1H), 6.64 (s, 1H), 6.46 (d, *J* = 8.0 Hz, 1H), 6.00–5.92 (m, 3H), 4.47 (s, 1H), 4.28 (d, *J* = 16.0 Hz, 1H), 4.02 (d, *J* = 16.0 Hz, 1H), 3.87 (s, 3H), 3.86 (s, 3H), 3.40–3.26 (m, 1H), 2.98–2.85 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 148.0, 146.9, 146.3, 143.4, 136.7, 136.4, 132.2, 130.4, 128.3, 127.6, 127.5, 125.8, 125.1, 122.4, 117.7, 111.4 (×2), 106.1, 101.4, 63.4, 56.1, 55.8, 51.0, 48.1, 28.1. IR (KBr) 3005.0, 2923.7, 2837.2, 1605.0, 1510.2, 1463.1, 1363.2, 1130.9, 1045.7, 913.1, 821.3, 731.8, 644.1, 512.4 cm^{-1}. HRMS (CI$^+$) (m/z) calcd. for $C_{27}H_{24}ClNO_4$ $[M+H]^+$ 462.1472; found 462.1422.



9c

9c (92.0 mg) was obtained by using **General Procedure F** from **4a** (86 mg, 0.2 mmol) in 93% as colorless oil: $[\alpha]^{25}_{D}$ = +123.0 (*c* = 0.15, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.43 (s, 1H), 7.37 (t, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 1H), 6.68 (s, 1H), 6.66–6.61 (m, 2H), 6.44 (d, *J* = 8.3, 1H), 5.99 (s, 3H), 4.49 (s, 1H), 4.30 (d, *J* = 17.3 Hz, 1H), 4.04 (d, *J* = 17.4, 1H), 3.89 (s, 3H), 3.87 (s, 3H), 3.34 (ddd, *J* = 9.2 Hz, 6.2, 3.7, 1H), 3.00–2.83 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 148.1, 147.0, 146.6, 143.5, 138.7, 137.9, 132.5, 130.6, 130.3, 128.4, 127.7, 127.2, 126.0 (d, *J* = 3.9 Hz), 125.7, 125.4, 124.6, 123.2 (d, *J* = 3.9 Hz), 122.7, 122.4, 117.9, 111.5, 106.0, 101.4, 63.4, 56.1, 55.8, 51.2, 48.1, 28.2. IR (KBr) 2924.4, 1608.2, 1513.7, 1464.9, 1329.6, 1258.6, 1163.0, 1125.6, 1043.9, 915.8, 808.6, 731.6, 652.3, 587.0, 472.2 cm^{-1}. HRMS (CI^+) (m/z) calcd. for $C_{28}H_{24}F_3NO_4$ $[M+H]^+$ 496.1376; found 496.1376.

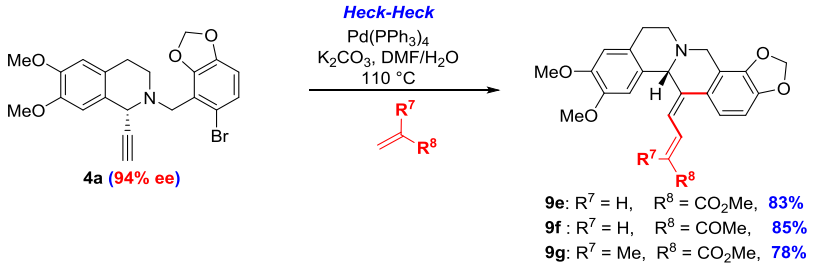


9k (86.0 mg) was obtained by using **General Procedure F** from **4e** (82.8 mg, 0.2 mmol) in 90% as colorless oil: $[\alpha]^{25}_{D}$ = +87.0 (*c* = 0.2, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.48 (s, 1H), 7.41 (s, 1H), 7.39 (s, 1H), 7.29 (d, J = 8.0 Hz, 1H), 6.65 (s, 1H), 6.64–6.60 (m, 2H), 6.43 (d, *J* = 8.0 Hz, 2H), 6.04 (s, 2H), 5.97 (s, 2H), 5.97–5.92 (m, 2H), 4.49 (s, 2H), 4.28 (d, *J* = 16.0 Hz, 2H), 4.01 (d, *J* = 16.0 Hz, 2H), 3.38–3.25 (m, 1H), 3.02–2.80 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 146.7, 146.5, 145.6, 143.5, 138.8, 137.5, 132.5, 130.7, 130.4, 128.7, 128.4, 128.1 (q, *J* = 10.8 Hz, 1C), 126.8, 125.98, 126.0, 125.4 (q, *J* = 10.8 Hz, 1C), 124.9, 123.23, 123.19, 122.7 (q, *J* = 10.8 Hz, 1C), 122.5, 120.0 (q, *J* = 10.8 Hz, 1C). 117.8, 108.7, 108.3, 106.1, 101.34, 100.78, 63.7, 50.4, 47.9, 28.20. IR (KBr) 2899.0, 1637.0, 1383.3, 1328.8, 1253.2, 1167.6, 1126.0, 1043.5, 996.7, 915.2, 812.5, 733.2, 653.8, 590.6, 523.2, 465.1 cm^{-1}. HRMS (CI^+) (m/z) calcd. for $C_{27}H_{20}F_3NO_4$ $[M+H]^+$ 480.1423; found 480.1379.

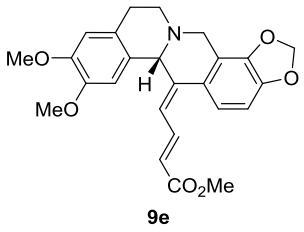
10[12] (41.4 mg) was obtained by using **General Procedure D** from **9k** (47.9 mg, 0.1 mmol) in 86% yield as colorless oil: $[\alpha]^{25}_{D}$ = +261.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, DMSO-d_6) δ = 7.47–7.32 (m, 2H), 7.17 (d, *J* = 7.2 HZ, 1H), 7.04 (s, 1H), 6.94 (s, 1H), 6.65 (s, 1H), 6.54 (d, *J* = 8.0, 1H), 6.02–5.96 (m, 2H), 5.95–5.90 (m, 2H), 5.89 (s, 1H), 4.04 (d, *J* = 15.6, 1H), 3.71 (s, 1H), 3.59–3.48 (m, 1H), 3.44 (d, *J* = 15.6 Hz, 1H), 3.15–3.06 (m, 1H), 2.95 (s, 1H), 2.72 (dd, *J* = 13.6 Hz, 4.8, 1H), 2.66–2.54 (m, 2H), 2.50–2.41 (m, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ = 145.9, 145.41, 144.7, 142.7, 142.4, 133.3, 132.7, 129.0, 128.65, 128.61, 128.56, 128.3 (q, *J* = 10.8 Hz, 1C), 125.6 (q, *J* = 10.8 Hz, 1C), 122.9 (q, *J* = 10.8 Hz, 1C), 122.2, 122.1, 121.7, 120.2 (q, *J* = 10.8 Hz, 1C), 116.2, 108.0, 105.9, 105.6, 100.9, 100.5, 62.9, 52.67, 50.4, 45.2, 38.5, 29.1. IR (KBr) 2917.9, 1648.3, 1473.8, 1370.7, 1329.2, 1261.6, 1165.4, 1123.2, 1069.1, 1038.8, 931.2, 806.3, 737.6, 702.0, 660.00 cm^{-1}; HRMS (CI^+) (m/z) calcd. for $C_{27}H_{22}F_3NO_4$ $[M]^+$ 482.1579; found 482.1547.

[12] Zhang, Z. H.; Zhang, H. J.; Deng, A. J.; Wang, B.; Li, Z. H.; Liu, Y.; Wu, L. Q.; Wang, W. J.; Qin, H. L. *J. Med.Chem.***2015,** *58,* 7557-7571.

2.7 Preparation of Compounds 9e–9g via Cascade Heck-Heck reaction

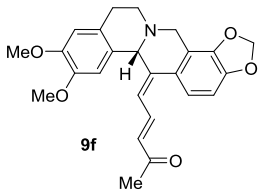


General Procedure G (Heck-Heck): To a solution of compound **4a** (86 mg, 0.2 mmol) and olefin (1 mmol) in DMF/H_2O (6.0 mL/2.0 mL) was added K_2CO_3 (55.2 mg, 0.4 mmol). The solution was bubbled with a stream of dry nitrogen gas for 15 min before the addition of $Pd(PPh_3)_4$ (11.5 mg, 0.01 mmol). The mixture was stirred at 110 °C for 3 h and then the mixture was diluted with EtOAc, washed with water and saturated brine. The organic layers were dried with Na_2SO_4 and concentrated. The residue was purified by column chromatography on silica gel to afford compounds **9e–9g**.



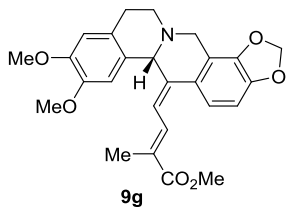
9e

9e(72.0 mg) was obtained by using **General Procedure G** from **4a** (86 mg, 0.2 mmol) in 83% yield as colorless oil: $[\alpha]^{25}_D$ = +261.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.79 (dd, *J* = 16.0 Hz, 12.0, 1H), 6.93 (d, *J* = 8.0 Hz, 1H), 6.80 (d, *J* = 8.0 Hz, 1H), 6.65 (s, 1H), 6.54 (s, 1H), 6.02 (d, *J* = 4.0 Hz, 2H), 5.83 (d, *J* = 16.0 Hz, 1H), 5.77 (d, *J* = 12.0 Hz, 1H), 4.49 (s, 1H), 4.23 (d, *J* = 16.0 Hz, 1H), 3.99 (d, *J* = 16.0 Hz, 1H), 3.89 (s, 3H), 3.83 (s, 3H), 3.70 (s, 3H), 3.18–3.08 (m, 1H), 2.93–2.77 (m, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ = 167.7, 148.1, 147.6, 147.0, 145.2, 141.9, 128.3, 127.8, 125.1, 123.3, 123.1, 121.9, 117.6, 111.5, 111.2, 106.5, 101.6, 63.2, 56.1, 55.8, 51.4, 51.3, 48.3, 28.4. IR (KBr) 2938.9, 2841.6, 1709.3, 1615.6, 1514.2, 1460.7, 1366.7, 1262.8, 1135.2, 1043.4, 912.9, 815.5, 731.0, 646.0, 567.3 cm^{-1}. HRMS (CI^+) (m/z) calcd. for $C_{25}H_{25}NO_6$ $[M+H]^+$ 436.1760; found 436.1739.



9f

9f (71.0 mg) was obtained by using **General Procedure G** from **4a** (86 mg, 0.2 mmol) in 85% yield as colorless oil: $[\alpha]^{25}_D$ = +135.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.65 (dd, *J* = 8.0, 4.0, 1H), 6.92 (d, *J* = 8.0 Hz, 1H), 6.81 (d, *J* = 8.0 Hz, 1H), 6.66 (s, 1H), 6.54 (s, 1H), 6.10 (d, *J* = 16.0 Hz, 1H), 6.05–6.0 (m, 2H), 5.79 (d, *J* = 16.0 Hz, 1H), 4.50 (s, 1H), 4.23 (d, *J* = 16.0 Hz, 1H), 4.01 (d, *J* = 16.0 Hz, 1H), 3.89 (s, 3H), 3.83 (s, 3H), 3.18–3.06 (m, 1H), 2.95–2.78 (m, 3H), 2.21 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 198.6, 148.2, 147.7, 146.4, 143.8, 140.6, 131.5, 128.4, 127.8, 125.0, 123.4, 123.1, 117.8, 111.5, 111.3, 106.5, 101.7, 63.3, 56.1, 55.9, 51.5, 48.5, 28.4, 27.5. IR (KBr) 2922.1, 1663.9, 1597.0, 1513.1, 1463.4, 1365.0, 1139.7, 1043.8, 993.0, 913.8, 816.9, 729.8, 646.0 cm^{-1}. HRMS (CI^+) (m/z) calcd. for $C_{25}H_{25}NO_5$ $[M+H]^+$ 420.1811; found 420.1700.

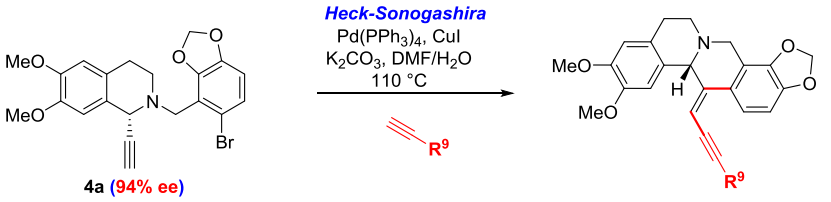


9g

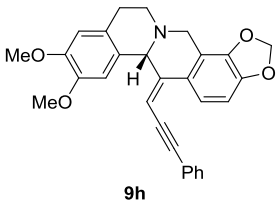
9g (70.0 mg) was obtained by using **General Procedure G** from **4a** (86 mg, 0.2 mmol) in 78% yield as colorless oil: $[\alpha]^{25}_D$ = +117.0 (*c* = 0.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.65 (d, *J* = 8.0 Hz, 1H), 6.93 (d, *J* = 8.0 Hz, 1H), 6.79 (d, *J* = 8.0 Hz, 1H), 6.66 (s, 1H), 6.61 (s, 1H), 6.01 (d, *J* = 8.0 Hz, 2H), 5.97 (d, *J* = 8.0 Hz, 1H), 4.51 (s, 1H), 4.20 (d, *J* = 16.0 Hz, 1H), 4.02 (d, *J* = 16.0 Hz, 1H), 3.89 (s, 3H), 3.84 (s, 3H), 3.72 (s, 3H), 3.20–3.01 (m, 1H), 2.97–2.76 (m, 3H), 1.78 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 169.1, 148.1,

147.3, 147.0, 143.6, 143.2, 135.7, 128.5, 128.0, 127.9, 125.4, 123.5, 120.9, 117.6, 111.5, 111.4, 106.4, 101.6, 63.56, 56.1, 55.8, 51.8, 51.4, 48.6, 28.4, 12.9. IR (KBr) 2925.6, 1704.5, 1608.2, 1513.1, 1464.5, 1353.3, 1258.4, 1114.4, 1042.9, 915.1, 816.5, 736.4, 646.7 cm^{-1}. HRMS (CI^+) (m/z) calcd. for $C_{26}H_{27}NO_6$ $[M+H]^+$ 450.1917; found 450.1893.

2.8. Preparation of Compounds 9h–9j via Cascade Heck-Sonogashira Reaction

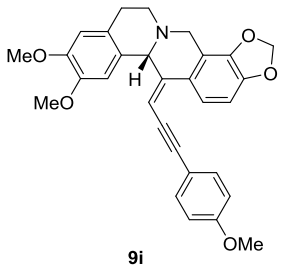


General Procedure H (Heck-Sonogashira): To a solution of compound **4a** (86 mg, 0.2 mmol) and alkyne (1 mmol) in DMF/H_2O (6.0 mL/2.0 mL) were added K_2CO_3 (55.2 mg, 0.4 mmol) and CuI (3.8 mg, 0.02 mmol). The solution was bubbled with a stream of dry nitrogen gas for 15 min before the addition of $Pd(PPh_3)_4$ (11.5 mg, 0.01 mmol). The mixture was stirred at 110 °C for 3 h. The reaction mixture was then diluted with EtOAc, washed with water and saturated brine. The organic layers were dried with Na_2SO_4 and concentrated. The residue was purified by column chromatography on silica gel to afford compounds **9h–9j**.



9h

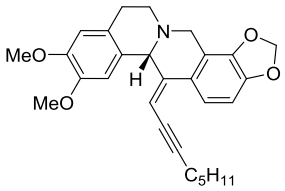
9h (71 mg) was obtained by using **General Procedure H** from **4a** (86 mg, 0.2 mmol) in 79% yield as colorless oil: $[\alpha]^{25}_D$ = +91.5 (*c* = 0.2, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ = 8.17 (d, *J* = 8.0 Hz, 1H), 7.41–7.34 (m, 2H), 7.34–7.27 (m, 3H), 6.81 (d, *J* = 8.0 Hz, 1H), 6.66 (s, 1H), 6.59 (s, 1H), 6.07–5.96 (m, 2H), 5.36 (s, 1H), 4.59 (s, 1H), 4.32 (d, *J* = 16.0 Hz, 1H), 4.00 (d, *J* = 16.0 Hz, 1H), 3.88 (s, 3H), 3.85 (s, 3H), 3.26–3.13 (m, 1H), 2.98–2.80 (m, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ = 148.2, 147.0, 146.9, 144.9, 143.4, 131.3, 128.3, 128.0, 127.7, 127.4, 125.8, 123.7, 121.7, 117.1, 111.7, 111.5, 106.2, 105.6, 101.5, 95.1, 88.9, 62.7, 56.0, 55.9, 51.0, 47.5, 28.1. IR (KBr) 2923.3, 2847.5, 1712.9, 1648.3, 1602.0, 1511.7, 1465.0, 1361.6, 1260.1, 1124.6, 1045.5, 912.8, 808.9, 729.5, 644.4, 532.7 cm^{-1}. HRMS (CI^+) (m/z) calcd. for $C_{29}H_{25}NO_4$ $[M+H]^+$ 452.1862; found 452.2018.



9i

9i (77 mg) was obtained by using **General Procedure H** from **4a** (86 mg, 0.2 mmol) in 80% yield as colorless oil: $[\alpha]^{25}_D$ = +77.5 (*c* = 0.2, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ = 8.18 (d, *J* = 8.0 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 2H), 6.83–6.79 (m, 3H), 6.65 (s, 1H), 6.59 (s, 1H), 6.00 (d, *J* = 0.4 Hz, 2H), 5.35 (s, 1H), 4.58 (s, 1H), 4.32 (d, *J* = 16.0 Hz, 1H), 3.99 (d, *J* = 16.0 Hz, 1H), 3.87 (s, 3H), 3.85 (s, 3H), 3.80 (s, 3H), 3.25–3.10 (m, 1H), 2.94–2.80 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 159.5, 148.1, 146.9, 143.8, 143.3, 132.7, 127.8, 127.4, 125.9, 121.5, 117.1, 115.9, 114.0, 111.7, 111.50, 106.2, 105.9, 101.4, 95.2, 87.6, 62.7, 56.0, 55.9, 55.3, 50.9, 47.4, 28.1. IR (KBr) 3003.5, 2925.2, 2837.6, 2189.3, 1604.5, 1511.1, 1465.0,

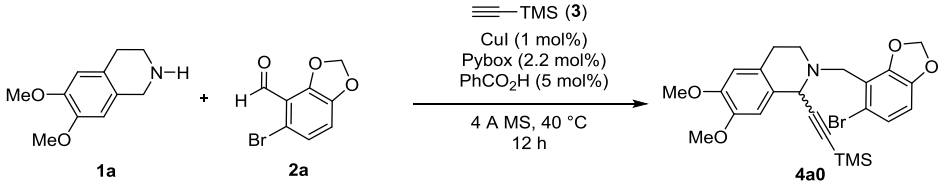
1363.7, 1254.0, 1178.9, 1121.9, 1042.2, 912.1, 824.1, 732.8, 648.3, 536.7, 472.4 cm^{-1}. HRMS (CI^+) (m/z) calcd. for $C_{30}H_{27}NO_5$ $[M+H]^+$ 482.1967; found 482.1891.



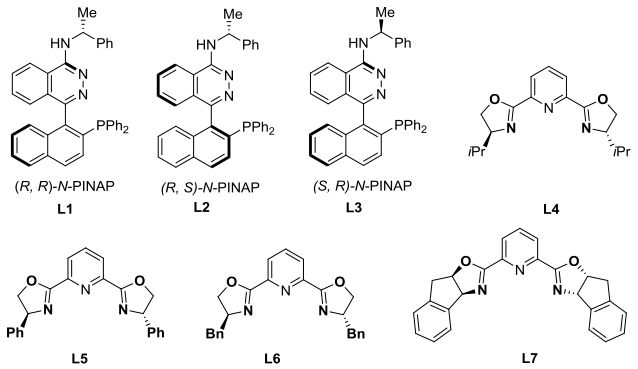
9j

9j (67 mg) was obtained by using **General Procedure H** from **4a** (86 mg, 0.2 mmol) in 75% yield as colorless oil: $[\alpha]^{25}_D$ = +134 (*c* = 0.2, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ = 8.14 (d, *J* = 8.0 Hz, 1H), 6.74 (d, *J* = 8.0, 1H), 6.63 (s, 1H), 6.54 (s, 1H), 5.98 (m, 2H), 5.15 (s, 1H), 4.52 (s, 1H), 4.27 (d, *J* = 16.0 Hz, 1H), 3.95 (d, *J* = 16.0 Hz, 1H), 3.86 (s, 3H), 3.82 (s, 3H), 3.23–3.10 (m, 1H), 2.90–2.86 (m, 3H), 2.35 (td, *J* = 7.0 Hz, 2.5 Hz, 2H), 1.60–1.45 (m, 2H) 1.44–1.22 (m, 4H), 0.89 (t, *J* = 8.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 148.1, 146.8, 146.6, 143.2, 142.6, 127.7, 127.3, 126.0, 121.2, 116.9, 111.6, 111.5, 106.5, 106.0, 101.3, 79.3, 62.7, 56.0, 55.9, 50.7, 47.4, 31.1, 28.3, 28.0, 22.2, 19.9, 14.0. IR (KBr) 2928.1, 2863.3, 1644.9, 1605.4, 1512.7, 1464.8, 1363.6, 1257.4, 1126.3, 1046.7, 992.8, 915.5, 808.1, 730.8, 637.4 cm^{-1}. HRMS (CI^+) (m/z) calcd. for $C_{28}H_{31}NO_4$ $[M]^+$ 445.2253; found 445.2254.

2.9. Screening of Chiral ligands for CuI-catalyzed Redox-A^3 Reaction (Table 1).



To a flame-dried bottom-rounded flask (25 mL) were added CuI (1.2 mg, 0.01 mmol), Pybox (0.022 mmol), and newly activated 4 Å molecular sieves (300 mg). To the reaction flask under nitrogen atmosphere were sequentially added toluene (10 mL), **1a** (270 mg, 1.4 mmol), **2a** (320.0 mg, 1.4 mmol), and **3a** (98 mg, 1.0 mmol). The reaction mixture was heated with an oil bath at 40 °C for 12 h. After TLC analysis indicated the completion of the reaction, the solvent was removed by evaporation, and the residue was purified by chromatography (hexane/EtOAc = 10:1) on silica gel to afford **4a0**. The HPLC spectra of different entries are shown in pages S-21–24.



Entry	Catalyst	Ligand	Conversion (%)	Yield (**4a0**, %)	*er*
1	CuI	**L1**	100	88	25:75
2	CuI	**L4**	58	72	55:45
3	$Cu(OTf)_2$	**L4**	0	0	0
4	CuI	**L5**	55	70	41:59
5	CuI	**L6**	64	78	30:70
6	CuI	**L7**	64	78	47:53
7	CuI	**L2**	100	94	97:3
8	CuI	**L3**	100	90	2.5:97.5

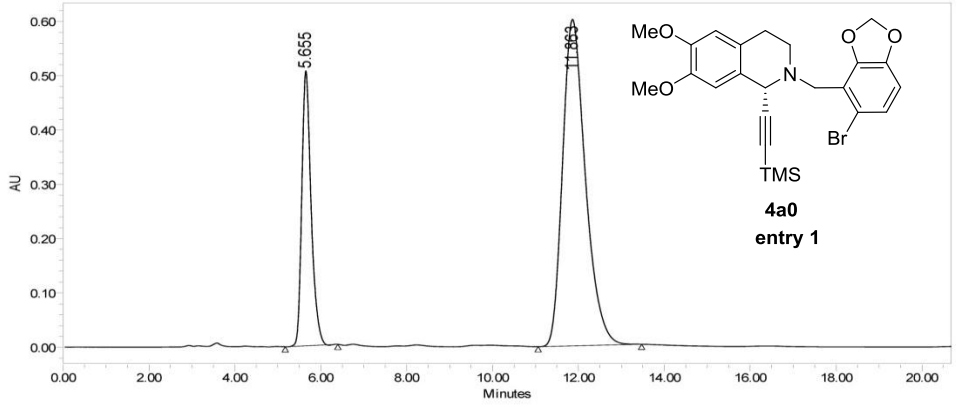
2.10. Copies of HPLC Chromatograms for Screening of Chiral ligands

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq6-40-1c(90/10)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	9/3/2016 2:50:17 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	3	Date Processed:	12/15/2016 10:39:24 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	40.00 Minutes	Sample Set Name	



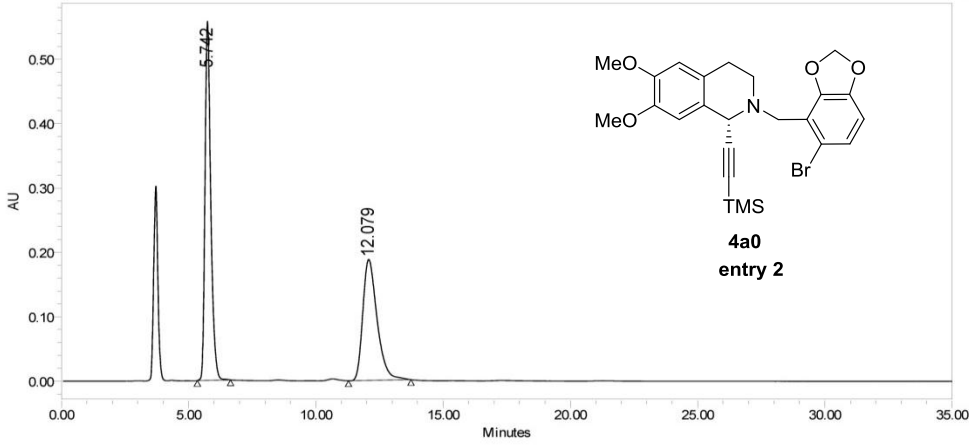
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	5.655	7632850	25.57	506223	45.71
2	11.863	22213595	74.43	601222	54.29

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq6-90-1-a(90/10)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	9/2/2016 3:31:53 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	3	Date Processed:	12/15/2016 10:24:59 AM HKT
Injection Volume:	5.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	35.00 Minutes	Sample Set Name	



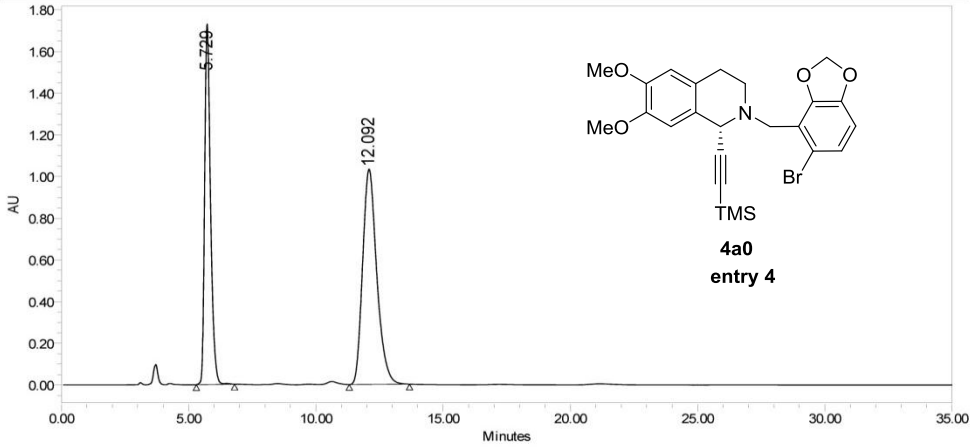
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	5.742	8765806	54.46	557616	74.80
2	12.079	7331146	45.54	187902	25.20

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq6-90-2(90/10)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	9/2/2016 4:19:18 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	4	Date Processed:	12/15/2016 10:26:06 AM HKT
Injection Volume:	5.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	35.00 Minutes	Sample Set Name	



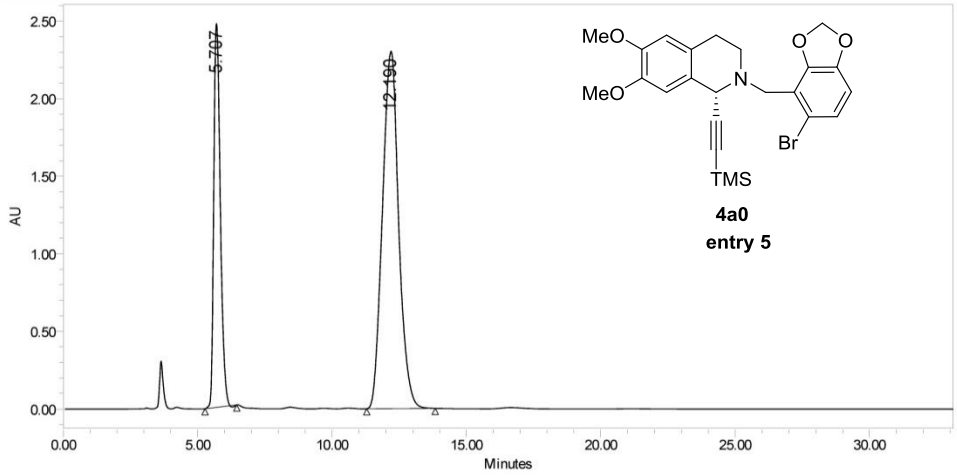
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	5.729	27803424	41.03	1729255	62.60
2	12.092	39953938	58.97	1033286	37.40

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq6-90-3(90/10)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	9/2/2016 5:17:46 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	5	Date Processed:	12/15/2016 10:27:04 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	35.00 Minutes	Sample Set Name	



4a0
entry 5

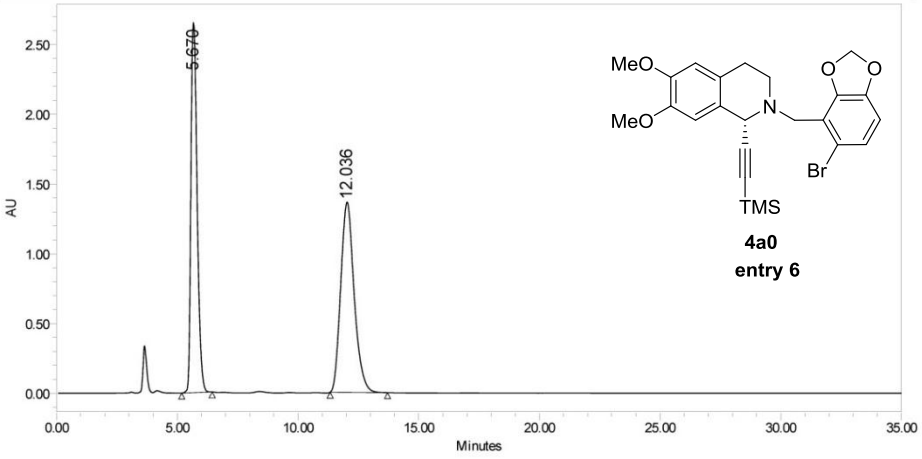
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	5.707	41945205	30.29	2471744	51.79
2	12.190	96552760	69.71	2301160	48.21

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq6-90-4(90/10)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	9/2/2016 5:51:25 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	6	Date Processed:	12/15/2016 10:28:04 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	35.00 Minutes	Sample Set Name	



4a0
entry 6

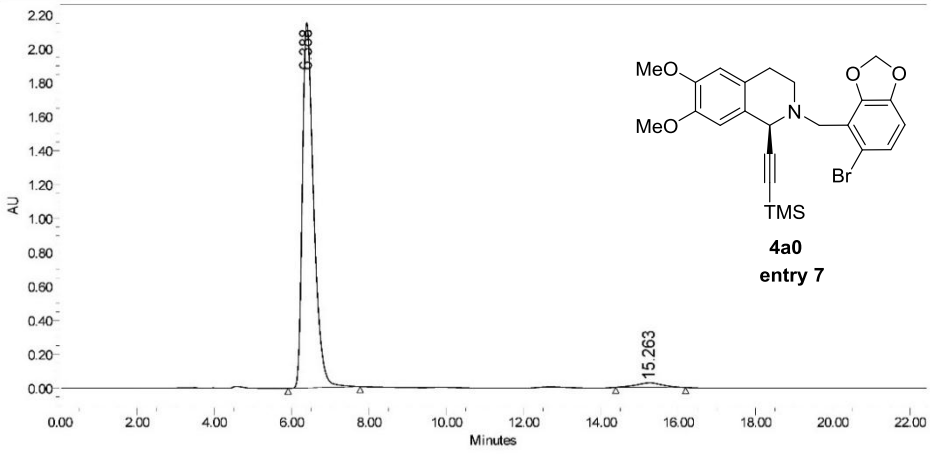
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	5.670	47617296	47.38	2653154	66.03
2	12.036	52892575	52.62	1364915	33.97

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	5-185-1	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	7/14/2016 2:38:18 AM HKT
Vial:	1	Acq. Method:	ZSQ_INDOL
Injection #:	1	Date Processed:	12/18/2016 1:02:24 PM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	30.00 Minutes	Sample Set Name	



4a0
entry 7

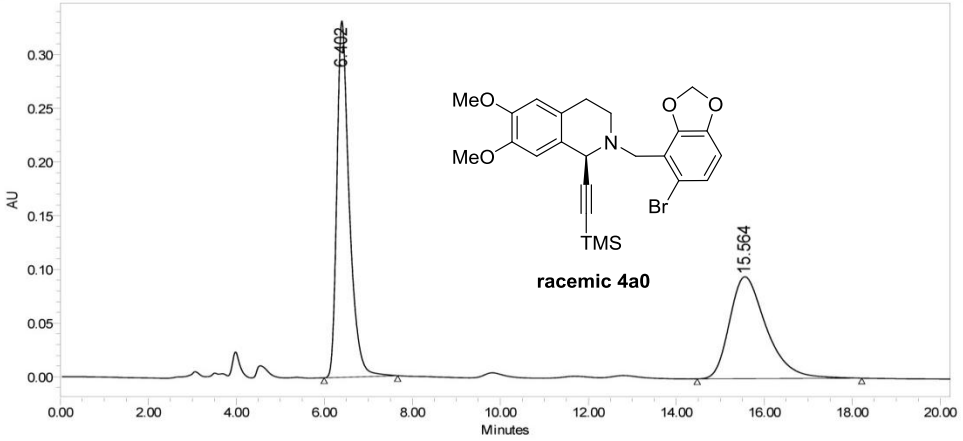
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	6.388	43494581	97.05	2151324	98.73
2	15.263	1322223	2.95	27721	1.27

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	2-99-1-A	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	7/13/2016 2:28:50 AM HKT
Vial:	1	Acq. Method:	ZSQ_INDOL
Injection #:	1	Date Processed:	12/15/2016 10:21:39 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	30.00 Minutes	Sample Set Name	



racemic 4a0

	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	6.402	6505087	54.14	331809	77.78
2	15.564	5509594	45.86	94812	22.22

3. X-ray data of Compound 4a (CCDC 1524047).

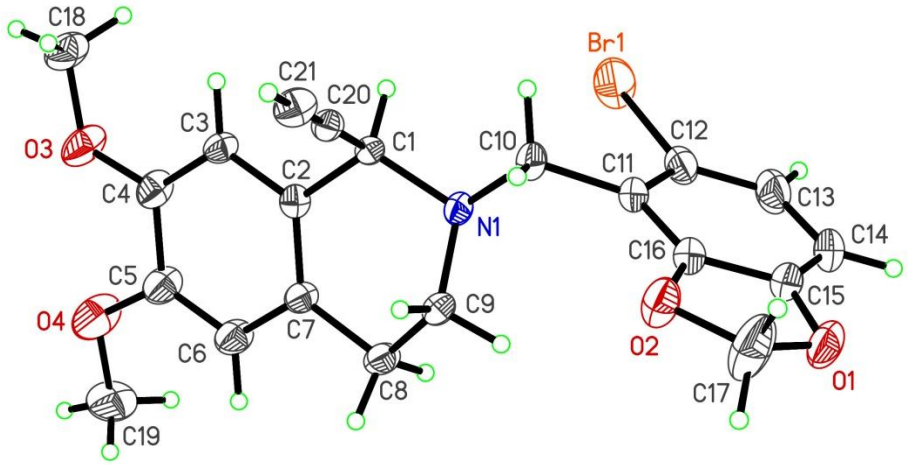


Table 1 Crystal data and structure refinement for qiang5CuLT.

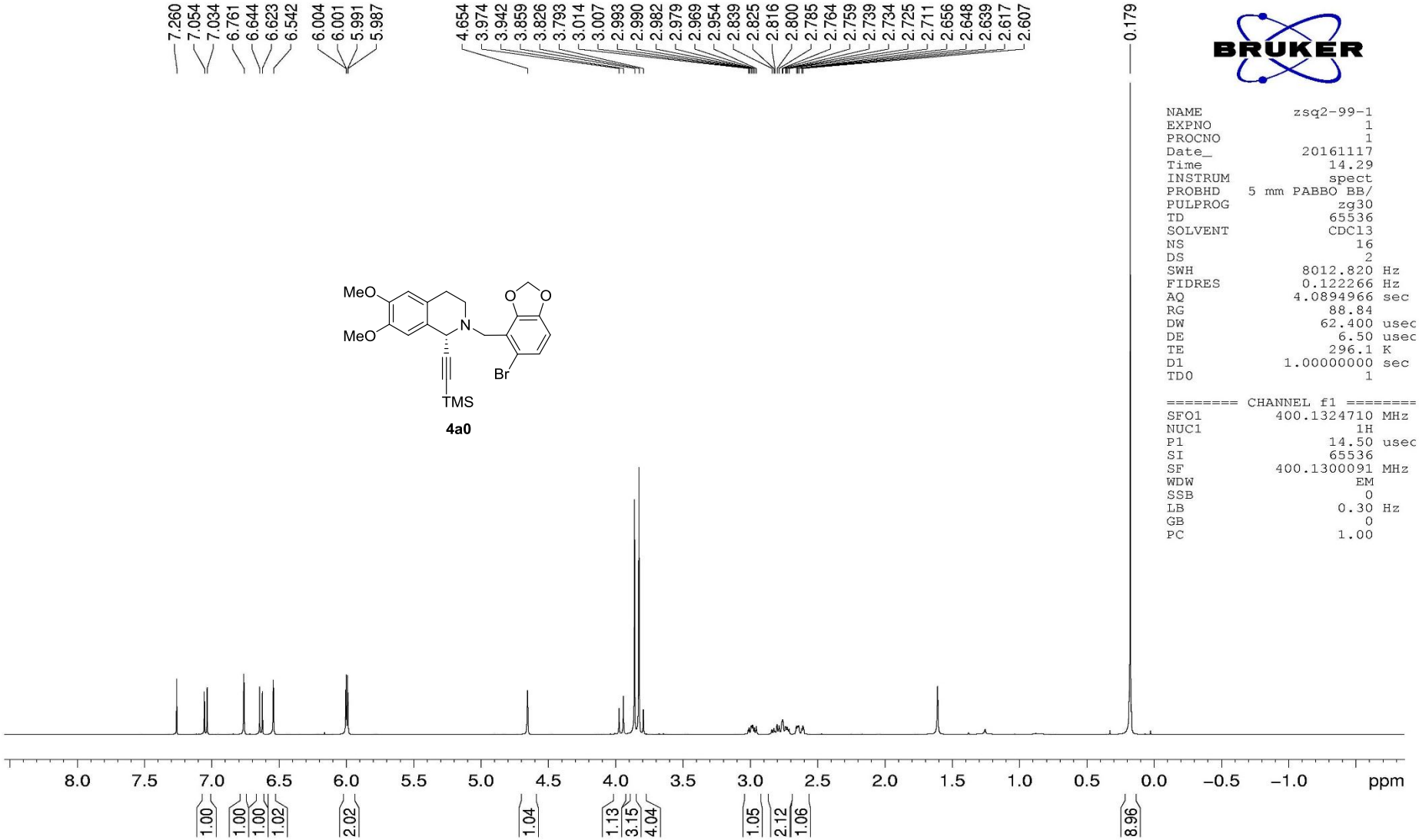
Identification code	qiang5CuLT
Empirical formula	$C_{21}H_{20}BrNO_4$
Formula weight	430.29
Temperature/K	173.15
Crystal system	monoclinic
Space group	$P2_1$
a/Å	9.4538(2)
b/Å	8.19410(10)
c/Å	12.3501(2)
α/°	90
β/°	90.0050(10)
γ/°	90
Volume/Å^3	956.71(3)
Z	2
ρ_{calc}g/cm^3	1.494
μ/mm^{-1}	3.155
F(000)	440.0
Crystal size/mm^3	0.2 × 0.2 × 0.18
Radiation	CuKα (λ = 1.54178)
2Θ range for data collection/°	7.158 to 133.56
Index ranges	-11 ≤ h ≤ 11, -9 ≤ k ≤ 9, -14 ≤ l ≤ 14
Reflections collected	12931
Independent reflections	3352 [R_{int} = 0.0216, R_{sigma} = 0.0163]
Data/restraints/parameters	3352/1/246
Completeness to theta = 66.5°	98.7%

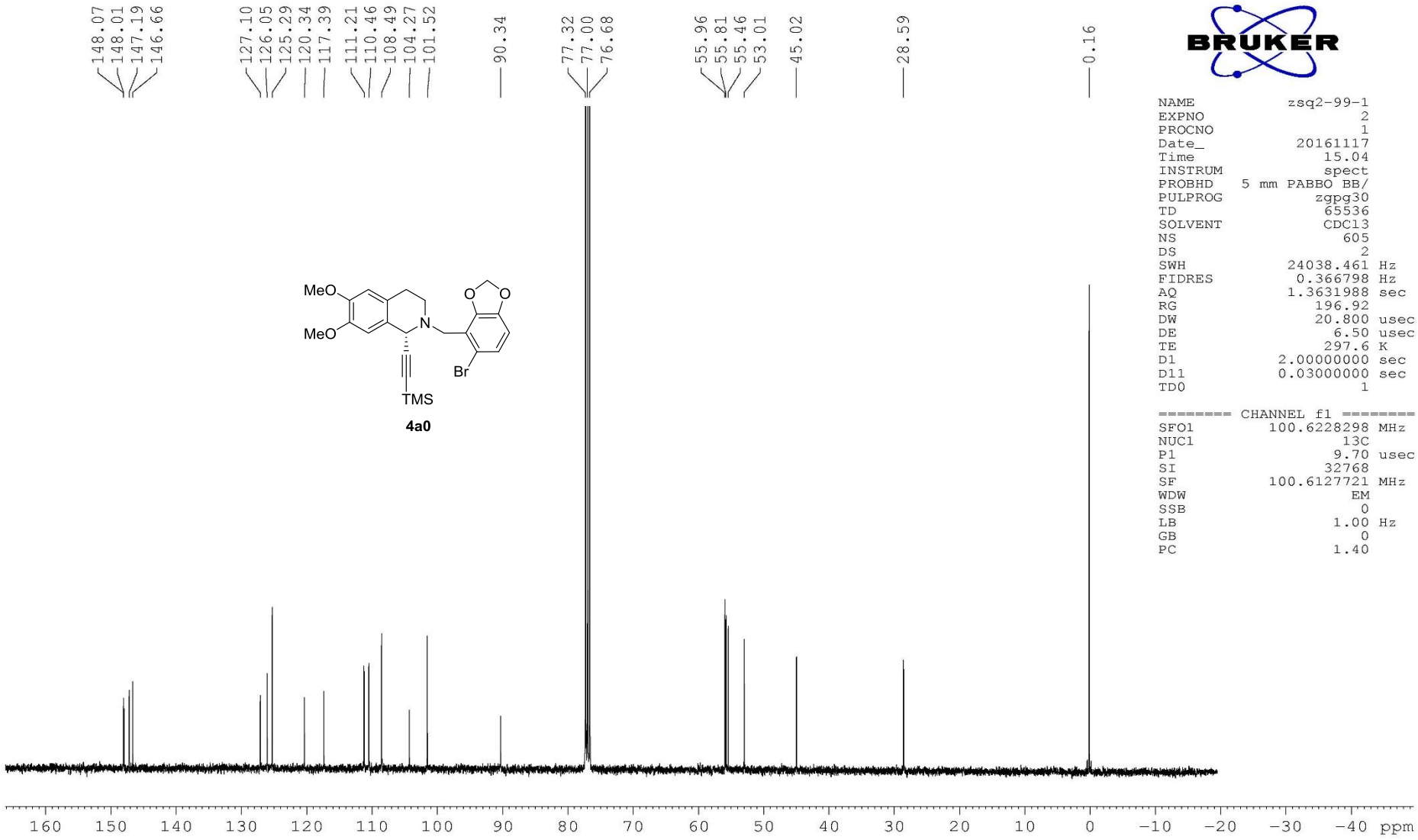
Goodness-of-fit on F^2	1.003
Final R indexes [I>=2σ (I)]	R_1 = 0.0245, wR_2 = 0.0650
Final R indexes [all data]	R_1 = 0.0248, wR_2 = 0.0653
Largest diff. peak/hole / e $Å^{-3}$	0.36/-0.36
Flack parameter	-0.024(5)

Crystal structure determination of [qiang5CuLT]

Crystal Data for $C_{21}H_{20}BrNO_4$ (M =430.29 g/mol): monoclinic, space group $P2_1$ (no. 4), a = 9.4538(2) Å, b = 8.19410(10) Å, c = 12.3501(2) Å, β = 90.0050(10)°, V = 956.71(3) $Å^3$, Z = 2, T = 173.15 K, μ(CuKα) = 3.155 mm^{-1}, *Dcalc* = 1.494 g/cm^3, 12931 reflections measured (7.158° ≤ 2Θ ≤ 133.56°), 3352 unique (R_{int} = 0.0216, R_{sigma} = 0.0163) which were used in all calculations. The final R_1 was 0.0245 (I > 2σ(I)) and wR_2 was 0.0653 (all data).

4. Copies of NMR Spectra

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126.05
125.29
120.34
117.39
111.21
110.46
108.49
104.27
101.52
90.34
77.32
77.00
76.68
55.96
55.81
55.46
53.01
45.02
28.59
0.16
MeO
MeO
O
O
N
Br
TMS
4a0
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EXPNO 2
PROCNO 1
Date_ 20161117
Time 15.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 605
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127721 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
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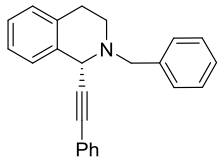

4a1

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PROCNO                   1
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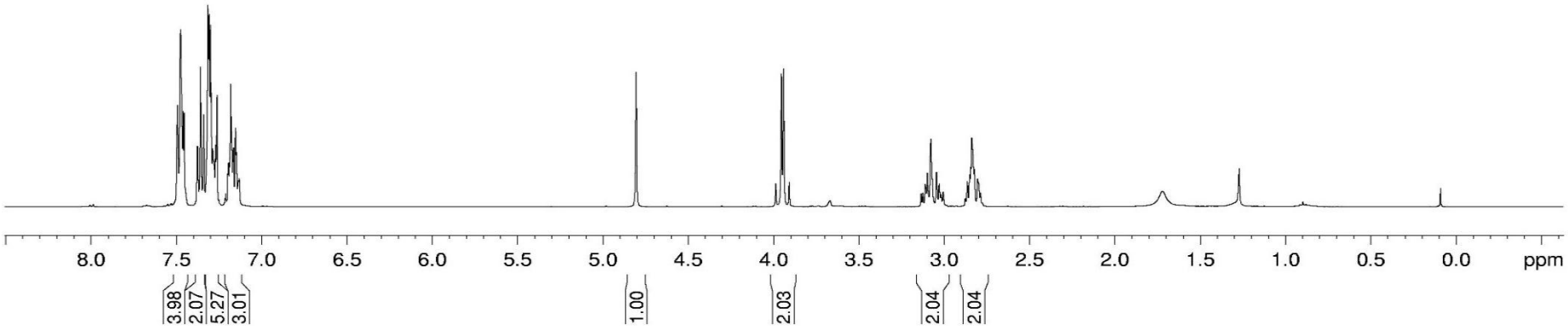
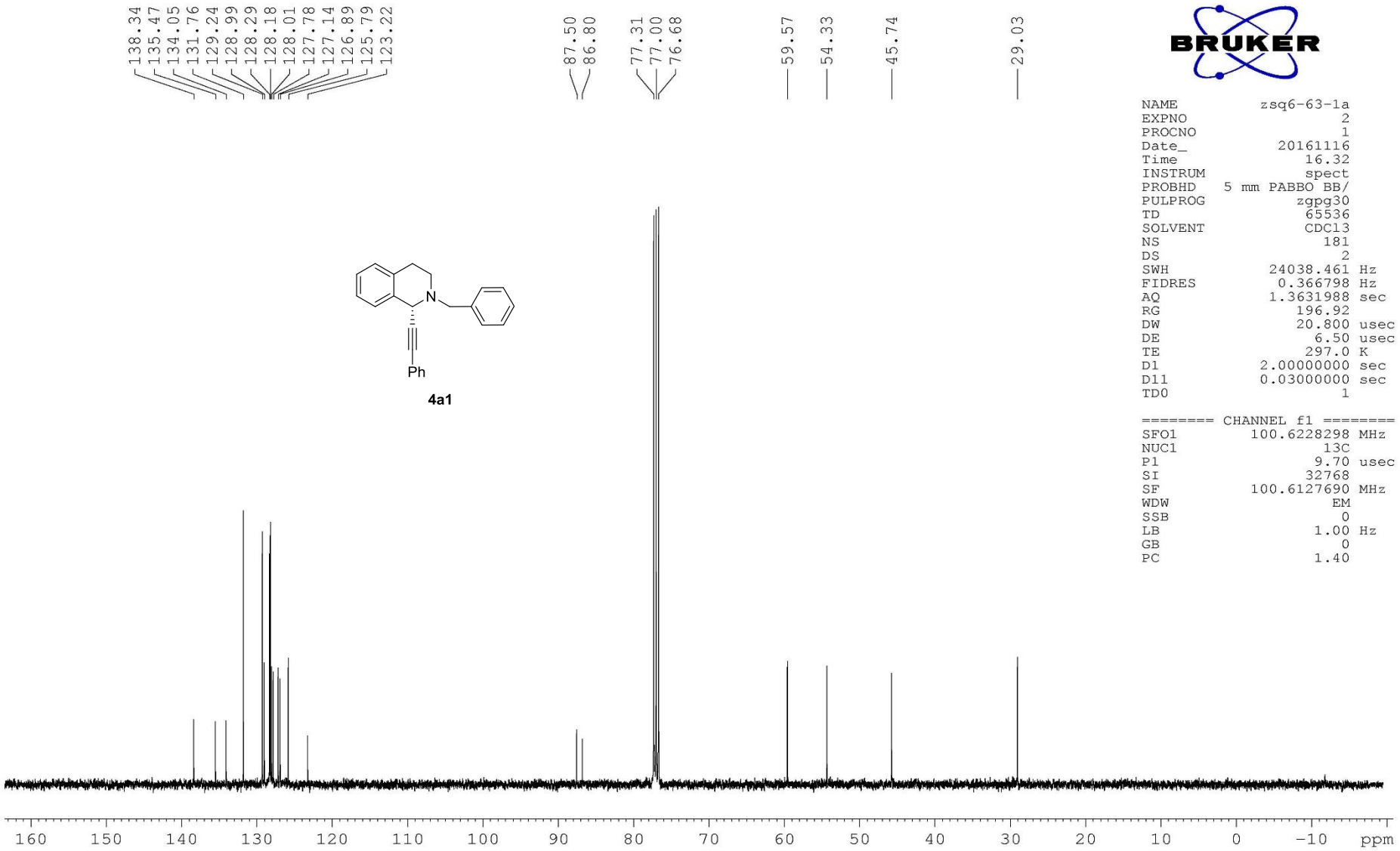
Time                 16.26
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8012.820 Hz
FIDRES               0.122266 Hz
AQ                4.0894966 sec
RG                   88.84
DW                   62.400 usec
DE                    6.50 usec
TE                   296.1 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                     1H
P1                   14.50 usec
SI                   65536
SF              400.1300094 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

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127.78
127.14
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125.79
123.22
87.50
86.80
77.31
77.00
76.68
59.57
54.33
45.74
29.03
BRUKER
NAME zsq6-63-1a
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PROCNO 1
Date_ 20161116
Time 16.32
INSTRUM spect
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PULPROG zgpg30
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SOLVENT CDC13
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FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
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GB 0
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Ph
4a1
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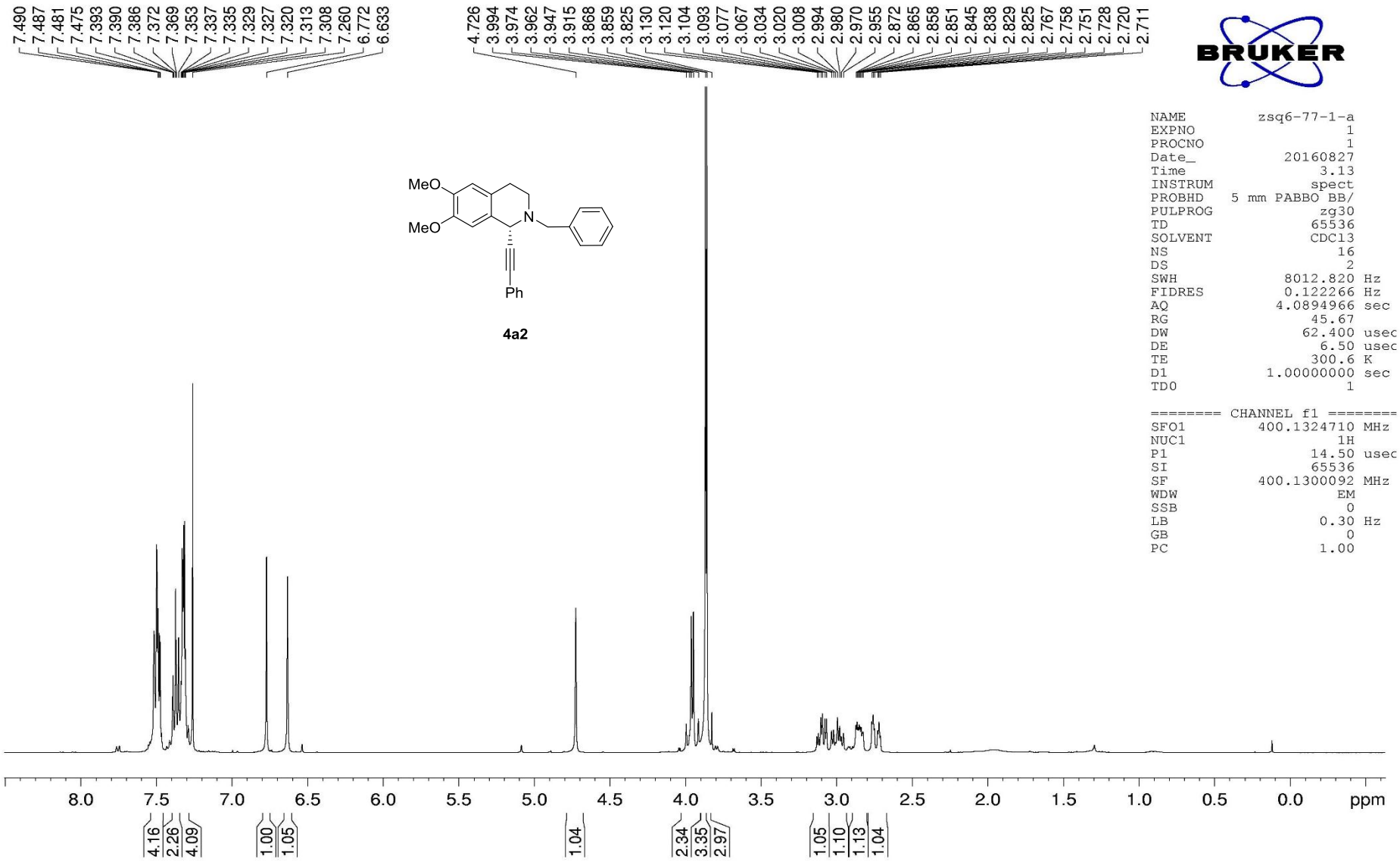
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7.327
7.320
7.313
7.308
7.260
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6.633
4.726
3.994
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3.962
3.947
3.915
3.868
3.859
3.825
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3.120
3.104
3.093
3.077
3.067
3.034
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2.865
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2.758
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2.728
2.720
2.711
MeO
MeO
N
Ph
4a2
NAME zsq6-77-1-a
EXPNO 1
PROCNO 1
Date_ 20160827
Time 3.13
INSTRUM spect
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PULPROG zg30
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SOLVENT CDCl3
NS 16
DS 2
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FIDRES 0.122266 Hz
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RG 45.67
DW 62.400 usec
DE 6.50 usec
TE 300.6 K
D1 1.00000000 sec
TD0 1
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NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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7.5
7.0
6.5
6.0
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5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
4.16
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1.00
1.05
1.04
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2.97
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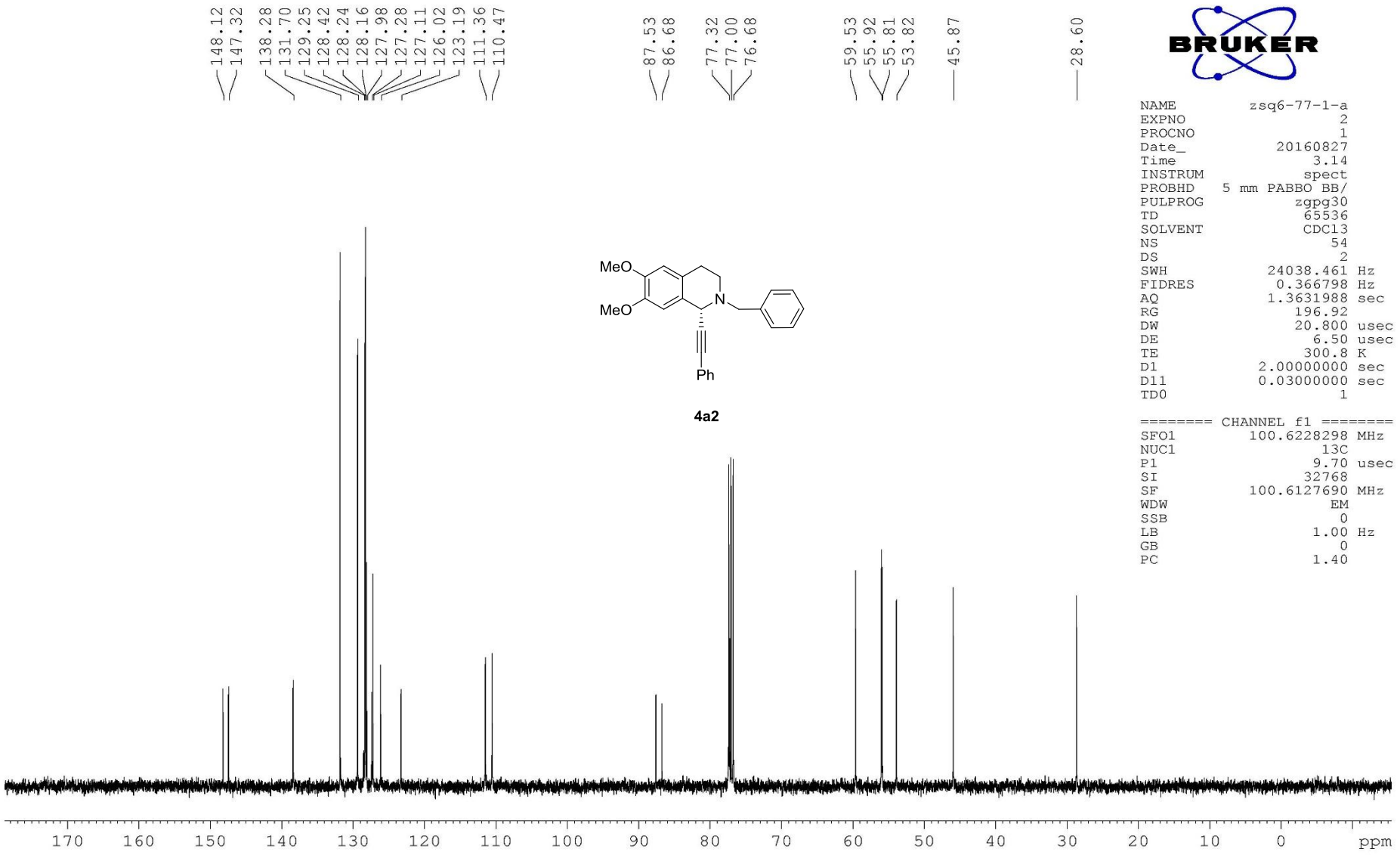
1.10
1.13
1.04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110.47
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86.68
77.32
77.00
76.68
59.53
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MeO
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4a2
NAME zsq6-77-1-a
EXPNO 2
PROCNO 1
Date_ 20160827
Time 3.14
INSTRUM spect
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 54
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 300.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
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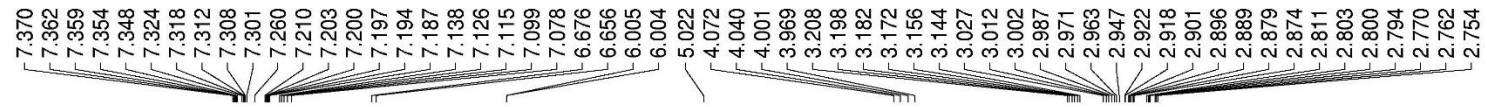

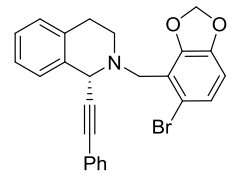

4a3

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PROCNO                  1
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Time                3.22
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894966 sec
RG                  49.32
DW                  62.400 usec
DE                   6.50 usec
TE                  300.6 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
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SI                  65536
SF           400.1300093 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
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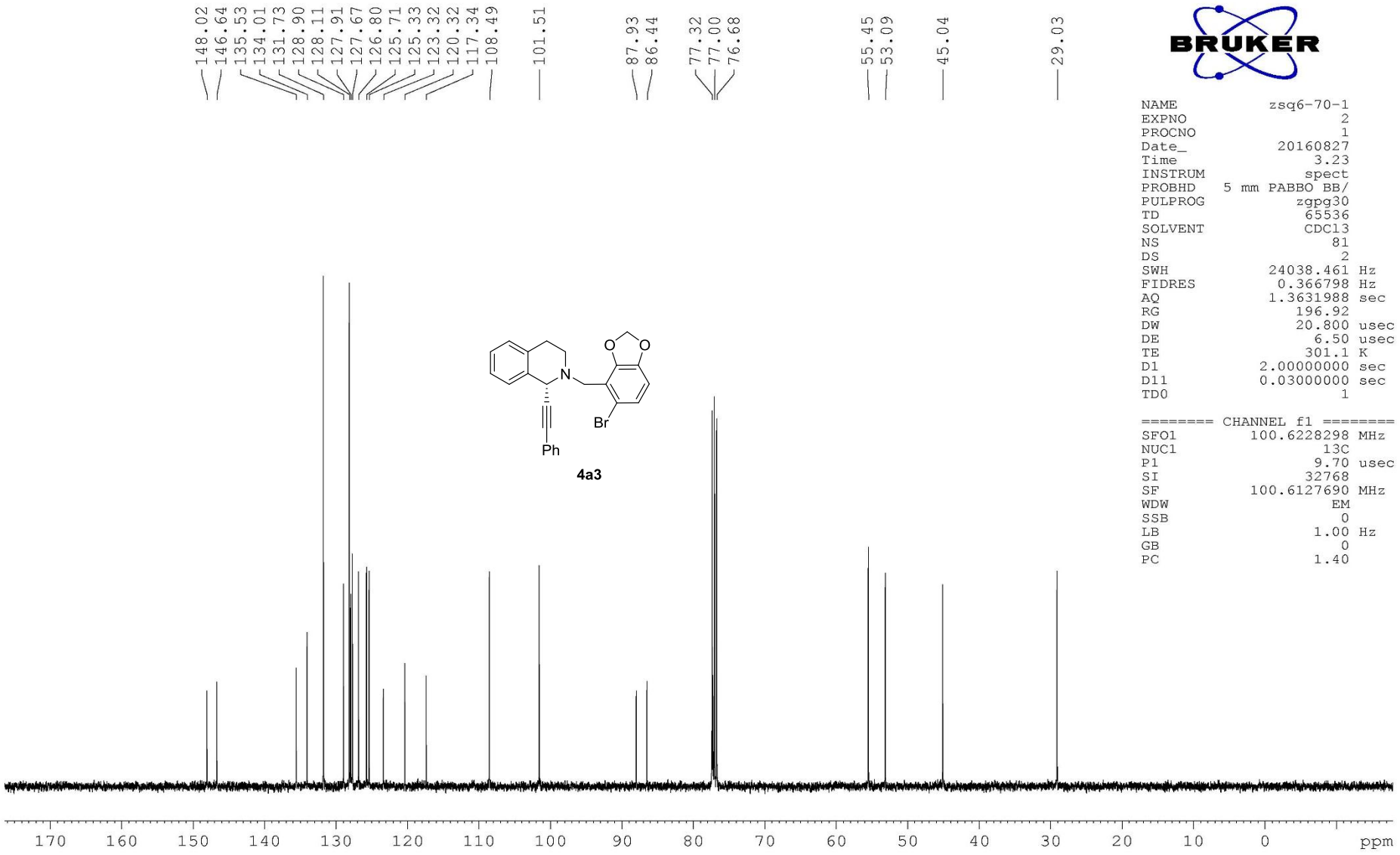
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123.32
120.32
117.34
108.49
101.51
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86.44
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77.00
76.68
55.45
53.09
45.04
29.03
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NAME zsq6-70-1
EXPNO 2
PROCNO 1
Date_ 20160827
Time 3.23
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 81
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 301.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
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O
N
Br
Ph
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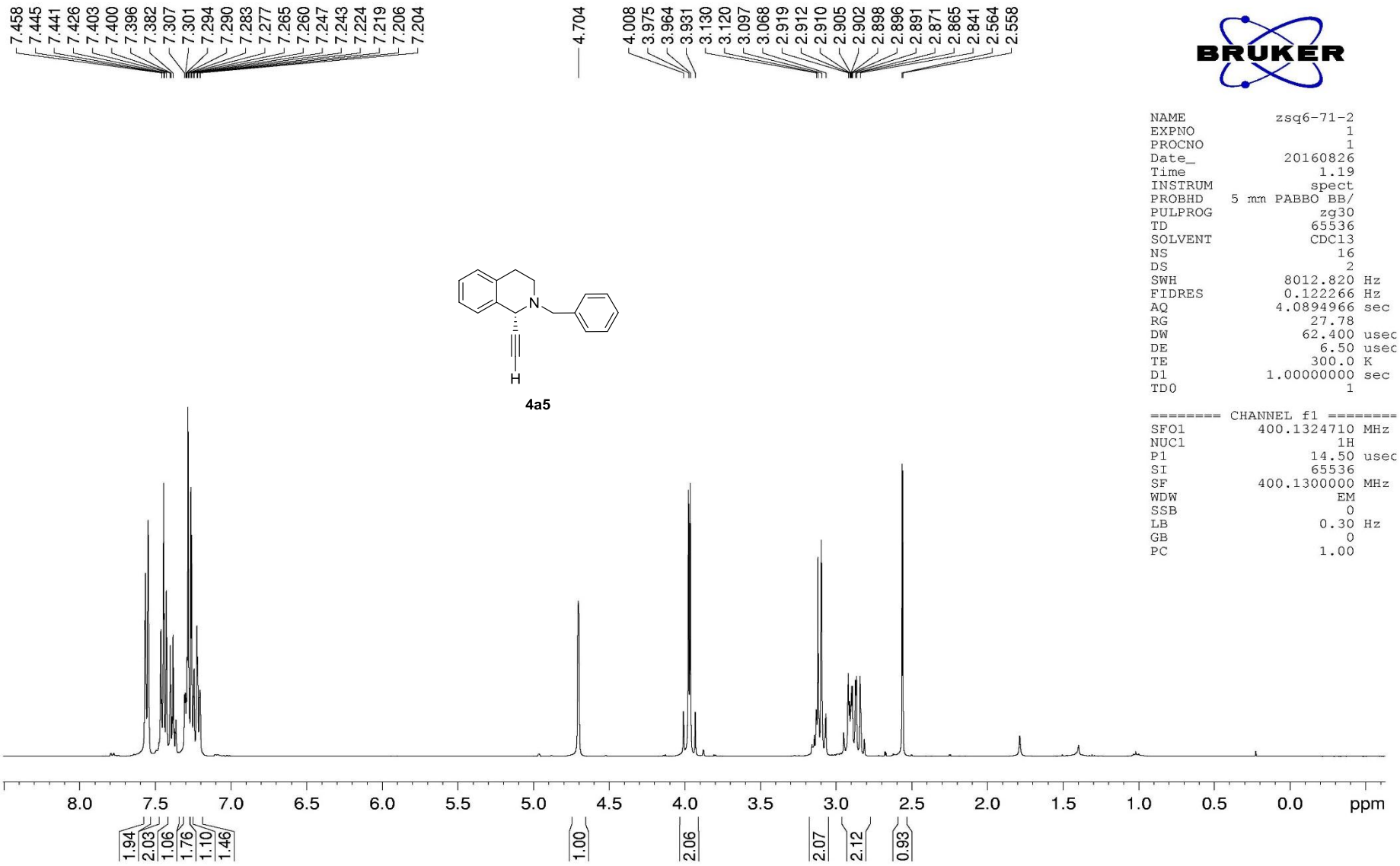
ppm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7.265
7.260
7.247
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7.224
7.219
7.206
7.204
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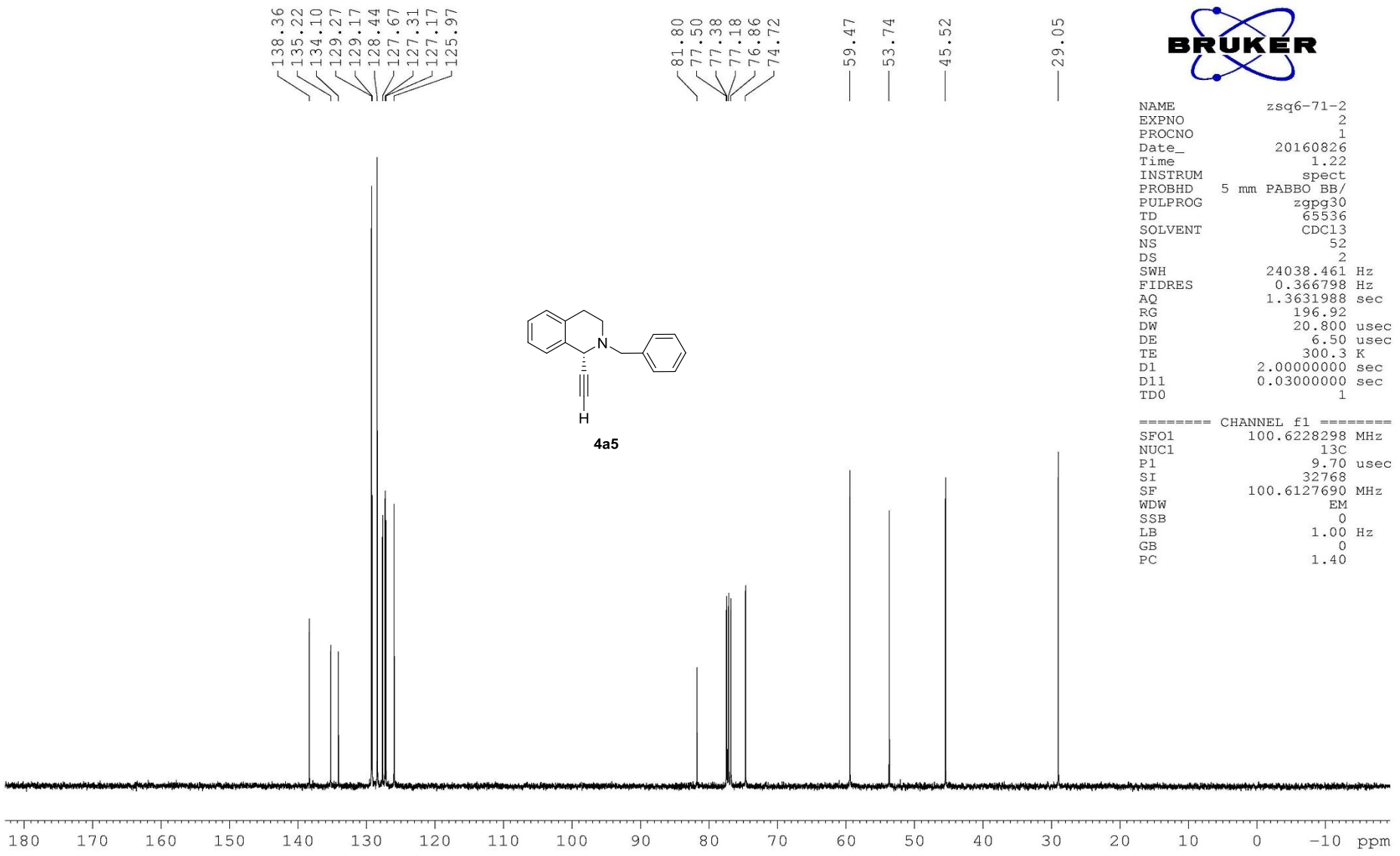
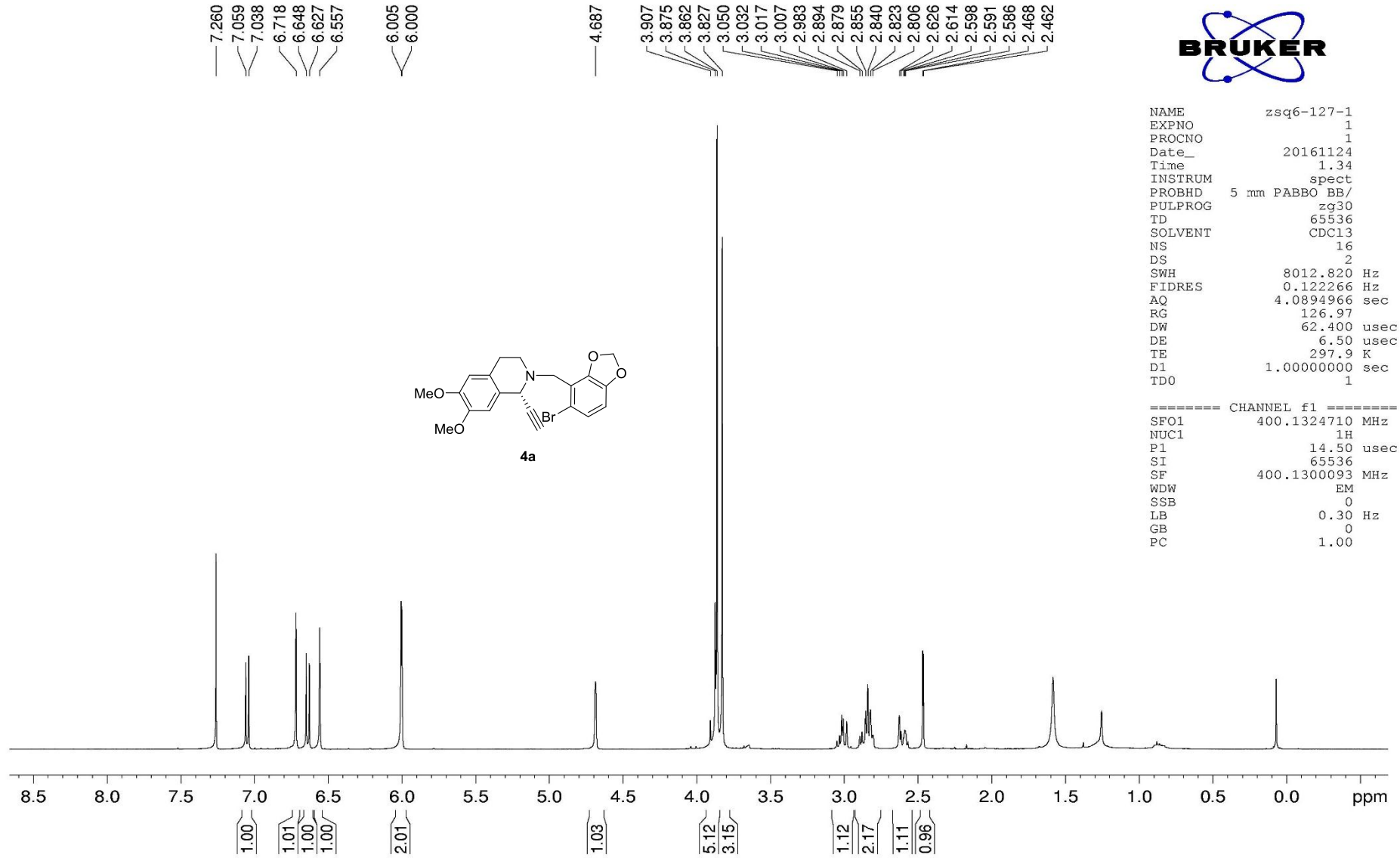
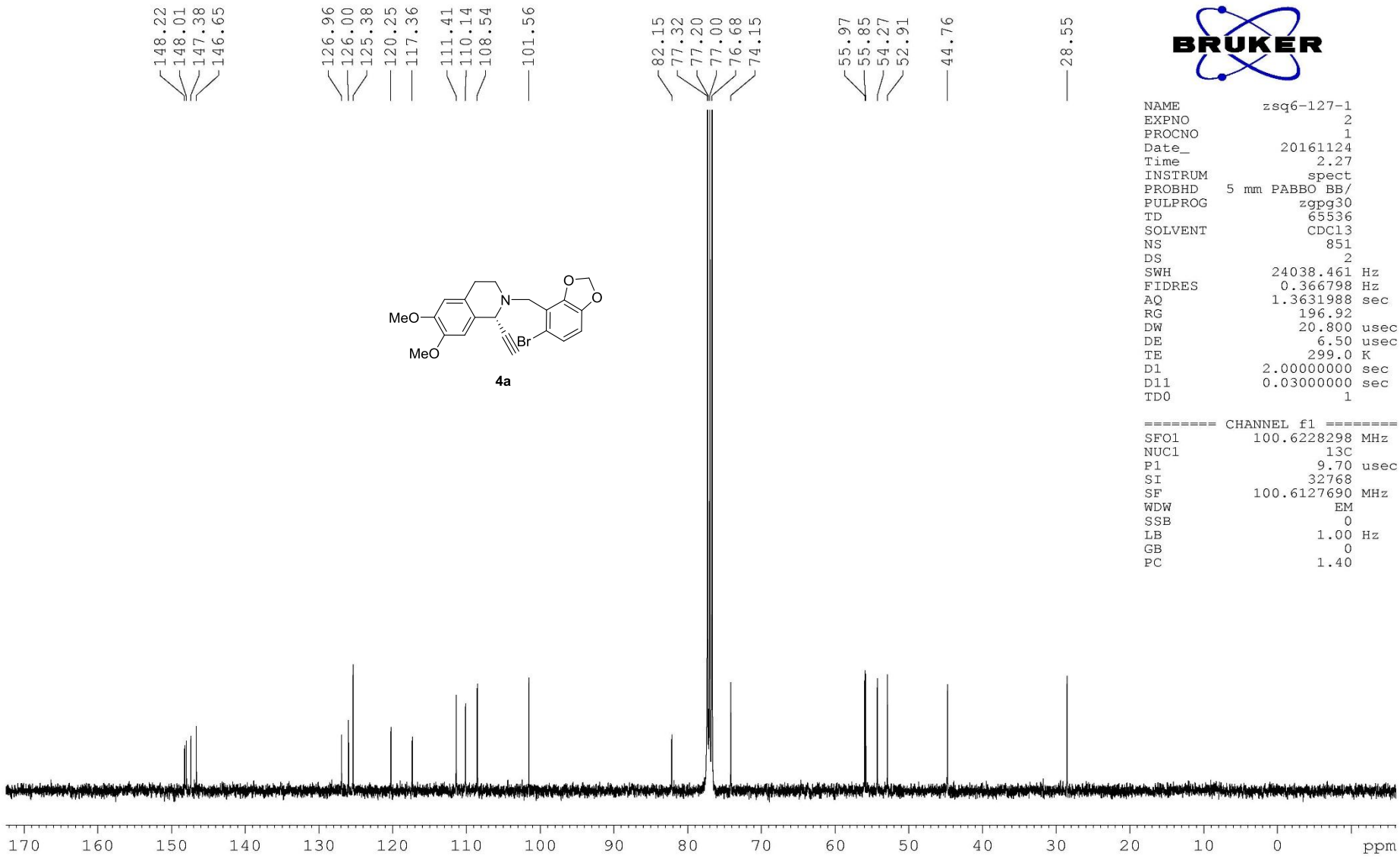
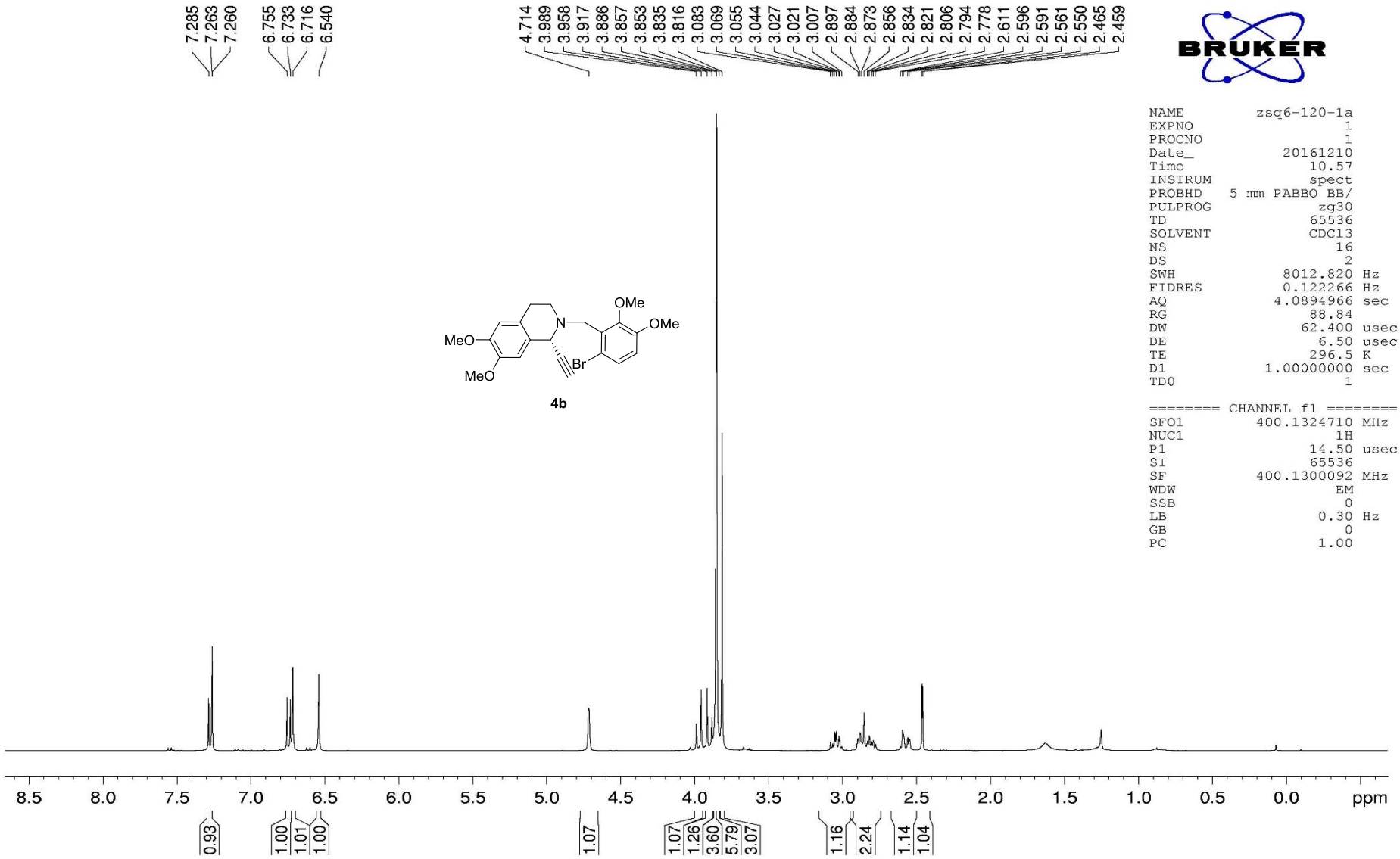
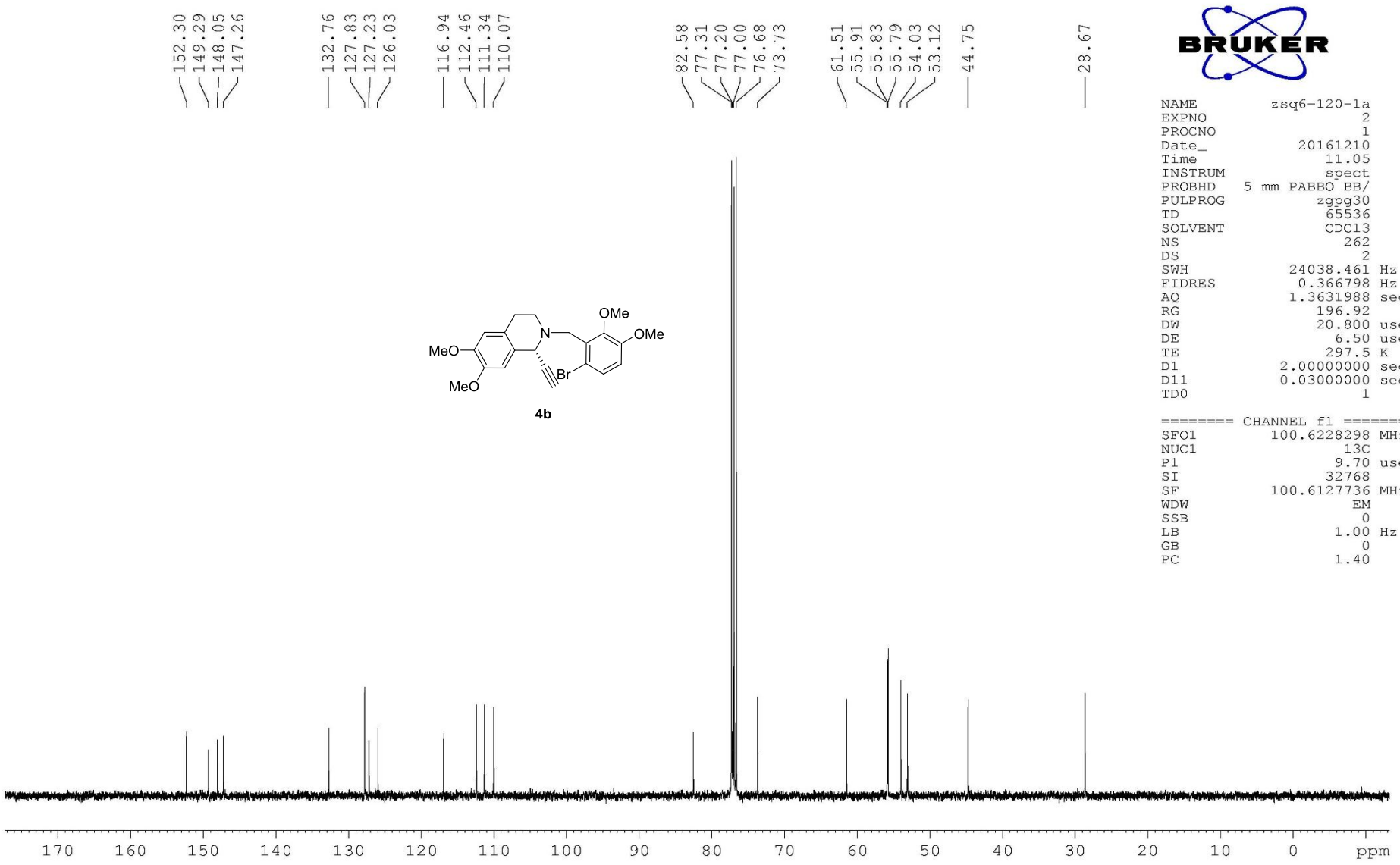
3.975
3.964
3.931
3.130
3.120
3.097
3.068
2.919
2.912
2.910
2.905
2.902
2.898
2.896
2.891
2.871
2.865
2.841
2.564
2.558
BRUKER
NAME zsq6-71-2
EXPNO 1
PROCNO 1
Date_ 20160826
Time 1.19
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 27.78
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
H
4a5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.94
2.03
1.06
1.76
1.10
1.46
1.00
2.06
2.07
2.12
0.93

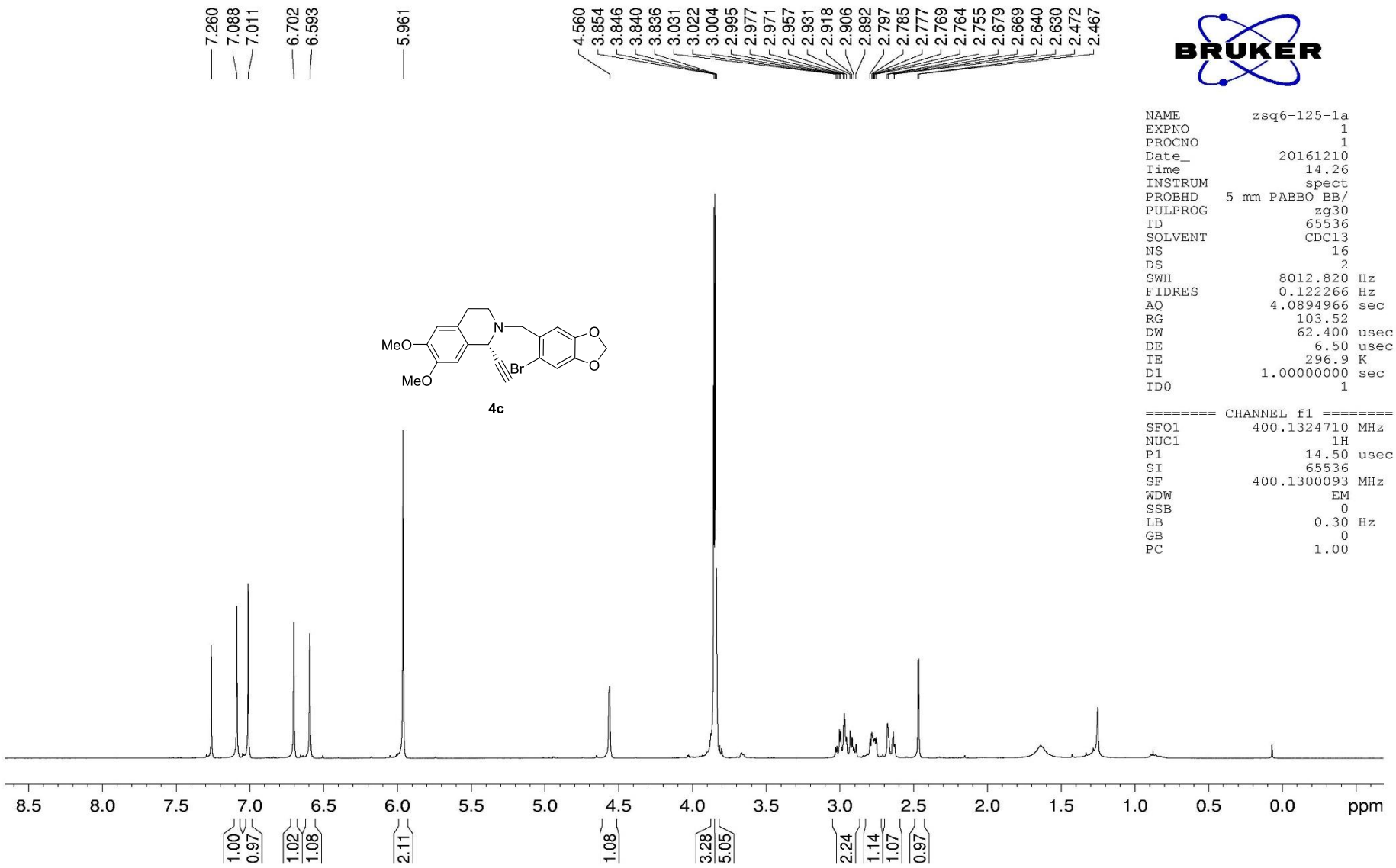

138.36
135.22
134.10
129.27
129.17
128.44
127.67
127.31
127.17
125.97
81.80
77.50
77.38
77.18
76.86
74.72
59.47
53.74
45.52
29.05
BRUKER
NAME zsq6-71-2
EXPNO 2
PROCNO 1
Date_ 20160826
Time 1.22
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 52
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 300.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
H
4a5
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm

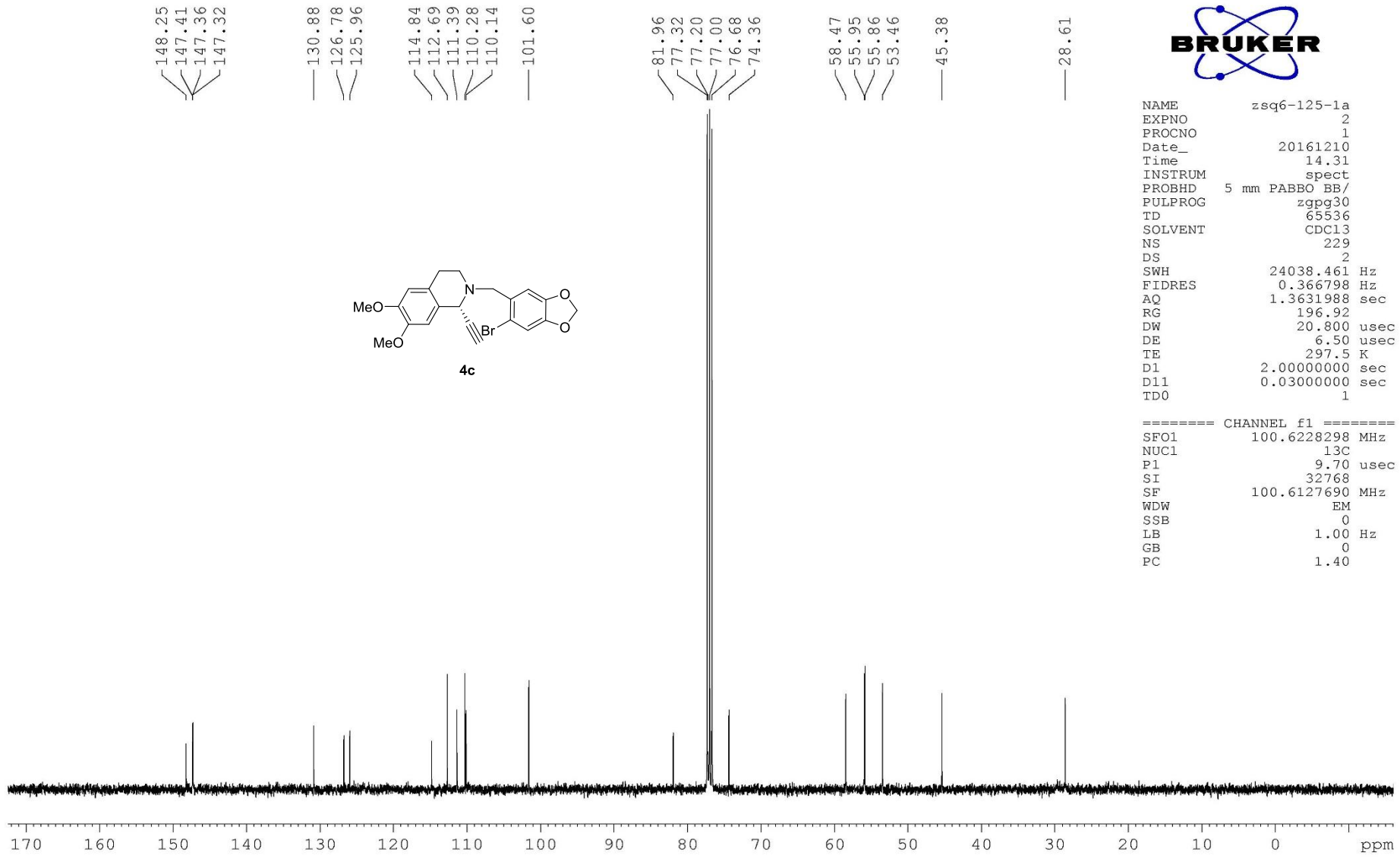

7.260
7.059
7.038
6.718
6.648
6.627
6.557
6.005
6.000
4.687
3.907
3.875
3.862
3.827
3.050
3.032
3.017
3.007
2.983
2.894
2.879
2.855
2.840
2.823
2.806
2.626
2.614
2.598
2.591
2.586
2.468
2.462
BRUKER
NAME zsq6-127-1
EXPNO 1
PROCNO 1
Date_ 20161124
Time 1.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 126.97
DW 62.400 usec
DE 6.50 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
MeO
N
O
O
Br
4a
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.01
1.00
1.00
2.01
1.03
5.12
3.15
1.12
2.17
1.11
0.96

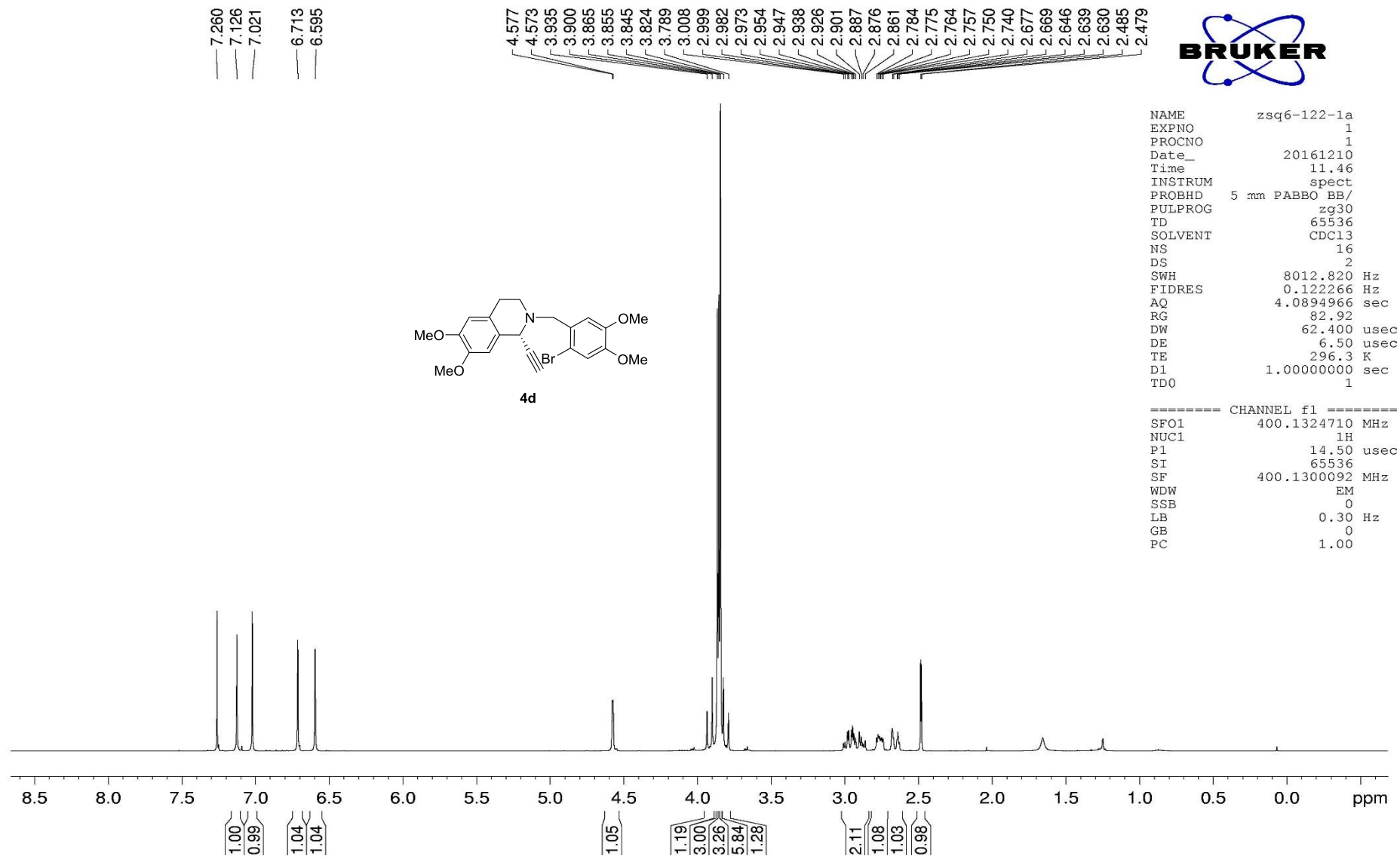

148.22
148.01
147.38
146.65
126.96
126.00
125.38
120.25
117.36
111.41
110.14
108.54
101.56
82.15
77.32
77.20
77.00
76.68
74.15
55.97
55.85
54.27
52.91
44.76
28.55
MeO
MeO
N
O
O
Br
4a
BRUKER
NAME zsq6-127-1
EXPNO 2
PROCNO 1
Date_ 20161124
Time 2.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 851
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

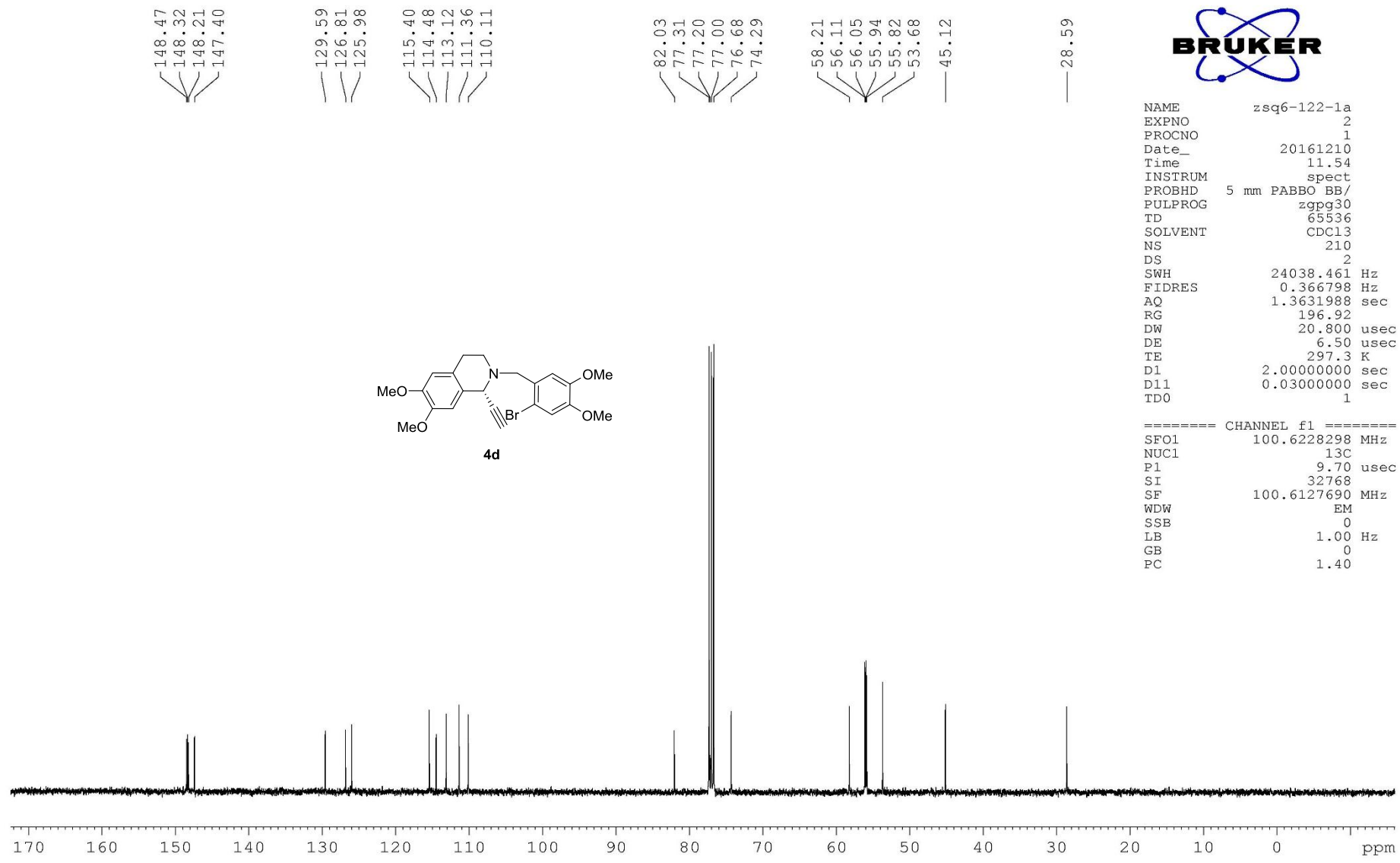

7.285
7.263
7.260
6.755
6.733
6.716
6.540
4.714
3.989
3.958
3.917
3.886
3.857
3.853
3.835
3.816
3.083
3.069
3.055
3.044
3.027
3.021
3.007
2.897
2.884
2.873
2.856
2.834
2.821
2.806
2.794
2.778
2.611
2.596
2.591
2.561
2.550
2.465
2.459
BRUKER
NAME zsq6-120-1a
EXPNO 1
PROCNO 1
Date_ 20161210
Time 10.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 88.84
DW 62.400 usec
DE 6.50 usec
TE 296.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OMe
OMe
MeO
MeO
N
Br
4b
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.93
1.00
1.01
1.00
1.07
1.07
1.26
3.60
5.79
3.07
1.16
2.24
1.14
1.04

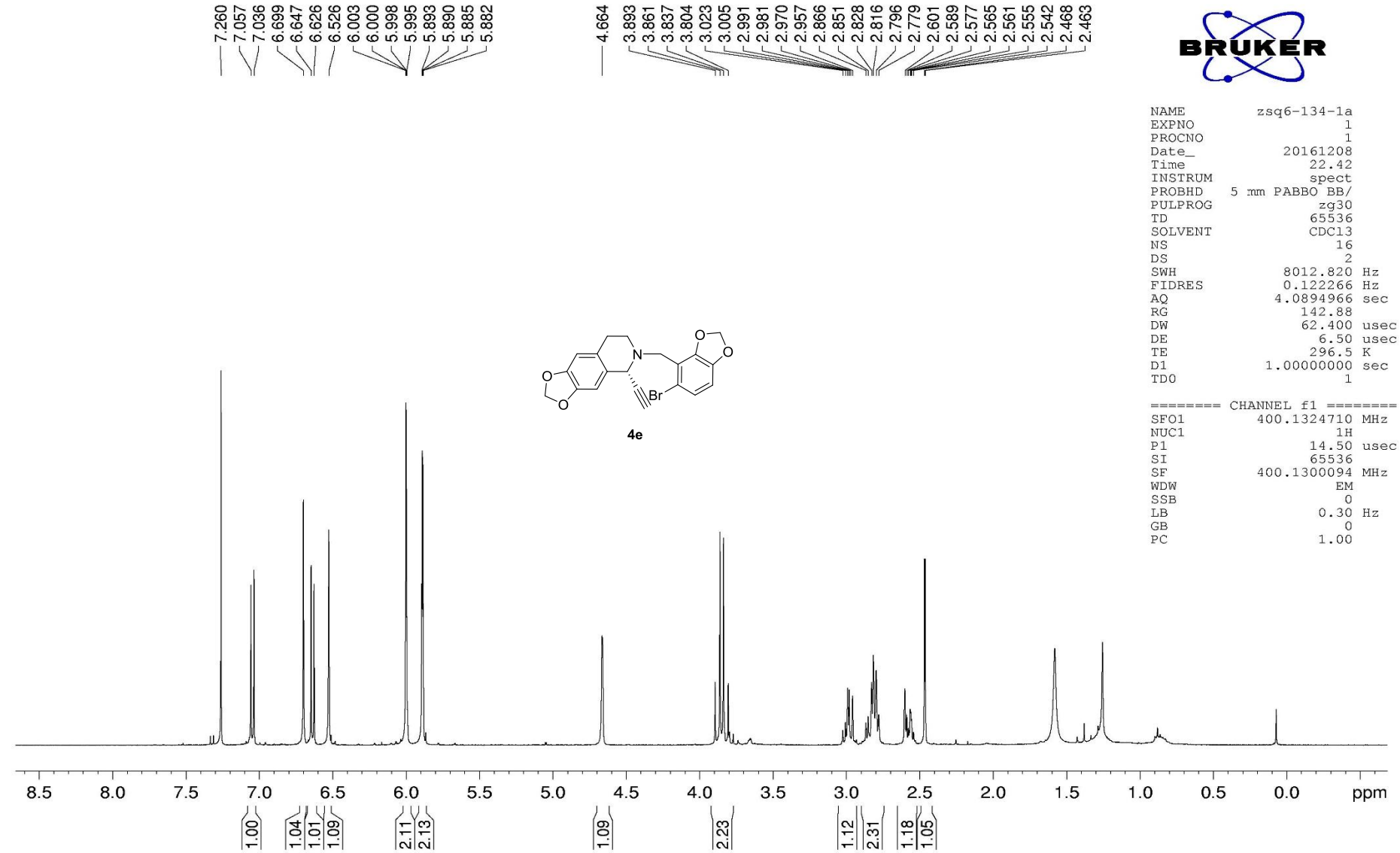

152.30
149.29
148.05
147.26
132.76
127.83
127.23
126.03
116.94
112.46
111.34
110.07
82.58
77.31
77.20
77.00
76.68
73.73
61.51
55.91
55.83
55.79
54.03
53.12
44.75
28.67
BRUKER
NAME zsq6-120-1a
EXPNO 2
PROCNO 1
Date_ 20161210
Time 11.05
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 262
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 use
DE 6.50 use
TE 297.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 =======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 use
SI 32768
SF 100.6127736 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OMe
OMe
MeO
MeO
N
Br
4b
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

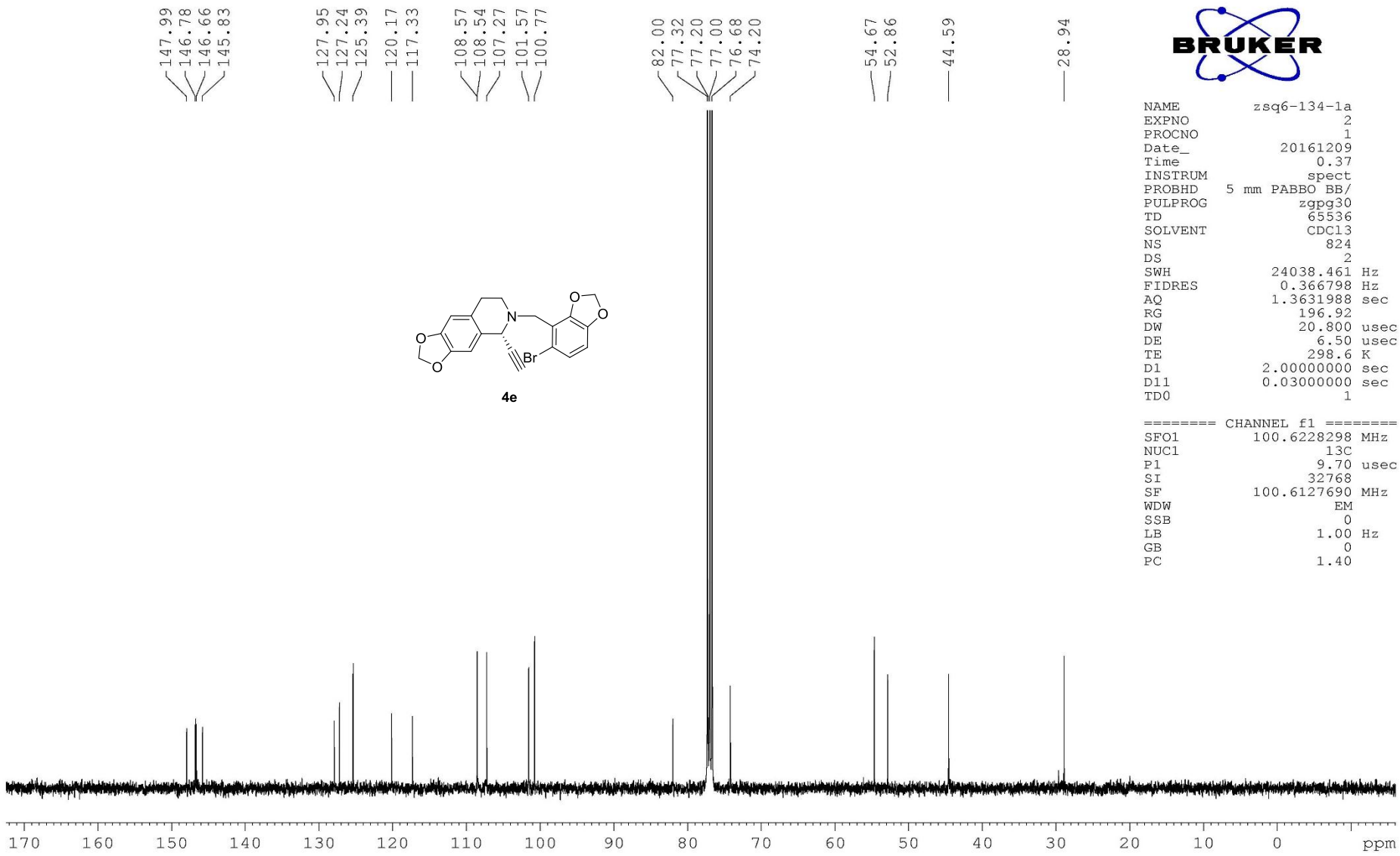

7.260
7.088
7.011
6.702
6.593
5.961
4.560
3.854
3.846
3.840
3.836
3.031
3.022
3.004
2.995
2.977
2.971
2.957
2.931
2.918
2.906
2.892
2.797
2.785
2.777
2.769
2.764
2.755
2.679
2.669
2.640
2.630
2.472
2.467
BRUKER
NAME zsq6-125-1a
EXPNO 1
PROCNO 1
Date_ 20161210
Time 14.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 103.52
DW 62.400 usec
DE 6.50 usec
TE 296.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
MeO
N
O
O
Br
4c
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
0.97
1.02
1.08
2.11
1.08
3.28
5.05
2.24
1.14
1.07
0.97

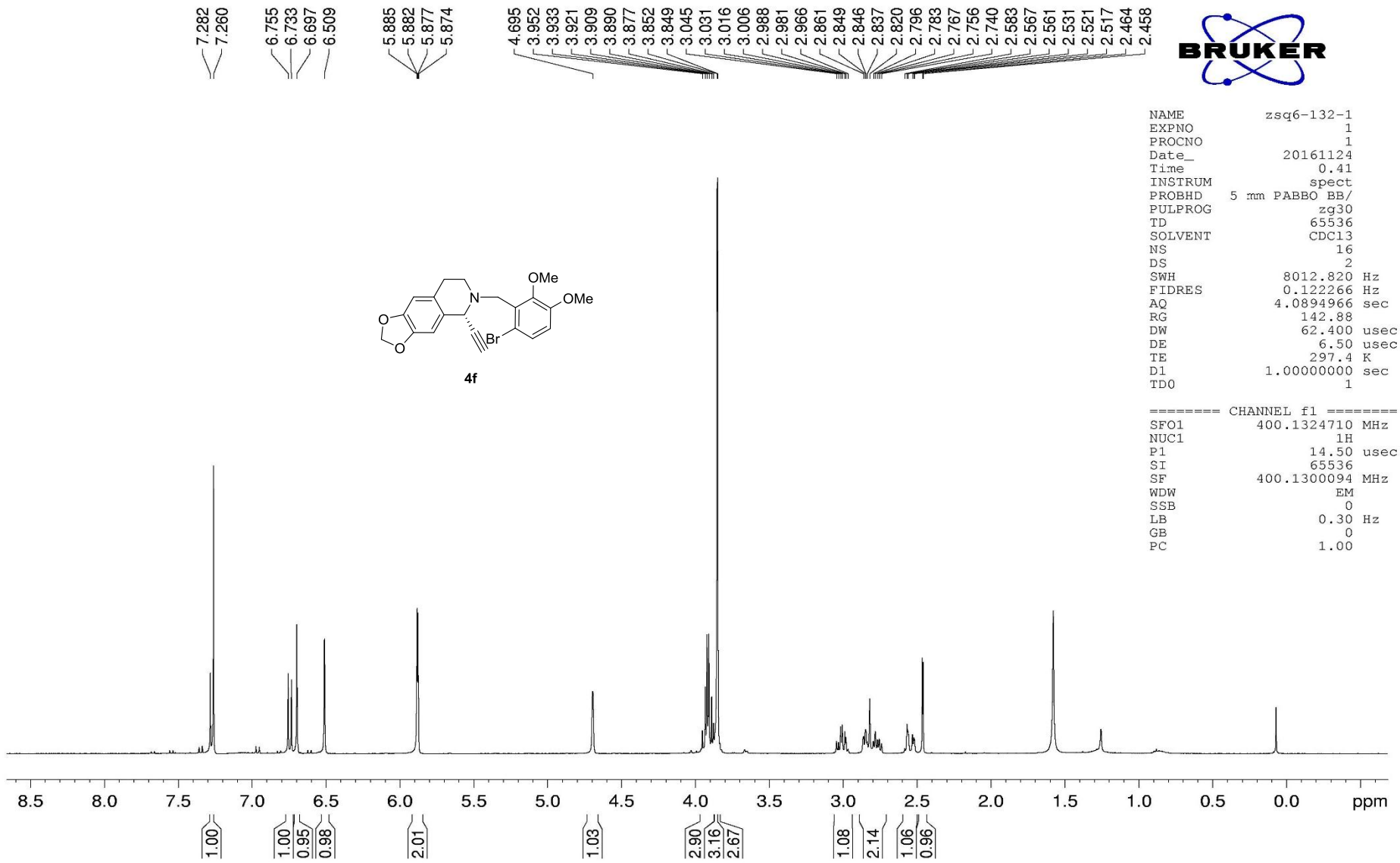

148.25
147.41
147.36
147.32
130.88
126.78
125.96
114.84
112.69
111.39
110.28
110.14
101.60
81.96
77.32
77.20
77.00
76.68
74.36
58.47
55.95
55.86
53.46
45.38
28.61
MeO
MeO
N
Br
O
O
4c
BRUKER
NAME zsq6-125-1a
EXPNO 2
PROCNO 1
Date_ 20161210
Time 14.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 229
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

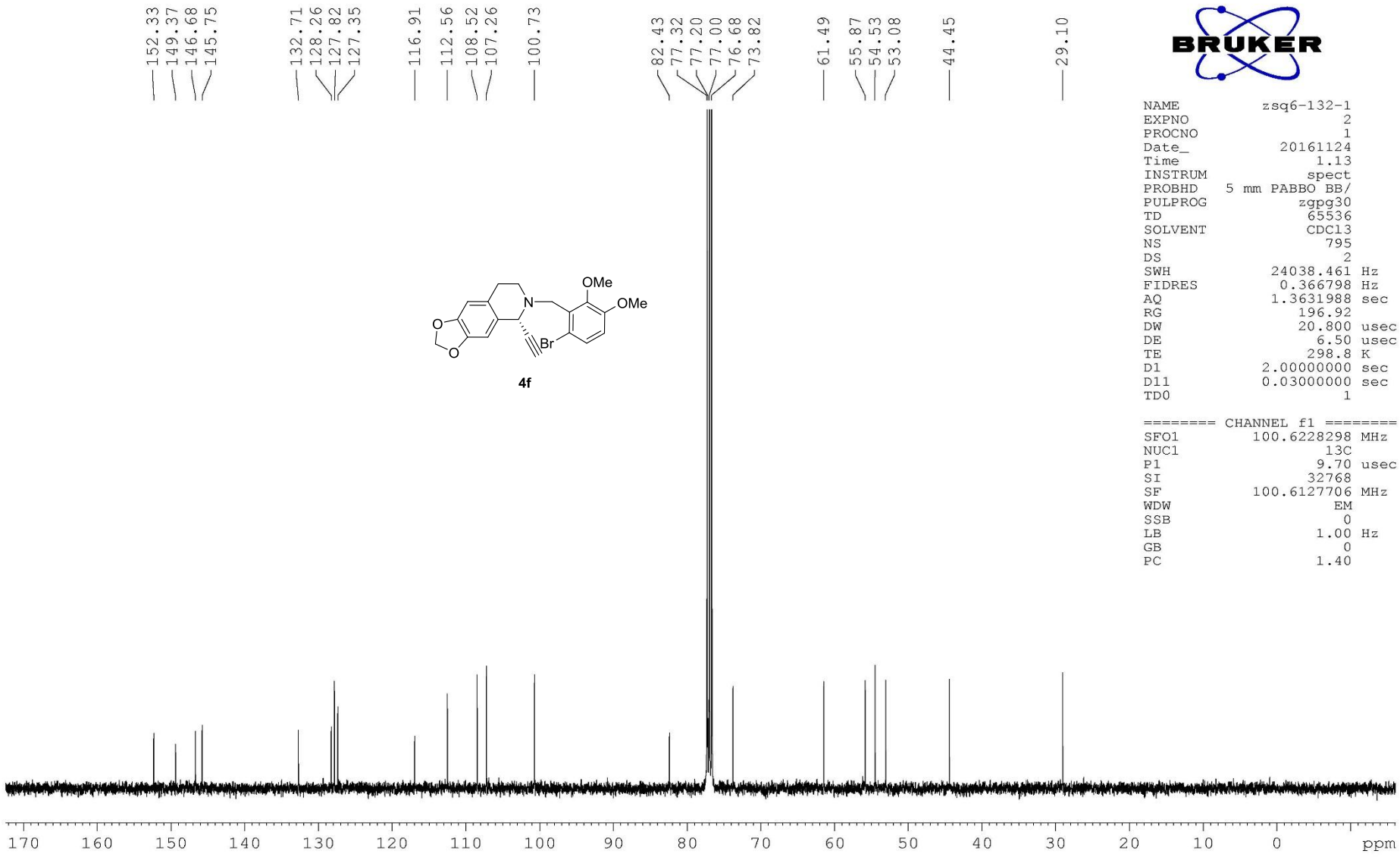

7.260
7.126
7.021
6.713
6.595
4.577
4.573
3.935
3.900
3.865
3.855
3.845
3.824
3.789
3.008
2.999
2.982
2.973
2.954
2.947
2.938
2.926
2.901
2.887
2.876
2.861
2.784
2.775
2.764
2.757
2.750
2.740
2.677
2.669
2.646
2.639
2.630
2.485
2.479
BRUKER
NAME zsq6-122-1a
EXPNO 1
PROCNO 1
Date_ 20161210
Time 11.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 82.92
DW 62.400 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
MeO
N
Br
OMe
OMe
4d
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
0.99
1.04
1.04
1.05
1.19
3.00
3.26
5.84
1.28
2.11
1.08
1.03
0.98

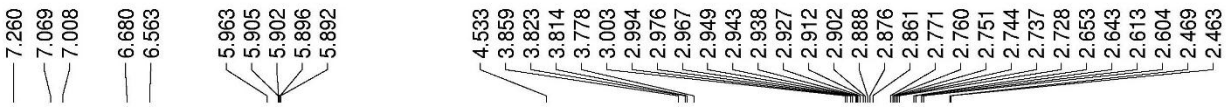

148.47
148.32
148.21
147.40
129.59
126.81
125.98
115.40
114.48
113.12
111.36
110.11
82.03
77.31
77.20
77.00
76.68
74.29
58.21
56.11
56.05
55.94
55.82
53.68
45.12
28.59
BRUKER
NAME zsq6-122-1a
EXPNO 2
PROCNO 1
Date_ 20161210
Time 11.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 210
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
MeO
MeO
N
Br
OMe
OMe
4d
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

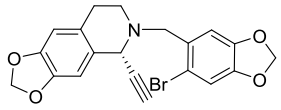

7.260
7.057
7.036
6.699
6.647
6.626
6.526
6.003
6.000
5.998
5.995
5.893
5.890
5.885
5.882
4.664
3.893
3.861
3.837
3.804
3.023
3.005
2.991
2.981
2.970
2.957
2.866
2.851
2.828
2.816
2.796
2.779
2.601
2.589
2.577
2.565
2.561
2.555
2.542
2.468
2.463
BRUKER
NAME zsq6-134-1a
EXPNO 1
PROCNO 1
Date_ 20161208
Time 22.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 142.88
DW 62.400 usec
DE 6.50 usec
TE 296.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
O
O
O
O
Br
4e
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.04
1.01
1.09
2.11
2.13
1.09
2.23
1.12
2.31
1.18
1.05


147.99
146.78
146.66
145.83
127.95
127.24
125.39
120.17
117.33
108.57
108.54
107.27
101.57
100.77
82.00
77.32
77.20
77.00
76.68
74.20
54.67
52.86
44.59
28.94
BRUKER
NAME zsq6-134-1a
EXPNO 2
PROCNO 1
Date_ 20161209
Time 0.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 824
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
O
O
O
N
Br
4e
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.282
7.260
6.755
6.733
6.697
6.509
5.885
5.882
5.877
5.874
4.695
3.952
3.933
3.921
3.909
3.890
3.877
3.852
3.849
3.045
3.031
3.016
3.006
2.988
2.981
2.966
2.861
2.849
2.846
2.837
2.820
2.796
2.783
2.767
2.756
2.740
2.583
2.567
2.561
2.531
2.521
2.517
2.464
2.458
BRUKER
OMe
OMe
N
O
O
Br
4f
NAME zsq6-132-1
EXPNO 1
PROCNO 1
Date_ 20161124
Time 0.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 142.88
DW 62.400 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.00
0.95
0.98
2.01
1.03
2.90
3.16
2.67
1.08
2.14
1.06
0.96


152.33
149.37
146.68
145.75
132.71
128.26
127.82
127.35
116.91
112.56
108.52
107.26
100.73
82.43
77.32
77.20
77.00
76.68
73.82
61.49
55.87
54.53
53.08
44.45
29.10
OMe
OMe
N
O
O
Br
4f
BRUKER
NAME zsq6-132-1
EXPNO 2
PROCNO 1
Date_ 20161124
Time 1.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 795
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127706 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

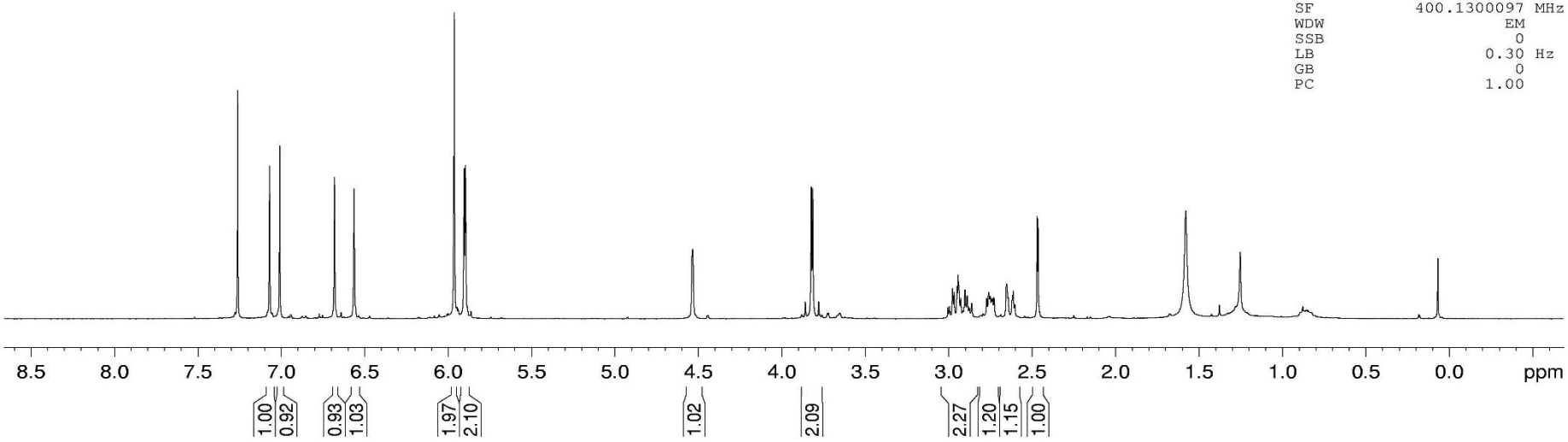


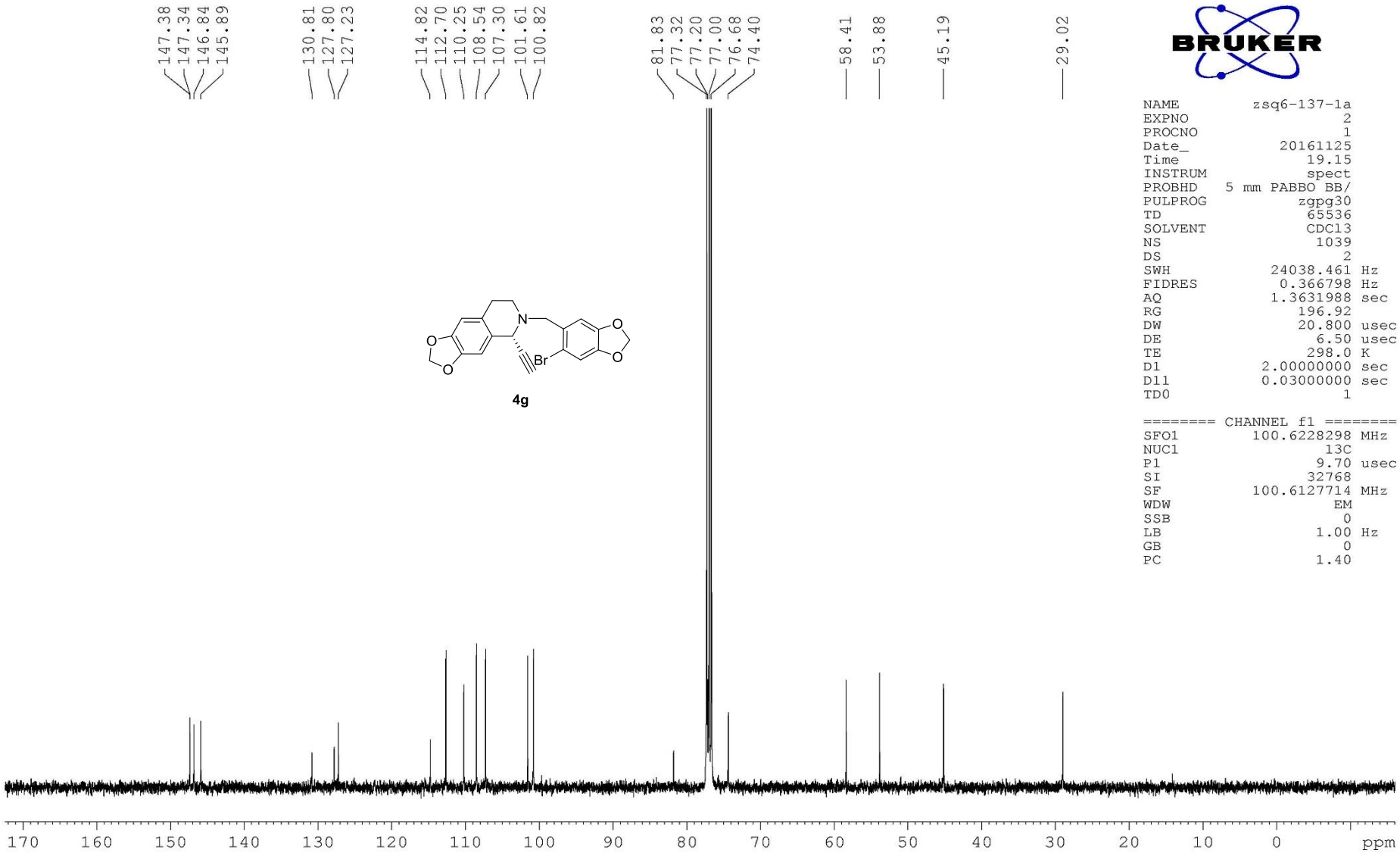


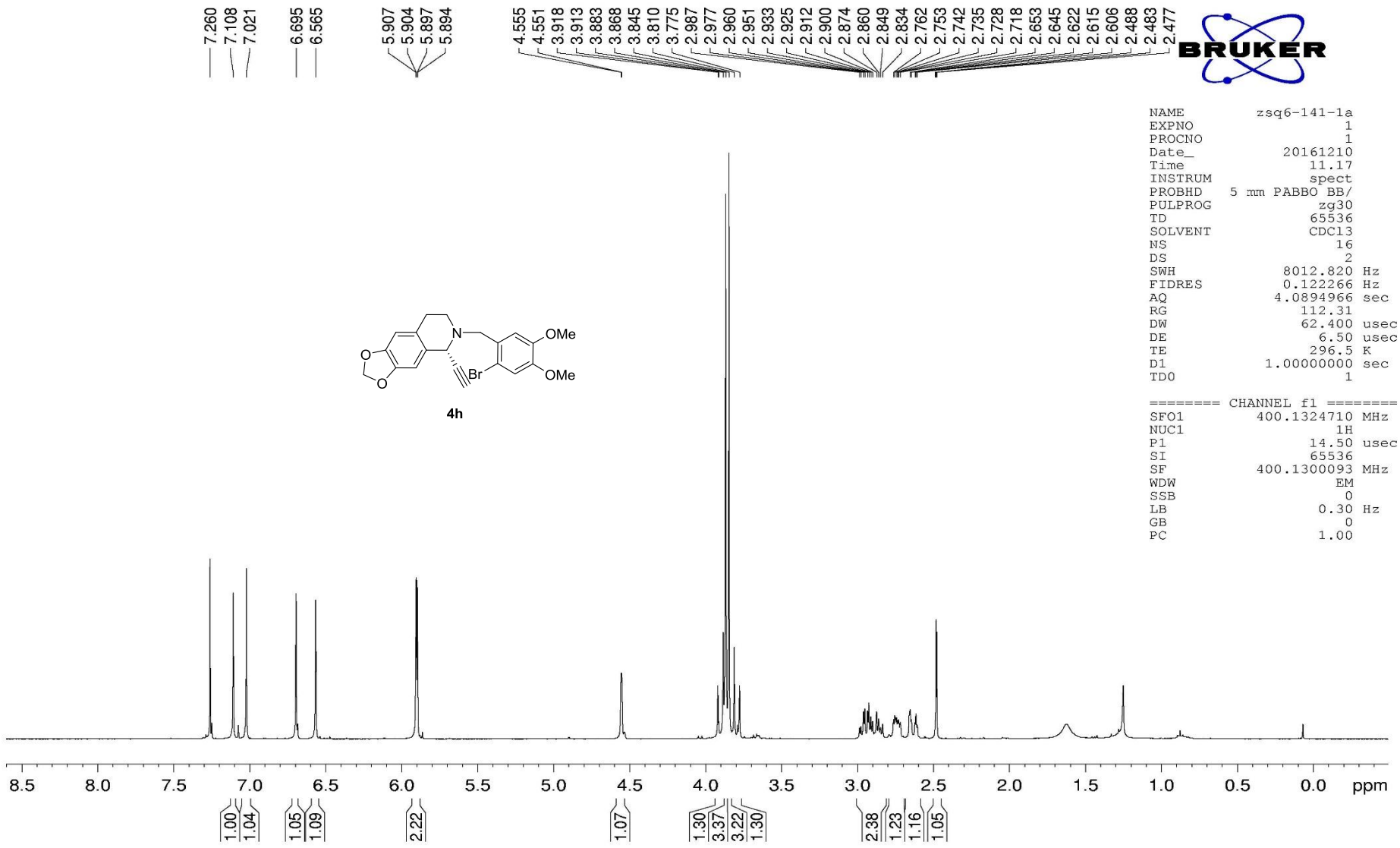
4g

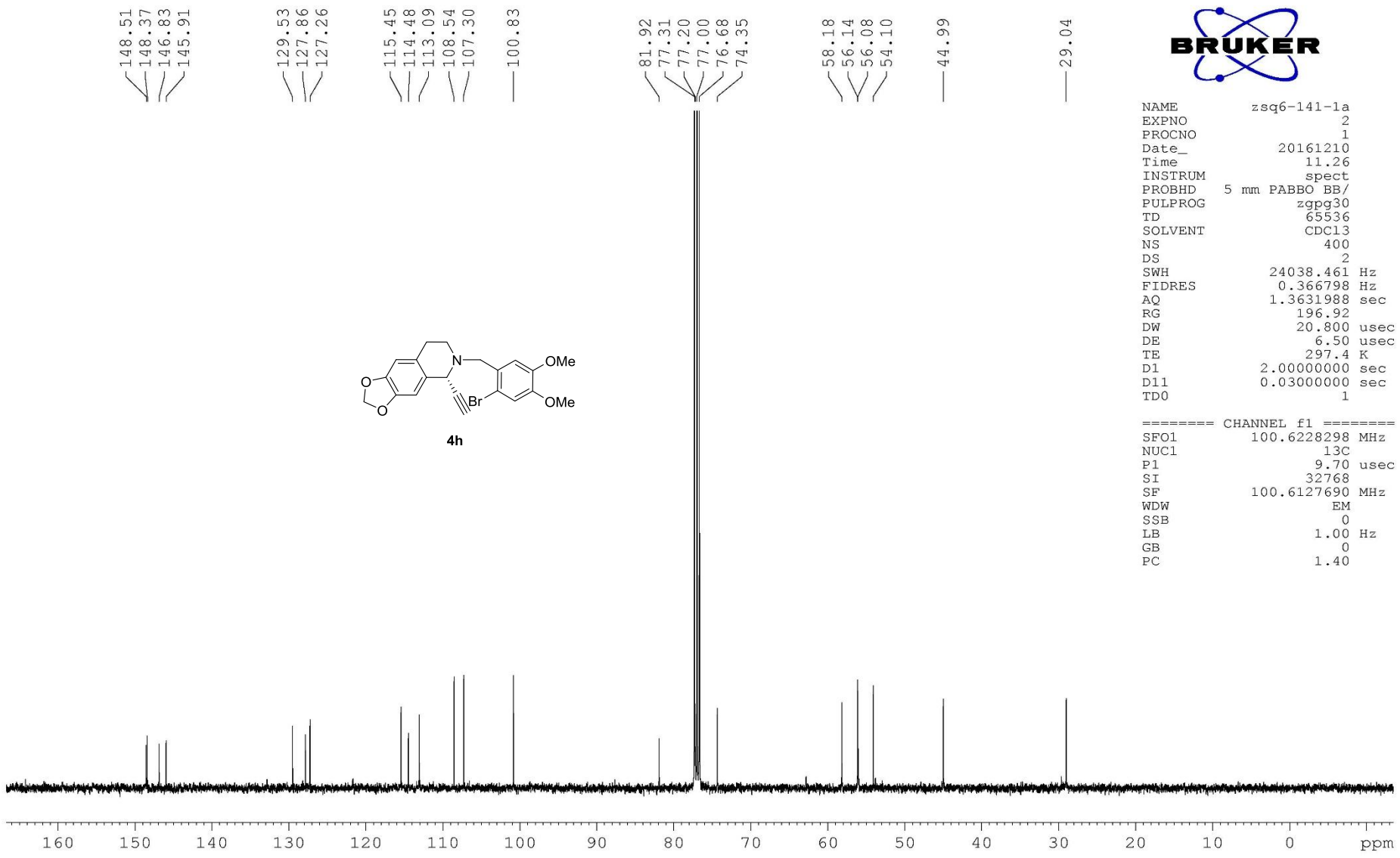
```
NAME          zsq6-137-1a
EXPNO                   1
PROCNO                  1
Date_            20161125
Time                19.09
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894966 sec
RG                  142.88
DW                  62.400 usec
DE                    6.50 usec
TE                   297.0 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                    1H
P1                   14.50 usec
SI                  65536
SF            400.1300097 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

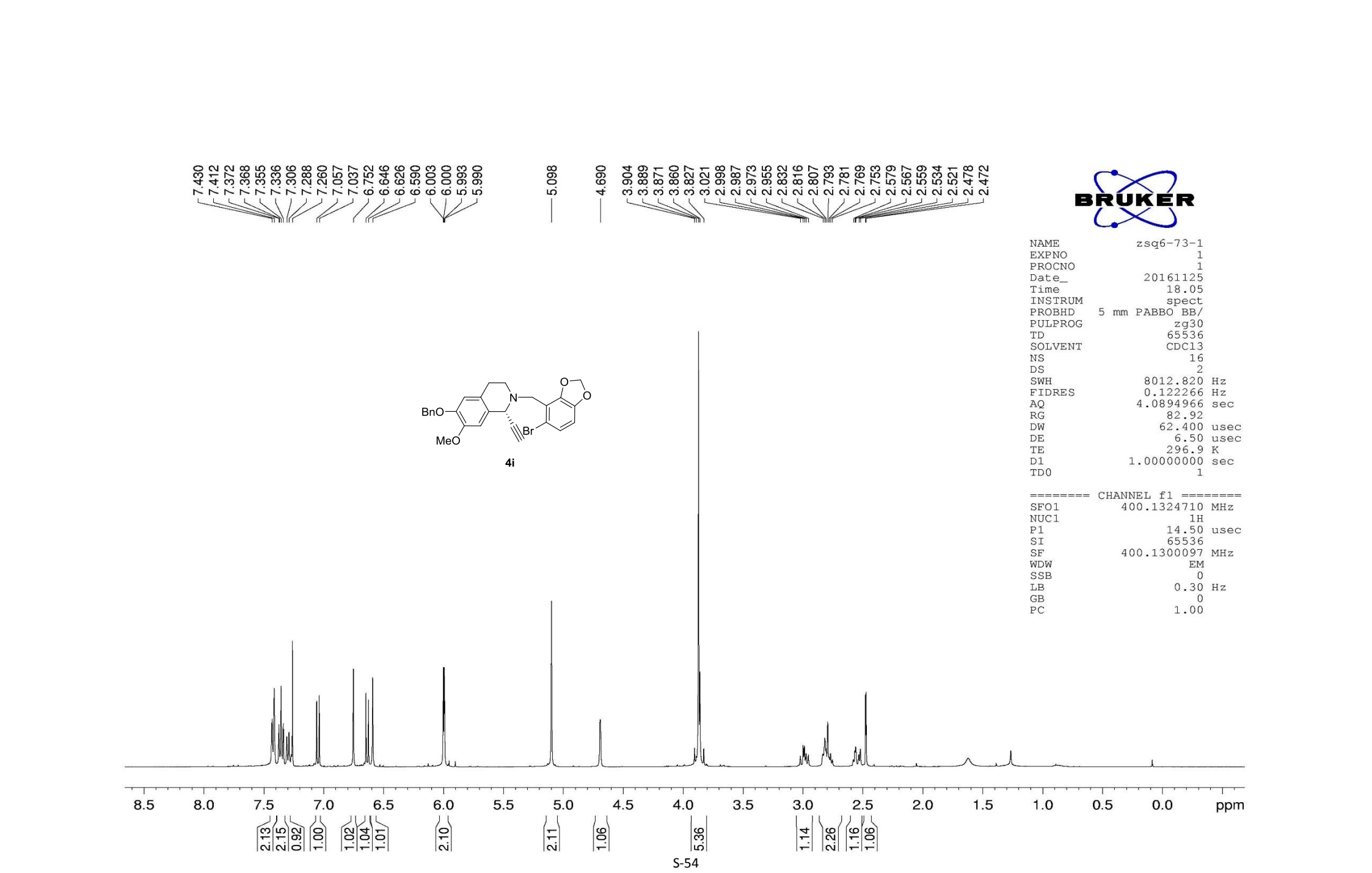


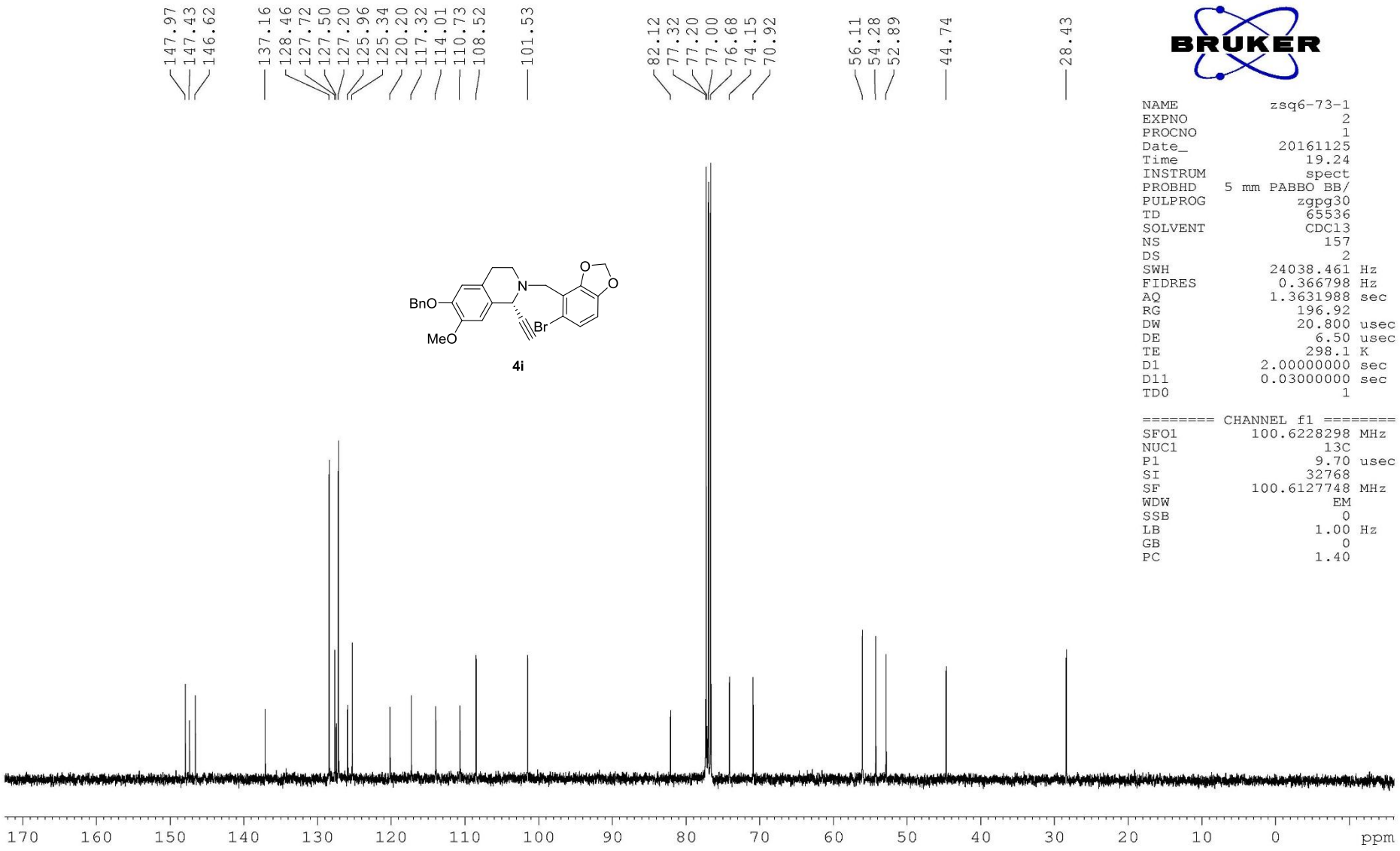

147.38
147.34
146.84
145.89
130.81
127.80
127.23
114.82
112.70
110.25
108.54
107.30
101.61
100.82
81.83
77.32
77.20
77.00
76.68
74.40
58.41
53.88
45.19
29.02
BRUKER
NAME zsq6-137-1a
EXPNO 2
PROCNO 1
Date_ 20161125
Time 19.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1039
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127714 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
O
O
O
O
Br
4g
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

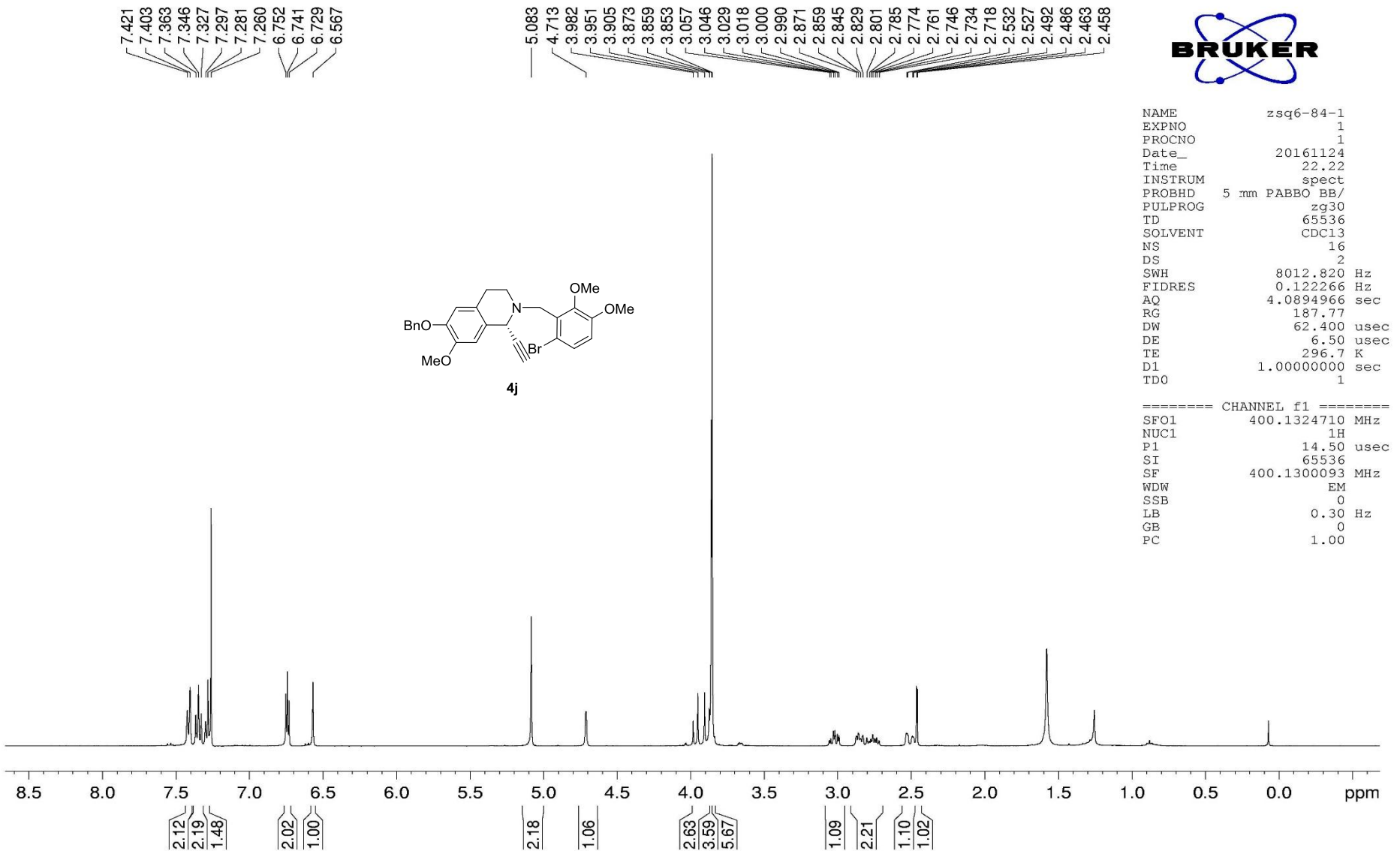

7.260
7.108
7.021
6.695
6.565
5.907
5.904
5.897
5.894
4.555
4.551
3.918
3.913
3.883
3.868
3.845
3.810
3.775
2.987
2.977
2.960
2.951
2.933
2.925
2.912
2.900
2.874
2.860
2.849
2.834
2.762
2.753
2.742
2.735
2.728
2.718
2.653
2.645
2.622
2.615
2.606
2.488
2.483
2.477
BRUKER
NAME zsq6-141-1a
EXPNO 1
PROCNO 1
Date_ 20161210
Time 11.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 112.31
DW 62.400 usec
DE 6.50 usec
TE 296.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OMe
OMe
Br
N
O
O
4h
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.04
1.05
1.09
2.22
1.07
1.30
3.37
3.22
1.30
2.38
1.23
1.16
1.05

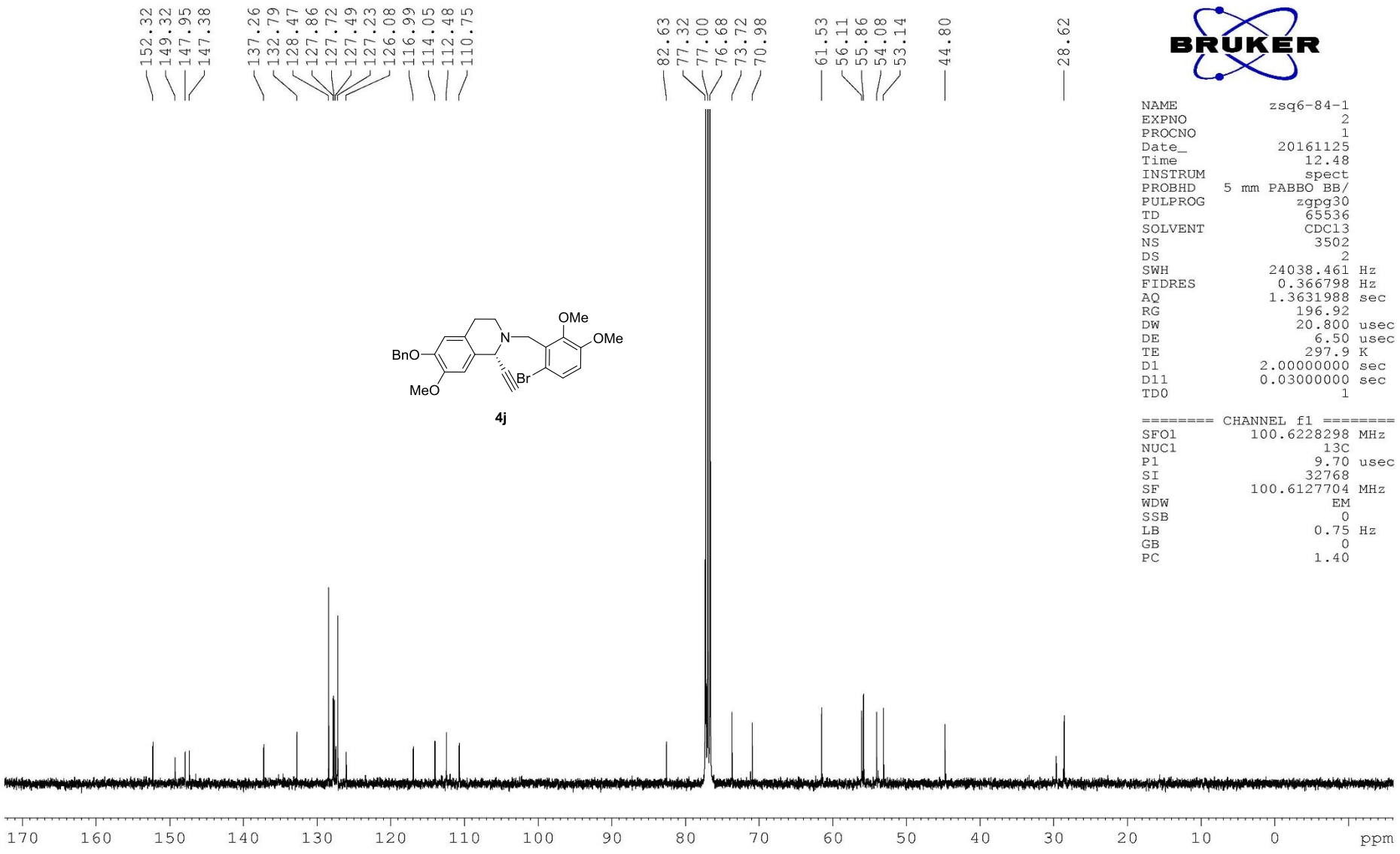

148.51
148.37
146.83
145.91
129.53
127.86
127.26
115.45
114.48
113.09
108.54
107.30
100.83
81.92
77.31
77.20
77.00
76.68
74.35
58.18
56.14
56.08
54.10
44.99
29.04
BRUKER
NAME zsq6-141-1a
EXPNO 2
PROCNO 1
Date_ 20161210
Time 11.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
O
N
Br
OMe
OMe
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

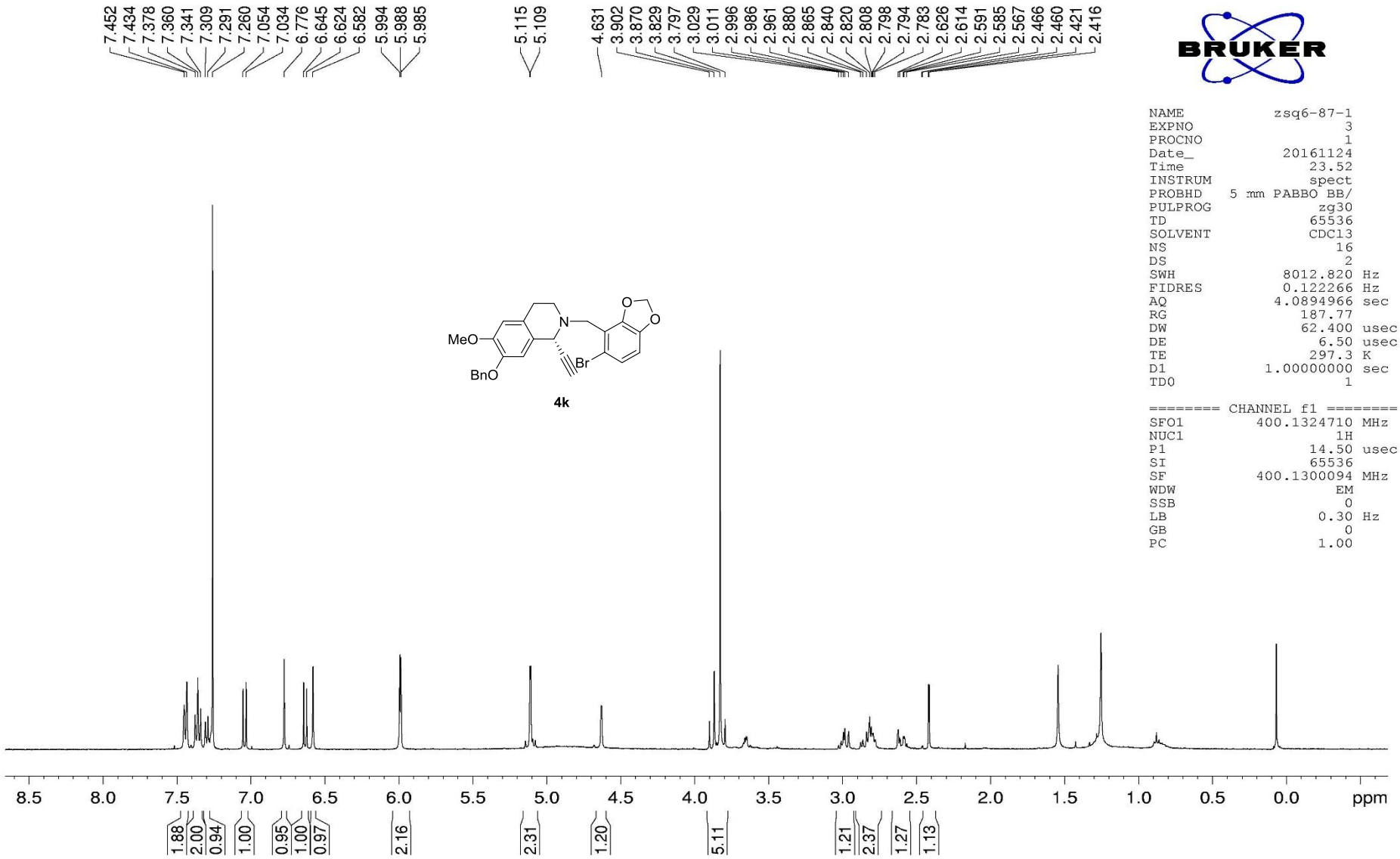
4h

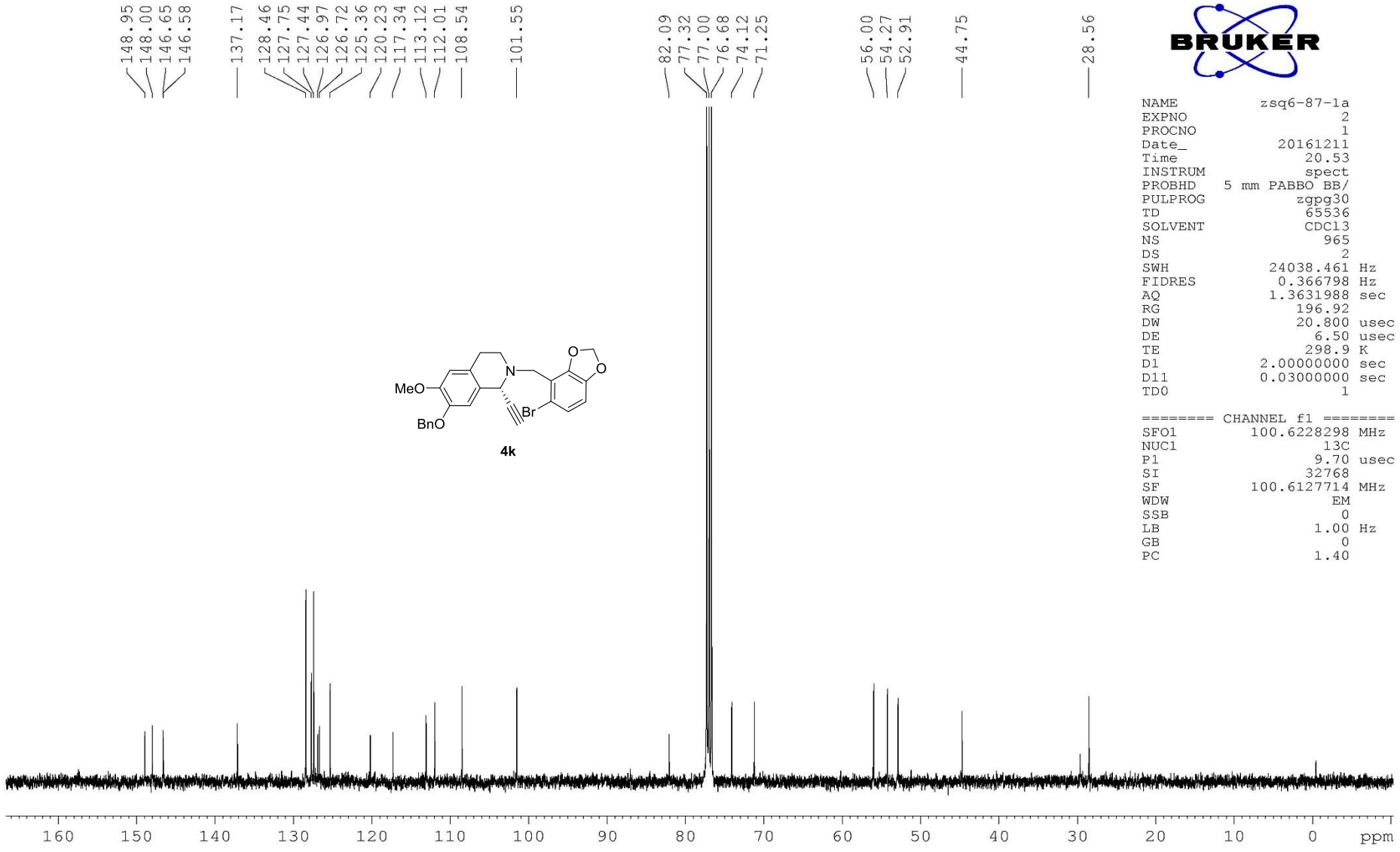

7.430
7.412
7.372
7.368
7.355
7.336
7.306
7.288
7.260
7.057
7.037
6.752
6.646
6.626
6.590
6.003
6.000
5.993
5.990
5.098
4.690
3.904
3.889
3.871
3.860
3.827
3.021
2.998
2.987
2.973
2.955
2.832
2.816
2.807
2.793
2.781
2.769
2.753
2.579
2.567
2.559
2.534
2.521
2.478
2.472
BnO
MeO
N
O
O
Br
4i
NAME zsq6-73-1
EXPNO 1
PROCNO 1
Date_ 20161125
Time 18.05
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 82.92
DW 62.400 usec
DE 6.50 usec
TE 296.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.13
2.15
0.92
1.00
1.02
1.04
1.01
2.10
2.11
1.06
5.36
1.14
2.26
1.16
1.06

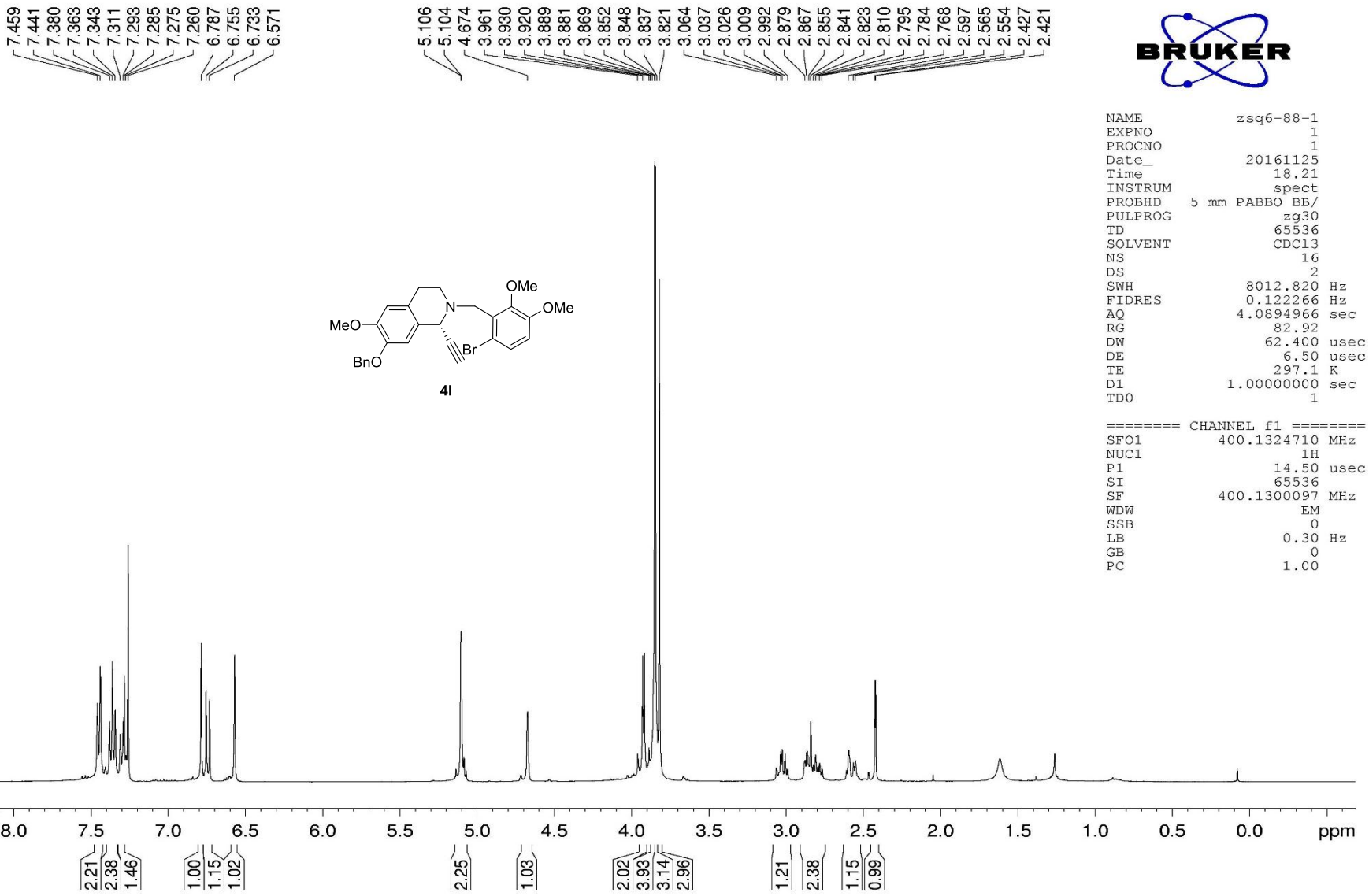

147.97
147.43
146.62
137.16
128.46
127.72
127.50
127.20
125.96
125.34
120.20
117.32
114.01
110.73
108.52
101.53
82.12
77.32
77.20
77.00
76.68
74.15
70.92
56.11
54.28
52.89
44.74
28.43
BnO
MeO
N
O
O
Br
4i
BRUKER
NAME zsq6-73-1
EXPNO 2
PROCNO 1
Date_ 20161125
Time 19.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 157
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127748 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

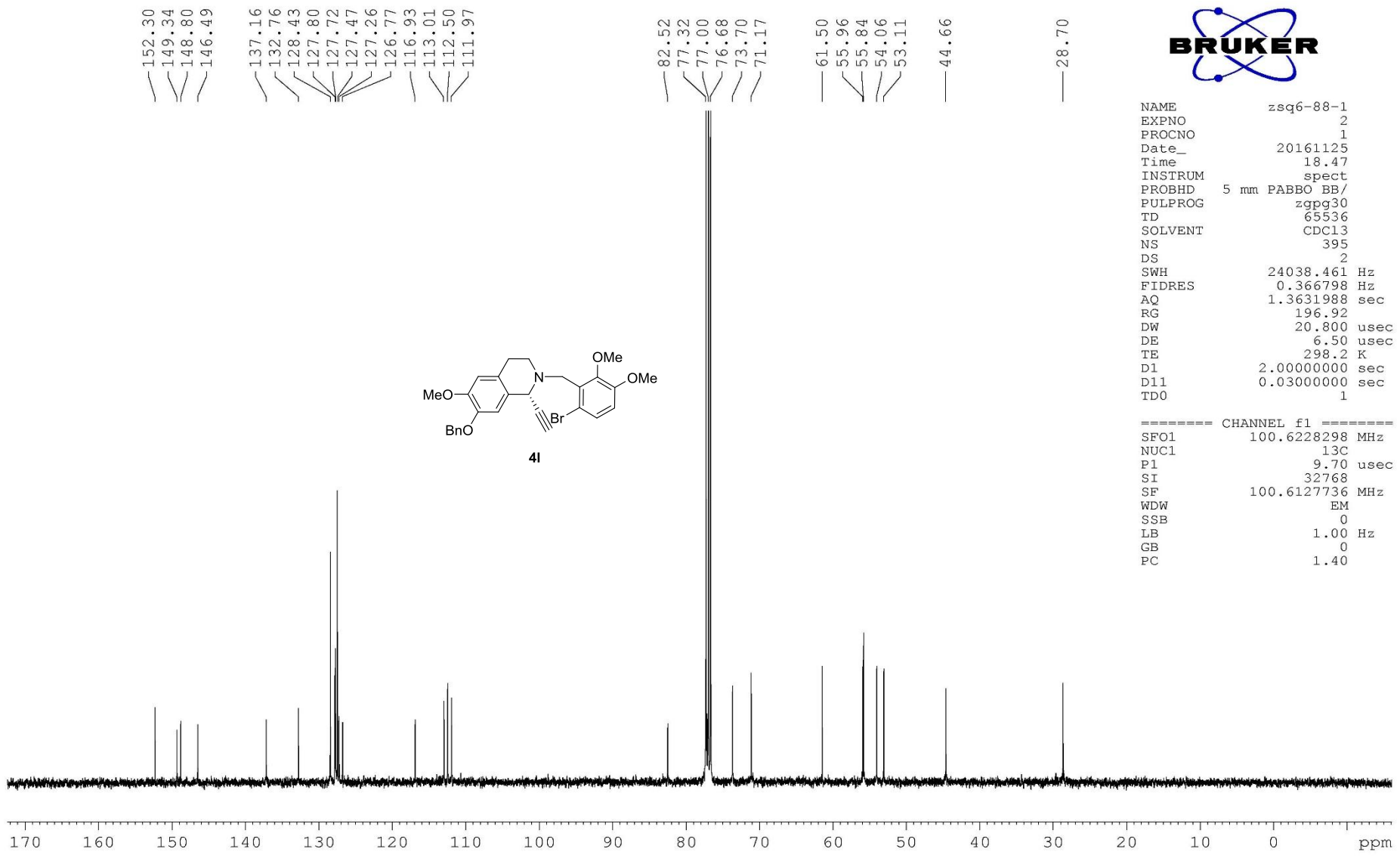

7.421
7.403
7.363
7.346
7.327
7.297
7.281
7.260
6.752
6.741
6.729
6.567
5.083
4.713
3.982
3.951
3.905
3.873
3.859
3.853
3.057
3.046
3.029
3.018
3.000
2.990
2.871
2.859
2.845
2.829
2.801
2.785
2.774
2.761
2.746
2.734
2.718
2.532
2.527
2.492
2.486
2.463
2.458
BRUKER
NAME zsq6-84-1
EXPNO 1
PROCNO 1
Date_ 20161124
Time 22.22
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 187.77
DW 62.400 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OMe
OMe
BnO
N
Br
MeO
4j
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.12
2.19
1.48
2.02
1.00
2.18
1.06
2.63
3.59
5.67
1.09
2.21
1.10
1.02

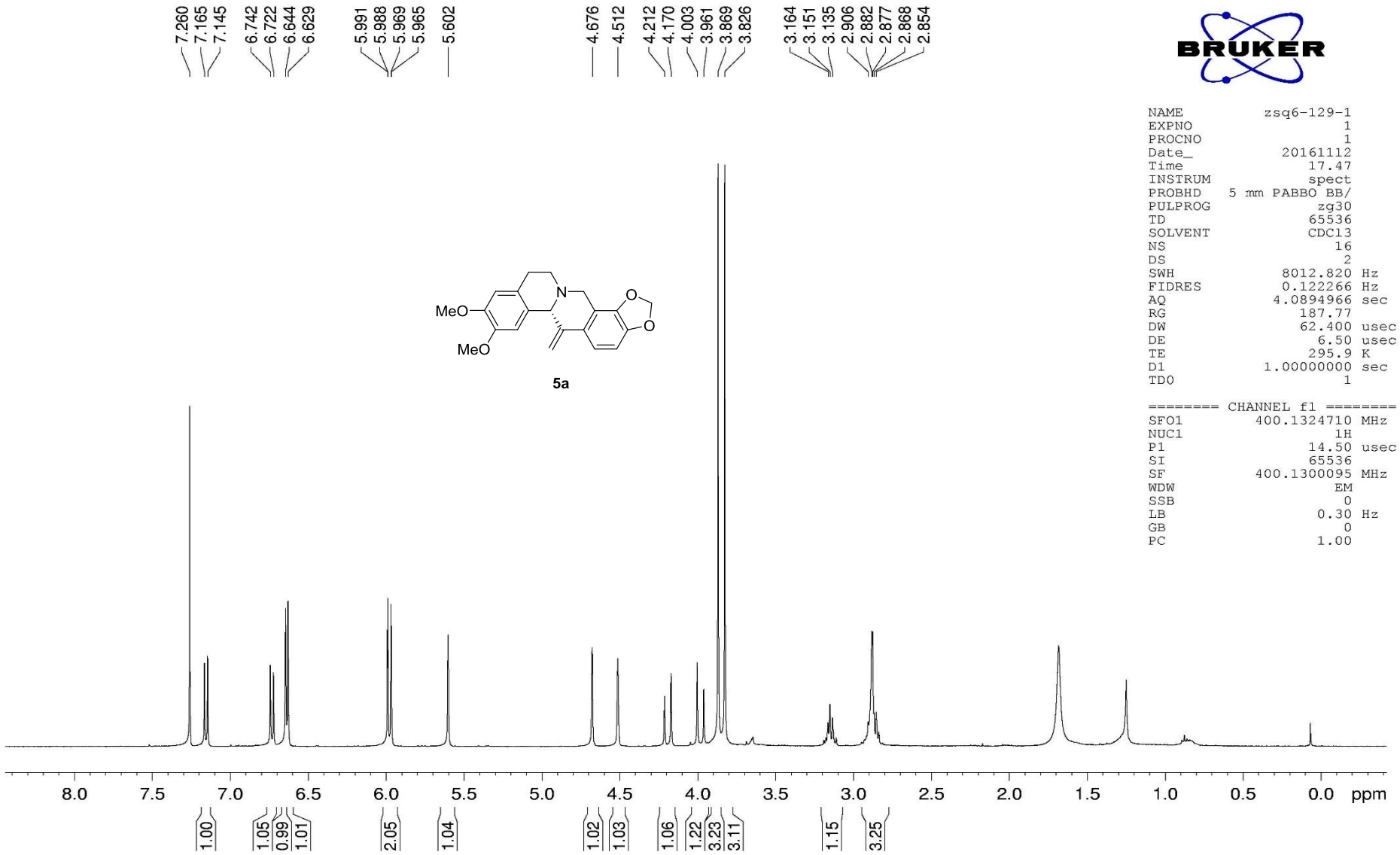

152.32
149.32
147.95
147.38
137.26
132.79
128.47
127.86
127.72
127.49
127.23
126.08
116.99
114.05
112.48
110.75
82.63
77.32
77.00
76.68
73.72
70.98
61.53
56.11
55.86
54.08
53.14
44.80
28.62
BRUKER
NAME zsq6-84-1
EXPNO 2
PROCNO 1
Date_ 20161125
Time 12.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3502
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127704 MHz
WDW EM
SSB 0
LB 0.75 Hz
GB 0
PC 1.40
OMe
OMe
BnO
MeO
Br
N
4j
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

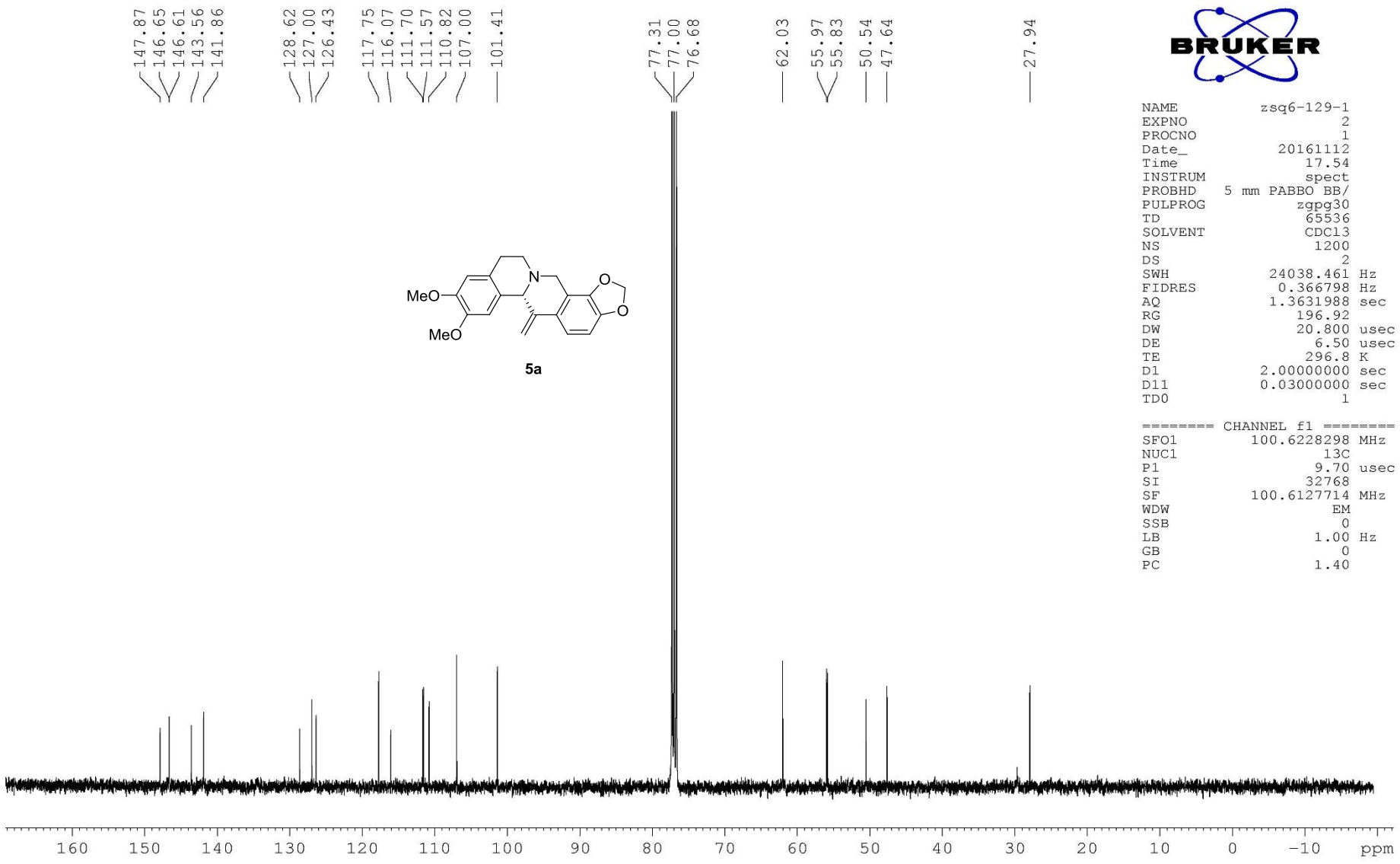

7.452
7.434
7.378
7.360
7.341
7.309
7.291
7.260
7.054
7.034
6.776
6.645
6.624
6.582
5.994
5.988
5.985
5.115
5.109
4.631
3.902
3.870
3.829
3.797
3.029
3.011
2.996
2.986
2.961
2.880
2.865
2.840
2.820
2.808
2.798
2.794
2.783
2.626
2.614
2.591
2.585
2.567
2.466
2.460
2.421
2.416
BRUKER
NAME zsq6-87-1
EXPNO 3
PROCNO 1
Date_ 20161124
Time 23.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 187.77
DW 62.400 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
BnO
N
O
O
Br
4k
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.88
2.00
0.94
1.00
0.95
1.00
0.97
2.16
2.31
1.20
5.11
1.21
2.37
1.27
1.13

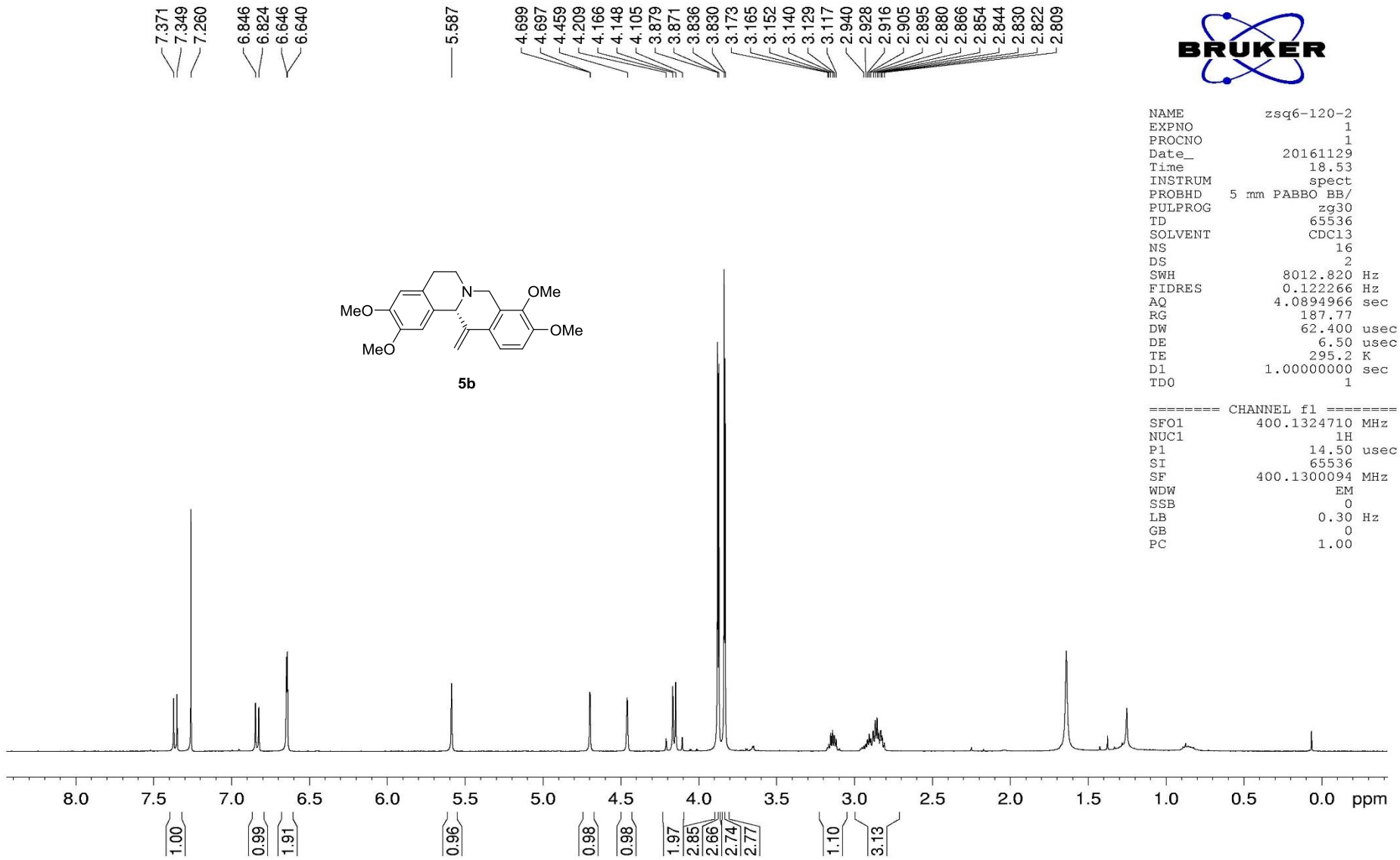

148.95
148.00
146.65
146.58
137.17
128.46
127.75
127.44
126.97
126.72
125.36
120.23
117.34
113.12
112.01
108.54
101.55
82.09
77.32
77.00
76.68
74.12
71.25
56.00
54.27
52.91
44.75
28.56
BRUKER
NAME zsq6-87-1a
EXPNO 2
PROCNO 1
Date_ 20161211
Time 20.53
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 965
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127714 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
MeO
BnO
Br
N
O
O
O
4k
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.459
7.441
7.380
7.363
7.343
7.311
7.293
7.285
7.275
7.260
6.787
6.755
6.733
6.571
5.106
5.104
4.674
3.961
3.930
3.920
3.889
3.881
3.869
3.852
3.848
3.837
3.821
3.064
3.037
3.026
3.009
2.992
2.879
2.867
2.855
2.841
2.823
2.810
2.795
2.784
2.768
2.597
2.565
2.554
2.427
2.421
BRUKER
NAME zsq6-88-1
EXPNO 1
PROCNO 1
Date_ 20161125
Time 18.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 82.92
DW 62.400 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OMe
OMe
MeO
N
Br
BnO
4l
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.21
2.38
1.46
1.00
1.15
1.02
2.25
1.03
2.02
3.93
3.14
2.96
1.21
2.38
1.15
0.99

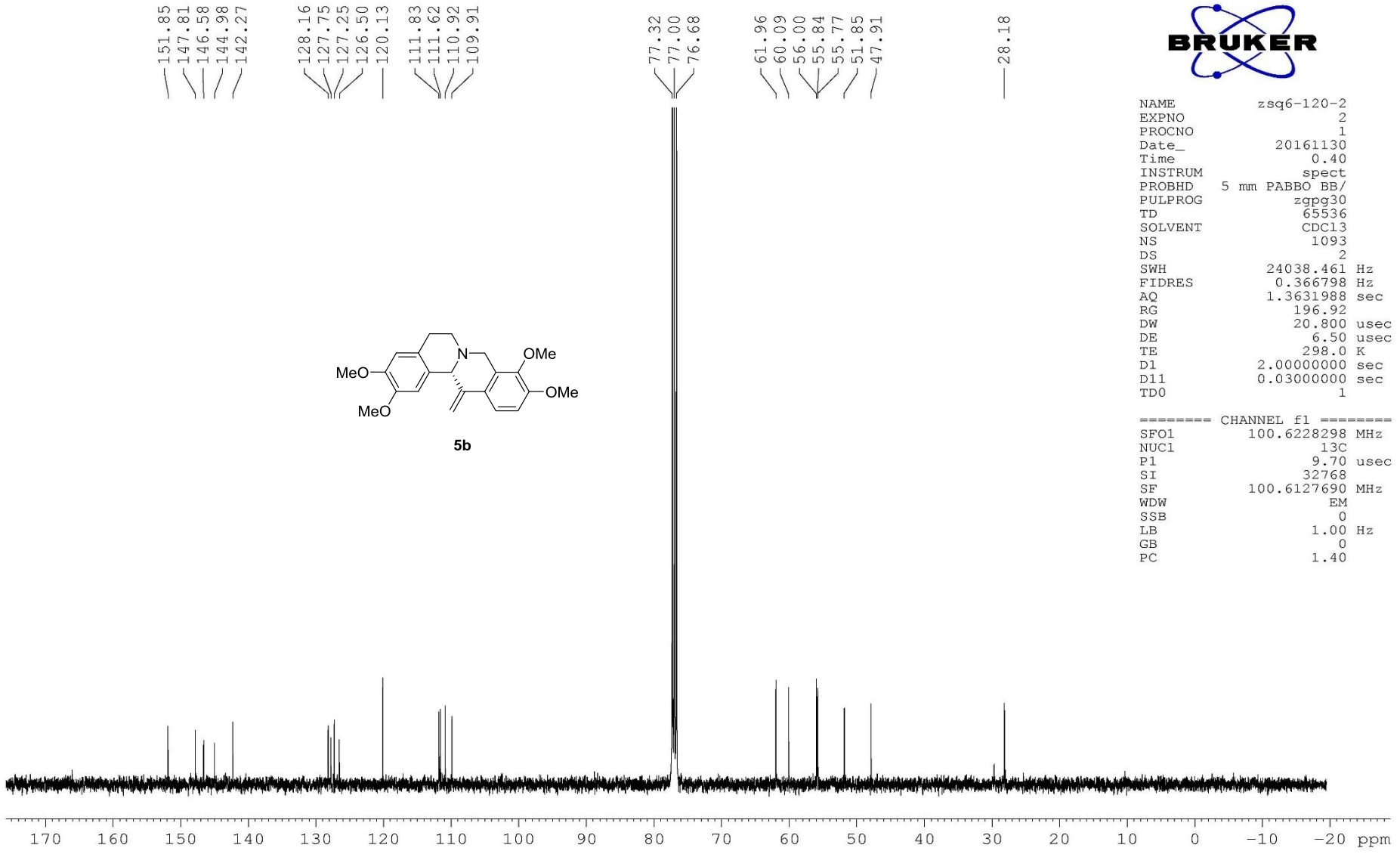

152.30
149.34
148.80
146.49
137.16
132.76
128.43
127.80
127.72
127.47
127.26
126.77
116.93
113.01
112.50
111.97
82.52
77.32
77.00
76.68
73.70
71.17
61.50
55.96
55.84
54.06
53.11
44.66
28.70
BRUKER
NAME zsq6-88-1
EXPNO 2
PROCNO 1
Date_ 20161125
Time 18.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 395
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127736 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OMe
OMe
MeO
N
Br
BnO
4l
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

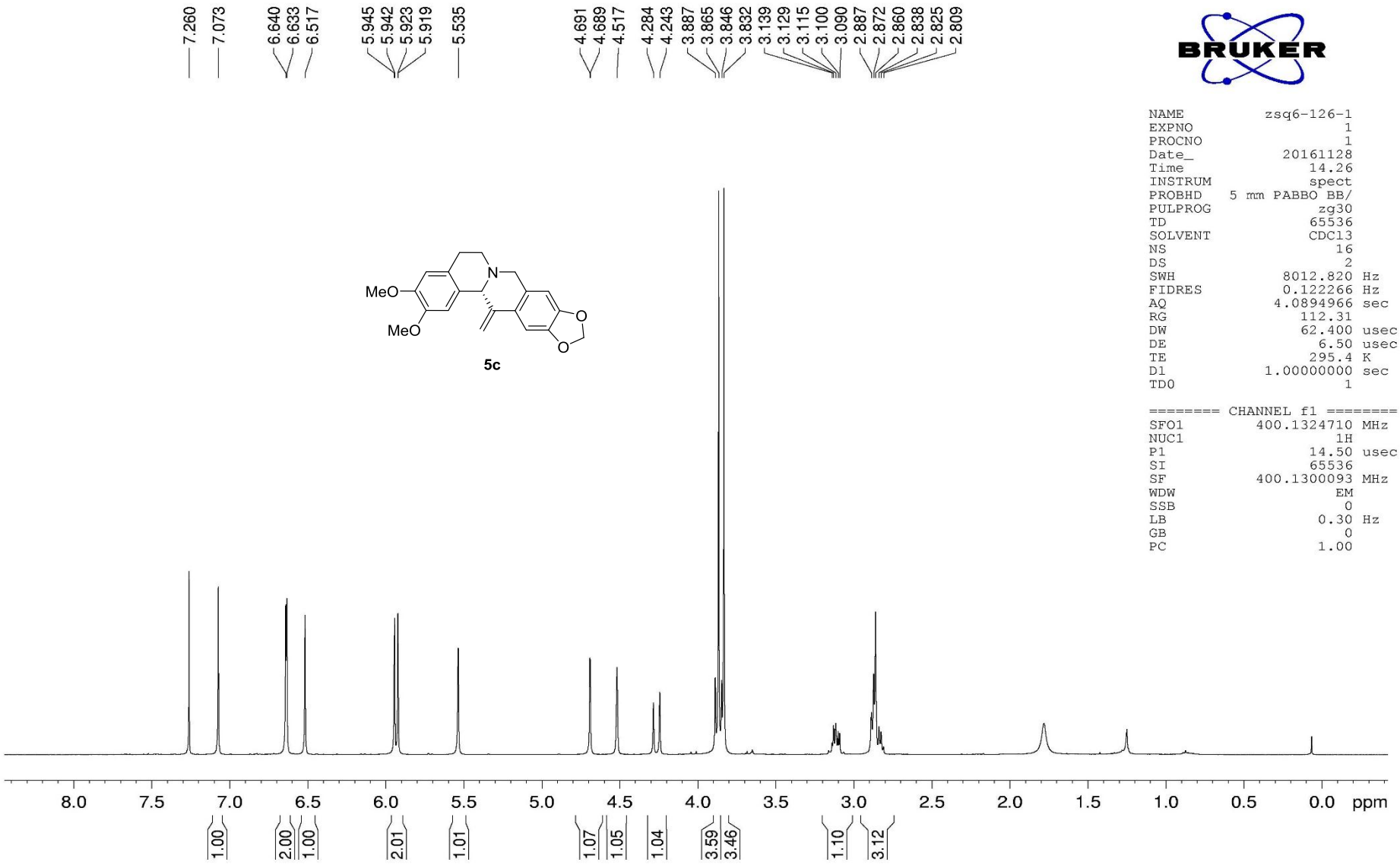

7.260
7.165
7.145
6.742
6.722
6.644
6.629
5.991
5.988
5.969
5.965
5.602
4.676
4.512
4.212
4.170
4.003
3.961
3.869
3.826
3.164
3.151
3.135
2.906
2.882
2.877
2.868
2.854
BRUKER
NAME zsq6-129-1
EXPNO 1
PROCNO 1
Date_ 20161112
Time 17.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 187.77
DW 62.400 usec
DE 6.50 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
MeO
N
O
O
5a
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.05
0.99
1.01
2.05
1.04
1.02
1.03
1.06
1.22
3.23
3.11
1.15
3.25

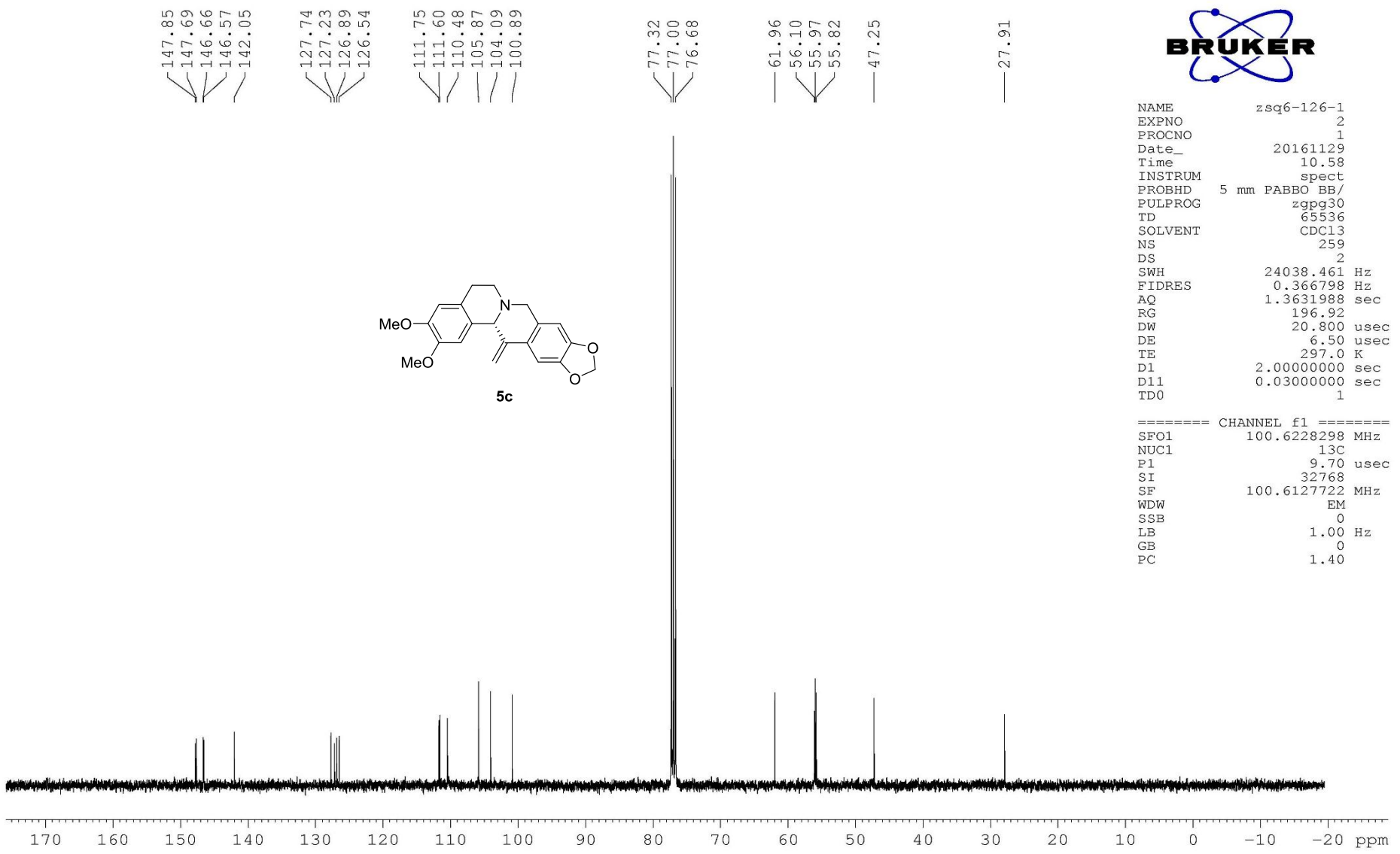

147.87
146.65
146.61
143.56
141.86
128.62
127.00
126.43
117.75
116.07
111.70
111.57
110.82
107.00
101.41
77.31
77.00
76.68
62.03
55.97
55.83
50.54
47.64
27.94
MeO
MeO
N
O
O
5a
NAME zsq6-129-1
EXPNO 2
PROCNO 1
Date_ 20161112
Time 17.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1200
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127714 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 ppm

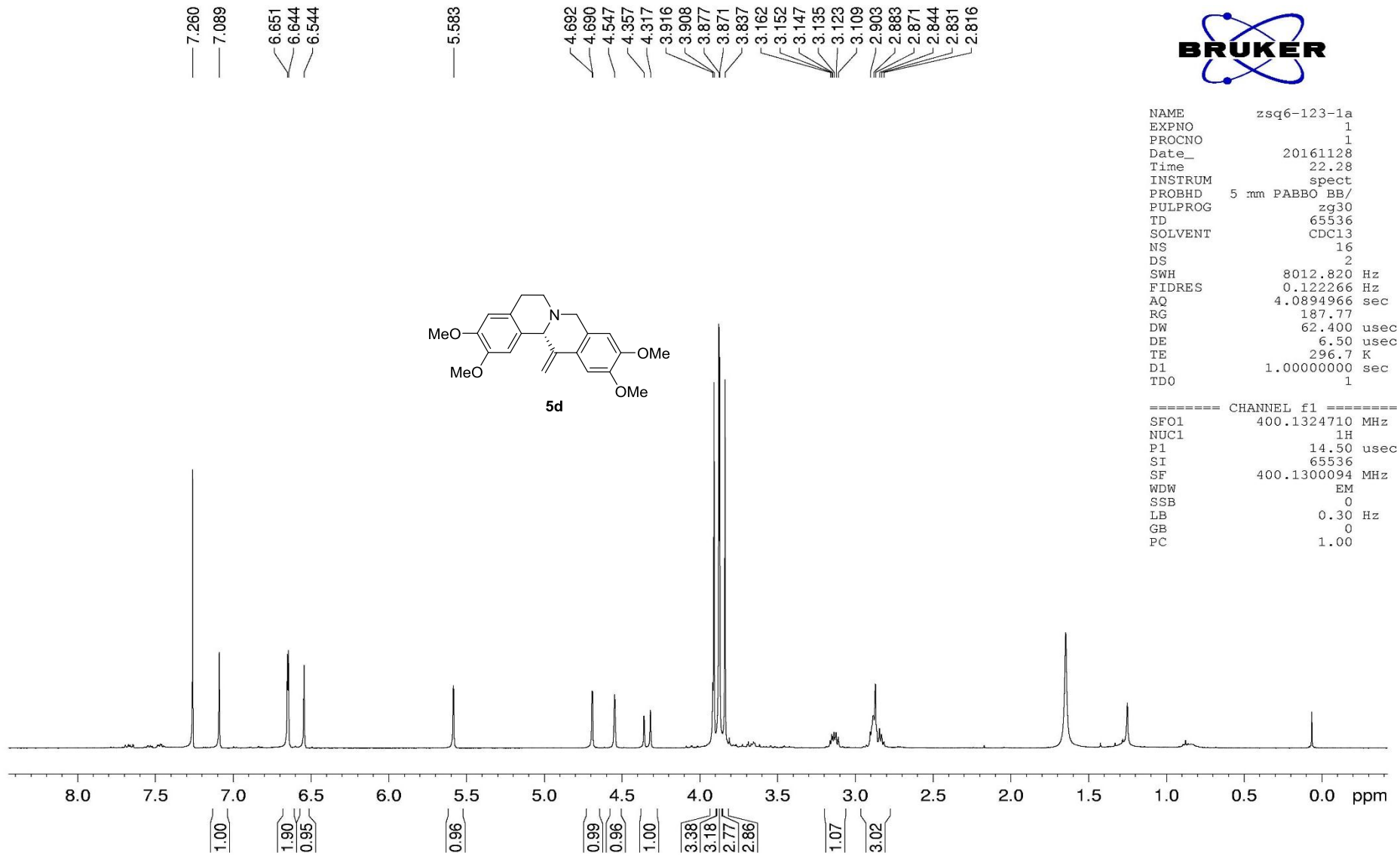

7.371
7.349
7.260
6.846
6.824
6.646
6.640
5.587
4.699
4.697
4.459
4.209
4.166
4.148
4.105
3.879
3.871
3.836
3.830
3.173
3.165
3.152
3.140
3.129
3.117
2.940
2.928
2.916
2.905
2.895
2.880
2.866
2.854
2.844
2.830
2.822
2.809
BRUKER
NAME zsq6-120-2
EXPNO 1
PROCNO 1
Date_ 20161129
Time 18.53
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 187.77
DW 62.400 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
MeO
N
OMe
OMe
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
0.99
1.91
0.96
0.98
0.98
1.97
2.85
2.66
2.74
2.77
1.10
3.13

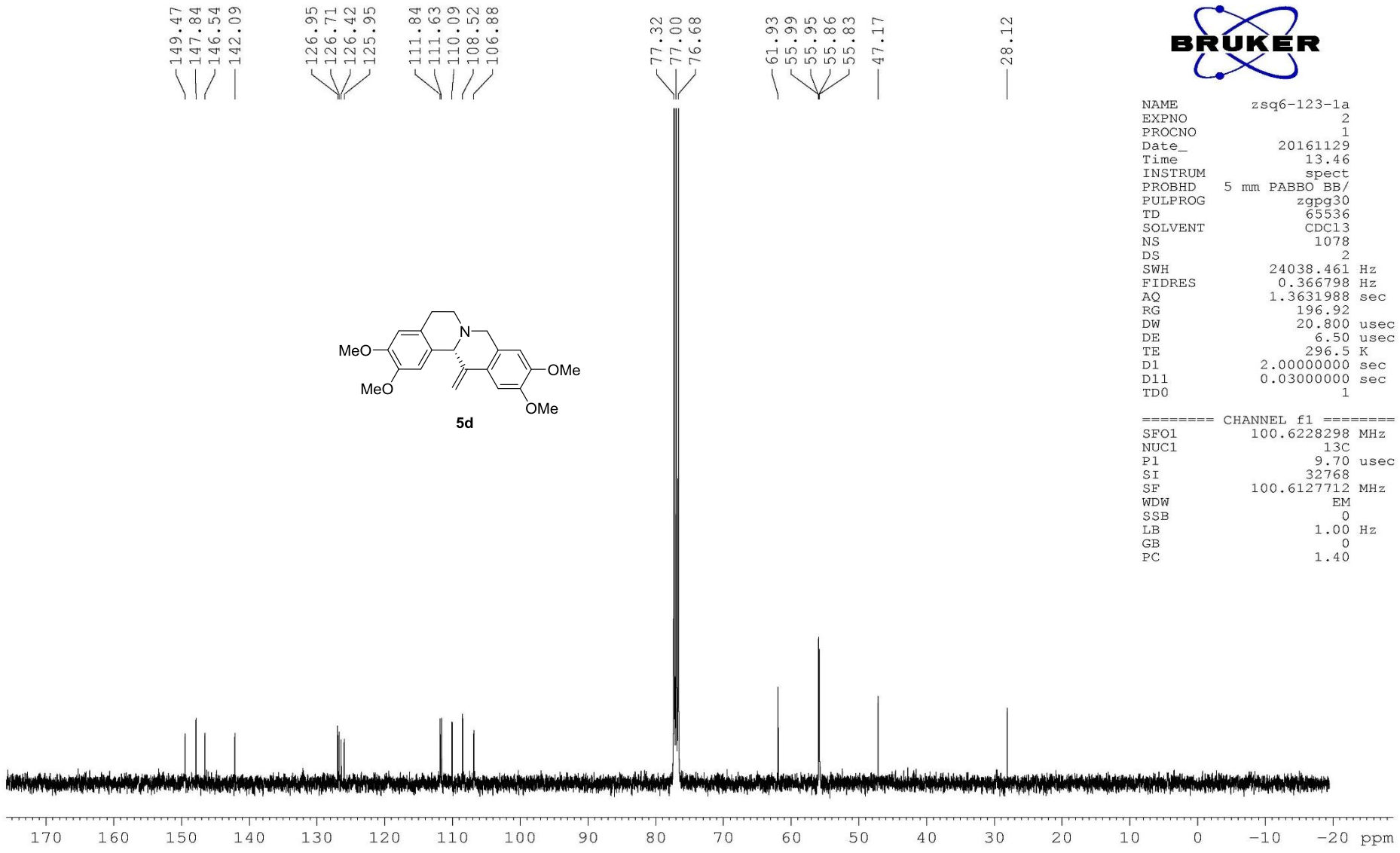
5b

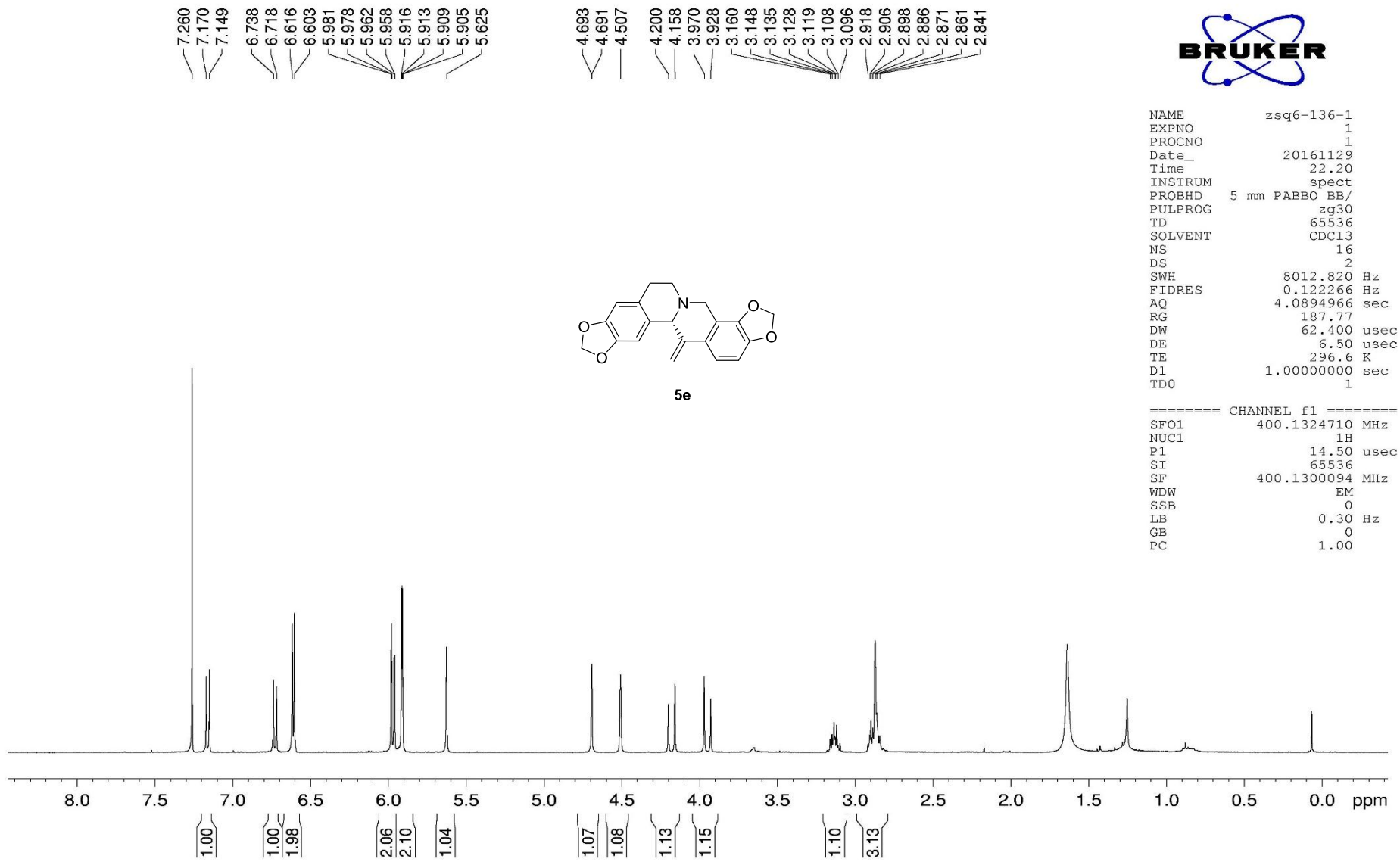

151.85
147.81
146.58
144.98
142.27
128.16
127.75
127.25
126.50
120.13
111.83
111.62
110.92
109.91
77.32
77.00
76.68
61.96
60.09
56.00
55.84
55.77
51.85
47.91
28.18
MeO
MeO
OMe
OMe
N
5b
BRUKER
NAME zsq6-120-2
EXPNO 2
PROCNO 1
Date_ 20161130
Time 0.40
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1093
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 ppm

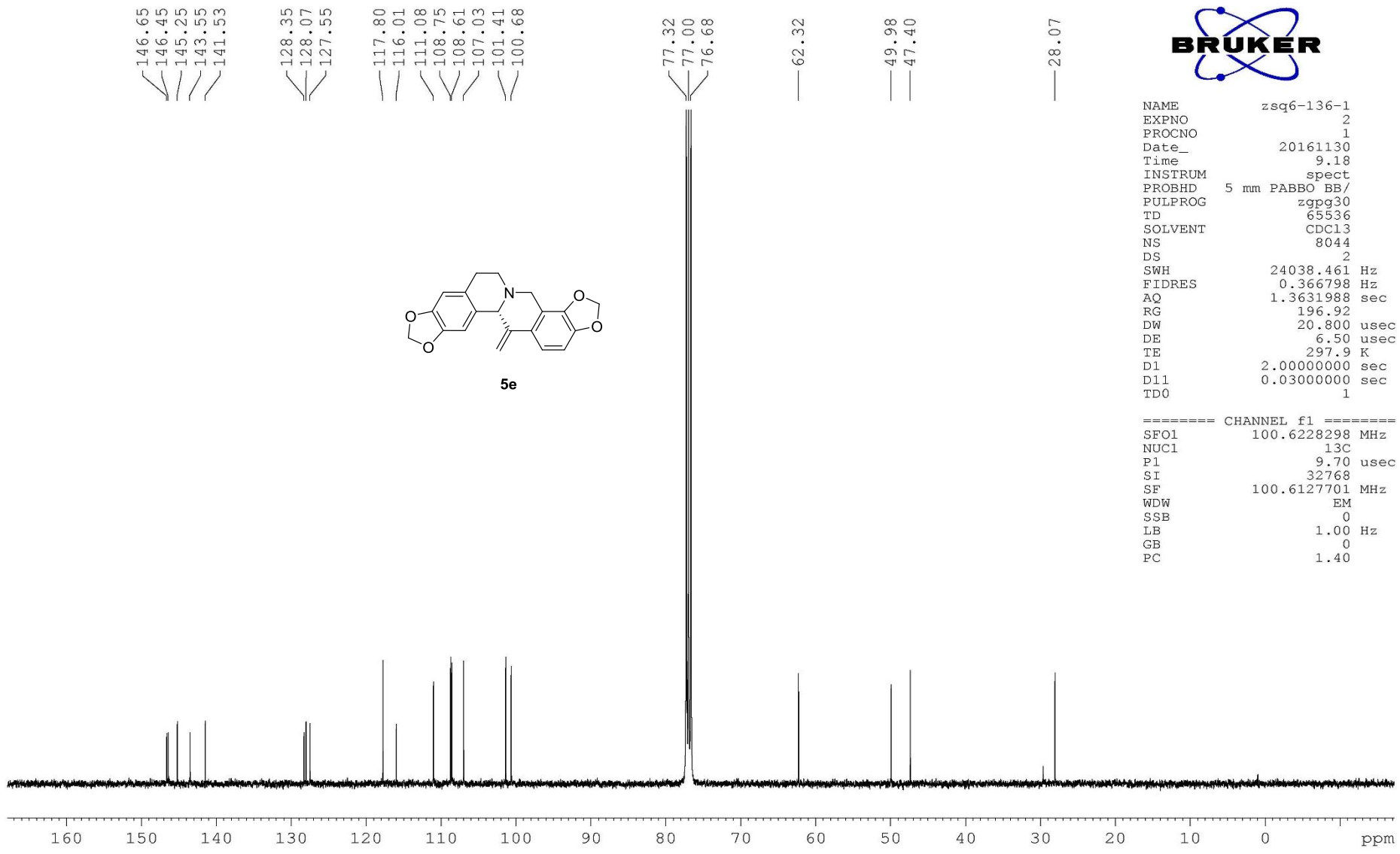

7.260
7.073
6.640
6.633
6.517
5.945
5.942
5.923
5.919
5.535
4.691
4.689
4.517
4.284
4.243
3.887
3.865
3.846
3.832
3.139
3.129
3.115
3.100
3.090
2.887
2.872
2.860
2.838
2.825
2.809
MeO
MeO
N
O
O
5c
NAME zsq6-126-1
EXPNO 1
PROCNO 1
Date_ 20161128
Time 14.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 112.31
DW 62.400 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
2.00
1.00
2.01
1.01
1.07
1.05
1.04
3.59
3.46
1.10
3.12


147.85
147.69
146.66
146.57
142.05
127.74
127.23
126.89
126.54
111.75
111.60
110.48
105.87
104.09
100.89
77.32
77.00
76.68
61.96
56.10
55.97
55.82
47.25
27.91
MeO
MeO
N
O
O
5c
BRUKER
NAME zsq6-126-1
EXPNO 2
PROCNO 1
Date_ 20161129
Time 10.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 259
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127722 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 ppm

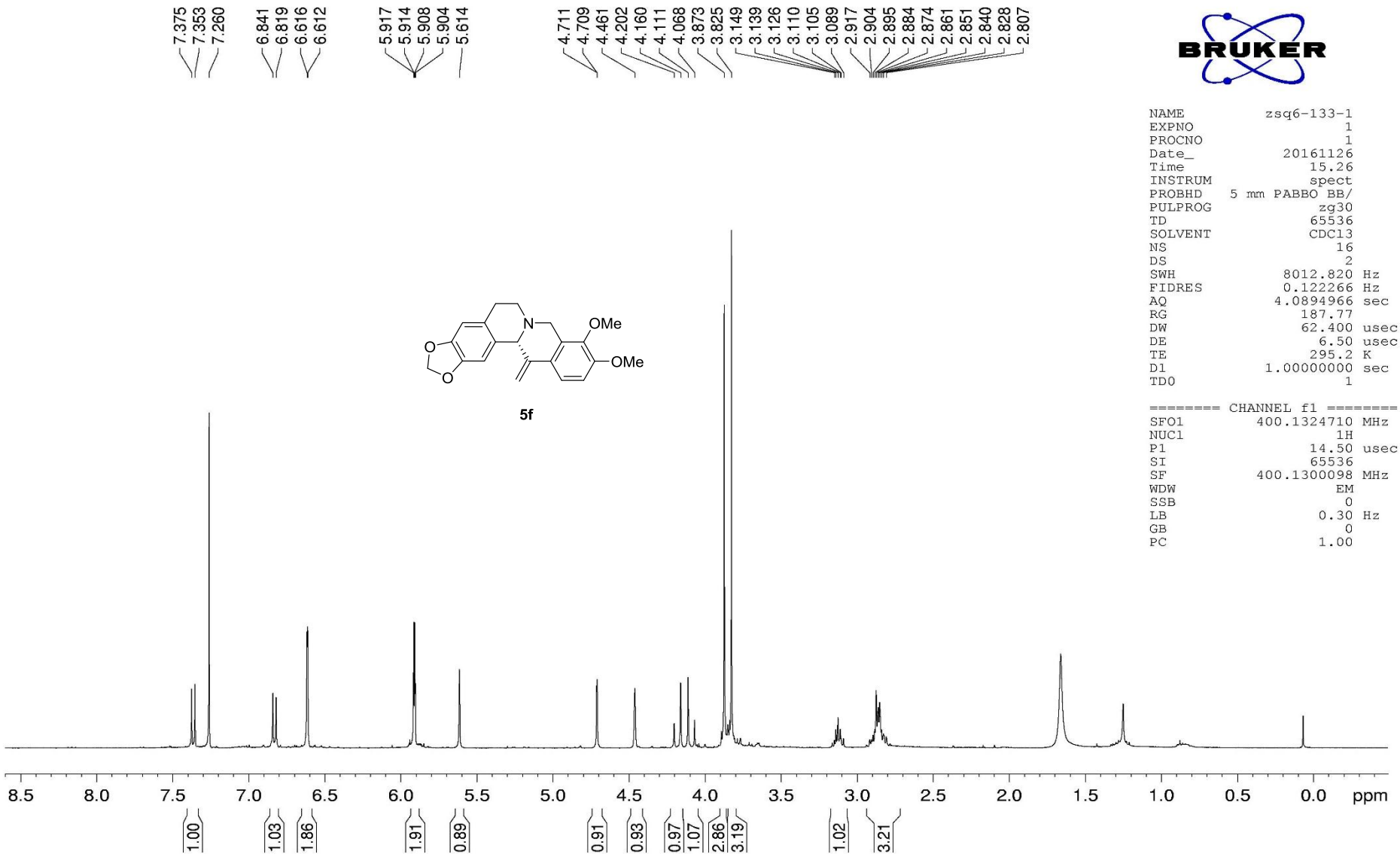

7.260
7.089
6.651
6.644
6.544
5.583
4.692
4.690
4.547
4.357
4.317
3.916
3.908
3.877
3.871
3.837
3.162
3.152
3.147
3.135
3.123
3.109
2.903
2.883
2.871
2.844
2.831
2.816
BRUKER
NAME zsq6-123-1a
EXPNO 1
PROCNO 1
Date_ 20161128
Time 22.28
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 187.77
DW 62.400 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
MeO
N
OMe
OMe
5d
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.90
0.95
0.96
0.99
0.96
1.00
3.38
3.18
2.77
2.86
1.07
3.02

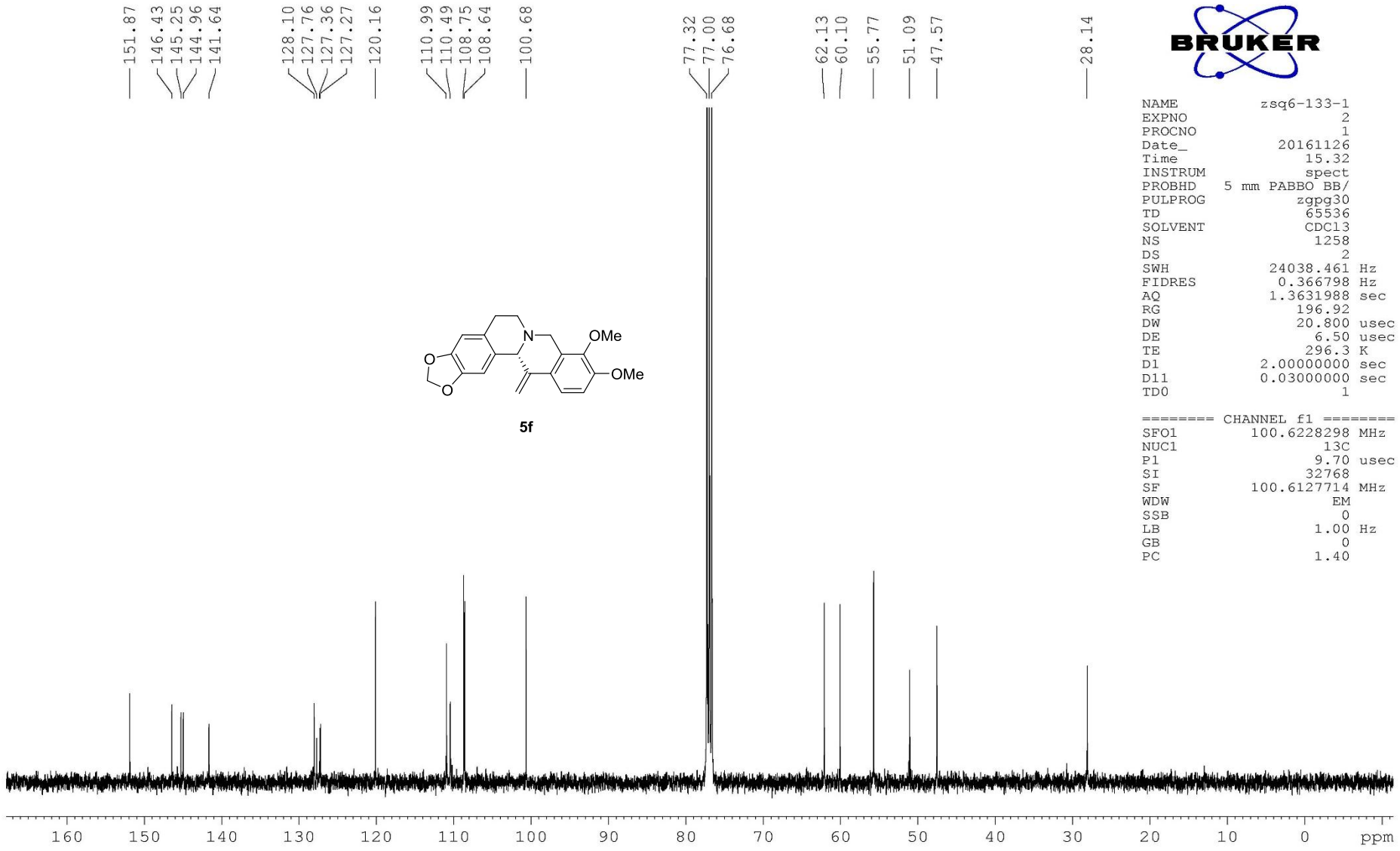

149.47
147.84
146.54
142.09
126.95
126.71
126.42
125.95
111.84
111.63
110.09
108.52
106.88
77.32
77.00
76.68
61.93
55.99
55.95
55.86
55.83
47.17
28.12
MeO
MeO
OMe
OMe
N
5d
BRUKER
NAME zsq6-123-1a
EXPNO 2
PROCNO 1
Date_ 20161129
Time 13.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1078
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127712 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 ppm


7.260
7.170
7.149
6.738
6.718
6.616
6.603
5.981
5.978
5.962
5.958
5.916
5.913
5.909
5.905
5.625
4.693
4.691
4.507
4.200
4.158
3.970
3.928
3.160
3.148
3.135
3.128
3.119
3.108
3.096
2.918
2.906
2.898
2.886
2.871
2.861
2.841
BRUKER
NAME zsq6-136-1
EXPNO 1
PROCNO 1
Date_ 20161129
Time 22.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 187.77
DW 62.400 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
O
O
O
O
5e
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.00
1.98
2.06
2.10
1.04
1.07
1.08
1.13
1.15
1.10
3.13

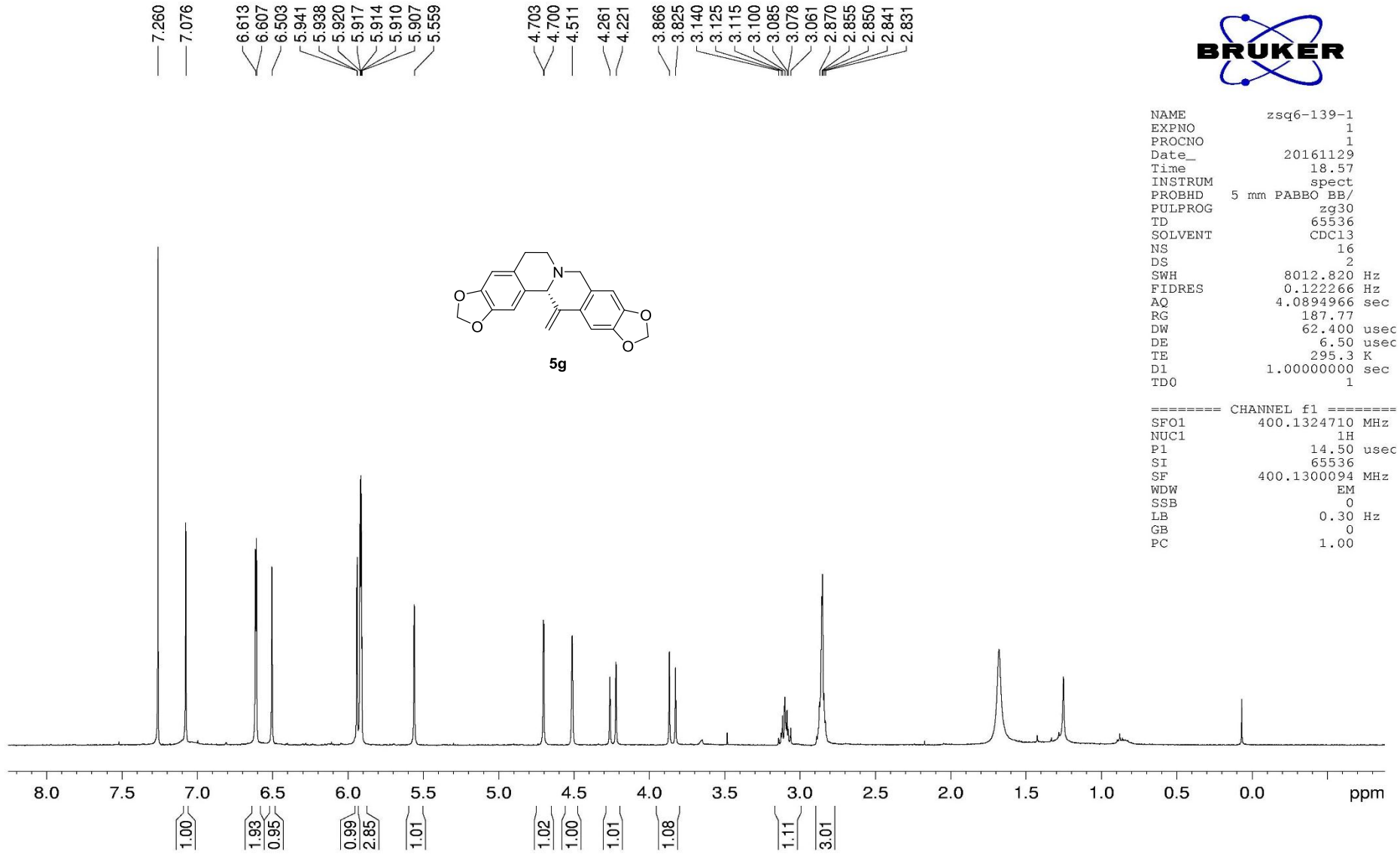

146.65
146.45
145.25
143.55
141.53
128.35
128.07
127.55
117.80
116.01
111.08
108.75
108.61
107.03
101.41
100.68
77.32
77.00
76.68
62.32
49.98
47.40
28.07
BRUKER
NAME zsq6-136-1
EXPNO 2
PROCNO 1
Date_ 20161130
Time 9.18
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 8044
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127701 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

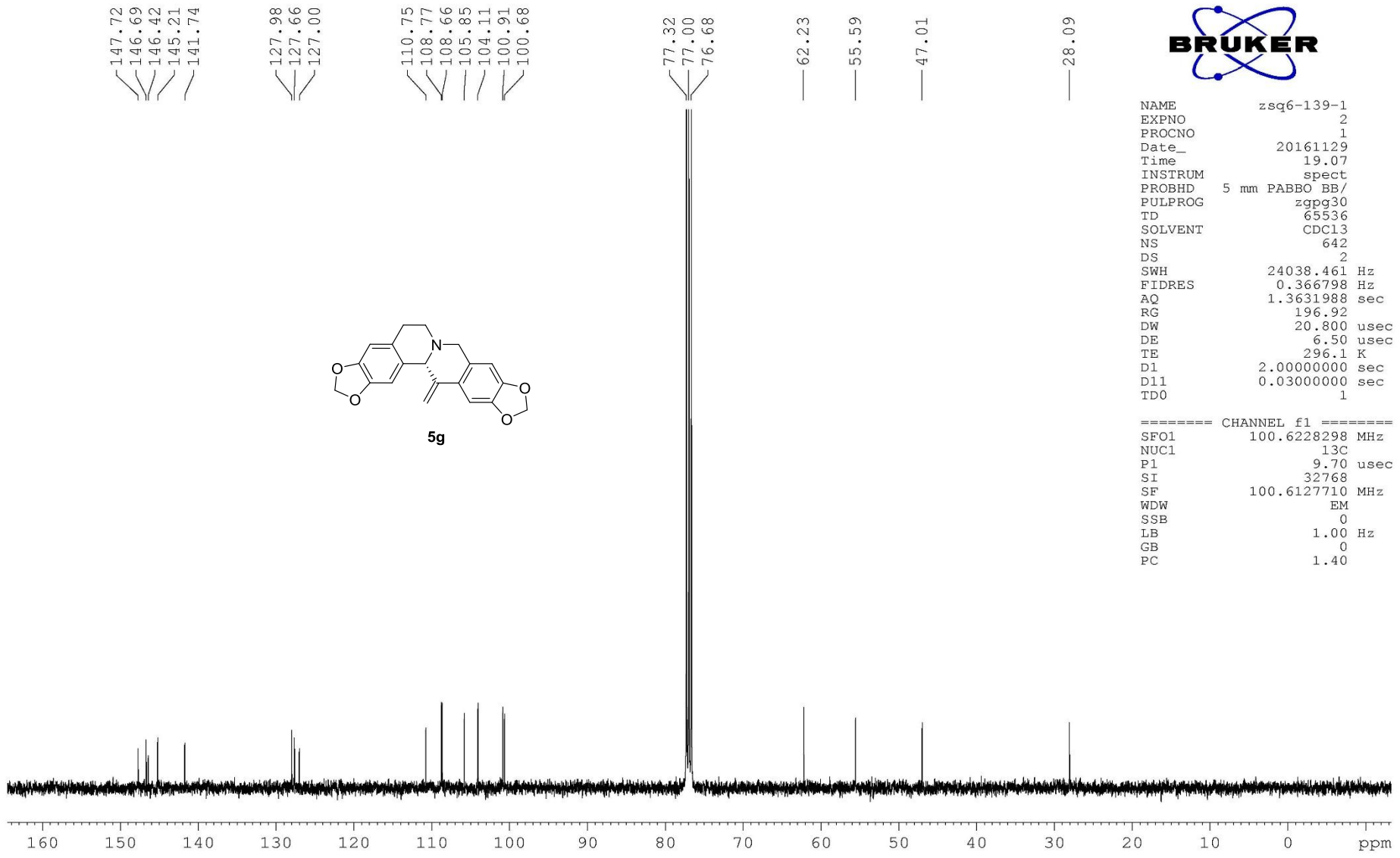
5e


7.375
7.353
7.260
6.841
6.819
6.616
6.612
5.917
5.914
5.908
5.904
5.614
4.711
4.709
4.461
4.202
4.160
4.111
4.068
3.873
3.825
3.149
3.139
3.126
3.110
3.105
3.089
2.917
2.904
2.895
2.884
2.874
2.861
2.851
2.840
2.828
2.807
BRUKER
NAME zsq6-133-1
EXPNO 1
PROCNO 1
Date_ 20161126
Time 15.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 187.77
DW 62.400 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OMe
OMe
N
O
O
5f
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.03
1.86
1.91
0.89
0.91
0.93
0.97
1.07
2.86
3.19
1.02
3.21

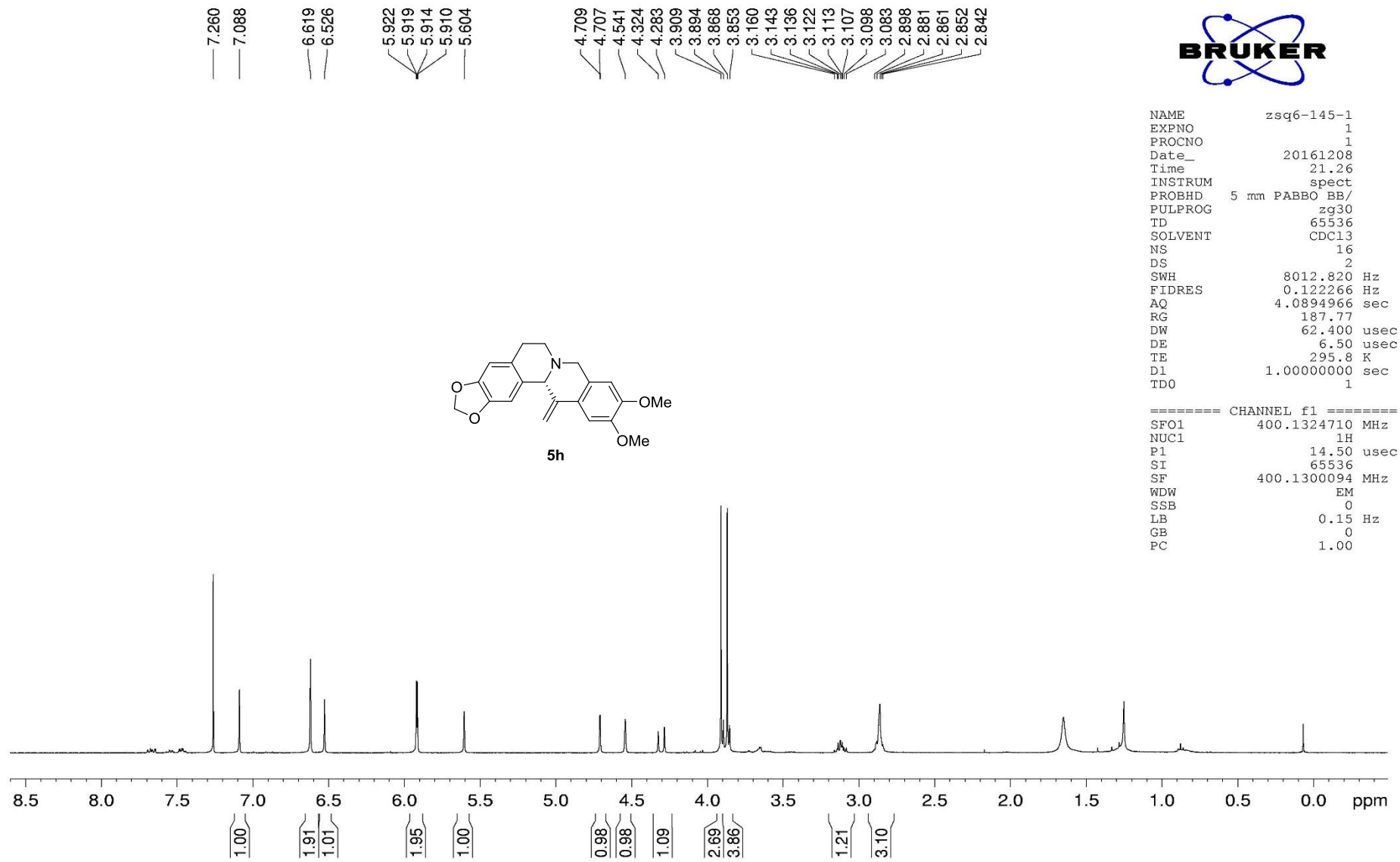

151.87
146.43
145.25
144.96
141.64
128.10
127.76
127.36
127.27
120.16
110.99
110.49
108.75
108.64
100.68
77.32
77.00
76.68
62.13
60.10
55.77
51.09
47.57
28.14
BRUKER
NAME zsq6-133-1
EXPNO 2
PROCNO 1
Date_ 20161126
Time 15.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1258
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127714 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
O
O
OMe
OMe
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

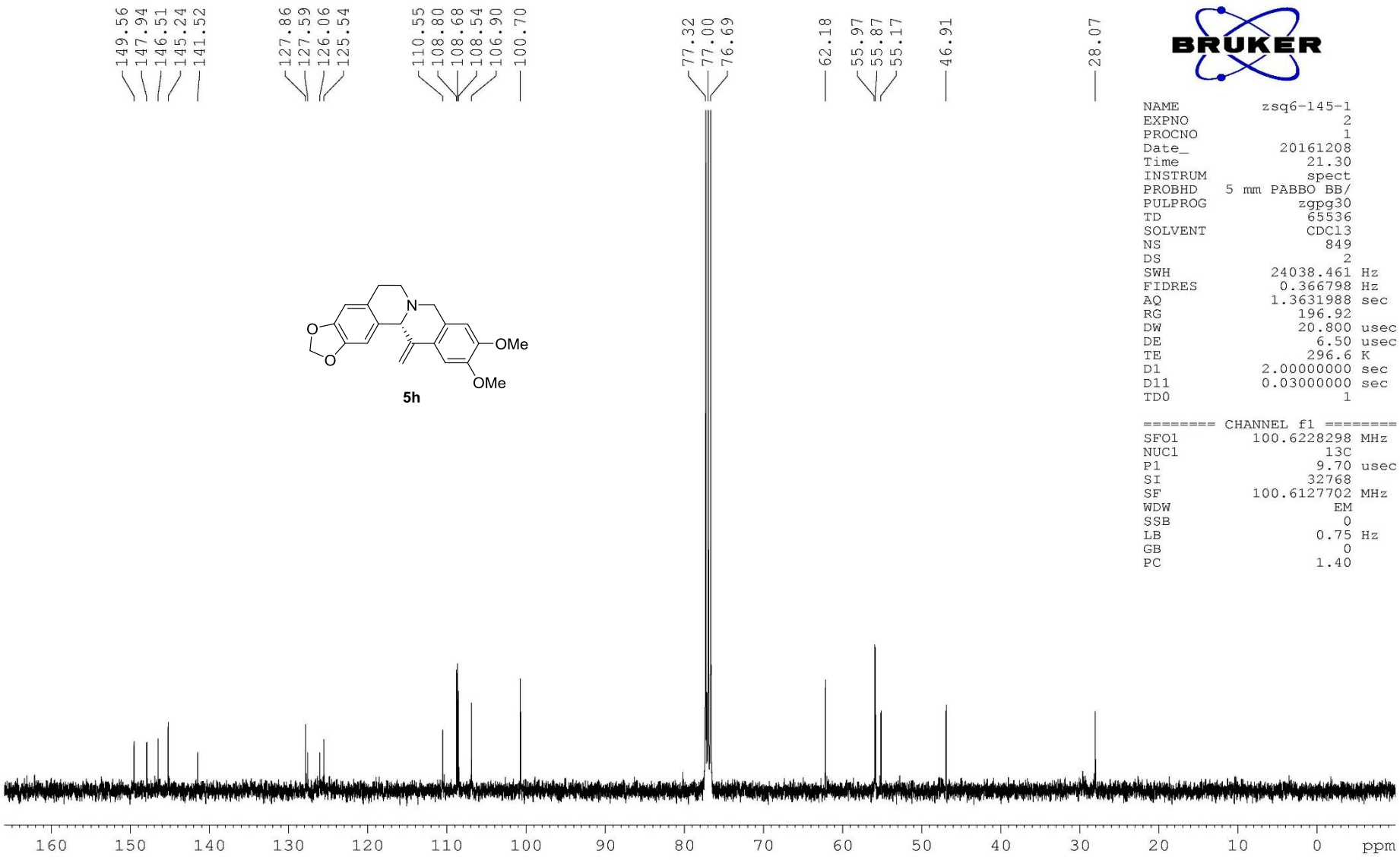
5f

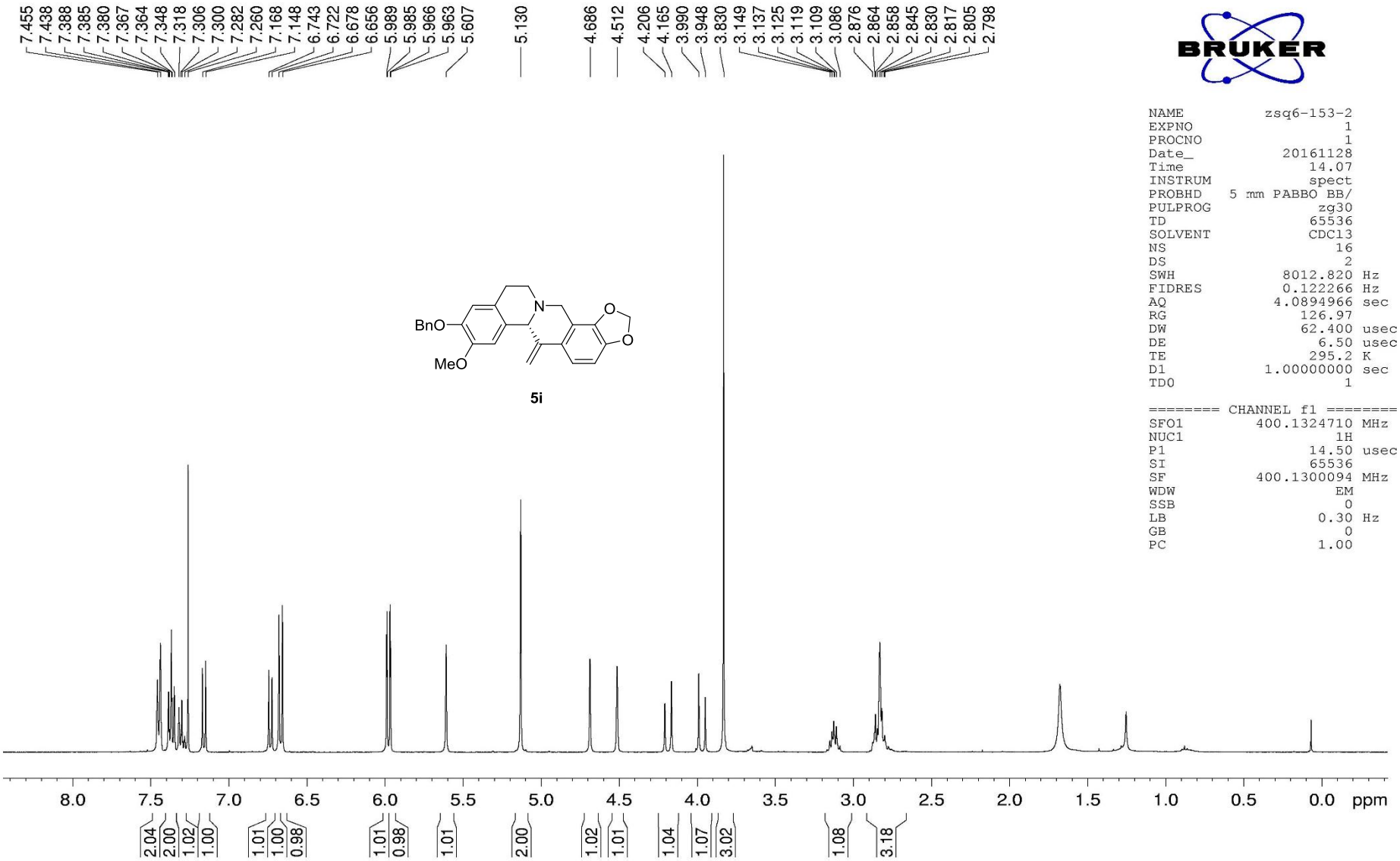

7.260
7.076
6.613
6.607
6.503
5.941
5.938
5.920
5.917
5.914
5.910
5.907
5.559
4.703
4.700
4.511
4.261
4.221
3.866
3.825
3.140
3.125
3.115
3.100
3.085
3.078
3.061
2.870
2.855
2.850
2.841
2.831
BRUKER
NAME zsq6-139-1
EXPNO 1
PROCNO 1
Date_ 20161129
Time 18.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 187.77
DW 62.400 usec
DE 6.50 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
5g
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.93
0.95
0.99
2.85
1.01
1.02
1.00
1.01
1.08
1.11
3.01

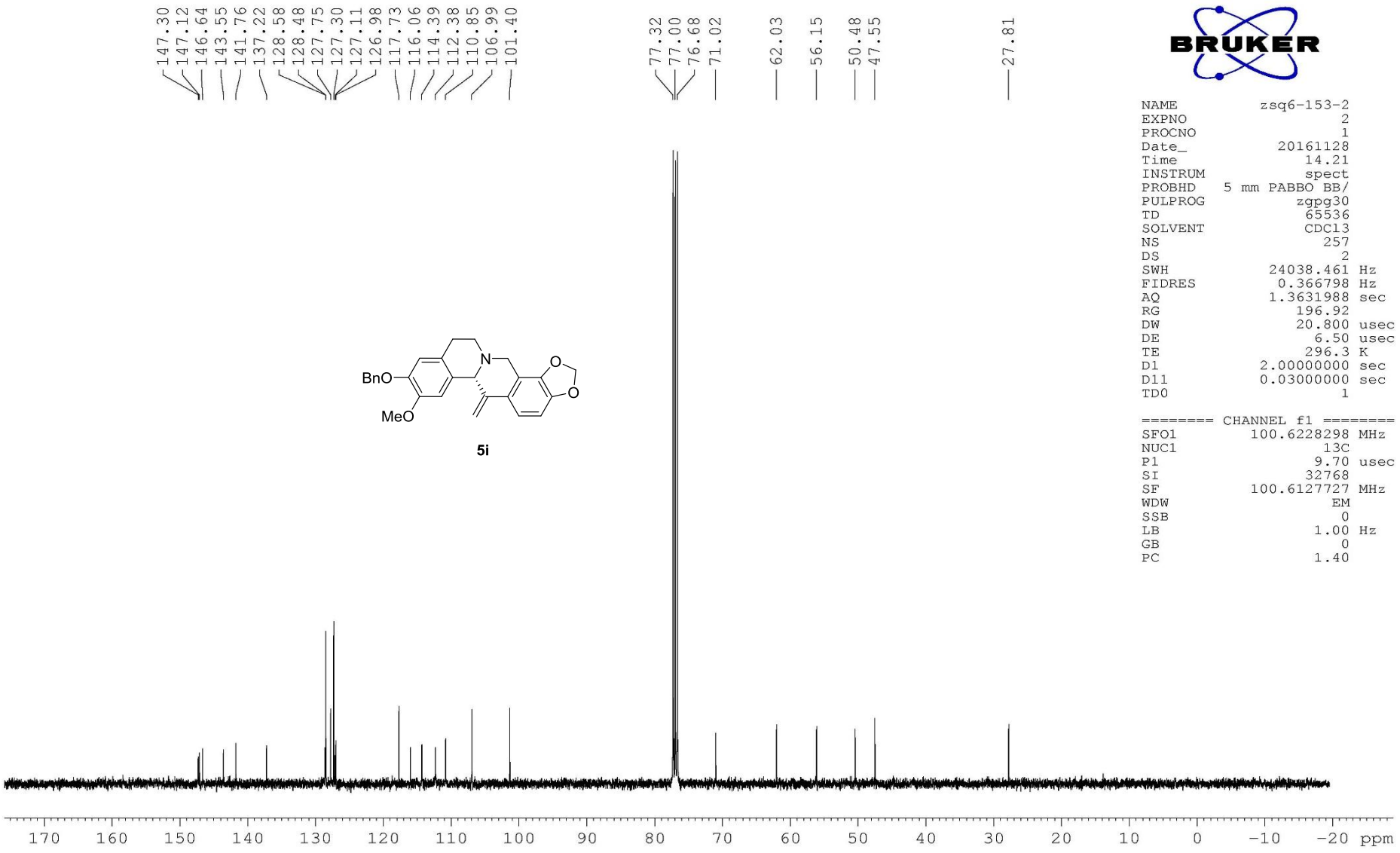

147.72
146.69
146.42
145.21
141.74
127.98
127.66
127.00
110.75
108.77
108.66
105.85
104.11
100.91
100.68
77.32
77.00
76.68
62.23
55.59
47.01
28.09
BRUKER
NAME zsq6-139-1
EXPNO 2
PROCNO 1
Date_ 20161129
Time 19.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 642
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127710 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

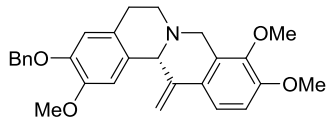
5g


7.260
7.088
6.619
6.526
5.922
5.919
5.914
5.910
5.604
4.709
4.707
4.541
4.324
4.283
3.909
3.894
3.868
3.853
3.160
3.143
3.136
3.122
3.113
3.107
3.098
3.083
2.898
2.881
2.861
2.852
2.842
BRUKER
NAME zsq6-145-1
EXPNO 1
PROCNO 1
Date_ 20161208
Time 21.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 187.77
DW 62.400 usec
DE 6.50 usec
TE 295.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.15 Hz
GB 0
PC 1.00
O
O
N
OMe
OMe
5h
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.91
1.01
1.95
1.00
0.98
0.98
1.09
2.69
3.86
1.21
3.10


149.56
147.94
146.51
145.24
141.52
127.86
127.59
126.06
125.54
110.55
108.80
108.68
108.54
106.90
100.70
77.32
77.00
76.69
62.18
55.97
55.87
55.17
46.91
28.07
BRUKER
NAME zsq6-145-1
EXPNO 2
PROCNO 1
Date_ 20161208
Time 21.30
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 849
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127702 MHz
WDW EM
SSB 0
LB 0.75 Hz
GB 0
PC 1.40
O
O
N
OMe
OMe
5h
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.455
7.438
7.388
7.385
7.380
7.367
7.364
7.348
7.318
7.306
7.300
7.282
7.260
7.168
7.148
6.743
6.722
6.678
6.656
5.989
5.985
5.966
5.963
5.607
5.130
4.686
4.512
4.206
4.165
3.990
3.948
3.830
3.149
3.137
3.125
3.119
3.109
3.086
2.876
2.864
2.858
2.845
2.830
2.817
2.805
2.798
BRUKER
NAME zsq6-153-2
EXPNO 1
PROCNO 1
Date_ 20161128
Time 14.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 126.97
DW 62.400 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
BnO
MeO
N
O
O
5i
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.04
2.00
1.02
1.00
1.01
1.00
0.98
1.01
0.98
1.01
2.00
1.02
1.01
1.04
1.07
3.02
1.08
3.18

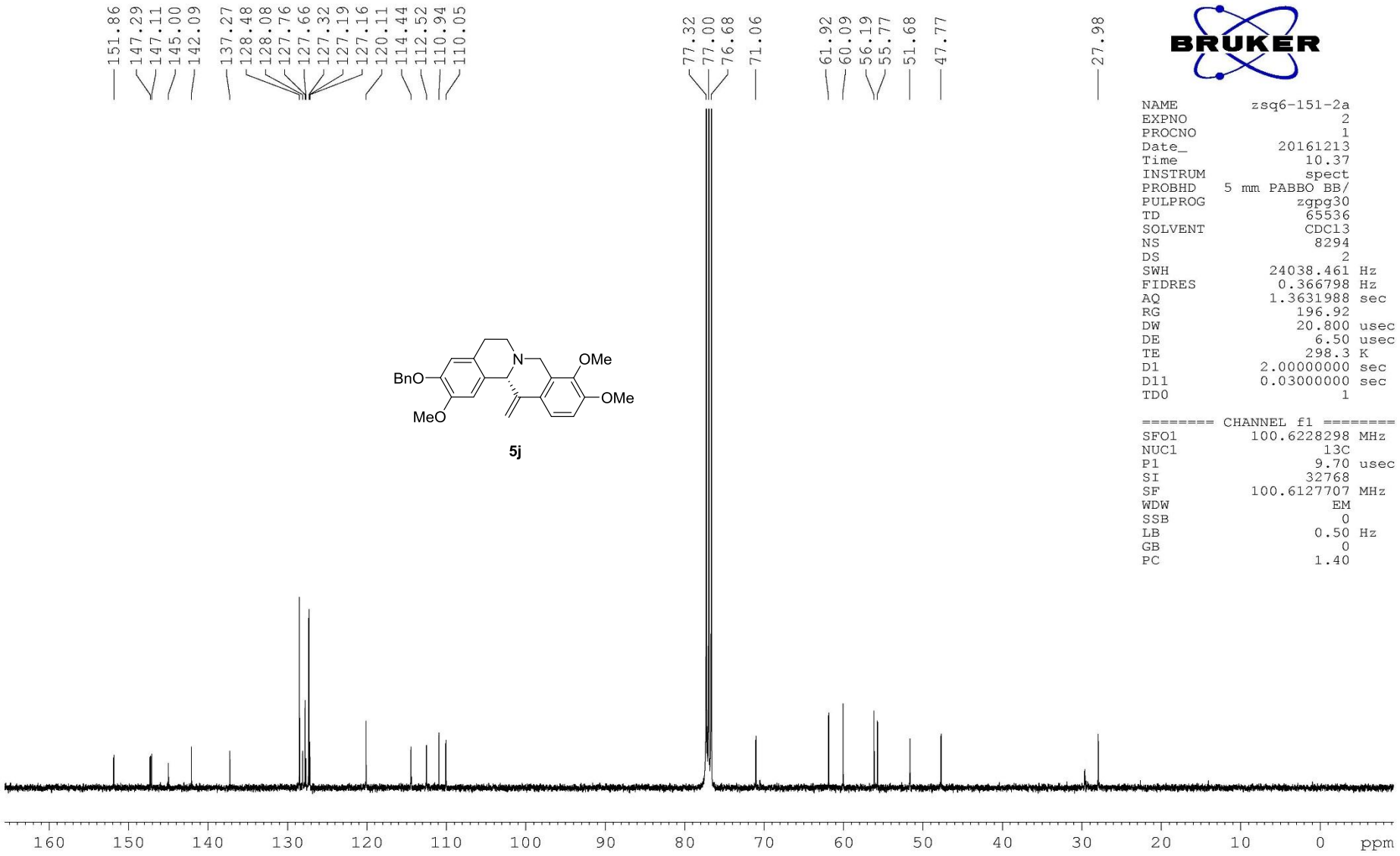

147.30
147.12
146.64
143.55
141.76
137.22
128.58
128.48
127.75
127.30
127.11
126.98
117.73
116.06
114.39
112.38
110.85
106.99
101.40
77.32
77.00
76.68
71.02
62.03
56.15
50.48
47.55
27.81
BnO
MeO
N
O
O
5i
NAME zsq6-153-2
EXPNO 2
PROCNO 1
Date_ 20161128
Time 14.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 257
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127727 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 ppm

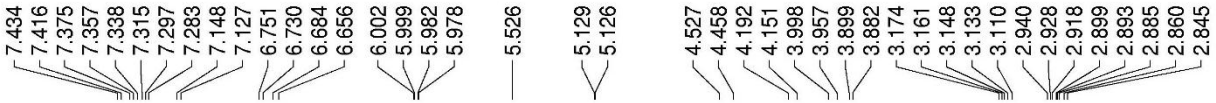


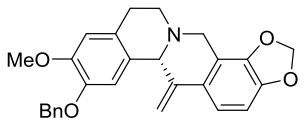
5j

```
NAME        zsq6-151-2maybe
EXPNO                     1
PROCNO                    1
Date_              20161126
Time                 20.03
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES                0.122266 Hz
AQ                 4.0894966 sec
RG                    142.88
DW                    62.400 usec
DE                     6.50 usec
TE                    297.1 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                    1H
P1                    14.50 usec
SI                    65536
SF            400.1300097 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```


151.86
147.29
147.11
145.00
142.09
137.27
128.48
128.08
127.76
127.66
127.32
127.19
127.16
120.11
114.44
112.52
110.94
110.05
77.32
77.00
76.68
71.06
61.92
60.09
56.19
55.77
51.68
47.77
27.98
BnO
MeO
N
OMe
OMe
5j
BRUKER
NAME zsq6-151-2a
EXPNO 2
PROCNO 1
Date_ 20161213
Time 10.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 8294
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127707 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

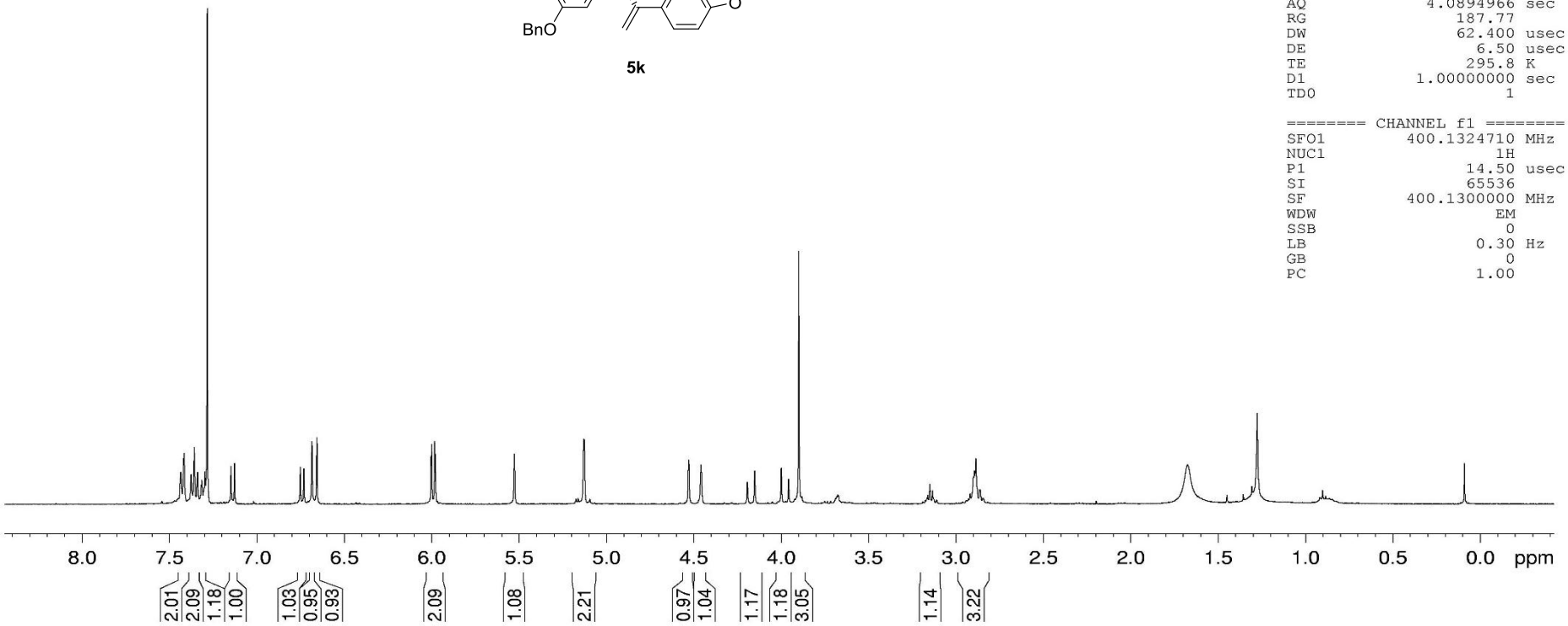


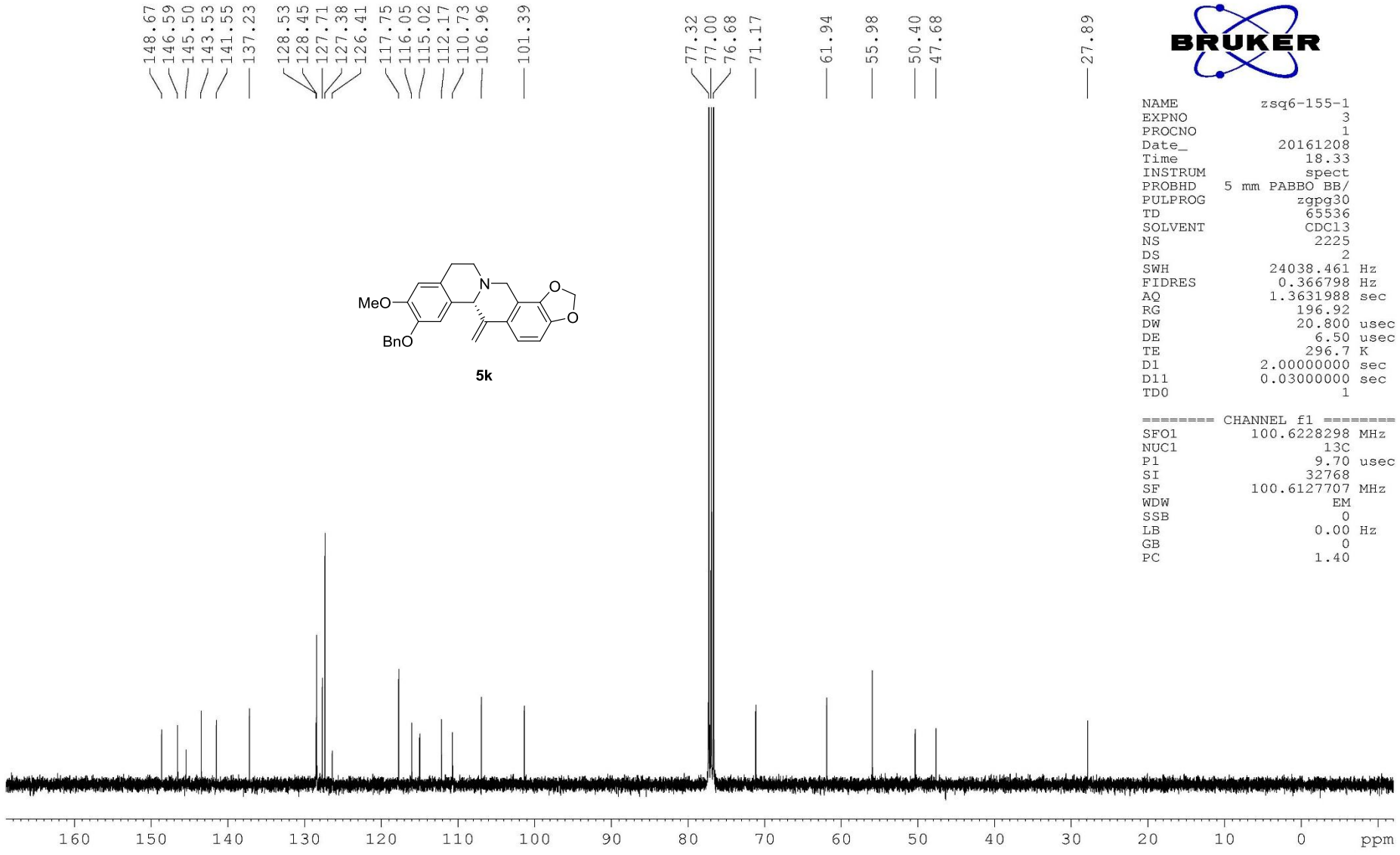


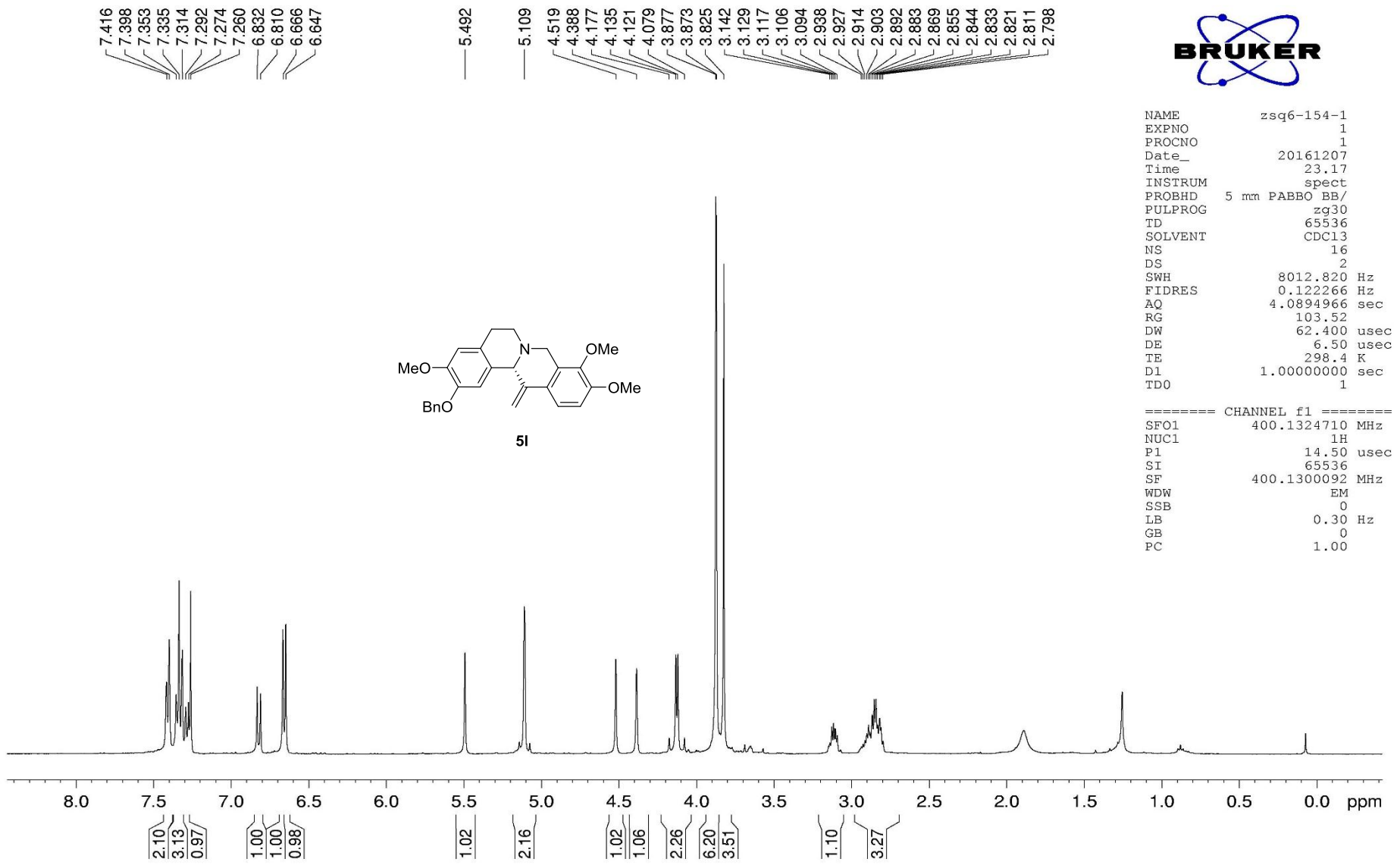
5k

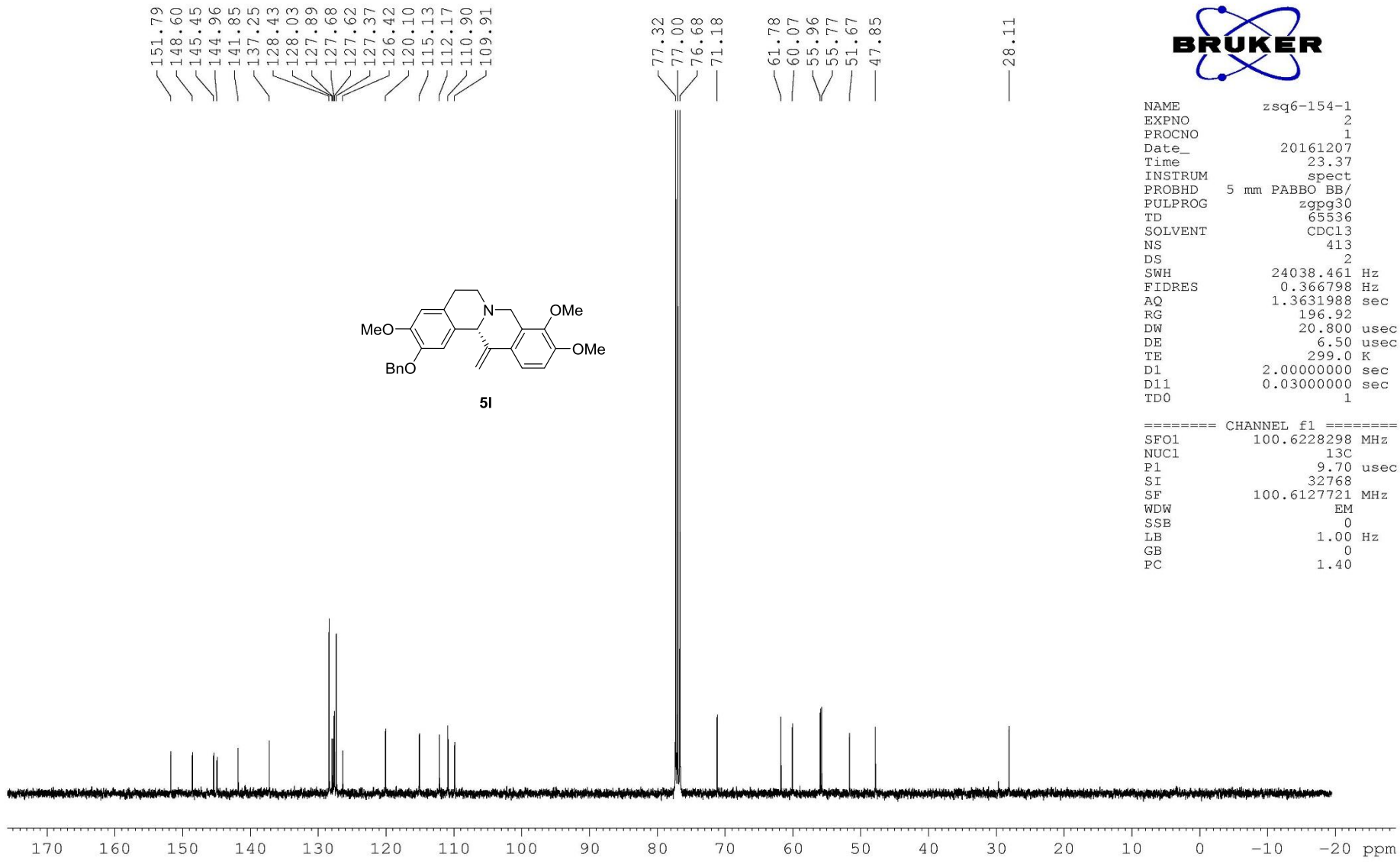
```
NAME            zsq6-155-1
EXPNO                    1
PROCNO                   1
Date_             20161207
Time                 12.15
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8012.820 Hz
FIDRES               0.122266 Hz
AQ                4.0894966 sec
RG                   187.77
DW                   62.400 usec
DE                    6.50 usec
TE                   295.8 K
D1              1.00000000 sec
TD0                      1

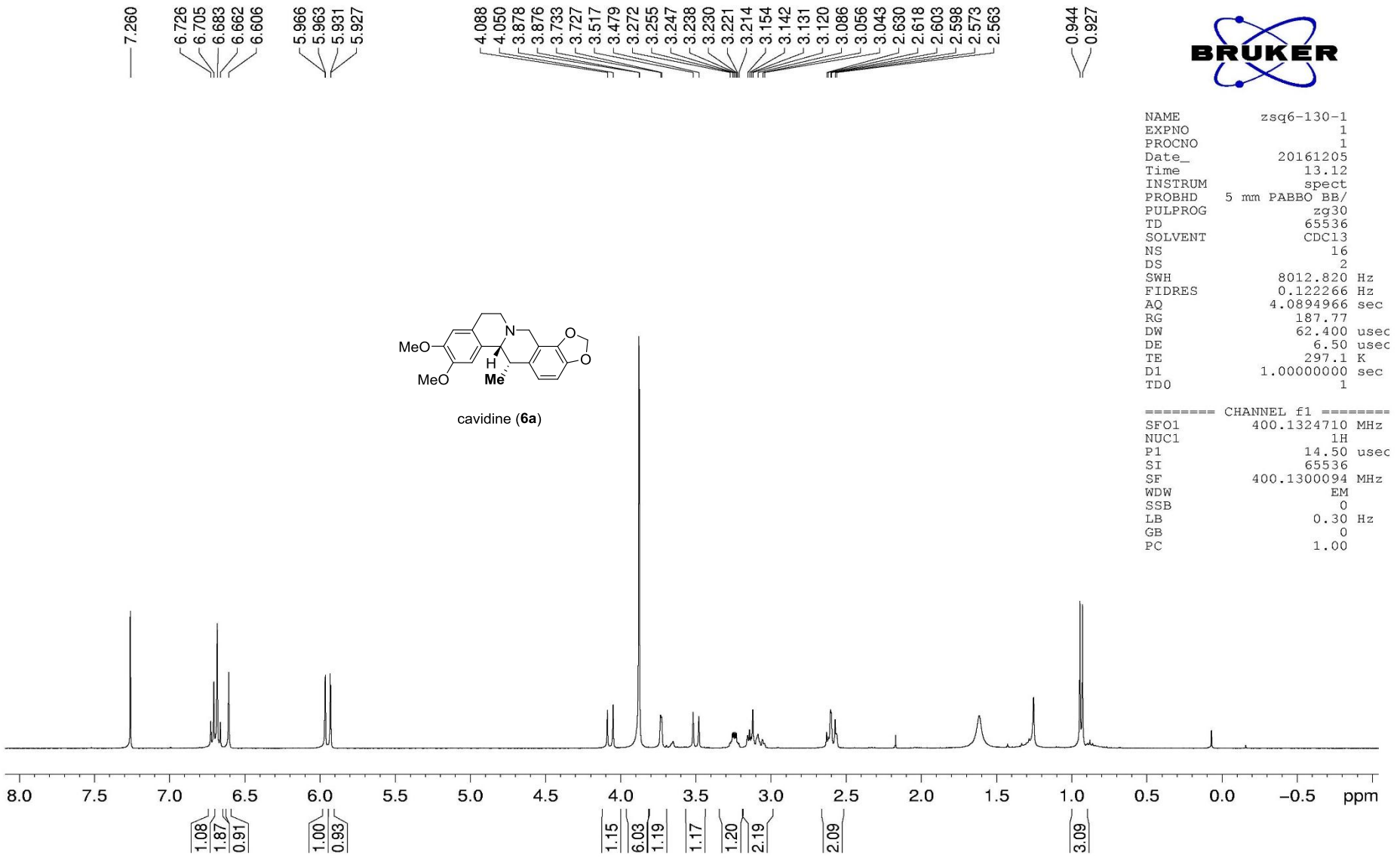
======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                    1H
P1                   14.50 usec
SI                   65536
SF            400.1300000 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

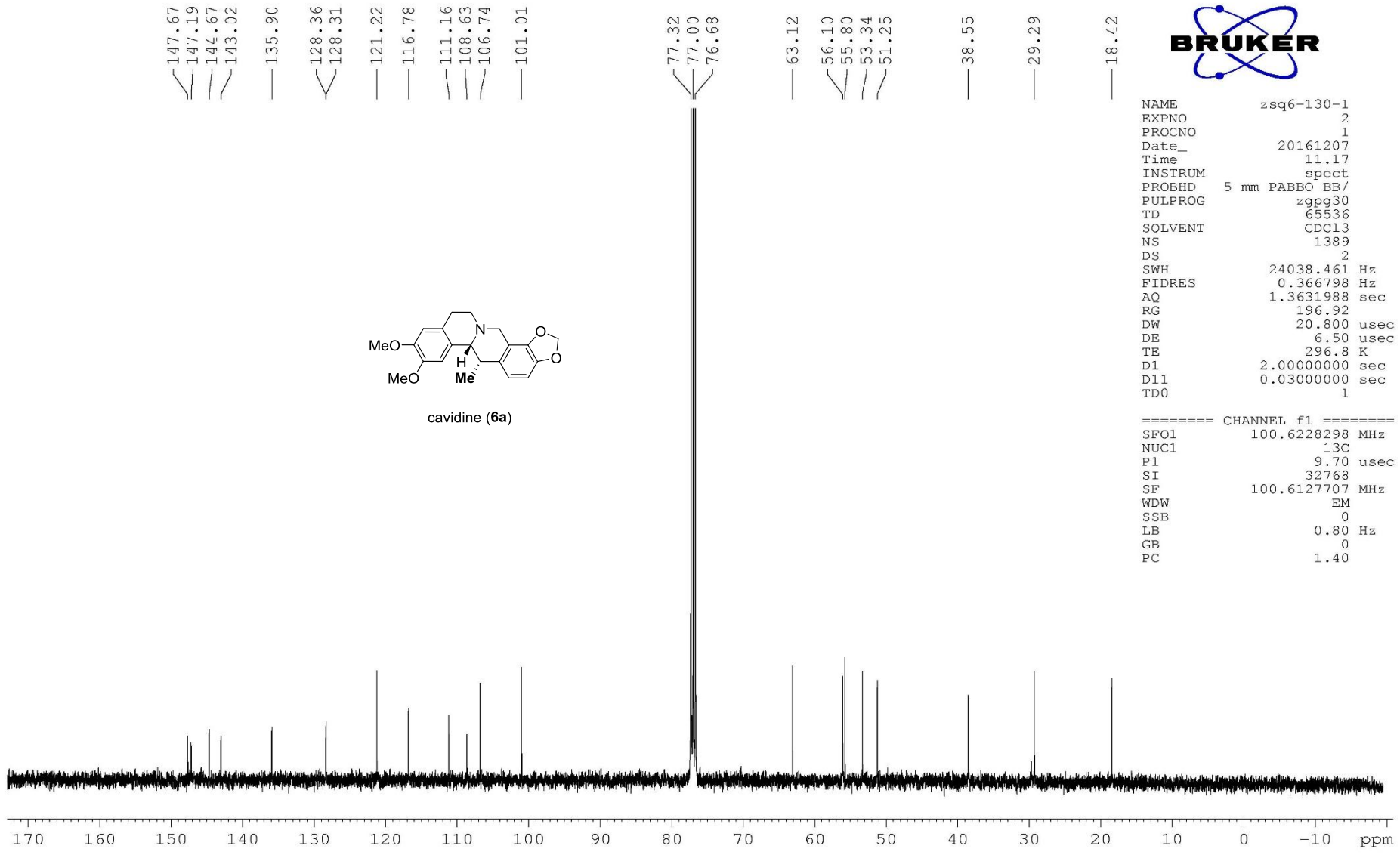



148.67
146.59
145.50
143.53
141.55
137.23
128.53
128.45
127.71
127.38
126.41
117.75
116.05
115.02
112.17
110.73
106.96
101.39
77.32
77.00
76.68
71.17
61.94
55.98
50.40
47.68
27.89
MeO
BnO
N
O
O
5k
BRUKER
NAME zsq6-155-1
EXPNO 3
PROCNO 1
Date_ 20161208
Time 18.33
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2225
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127707 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

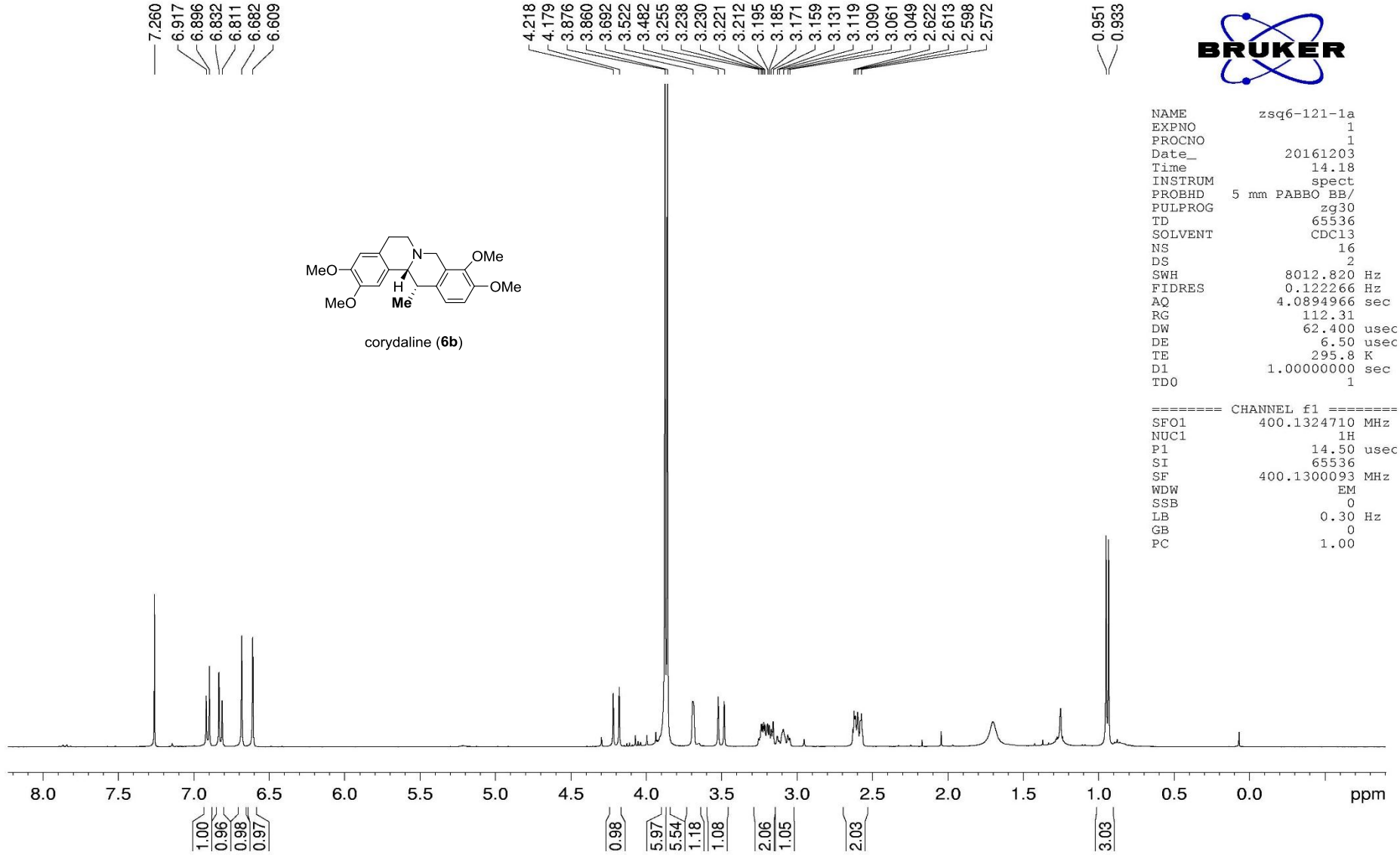

7.416
7.398
7.353
7.335
7.314
7.292
7.274
7.260
6.832
6.810
6.666
6.647
5.492
5.109
4.519
4.388
4.177
4.135
4.121
4.079
3.877
3.873
3.825
3.142
3.129
3.117
3.106
3.094
2.938
2.927
2.914
2.903
2.892
2.883
2.869
2.855
2.844
2.833
2.821
2.811
2.798
BRUKER
NAME zsq6-154-1
EXPNO 1
PROCNO 1
Date_ 20161207
Time 23.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 103.52
DW 62.400 usec
DE 6.50 usec
TE 298.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
BnO
N
OMe
OMe
5l
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.10
3.13
0.97
1.00
1.00
0.98
1.02
2.16
1.02
1.06
2.26
6.20
3.51
1.10
3.27


151.79
148.60
145.45
144.96
141.85
137.25
128.43
128.03
127.89
127.68
127.62
127.37
126.42
120.10
115.13
112.17
110.90
109.91
77.32
77.00
76.68
71.18
61.78
60.07
55.96
55.77
51.67
47.85
28.11
MeO
BnO
N
OMe
OMe
5l
BRUKER
NAME zsq6-154-1
EXPNO 2
PROCNO 1
Date_ 20161207
Time 23.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 413
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127721 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 ppm

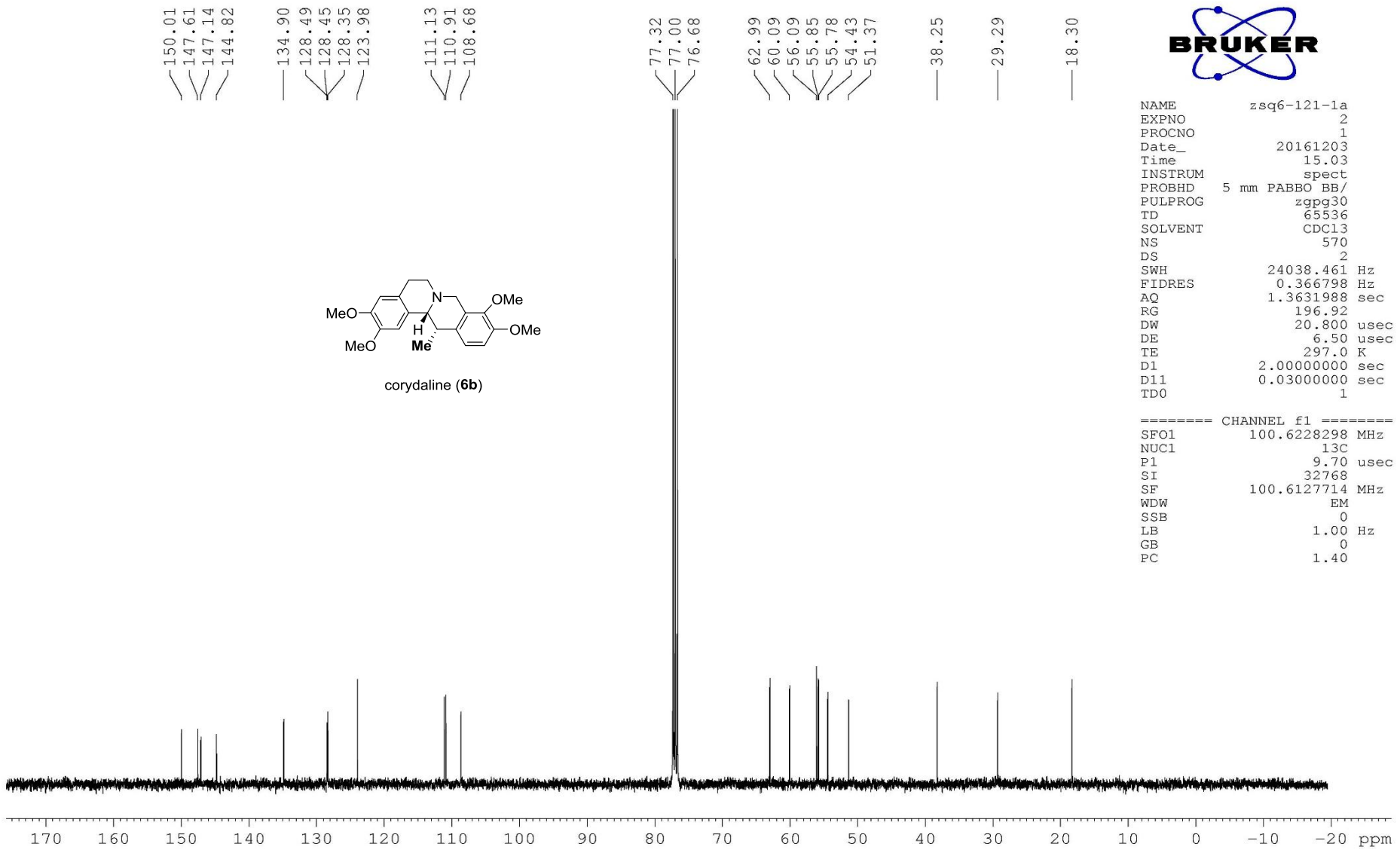

7.260
6.726
6.705
6.683
6.662
6.606
5.966
5.963
5.931
5.927
4.088
4.050
3.878
3.876
3.733
3.727
3.517
3.479
3.272
3.255
3.247
3.238
3.230
3.221
3.214
3.154
3.142
3.131
3.120
3.086
3.056
3.043
2.630
2.618
2.603
2.598
2.573
2.563
0.944
0.927
BRUKER
NAME zsq6-130-1
EXPNO 1
PROCNO 1
Date_ 20161205
Time 13.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 187.77
DW 62.400 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
MeO
N
O
O
H
Me
cavidine (6a)
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
ppm
1.08
1.87
0.91
1.00
0.93
1.15
6.03
1.19
1.17
1.20
2.19
2.09
3.09


147.67
147.19
144.67
143.02
135.90
128.36
128.31
121.22
116.78
111.16
108.63
106.74
101.01
77.32
77.00
76.68
63.12
56.10
55.80
53.34
51.25
38.55
29.29
18.42
BRUKER
NAME zsq6-130-1
EXPNO 2
PROCNO 1
Date_ 20161207
Time 11.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1389
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127707 MHz
WDW EM
SSB 0
LB 0.80 Hz
GB 0
PC 1.40
MeO
MeO
N
H
Me
O
O
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 ppm

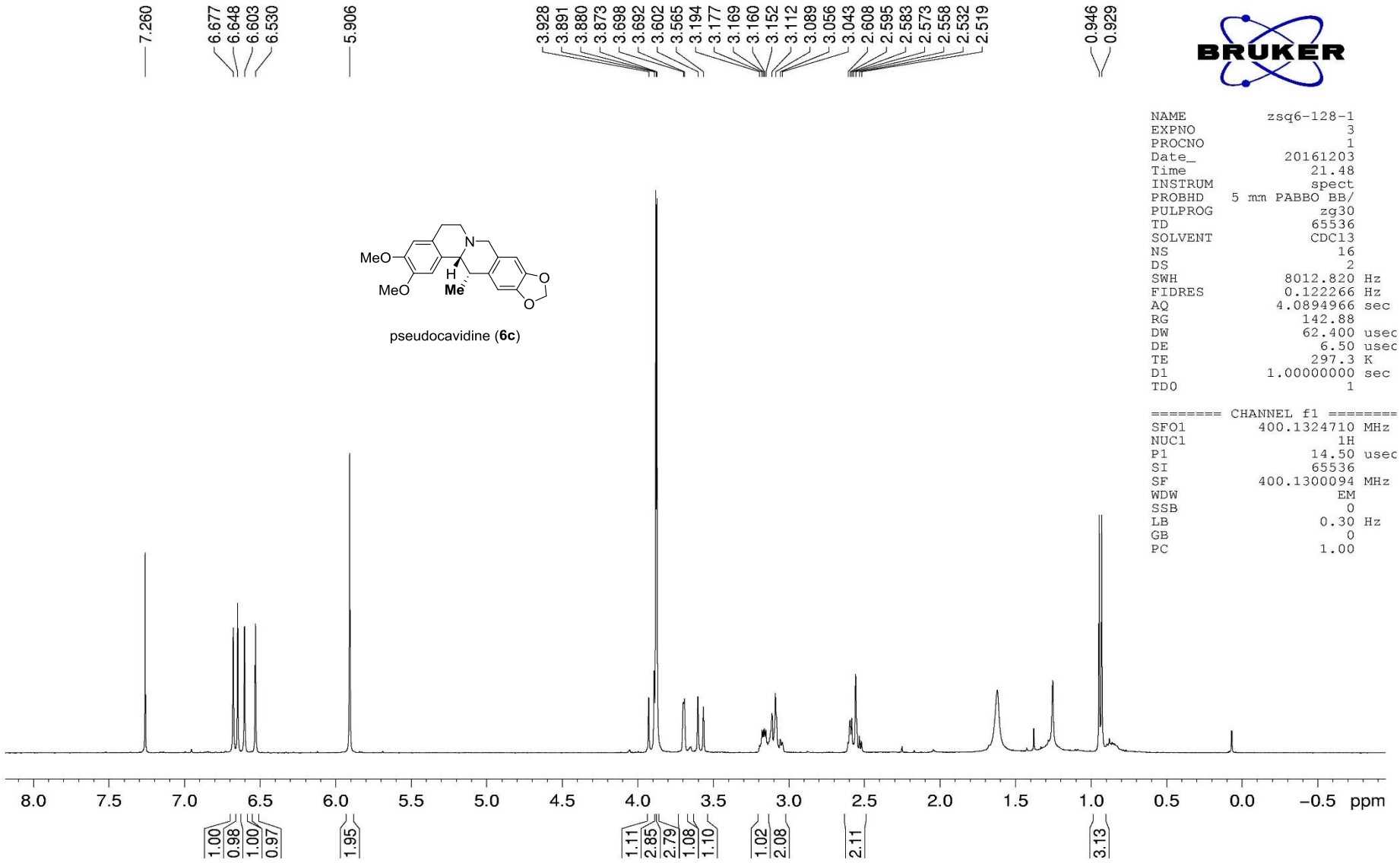
cavidine (**6a**)

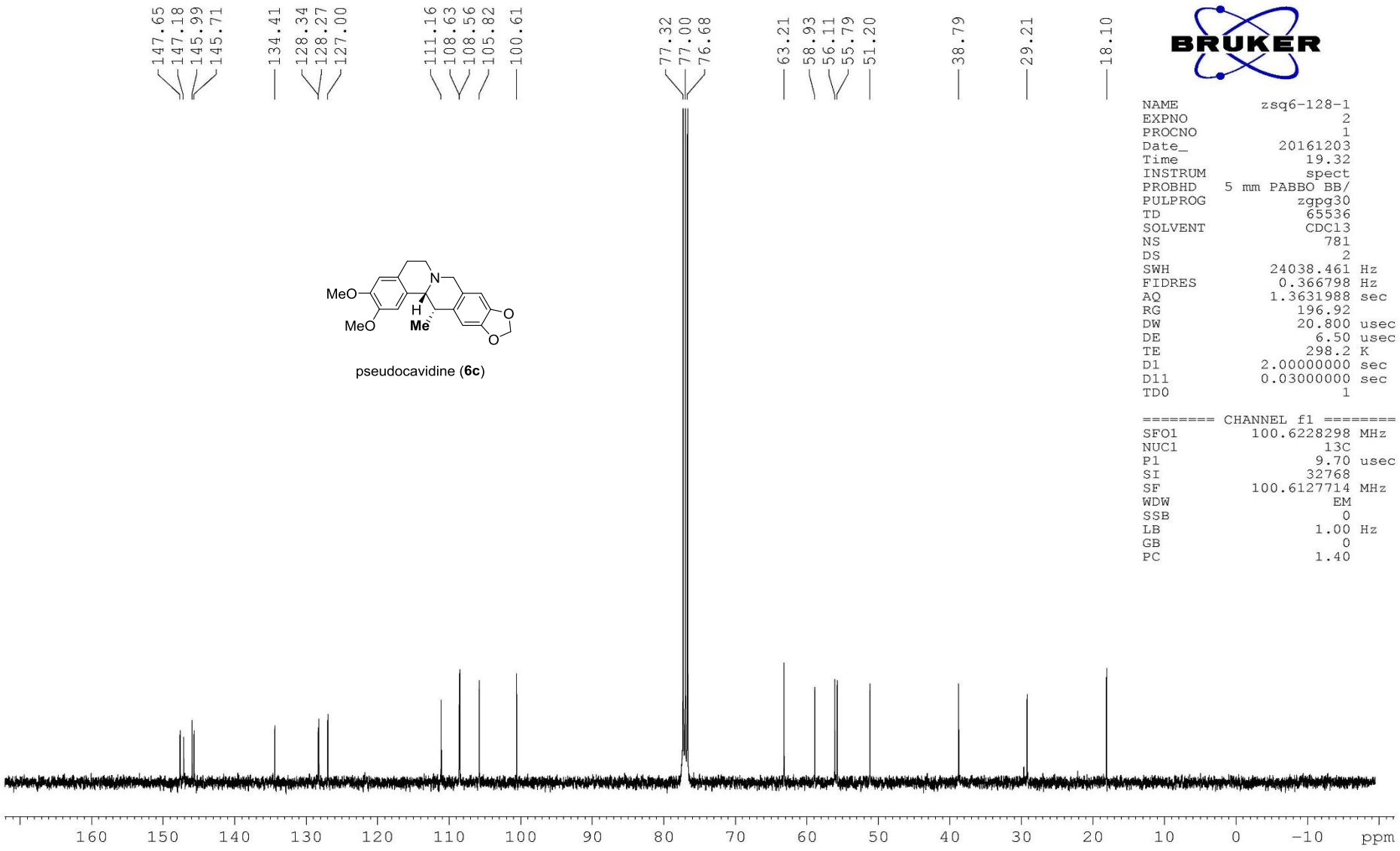


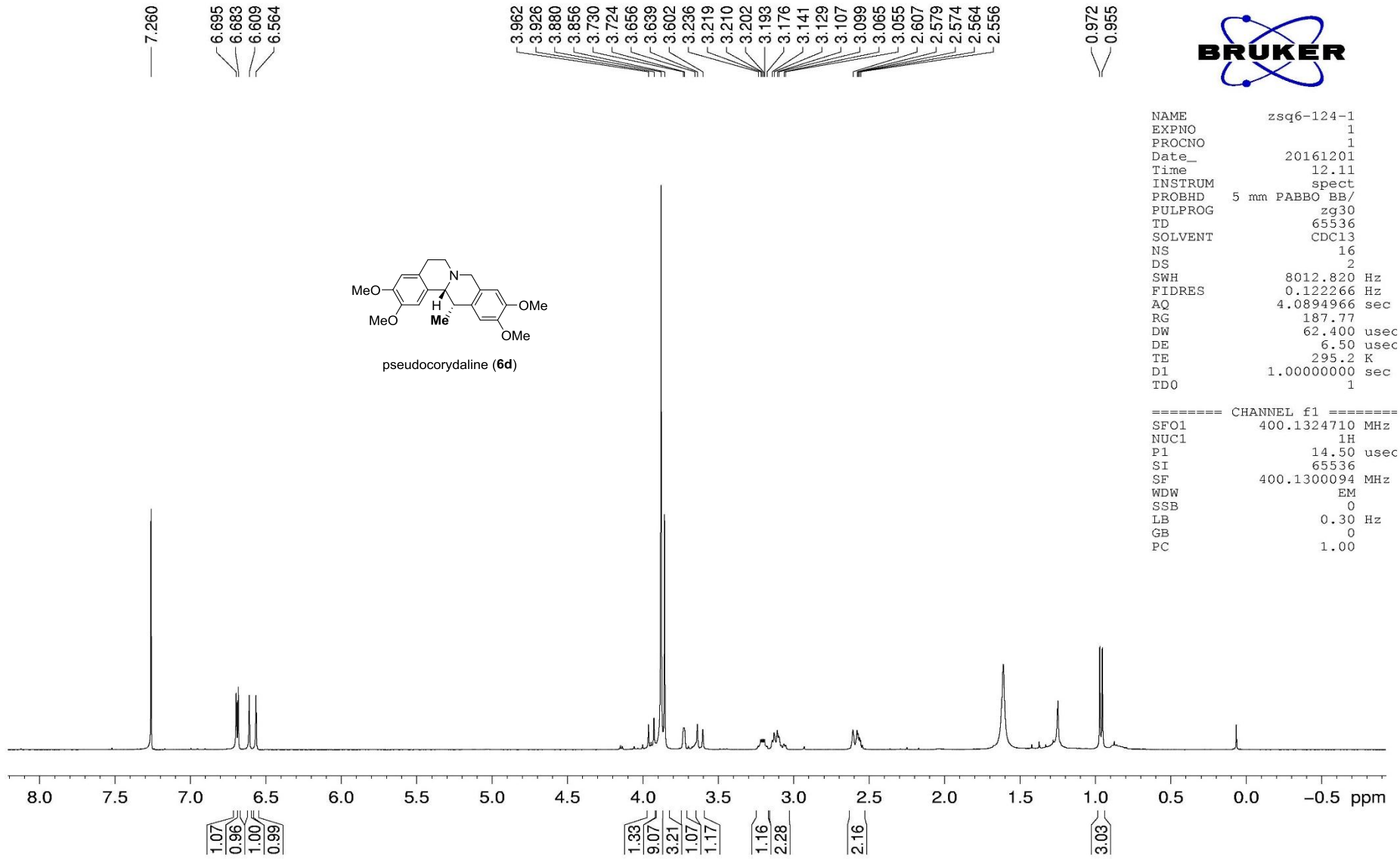
corydaline (**6b**)



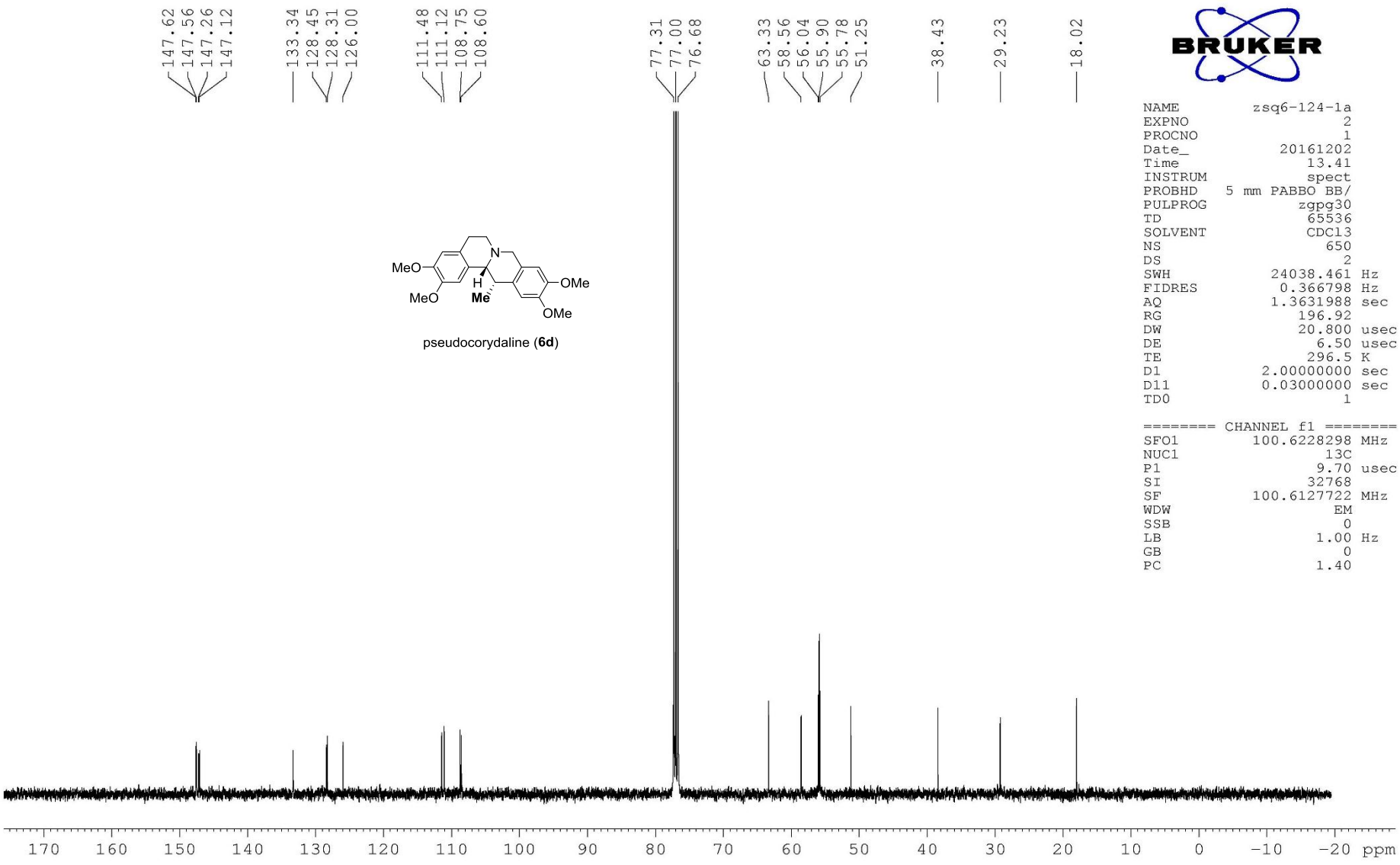
corydaline (**6b**)

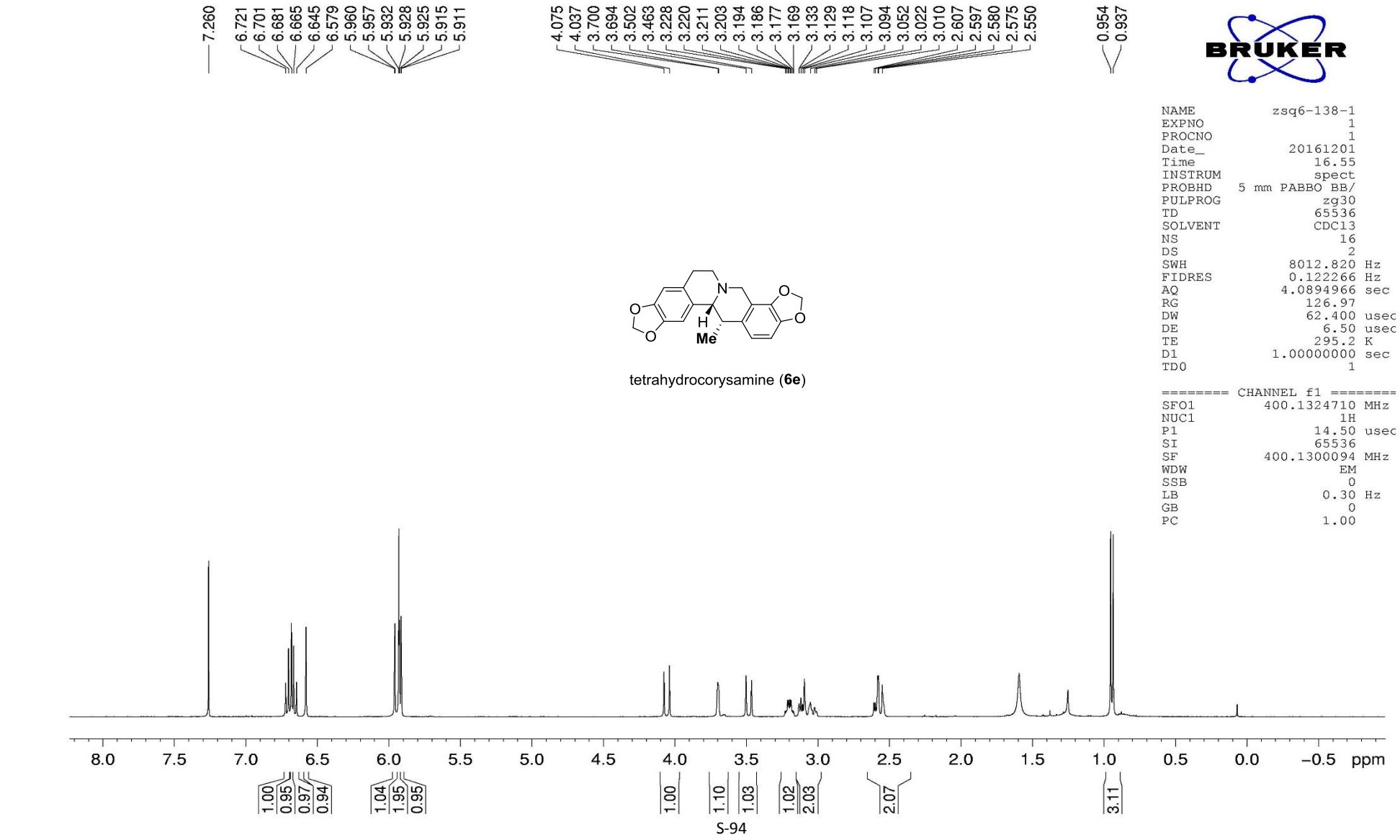

7.260
6.677
6.648
6.603
6.530
5.906
3.928
3.891
3.880
3.873
3.698
3.692
3.602
3.565
3.194
3.177
3.169
3.160
3.152
3.112
3.089
3.056
3.043
2.608
2.595
2.583
2.573
2.558
2.532
2.519
0.946
0.929
BRUKER
NAME zsq6-128-1
EXPNO 3
PROCNO 1
Date_ 20161203
Time 21.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 142.88
DW 62.400 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
MeO
H
Me
N
O
O
pseudocavidine (6c)
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
ppm
1.00
0.98
1.00
0.97
1.95
1.11
2.85
2.79
1.08
1.10
1.02
2.08
2.11
3.13


147.65
147.18
145.99
145.71
134.41
128.34
128.27
127.00
111.16
108.63
108.56
105.82
100.61
77.32
77.00
76.68
63.21
58.93
56.11
55.79
51.20
38.79
29.21
18.10
MeO
MeO
H
Me
N
O
O
pseudocavidine (6c)
BRUKER
NAME zsq6-128-1
EXPNO 2
PROCNO 1
Date_ 20161203
Time 19.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 781
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127714 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 ppm

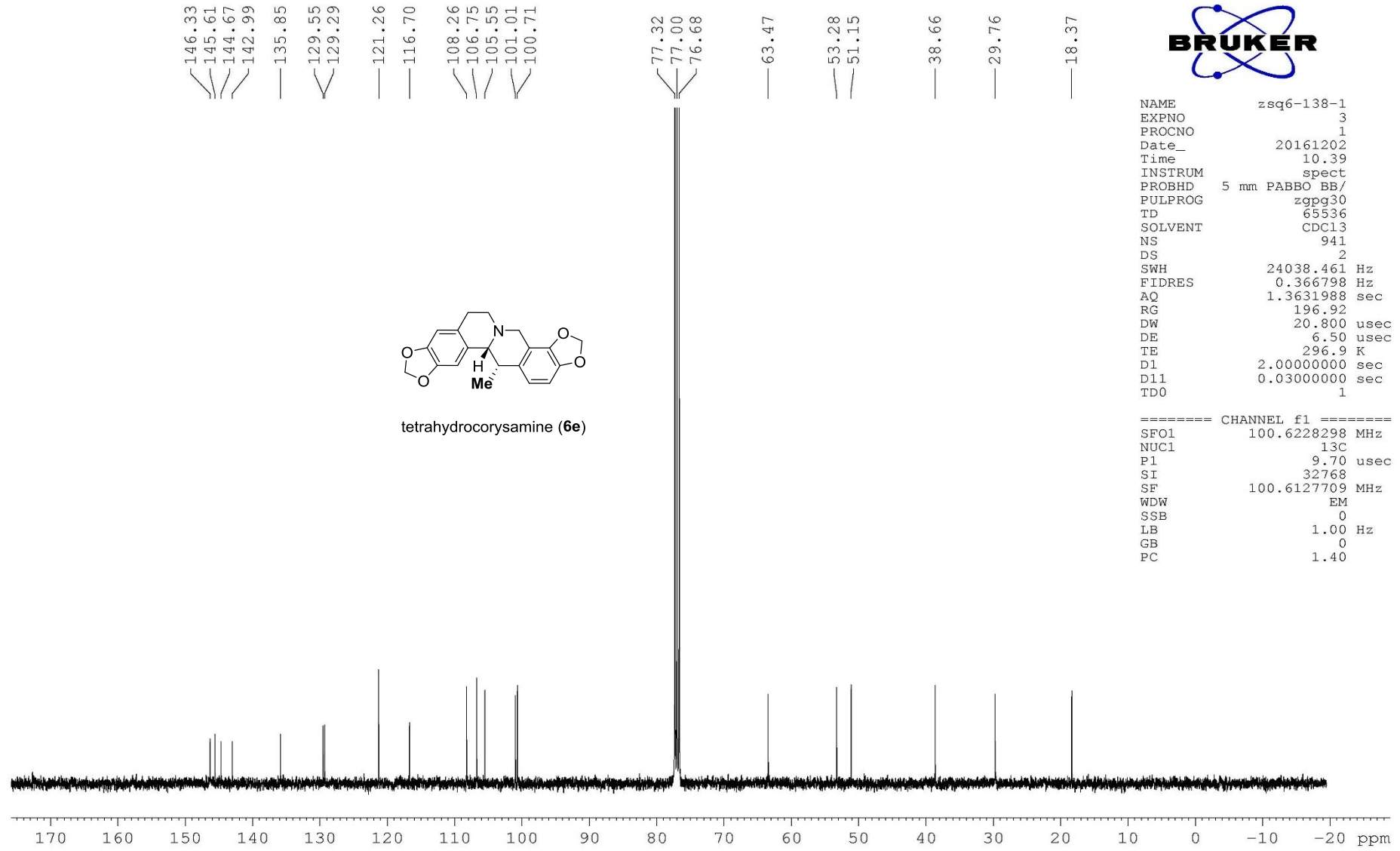


pseudocorydaline (**6d**)

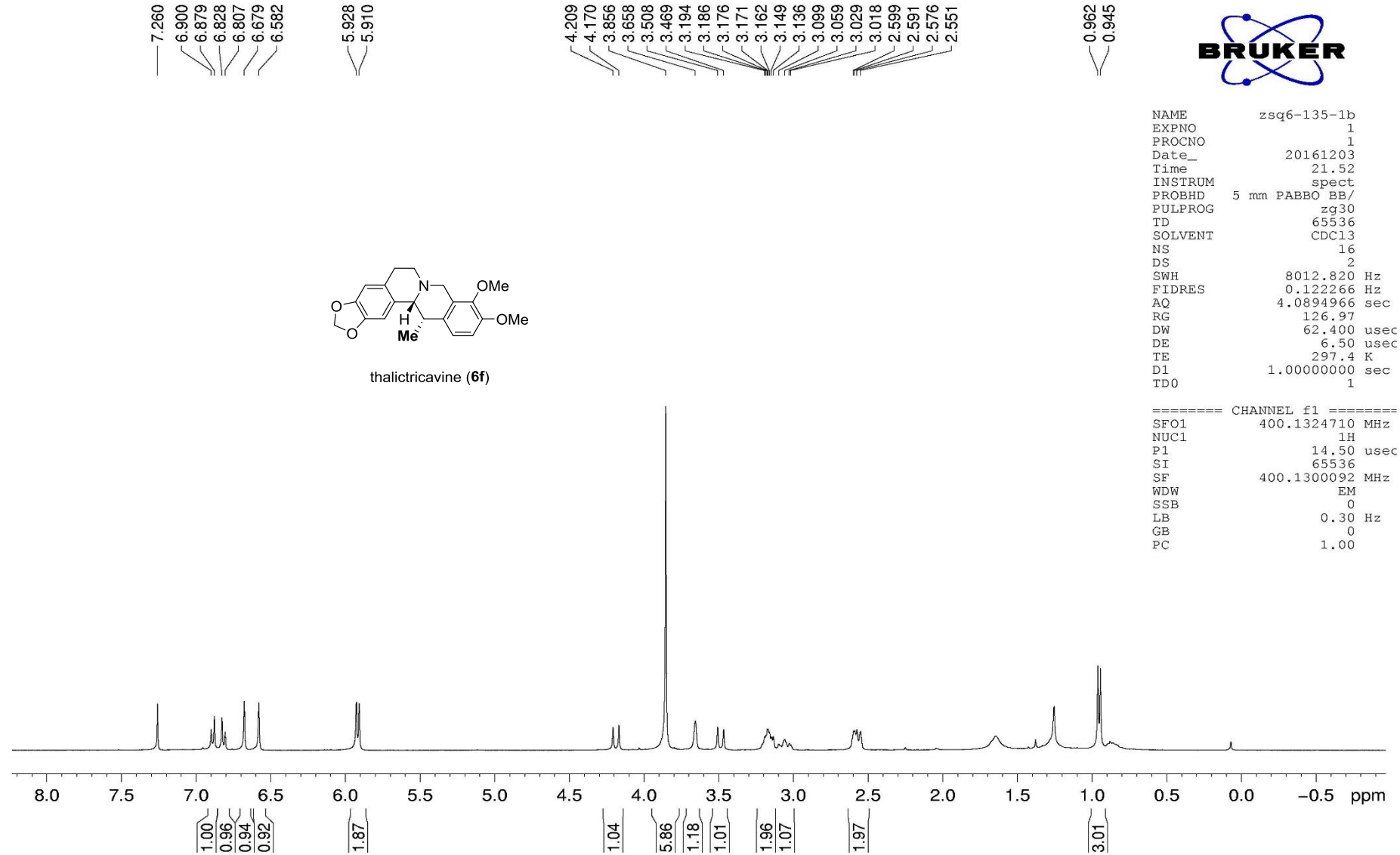

147.62
147.56
147.26
147.12
133.34
128.45
128.31
126.00
111.48
111.12
108.75
108.60
77.31
77.00
76.68
63.33
58.56
56.04
55.90
55.78
51.25
38.43
29.23
18.02
MeO
MeO
H
Me
N
OMe
OMe
pseudocorydaline (6d)
BRUKER
NAME zsq6-124-1a
EXPNO 2
PROCNO 1
Date_ 20161202
Time 13.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 650
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127722 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 ppm



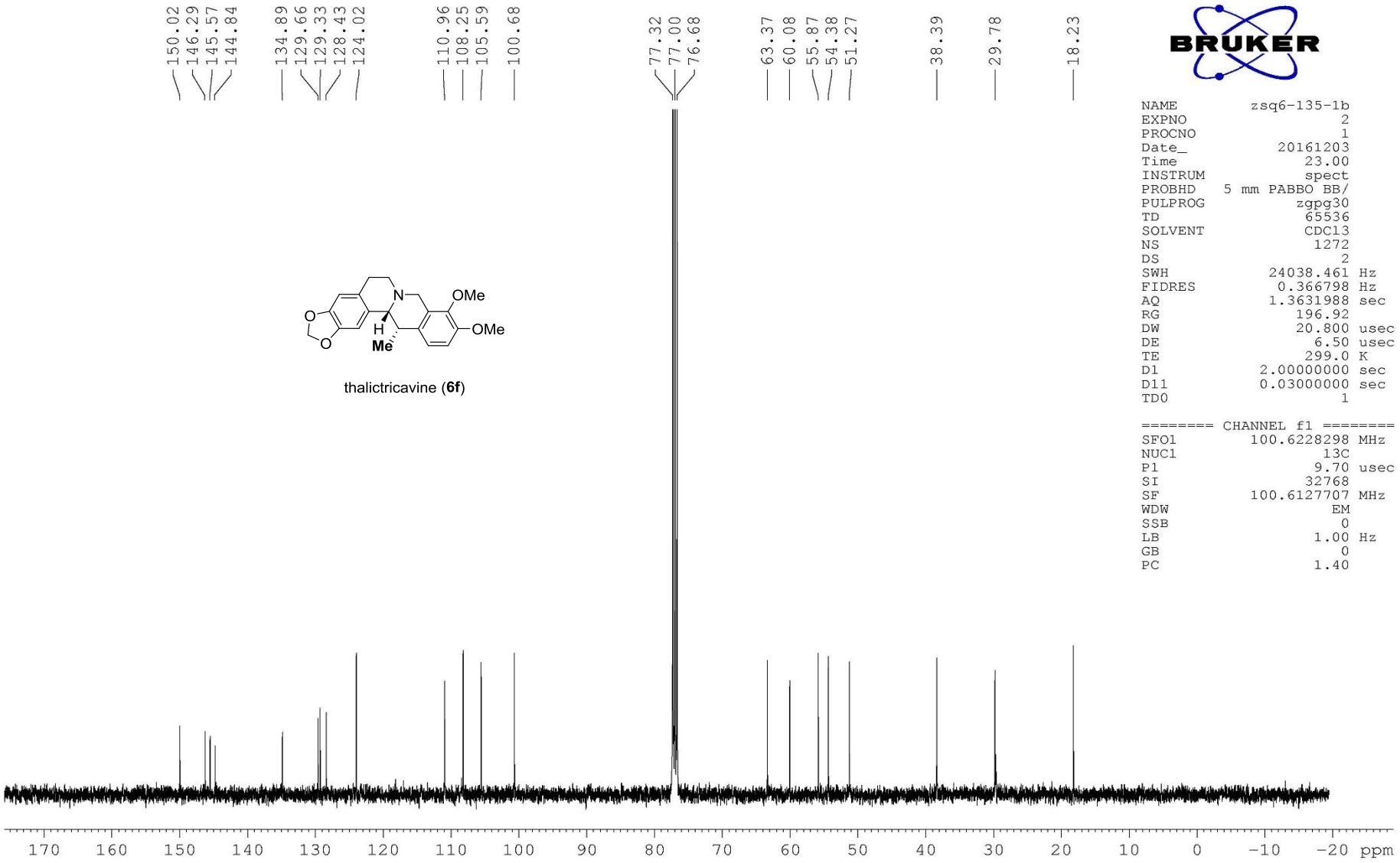
tetrahydrocorysamine (**6e**)



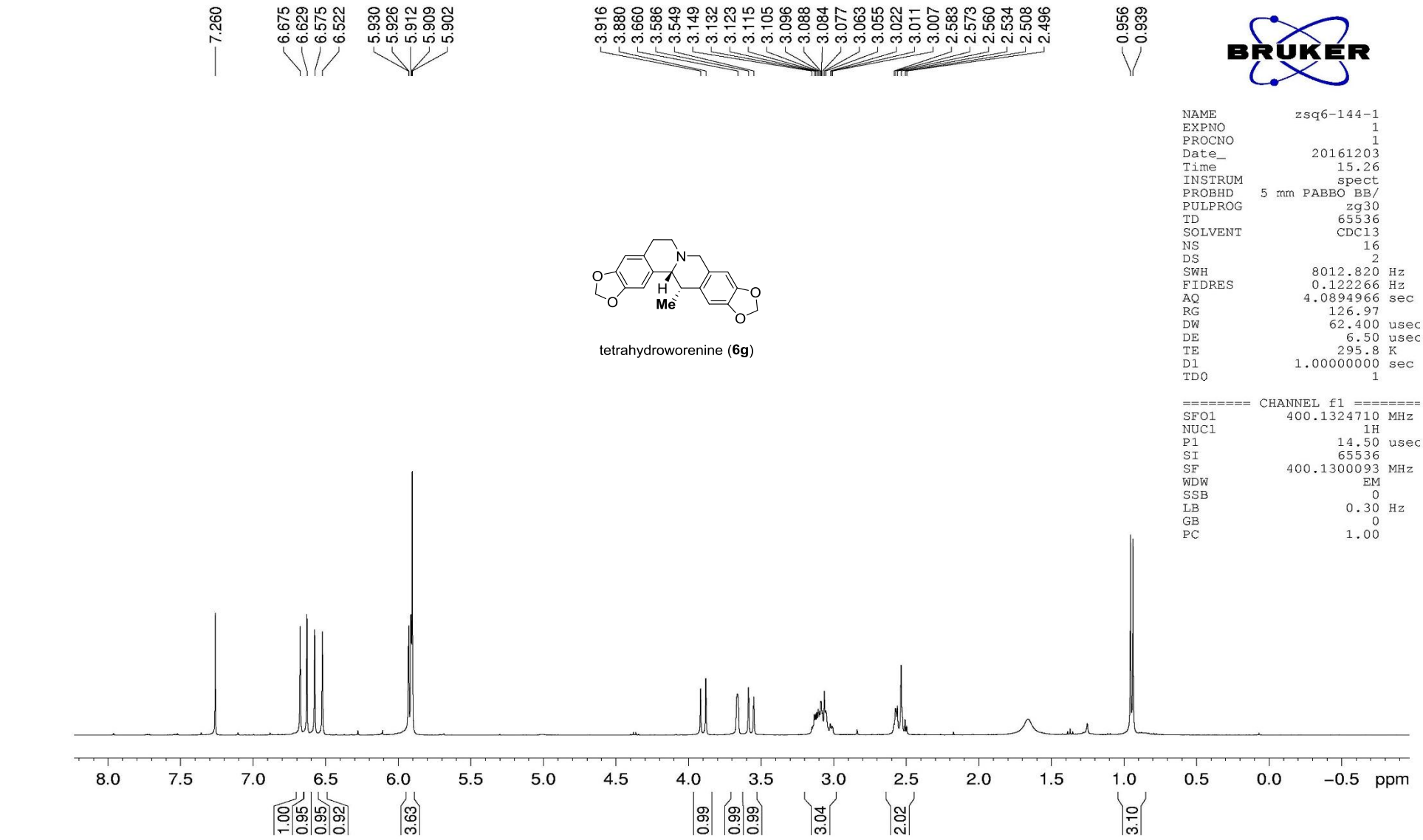
tetrahydrocorysamine (**6e**)



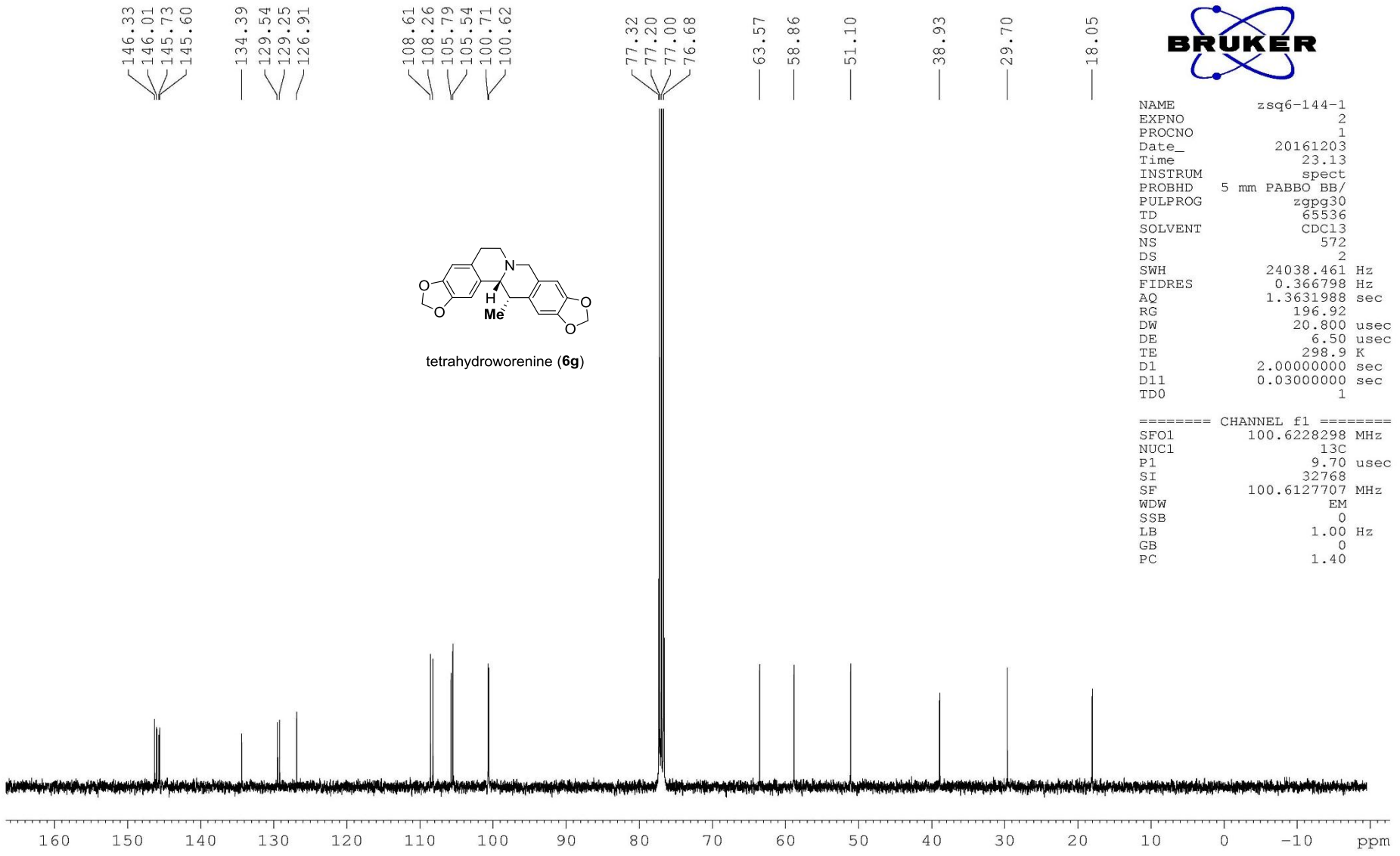
thalictricavine (**6f**)



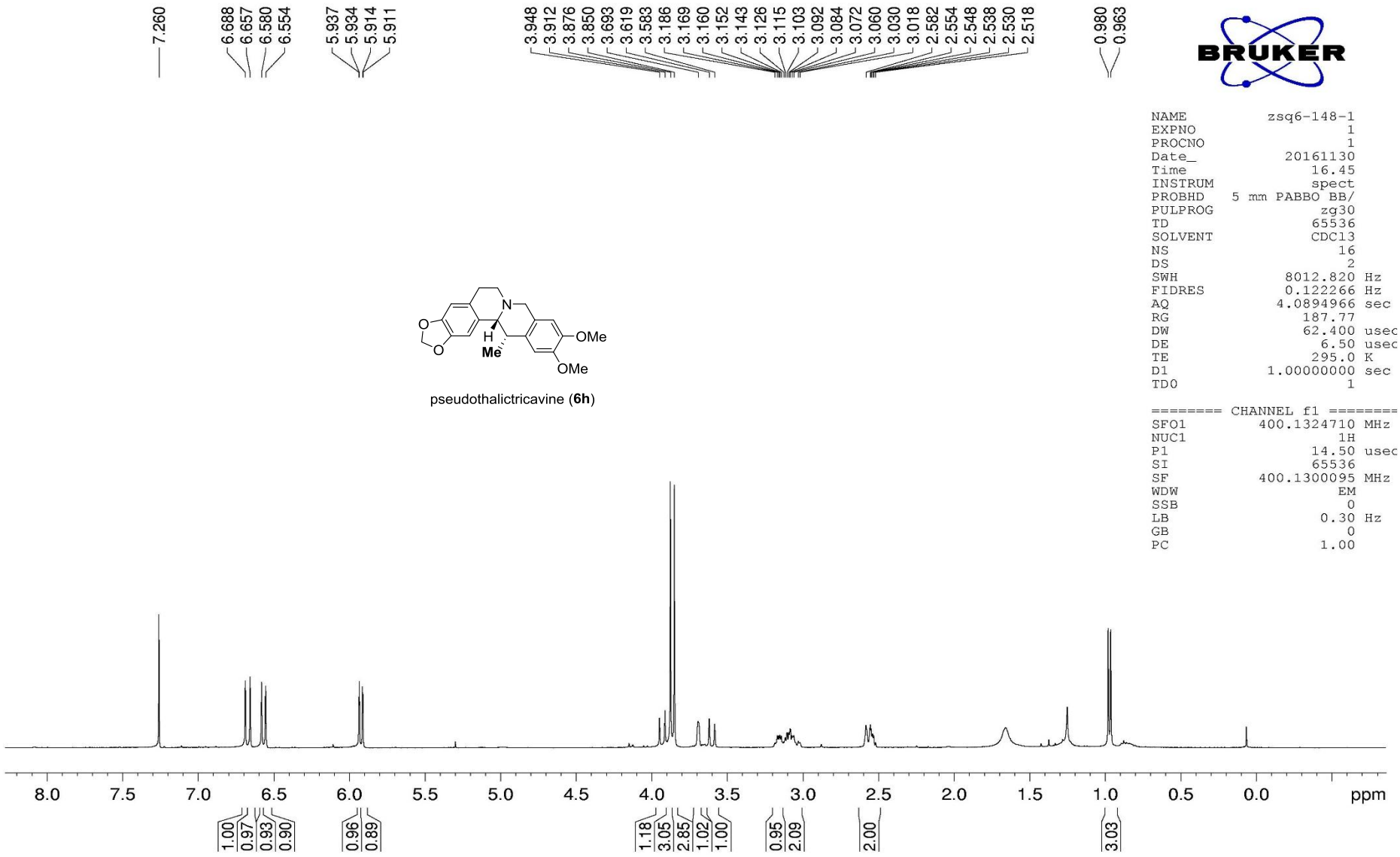
thalictricavine (**6f**)



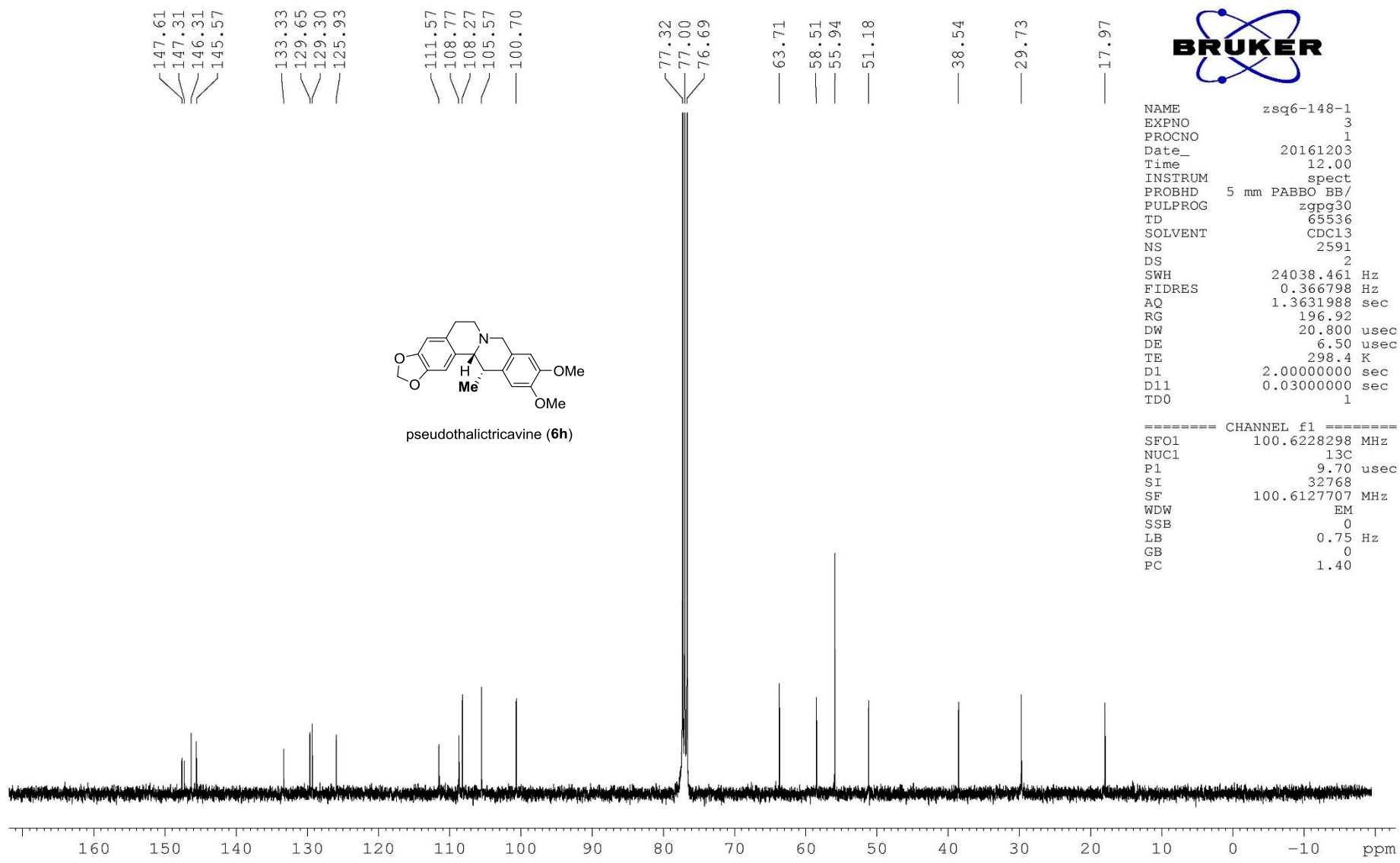
tetrahydroworenine (**6g**)



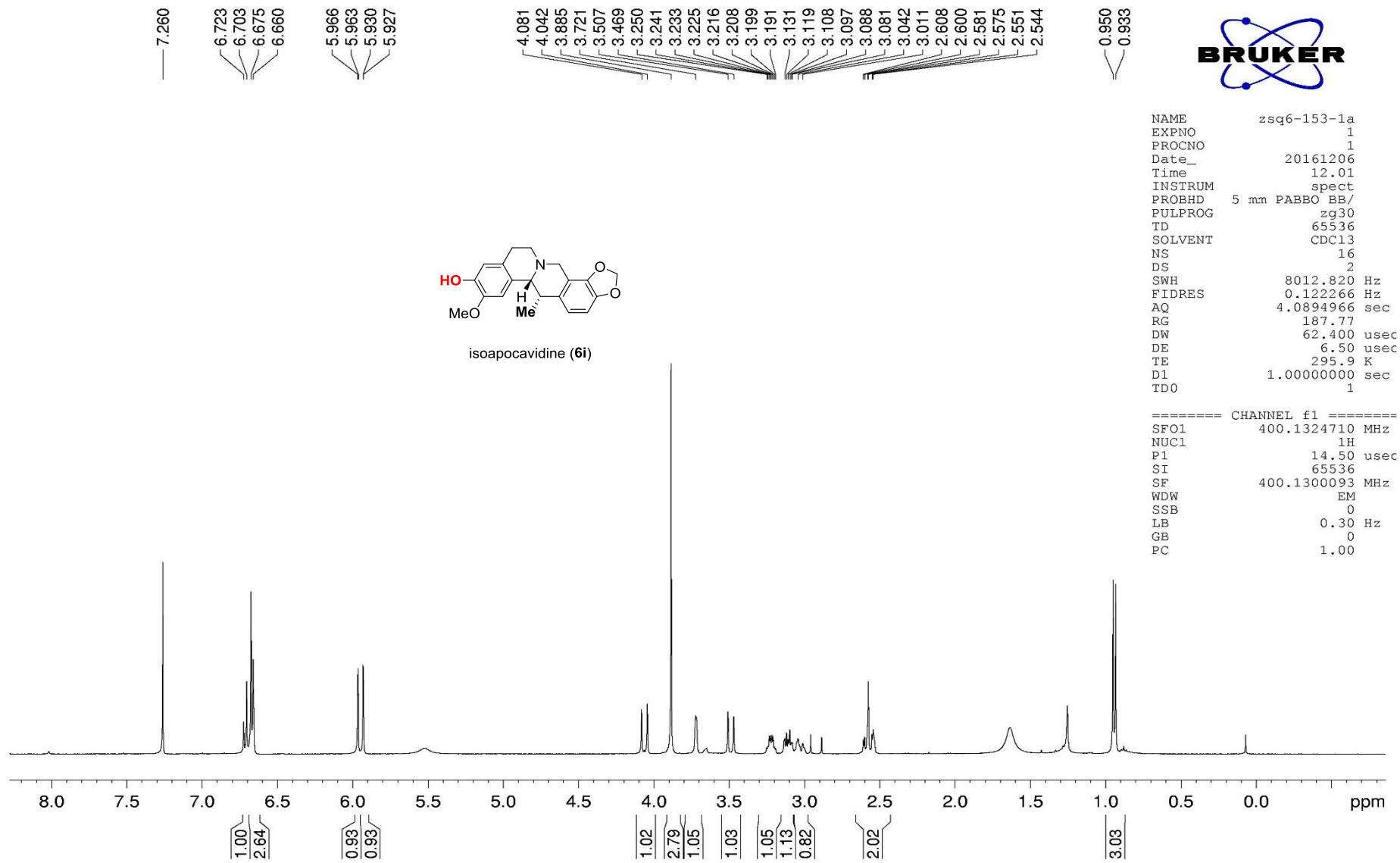
tetrahydroworenine (**6g**)



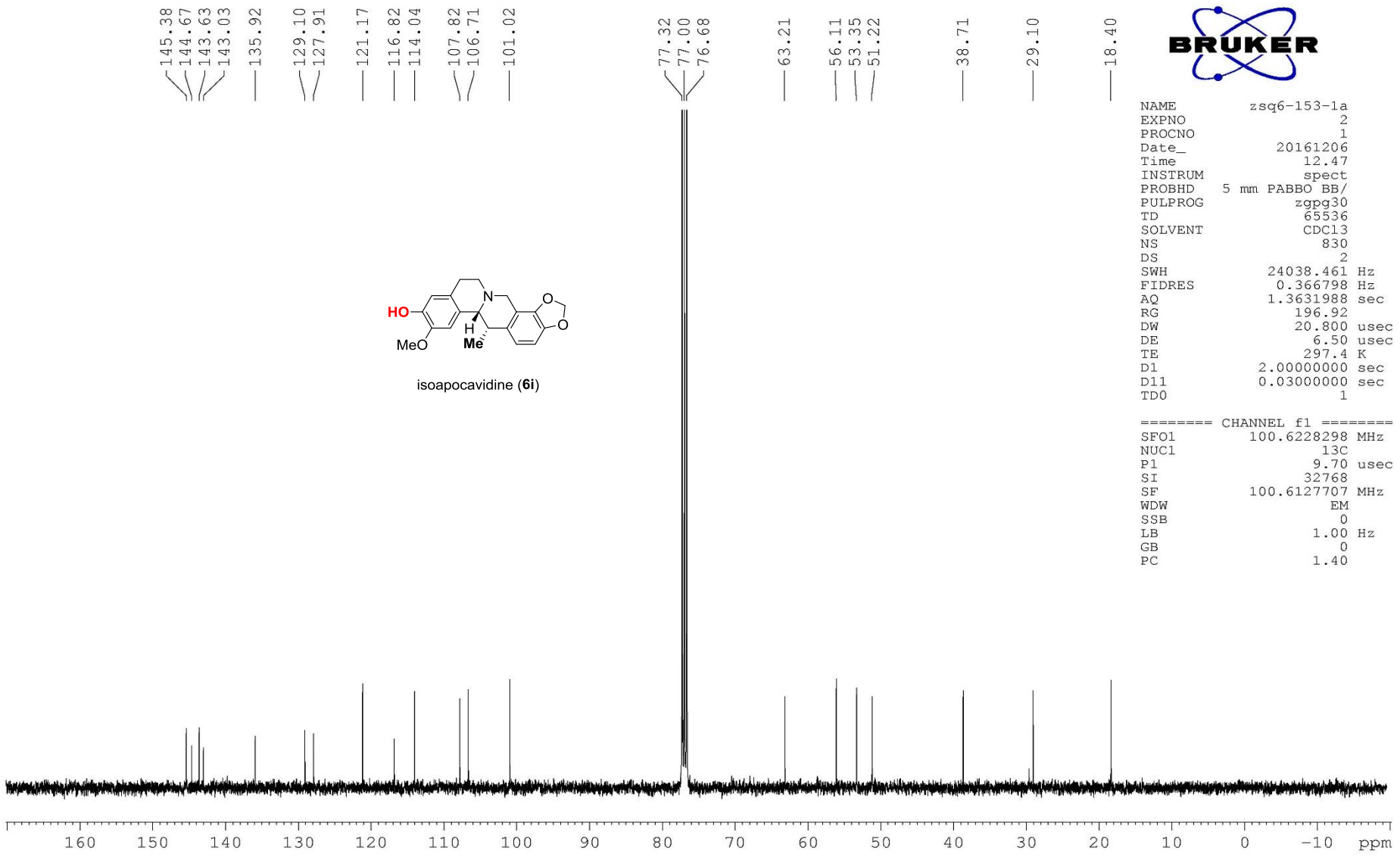
pseudothalictricavine (**6h**)

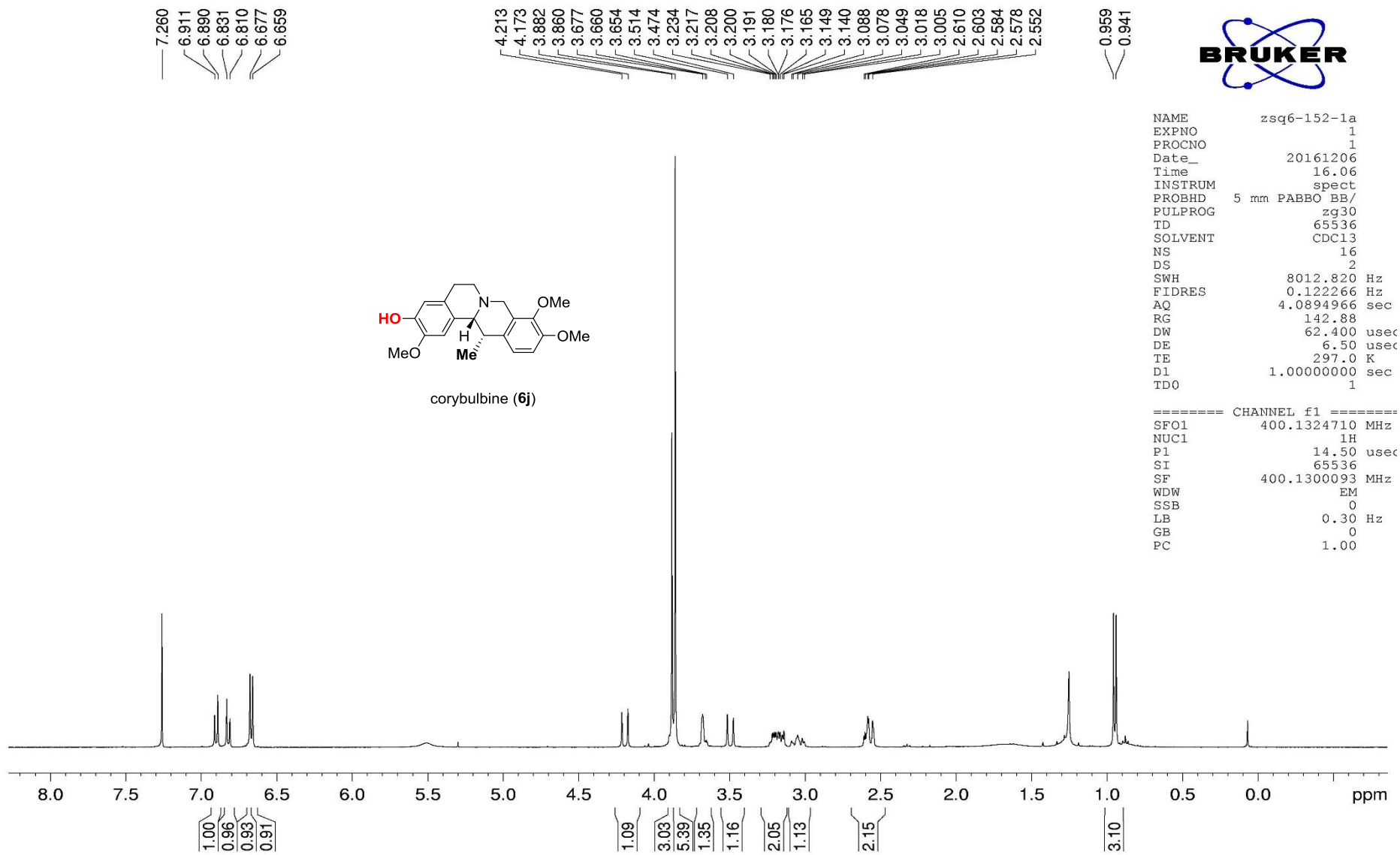


pseudothalictricavine (**6h**)

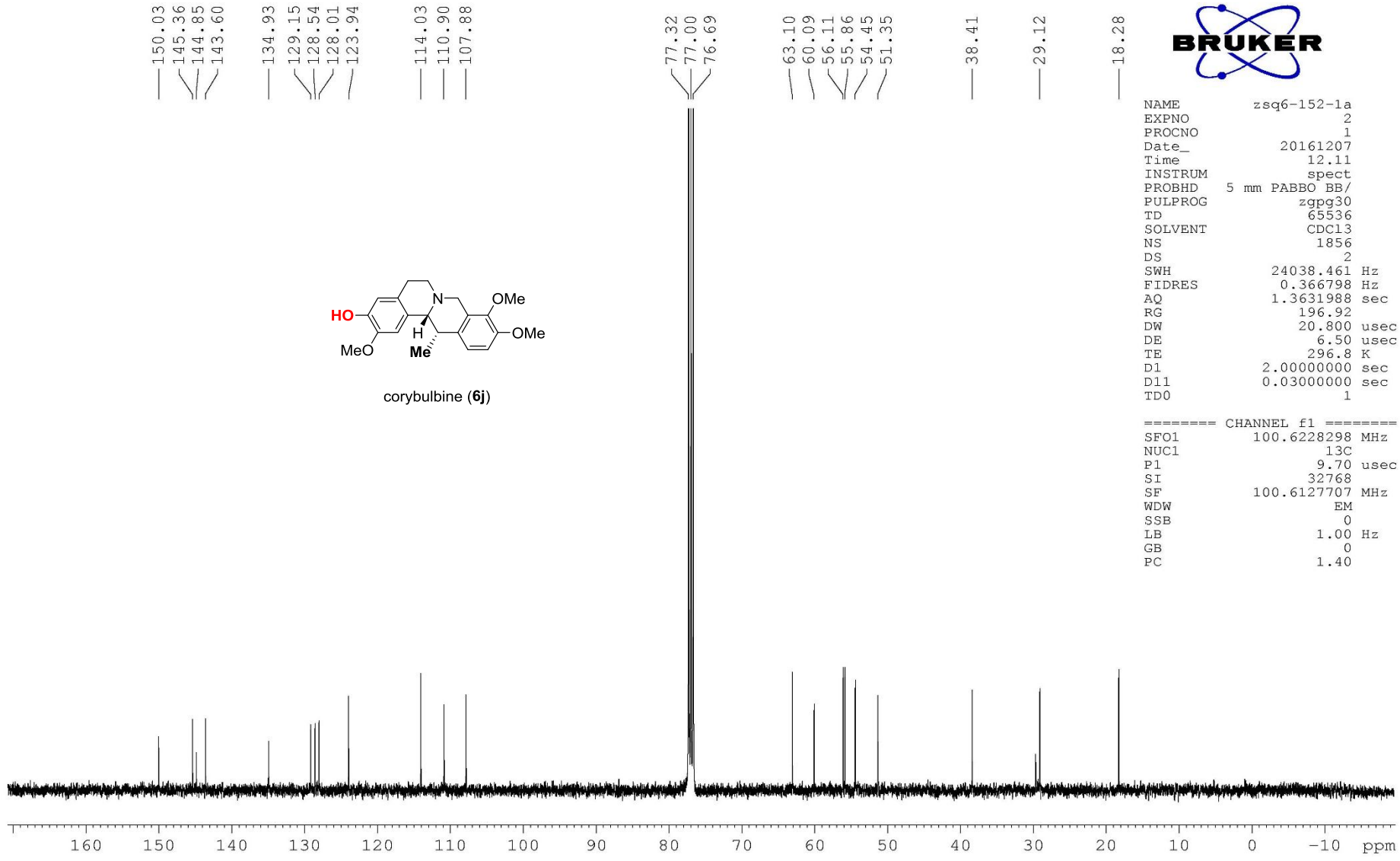


isoapocavidine (**6i**)

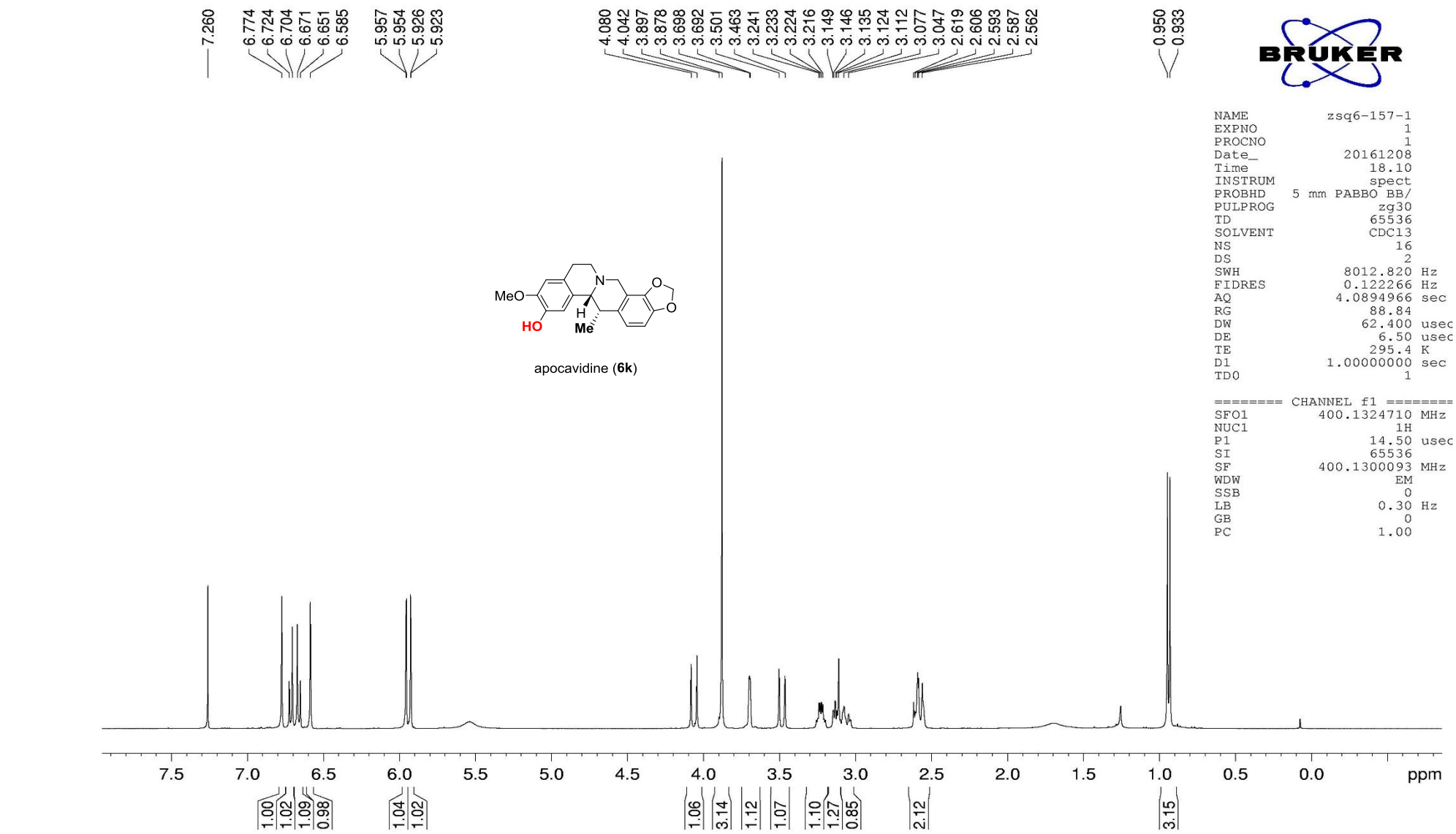

145.38
144.67
143.63
143.03
135.92
129.10
127.91
121.17
116.82
114.04
107.82
106.71
101.02
77.32
77.00
76.68
63.21
56.11
53.35
51.22
38.71
29.10
18.40
HO
MeO
N
O
O
H
Me
isoapocavidine (6i)
BRUKER
NAME zsq6-153-1a
EXPNO 2
PROCNO 1
Date_ 20161206
Time 12.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 830
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127707 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm

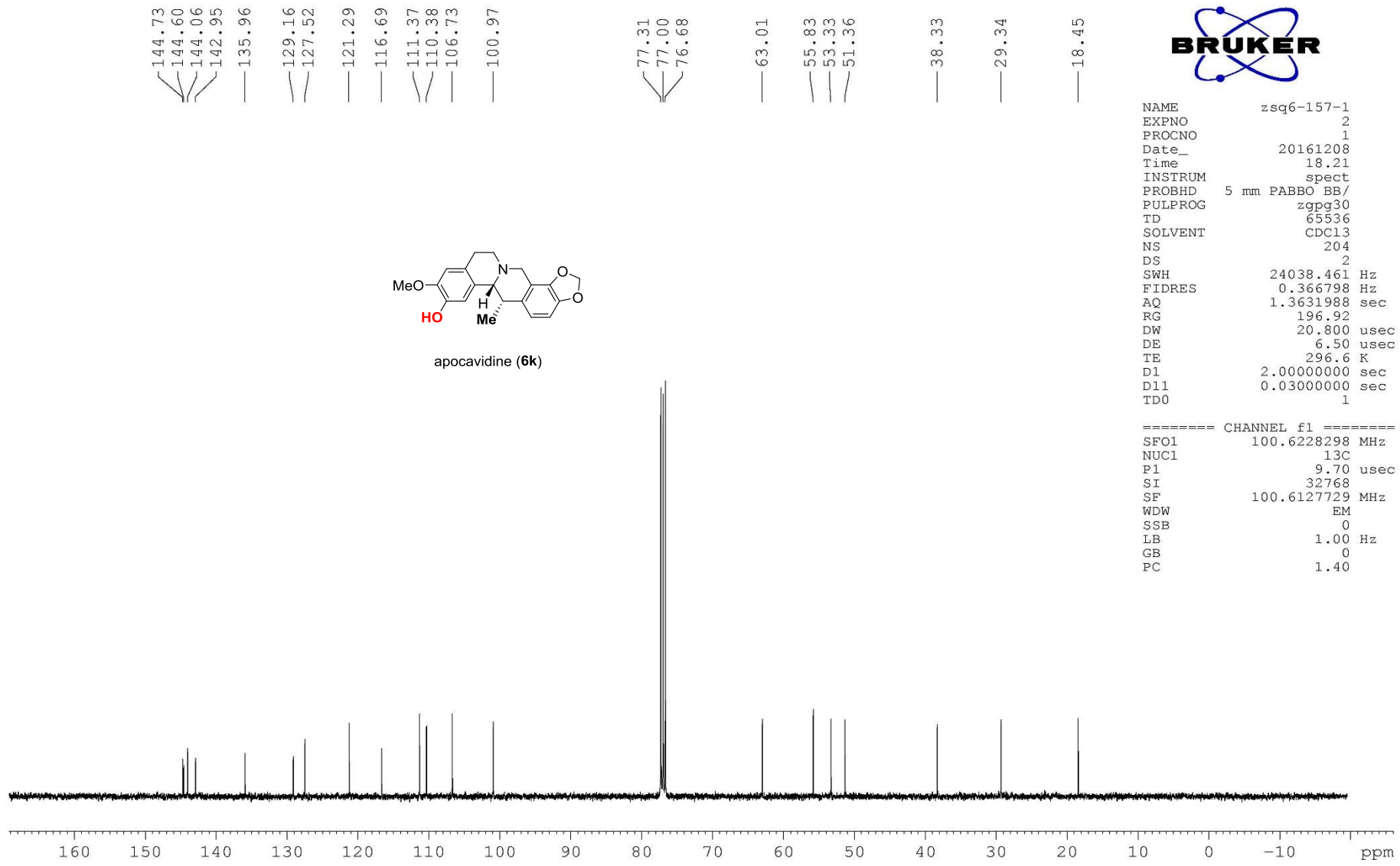


corybulbine (**6j**)

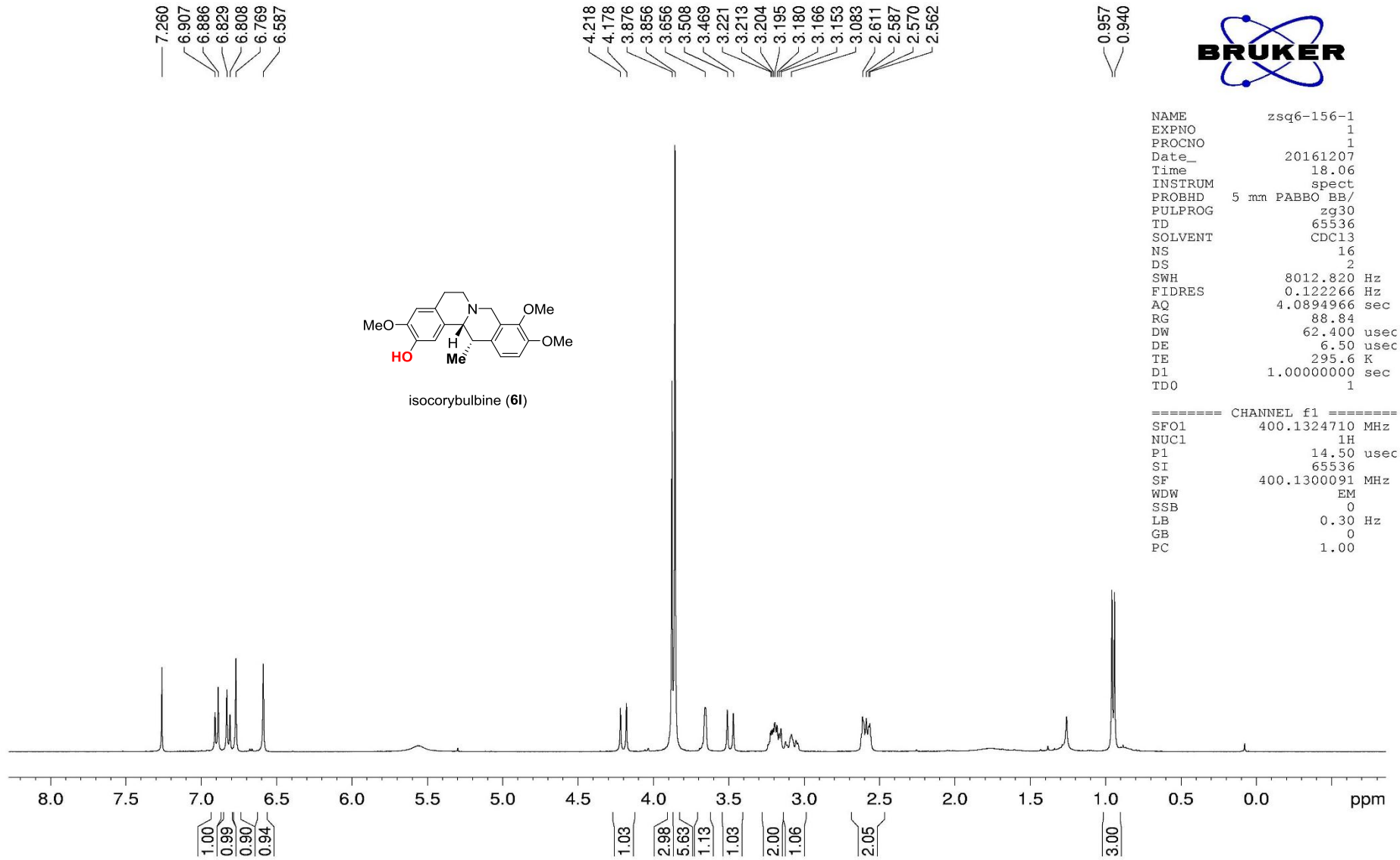


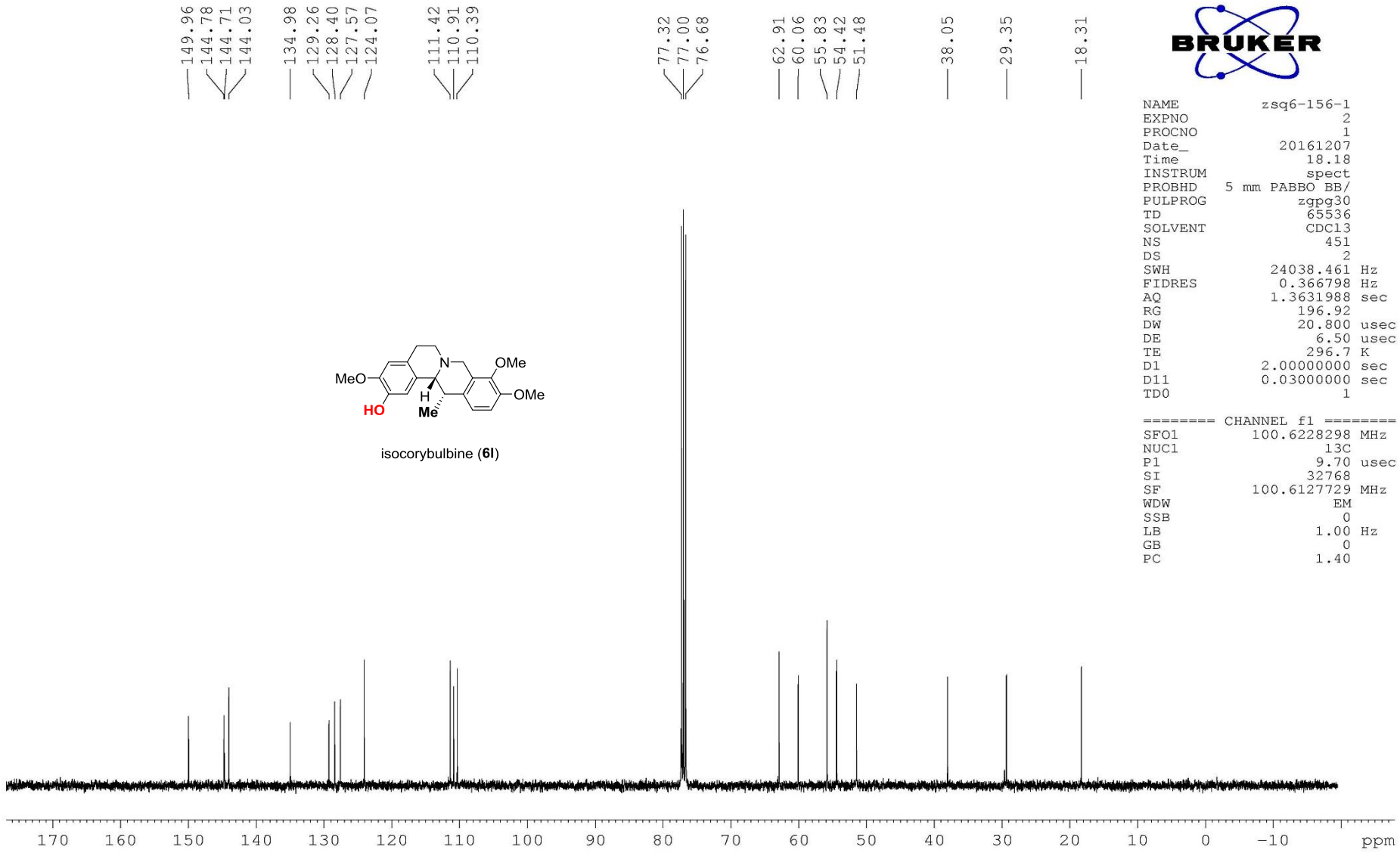
corybulbine (**6j**)


7.260
6.774
6.724
6.704
6.671
6.651
6.585
5.957
5.954
5.926
5.923
4.080
4.042
3.897
3.878
3.698
3.692
3.501
3.463
3.241
3.233
3.224
3.216
3.149
3.146
3.135
3.124
3.112
3.077
3.047
2.619
2.606
2.593
2.587
2.562
0.950
0.933
BRUKER
NAME zsq6-157-1
EXPNO 1
PROCNO 1
Date_ 20161208
Time 18.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 88.84
DW 62.400 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
HO
N
O
O
H
Me
apocavidine (6k)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.02
1.09
0.98
1.04
1.02
1.06
3.14
1.12
1.07
1.10
1.27
0.85
2.12
3.15

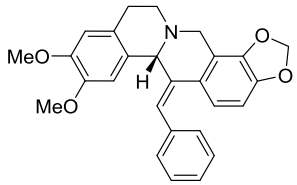


apocavidine (**6k**)


7.260
6.907
6.886
6.829
6.808
6.769
6.587
4.218
4.178
3.876
3.856
3.656
3.508
3.469
3.221
3.213
3.204
3.195
3.180
3.166
3.153
3.083
2.611
2.587
2.570
2.562
0.957
0.940
MeO
OMe
OMe
HO
H
Me
isocorybulbine (6l)
NAME zsq6-156-1
EXPNO 1
PROCNO 1
Date_ 20161207
Time 18.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 88.84
DW 62.400 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
0.99
0.90
0.94
1.03
2.98
5.63
1.13
1.03
2.00
1.06
2.05
3.00



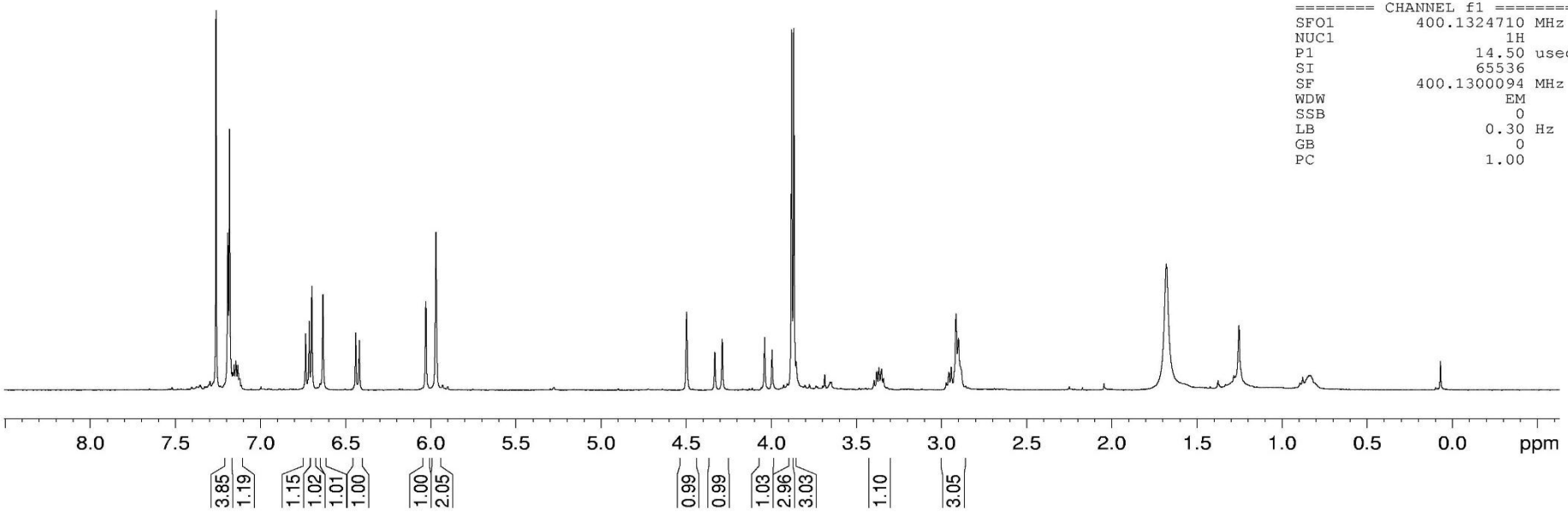
isocorybulbine (**6l**)

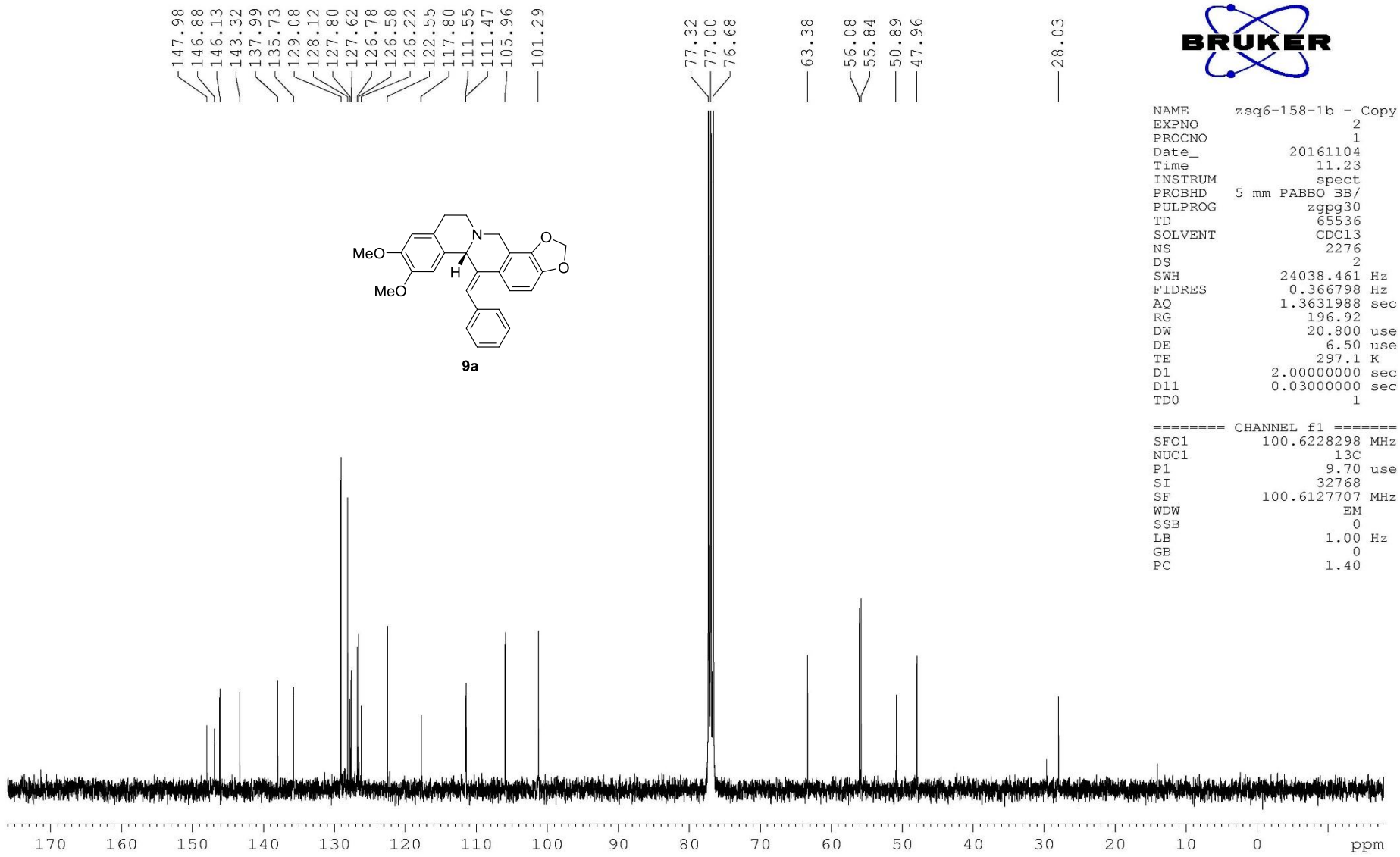


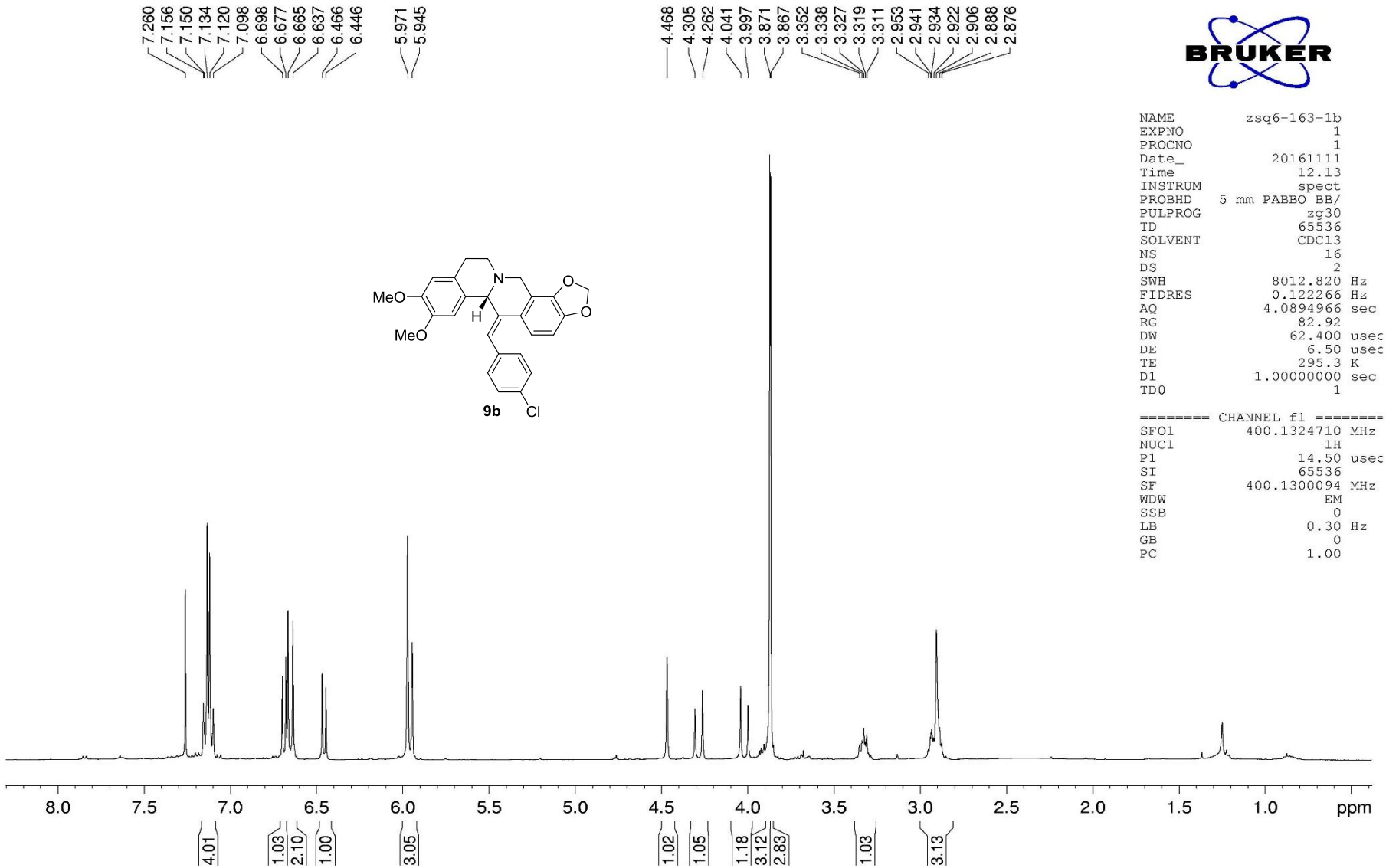
9a

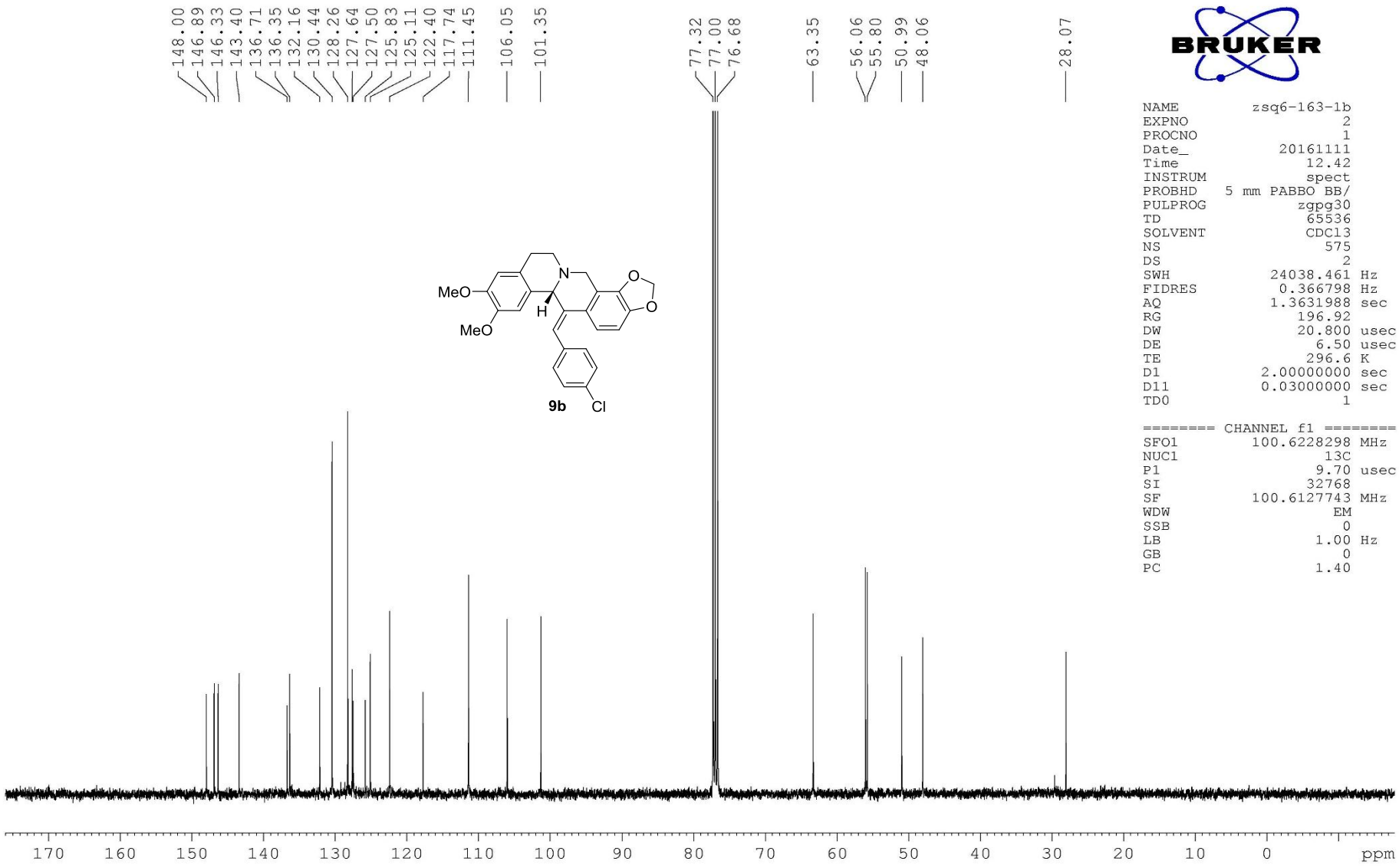
```
NAME        zsq6-158-1b - Copy
EXPNO                    1
PROCNO                   1
Date_             20161029
Time                 16.04
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8012.820 Hz
FIDRES               0.122266 Hz
AQ                4.0894966 sec
RG                   187.77
DW                   62.400 usec
DE                    6.50 usec
TE                   296.7 K
D1              1.00000000 sec
TD0                      1

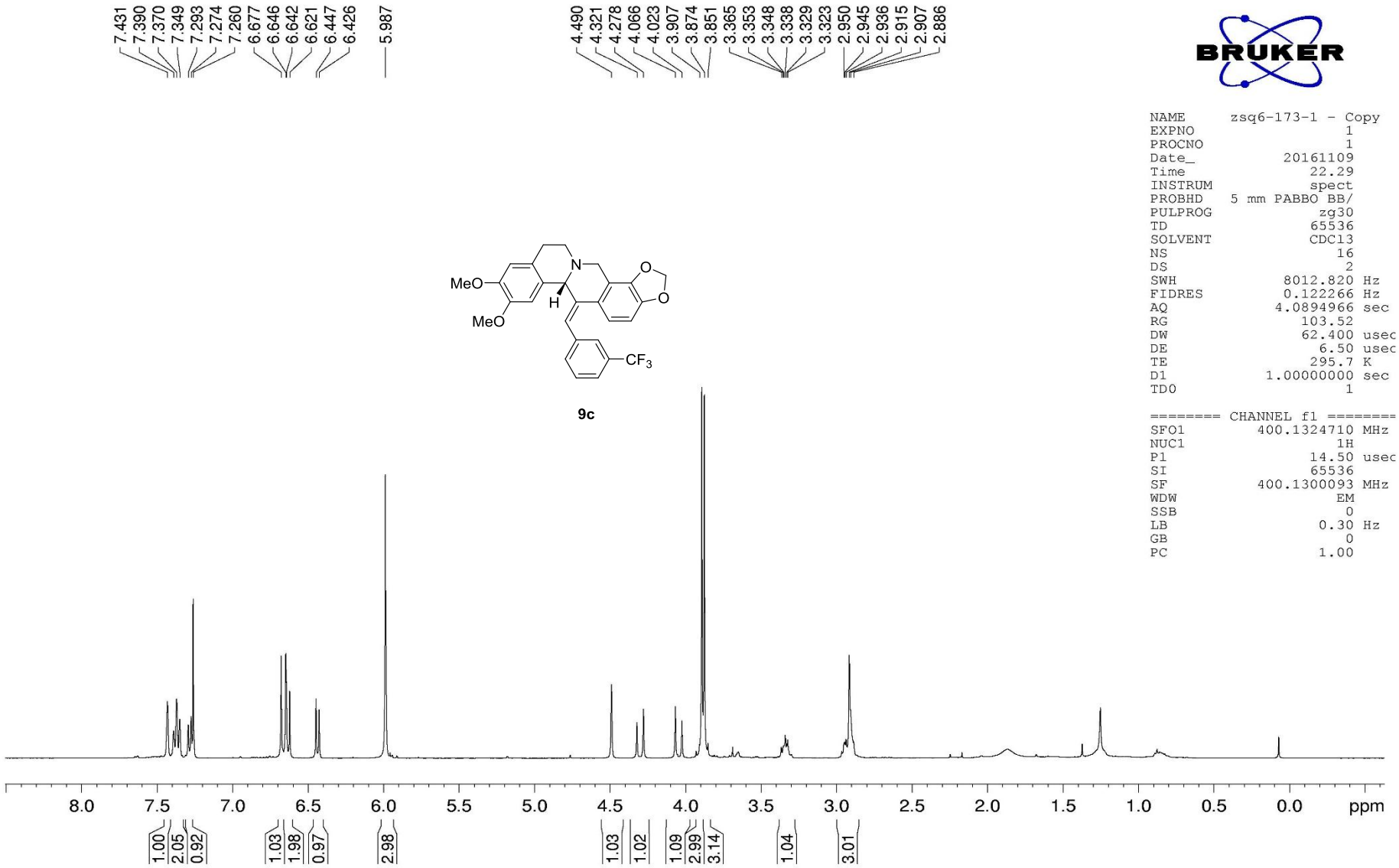
======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                     1H
P1                    14.50 usec
SI                    65536
SF              400.1300094 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

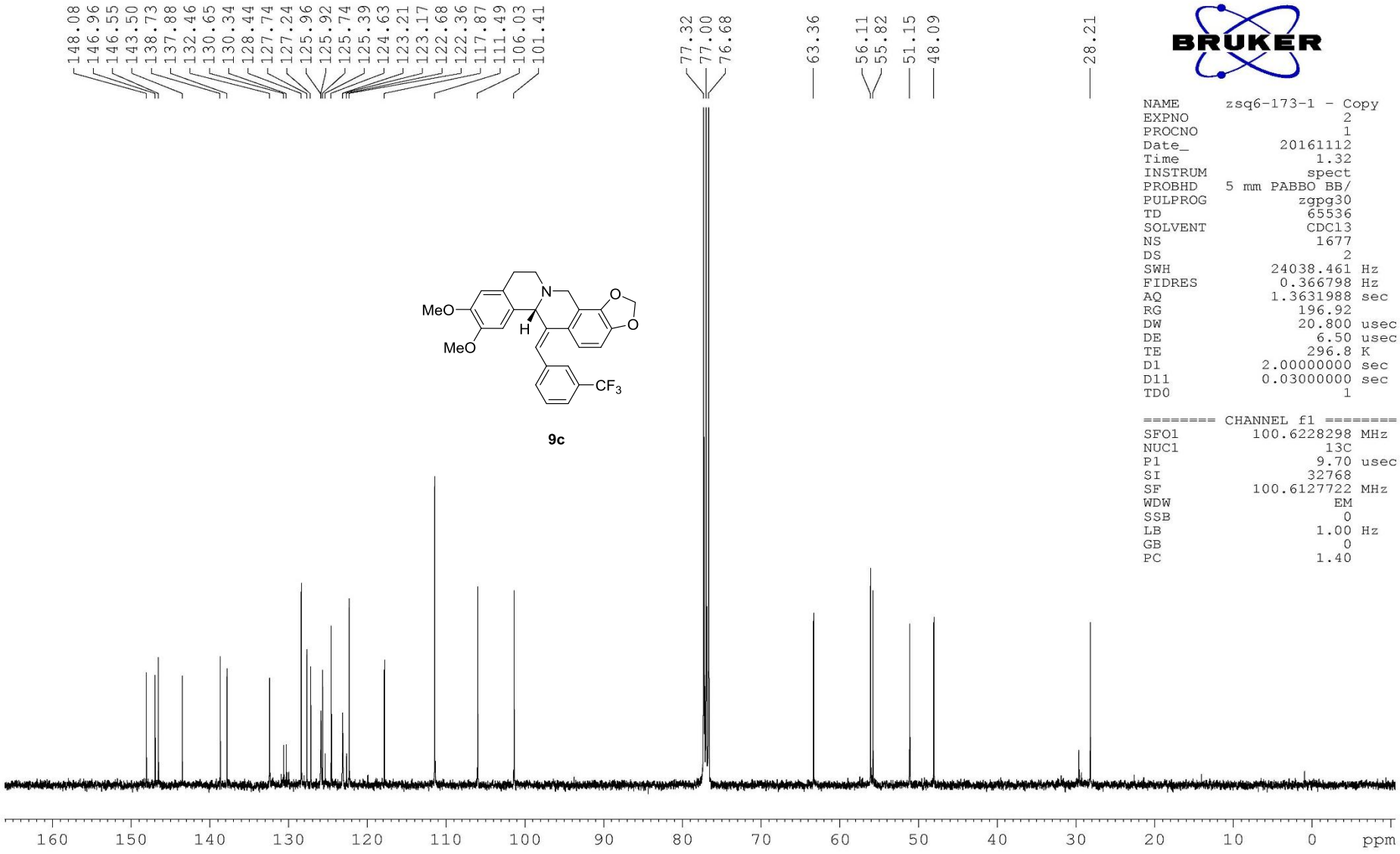


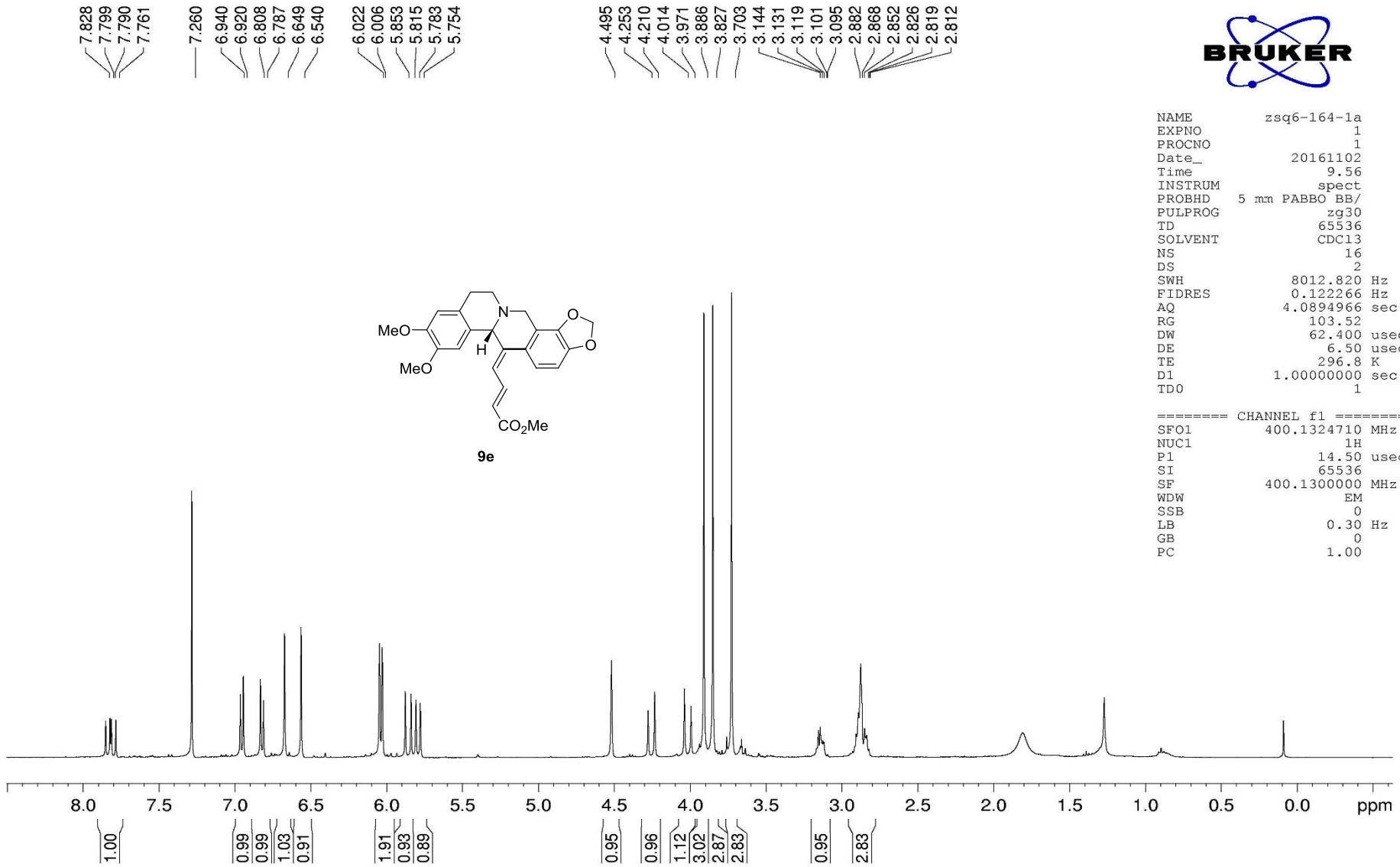

147.98
146.88
146.13
143.32
137.99
135.73
129.08
128.12
127.80
127.62
126.78
126.58
126.22
122.55
117.80
111.55
111.47
105.96
101.29
77.32
77.00
76.68
63.38
56.08
55.84
50.89
47.96
28.03
MeO
MeO
H
N
O
O
9a
BRUKER
NAME zsq6-158-1b - Copy
EXPNO 2
PROCNO 1
Date_ 20161104
Time 11.23
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2276
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 use
DE 6.50 use
TE 297.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 =======
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 use
SI 32768
SF 100.6127707 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

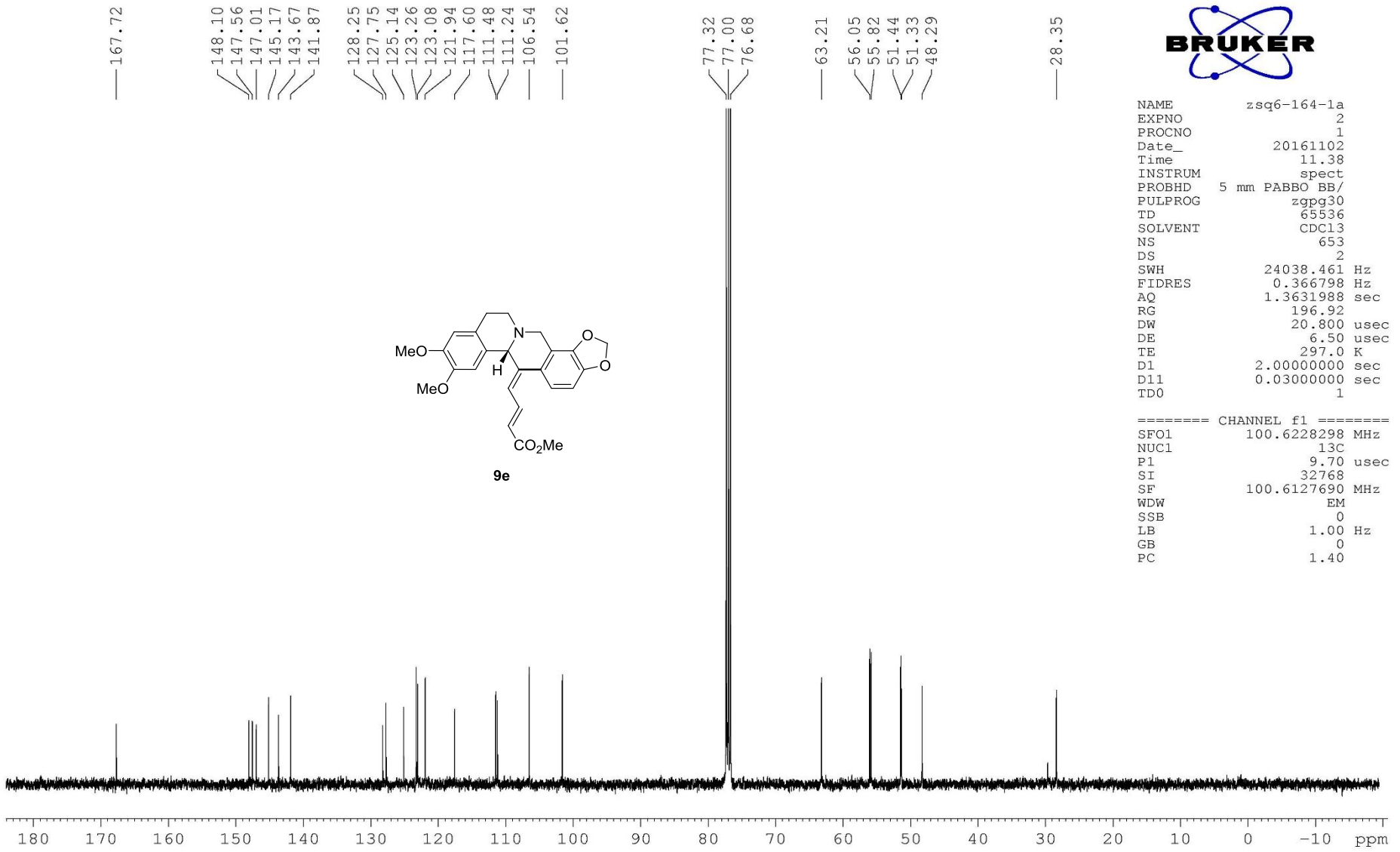

7.260
7.156
7.150
7.134
7.120
7.098
6.698
6.677
6.665
6.637
6.466
6.446
5.971
5.945
4.468
4.305
4.262
4.041
3.997
3.871
3.867
3.352
3.338
3.327
3.319
3.311
2.953
2.941
2.934
2.922
2.906
2.888
2.876
BRUKER
NAME zsq6-163-1b
EXPNO 1
PROCNO 1
Date_ 20161111
Time 12.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 82.92
DW 62.400 usec
DE 6.50 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
MeO
N
H
O
O
9b
Cl
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
4.01
1.03
2.10
1.00
3.05
1.02
1.05
1.18
3.12
2.83
1.03
3.13

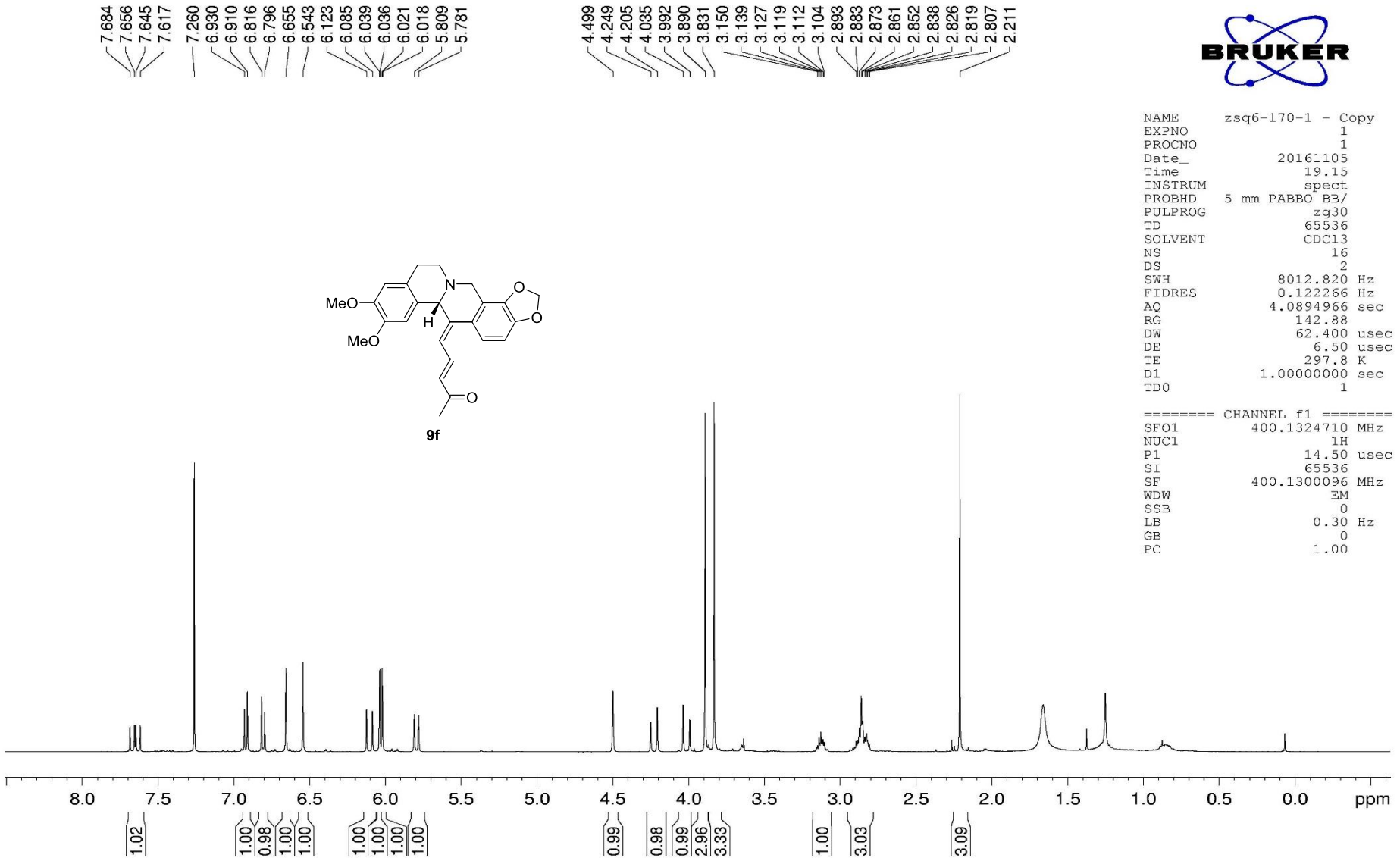

148.00
146.89
146.33
143.40
136.71
136.35
132.16
130.44
128.26
127.64
127.50
125.83
125.11
122.40
117.74
111.45
106.05
101.35
77.32
77.00
76.68
63.35
56.06
55.80
50.99
48.06
28.07
MeO
MeO
N
H
O
O
9b
Cl
BRUKER
NAME zsq6-163-1b
EXPNO 2
PROCNO 1
Date_ 20161111
Time 12.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 575
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127743 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

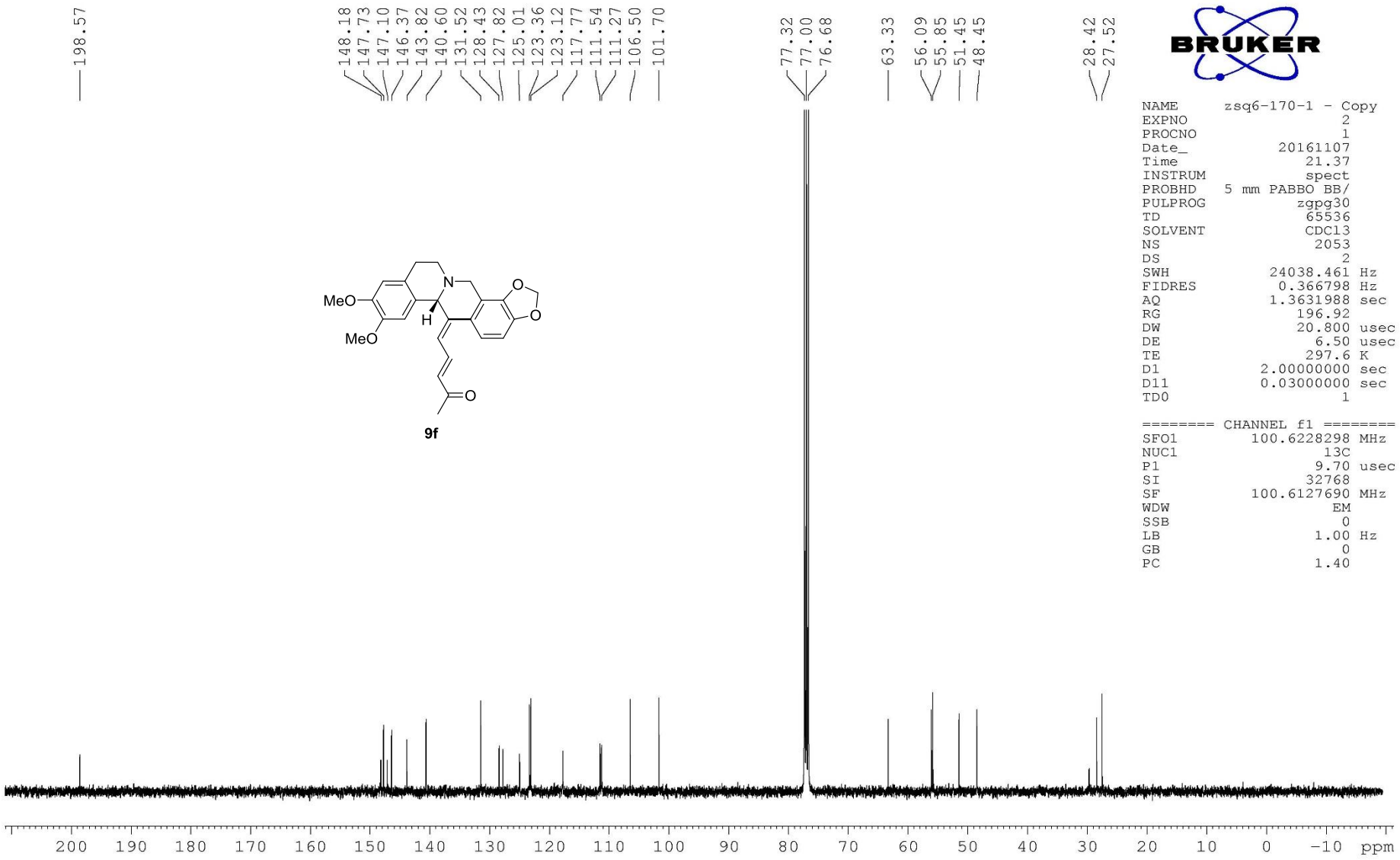

7.431
7.390
7.370
7.349
7.293
7.274
7.260
6.677
6.646
6.642
6.621
6.447
6.426
5.987
4.490
4.321
4.278
4.066
4.023
3.907
3.874
3.851
3.365
3.353
3.348
3.338
3.329
3.323
2.950
2.945
2.936
2.915
2.907
2.886
BRUKER
NAME zsq6-173-1 - Copy
EXPNO 1
PROCNO 1
Date_ 20161109
Time 22.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 103.52
DW 62.400 usec
DE 6.50 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
MeO
N
O
O
H
CF3
9c
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
2.05
0.92
1.03
1.98
0.97
2.98
1.03
1.02
1.09
2.99
3.14
1.04
3.01

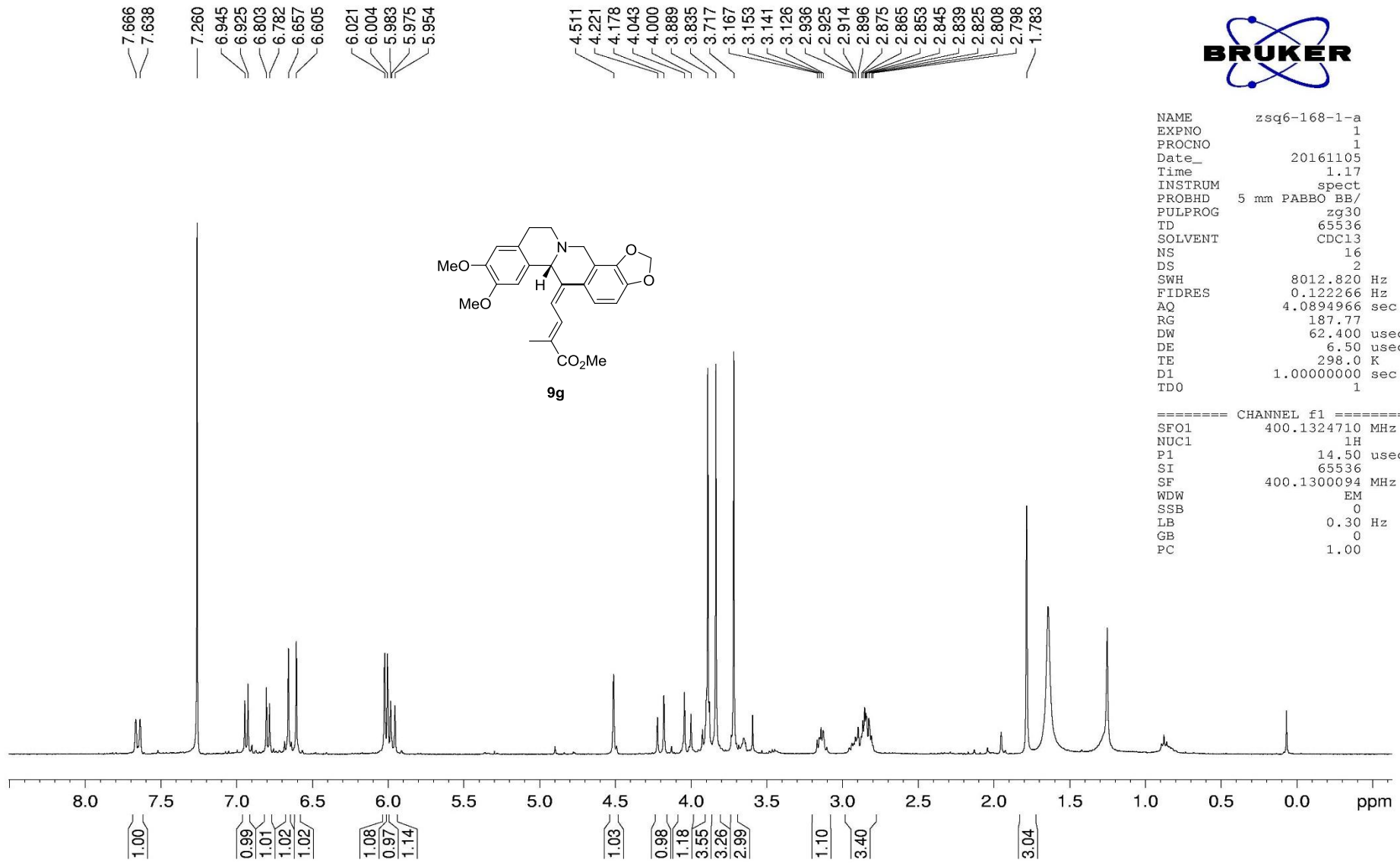

148.08
146.96
146.55
143.50
138.73
137.88
132.46
130.65
130.34
128.44
127.74
127.24
125.96
125.92
125.74
125.39
124.63
123.21
123.17
122.68
122.36
117.87
111.49
106.03
101.41
77.32
77.00
76.68
63.36
56.11
55.82
51.15
48.09
28.21
MeO
MeO
N
H
O
O
CF3
9c
NAME zsq6-173-1 - Copy
EXPNO 2
PROCNO 1
Date_ 20161112
Time 1.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1677
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127722 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

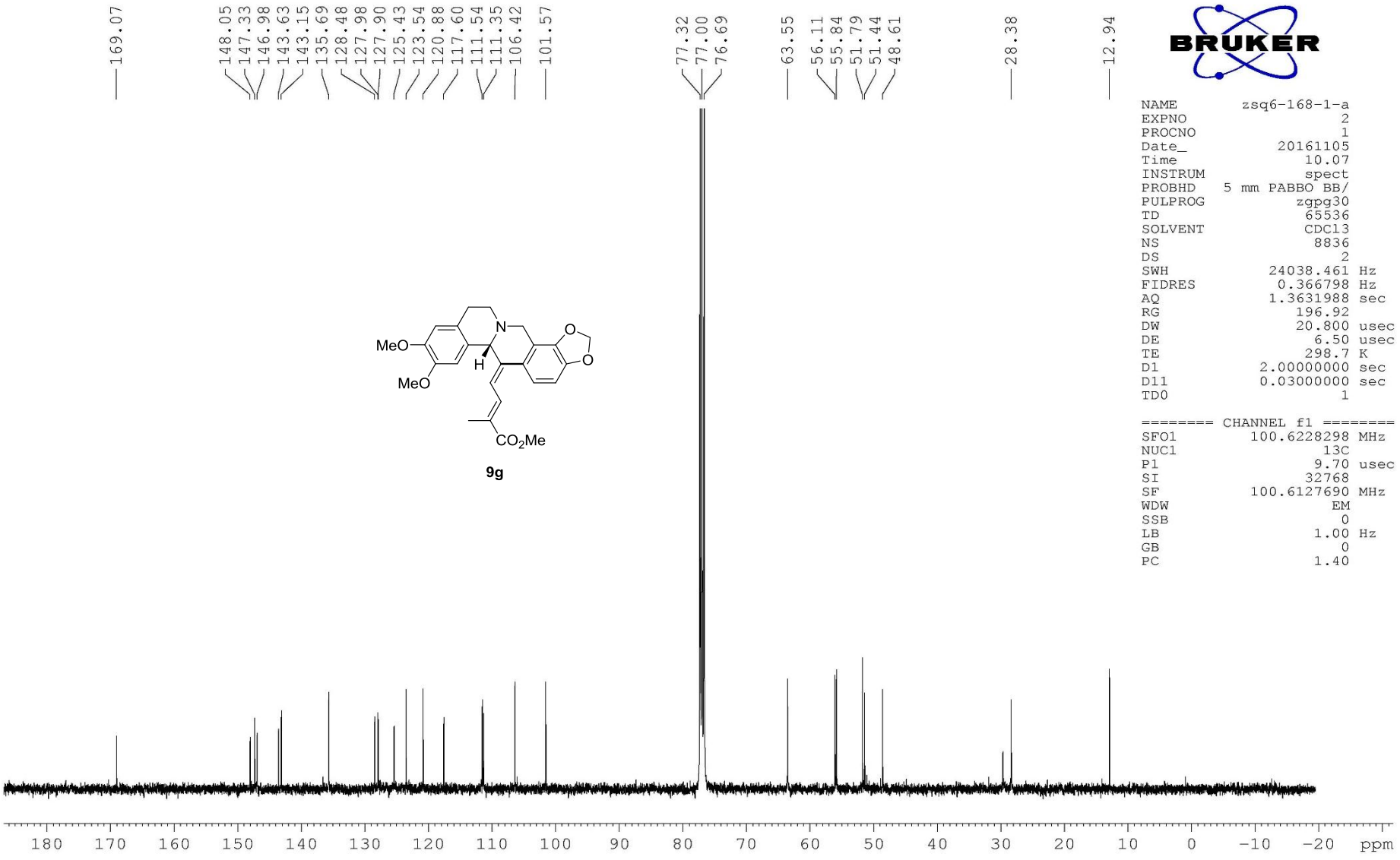

7.828
7.799
7.790
7.761
7.260
6.940
6.920
6.808
6.787
6.649
6.540
6.022
6.006
5.853
5.815
5.783
5.754
4.495
4.253
4.210
4.014
3.971
3.886
3.827
3.703
3.144
3.131
3.119
3.101
3.095
2.882
2.868
2.852
2.826
2.819
2.812
BRUKER
NAME zsq6-164-1a
EXPNO 1
PROCNO 1
Date_ 20161102
Time 9.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 103.52
DW 62.400 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
MeO
N
O
O
H
CO2Me
9e
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
0.99
0.99
1.03
0.91
1.91
0.93
0.89
0.95
0.96
1.12
3.02
2.87
2.83
0.95
2.83

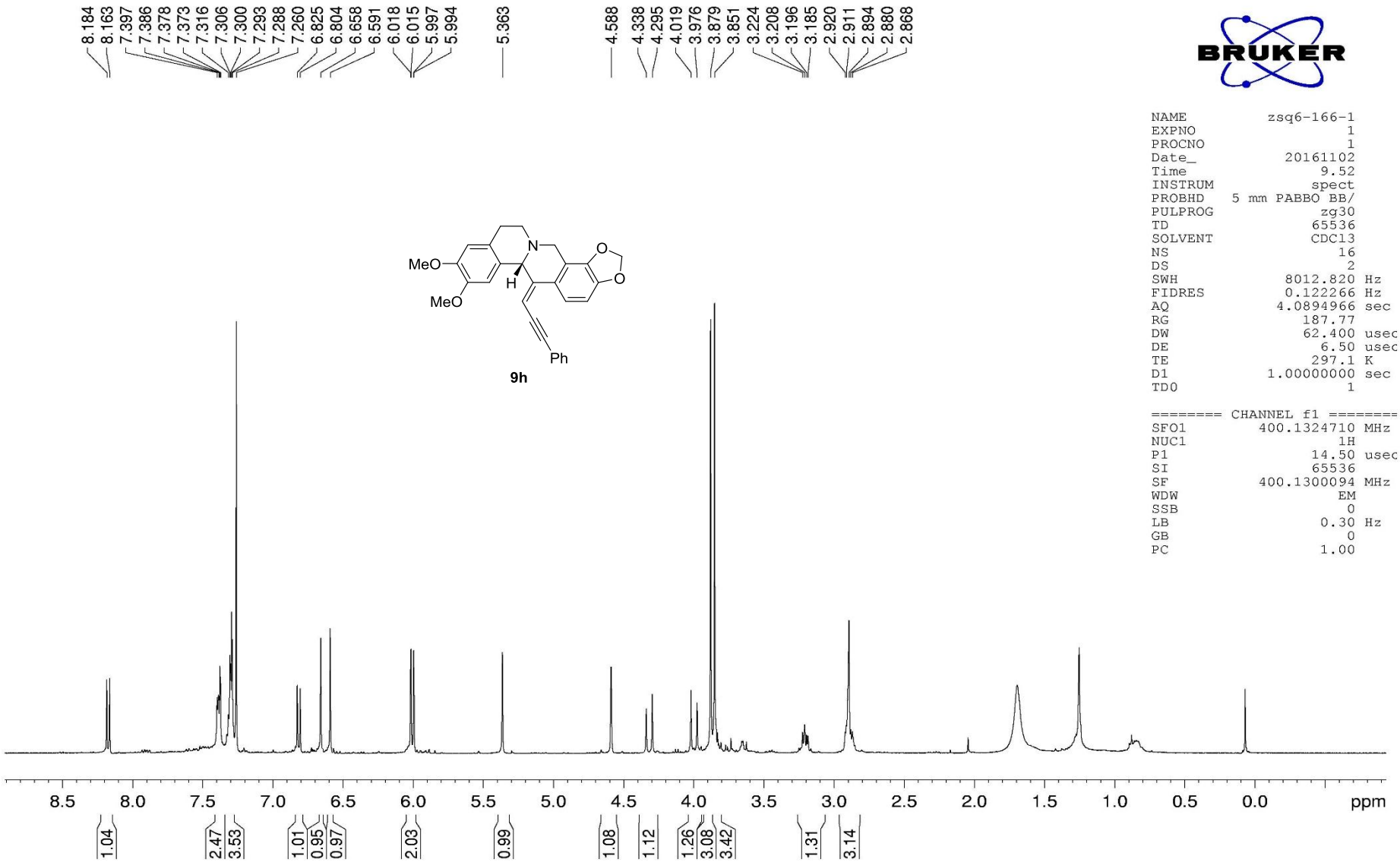

167.72
148.10
147.56
147.01
145.17
143.67
141.87
128.25
127.75
125.14
123.26
123.08
121.94
117.60
111.48
111.24
106.54
101.62
77.32
77.00
76.68
63.21
56.05
55.82
51.44
51.33
48.29
28.35
MeO
MeO
N
H
O
O
CO2Me
9e
BRUKER
NAME zsq6-164-1a
EXPNO 2
PROCNO 1
Date_ 20161102
Time 11.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 653
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm

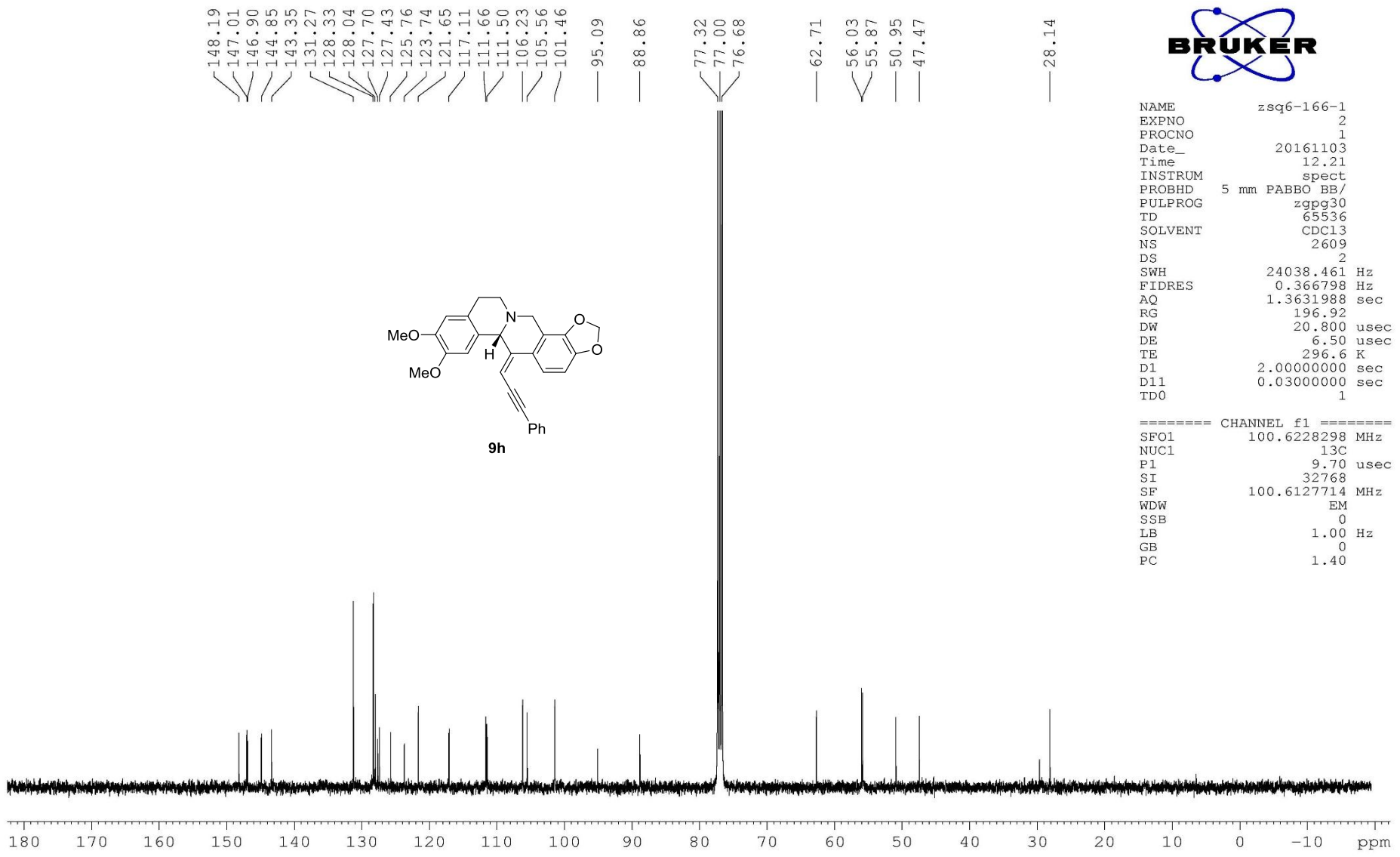

7.684
7.656
7.645
7.617
7.260
6.930
6.910
6.816
6.796
6.655
6.543
6.123
6.085
6.039
6.036
6.021
6.018
5.809
5.781
4.499
4.249
4.205
4.035
3.992
3.890
3.831
3.150
3.139
3.127
3.119
3.112
3.104
2.893
2.883
2.873
2.861
2.852
2.838
2.826
2.819
2.807
2.211
BRUKER
NAME zsq6-170-1 - Copy
EXPNO 1
PROCNO 1
Date_ 20161105
Time 19.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 142.88
DW 62.400 usec
DE 6.50 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
MeO
N
H
O
O
O
9f
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.02
1.00
0.98
1.00
1.00
1.00
1.00
1.00
1.00
0.99
0.98
0.99
2.96
3.33
1.00
3.03
3.09

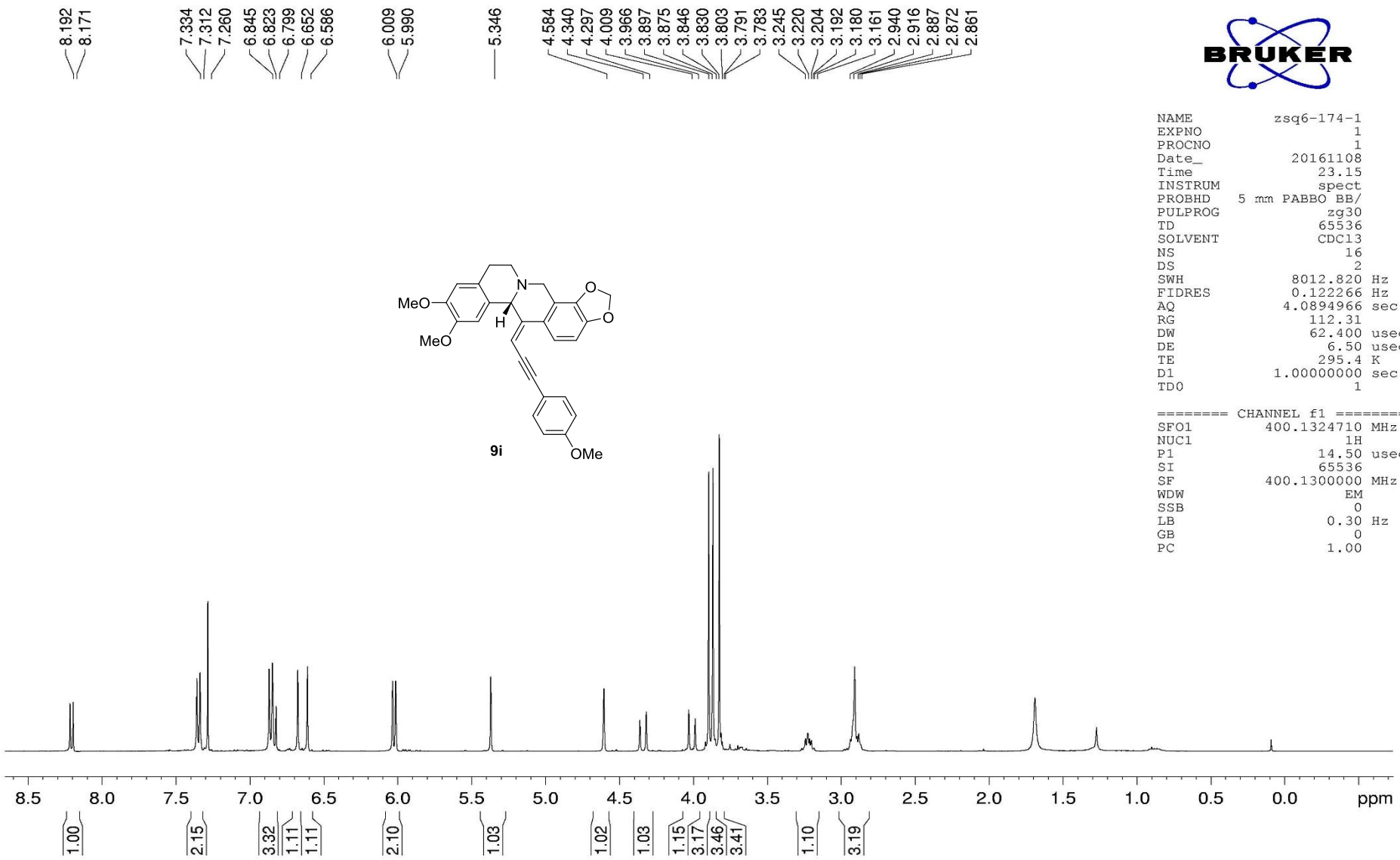

198.57
148.18
147.73
147.10
146.37
143.82
140.60
131.52
128.43
127.82
125.01
123.36
123.12
117.77
111.54
111.27
106.50
101.70
77.32
77.00
76.68
63.33
56.09
55.85
51.45
48.45
28.42
27.52
MeO
MeO
H
N
O
O
O
9f
NAME zsq6-170-1 - Copy
EXPNO 2
PROCNO 1
Date_ 20161107
Time 21.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2053
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm

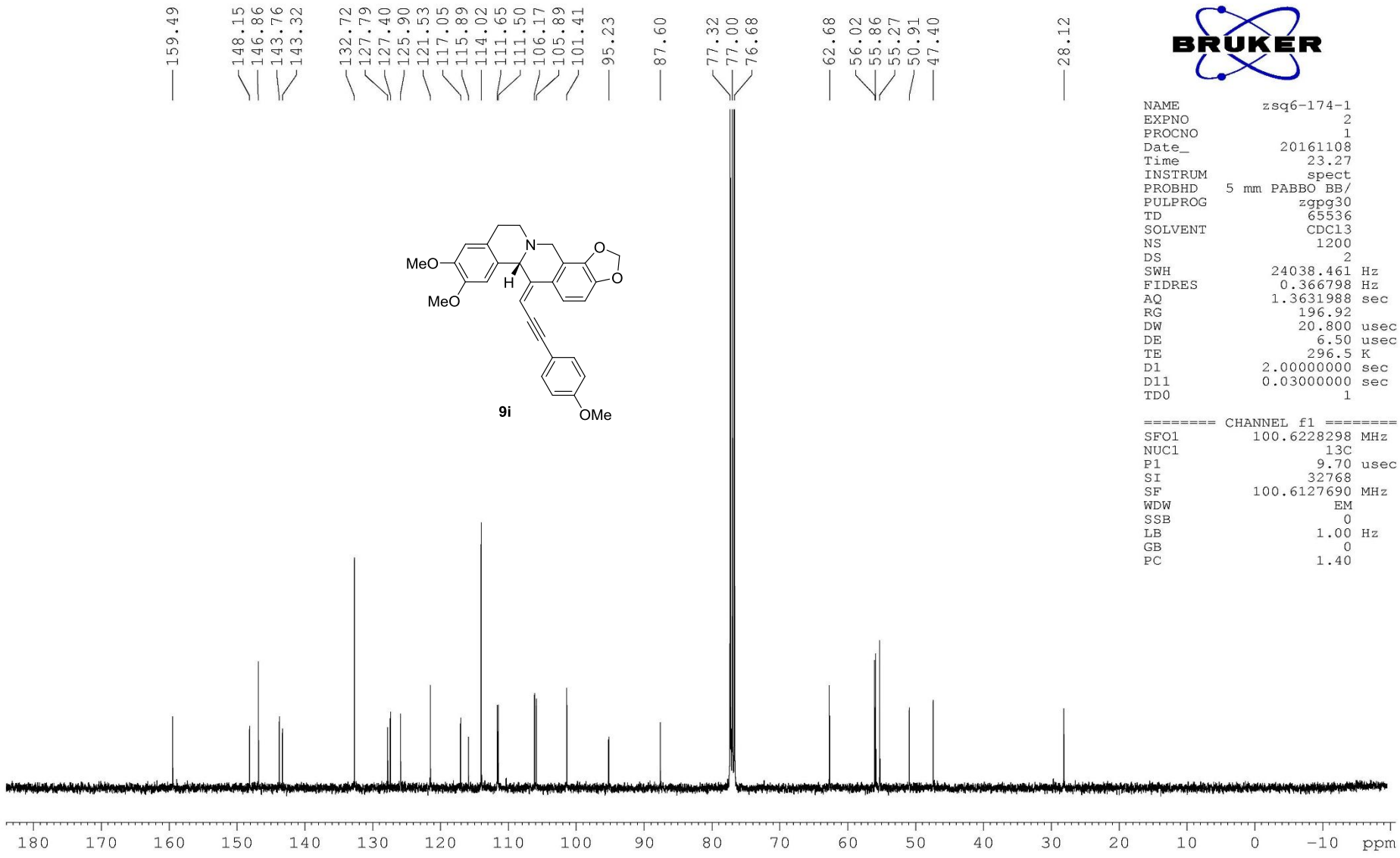

7.666
7.638
7.260
6.945
6.925
6.803
6.782
6.657
6.605
6.021
6.004
5.983
5.975
5.954
4.511
4.221
4.178
4.043
4.000
3.889
3.835
3.717
3.167
3.153
3.141
3.126
2.936
2.925
2.914
2.896
2.875
2.865
2.853
2.845
2.839
2.825
2.808
2.798
1.783
BRUKER
NAME zsq6-168-1-a
EXPNO 1
PROCNO 1
Date_ 20161105
Time 1.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 187.77
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
MeO
N
H
O
O
CO2Me
9g
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
0.99
1.01
1.02
1.02
1.08
0.97
1.14
1.03
0.98
1.18
3.55
3.26
2.99
1.10
3.40
3.04


169.07
148.05
147.33
146.98
143.63
143.15
135.69
128.48
127.98
127.90
125.43
123.54
120.88
117.60
111.54
111.35
106.42
101.57
77.32
77.00
76.69
63.55
56.11
55.84
51.79
51.44
48.61
28.38
12.94
MeO
MeO
N
H
O
O
CO2Me
9g
BRUKER
NAME zsq6-168-1-a
EXPNO 2
PROCNO 1
Date_ 20161105
Time 10.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 8836
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 ppm


8.184
8.163
7.397
7.386
7.378
7.373
7.316
7.306
7.300
7.293
7.288
7.260
6.825
6.804
6.658
6.591
6.018
6.015
5.997
5.994
5.363
4.588
4.338
4.295
4.019
3.976
3.879
3.851
3.224
3.208
3.196
3.185
2.920
2.911
2.894
2.880
2.868
MeO
MeO
H
N
O
O
Ph
9h
NAME zsq6-166-1
EXPNO 1
PROCNO 1
Date_ 20161102
Time 9.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 187.77
DW 62.400 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.04
2.47
3.53
1.01
0.95
0.97
2.03
0.99
1.08
1.12
1.26
3.08
3.42
1.31
3.14


148.19
147.01
146.90
144.85
143.35
131.27
128.33
128.04
127.70
127.43
125.76
123.74
121.65
117.11
111.66
111.50
106.23
105.56
101.46
95.09
88.86
77.32
77.00
76.68
62.71
56.03
55.87
50.95
47.47
28.14
MeO
MeO
N
H
O
O
Ph
9h
BRUKER
NAME zsq6-166-1
EXPNO 2
PROCNO 1
Date_ 20161103
Time 12.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2609
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127714 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm


8.192
8.171
7.334
7.312
7.260
6.845
6.823
6.799
6.652
6.586
6.009
5.990
5.346
4.584
4.340
4.297
4.009
3.966
3.897
3.875
3.846
3.830
3.803
3.791
3.783
3.245
3.220
3.204
3.192
3.180
3.161
2.940
2.916
2.887
2.872
2.861
BRUKER
NAME zsq6-174-1
EXPNO 1
PROCNO 1
Date_ 20161108
Time 23.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 112.31
DW 62.400 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
MeO
N
H
O
O
OMe
9i
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
2.15
3.32
1.11
1.11
2.10
1.03
1.02
1.03
1.15
3.17
3.46
3.41
1.10
3.19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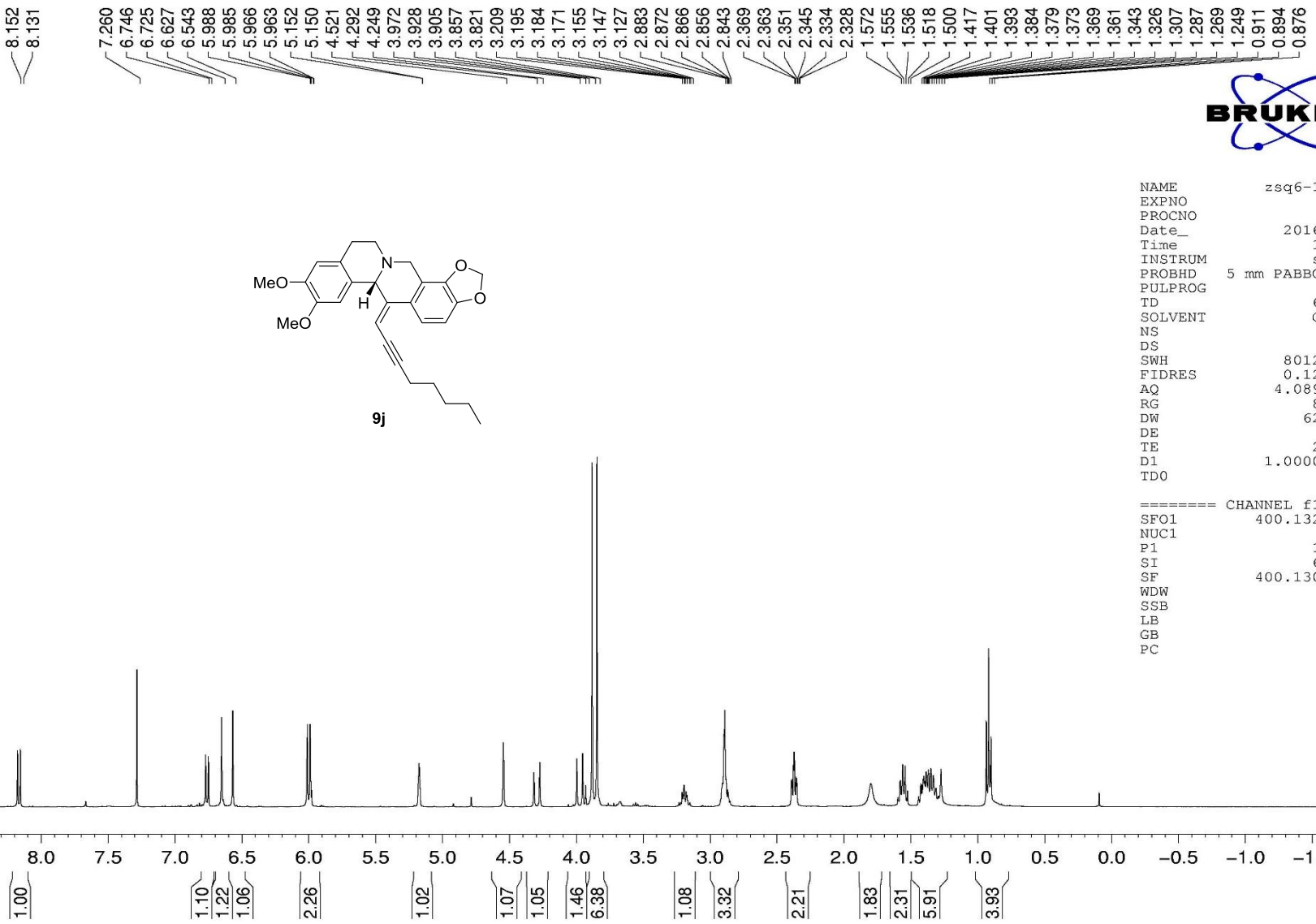
148.15
146.86
143.76
143.32
132.72
127.79
127.40
125.90
121.53
117.05
115.89
114.02
111.65
111.50
106.17
105.89
101.41
95.23
87.60
77.32
77.00
76.68
62.68
56.02
55.86
55.27
50.91
47.40
28.12
MeO
MeO
N
H
O
O
OMe
9i
BRUKER
NAME zsq6-174-1
EXPNO 2
PROCNO 1
Date_ 20161108
Time 23.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1200
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 ppm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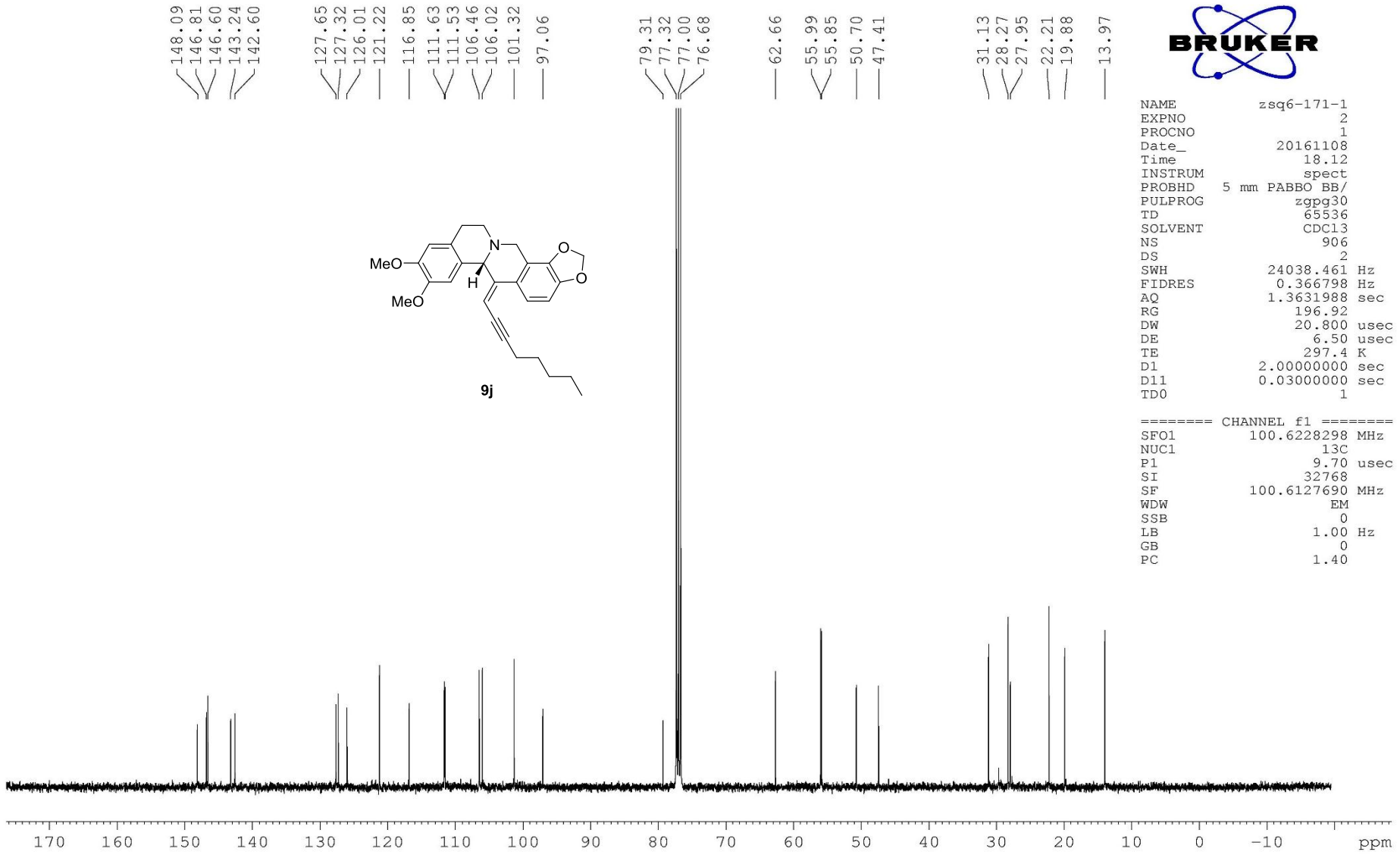
8.131
7.260
6.746
6.725
6.627
6.543
5.988
5.985
5.966
5.963
5.152
5.150
4.521
4.292
4.249
3.972
3.928
3.905
3.857
3.821
3.209
3.195
3.184
3.171
3.155
3.147
3.127
2.883
2.872
2.866
2.856
2.843
2.369
2.363
2.351
2.345
2.334
2.328
1.572
1.555
1.536
1.518
1.500
1.417
1.401
1.393
1.384
1.379
1.373
1.369
1.361
1.343
1.326
1.307
1.287
1.269
1.249
0.911
0.894
0.876
BRUKER
MeO
MeO
N
H
O
O
9j
NAME zsq6-171-1
EXPNO 1
PROCNO 1
Date_ 20161108
Time 15.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 88.84
DW 62.400 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0 −1.5 ppm
1.00
1.10
1.22
1.06
2.26
1.02
1.07
1.05
1.46
6.38
1.08
3.32
2.21
1.83
2.31
5.91
3.93


148.09
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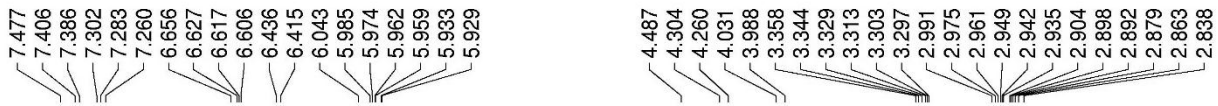
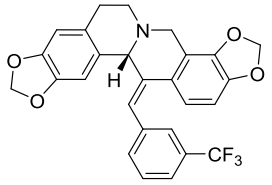
146.60
143.24
142.60
127.65
127.32
126.01
121.22
116.85
111.63
111.53
106.46
106.02
101.32
97.06
79.31
77.32
77.00
76.68
62.66
55.99
55.85
50.70
47.41
31.13
28.27
27.95
22.21
19.88
13.97
MeO
MeO
H
N
O
O
9j
BRUKER
NAME zsq6-171-1
EXPNO 2
PROCNO 1
Date_ 20161108
Time 18.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 906
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 ppm

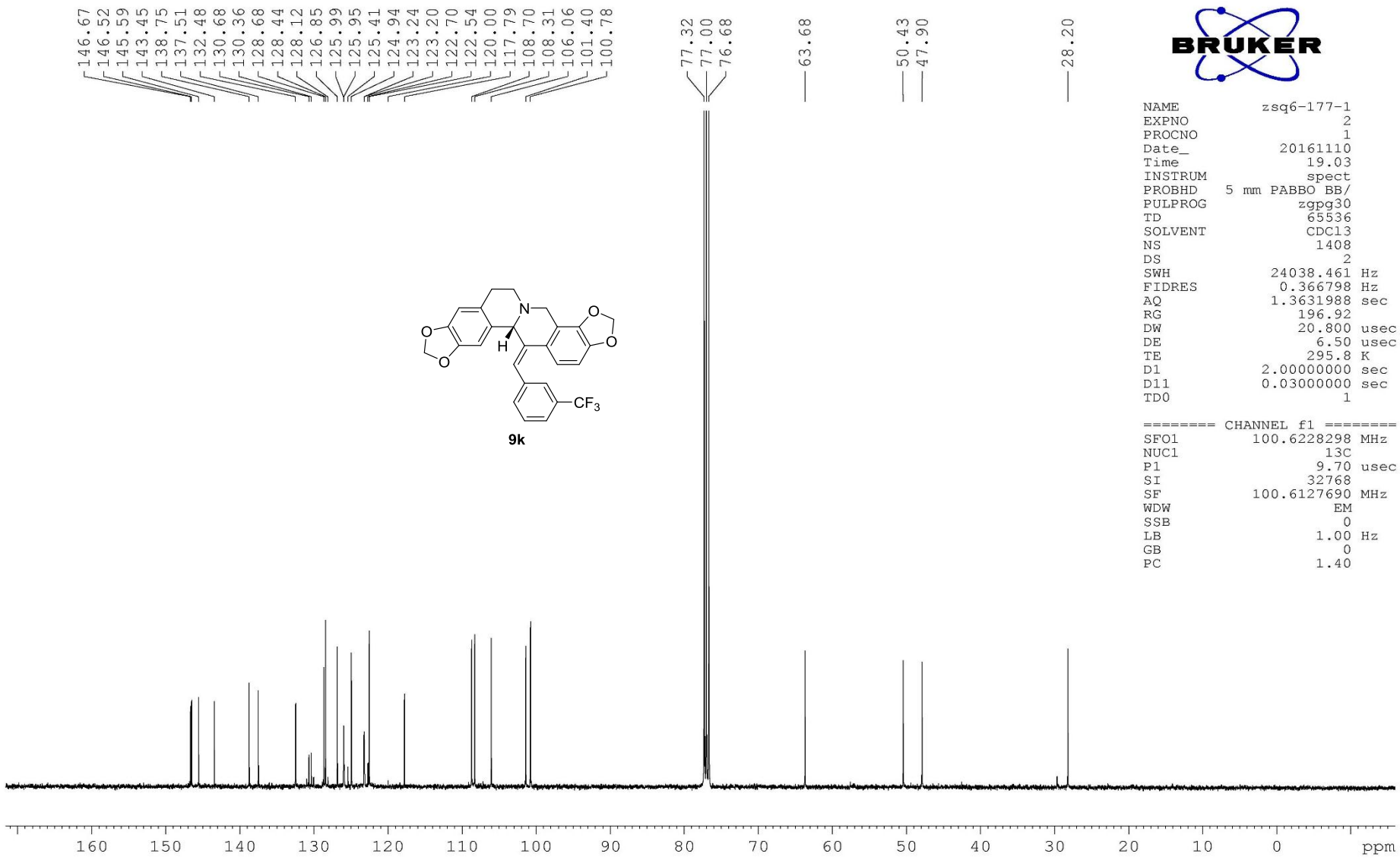




9k

```
NAME            zsq6-177-1
EXPNO                    1
PROCNO                   1
Date_             20161110
Time                 17.57
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8012.820 Hz
FIDRES               0.122266 Hz
AQ                4.0894966 sec
RG                   82.92
DW                   62.400 usec
DE                    6.50 usec
TE                   294.3 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                    1H
P1                   14.50 usec
SI                   65536
SF              400.1300094 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```


146.67
146.52
145.59
143.45
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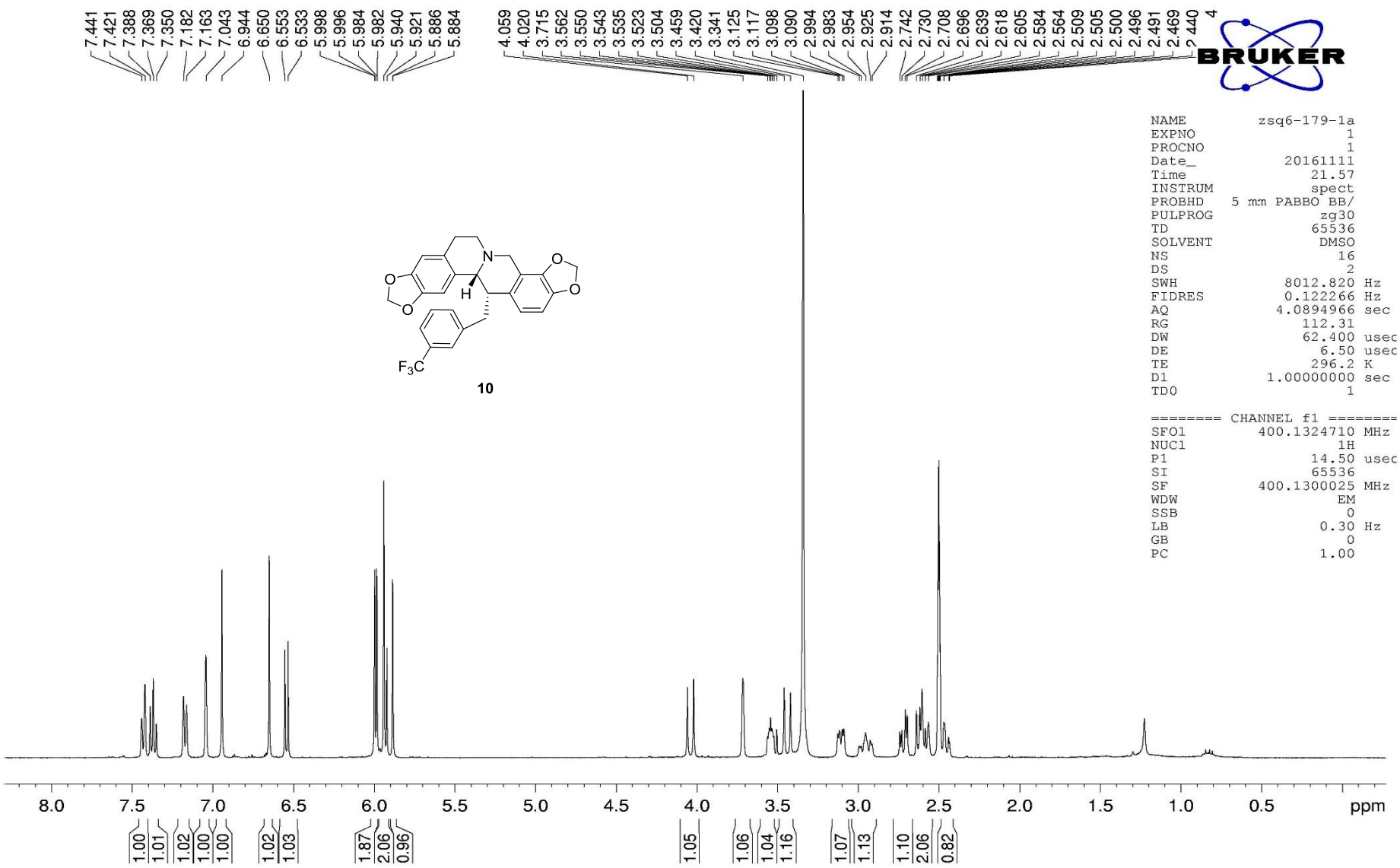
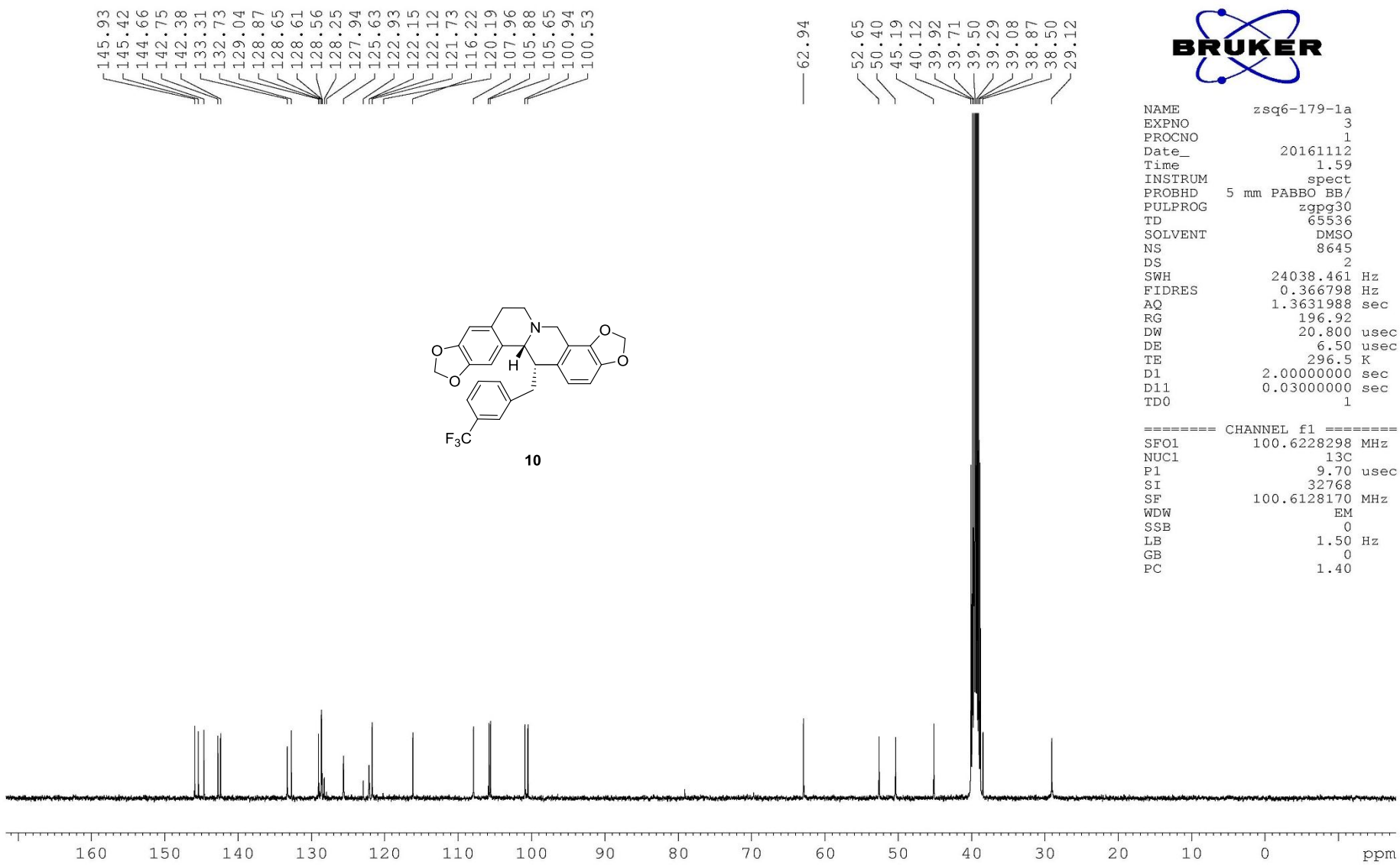
137.51
132.48
130.68
130.36
128.68
128.44
128.12
126.85
125.99
125.95
125.41
124.94
123.24
123.20
122.70
122.54
120.00
117.79
108.70
108.31
106.06
101.40
100.78
77.32
77.00
76.68
63.68
50.43
47.90
28.20
BRUKER
NAME zsq6-177-1
EXPNO 2
PROCNO 1
Date_ 20161110
Time 19.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1408
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 295.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
O
O
O
O
H
CF3
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

9k


7.441 7.421 7.388 7.369 7.350 7.182 7.163 7.043 6.944 6.650 6.553 6.533 5.998 5.996 5.984 5.982 5.940 5.921 5.886 5.884
4.059 4.020 3.715 3.562 3.550 3.543 3.535 3.523 3.504 3.459 3.420 3.341 3.125 3.117 3.098 3.090 2.994 2.983 2.954 2.925 2.914 2.742 2.730 2.708 2.696 2.639 2.618 2.605 2.584 2.564 2.509 2.505 2.500 2.496 2.491 2.469 2.440
BRUKER
NAME zsq6-179-1a
EXPNO 1
PROCNO 1
Date_ 20161111
Time 21.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 112.31
DW 62.400 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300025 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
O
O
O
O
H
F3C
10
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00 1.01 1.02 1.00 1.00 1.02 1.03 1.87 2.06 0.96 1.05 1.06 1.04 1.16 1.07 1.13 1.10 2.06 0.82


145.93
145.42
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142.75
142.38
133.31
132.73
129.04
128.87
128.65
128.61
128.56
128.25
127.94
125.63
122.93
122.15
122.12
121.73
116.22
120.19
107.96
105.88
105.65
100.94
100.53
62.94
52.65
50.40
45.19
40.12
39.92
39.71
39.50
39.29
39.08
38.87
38.50
29.12
BRUKER
NAME zsq6-179-1a
EXPNO 3
PROCNO 1
Date_ 20161112
Time 1.59
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 8645
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6128170 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
N
O
O
O
O
H
F3C
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

10

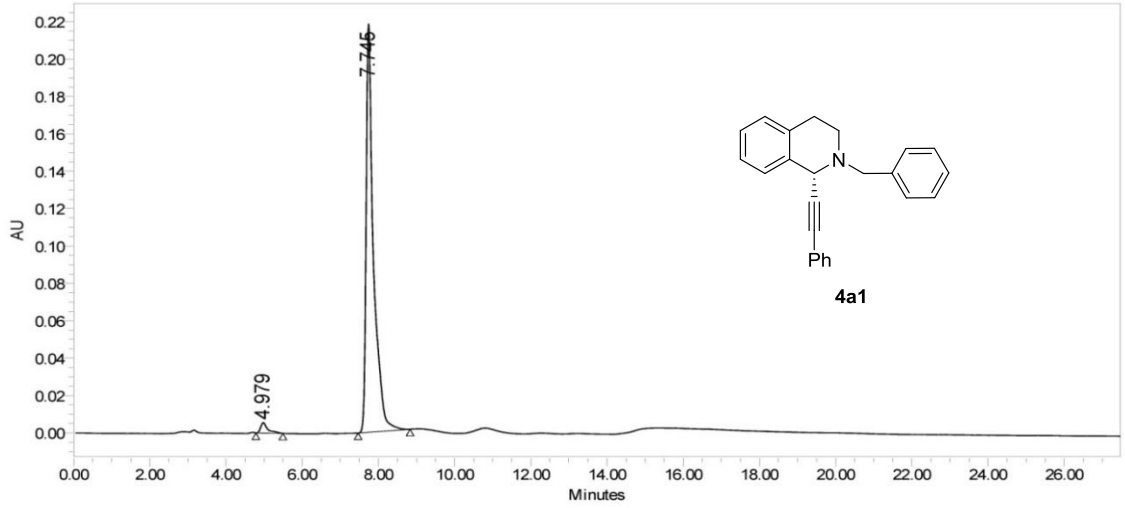
5. Copies of HPCL Chromatograms

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	6-63-1-g	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	8/20/2016 7:32:55 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	3	Date Processed:	12/15/2016 9:44:11 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	40.00 Minutes	Sample Set Name	



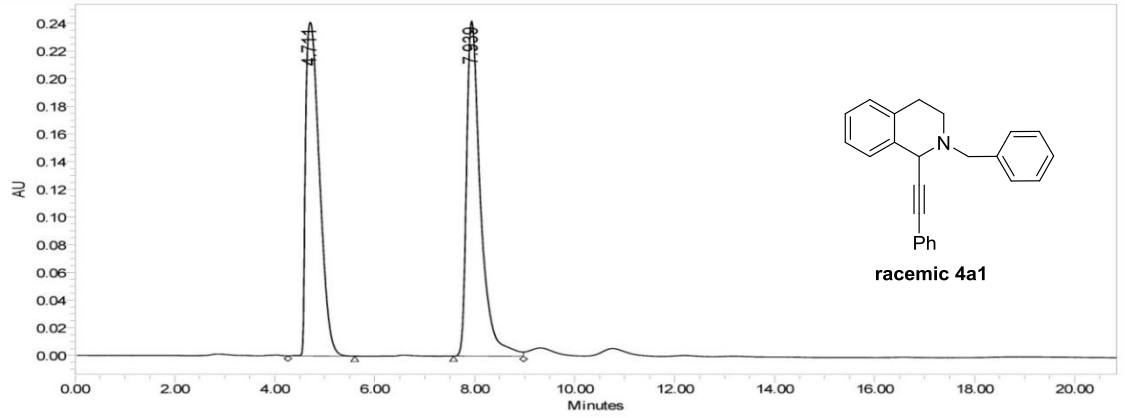
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	4.979	71781	2.39	5603	2.50
2	7.745	2932952	97.61	218319	97.50

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	6-63-1-racimic	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	8/20/2016 7:10:57 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	2	Date Processed:	12/15/2016 9:47:24 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	40.00 Minutes	Sample Set Name	



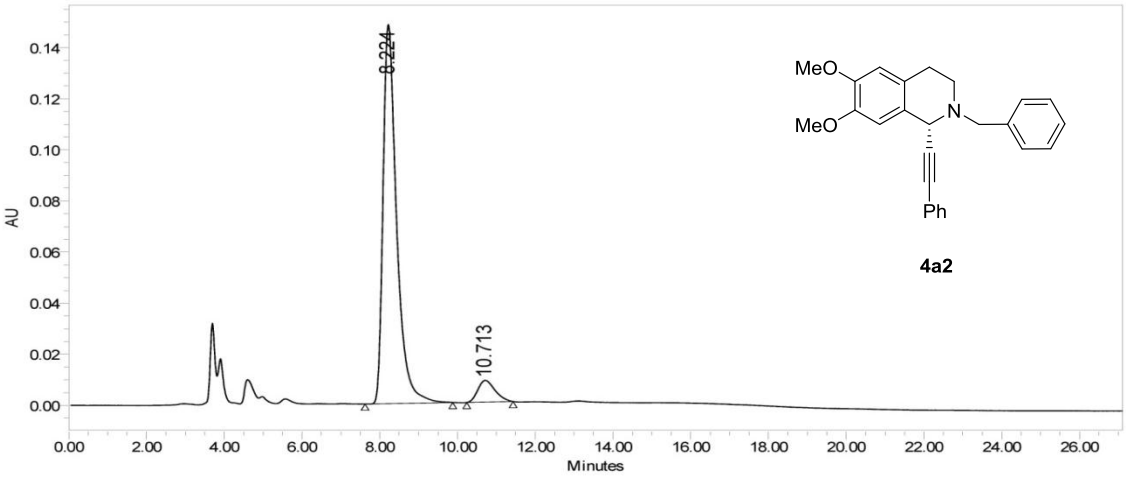
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	4.711	4632083	49.66	240965	49.89
2	7.939	4695830	50.34	242015	50.11

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	6-77-1-asymmetric(95:5)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	8/27/2016 3:18:44 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	2	Date Processed:	12/15/2016 10:08:39 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	50.00 Minutes	Sample Set Name	



4a2

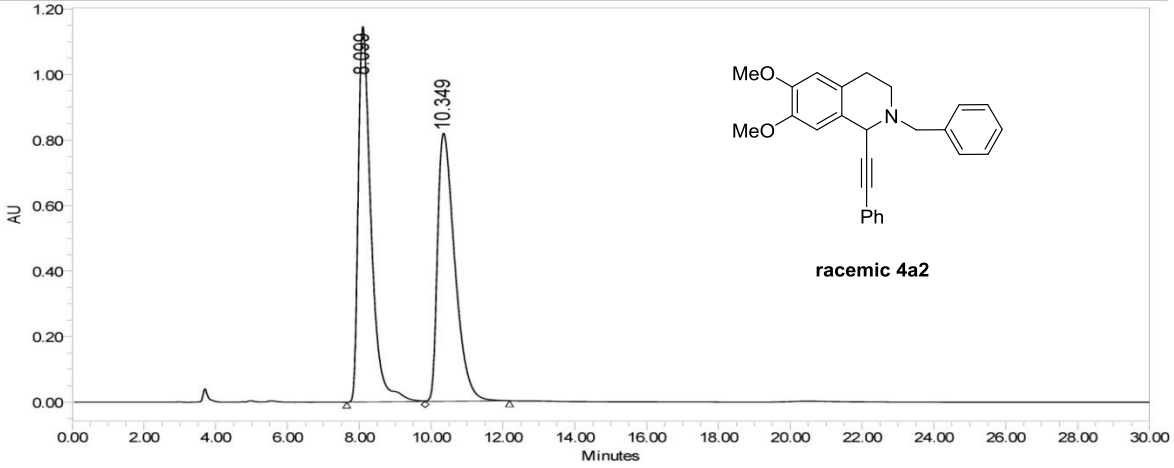
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	8.224	3585169	93.21	148362	94.58
2	10.713	261316	6.79	8501	5.42

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	6-77-1a-racimic(95:5)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	8/27/2016 4:18:25 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	4	Date Processed:	12/15/2016 10:11:33 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	30.00 Minutes	Sample Set Name	



racemic 4a2

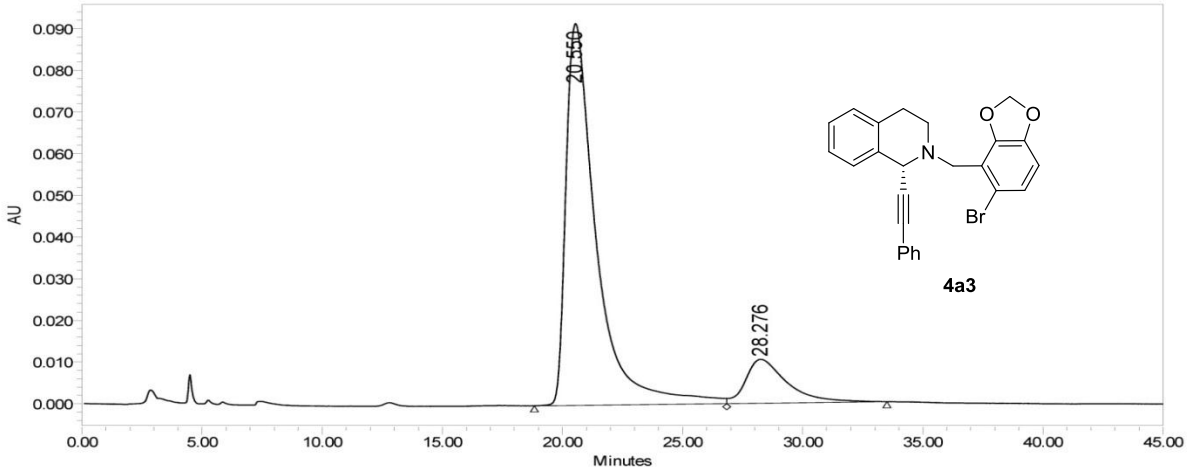
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	8.099	28370375	50.95	1146175	58.33
2	10.349	27307421	49.05	818770	41.67

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	6-70-1d-(500:1)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	8/27/2016 11:55:07 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	1	Date Processed:	12/15/2016 9:51:15 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	45.00 Minutes	Sample Set Name	



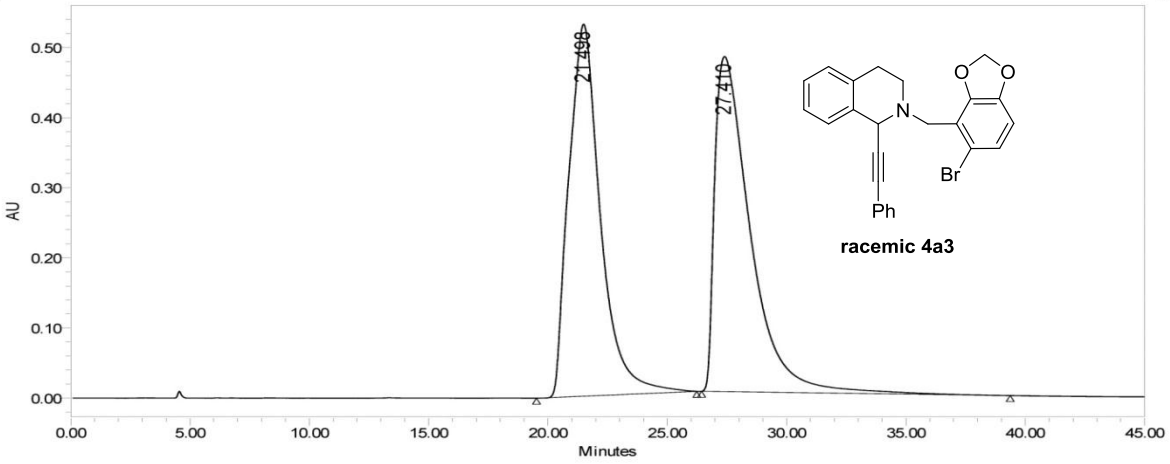
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	20.550	8039556	86.46	91641	89.68
2	28.276	1258672	13.54	10548	10.32

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	6-70-1d-racimic(500:1)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	8/27/2016 12:53:13 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	2	Date Processed:	12/15/2016 9:50:15 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	45.00 Minutes	Sample Set Name	



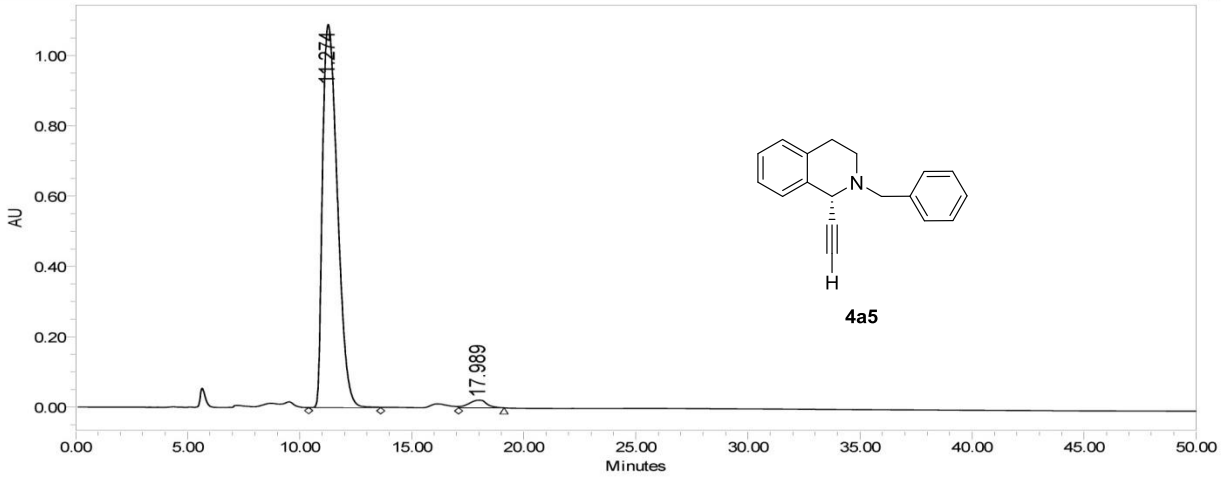
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	21.498	48799969	49.62	530241	52.61
2	27.410	49539975	50.38	477691	47.39

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Y6-71-2-b(500:1)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	8/25/2016 12:46:12 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	1	Date Processed:	12/15/2016 10:03:19 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	50.00 Minutes	Sample Set Name	



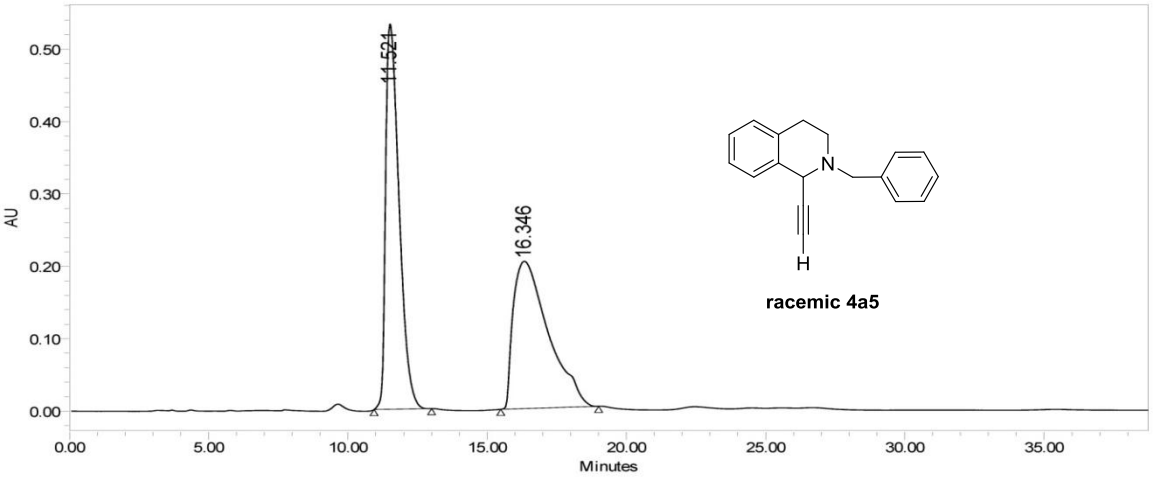
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	11.274	50119390	97.66	1089024	98.06
2	17.989	1202198	2.34	21537	1.94

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	6-71-2a-racimic(500:1)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	8/25/2016 4:10:59 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	4	Date Processed:	12/15/2016 10:04:25 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	50.00 Minutes	Sample Set Name	



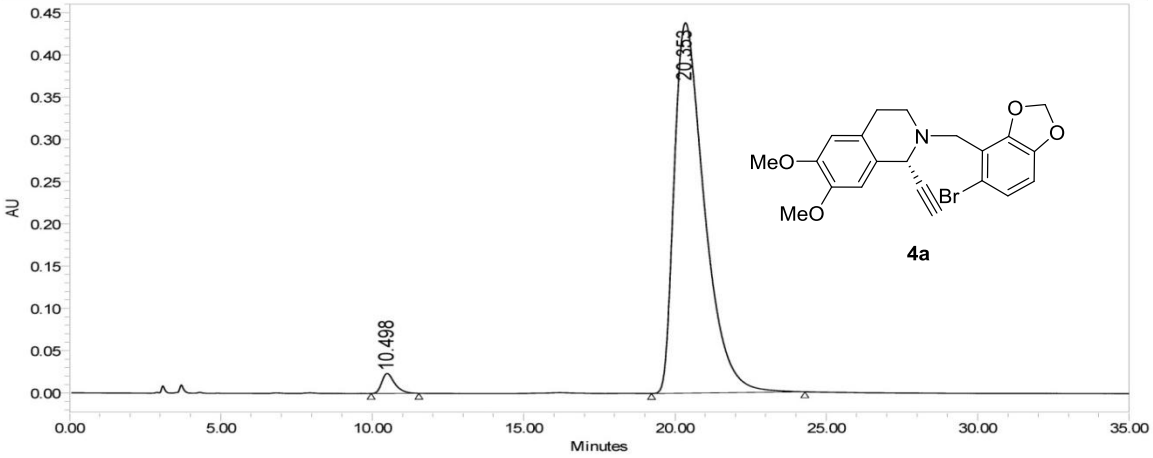
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	11.521	17917791	50.12	532033	72.34
2	16.346	17829169	49.88	203424	27.66

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq6-127-1c (90/10 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	11/18/2016 8:37:34 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	2	Date Processed:	12/15/2016 8:11:27 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	35.00 Minutes	Sample Set Name	



4a

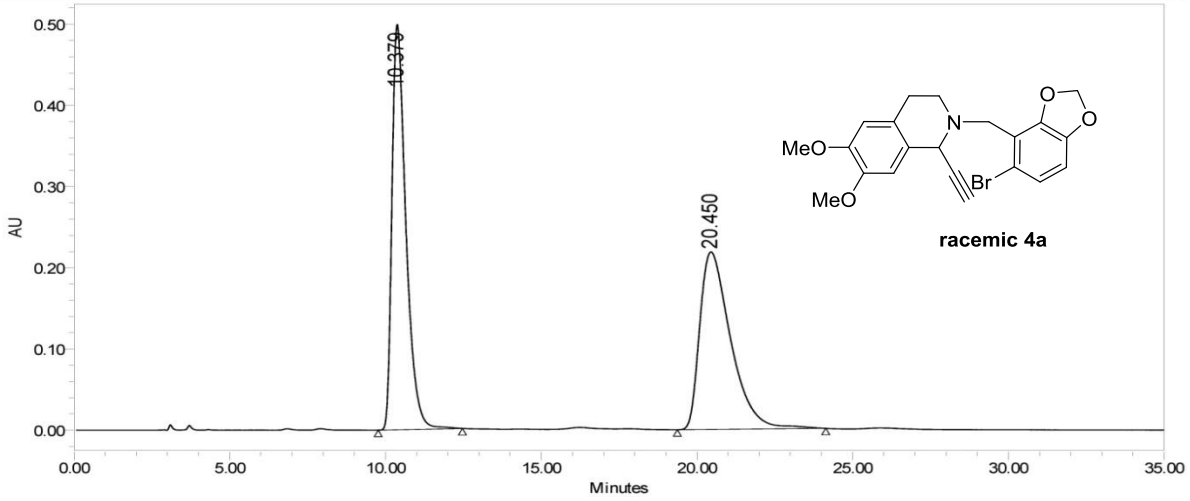
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	10.498	700416	2.25	23566	5.10
2	20.353	30388261	97.75	438369	94.90

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq2-105-1c2 (90/10 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	11/18/2016 11:29:09 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	5	Date Processed:	12/15/2016 8:14:01 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	35.00 Minutes	Sample Set Name	



racemic 4a

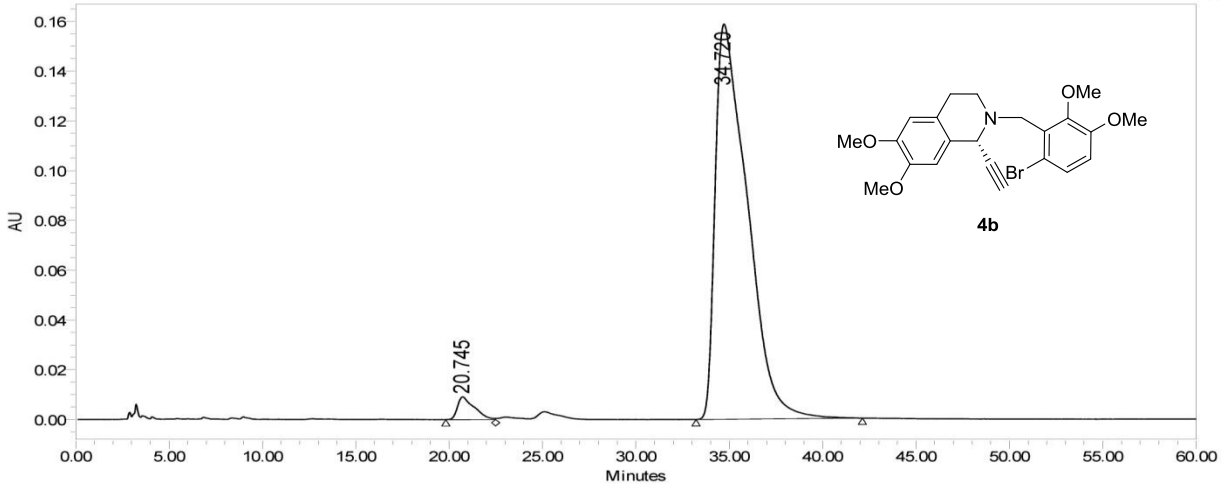
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	10.379	15518983	50.65	498959	69.53
2	20.450	15123163	49.35	218643	30.47

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq6-120-1 ((95/5 AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/20/2016 7:50:36 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	2	Date Processed:	12/15/2016 7:35:52 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



4b

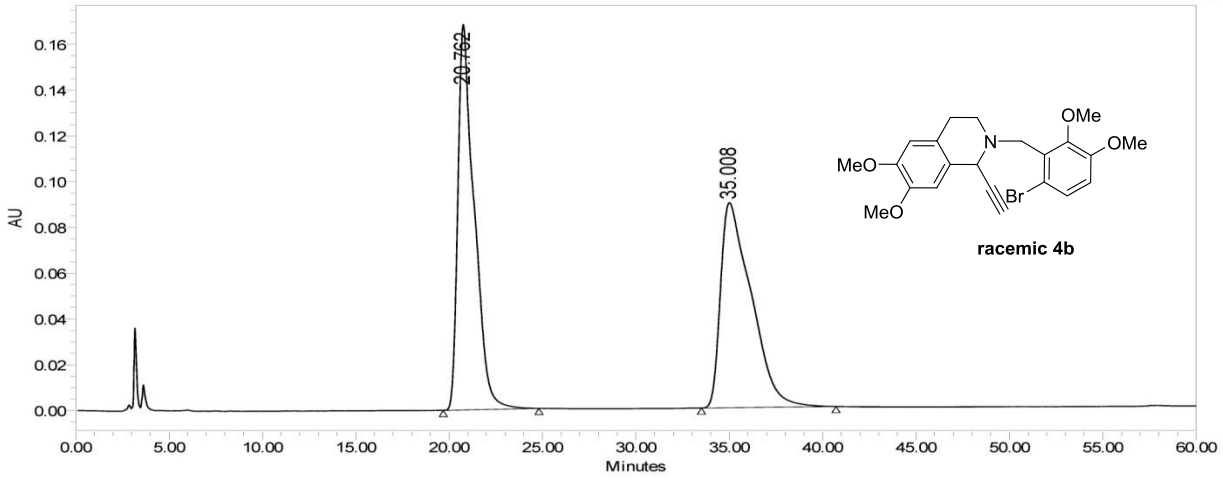
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	20.745	571581	2.94	9169	5.46
2	34.720	18893146	97.06	158800	94.54

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq2-104-1new made ((95/5 AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/20/2016 6:40:31 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	1	Date Processed:	12/15/2016 7:37:16 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



racemic 4b

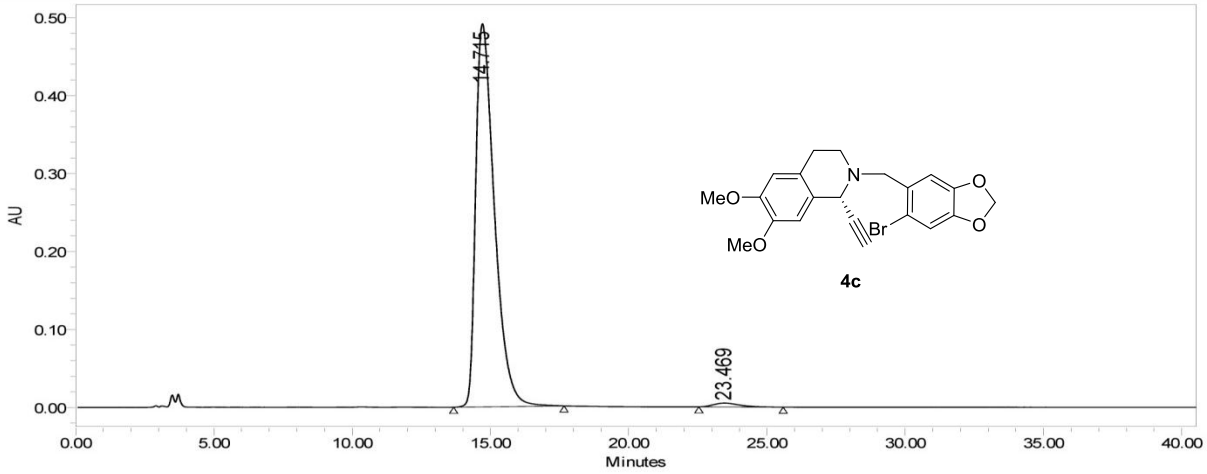
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	20.762	10237587	49.96	168427	65.26
2	35.008	10252371	50.04	89651	34.74

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq6-125-1 up (90/10 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/21/2016 5:36:01 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	4	Date Processed:	12/15/2016 8:23:45 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



4c

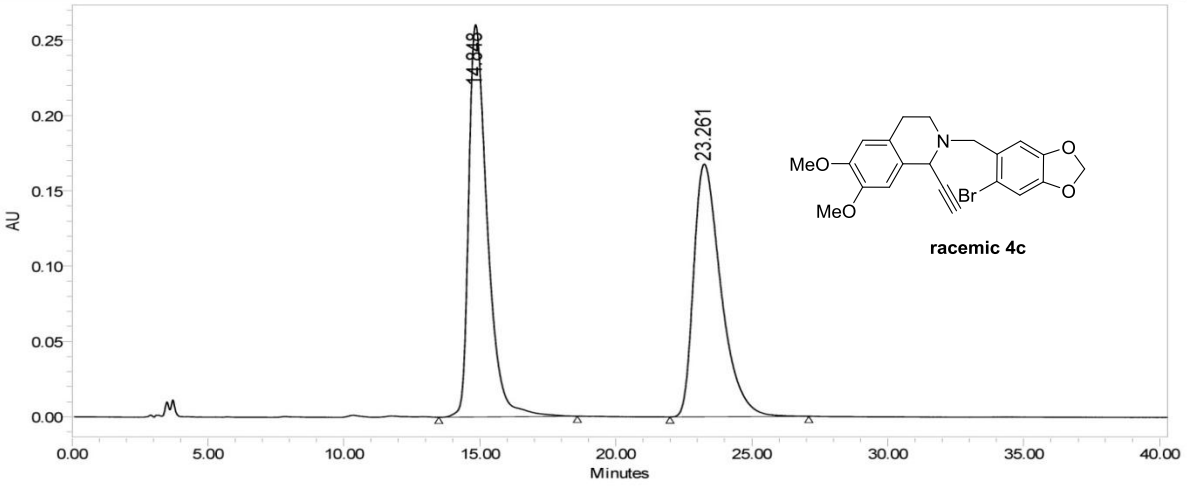
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	14.715	23142231	98.64	491402	99.00
2	23.469	318052	1.36	4961	1.00

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq3-45-2 up (90/10 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/21/2016 4:55:12 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	3	Date Processed:	12/15/2016 8:22:31 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



racemic 4c

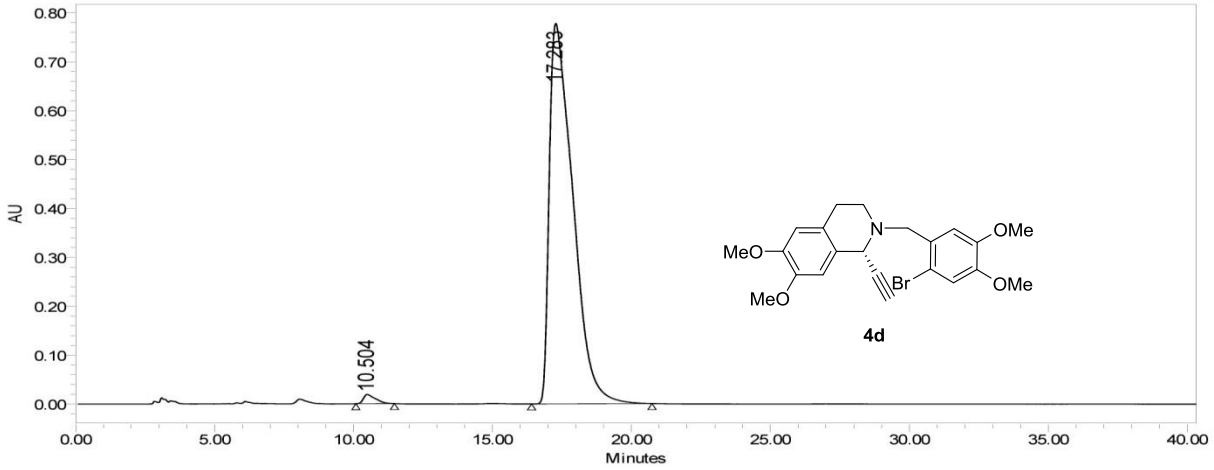
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	14.848	12390424	51.39	260432	60.82
2	23.261	11718759	48.61	167800	39.18

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq6-122-1 ((80/20 AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/20/2016 1:25:08 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	2	Date Processed:	12/15/2016 7:56:09 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



4d

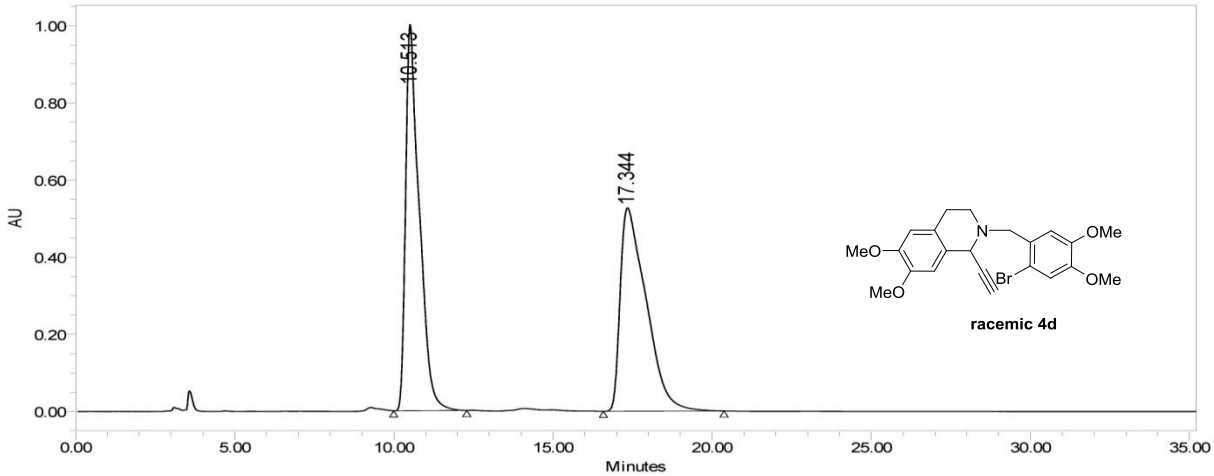
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	10.504	562372	1.22	18840	2.37
2	17.283	45641914	98.78	777629	97.63

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq2-105-2 ((80/20 AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/20/2016 12:49:16 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	1	Date Processed:	12/15/2016 7:57:02 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



racemic 4d

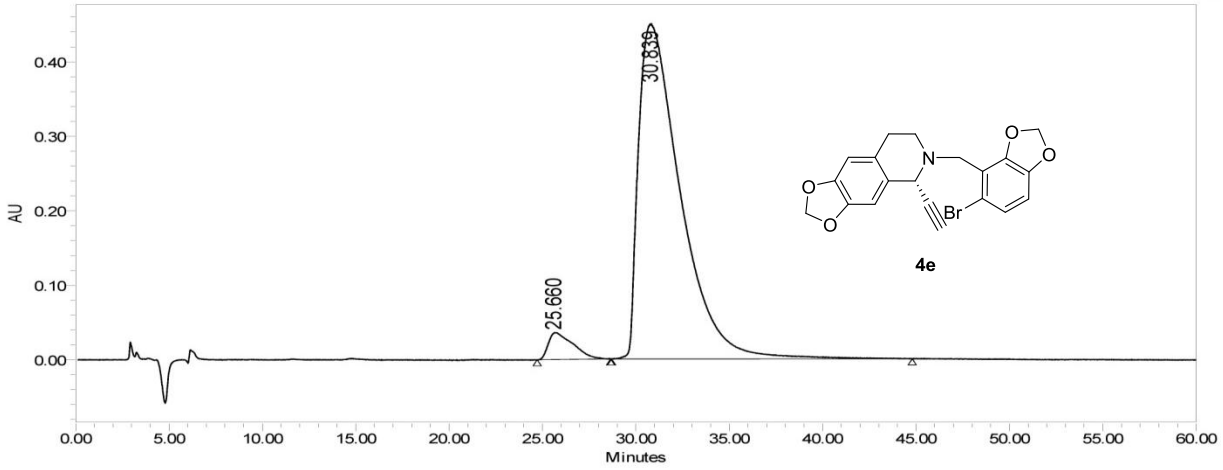
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	10.513	29862365	50.07	1000742	65.50
2	17.344	29779848	49.93	527000	34.50

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq6-134-1C(99/1 Et2NH AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/6/2016 11:01:46 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	3	Date Processed:	12/15/2016 5:44:41 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch4 210nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



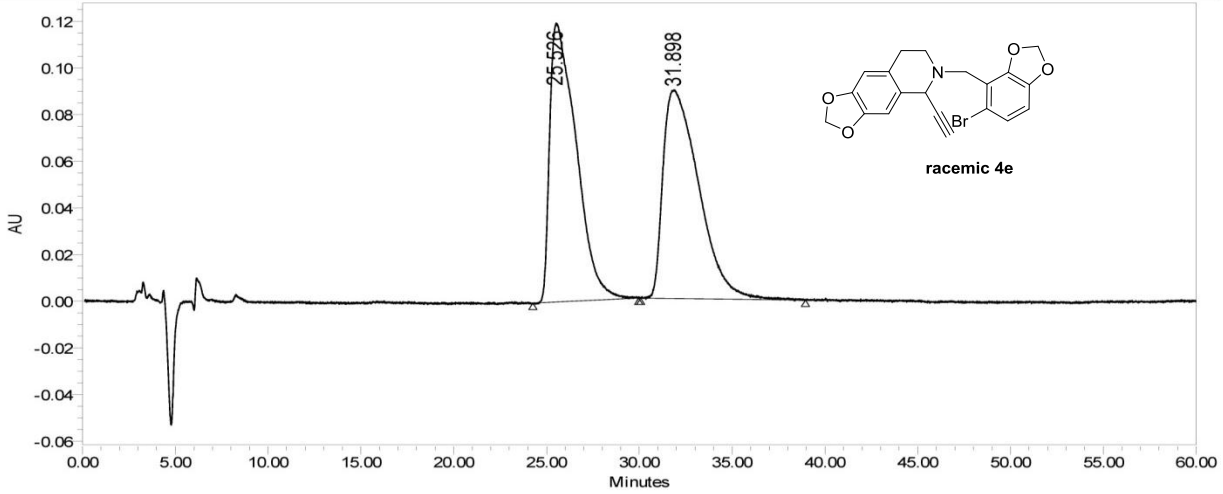
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	25.660	3337468	4.53	36325	7.46
2	30.839	70268922	95.47	450293	92.54

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq2-106-2C(99/1 Et2NH AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/6/2016 10:01:07 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	2	Date Processed:	12/15/2016 5:46:02 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch4 210nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



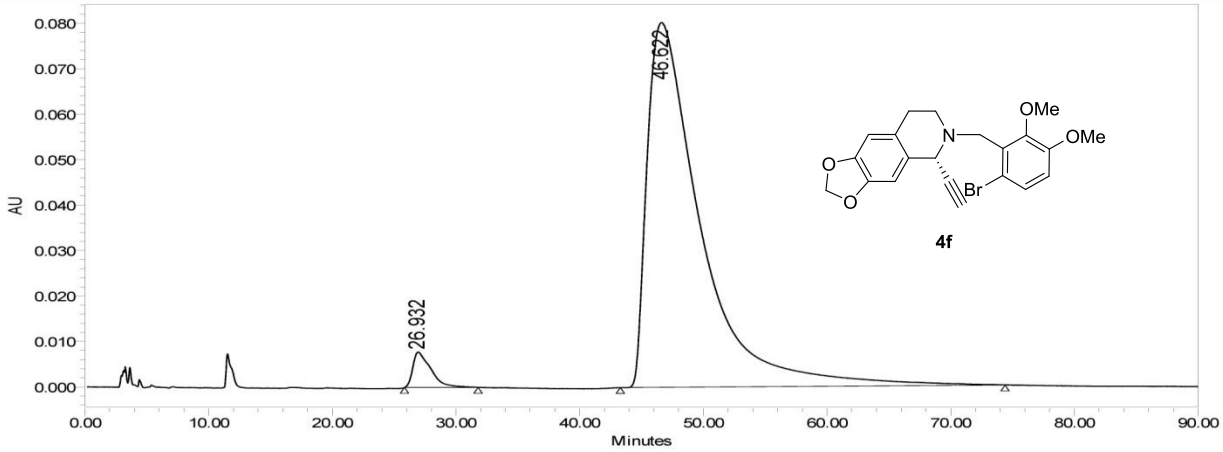
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	25.526	11829054	49.70	119619	57.22
2	31.898	11971513	50.30	89441	42.78

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq6-132-1f (100/1 AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/5/2016 6:21:48 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	3	Date Processed:	12/15/2016 6:21:26 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	90.00 Minutes	Sample Set Name	



4f

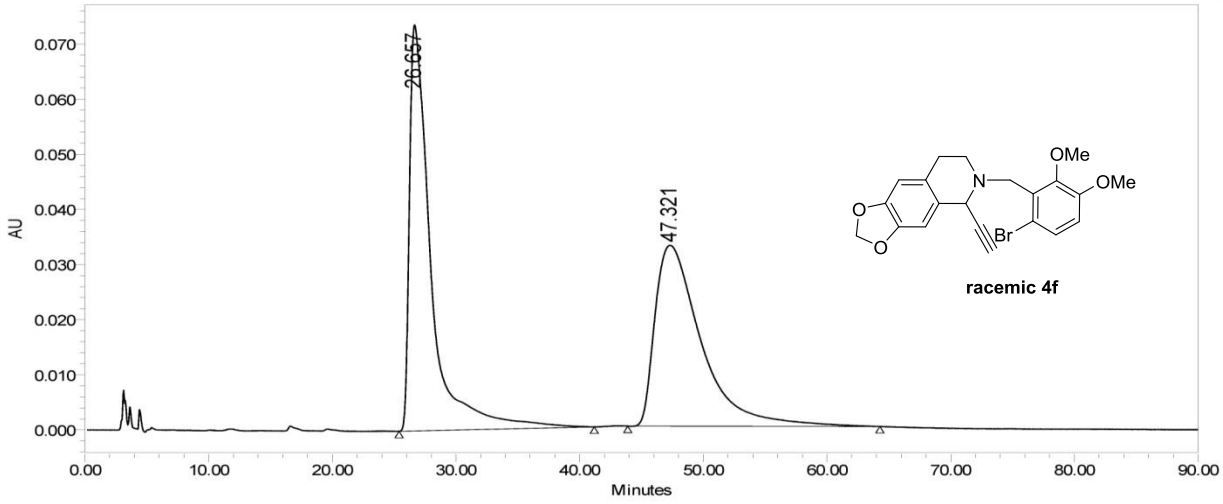
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	26.932	785483	3.15	7790	8.85
2	46.622	24141417	96.85	80260	91.15

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq2-106-1f (100/1 AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/5/2016 7:52:35 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	4	Date Processed:	12/15/2016 6:20:06 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	90.00 Minutes	Sample Set Name	



racemic 4f

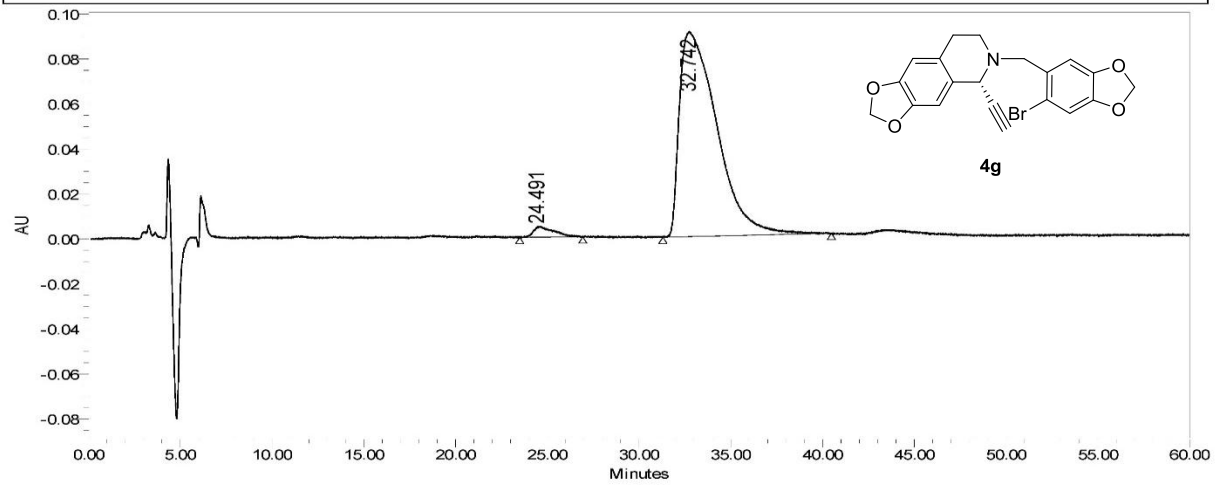
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	26.657	8912785	51.04	73628	69.17
2	47.321	8547995	48.96	32821	30.83

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq6-137-1(100/1+Et2NH AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/7/2016 4:53:03 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	3	Date Processed:	12/15/2016 7:27:04 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



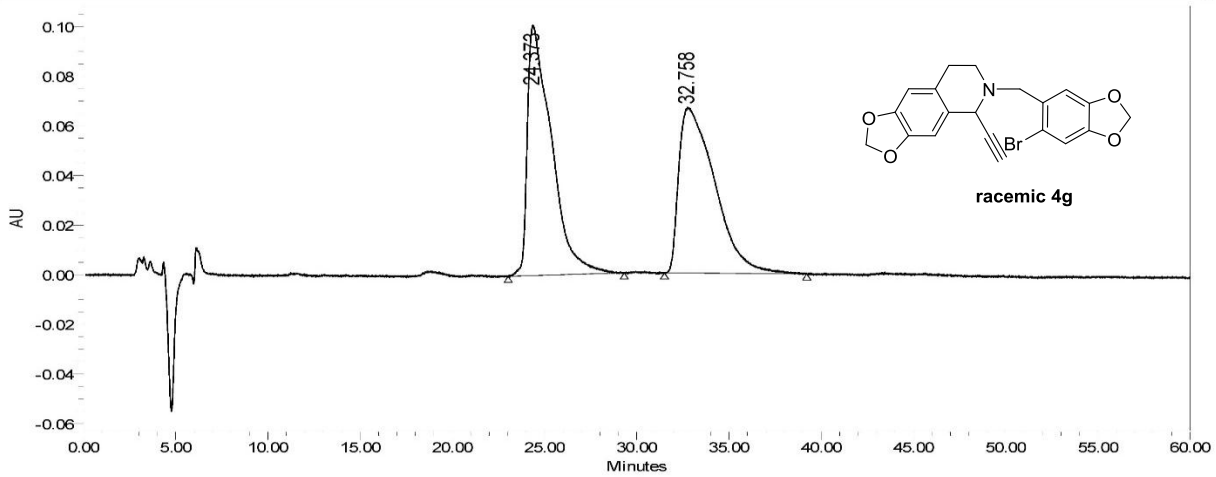
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	24.491	376056	2.83	4858	5.06
2	32.742	12896314	97.17	91191	94.94

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq3-93-2b(100/1+Et2NH AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/7/2016 3:52:08 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	2	Date Processed:	12/15/2016 7:28:23 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



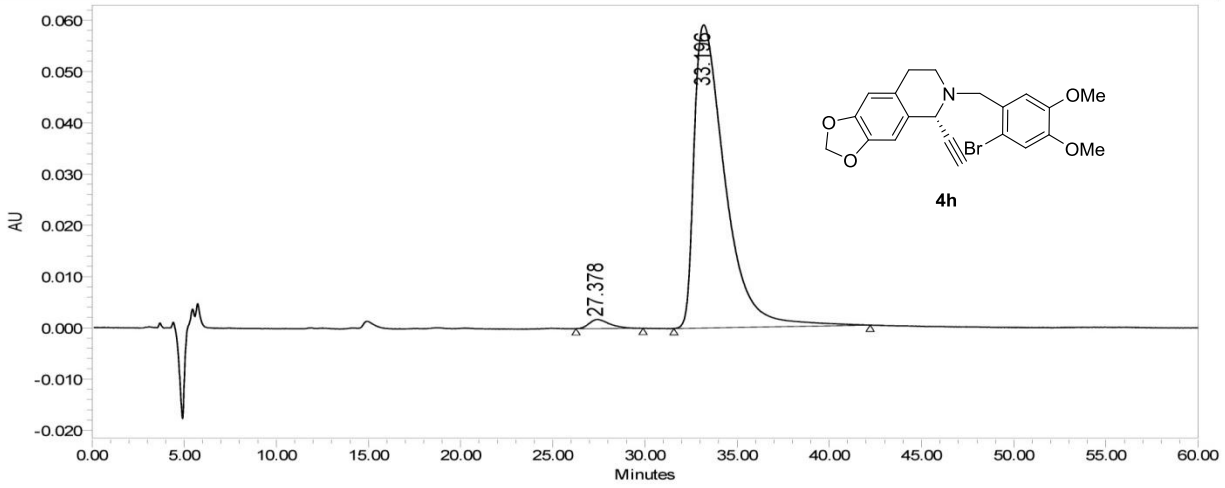
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	24.373	9144380	50.32	100896	60.17
2	32.758	9029440	49.68	66778	39.83

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq6-141-1a4 (99/1 Et2NH OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/23/2016 10:43:18 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	9	Date Processed:	12/15/2016 6:55:29 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch3 230nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



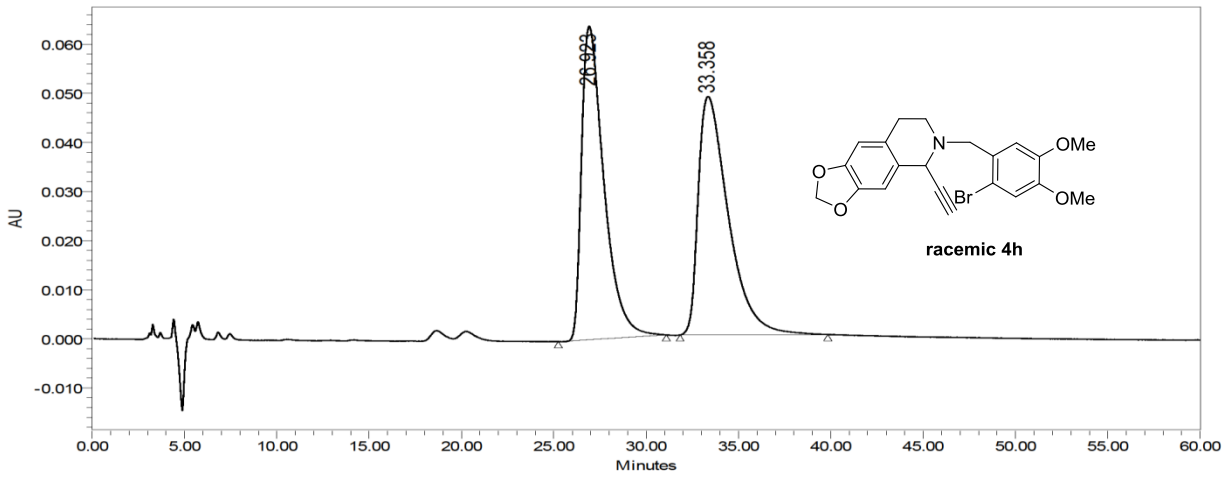
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	27.378	136498	1.93	1777	2.92
2	33.196	6929670	98.07	59165	97.08

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq2-107-1d2 (99/1 Et2NH OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/23/2016 9:42:07 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	8	Date Processed:	12/15/2016 6:57:23 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch3 230nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



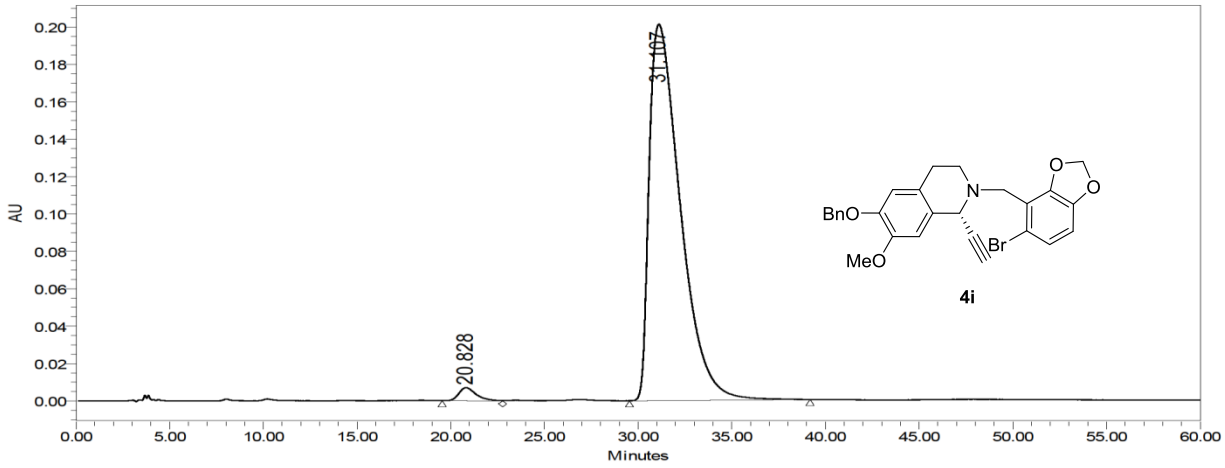
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	26.923	5366776	49.40	63837	56.80
2	33.358	5497782	50.60	48545	43.20

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq6-147-1 (95/5 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/11/2016 7:33:54 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	2	Date Processed:	12/15/2016 9:33:24 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



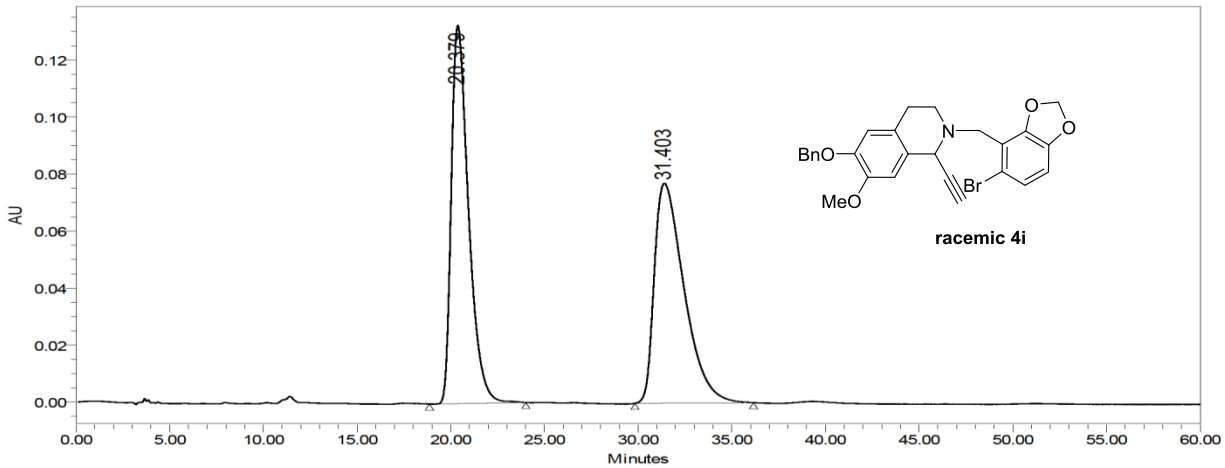
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	20.828	448330	1.89	6980	3.35
2	31.107	23238832	98.11	201286	96.65

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq4-120-2b (95/5 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/11/2016 9:50:33 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	5	Date Processed:	12/15/2016 9:35:45 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



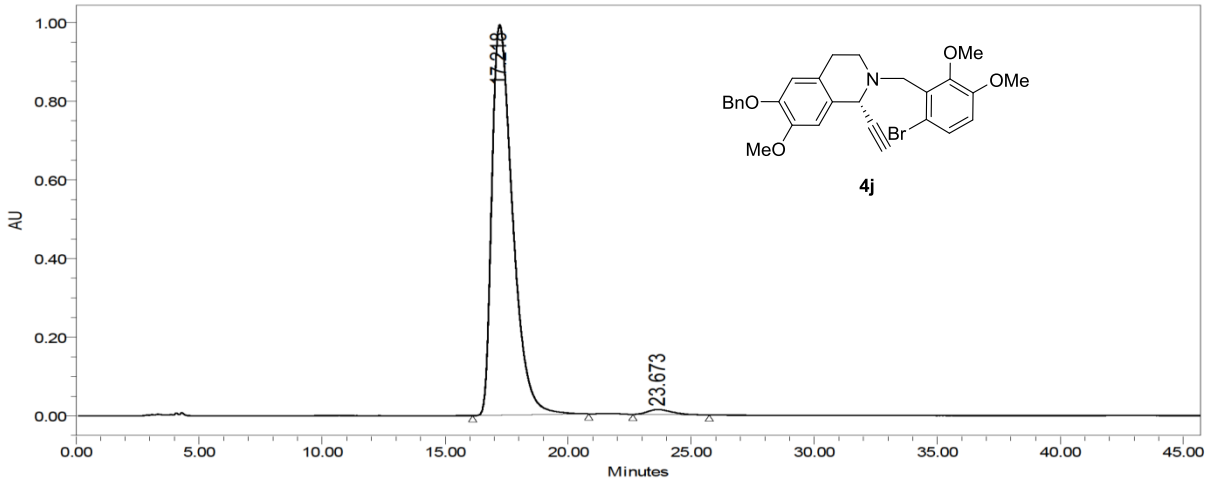
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	20.379	8477724	49.88	132661	63.24
2	31.403	8517374	50.12	77123	36.76

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq6-146-1a (95/5 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/10/2016 4:34:51 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	8	Date Processed:	12/15/2016 9:14:15 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	50.00 Minutes	Sample Set Name	



4j

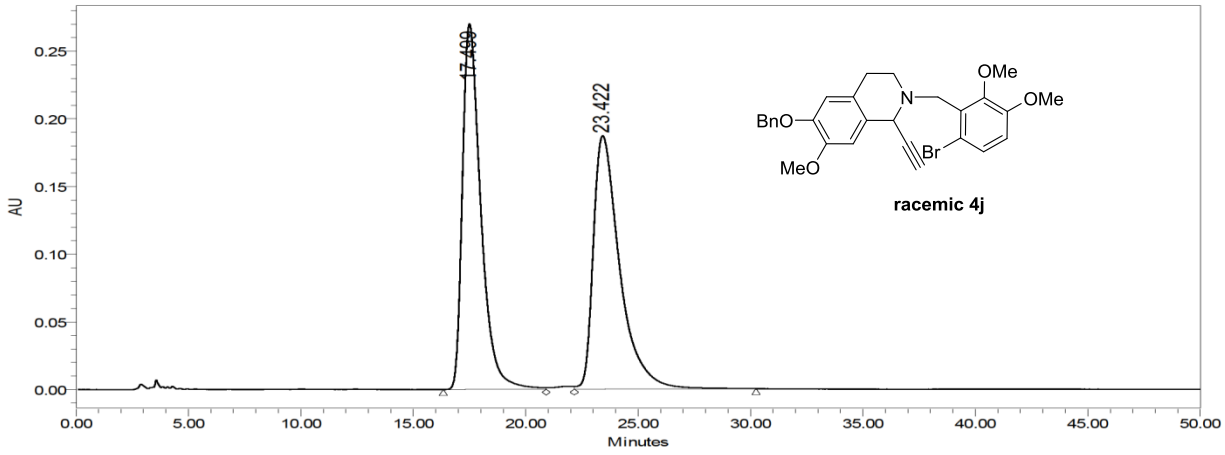
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	17.218	58169883	98.51	993001	98.72
2	23.673	882463	1.49	12841	1.28

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq4-94-2d (95/5 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/10/2016 3:43:55 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	7	Date Processed:	12/15/2016 9:17:00 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	50.00 Minutes	Sample Set Name	



racemic 4j

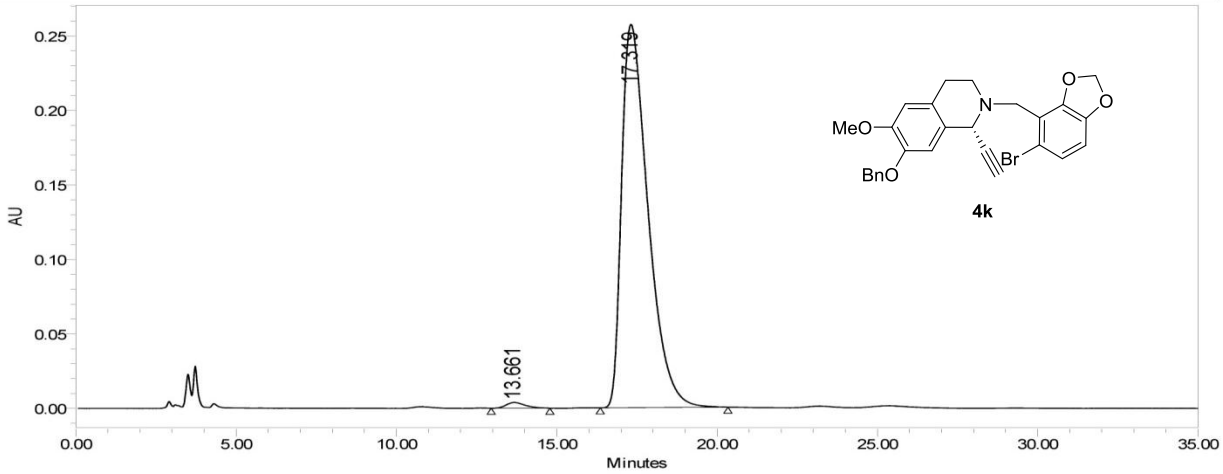
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	17.499	15357856	50.10	270105	59.07
2	23.422	15297733	49.90	187190	40.93

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq6-150-1 down prd ((90/10 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/16/2016 2:04:07 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	13	Date Processed:	12/15/2016 9:04:35 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	35.00 Minutes	Sample Set Name	



4k

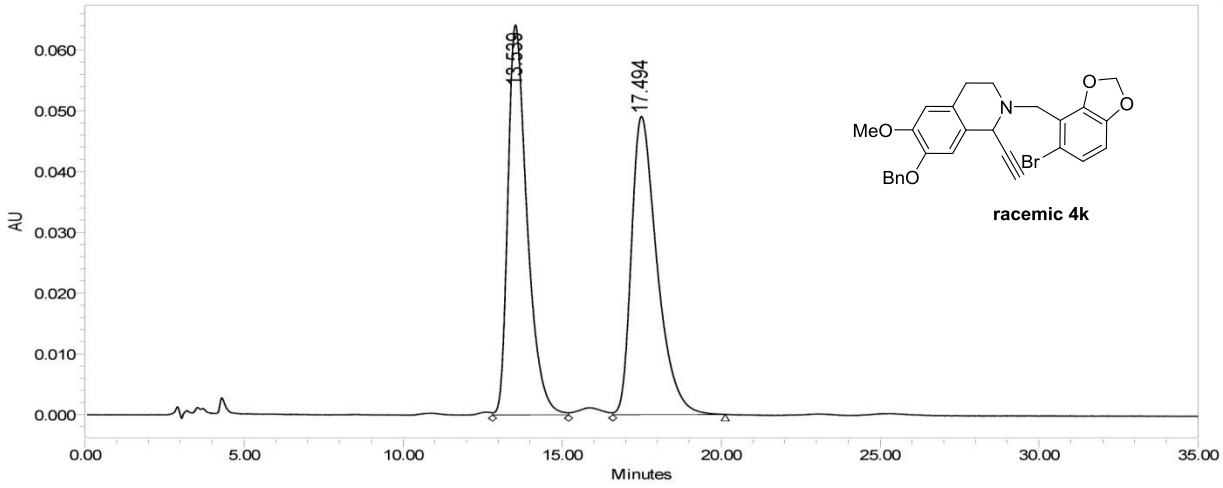
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	13.661	155060	1.04	3822	1.46
2	17.319	14721853	98.96	257250	98.54

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq4-93-1a1 ((90/10 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/16/2016 1:28:02 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	12	Date Processed:	12/15/2016 9:06:42 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	35.00 Minutes	Sample Set Name	



racemic 4k

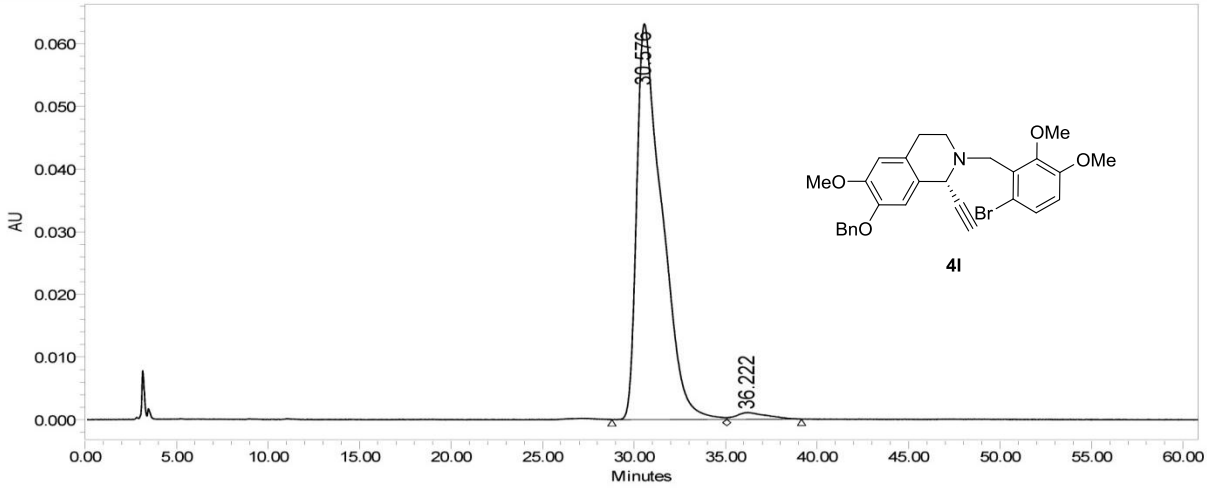
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	13.539	2694388	49.51	64174	56.67
2	17.494	2747913	50.49	49069	43.33

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq6-149-1a ((875/125 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/15/2016 5:19:34 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	1	Date Processed:	12/15/2016 8:48:35 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	80.00 Minutes	Sample Set Name	



4l

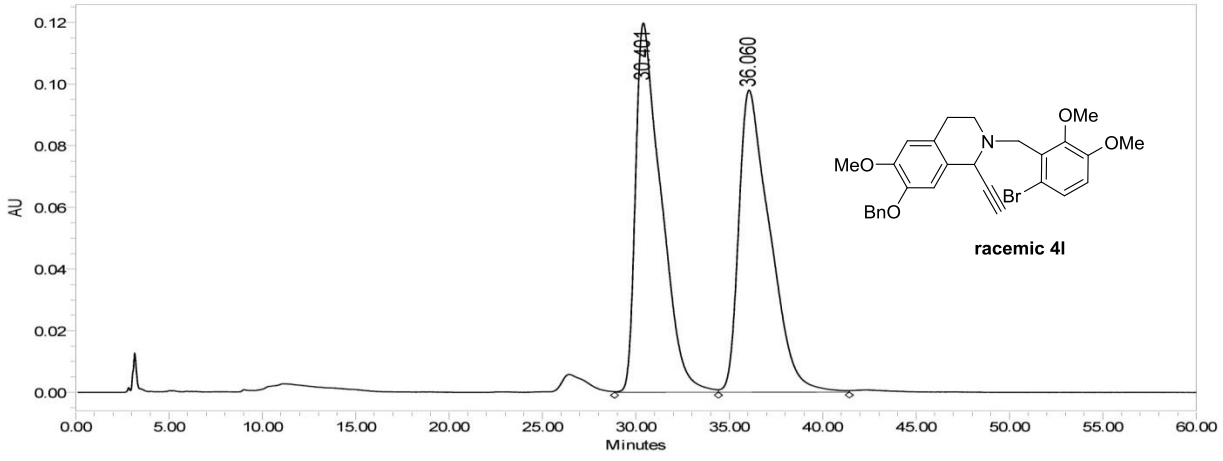
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	30.576	6023778	97.95	63143	98.29
2	36.222	126337	2.05	1101	1.71

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq4-89-1a ((875/125 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/15/2016 6:22:28 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	2	Date Processed:	12/15/2016 8:50:41 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



racemic 4l

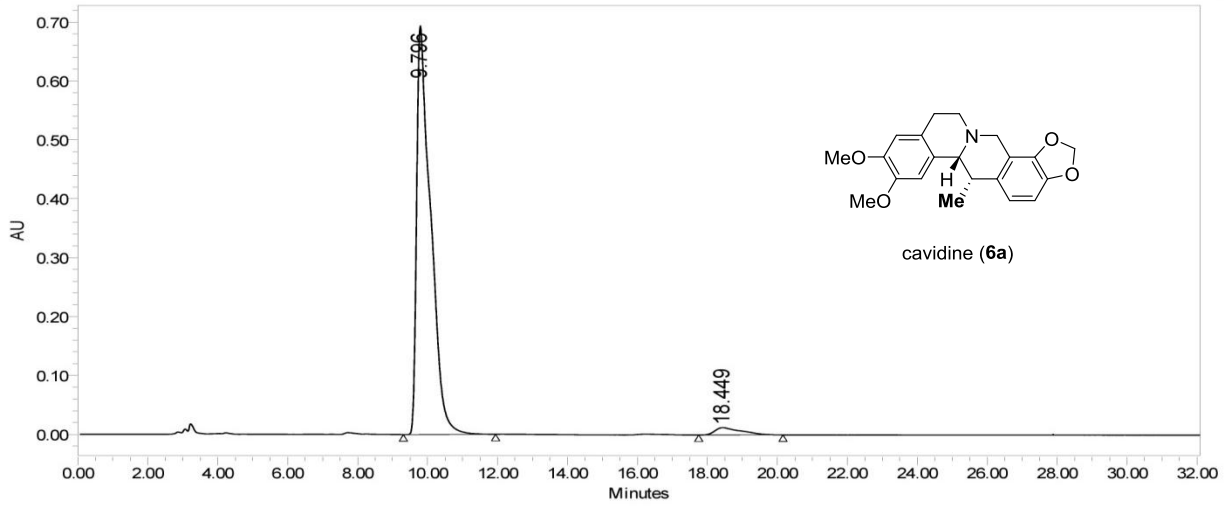
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	30.401	11381724	50.13	119767	55.00
2	36.060	11322054	49.87	97994	45.00

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq6-130-1a (95/5 AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	11/5/2016 4:58:49 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	3	Date Processed:	12/15/2016 8:16:04 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	35.00 Minutes	Sample Set Name	

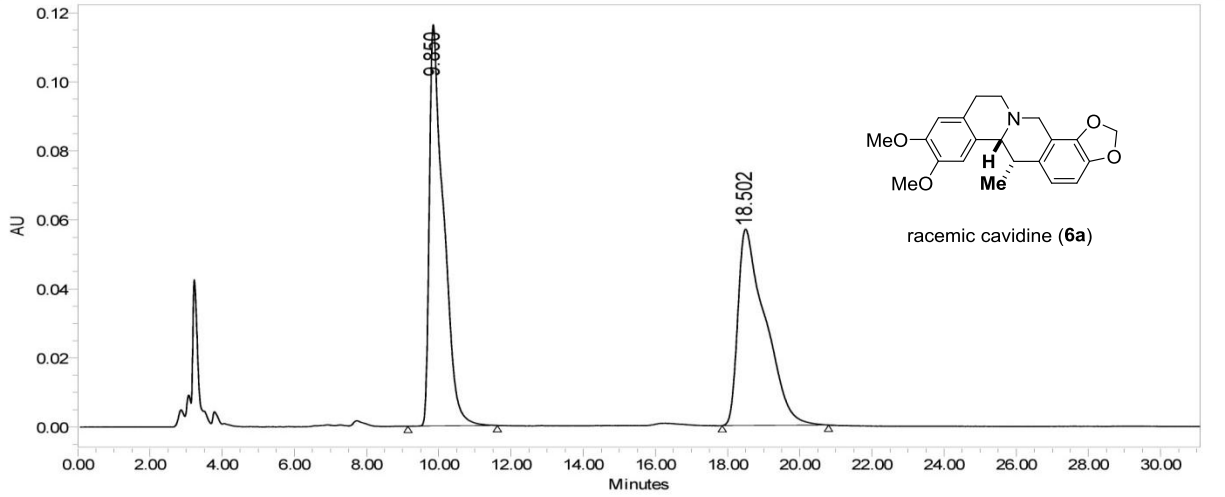


cavidine (**6a**)

	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	9.796	19267790	96.81	694007	98.25
2	18.449	633947	3.19	12370	1.75

SAMPLE INFORMATION

Sample Name:	Yzsq2-120-1a (95/5 AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	11/5/2016 5:31:24 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	4	Date Processed:	12/15/2016 8:17:22 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	35.00 Minutes	Sample Set Name	



racemic cavidine (**6a**)

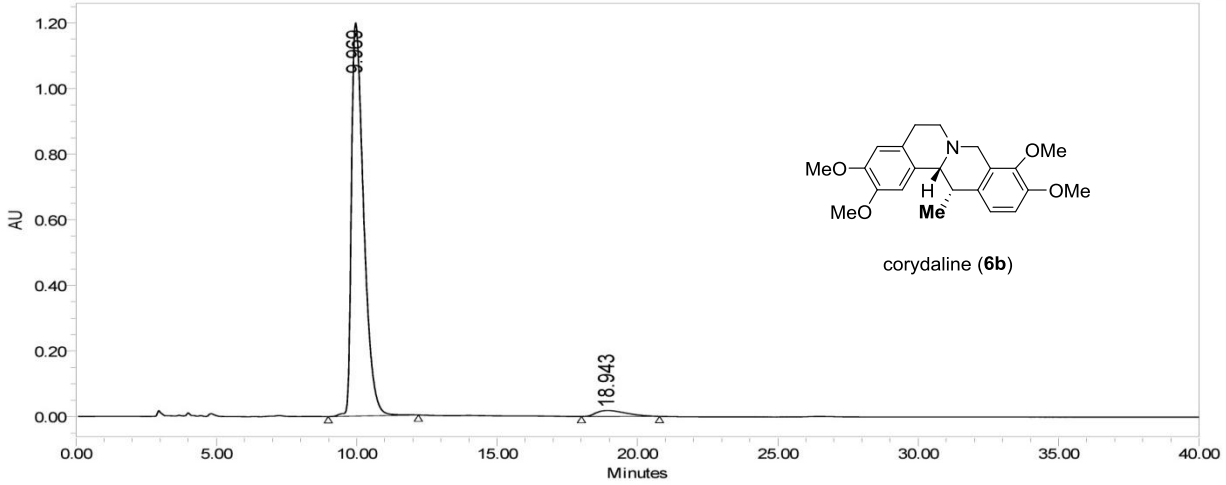
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	9.850	3158595	51.22	116275	67.14
2	18.502	3007599	48.78	56914	32.86

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq6-121-1-b(95/5 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	9/25/2016 12:09:47 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	1	Date Processed:	12/15/2016 7:49:43 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	40.00 Minutes	Sample Set Name	



corydaline (**6b**)

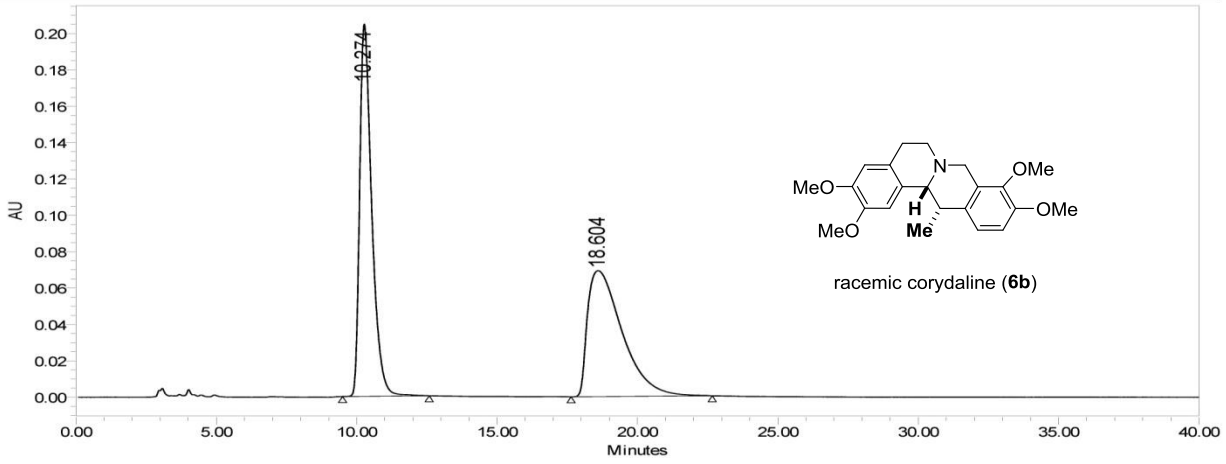
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	9.969	36316295	96.43	1198317	98.51
2	18.943	1346398	3.57	18101	1.49

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq2-109-1-b(95/5 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	9/25/2016 1:39:02 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	3	Date Processed:	12/15/2016 7:51:09 AM HKT
Injection Volume:	5.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	40.00 Minutes	Sample Set Name	



racemic corydaline (**6b**)

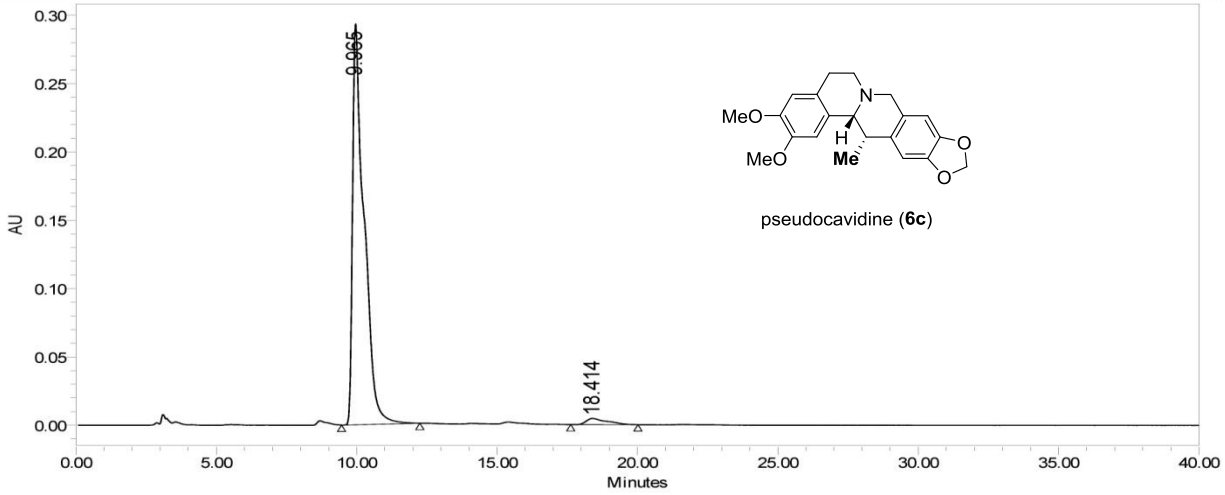
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	10.274	6134925	50.85	204635	74.71
2	18.604	5929826	49.15	69256	25.29

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq6-128-1f(90/10 AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	9/30/2016 4:31:36 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	5	Date Processed:	12/15/2016 8:32:15 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	40.00 Minutes	Sample Set Name	



pseudocavidine (**6c**)

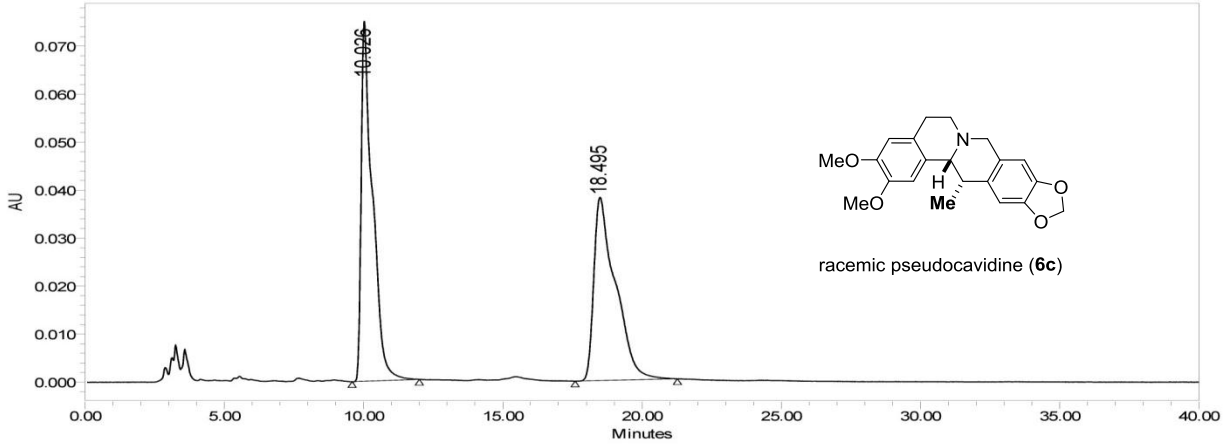
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	9.965	8316950	97.31	293256	98.49
2	18.414	229982	2.69	4504	1.51

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq3-174-1f(90/10 AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	9/30/2016 5:17:10 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	6	Date Processed:	12/15/2016 8:33:55 AM HKT
Injection Volume:	15.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	40.00 Minutes	Sample Set Name	



racemic pseudocavidine (**6c**)

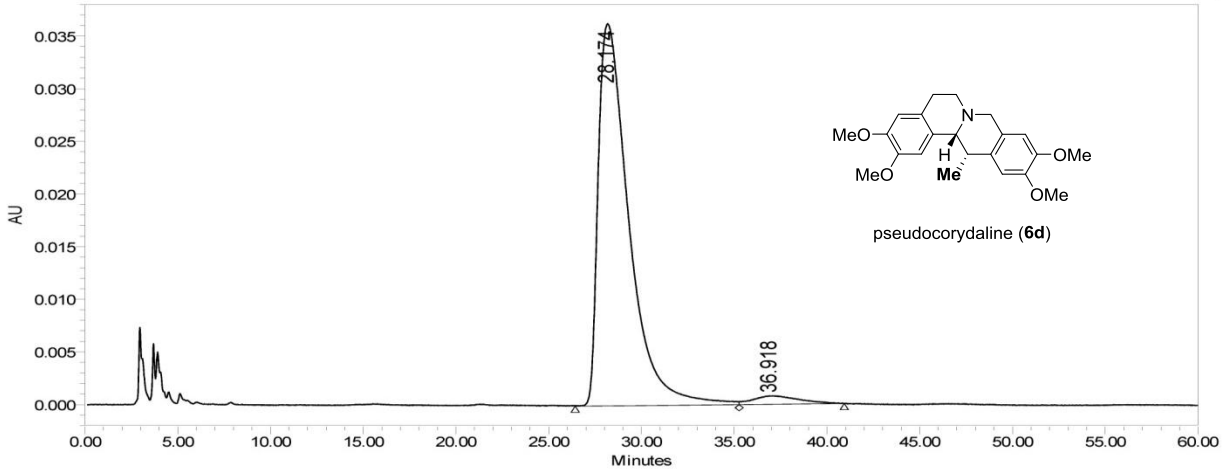
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	10.026	2116860	50.65	74982	66.26
2	18.495	2062234	49.35	38174	33.74

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq6-124-1a(97.5/2.5 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	9/28/2016 9:34:22 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	4	Date Processed:	12/15/2016 8:04:18 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



pseudocorydaline (**6d**)

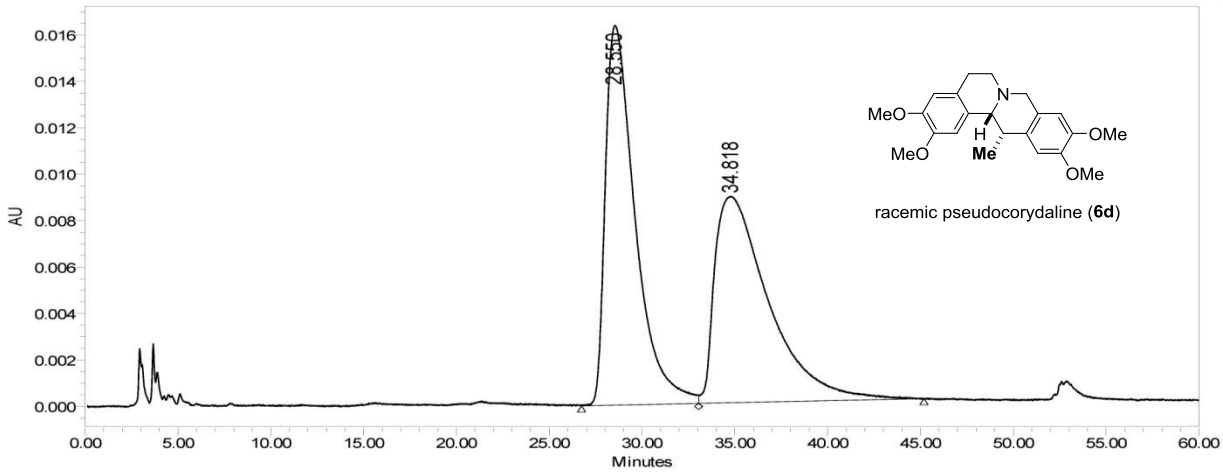
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	28.174	4172335	96.76	36322	97.80
2	36.918	139661	3.24	817	2.20

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq2-127-1b(97.5/2.5 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	9/28/2016 10:36:36 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	5	Date Processed:	12/15/2016 8:07:25 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



racemic pseudocorydaline (**6d**)

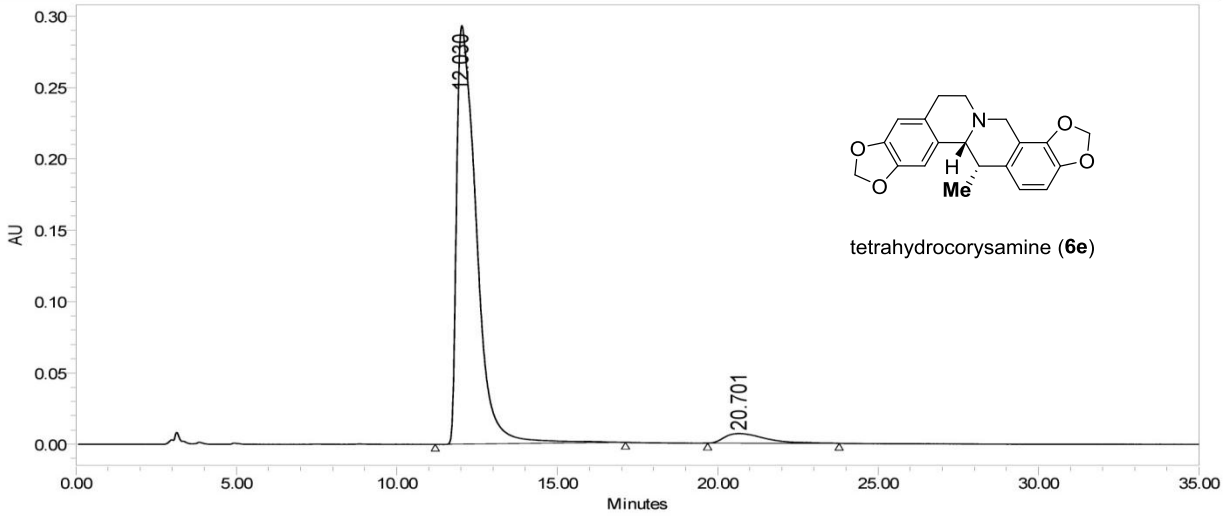
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	28.550	1826027	50.34	16354	64.79
2	34.818	1801592	49.66	8889	35.21

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq6-138-1b2 (200/1 AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	11/19/2016 5:26:35 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	5	Date Processed:	12/15/2016 5:32:18 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	35.00 Minutes	Sample Set Name	



tetrahydrocorysamine (**6e**)

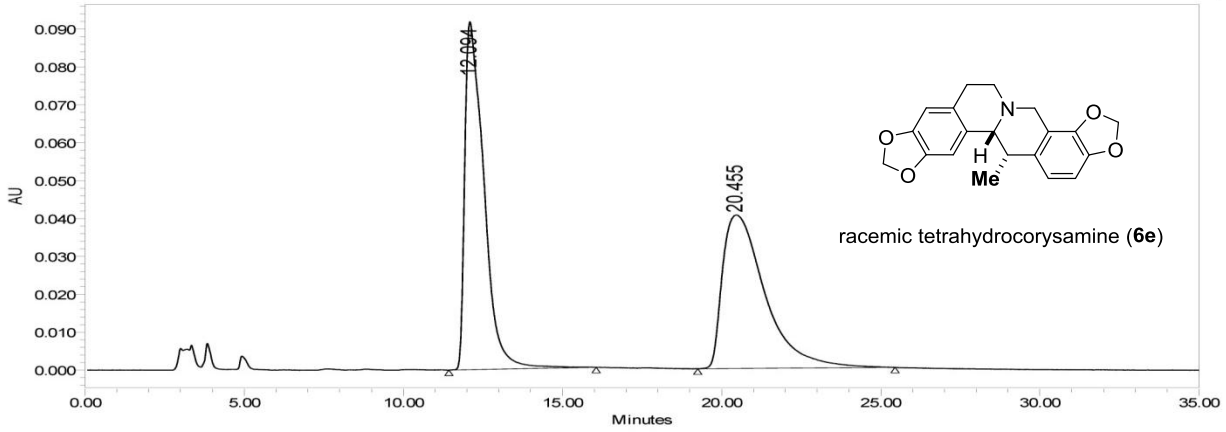
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	12.030	12505553	95.44	293172	97.75
2	20.701	597662	4.56	6760	2.25

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq2-130-1b3 (200/1 AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	11/19/2016 6:02:08 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	6	Date Processed:	12/15/2016 5:33:55 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	35.00 Minutes	Sample Set Name	



racemic tetrahydrocorysamine (**6e**)

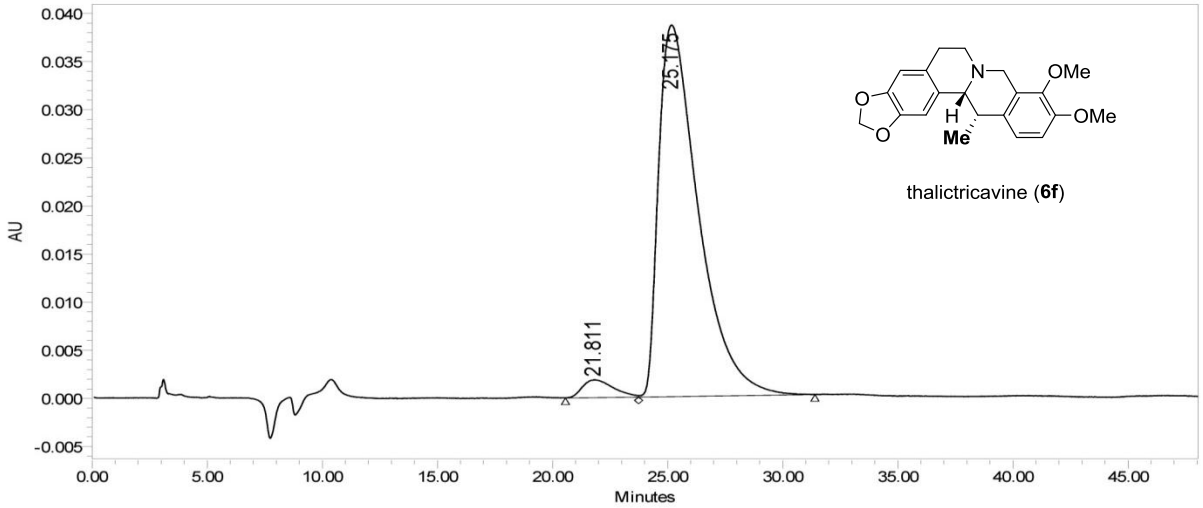
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	12.094	3832575	50.40	91808	69.39
2	20.455	3771173	49.60	40506	30.61

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq6-135-1a10 (250/1Et2NH	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/26/2016 5:53:47 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	10	Date Processed:	12/15/2016 6:41:55 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch3 230nm@1.2nm
Run Time:	50.00 Minutes	Sample Set Name	



thalictricavine (**6f**)

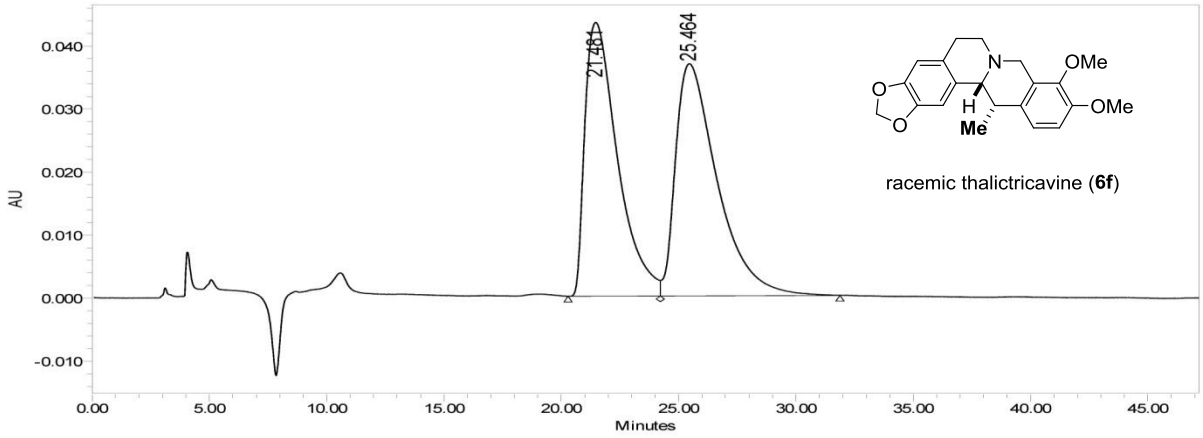
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	21.811	173006	3.59	1875	4.63
2	25.175	4640033	96.41	38618	95.37

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq2-112-1a11 (250/1Et2NH	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/26/2016 6:42:26 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	11	Date Processed:	12/15/2016 6:40:53 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch3 230nm@1.2nm
Run Time:	50.00 Minutes	Sample Set Name	



racemic thalictricavine (**6f**)

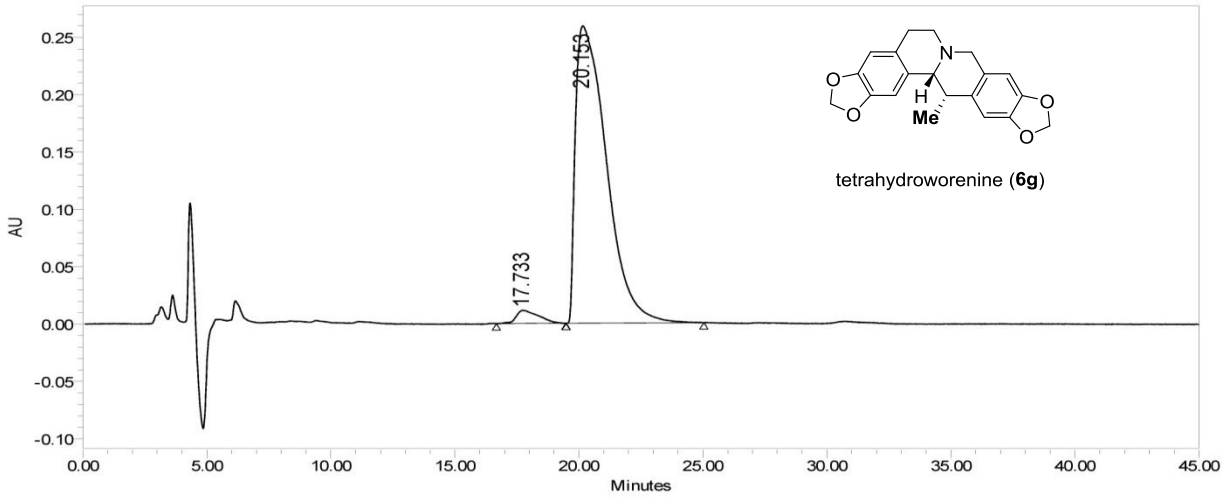
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	21.481	4192186	47.91	43446	54.11
2	25.464	4557091	52.09	36848	45.89

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq6-144-1a(100/1+Et2NH AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/8/2016 11:18:41 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	4	Date Processed:	12/15/2016 7:20:08 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	45.00 Minutes	Sample Set Name	



tetrahydroworenine (**6g**)

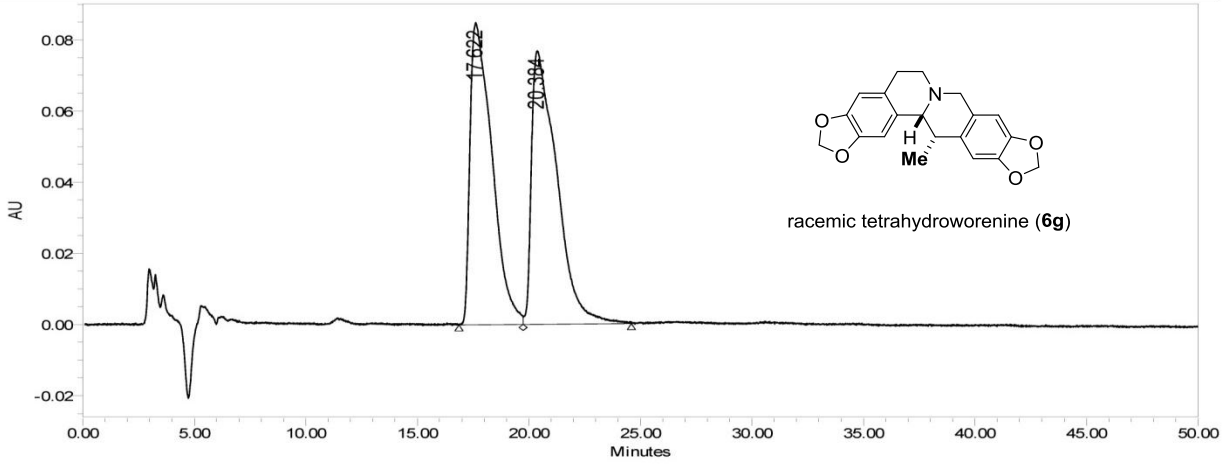
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	17.733	716144	3.08	11560	4.27
2	20.153	22517632	96.92	259235	95.73

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq3-96-1c(100/1+Et2NH AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/8/2016 9:27:12 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	2	Date Processed:	12/15/2016 7:21:45 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	50.00 Minutes	Sample Set Name	



racemic tetrahydroworenine (**6g**)

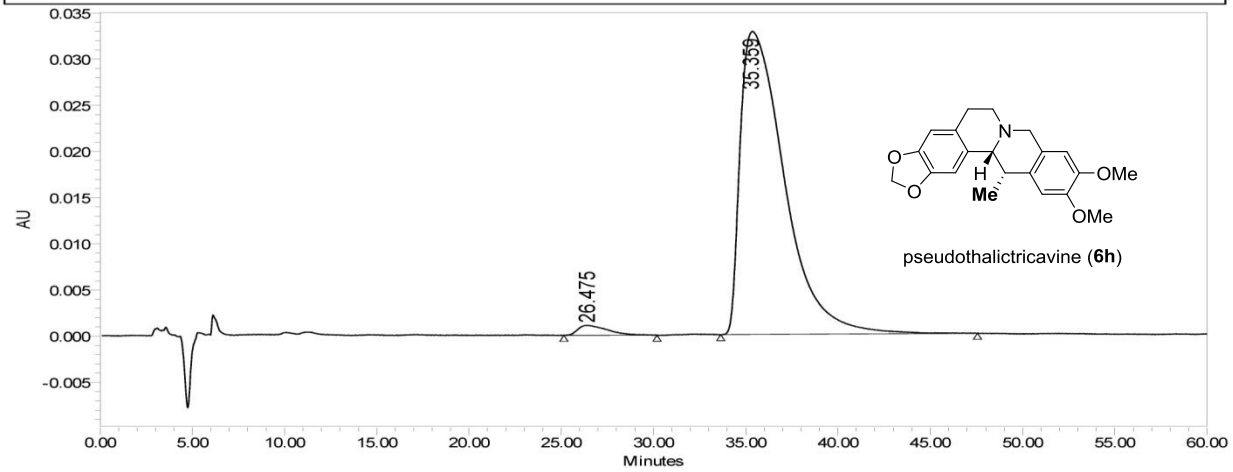
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	17.622	5807591	49.02	84969	52.50
2	20.384	6038713	50.98	76879	47.50

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq6-148-1a (99/1+Et2NH AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/9/2016 6:08:52 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	2	Date Processed:	12/15/2016 7:04:45 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch3 230nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



pseudothalictricavine (**6h**)

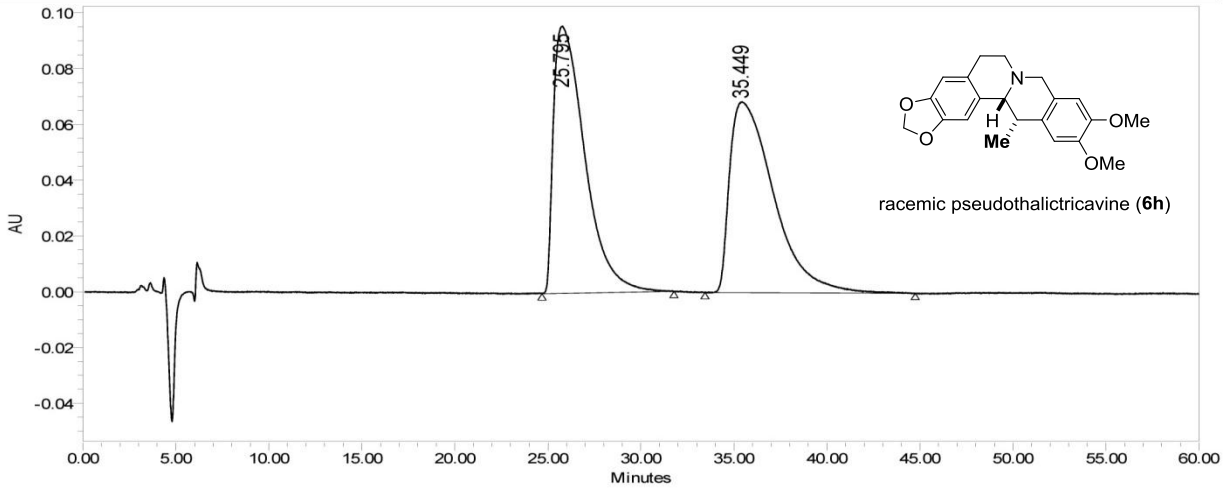
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	26.475	120313	2.15	1088	3.21
2	35.359	5476794	97.85	32841	96.79

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq2-130-2 (99/1+Et2NH AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/9/2016 7:09:29 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	3	Date Processed:	12/15/2016 7:06:37 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



racemic pseudothalictricavine (**6h**)

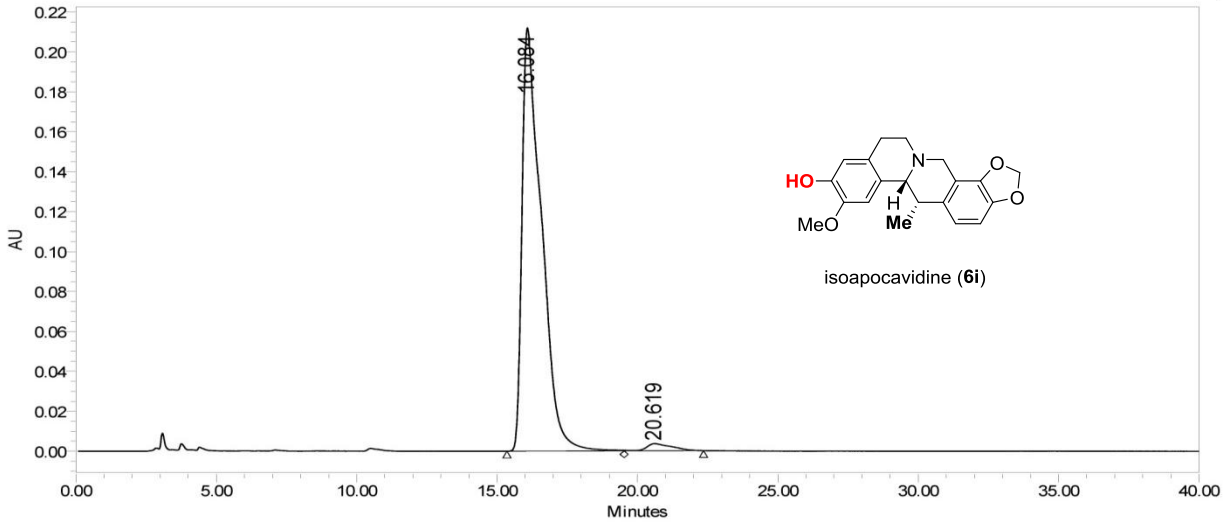
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	25.795	11225243	49.94	95877	58.35
2	35.449	11252716	50.06	68449	41.65

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq6-153-1a3 (95/5 AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	11/18/2016 3:16:04 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	4	Date Processed:	12/15/2016 9:29:16 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	40.00 Minutes	Sample Set Name	



isoapocavidine (**6i**)

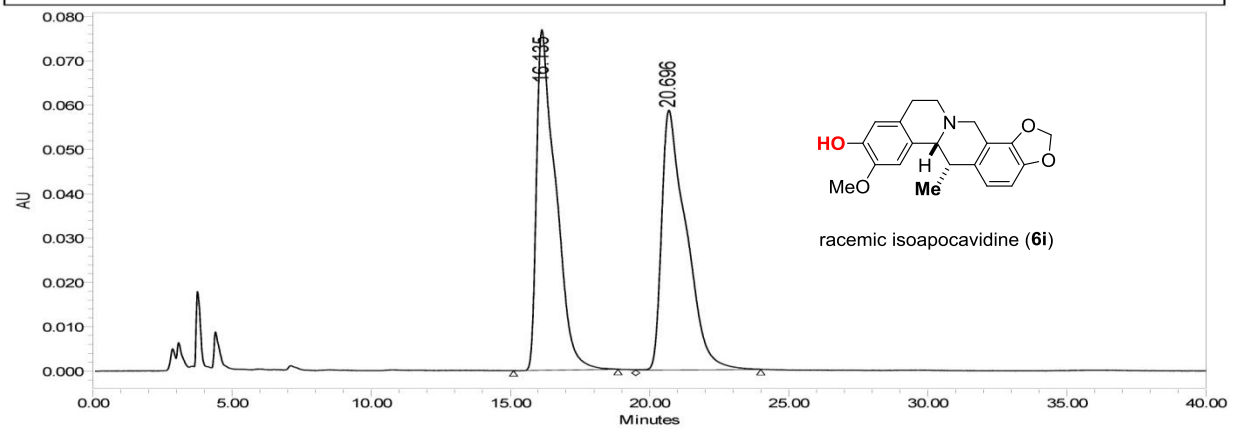
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	16.084	10201069	97.82	211971	98.31
2	20.619	227842	2.18	3654	1.69

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq4-121-2a3 (95/5 AD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	11/18/2016 4:00:09 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	5	Date Processed:	12/15/2016 9:31:14 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	40.00 Minutes	Sample Set Name	



racemic isoapocavidine (**6i**)

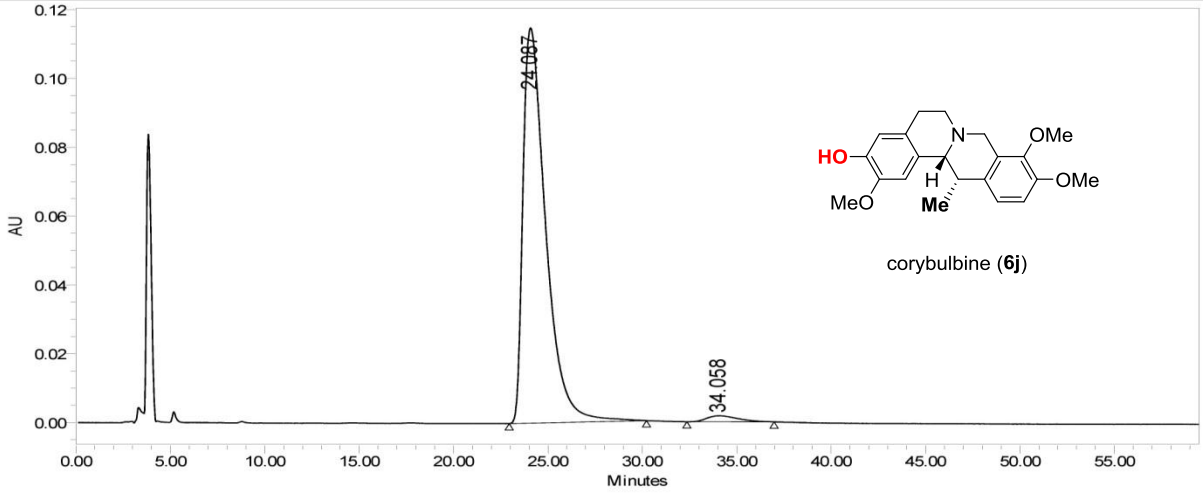
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	16.135	3661828	50.42	76854	56.73
2	20.696	3600902	49.58	58620	43.27

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq6-152-1a (97.5/2.5 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/12/2016 7:16:12 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	2	Date Processed:	12/15/2016 9:25:47 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



corybulbine (**6j**)

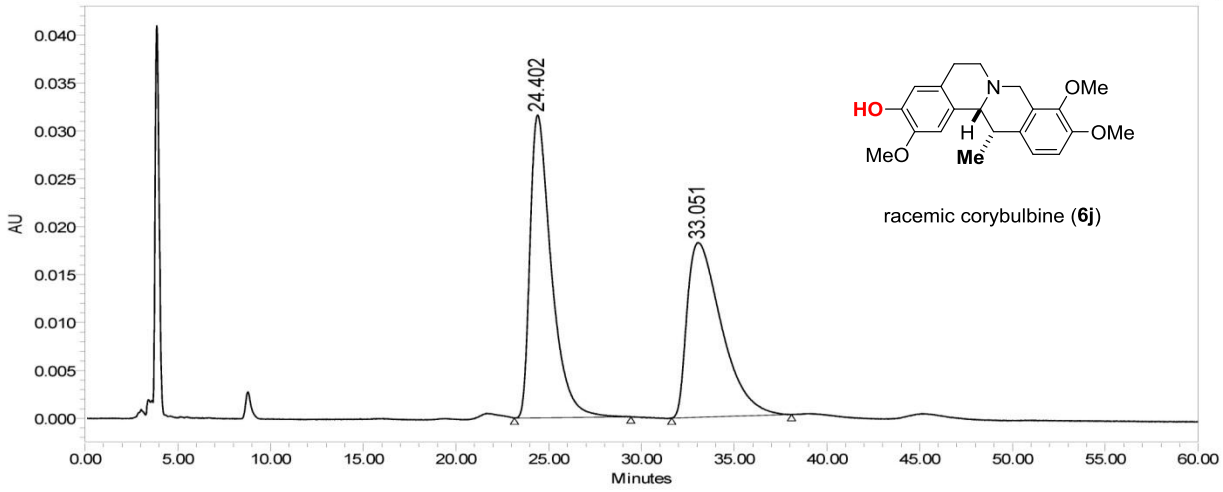
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	24.087	9613188	97.93	114839	98.48
2	34.058	202788	2.07	1777	1.52

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq4-96-2a (97.5/2.5 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/12/2016 8:16:15 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	3	Date Processed:	12/15/2016 9:23:50 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	60.00 Minutes	Sample Set Name	



racemic corybulbine (**6j**)

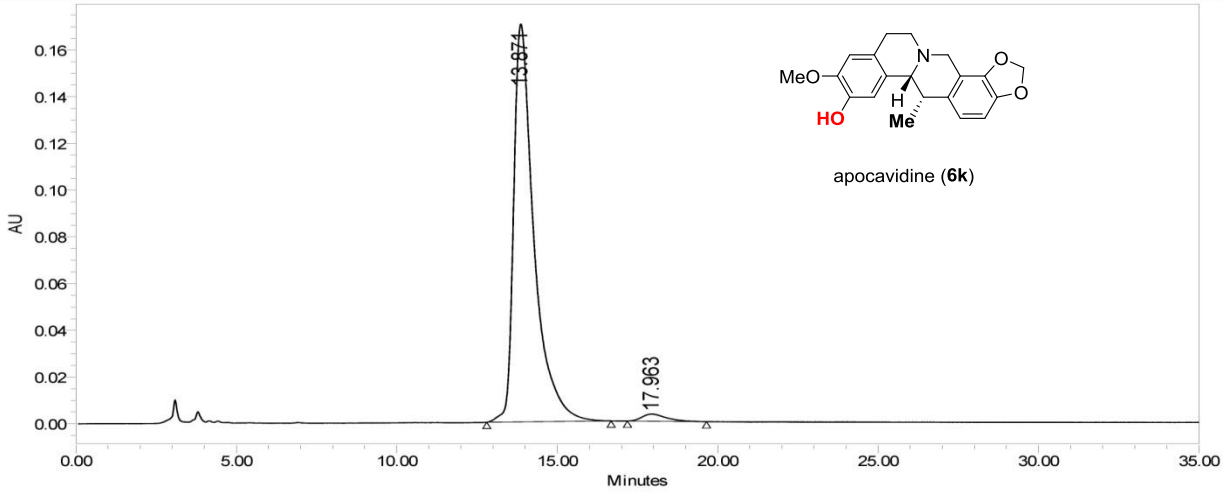
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	24.402	2528143	51.77	31608	63.41
2	33.051	2354978	48.23	18241	36.59

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq6-157-1a ((95/5 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/19/2016 4:48:53 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	2	Date Processed:	12/15/2016 9:08:59 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	35.00 Minutes	Sample Set Name	



apocavidine (**6k**)

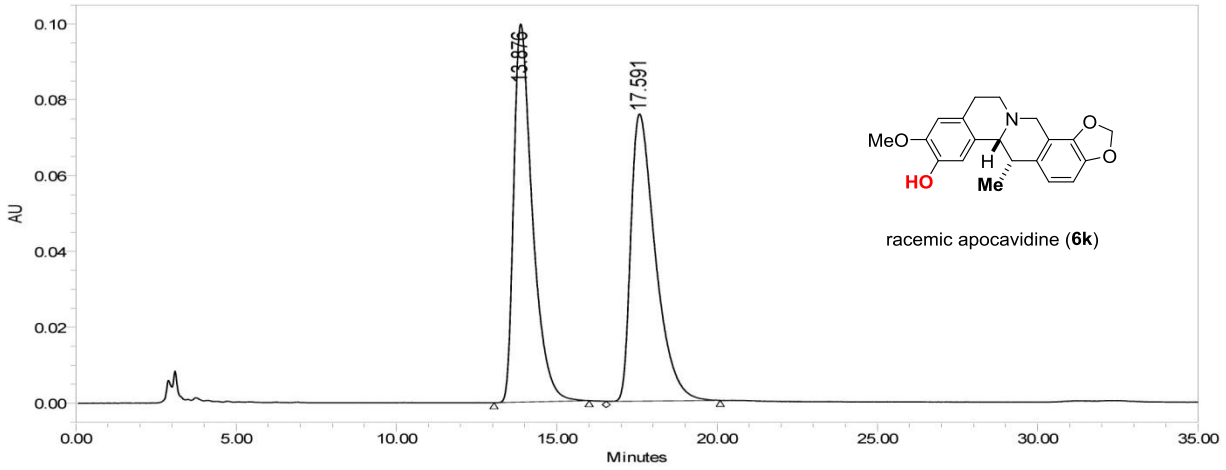
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	13.871	7486077	97.90	170276	98.23
2	17.963	160500	2.10	3067	1.77

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq4-95-2 ((95/5 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/19/2016 5:28:11 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	3	Date Processed:	12/15/2016 9:10:32 AM HKT
Injection Volume:	10.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	35.00 Minutes	Sample Set Name	



racemic apocavidine (**6k**)

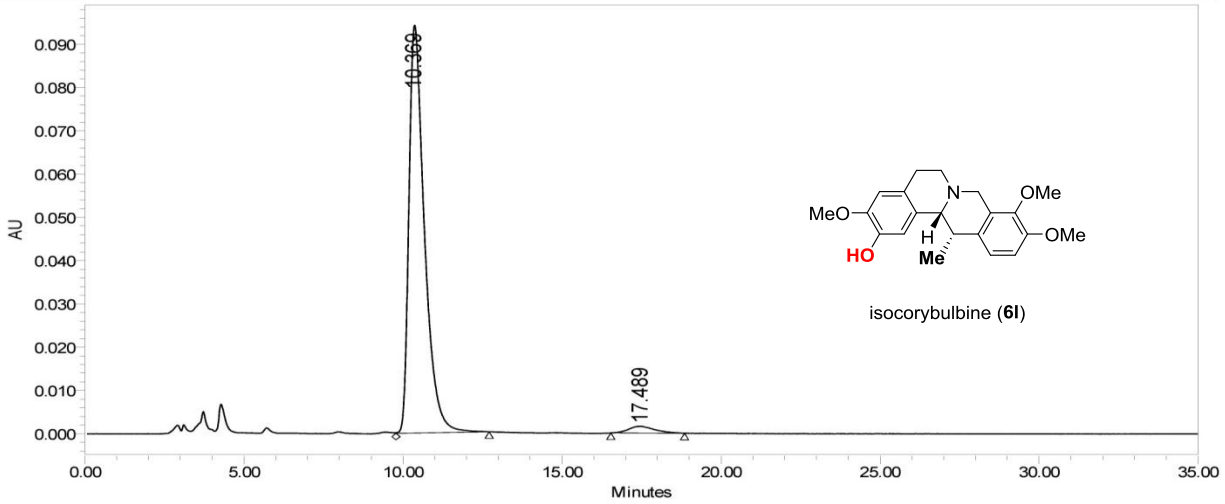
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	13.876	4045621	50.05	99719	56.82
2	17.591	4037926	49.95	75783	43.18

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	Yzsq6-156-1c ((90/10 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/18/2016 4:28:03 PM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	3	Date Processed:	12/15/2016 9:00:02 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	35.00 Minutes	Sample Set Name	



isocorybulbine (**6l**)

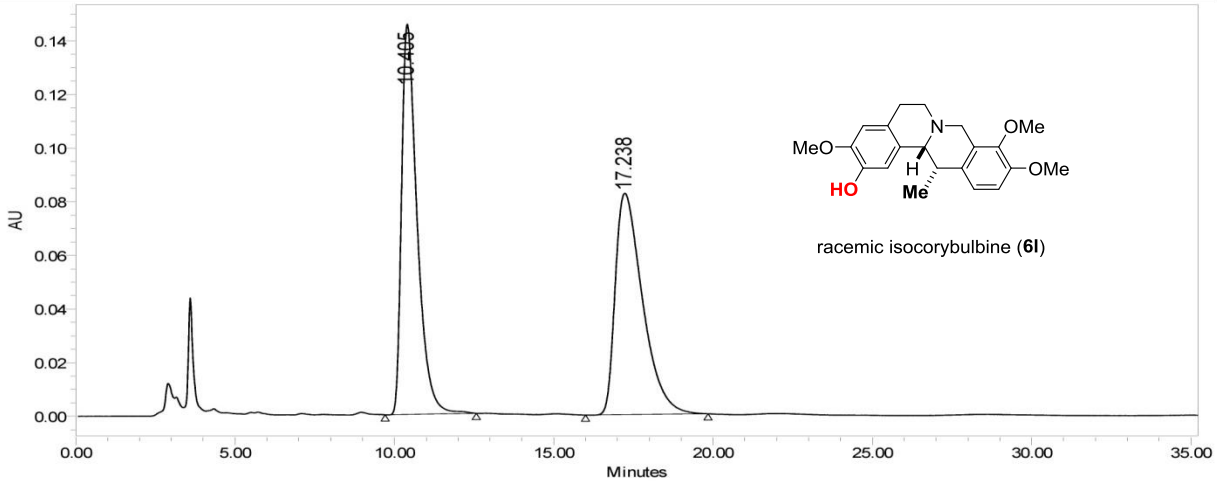
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	10.369	3186626	97.25	94205	98.34
2	17.489	90048	2.75	1586	1.66

Project Name Defaults
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	zsq4-92-2a ((90/10 OD)	Acquired By:	Breeze
Sample Type:	Unknown	Date Acquired:	10/18/2016 11:35:45 AM HKT
Vial:	1	Acq. Method:	zsq project 2
Injection #:	2	Date Processed:	12/15/2016 9:02:41 AM HKT
Injection Volume:	20.00 ul	Channel Name:	2998 Ch2 214nm@1.2nm
Run Time:	40.00 Minutes	Sample Set Name	



racemic isocorybulbine (**6l**)

	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	10.405	4928479	50.05	145459	63.81
2	17.238	4919345	49.95	82506	36.19