Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Zinc Diiodide-Promoted Synthesis of Trisubstituted Allenes From Propargylic Amines

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Supporting Information

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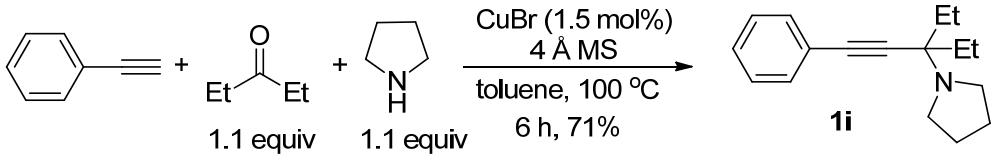
General Information. CuBr (98.5%) was purchased from Sinopharm Chemical Reagent Co., Ltd and used without further treatment. ZnI_2 (98%) was purchased from Acros and kept in a glove box. 4 Å molecular sieves was purchased from Alfa Aesar and kept in glove box after activation (heated at 450 ^oC for 10 h in Muffle furnace, taken out after cooling to 200 ^oC and then kept in a glove box to allow to cool to room temperature) under N_2. Toluene was dried over sodium wire with benzophenone as the indicator and distilled freshly before use. Anhydrous benzene was purchased from Aladdin and used without further treatment. Other reagents were used without further treatment. All the temperatures are referred to the oil baths used.

Propargylic amines were all synthesized according to our previously reported method.[1] **1a-1h**, **1k-1n**, and **1p** are known compounds. [1]

Part I Synthesis of propargylic amines **1i-1l**, **1q**, **1s-1u**, and **1w-1z**.

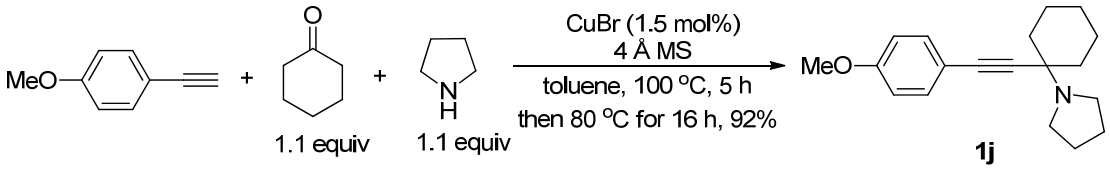
(1) Synthesis of 1-phenyl-3-ethylpent-1-yn-3-yl)pyrrolidine **1i** (kjq-2-175)



Typical Procedure I: To a dried Schlenk tube was added 4 Å MS (2.9995 g). The Schlenk tube was then dried under vacuum with a heating gun. CuBr (0.0215 g, 0.15 mmol), phenylacetylene (1.0208 g, 10 mmol)/toluene (3 mL), 3-pentanone (0.9471 g, 11 mmol)/toluene (3 mL), and pyrrolidine (0.7820 g, 11 mmol)/toluene (4 mL) were then added sequentially under Ar atmosphere. The Schlenk tube was then placed in a pre-heated oil bath at 100 ^oC with stirring for 6 h as monitored by TLC. After cooling to room temperature, the crude reaction mixture was filtrated through a short pad of silica gel with a sand-core funnel eluted with acetone (50 mL). After evaporation, the residue was purified by chromatography on silica gel to afford **1i** (1.7092 g, 71%) (eluent: petroleum ether/ethyl acetate = 50/1; then petroleum ether/ethyl acetate = 20/1) as a liquid: ^1H NMR (400 MHz, $CDCl_3$) δ 7.44-7.38 (m, 2 H, Ar-H), 7.31-7.23 (m, 3 H, Ar-H), 2.79-2.72 (m, 4 H, CH_2NCH_2), 1.85-1.67 (m, 8 H, 4×CH_2), 0.96 (t, *J* = 7.4 Hz, 6 H, 2×CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 131.7, 128.1, 127.5, 123.7,

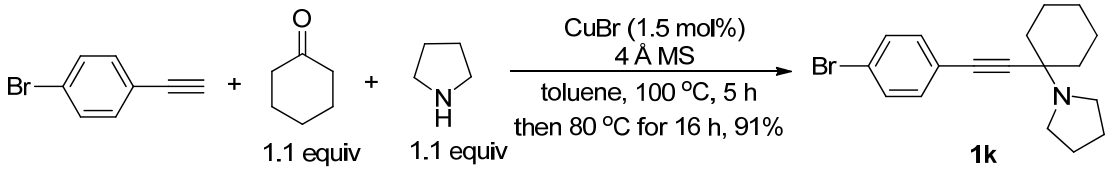
91.4, 84.8, 61.9, 47.4, 28.8, 23.6, 8.1; MS (ESI) *m/z* 242 ($M+H^+$), 171 ($M+H^+$-pyrrolidine); IR (neat): 3080, 3056, 2968, 2876, 2808, 1598, 1489, 1458, 1443, 1376, 1335, 1324, 1280, 1247, 1191, 1157, 1144, 1120, 1070, 1029, 1009 cm^{-1}; HRMS calcd for $C_{17}H_{24}N$ ($M+H^+$): 242.1903. Found: 242.1904.

(2) Synthesis of 1-(1-(4-methoxyphenylethynyl)cyclohexyl)pyrrolidine **1j** (kjq-2-23)



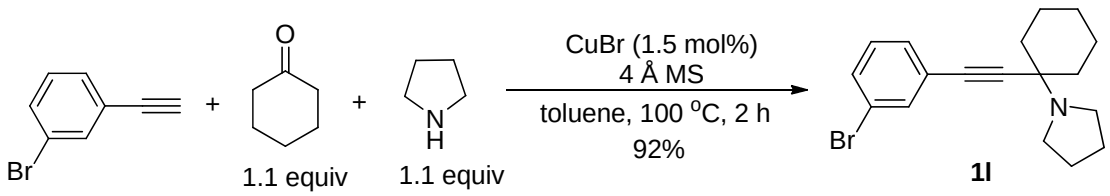
Typical Procedure II: To a dried Schlenk tube was added 4 Å MS (3.0150 g). The Schlenk tube was then dried under vacuum with a heating gun. CuBr (0.0215 g, 0.15 mmol), 4-methoxyphenylacetylene (1.3225 g, 10 mmol)/toluene (3 mL), cyclohexanone (1.0770 g, 11 mmol)/toluene (3 mL), and pyrrolidine (0.7830 g, 11 mmol)/toluene (4 mL) were then added sequentially under Ar atmosphere. The Schlenk tube was then placed in a pre-heated oil bath at 100 ^oC with stirring for 5 h and 80 ^oC for additional 16 h. After cooling to room temperature, the crude reaction mixture was filtrated through a short pad of silica gel with a sand-core funnel eluted with acetone (50 mL). After evaporation, the residue was purified by chromatography on silica gel to afford **1j** (2.6171 g, 92%) (eluent: petroleum ether; then petroleum ether/ethyl acetate = 4/1; finally petroleum ether/ethyl acetate = 1/1) as a yellow solid: m.p. 52-54 ^oC (petroleum ether & ethyl ether); ^1H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.8 Hz, 2 H, Ar-H), 6.81 (d, *J* = 8.8 Hz, 2 H, Ar-H), 3.79 (s, 3 H, OCH_3), 2.78 (t, *J* = 5.8 Hz, 4 H, CH_2NCH_2), 2.06-1.96 (m, 2 H, 2 H in Cy), 1.85-1.73 (m, 4 H, 2×CH_2), 1.72-1.57 (m, 5 H, 2×CH_2 + one proton of CH_2), 1.56-1.45 (m, 2 H, CH_2), 1.29-1.16 (m, 1 H, one proton of CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.1, 133.0, 115.9, 113.7, 88.7, 85.7, 59.3, 55.2, 47.0, 37.9, 25.7, 23.5, 23.0; MS (ESI) *m/z* 284 ($M+H^+$), 213 ($M+H^+$-pyrrolidine); IR (neat): 2924, 2855, 2803, 1604, 1570, 1507, 1439, 1285, 1243, 1173, 1154, 1123, 1106, 1034 cm^{-1}; Anal. calcd for $C_{19}H_{25}NO$ (%): C 80.52, H 8.89, N 4.94; Found: C 80.34, H 9.04, N 4.91.

(3) Synthesis of 1-(1-(4-bromophenylethynyl)cyclohexyl)pyrrolidine **1k** (kjq-2-22)



Following **Typical Procedure II**, the reaction of 4 Å MS (3.0100 g), CuBr (0.0213 g, 0.15 mmol), 4-bromophenylacetylene (1.8135 g, 10 mmol)/toluene (3 mL), cyclohexanone (1.0813 g, 11 mmol)/toluene (3 mL), and pyrrolidine (0.7816 g, 11 mmol)/toluene (4 mL) afforded **1k** (3.0359 g, 91%) (eluent: petroleum ether; then petroleum ether/ethyl acetate = 4/1; finally petroleum ether/ethyl acetate = 1/1) as a yellow solid: m.p. 85-87 °C (petroleum ether & ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, *J* = 8.4 Hz, 2 H, Ar-H), 7.28 (d, *J* = 8.4 Hz, 2 H, Ar-H), 2.84-2.72 (m, 4 H, CH_2NCH_2), 2.06-1.96 (m, 2 H, 2 H in Cy), 1.86-1.46 (m, 11 H, 5×CH_2 + one proton of CH_2), 1.30-1.17 (m, 1 H, one proton of CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 133.2, 131.4, 122.6, 121.7, 91.9, 85.0, 59.3, 47.0, 37.8, 25.6, 23.5, 23.0; MS (ESI) *m/z* 334 (M(^{81}Br)+H^+), 332 (M(^{79}Br)+H^+); IR (neat): 2932, 2857, 2798, 1482, 1446, 1390, 1283, 1123, 1069, 1010 cm^{-1}; Anal. calcd for $C_{18}H_{22}NBr$ (%): C 65.06, H 6.67, N 4.22; Found: C 64.96, H 6.64, N 4.30.

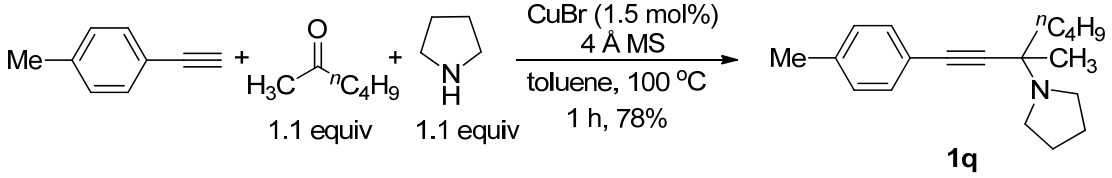
(4) Synthesis of 1-(1-(3-bromophenylethynyl)cyclohexyl)pyrrolidine **1l** (kjq-2-26)



Following **Typical Procedure I**, the reaction of 4 Å MS (3.0100 g), CuBr (0.0220 g, 0.15 mmol), 3-bromophenylacetylene (1.8071 g, 10 mmol)/toluene (3 mL), cyclohexanone (1.0777 g, 11 mmol)/toluene (3 mL), and pyrrolidine (0.7820 g, 11 mmol)/toluene (4 mL) afforded **1l** (3.0583 g, 92%) (eluent: petroleum ether; then petroleum ether/ethyl acetate = 4/1; finally petroleum ether/ethyl acetate = 1/1) as a yellow solid: m.p. 76-78 °C (petroleum ether & ethyl acetate); ^{1}H NMR (400 MHz,

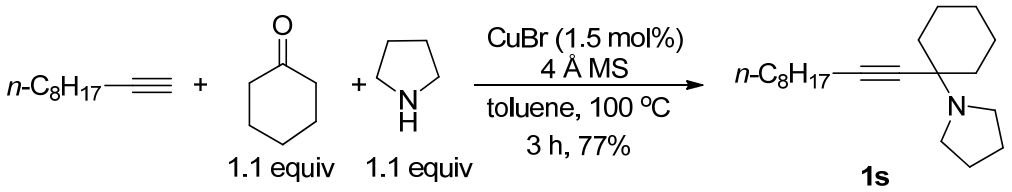
$CDCl_3$) δ 7.57 (t, *J* = 1.8 Hz, 1 H, Ar-H), 7.43-7.38 (m, 1 H, Ar-H), 7.35 (dt, *J* = 7.8, 1.2 Hz, 1 H, Ar-H), 7.15 (t, *J* = 8.0 Hz, 1 H, Ar-H), 2.77 (t, *J* = 6.6 Hz, 4 H, CH_2NCH_2), 2.06-1.98 (m, 2 H, 2 H in Cy), 1.85-1.75 (m, 4 H, 2×CH_2), 1.75-1.58 (m, 5 H, 2×CH_2 + one proton of CH_2), 1.58-1.47 (m, 2 H, CH_2), 1.30-1.17 (m, 1 H, one proton of CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.2, 130.6, 130.1, 129.4, 125.5, 121.8, 92.0, 84.4, 59.0, 46.9, 37.6, 25.5, 23.3, 22.8; MS (ESI) *m/z* 334 (M(^{81}Br)+H^+), 332 (M(^{79}Br)+H^+); IR (neat): 3031, 2945, 2927, 2851, 2825, 1589, 1556, 1473, 1447, 1404, 1331, 1290, 1260, 1224, 1155, 1123, 1089, 1072, 1041 cm^{-1}; Anal. calcd for $C_{18}H_{22}NBr$ (%): C 65.06, H 6.67, N 4.22; Found: C 65.10, H 6.79, N 4.19.

(5) Synthesis of 1-(1-(4-methylphenyl)-3-methylhept-1-yn-3-yl)pyrrolidine **1q** (tangxj-4-143)



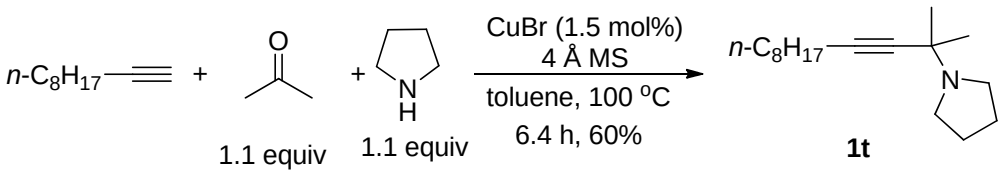
Following **Typical Procedure I**, the reaction of 4 Å MS (3.0031 g), CuBr (0.0219 g, 0.15 mmol), 4-methylphenylacetylene (1.1602 g, 10 mmol)/toluene (3 mL), 2-hexanone (1.1001 g, 11 mmol)/toluene (3 mL), and pyrrolidine (0.7830 g, 11 mmol)/toluene (4 mL) afforded **1q** (2.0992 g, 78%) (eluent: petroleum ether; then petroleum ether/ethyl acetate = 150/1; finally petroleum ether/ethyl acetate/Et_3N = 900/180/1) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, *J* = 8.0 Hz, 2 H, Ar-H), 7.08 (d, *J* = 8.0 Hz, 2 H, Ar-H), 2.85-2.72 (m, 4 H, CH_2NCH_2), 2.33 (s, 3 H, $ArCH_3$), 1.87-1.73 (m, 5 H, 2×CH_2 + one proton of CH_2), 1.67 (td, *J* = 12.2, 5.2 Hz, 1 H, one proton of CH_2), 1.57-1.29 (m, 7 H, 2×CH_2 + CH_3), 0.94 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.5, 131.5, 128.8, 120.5, 90.5, 84.2, 57.8, 47.6, 41.2, 26.5, 25.8, 23.6, 23.1, 21.3, 14.0; MS (ESI) *m/z* 270 (M+H^+), 199 (M+H^+-pyrrolidine); IR (neat): 3028, 2956, 2870, 2807, 2358, 1509, 1461, 1370, 1302, 1264, 1190, 1144, 1091, 1038, 1000 cm^{-1}; HRMS calcd for $C_{19}H_{27}N$ [M^+]: 269.2143. Found: 269.2151.

(6) Synthesis of 1-(1-(dec-1-yn-1-yl)cyclohexyl)pyrrolidine **1s** (kjq-2-33)



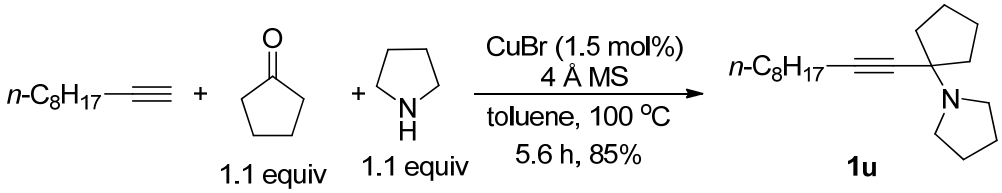
Following **Typical Procedure I**, the reaction of 4 Å MS (3.0009 g), CuBr (0.0219 g, 0.15 mmol), 1-decyne (1.3790 g, 10 mmol)/toluene (3 mL), cyclohexanone (1.0770 g, 11 mmol)/toluene (3 mL), and pyrrolidine (0.7825 g, 11 mmol)/toluene (4 mL) afforded **1s** (2.2322 g, 77%) (eluent: petroleum ether/ethyl acetate = 10/1; then petroleum ether/ethyl acetate = 3/1) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.75-2.62 (m, 4 H, CH_2NCH_2), 2.25-2.17 (m, 2 H, CH_2), 1.93-1.82 (m, 2 H, 2 H in Cy), 1.80-1.69 (m, 4 H, 2×CH_2), 1.67-1.06 (m, 20 H, 10×CH_2), 0.87 (t, J = 5.2 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 85.8, 80.0, 58.8, 46.8, 38.1, 31.8, 29.3, 29.2, 29.0, 28.7, 25.7, 23.4, 23.0, 22.6, 18.6, 14.1; MS (ESI) *m/z* 290 ($M+H^+$); IR (neat): 2931, 2855, 2809, 1447, 1378, 1328, 1282, 1263, 1224, 1163, 1127, 1079, 1014 cm^{-1}; HRMS calcd for $C_{20}H_{36}N$ [$M+H^+$]: 290.2842. Found: 290.2849.

(7) Synthesis of 1-(2-methyldodec-3-yn-2-yl)pyrrolidine **1t** (kjq-2-46)



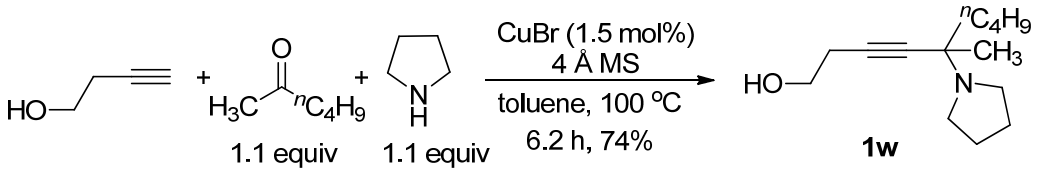
Following **Typical Procedure I**, the reaction of 4 Å MS (1.5008 g), CuBr (0.0115 g, 0.080 mmol), 1-decyne (0.6905 g, 5 mmol)/toluene (2 mL), acetone (0.4 mL, d = 0.80 g/mL, 0.3200 g, 5.5 mmol), and pyrrolidine (0.3915 g, 5.5 mmol)/toluene (3 mL) afforded **1t** (0.7426 g, 60%) (eluent: petroleum ether; then petroleum ether/ethyl acetate = 5/1) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.69 (t, J = 6.2 Hz, 4 H, CH_2NCH_2), 2.18 (t, J = 7.0 Hz, 2 H, CH_2), 1.83-1.73 (m, 4 H, 2×CH_2), 1.52-1.19 (m, 18 H, 2×CH_3 + 6×CH_2), 0.88 (t, J = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 83.6, 81.3, 53.6, 47.9, 31.8, 29.9, 29.2, 29.1, 29.0, 28.7, 23.6, 22.6, 18.5, 14.0; MS (ESI) *m/z* 250 ($M+H^+$); IR (neat): 2960, 2928, 2856, 2809, 1459, 1377, 1359, 1328, 1223, 1185, 1118, 1074, 1014 cm^{-1}; HRMS calcd for $C_{17}H_{32}N$ [$M+H^+$]: 250.2529. Found: 250.2535.

(8) Synthesis of 1-(1-(dec-1-yn-1-yl)cyclopentyl)pyrrolidine **1u** (kjq-2-50)



Following **Typical Procedure I**, the reaction of 4 Å MS (3.0150 g), CuBr (0.0210 g, 0.15 mmol), 1-decyne (1.3830 g, 10 mmol)/toluene (3 mL), cyclopentanone (0.9248 g, 11 mmol)/toluene (3 mL), and pyrrolidine (0.7820 g, 11 mmol)/toluene (4 mL) afforded **1u** (2.3531 g, 85%) (eluent: petroleum ether/ethyl acetate = 10/1; then petroleum ether/ethyl acetate = 5/1) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.67 (t, J = 6.2 Hz, 4 H, CH_2NCH_2), 2.19 (t, J = 6.8 Hz, 2 H, CH_2), 1.96-1.65 (m, 12 H, 6×CH_2), 1.52-1.21 (m, 12 H, 6×CH_2), 0.88 (t, J = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 84.5, 80.9, 65.0, 48.9, 40.6, 31.7, 29.19, 29.16, 29.0, 28.7, 23.5, 23.3, 22.6, 18.5, 14.0; MS (ESI) *m/z* 276 ($M+H^+$); IR (neat): 2961, 2929, 2871, 2856, 2810, 1458, 1378, 1354, 1321, 1213, 1149, 1084 cm^{-1}; HRMS calcd for $C_{19}H_{34}N$ [$M+H^+$]: 276.2686. Found: 276.2692.

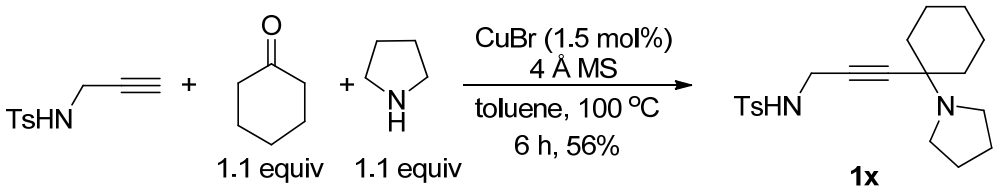
(9) Synthesis of 5-methyl-5-(pyrrolidin-1-yl)non-3-yn-1-ol **1w** (kjq-2-45)



Following **Typical Procedure I**, the reaction of 4 Å MS (3.0100 g), CuBr (0.0214 g, 0.15 mmol), 3-butyn-1-ol (0.6997 g, 10 mmol)/toluene (3 mL), 2-hexanone (1.0961 g, 11 mmol)/toluene (3 mL), and pyrrolidine (0.7808 g, 11 mmol)/toluene (4 mL) afforded **1w** (1.6608 g, 74%) (eluent: petroleum ether/ethyl acetate = 10/1, 330 mL; then petroleum ether/ethanol = 10/1, 880 mL) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.67 (t, J = 6.6 Hz, 2 H, OCH_2), 2.72-2.60 (m, 4 H, CH_2NCH_2), 2.47 (t, J = 6.4 Hz, 2 H, C≡CCH_2), 2.17 (brs, 1 H, OH), 1.81-1.70 (m, 4 H, 2×CH_2), 1.68-1.48 (m, 2 H, CH_2), 1.46-1.22 (m, 7 H, 2×CH_2 + CH_3), 0.90 (t, J = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 83.2, 80.3, 61.3, 57.4, 47.5, 41.1, 26.6, 25.7, 23.4, 23.0, 22.9,

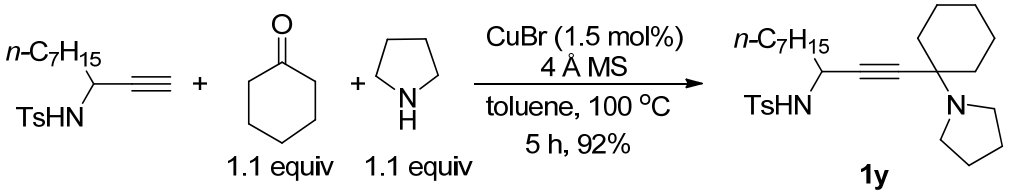
14.0; MS (ESI) *m/z* 224 ($M+H^+$); IR (neat): 3419, 2956, 2872, 1466, 1375, 1333, 1261, 1241, 1180, 1147, 1118, 1052 cm^{-1}; HRMS calcd for $C_{14}H_{26}NO$ [$M+H^+$]: 224.2009. Found: 224.2010.

(10) Synthesis of *N*-(3-(1-(pyrrolidin-1-yl)cyclohexyl)prop-2-yn-1-yl)-4-methylbenzenesulfonamide **1x** (kjq-2-96)



Following **Typical Procedure I**, the reaction of 4 Å MS (2.9998 g), CuBr (0.0218 g, 0.15 mmol), *N*-(prop-2-yn-1-yl)-*p*-toluenesulfonamide (2.0907 g, 10 mmol)/toluene (3 mL), cyclohexanone (1.0796 g, 11 mmol)/toluene (3 mL), and pyrrolidine (0.7820 g, 11 mmol)/toluene (4 mL) afforded **1x** (2.0160 g, 56%) (eluent: DCM/MeOH = 100/1; then DCM/MeOH = 15/1) as a solid: m.p. 130-132 °C (petroleum ether & ethyl acetate); ^1H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.0 Hz, 2 H, Ar-H), 7.31 (d, *J* = 7.8 Hz, 2 H, Ar-H), 4.87 (brs, 1 H, NH), 3.93 (s, 2 H, C≡CCH_2), 2.60-2.46 (m, 4 H, CH_2NCH_2), 2.43 (s, 3 H, CH_3), 1.78-1.61 (m, 6 H, 3×CH_2), 1.60-1.44 (m, 3 H, CH_2 + one proton of CH_2), 1.41-1.23 (m, 4 H, 2×CH_2), 1.20-1.06 (m, 1 H, one proton of CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.2, 136.9, 129.5, 127.0, 84.4, 80.0, 58.6, 46.6, 37.0, 32.8, 25.2, 23.1, 22.5, 21.3; MS (ESI) *m/z* 361 ($M+H^+$); IR (neat): 3279, 2932, 2855, 1598, 1495, 1448, 1330, 1289, 1263, 1160, 1094, 1049 cm^{-1}; Anal. calcd for $C_{20}H_{28}N_2O_2S$ (%): C 66.63, H 7.83, N 7.77; Found: C 66.41, H 7.86, N 7.46.

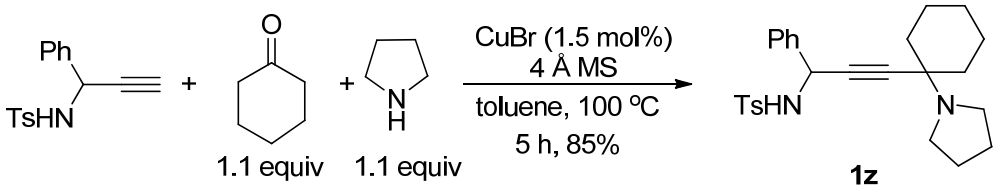
(11) Synthesis of *N*-(1-(1-(pyrrolidin-1-yl)cyclohexyl)dec-1-yn-3-yl)-4-methyl benzenesulfonamide **1w** (kjq-2-116)



Following **Typical Procedure I**, the reaction of 4 Å MS (1.5050 g), CuBr (0.0108 g, 0.075 mmol), *N*-(dec-1-yn-3-yl)-*p*-toluenesulfonamide (1.5372 g, 5 mmol), cyclohexanone (0.5400 g, 5.5 mmol)/toluene (2 mL), and pyrrolidine (0.3910 g, 5.5

mmol)/toluene (3 mL) afforded **1y** (2.1143 g, 92%) (eluent: ethyl acetate/DCM = 2/1) as a solid: m.p. 60-62 ^{o}C (petroleum ether & ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.4 Hz, 2 H, Ar-H), 7.29 (d, *J* = 8.4 Hz, 2 H, Ar-H), 4.77-4.62 (m, 1 H, NH), 4.13 (q, *J* = 7.3 Hz, 1 H, C≡CCH), 2.52-2.36 (m, 7 H, CH_2NCH_2 + CH_3), 1.75-1.01 (m, 26 H, 13×CH_2), 0.88 (t, *J* = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.0, 137.7, 129.4, 127.0, 84.4, 83.9, 58.3, 46.6, 45.7, 37.6, 37.3, 31.6, 29.0, 28.8, 25.3, 23.2, 22.6, 22.5, 21.4, 14.0; MS (ESI) *m/z* 459 ($M+H^+$); IR (neat): 3269, 2930, 2856, 1599, 1496, 1448, 1334, 1286, 1263, 1184, 1162, 1126, 1095, 1064 cm^{-1}; Anal. calcd for $C_{27}H_{42}N_2O_2S$ (%): C 70.70, H 9.23, N 6.11; Found: C 70.53, H 9.47, N 6.06.

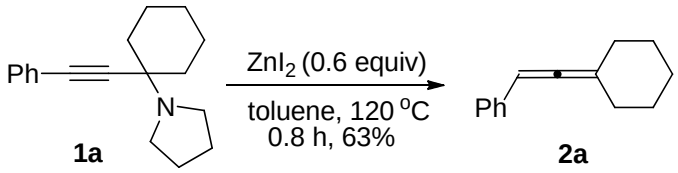
(12) Synthesis of *N*-(1-phenyl-3-(1-(pyrrolidin-1-yl)cyclohexyl)prop-2-yn-1-yl)-4-methylbenzenesulfonamide **1z** (kjq-2-117)



Following **Typical Procedure I**, the reaction of 4 Å MS (1.2100 g), CuBr (0.0087 g, 0.06 mmol), *N*-(dec-1-yn-3-yl)-*p*-toluenesulfonamide (1.1426 g, 4 mmol), cyclohexanone (0.4310 g, 4.4 mmol)/toluene (2 mL), and pyrrolidine (0.3128 g, 4.4 mmol)/toluene (2 mL) afforded **1z** (1.4802 g, 85%) (eluent: DCM/MeOH = 50/1; then DCM/MeOH = 15/1) as a solid: m.p. 145-147 ^{o}C (petroleum ether & ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.0 Hz, 2 H, Ar-H), 7.52 (d, *J* = 7.2 Hz, 2 H, Ar-H), 7.37-7.23 (m, 5 H, Ar-H), 5.41 (s, 1 H, C≡CCH), 5.25 (brs, 1 H, NH), 2.58-2.44 (m, 4 H, CH_2NCH_2), 2.41 (s, 3 H, CH_3), 1.76-1.60 (m, 6 H, 3×CH_2), 1.59-1.42 (m, 3 H, CH_2 + one proton of CH_2), 1.37-1.19 (m, 4 H, 2×CH_2), 1.19-1.04 (m, 1 H, one proton of CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.9, 138.4, 137.6, 129.3, 128.2, 127.8, 127.0, 126.9, 86.9, 82.2, 58.5, 48.8, 46.6, 37.1, 25.2, 23.1, 22.5, 21.3; MS (ESI) *m/z* 437 ($M+H^+$); IR (neat): 3268, 3062, 3031, 2933, 2855, 1599, 1494, 1450, 1332, 1306, 1288, 1264, 1160, 1126, 1092, 1068, 1044, 1021 cm^{-1}; Anal. calcd for $C_{26}H_{32}N_2O_2S$ (%): C 71.52, H 7.39, N 6.42; Found: C 71.44, H 7.53, N 6.30.

Part II ZnI_2-Promoted synthesis of trisubstituted allenes from propargylic amines.

(1) Synthesis of 1,1-pentamethylene-3-phenylpropadiene **2a** (kjq-2-37)

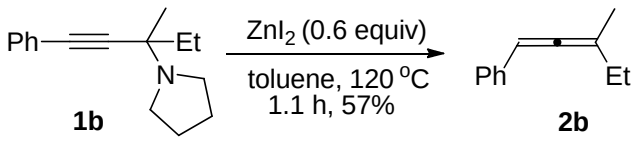


1a **2a**

Typical Procedure III: To a flame-dried Schlenk tube was added anhydrous ZnI_2 (191.3 mg, 0.6 mmol). The Schlenk tube was then taken out and dried under vacuum with a heating gun. **1a** (252.9 mg. 1.0 mmol) and 3 mL of toluene were added under the atmosphere of Ar. The Schlenk tube was then equipped with a condenser and placed in a pre-heated oil bath of 120 °C with stirring for 0.8 h as monitored by TLC. After cooling to room temperature, the crude reaction mixture was filtrated through a short pad of silica gel eluted with ethyl acetate (15 mL). After evaporation, the residue was purified by chromatography on silica gel (eluent: 30-60 °C petroleum ether) to afford **2a**[2] (116.7 mg, 63%) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32-7.23 (m, 4 H, ArH), 7.18-7.10 (m, 1 H, ArH), 6.02-5.96 (m, 1 H, =CH), 2.32-2.12 (m, 4 H, 2×CH_2), 1.77-1.48 (m, 6 H, 3×CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.6, 136.1, 128.5, 126.5, 126.2, 106.4, 92.3, 31.3, 27.7, 26.1; MS (EI) *m/z* 184 (M^+, 70.53), 141 (100); IR (neat) 3030, 2929, 2887, 2853, 1951, 1598, 1496, 1459, 1446, 1256, 1237, 1198, 1069, 1027 cm^{-1}.

The following compounds were prepared according to **Typical Procedure III.**

(2) Synthesis of 1-phenyl-3-methyl-1,2-pentadiene **2b** (kjq-2-38)

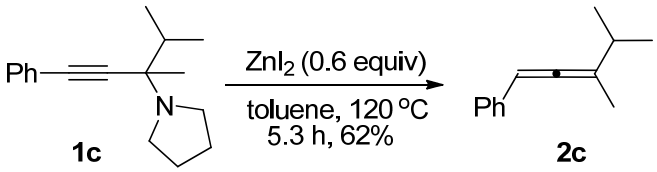


1b **2b**

The reaction of **1b** (226.3 mg, 1.0 mmol), ZnI_2 (191.6, 0.6 mmol), and toluene (3 mL) afforded allene **2b**[3] (90.3 mg, 57%) (eluent: 30-60 °C petroleum ether) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.23 (m, 4 H, ArH), 7.20-7.12 (m, 1 H, ArH), 6.12-6.05 (m, 1 H, =CH), 2.15-2.03 (m, 2 H, CH_2), 1.81 (d, *J* = 2.8 Hz, 3 H, =CCH_3),

1.06 (t, J = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.3, 136.1, 128.4, 126.4, 126.3, 105.4, 94.4, 27.2, 18.7, 12.3; MS (EI) *m/z* 158 (M^+, 70.99), 143 (100); IR (neat) 3083, 3064, 3029, 2967, 2933, 2905, 2846, 1950, 1597, 1496, 1458, 1400, 1368, 1327, 1218, 1206, 1150, 1073, 1028, 1002 cm^{-1}.

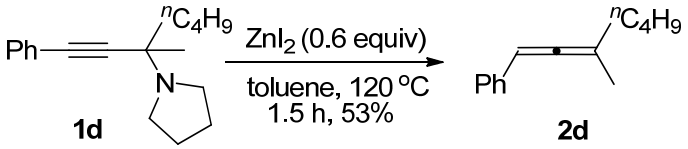
(3) Synthesis of 1-phenyl-3,4-dimethyl-1,2-pentadiene **2c** (kjq-2-39)



1c **2c**

The reaction of **1c** (240.8 mg, 1.0 mmol), ZnI_2 (191.0, 0.6 mmol), and toluene (3 mL) afforded allene **2c**[4] (105.8 mg, 62%) (eluent: 30-60 °C petroleum ether) as a liquid: ^1H NMR (400 MHz, $CDCl_3$) δ 7.31-7.25 (m, 4 H, ArH), 7.19-7.13 (m, 1 H, ArH), 6.08 (q, J = 2.7 Hz, 1 H, =CH), 2.29-2.21 (m, 1 H, CH in *i*-Pr), 1.81 (d, J = 3.2 Hz, 3 H, $=CCH_3$), 1.10 (d, J = 6.8 Hz, 3 H, CH_3), 1.08 (d, J = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.7, 136.1, 128.5, 126.34, 126.30, 109.8, 94.7, 32.5, 21.6, 21.5, 17.0; MS (EI) *m/z* 172 (M^+, 88.17), 129 (100); IR (neat) 3082, 3063, 3029, 2962, 2926, 2869, 1950, 1598, 1496, 1462, 1398, 1382, 1369, 1324, 1298, 1219, 1202, 1129, 1101, 1075, 1028 cm^{-1}.

(4) Synthesis of 1-phenyl-3-methyl-1,2-heptadiene **2d** (kjq-2-19)

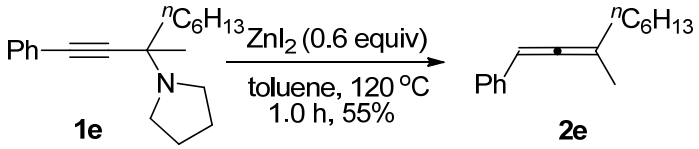


1d **2d**

The reaction of **1d** (254.5 mg, 1.0 mmol), ZnI_2 (192.5, 0.6 mmol), and toluene (3 mL) afforded allene **2d**[2] (97.7 mg, 53%) (eluent: petroleum ether) as a liquid: ^1H NMR (400 MHz, $CDCl_3$) δ 7.33-7.22 (m, 4 H, ArH), 7.20-7.12 (m, 1 H, ArH), 6.07-6.01 (m, 1 H, =CH), 2.12-2.02 (m, 2 H, $=CCH_2$), 1.80 (d, J = 2.8 Hz, 3 H, $=CCH_3$), 1.51-1.30 (m, 4 H, 2×CH_2), 0.89 (t, J = 7.2 Hz, 3 H, CH_3 in Bu); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.6, 136.1, 128.4, 126.5, 126.3, 103.7, 93.7, 33.8, 29.7, 22.4, 18.8, 13.9; MS (EI) *m/z* 186 (M^+, 1.92), 129 (100); IR (neat) 3029, 2957, 2929, 2872,

2859, 1951, 1598, 1496, 1464, 1399, 1378, 1369, 1227, 1200, 1151, 1071, 1028 cm^{-1}.

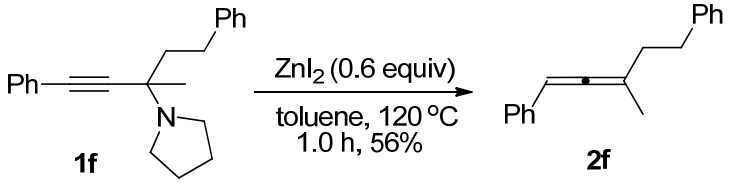
(5) Synthesis of 1-phenyl-3-methyl-1,2-nonadiene **2e** (kjq-2-41)



1e **2e**

The reaction of **1e** (282.5 mg, 1.0 mmol), ZnI_2 (191.0, 0.6 mmol), and toluene (3 mL) afforded allene **2e**[5] (116.5 mg, 55%) (eluent: 30-60 °C petroleum ether) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32-7.23 (m, 4 H, ArH), 7.19-7.12 (m, 1 H, ArH), 6.07-6.01 (m, 1 H, =CH), 2.12-2.02 (m, 2 H, $=CCH_2$), 1.80 (d, J = 2.4 Hz, 3 H, $=CCH_3$), 1.52-1.41 (m, 2 H, CH_2), 1.38-1.18 (m, 6 H, 2×CH_2), 0.86 (t, J = 6.4 Hz, 3 H, CH_3 in Hex); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.7, 136.1, 128.4, 126.5, 126.3, 103.7, 93.7, 34.1, 31.7, 29.1, 27.5, 22.6, 18.8, 14.1; MS (EI) *m/z* 214 (M^+, 0.69), 171 (M^+-C_3H_7, 3.70), 157 (M^+-C_4H_9, 5.74), 129 (100); IR (neat) 3064, 3029, 2956, 2926, 2856, 1951, 1598, 1496, 1465, 1399, 1369, 1210, 1149, 1072, 1028 cm^{-1}.

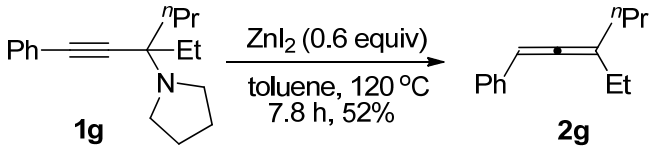
(6) Synthesis of 1-phenyl-3-ethyl-1,2-hexadiene **2f** (kjq-2-44)



1f **2f**

The reaction of **1f** (302.3 mg, 1.0 mmol), ZnI_2 (191.2, 0.6 mmol), and toluene (3 mL) afforded allene **2f**[6] (130.5 mg, 56%) (eluent: petroleum ether) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.09 (m, 10 H, ArH), 6.10-6.02 (m, 1 H, =CH), 2.80 (t, J = 7.8 Hz, 2 H, $PhCH_2$), 2.47-2.33 (m, 2 H, $=CCH_2$), 1.84 (d, J = 2.8 Hz, 3 H, $=CCH_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.7, 141.8, 135.7, 128.42, 128.38, 128.3, 126.5, 126.4, 125.8, 103.0, 94.4, 35.7, 33.8, 18.9; MS (EI) *m/z* 234 (M^+, 46.46), 128 (100); IR (neat) 3062, 3027, 2979, 2917, 2855, 1950, 1598, 1496, 1463, 1453, 1400, 1369, 1336, 1310, 1212, 1161, 1073, 1029 cm^{-1}.

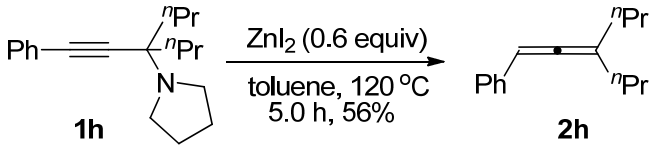
(7) Synthesis of 1-phenyl-3-ethyl-1,2-hexadiene **2g** (kjq-2-43)



1g 2g

The reaction of **1g** (256.0 mg, 1.0 mmol), ZnI_2 (191.7, 0.6 mmol), and toluene (3 mL) afforded allene **2g**[7] (97.3 mg, 52%) (eluent: 30-60 °C petroleum ether) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.26 (m, 4 H, ArH), 7.19-7.12 (m, 1 H, ArH), 6.15 (q, *J* = 3.0 Hz, 1 H, =CH), 2.15-2.00 (m, 4 H, 2×CH_2), 1.55-1.45 (m, 2 H, CH_2), 1.05 (t, *J* = 7.4 Hz, 3 H, CH_3), 0.93 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.0, 136.3, 128.5, 126.31, 126.29, 110.4, 95.8, 34.9, 25.8, 21.0, 14.0, 12.3; MS (EI) *m/z* 186 (M^+, 31.78), 129 (100); IR (neat) 3031, 2963, 2932, 2873, 1947, 1598, 1496, 1459, 1377, 1327, 1201, 1071, 1028 cm^{-1}.

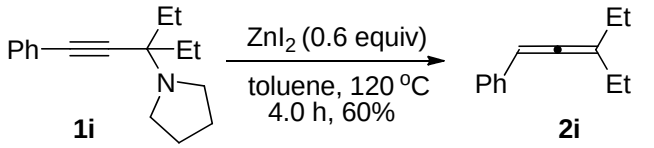
(8) Synthesis of 1-phenyl-3-(*n*-propyl)-1,2-hexadiene **2h** (kjq-2-170)



1h 2h

The reaction of **1h** (269.6 mg, 1.0 mmol), ZnI_2 (192.0, 0.6 mmol), and toluene (3 mL) afforded allene **2h**[5] (112.9 mg, 56%) (eluent: petroleum ether) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) 7.30-7.24 (m, 4 H, Ar-H), 7.18-7.12 (m, 1 H, Ar-H), 6.11 (q, *J* = 3.0 Hz, 1 H, =CH), 2.12-1.98 (m, 4 H, 2×CH_2), 1.57-1.43 (m, 4 H, 2×CH_2), 0.93 (t, *J* = 7.4 Hz, 6 H, 2×CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) 202.3, 136.2, 128.5, 126.4, 126.3, 108.4, 95.2, 34.9, 20.9, 14.0; MS (EI) *m/z* (%) 200 (M^+, 18.35), 129 (100); IR (neat): 3082, 3063, 3031, 2958, 2931, 2872, 1947, 1598, 1496, 1461, 1377, 1242, 1199, 1094, 1071, 1028 cm^{-1}.

(9) Synthesis of 1-phenyl-3-ethyl-1,2-pentadiene **2i** (kjq-2-180)

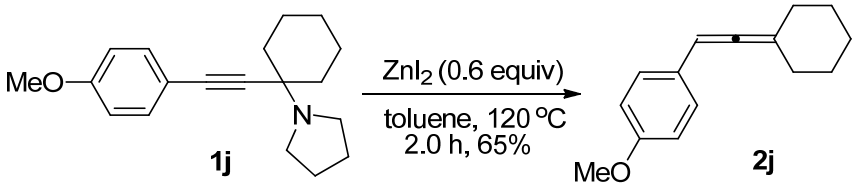


1i 2i

The reaction of **1i** (240.9 mg, 1.0 mmol), ZnI_2 (192.0, 0.6 mmol), and toluene (3 mL) afforded allene **2i**[2] (102.7 mg, 60%) (eluent: *n*-hexane) as a liquid: ^{1}H NMR (400

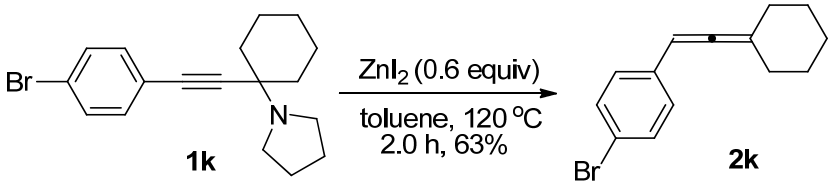
MHz, $CDCl_3$) 7.31-7.24 (m, 4 H, Ar-H), 7.19-7.13 (m, 1 H, Ar-H), 6.18 (q, *J* = 3.2 Hz, 1 H, =CH), 2.18-2.02 (m, 4 H, 2×CH_2), 1.06 (t, *J* = 7.6 Hz, 6 H, 2×CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) 201.6, 136.3, 128.5, 126.31, 126.26, 112.4, 96.4, 25.8, 12.3; MS (EI) *m/z* (%) 172 (M^+, 36.46), 128 (100); IR (neat): 3064, 3031, 2966, 2932, 2875, 1948, 1597, 1496, 1459, 1406，1376, 1328, 1206, 1074, 1045 cm^{-1}.

(10) Synthesis of 1,1-pentamethylene-3-(4-methoxyphenyl)propadiene **2j** (kjq-2-28)



The reaction of **1j** (282.6 mg, 1.0 mmol), ZnI_2 (192.2, 0.6 mmol), and toluene (3 mL) afforded allene **2j**[5] (139.5 mg, 65%) (eluent: petroleum ether) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.20 (d, *J* = 8.8 Hz, 2 H, ArH), 6.83 (d, *J* = 8.8 Hz, 2 H, ArH), 5.98-5.93 (m, 1 H, =CH), 3.78 (s, 3 H, OCH_3), 2.30-2.12 (m, 4 H, 2×CH_2), 1.75-1.47 (m, 6 H, 3×CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.8, 158.3, 128.4, 127.5, 114.0, 106.4, 91.7, 55.3, 31.5, 27.7, 26.1; MS (EI) *m/z* 214 (M^+, 100); IR (neat) 2928, 2852, 2836, 1951, 1609, 1581, 1509, 1463, 1444, 1402, 1296, 1247, 1200, 1180, 1170, 1106, 1036 cm^{-1}.

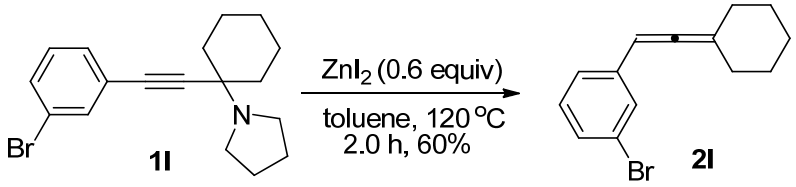
(11) Synthesis of 1,1-pentamethylene-3-(4-bromophenyl)propadiene **2k**(kjq-2-27)



The reaction of **1k** (333.0 mg, 1.0 mmol), ZnI_2 (191.9, 0.6 mmol), and toluene (3 mL) afforded allene **2k**[5] (167.0 mg, 63%) (eluent: petroleum ether) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.4 Hz, 2 H, ArH), 7.15 (d, *J* = 8.4 Hz, 2 H, ArH), 5.99-5.92 (m, 1 H, =CH), 2.34-2.15 (m, 4 H, 2×CH_2), 1.79-1.50 (m, 6 H, 3×CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.8, 135.2, 131.5, 128.0, 119.7, 106.9, 91.6, 31.1, 27.6, 26.0; MS (EI) *m/z* 264 (M^+(^{81}Br), 31.39), 262 (M^+(^{79}Br), 31.2), 141 (100); IR (neat) 2929, 2852, 1951, 1486, 1444, 1386, 1343, 1237, 1198, 1097, 1070, 1009

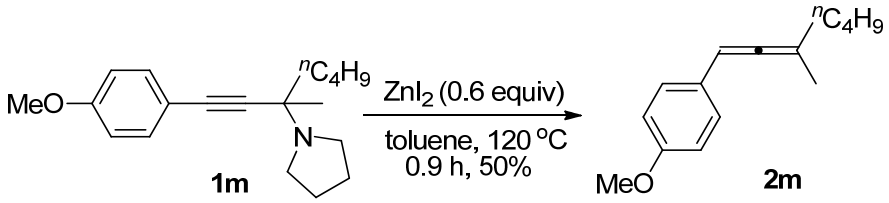
cm^{-1}.

(12) Synthesis of 1,1-pentamethylene-3-(3-bromophenyl)propadiene **2l** (kjq-2-40)



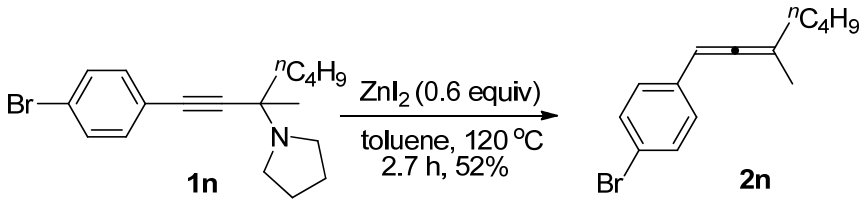
The reaction of **1l** (332.1 mg, 1.0 mmol), ZnI_2 (191.3, 0.6 mmol), and toluene (3 mL) afforded allene **2l**[5] (158.0 mg, 60%) (eluent: 30-60 °C petroleum ether) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (s, 1 H, ArH), 7.27 (d, *J* = 7.6 Hz, 1 H, ArH), 7.21-7.10 (m, 2 H, ArH), 5.95-5.89 (m, 1 H, =CH), 2.32-2.14 (m, 4 H, 2×CH_2), 1.76-1.49 (m, 6 H, 3×CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.0, 138.6, 129.9, 129.3, 129.1, 125.1, 122.7, 107.1, 91.4, 31.2, 27.6, 26.0; MS (EI) *m/z* 264 (M^+(^{81}Br), 9.23), 262 (M^+(^{79}Br), 9.91), 129 (100); IR (neat) 3056, 2930, 2852, 1952, 1591, 1564, 1475, 1446, 1401, 1384, 1343, 1277, 1255, 1236, 1200, 1165, 1088, 1068, 1022 cm^{-1}.

(13) Synthesis of 1-(4-methoxyphenyl)-3-methyl-1,2-heptadiene **2m** (kjq-2-3)



The reaction of **1m** (285.4 mg, 1.0 mmol), ZnI_2 (191.8, 0.6 mmol), and toluene (3 mL) afforded allene **2m** (107.6 mg, 50%) (eluent: petroleum ether) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (d, *J* = 8.4 Hz, 2 H, ArH), 6.82 (d, *J* = 8.8 Hz, 2 H, ArH), 6.05-5.97 (m, 1 H, =CH), 3.76 (s, 3 H, OMe), 2.12-2.00 (m, 2 H, =CCH_2), 1.78 (d, *J* = 2.0 Hz, 3 H, Me), 1.52-1.28 (m, 4 H, 2×CH_2), 0.89 (t, *J* = 7.2 Hz, 3 H, CH_3 in Bu); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.9, 158.3, 128.4, 127.5, 114.0, 103.5, 93.1, 55.2, 33.9, 29.7, 22.4, 18.9, 13.9; MS (EI) *m/z* 216 (M^+, 12.06), 159 (100); IR (neat) 2956, 2931, 2872, 2858, 2835, 1951, 1608, 1581, 1510, 1464, 1441, 1395, 1369, 1296, 1249, 1201, 1170, 1106, 1036 cm^{-1}; HRMS calcd for $C_{15}H_{20}O$ [M^+]: 216.1514. Found: 216.1512.

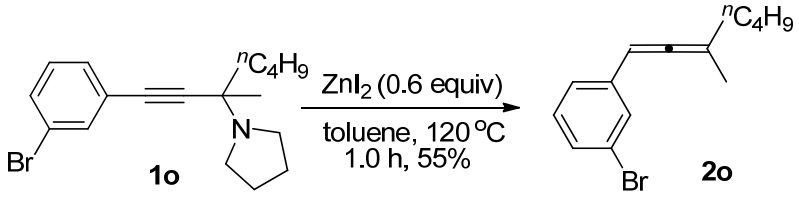
(14) Synthesis of 1-(4-bromophenyl)-3-methyl-1,2-heptadiene **2n** (kjq-2-4)



1n **2n**

The reaction of **1n** (335.0 mg, 1.0 mmol), ZnI_2 (192.0, 0.6 mmol), and toluene (3 mL) afforded allene **2n** (138.4 mg, 50%) (eluent: petroleum ether) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (dt, *J* = 8.4, 2.1 Hz, 2 H, ArH), 7.13 (dt, *J* = 8.4, 2.0 Hz, 2 H, ArH), 6.01-5.97 (m, 1 H, C=CH), 2.08 (td, *J* = 7.4, 2.7 Hz, 2 H, C=CCH_2), 1.80 (d, *J* = 2.8 Hz, 3 H, Me), 1.51-1.30 (m, 4 H, 2×CH_2), 0.89 (t, *J* = 7.2 Hz, 3 H, CH_3 in Bu); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.8, 135.1, 131.5, 128.0, 119.8, 104.2, 93.0, 33.7, 29.6, 22.4, 18.7, 13.9; MS (EI) *m/z* 266 (M^+(^{81}Br), 2.07), 264 (M^+(^{79}Br), 2.12), 143 (100); IR (neat) 2957, 2927, 2871, 2858, 1951, 1488, 1465, 1456, 1443, 1381, 1369, 1223, 1201, 1098, 1071, 1009 cm^{-1}; HRMS calcd for $C_{14}H_{17}Br$ [M^+(^{79}Br)]: 264.0514. Found: 264.0512.

(15) Synthesis of 1-(3-bromophenyl)-3-methyl-1,2-heptadiene **2o** (kjq-2-21)

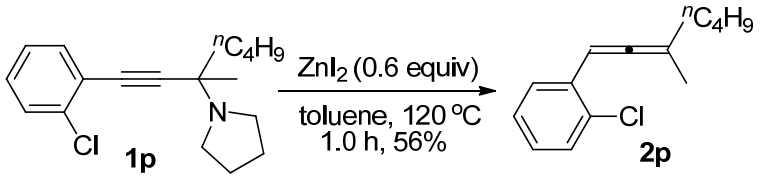


1o **2o**

The reaction of **1o** (334.5 mg, 1.0 mmol), ZnI_2 (191.8, 0.6 mmol), and toluene (3 mL) afforded allene **2o** (144.7 mg, 55%) (eluent: petroleum ether) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (s, 1 H, ArH), 7.31-7.22 (m, 1 H, ArH), 7.20-7.09 (m, 2 H, ArH), 6.02-5.92 (m, 1 H, =CH), 2.15-2.01 (m, 2 H, =CCH_2), 1.81 (d, *J* = 2.8 Hz, 3 H, Me), 1.51-1.30 (m, 4 H, 2×CH_2), 0.90 (t, *J* = 7.0 Hz, 3 H, CH_3 in Bu); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.0, 138.5, 129.9, 129.23, 129.17, 125.1, 122.7, 104.4, 92.8, 33.7, 29.6, 22.4, 18.7, 13.9; MS (EI) *m/z* 266 (M^+(^{81}Br), 1.02), 264 (M^+(^{79}Br), 1.21), 143 (100); IR (neat) 3057, 2957, 2927, 2871, 2858, 1951, 1590, 1565, 1475, 1465, 1392, 1377, 1298, 1225, 1191, 1164, 1150, 1087, 1069 cm^{-1}; HRMS calcd for

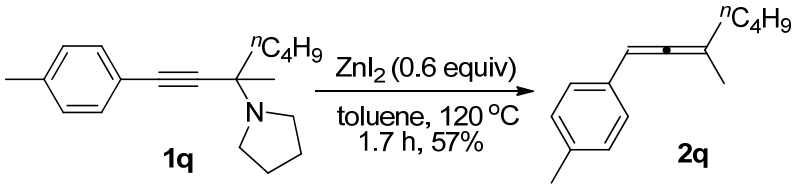
$C_{14}H_{17}Br$ [M^+(^{79}Br)]: 264.0514. Found: 264.0519.

(16) Synthesis of 1-(2-chlorophenyl)-3-methyl-1,2-heptadiene **2p** (kjq-2-20)



The reaction of **1p** (289.8 mg, 1.0 mmol), ZnI_2 (192.0, 0.6 mmol), and toluene (3 mL) afforded allene **2p** (123.6 mg, 56%) (eluent: petroleum ether) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, *J* = 7.6 Hz, 1 H, ArH), 7.31 (d, *J* = 8.4 Hz, 1 H, ArH), 7.17 (t, *J* = 7.4 Hz, 1 H, ArH), 7.08 (t, *J* = 7.4 Hz, 1 H, ArH), 6.53-6.45 (m, 1 H, =CH), 2.09 (t, *J* = 6.6 Hz, 2 H, =CCH_2), 1.81 (d, *J* = 2.8 Hz, 3 H, Me), 1.53-1.29 (m, 4 H, 2×CH_2), 0.90 (t, *J* = 7.2 Hz, 3 H, CH_3 in Bu); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.7, 133.6, 131.8, 129.6, 128.0, 127.3, 126.6, 104.0, 90.1, 33.6, 29.6, 22.4, 18.6, 13.9; MS (EI) *m/z* 222 (M^+(^{37}Cl), 0.37), 220 (M^+(^{35}Cl), 1.15), 143 (100); IR (neat) 3066, 2957, 2929, 2872, 2859, 1951, 1591, 1567, 1478, 1466, 1456, 1441, 1397, 1379, 1369, 1284, 1227, 1199, 1150, 1125, 1048, 1032 cm^{-1}; HRMS calcd for $C_{14}H_{17}Cl$ [M^+(^{35}Cl)]: 220.1019. Found: 220.1022.

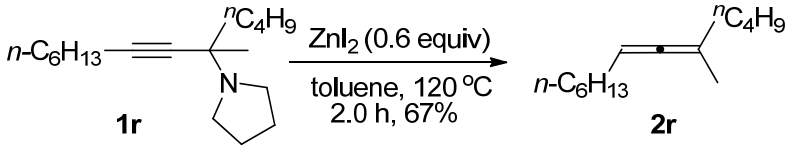
(17) Synthesis of 1-(4-methylphenyl)-3-methyl-1,2-heptadiene **2q** (kjq-2-25)



The reaction of **1q** (269.1 mg, 1.0 mmol), ZnI_2 (192.5, 0.6 mmol), and toluene (3 mL) afforded allene **2q** (114.0 mg, 57%) (eluent: petroleum ether) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (d, *J* = 8.0 Hz, 2 H, ArH), 7.11 (d, *J* = 8.0 Hz, 2 H, ArH), 6.07-6.00 (m, 1 H, =CH), 2.34 (s, 3 H, ArCH_3), 2.13-2.04 (m, 2 H, =CCH_2), 1.81 (d, *J* = 2.8 Hz, 3 H, Me), 1.52-1.31 (m, 4 H, 2×CH_2), 0.91 (t, *J* = 7.2 Hz, 3 H, CH_3 in Bu); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.3, 136.0, 133.1, 129.2, 126.4, 103.5, 93.5, 33.8, 29.7, 22.4, 21.1, 18.9, 13.9; MS (EI) *m/z* 200 (M^+, 5.37), 143 (100); IR

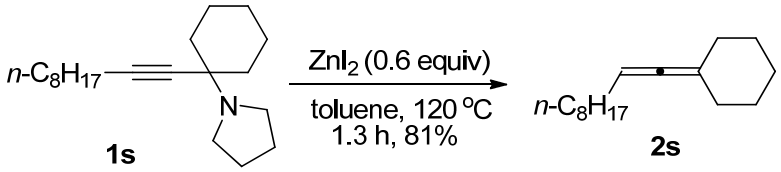
(neat) 3018, 2957, 2926, 2872, 2859, 1952, 1513, 1458, 1390, 1378, 1368, 1308, 1227, 1202, 1176, 1150, 1118, 1106, 1039, 1019 cm^{-1}; HRMS calcd for $C_{15}H_{20}$ (M^+): 200.1565. Found: 200.1567.

(18) Synthesis of 5-methyl-5,6-tridecadiene **2r** (kjq-2-30)



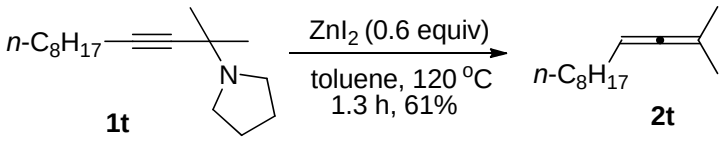
The reaction of **1r** (263.6 mg, 1.0 mmol), ZnI_2 (191.7, 0.6 mmol), and toluene (3 mL) afforded allene **2r** (130.1 mg, 67%) (eluent: 30-60 ^oC petroleum ether) as a liquid: ^1H NMR (400 MHz, $CDCl_3$) δ 5.02-4.94 (m, 1 H, =CH), 2.00-1.85 (m, 4 H, 2× $=CCH_2$), 1.66 (d, J = 2.8 Hz, 3 H, Me), 1.44-1.21 (m, 12 H, 6×CH_2), 0.96-0.81 (m, 6 H, 2×CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.2, 99.1, 90.1, 33.8, 31.8, 29.8, 29.4, 29.3, 28.8, 22.7, 22.4, 19.3, 14.1, 14.0; MS (EI) *m/z* 194 (M^+, 1.23), 95 (100); IR (neat) 2958, 2927, 2873, 2856, 1965, 1466, 1378, 1369, 1231, 1154, 1106 cm^{-1}; HRMS calcd for $C_{14}H_{26}$ (M^+): 194.2035. Found: 194.2034.

(19) Synthesis of 1,1-pentamethylene-1,2-undecadiene **2s** (kjq-2-48)



The reaction of **1s** (289.7 mg, 1.0 mmol), ZnI_2 (191.8, 0.6 mmol), and toluene (3 mL) afforded allene **2s**[5] (178.2 mg, 81%) (eluent: 30-60 ^oC petroleum ether) as a liquid: ^1H NMR (400 MHz, $CDCl_3$) δ 4.98-4.92 (m, 1 H, =CH), 2.16-2.04 (m, 4 H, 2×CH_2), 1.95 (q, J = 6.8 Hz, 2 H, CH_2), 1.66-1.45 (m, 6 H, 3×CH_2), 1.44-1.20 (m, 12 H, 6×CH_2), 0.89 (t, J = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) 198.3, 102.2, 88.7, 31.9, 31.8, 29.5, 29.4, 29.3, 29.1, 29.0, 27.5, 26.2, 22.7, 14.1; MS (EI) *m/z* (%) 220 (M^+, 1.85), 122 (100); IR (neat) 2925, 2853, 1965, 1446, 1378, 1344, 1261, 1239 cm^{-1}.

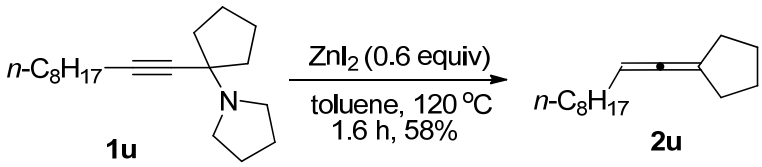
(20) Synthesis of 2-methyl-2,3-dodecadiene **2t** (kjq-2-47)



1t **2t**

The reaction of **1t** (249.5 mg, 1.0 mmol), ZnI_2 (192.0, 0.6 mmol), and toluene (3 mL) afforded allene **2t**[8] (110.3 mg, 61%) (eluent: 30-60 °C petroleum ether) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.96-4.89 (m, 1 H, =CH), 1.93 (q, *J* = 6.9 Hz, 2 H, CH_2), 1.67 (d, *J* = 3.2 Hz, 6 H, 2×CH_3), 1.42-1.21 (m, 12 H, 6×CH_2), 0.88 (t, *J* = 7.0 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.6, 94.7, 88.8, 31.9, 29.5, 29.3, 29.23, 29.22, 29.1, 22.7, 20.8, 14.1; MS (EI) *m/z* 180 (M^+, 0.5), 137 (M^+-C_3H_7, 1.19), 82 (100); IR (neat) 2958, 2925, 2855, 1969, 1456, 1406, 1377, 1362, 1229, 1190 cm^{-1}.

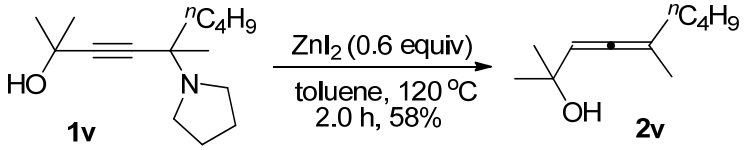
(21) Synthesis of 1,1-tetramethylene-1,2-undecadiene **2u** (kjq-2-54)



1u **2u**

The reaction of **1u** (274.9 mg, 1.0 mmol), ZnI_2 (192.0, 0.6 mmol), and toluene (3 mL) afforded allene **2u** (118.6 mg, 58%) (eluent: 30-60 °C petroleum ether) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.09-5.01 (m, 1 H, =CH), 2.41-2.24 (m, 4 H, 2×CH_2), 1.96 (q, *J* = 6.8 Hz, 2 H, CH_2), 1.71-1.59 (m, 4 H, 2×CH_2), 1.44-1.17 (m, 12 H, 6×CH_2), 0.88 (t, *J* = 6.4 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) 197.0, 103.4, 91.4, 31.9, 31.2, 29.5, 29.36, 29.35, 29.2, 29.1, 27.1, 22.7, 14.1; MS (EI) *m/z* (%) 206 (M^+, 1.61), 93 (100); IR (neat) 2956, 2925, 2854, 1964, 1466, 1437, 1378, 1212, 1133, 1015 cm^{-1}; HRMS calcd for $C_{15}H_{26}$ (M^+): 206.2035. Found: 206.2039.

(22) Synthesis of 2,5-dimethyl-3,4-nonadien-2-ol **2v** (kjq-2-29)

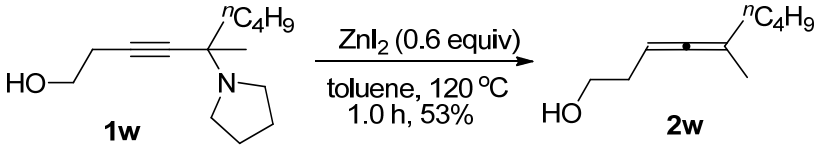


1v **2v**

The reaction of **1v** (237.2 mg, 1.0 mmol), ZnI_2 (192.0, 0.6 mmol), and toluene (3

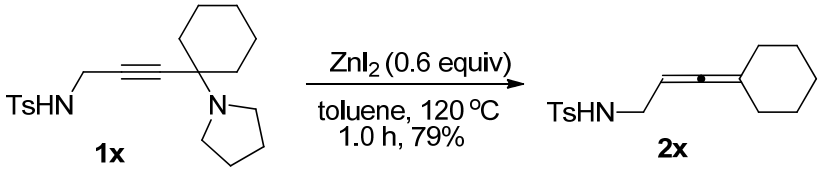
mL) afforded allene **2v** (97.3 mg, 58%) (eluent: 30-60 °C petroleum ether/ethyl ether = 10/1 to 5/1) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.25-5.20 (m, 1 H, =CH), 1.99-1.91 (m, 2 H, CH_2), 1.75-1.65 (m, 4 H, OH and CH_3), 1.43-1.22 (m, 10 H, 2×CH_3 and 2×CH_2), 0.89 (t, *J* = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) 197.2, 104.0, 100.6, 69.8, 33.7, 30.03, 29.98, 29.7, 22.4, 19.2, 13.9; MS (EI) *m/z* (%) 168 (M^+, 4.30), 68 (100); IR (neat) 3362, 2972, 2930, 2874, 2860, 1966, 1459, 1400, 1370, 1148 cm^{-1}; HRMS calcd for $C_{11}H_{20}O$ (M^+): 168.1514. Found: 168.1510.

(23) Synthesis of 5-methyl-3,4-nonadien-1-ol **2w** (kjq-2-49)



The reaction of **1w** (222.7 mg, 1.0 mmol), ZnI_2 (191.0, 0.6 mmol), and toluene (3 mL) afforded allene **2w**[9] (81.8 mg, 53%) (eluent: petroleum ether/ethyl acetate = 15/1) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.05-4.96 (m, 1 H, =CH), 3.68 (q, *J* = 5.7 Hz, 2 H, CH_2), 2.22 (q, *J* = 6.3 Hz, 2 H, CH_2), 1.99-1.89 (m, 2 H, CH_2), 1.68 (d, *J* = 2.4 Hz, 3 H, =CCH_3), 1.62 (brs, 1 H, OH), 1.47-1.26 (m, 4 H, 2×CH_2), 0.89 (t, *J* = 7.0 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) 202.1, 100.2, 86.2, 62.1, 33.7, 32.6, 29.7, 22.3, 19.2, 13.9; MS (EI) *m/z* (%) 154 (M^+, 0.64), 139 (M^+-CH_3, 2.75), 121 (M^+-CH_3-H_2O, 6.96), 68 (100); IR (neat) 3346, 2957, 2928, 2873, 1966, 1466, 1443, 1370, 1230, 1177, 1049 cm^{-1}.

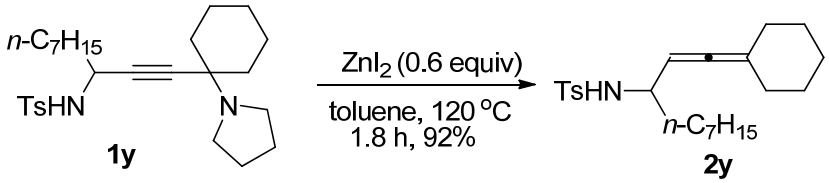
(24) Synthesis of *N*-(1,1-pentamethylene-1,2-butadien-4-yl)-4-methyl benzenesulfonamide **2x** (kjq-2-105)



The reaction of **1x** (359.8 mg, 1.0 mmol), ZnI_2 (191.8, 0.6 mmol), and toluene (3 mL) afforded allene **2x**[10] (228.6 mg, 79%) (eluent: *n*-hexane/ethyl acetate = 10/1) as a liquid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.4 Hz, 2 H, ArH), 7.30 (d, *J* = 8.0

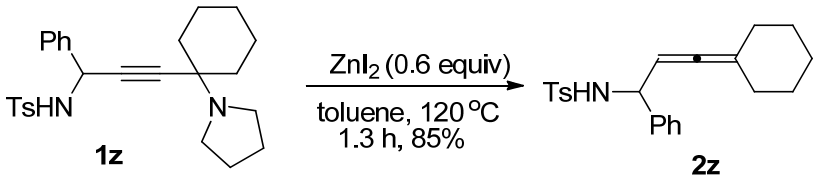
Hz, 2 H, ArH), 4.95-4.88 (m, 1 H, =CH), 4.51-4.41 (m, 1 H, NH), 3.52 (t, J = 5.6 Hz, 2 H, CH_2), 2.42 (s, 3 H, CH_3), 2.08-1.99 (m, 4 H, 2×CH_2), 1.62-1.45 (m, 6 H, 3×CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) 197.7, 143.2, 136.9, 129.6, 127.0, 106.2, 85.3, 42.3, 31.1, 27.2, 25.8, 21.4; MS (EI) *m/z* (%) 291 (M^+, 1.52), 155 (100); IR (neat) 3271, 2924, 2852, 1965, 1598, 1496, 1444, 1325, 1290, 1265, 1240, 1184, 1160, 1093, 1070, 1038, 1020 cm^{-1}.

(25) Synthesis of *N*-(1,1-pentamethylene-1,2-undecadien-4-yl)-4-methyl benzenesulfonamide **2y** (kjq-2-119)



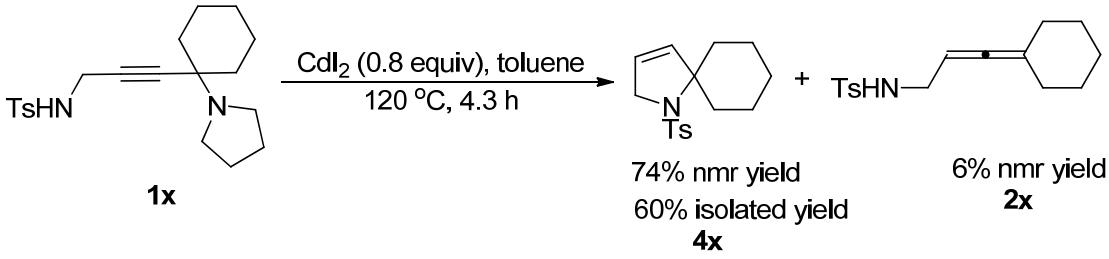
The reaction of **1y** (458.9 mg, 1.0 mmol), ZnI_2 (192.3, 0.6 mmol), and toluene (3 mL) afforded allene **2y** (357.7 mg, 92%) (eluent: *n*-hexane/ethyl acetate = 10/1 to 5/1) as a white solid: m.p. 97-99 ^{o}C (petroleum ether & ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, J = 8.4 Hz, 2 H, ArH), 7.28 (d, J = 8.8 Hz, 2 H, ArH), 4.87-4.80 (m, 1 H, =CH), 4.49-4.40 (m, 1 H, NH), 3.75-3.67 (m, 1 H, NCH), 2.42 (s, 3 H, CH_3), 2.08-1.89 (m, 4 H, 2×CH_2), 1.65-1.41 (m, 8 H, 4×CH_2), 1.36-1.13 (m, 10 H, 5×CH_2), 0.87 (t, J = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) 196.5, 142.8, 138.0, 129.3, 127.0, 106.4, 90.6, 52.8, 36.1, 31.6, 31.13, 31.10, 29.08, 29.06, 27.2, 25.8, 25.1, 22.5, 21.3, 13.9; MS (EI) *m/z* (%) 389 (M^+, 0.55), 290 (M^+-C_7H_{15}, 4.18), 91 (100); IR (neat) 3276, 2927, 2854, 1967, 1599, 1496, 1446, 1331, 1306, 1239, 1184, 1161, 1094, 1049 cm^{-1}; Anal. calcd for $C_{23}H_{35}NO_2S$ (%): C 70.91, H 9.06, N 3.60; Found: C 70.81, H 9.47, N 3.49.

(26) Synthesis of *N*-(1,1-pentamethylene-4-phenyl-1,2-undecadien-4-yl)-4-methyl benzenesulfonamide **2z** (kjq-2-120)



The reaction of **1z** (437.0 mg, 1.0 mmol), ZnI_2 (191.0, 0.6 mmol), and toluene (3 mL) afforded allene **2z** (312.0 mg, 85%) (eluent: *n*-hexane/ethyl acetate = 10/1 to 5/1) as a white solid: m.p. 124-126 ^{o}C (petroleum ether & ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 8.0 Hz, 2 H, ArH), 7.25-7.14 (m, 7 H, ArH), 5.14-5.08 (m, 1 H, =CH), 5.06-4.98 (m, 1 H, NH), 4.89 (dd, *J* = 6.8, 4.8 Hz, 1 H, NCH), 2.38 (s, 3 H, CH_3), 2.06-1.91 (m, 4 H, 2×CH_2), 1.62-1.33 (m, 6 H, 3×CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) 196.9, 142.7, 140.4, 137.6, 129.1, 128.0, 127.1, 126.9, 126.8, 107.5, 91.3, 56.4, 31.0, 30.8, 27.0, 25.6, 21.2; MS (EI) *m/z* (%) 367 (M^+, 0.08), 260 (M^+-CH=C=C$(CH_2)_5$, 67.77), 91 (100); IR (neat) 3227, 3063, 3030, 2929, 2853, 1967, 1599, 1495, 1448, 1328, 1289, 1163, 1093, 1051, 1028 cm^{-1}; Anal. calcd for $C_{22}H_{25}NO_2S$ (%): C 71.90, H 6.86, N 3.81; Found: C 71.64, H 6.78, N 3.70.

CdI_2-mediated reaction of propargylic amine **1x**-Synthesis of **4x** and **2x** (kjq-2-188).

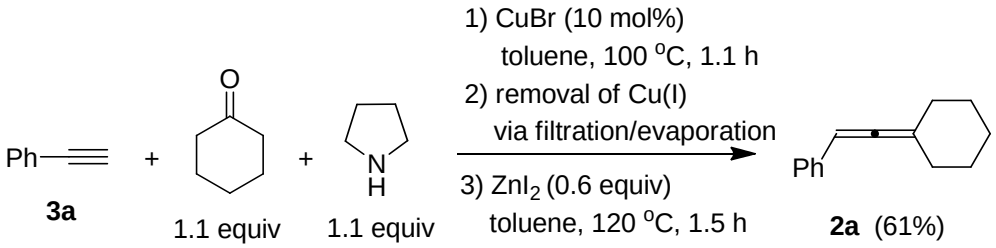


To a flame-dried Schlenk tube was added CdI_2 (146.0 mg, 0.4 mmol) inside a glove box. The Schlenk tube was then taken out and dried under vacuum with a heating gun. After cooling to room temperature, propargylic amine **1x** (181.1 mg. 0.5 mmol) and 1.5 mL of toluene were added under the atmosphere of Ar. The Schlenk tube equipped with a condenser was then placed in a pre-heated oil bath of 130 ^{o}C with stirring for 4.3 h as monitored TLC. After cooling to room temperature, the crude reaction mixture was filtrated through a short pad of silica gel eluted with ethyl acetate (15 mL). After evaporation, to the residue was added 27 μL of CH_3NO_2 as the internal

standard, the NMR yields of **4x** and **2x** were determined based on the ^{1}H NMR analysis. Then the residue was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 10/1) to afford **4x**[13] (87.3 mg, 60%) as a solid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79-7.72 (m, 2 H, Ar-H), 7.29-7.23 (m, 2 H, Ar-H), 6.12 (dt, *J* = 6.6, 2.3 Hz, 1 H, CH=), 5.67 (dt, *J* = 6.4, 2.0 Hz, 1 H, CH=), 4.12 (t, *J* = 2.2 Hz, 2 H, CH_2), 2.52-2.36 (m, 5 H, CH_3 + two protons of Cy), 1.82-1.57 (m, 5 H, five protons of Cy), 1.42-1.22 (m, 3 H, three protons of Cy); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.5, 138.4, 132.6, 129.22, 127.0, 122.1, 75.7, 55.0, 37.1, 25.0, 24.4, 21.3; MS (EI) *m/z* (%) 291 (M^+, 18.01), 91 (100); IR (neat) 2927, 2863, 1598, 1495, 1455, 1368, 1160, 1127, 1100, 1071cm^{-1}.

Part III ZnI_2-Promoted synthesis of trisubstituted allenes from 1-alkynes, ketones, and pyrrolidine.

(1) Synthesis of 1,1-pentamethylene-3-phenylpropadiene (**2a**) (kjq-13-56)



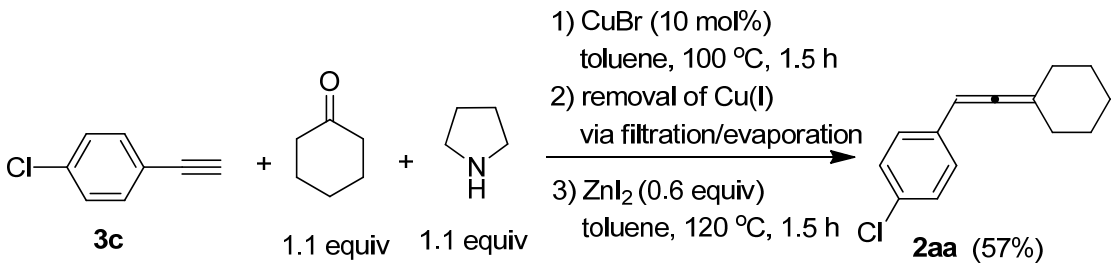
Typically Procedure IV: To a flame-dried Schlenk tube were added CuBr (14.6 mg, 0.1 mmol), phenylacetylene **3a** (102.4 mg, 1.0 mmol)/toluene(0.5 mL), cyclohexanone (107.8 mg, 1.1 mmol)/ toluene(0.5 mL), and pyrrolidine (93.0 μL, d = 0.862 g/mL, 80.2 mg, 1.1 mmol) sequentially. The Schlenk tube was then stirred at 100 °C until completion of the reaction as monitored by TLC (1.1 h). After cooling to room temperature, the crude reaction mixture was filtrated through a short pad of silica gel eluted with acetone (20 mL). After evaporation, the crude product was used in the next step without further treatment.

To another Schlenk tube were added anhydrous ZnI_2 (191.4 mg, 0.6 mmol). The

Schlenk tube was then dried under vacuum with a heating gun. The above crude product was then dissolved in toluene (3 mL) and transferred to the Schlenk tube via a syringe under Ar atmosphere. The Schlenk tube was then equipped with a condenser and placed in a pre-heated oil bath of 120 ^{o}C with stirring. After 1.5 h, the reaction was complete as monitored by TLC, the crude reaction mixture was cooled to room temperature and filtrated through a short pad of silica gel eluted with ethyl ether (20 mL). After evaporation, the residue was purified by chromatography on silica gel to afford **2a**[2] (112.1 mg, 61%) (eluent: petroleum ether) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33-7.22 (m, 4 H, ArH), 7.20-7.10 (m, 1 H, ArH), 6.02-5.96 (m, 1 H, =CH), 2.34-2.12 (m, 4 H, 2×CH_2), 1.80-1.46 (m, 6 H, 3×CH_2).

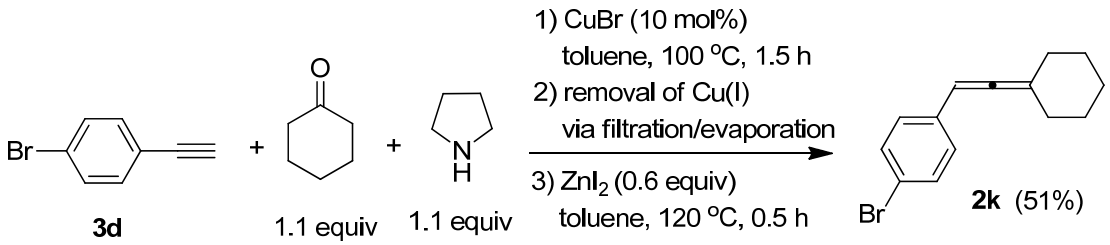
The following compounds were prepared according to **Typically Procedure IV**.

(2) Synthesis of 1,1-pentamethylene-3-(4-chlorophenyl)propadiene (**2aa**) (kjq-13-48)



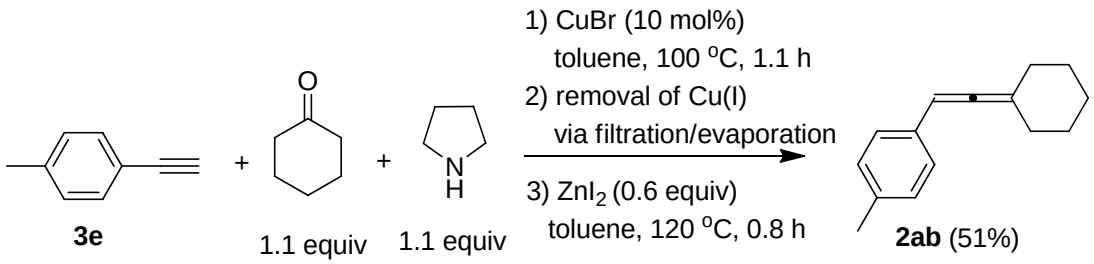
The reaction of CuBr (14.3 mg, 0.1 mmol), **3c** (136.3 mg, 1.0 mmol)/toluene (0.5 mL), cyclohexanone (107.1 mg, 1.1 mmol)/toluene (0.5 mL), pyrrolidine (93.0 μL, d = 0.862 g/mL, 80.2 mg, 1.1 mmol) afforded the crude product after filtration and evaporation. The reaction of the crude product/toluene (3 mL) and ZnI_2 (191.8 mg, 0.6 mmol) afforded **2aa** (124.3 mg, 57%) (eluent: petroleum ether) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29-7.14 (m, 4 H, ArH), 5.99-5.92 (m, 1 H, =CH), 2.35-2.12 (m, 4 H, 2×CH_2), 1.80-1.47 (m, 6 H, 3×CH_2); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 199.7, 134.7, 131.7, 128.6, 127.6, 106.9, 91.5, 31.2, 27.6, 26.0; MS (EI) *m/z* 220 [M^+(^{37}Cl), 22.82], 218 [M^+(^{35}Cl), 68.91], 141 (100); IR (neat) 2929, 2853, 1952, 1489, 1444, 1390, 1343, 1238, 1091, 1012 cm^{-1}; HRMS (EI) calcd for $C_{14}H_{15}{}^{35}Cl$ (M^+): 218.0862. Found: 218.0860.

(3) Synthesis of 1,1-pentamethylene-3-(4-bromophenyl)propadiene (**2k**) (kjq-1-39)



The reaction of CuBr (14.4 mg, 0.1 mmol), **3d** (181.2 mg, 1.0 mmol)/toluene (0.5 mL), cyclohexanone (107.5 mg, 1.1 mmol)/toluene (0.5 mL), pyrrolidine (93.0 μL, d = 0.862 g/mL, 80.2 mg, 1.1 mmol) afforded the crude product after filtration and evaporation. The reaction of the crude product/toluene (3 mL) and ZnI_2 (191.4 mg, 0.6 mmol) afforded **2k**[5] (135.6 mg, 51%) (eluent: petroleum ether) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.4 Hz, 2 H, ArH), 7.15 (d, *J* = 8.0 Hz, 2 H, ArH), 5.99-5.93 (m, 1 H, =CH), 2.34-2.16 (m, 4 H, 2×CH_2), 1.79-1.51 (m, 6 H, 3×CH_2).

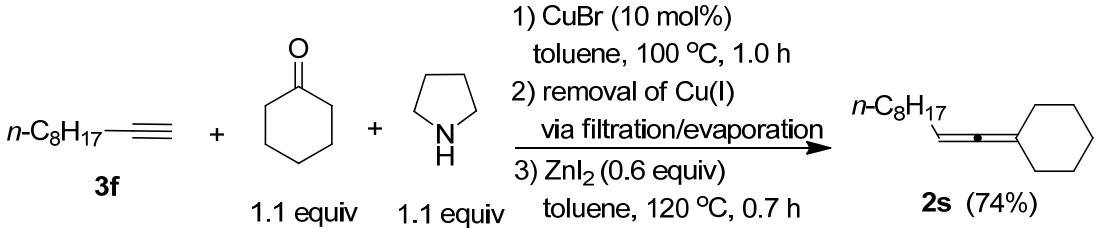
(4) Synthesis of 1,1-pentamethylene-3-(4-methylphenyl)propadiene (**2ab**) (kjq-13-47)



The reaction of CuBr (14.7 mg, 0.1 mmol), **3e** (115.7 mg, 1.0 mmol)/toluene (0.5 mL), cyclohexanone (106.8 mg, 1.1 mmol)/toluene (0.5 mL), pyrrolidine (93.0 μL, d = 0.862 g/mL, 80.2 mg, 1.1 mmol) afforded the crude product after filtration and evaporation. The reaction of the crude product/toluene (3 mL) and ZnI_2 (191.9 mg, 0.6 mmol) afforded **2ab** (101.3 mg, 51%) (eluent: petroleum ether) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.17 (d, *J* = 8.1 Hz, 2 H, ArH), 7.09 (d, *J* = 7.8 Hz, 2 H, ArH), 6.00-5.94 (m, 1 H, =CH), 2.37-2.10 (m, 7 H, CH_3 + 2×CH_2), 1.77-1.46 (m, 6 H, 3×CH_2); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 199.3, 136.0, 133.1, 129.2, 126.4, 106.3, 92.1, 31.4, 27.7, 26.1, 21.1; MS (EI) *m/z* 198 (M^+, 74.69), 155 (100); IR (neat) 2926,

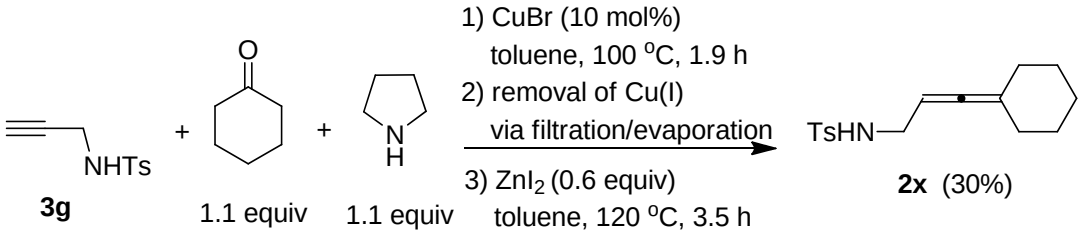
2853, 1951, 1513, 1445, 1257, 1237 cm^{-1}; HRMS (EI) calcd for $C_{15}H_{18}$ (M^+): 198.1409. Found: 198.1410.

(5) Synthesis of 1,1-pentamethylene-1,2-undecadiene (**2s**) (kjq-13-64)



The reaction of CuBr (14.4 mg, 0.1 mmol), **3f** (138.6 mg, 1.0 mmol)/toluene (0.5 mL), cyclohexanone (108.0 mg, 1.1 mmol) /toluene (0.5 mL), pyrrolidine (93.0 μL, d = 0.862 g/mL, 80.2 mg, 1.1 mmol) afforded the crude product after filtration and evaporation. The reaction of the crude product/toluene (3 mL) and ZnI_2 (191.2 mg, 0.6 mmol) afforded **2s**[5] (164.5 mg, 74%) (eluent: petroleum ether) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.00-4.90 (m, 1 H, =CH), 2.20-2.01 (m, 4 H, 2×CH_2), 2.00-1.88 (m, 2 H, CH_2), 1.68-1.15 (m, 18 H, 9×CH_2), 0.88 (t, J = 6.8 Hz, 3 H, CH_3).

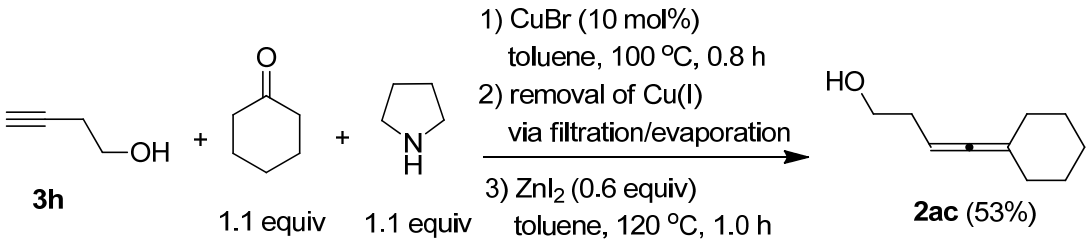
(6) Synthesis of *N*-(1,1-pentamethylene-1,2-butadien-4-yl)-4-methyl-benzenesulfonamide (**2x**) (kjq-13-53)



The reaction of CuBr (14.1 mg, 0.1 mmol), **3g** (208.5 mg, 1.0 mmol)/toluene (0.5 mL), cyclohexanone (107.7 mg, 1.1 mmol)/toluene (0.5 mL), pyrroline (93.0 μL, d = 0.862 g/mL, 80.2 mg, 1.1 mmol) afforded the crude product after filtration and evaporation. The reaction of the crude product/toluene (3 mL) and ZnI_2 (191.0 mg, 0.6 mmol) afforded **2x**[10] (88.5 mg, 30%) (eluent: petroleum ether/ethyl acetate = 10/1) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76 (d, J = 8.4 Hz, 2 H, ArH), 7.30 (d, J = 8.4 Hz, 2 H, ArH), 4.96-4.86 (m, 1 H, =CH), 4.76-4.60 (bs, 1 H, NH), 3.52 (t, J = 5.9 Hz, 2 H, NCH_2), 2.42 (s, 3 H, Me), 2.10-1.96 (m, 4 H, 2×CH_2), 1.64-1.42 (m, 6 H,

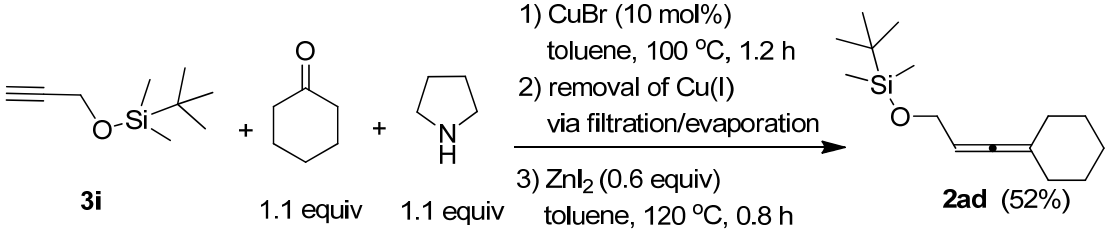
3×CH_2).

(7) Synthesis of 4-cyclohexylidenebut-3-en-1-ol (**2ac**) (kjq-13-67)



The reaction of CuBr (14.3 mg, 0.1 mmol), **3h** (69.8 mg, 1.0 mmol)/toluene (0.5 mL), cyclohexanone (108.6 mg, 1.1 mmol)/toluene (0.5 mL), pyrrolidine (93.0 μL, d = 0.862 g/mL, 80.2 mg, 1.1 mmol) afforded the crude product after filtration and evaporation. The reaction of the crude product/toluene (3 mL) and ZnI_2 (191.7 mg, 0.6 mmol) afforded **2ac**[11] (80.0 mg, 53%) (eluent: petroleum ether/ethyl acetate = 4/1) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.00-4.91 (m, 1 H, =CH), 3.74-3.61 (m, 2 H, OCH_2), 2.22 (q, *J* = 6.2 Hz, 2 H, CH_2), 2.16-1.96 (m, 4 H, 2×CH_2), 1.76-1.42 (m, 7 H, OH + 3×CH_2); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 199.0, 103.1, 84.8, 62.0, 32.5, 31.7, 27.4, 26.0; MS (EI) *m/z* 152 (M^+, 13.39), 79 (100); IR (neat) 3335, 2924, 2852, 1965, 1708, 1445, 1264, 1238, 1176, 1130, 1048 cm^{-1}.

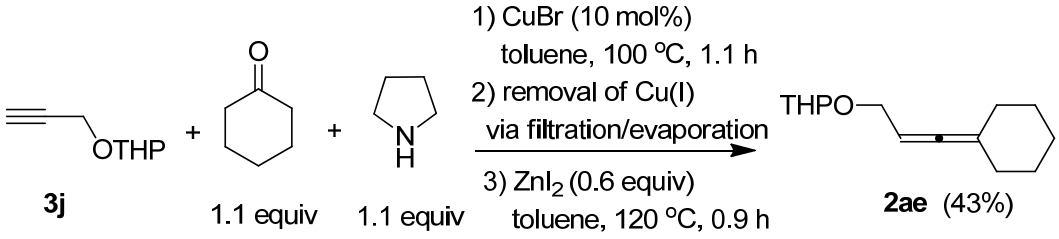
(8) Synthesis of tert-butyl(3-cyclohexylideneallyloxy)dimethylsilane (**2ad**) (kjq-13-69)



The reaction of CuBr (14.2 mg, 0.1 mmol), **3i** (170.4 mg, 1.0 mmol)/toluene (0.5 mL), cyclohexanone (107.4 mg, 1.1 mmol)/toluene (0.5 mL), pyrrolidine (93.0 μL, d = 0.862 g/mL, 80.2 mg, 1.1 mmol) afforded the crude product after filtration and evaporation. The reaction of the crude product/toluene (3 mL) and ZnI_2 (191.7 mg, 0.6 mmol) afforded **2ad**[12] (132.2 mg, 52%) (eluent: petroleum ether/ethyl acetate =

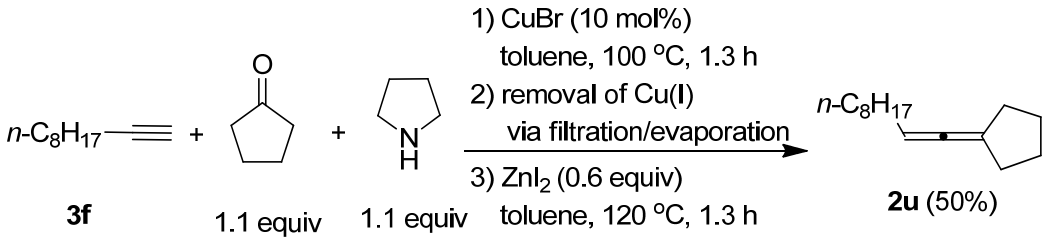
50/1) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.15-5.04 (m, 1 H, =CH), 4.14 (d, *J* = 6.3 Hz, 2 H, OCH_2), 2.16-2.06 (m, 4 H, 2×CH_2), 1.64-1.45 (m, 6 H, 3×CH_2), 0.90 (s, 9 H, $C(CH_3)_3$), 0.08 (s, 6 H, $Si(CH_3)_2$); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 197.9, 103.6, 89.6, 62.7, 31.3, 27.3, 26.1, 26.0, 18.4, -5.0; MS (EI) *m/z* 252 (M^+, 0.05), 237 (M^+-CH_3, 1.98), 75 (100); IR (neat) 2928, 2855, 1966, 1472, 1446, 1361, 1253, 1176, 1096, 1078, 1047, 1006 cm^{-1}.

(9) Synthesis of 2-(3-cyclohexylideneallyloxy)-tetrahydro-2H-pyran (**2ae**) (kjq-13-81)



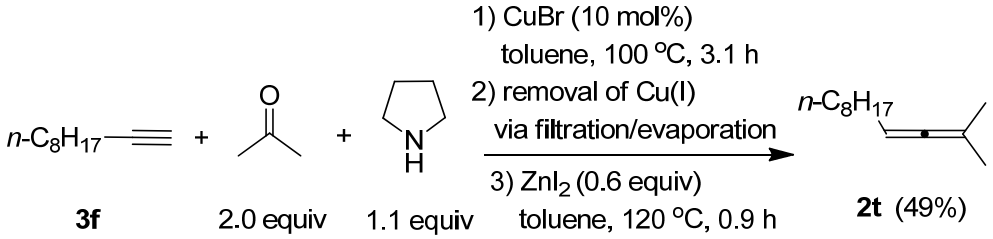
The reaction of CuBr (14.5 mg, 0.1 mmol), **3j** (140.3 mg, 1.0 mmol)/toluene (0.5 mL), cyclohexanone (107.8 mg, 1.1 mmol)/toluene (0.5 mL), pyrrolidine (93.0 μL, d = 0.862 g/mL, 80.2 mg, 1.1 mmol) afforded the crude product after filtration and evaporation. The reaction of the crude product/toluene (3 mL) and ZnI_2 (191.6 mg, 0.6 mmol) afforded **2ae** (95.8 mg, 43%) (eluent: petroleum ether/ethyl acetate = 20/1) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.12-5.03 (m, 1 H, =CH), 4.72-4.66 (m, 1 H, OCHO in THP), 4.18 (dd, *J* = 11.6, 6.2 Hz, 1 H, one proton of OCH_2), 4.00 (dd, *J* = 11.6, 7.4 Hz, 1 H, one proton of OCH_2), 3.94-3.83 (m, 1 H, one proton of OCH_2), 3.55-3.44 (m, 1 H, one proton of OCH_2), 2.18-2.04 (m, 4 H, 2×CH_2), 1.94-1.42 (m, 12 H, 6×CH_2); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 199.4, 103.0, 97.2, 86.0, 65.8, 62.1, 31.3, 31.2, 30.6, 27.17, 27.16, 25.9, 25.4, 19.5; MS (EI) *m/z* 222 (M^+, 1.08), 85 (100); IR (neat) 2922, 2849, 1966, 1622, 1579, 1443, 1385, 1353, 1321, 1269, 1201, 1182, 1118, 1078, 1053, 1023 cm^{-1}; HRMS (EI) calcd for $C_{14}H_{22}O_2$ (M^+): 222.1620. Found: 222.1622.

(10) Synthesis of 1,1-tetramethylene-1,2-undecadiene (**2u**) (kjq-13-89)



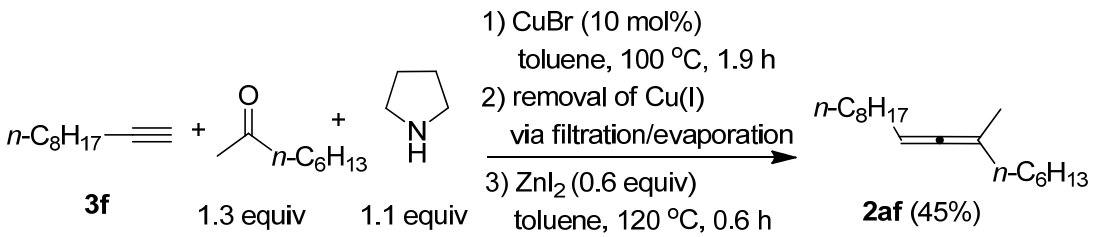
The reaction of CuBr (14.4 mg, 0.1 mmol), **3f** (138.6 mg, 1.0 mmol)/toluene (0.5 mL), cyclopentanone (92.2 mg, 1.1 mmol)/toluene (0.5 mL), pyrrolidine (93.0 μL, d = 0.862 g/mL, 80.2 mg, 1.1 mmol) afforded the crude product after filtration and evaporation. The reaction of the crude product/toluene (3 mL) and ZnI_2 (192.0 mg, 0.6 mmol) afforded **2u** (102.9 mg, 50%) (eluent: petroleum ether) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.11-5.00 (m, 1 H, =CH), 2.40-2.24 (m, 4 H, 2×CH_2), 2.02-1.90 (m, 2 H, CH_2), 1.73-1.58 (m, 4 H, 2×CH_2), 1.45-1.15 (m, 12 H, 6×CH_2), 0.88 (t, J = 6.6 Hz, 3 H, CH_3).

(11) Synthesis of 2-methyl-2,3-dodecadiene (**2r**) (kjq-13-101)



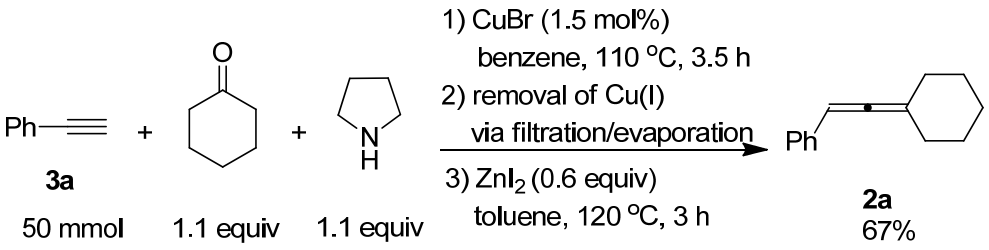
The reaction of CuBr (14.2 mg, 0.1 mmol), **3f** (137.2 mg, 1.0 mmol)/toluene (0.5 mL), acetone (148.0 μL, d = 0.7845 g/mL, 116.1 mg, 2.0 mmol)/toluene (0.5 mL), pyrrolidine (93.0 μL, d = 0.862 g/mL, 80.2 mg, 1.1 mmol) afforded the crude product after filtration and evaporation. The reaction of the crude product/toluene (3 mL) and ZnI_2 (192.7 mg, 0.6 mmol) afforded **2t**[8] (87.5 mg, 49%) (eluent: 30-60 °C petroleum ether) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.98-4.87 (m, 1 H, C=CH), 1.98-1.86 (q, J = 6.8 Hz, 2 H, CH_2), 1.67 (d, J = 2.7 Hz, 6 H, 2×CH_3), 1.44-1.19 (m, 12 H, 6×CH_2), 0.88 (t, J = 6.5 Hz, 3 H, CH_3 in C_8H_{17}).

(12) Synthesis of 7-methylheptadeca-7,8-diene (**2af**) (kjq-13-145)



The reaction of CuBr (14.7 mg, 0.1 mmol), **3f** (138.5 mg, 1.0 mmol)/toluene (0.5 mL), 2-octanone (166.1 mg, 1.3 mmol)/toluene (0.5 mL), pyrrolidine (92.6 μL, d = 0.862 g/mL, 79.8 mg, 1.1 mmol) afforded the crude product after filtration and evaporation. The reaction of the crude product/toluene (3 mL) and ZnI_2 (192.1 mg, 0.6 mmol) afforded **2af** (112.3 mg, 45%) (eluent: petroleum ether) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.04-4.94 (m, 1 H, =CH), 2.01-1.84 (m, 4 H, 2×CH_2), 1.66 (d, J = 3.0 Hz, 3 H, =CCH_3), 1.50-1.17 (m, 20 H, 10×CH_2), 0.89 (t, J = 6.3 Hz, 6 H, 2×CH_3); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 201.1, 99.1, 90.1, 34.1, 31.9, 31.8, 29.5, 29.39, 29.35, 29.1, 29.0, 27.6, 22.7, 19.3, 14.1; MS (EI) *m/z* 250 (M^+, 1.19), 81 (100); IR (neat) 2957, 2924, 2854, 1966, 1465, 1378, 1232, 1153, 1115, 1056 cm^{-1}; HRMS (EI) calcd for $C_{18}H_{34}$ (M^+): 250.2661. Found: 250.2662.

Fifty mmol-scale reaction for the synthesis of 1,1-pentamethylene-3-phenylpropadiene from phenylacetylene, cyclohexanone, and pyrrolidine (2a) (kjq-1-164)



To a three-necked flask equipped with a Dean-Stark trap and a condenser dried under vacuum with a heating gun were added CuBr (0.11 g, 0.75 mmol), **3a** (5.65 mL, d = 0.93 g/mL, 5.25 g, 97%, 50 mmol), cyclohexanone (5.67 mL, d = 0.95 g/mL, 5.39 g, 55 mmol), pyrrolidine (4.54 mL, d = 0.86 g/mL, 3.90 g, 55 mmol), and benzene (50 mL) sequentially under Ar atmosphere. The flask was then placed in a pre-heated oil bath at 110 °C with stirring for 3.5 h as monitored by TLC. After cooling to room

temperature, the crude reaction mixture was filtrated through a short pad of silica gel eluted with ethyl acetate (120 mL). After evaporation, the crude product was used in the next step without further treatment.

To another three-necked flask equipped with a condenser was added anhydrous ZnI_2 (9.78 g, 30 mmol), the flask was then dried under vacuum with a heating gun. The above crude product was then dissolved in toluene (150 mL) and transferred to the flask via a syringe under Ar atmosphere. The flask was then placed in a pre-heated oil bath of 120 ^oC with stirring. After 3.0 h, the reaction was complete as monitored by TLC, the crude reaction mixture was cooled to room temperature and filtrated through a short pad of silica gel eluted with ethyl ether (100 mL). After evaporation, the residue was purified by chromatography on silica gel to afford **2a**[2] (6.13 g, 67%) (eluent: petroleum ether) as a liquid: ^1H NMR (400 MHz, $CDCl_3$) δ 7.32-7.25 (m, 4 H, ArH), 7.20-7.12 (m, 1 H, ArH), 6.02-5.97 (m, 1 H, C=CH), 2.32-2.12 (m, 4 H, 2×CH_2), 1.77-1.47 (m, 6 H, 3×CH_2).

References:

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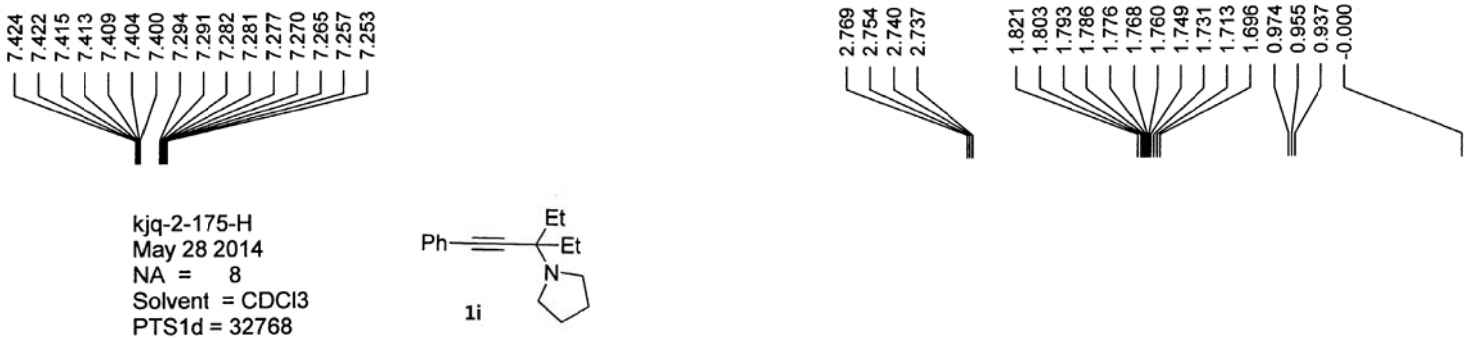
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7.281
7.277
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7.265
7.257
7.253
2.769
2.754
2.740
2.737
1.821
1.803
1.793
1.786
1.776
1.768
1.760
1.749
1.731
1.713
1.696
0.974
0.955
0.937
-0.000
kjq-2-175-H
May 28 2014
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Solvent = CDCl3
PTS1d = 32768
F1 = 399.722412 MHz
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Et
Et
N
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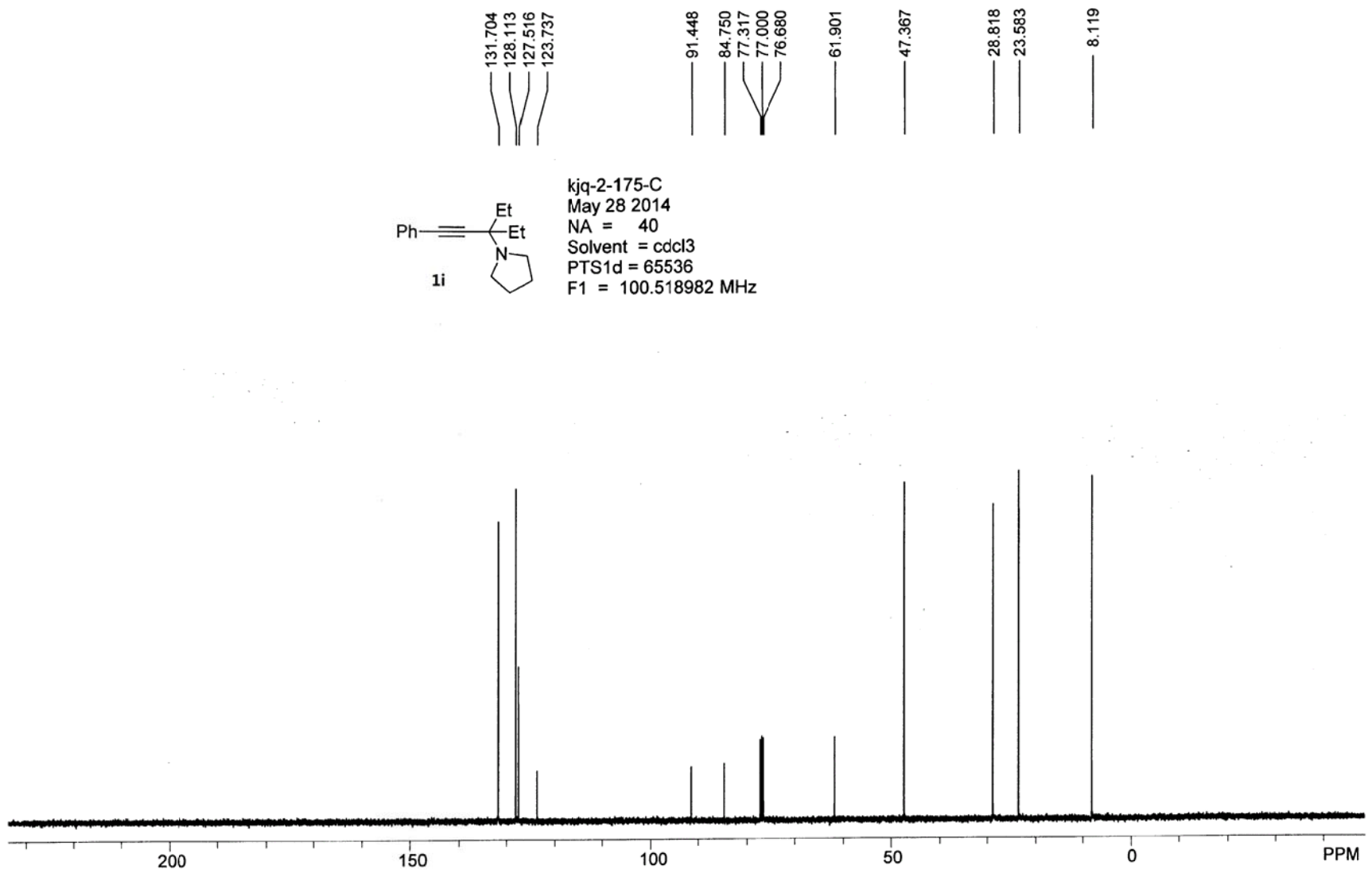
8.60
6.29
8
7
6
5
4
3
2
1
0
PPM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47.367
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8.119
Et
Ph
Et
N
1i
kjq-2-175-C
May 28 2014
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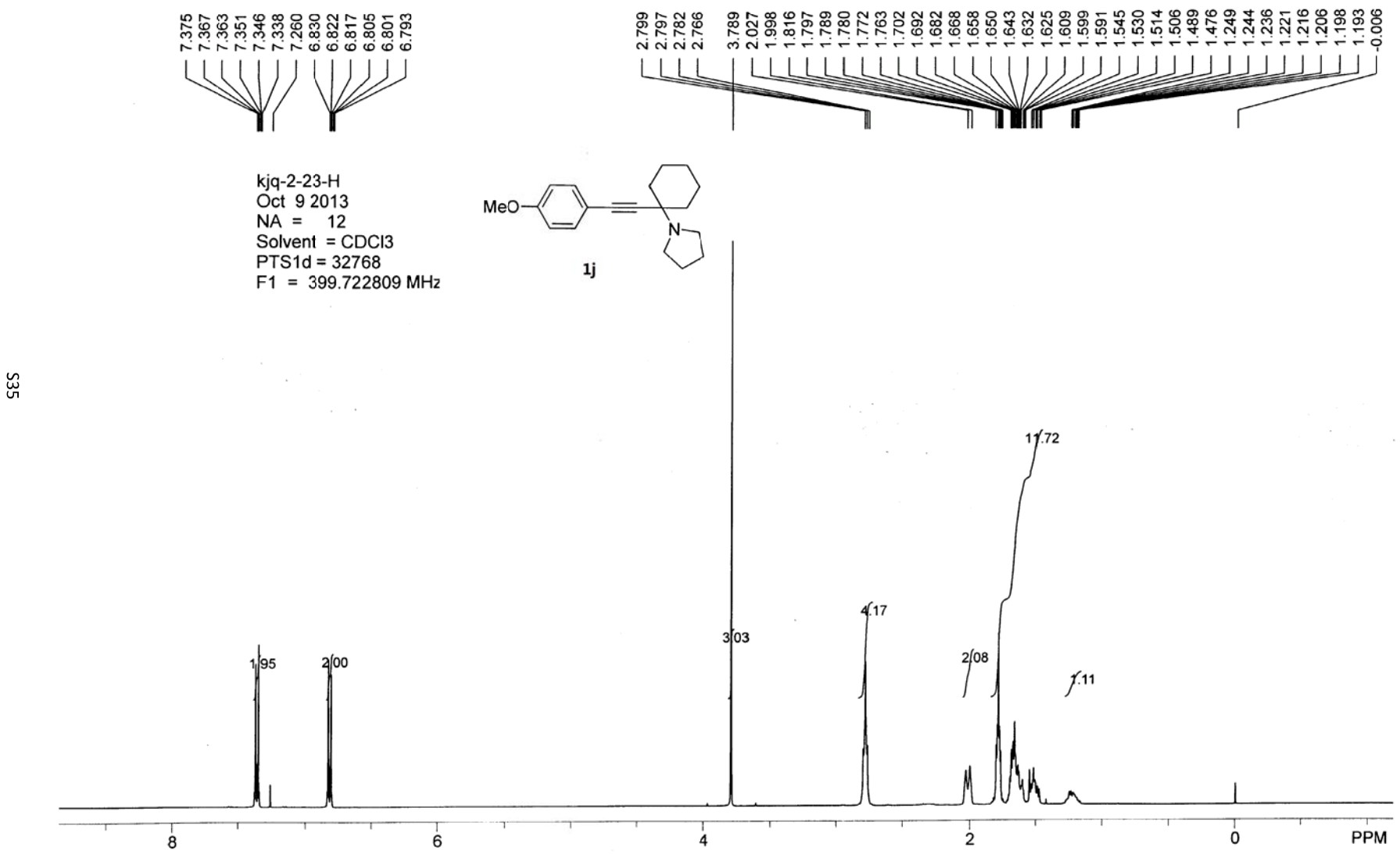
150
100
50
0
PPM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6.793
2.799
2.797
2.782
2.766
3.789
2.027
1.998
1.816
1.797
1.789
1.780
1.772
1.763
1.702
1.692
1.682
1.668
1.658
1.650
1.643
1.632
1.625
1.609
1.599
1.591
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1.514
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1.249
1.244
1.236
1.221
1.216
1.206
1.198
1.193
-0.006
kjq-2-23-H
Oct 9 2013
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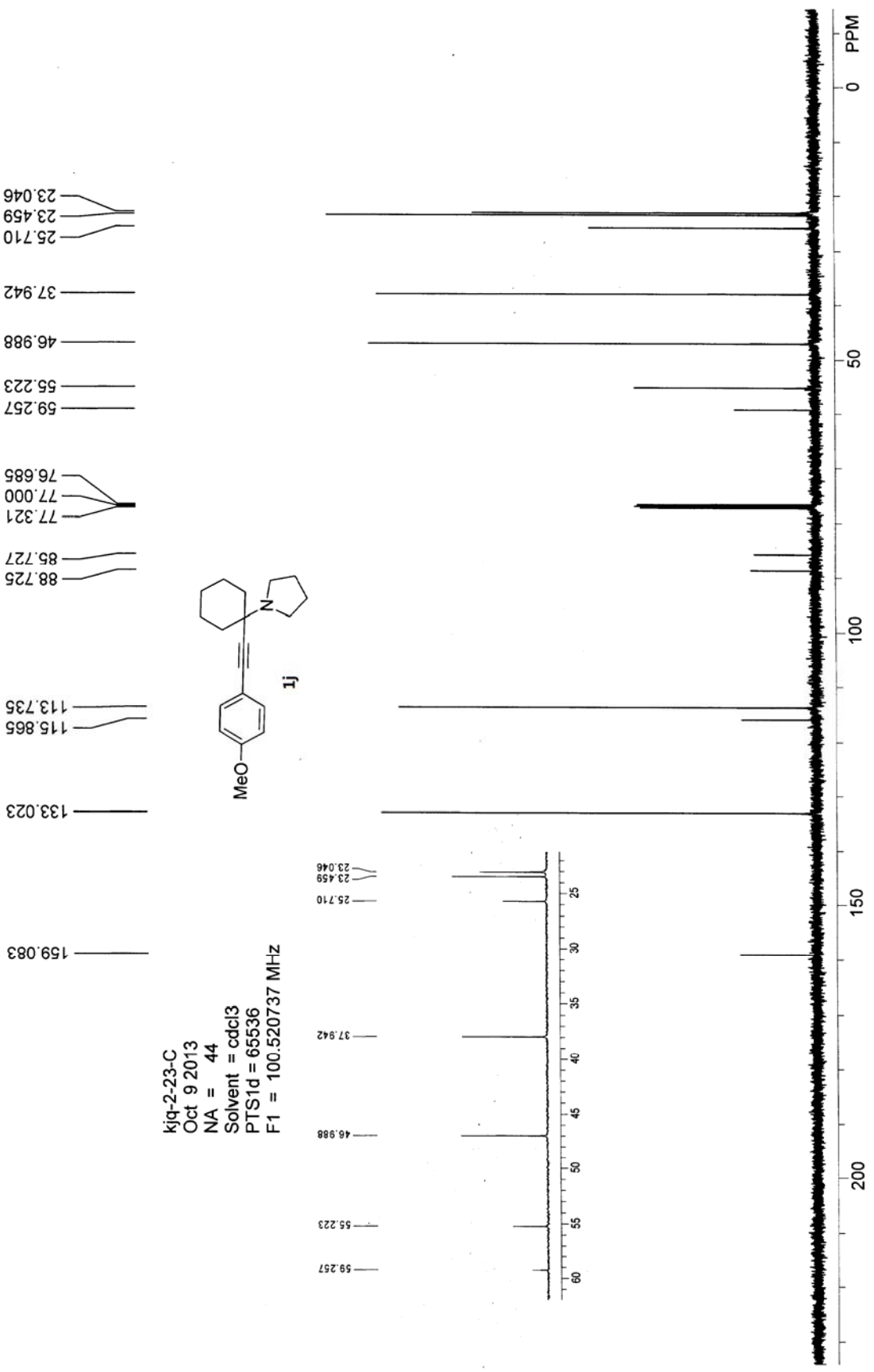
4
2
0
PPM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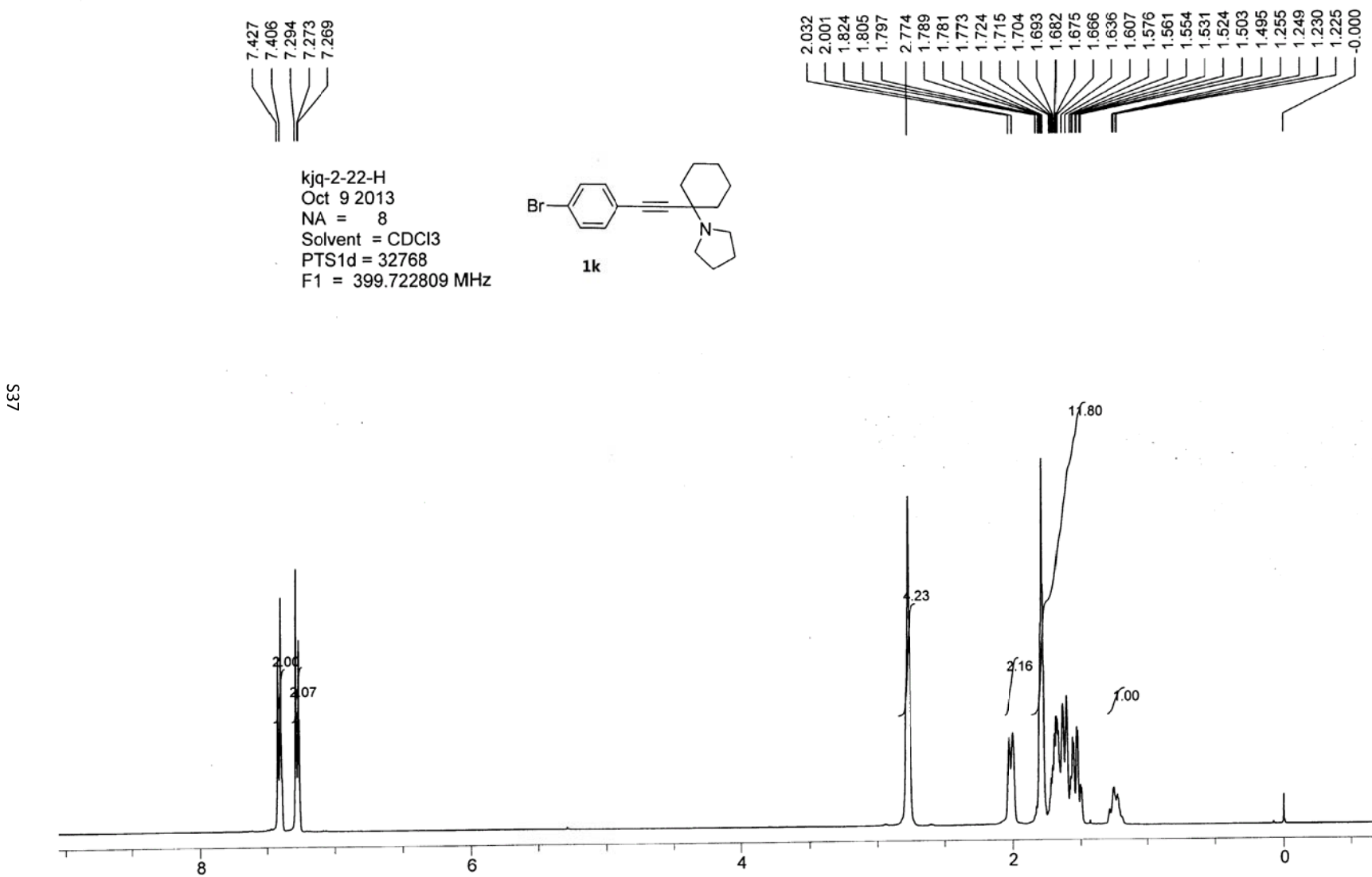
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1.724
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1.704
1.693
1.682
1.675
1.666
1.636
1.607
1.576
1.561
1.554
1.531
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1.495
1.255
1.249
1.230
1.225
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kjq-2-22-H
Oct 9 2013
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PTS1d = 32768
F1 = 399.722809 MHz
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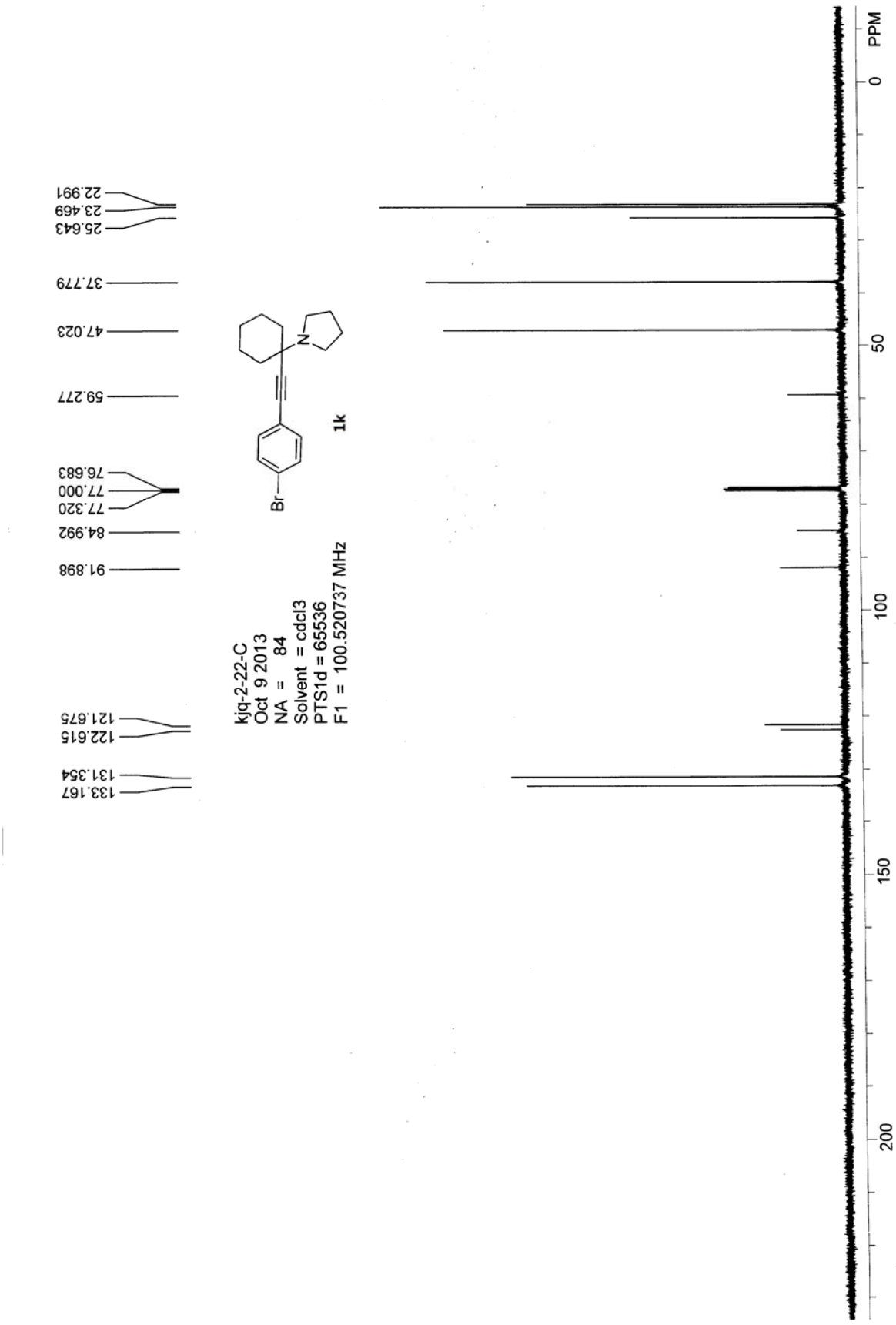
1.00
8
6
4
2
0
PPM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77.000
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84.992
91.898
121.675
122.615
131.354
133.167
kjq-2-22-C
Oct 9 2013
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Br
N
PPM
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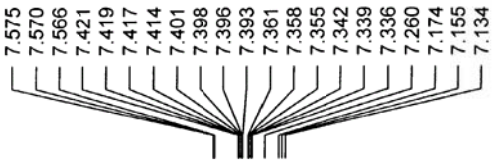
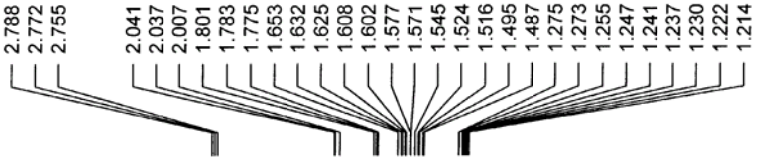
100
150
200





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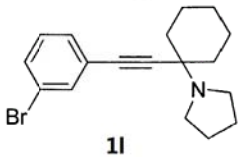

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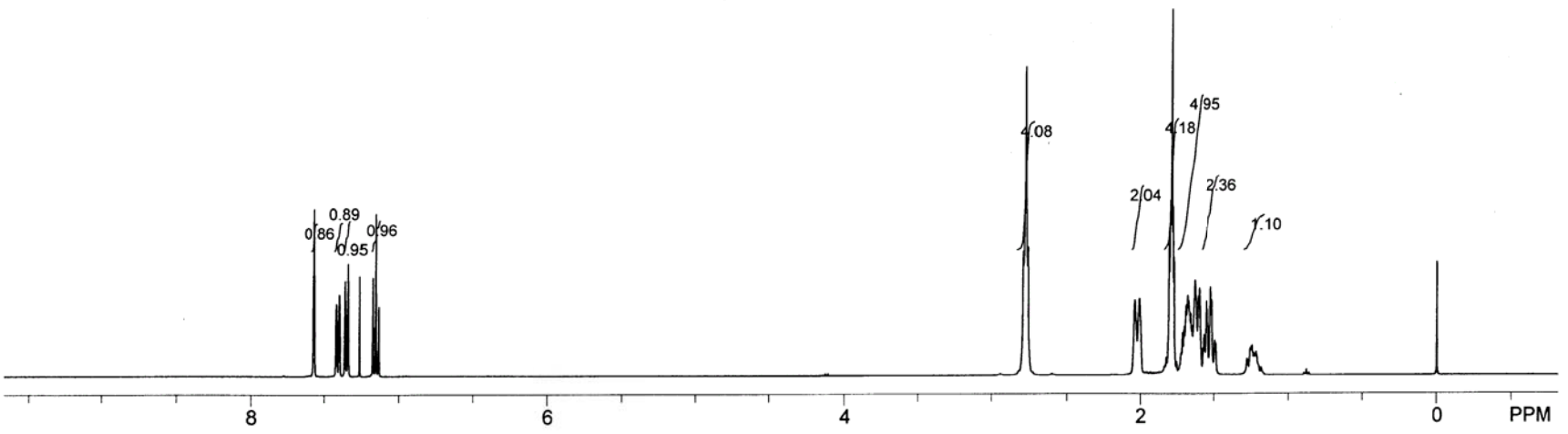

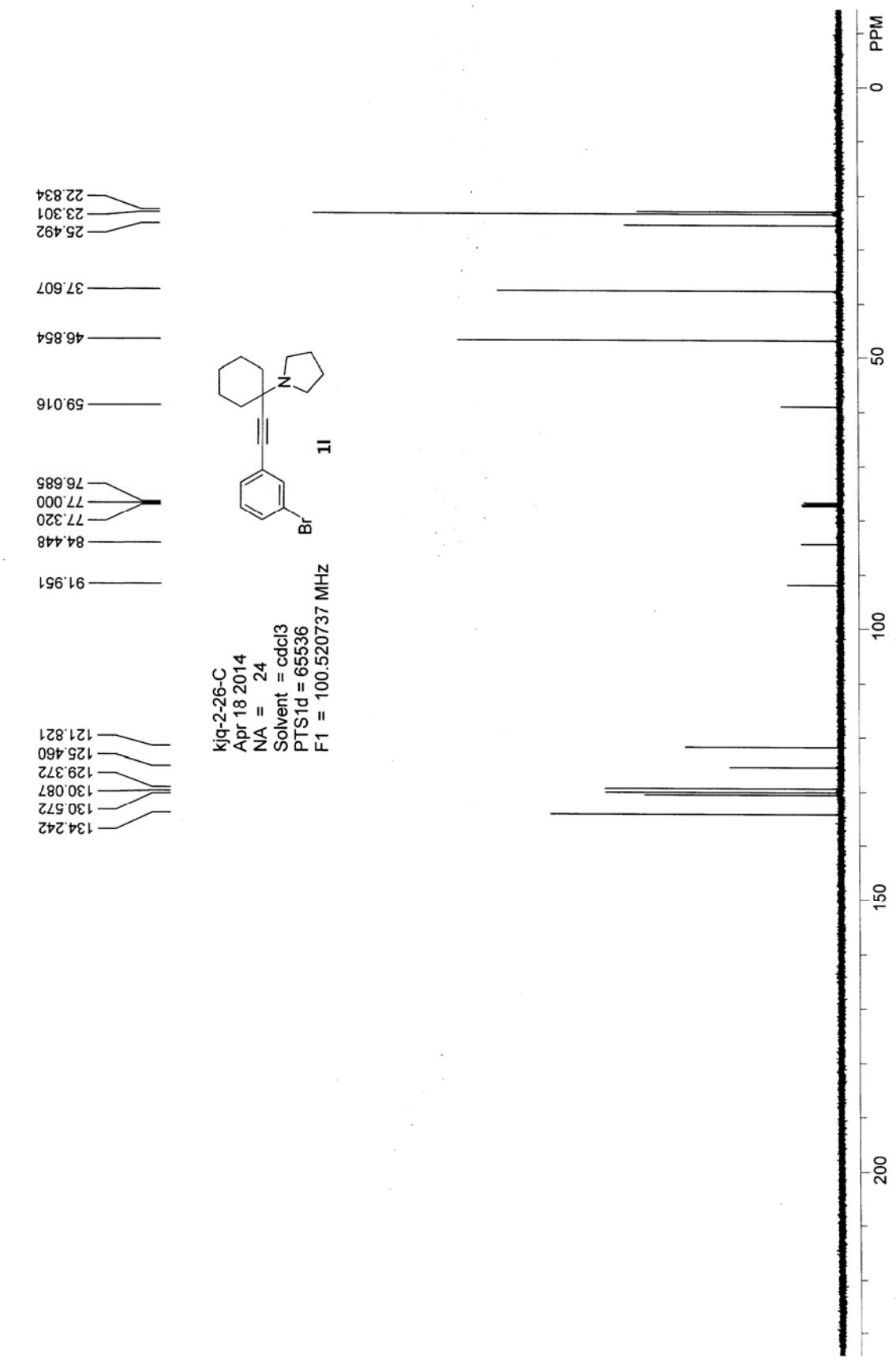
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130.087
130.572
134.242
kjq-2-26-C
Apr 18 2014
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Br
1l
PPM
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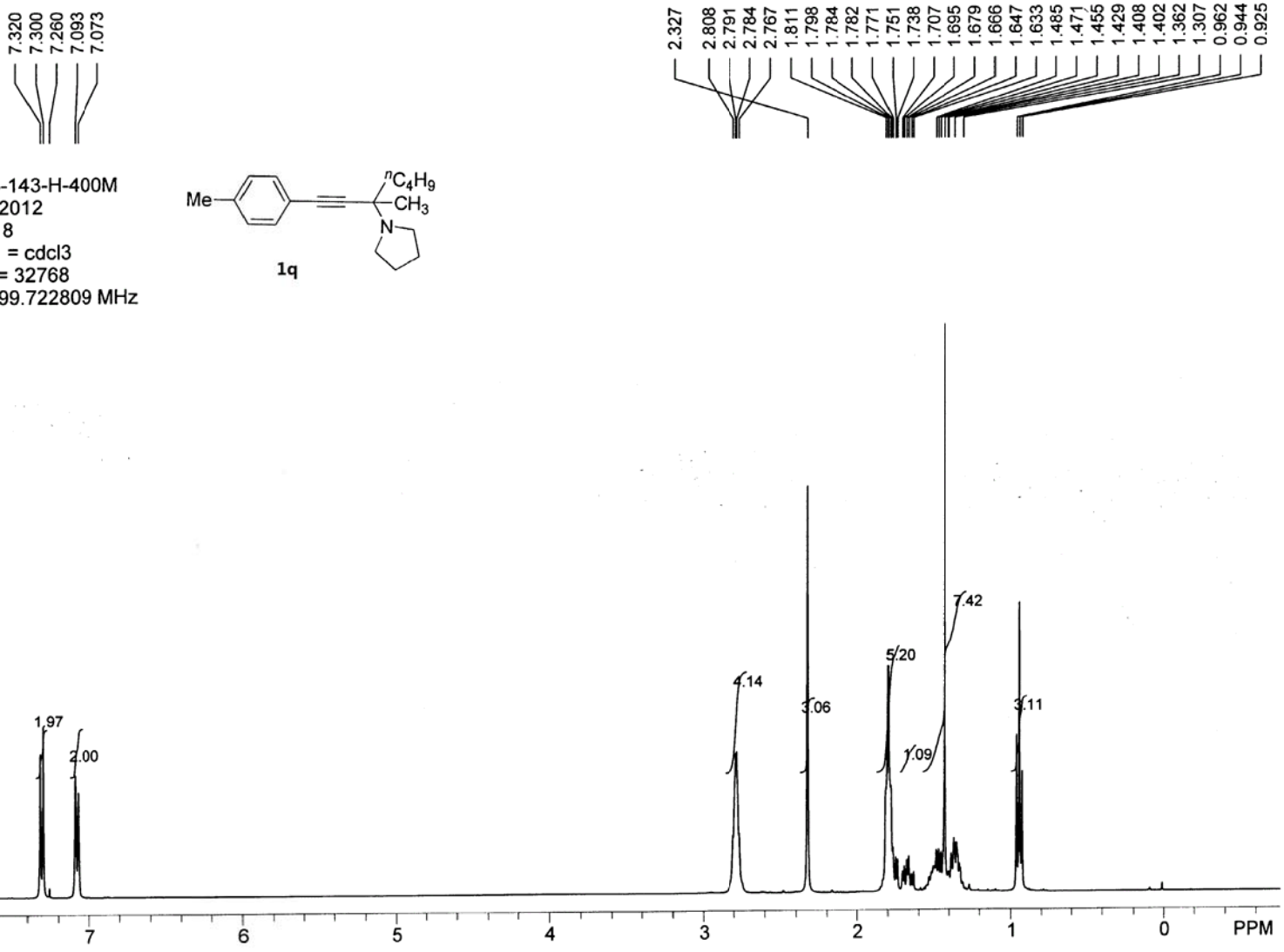
100
150
200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2.767
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1.784
1.782
1.771
1.751
1.738
1.707
1.695
1.679
1.666
1.647
1.633
1.485
1.471
1.455
1.429
1.408
1.402
1.362
1.307
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0.944
0.925
tangxj-4-143-H-400M
Nov 13 2012
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Solvent = cdcl3
PTS1d = 32768
F1 = 399.722809 MHz
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CH_3
N
1q
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5.20
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8
7
6
5
4
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2
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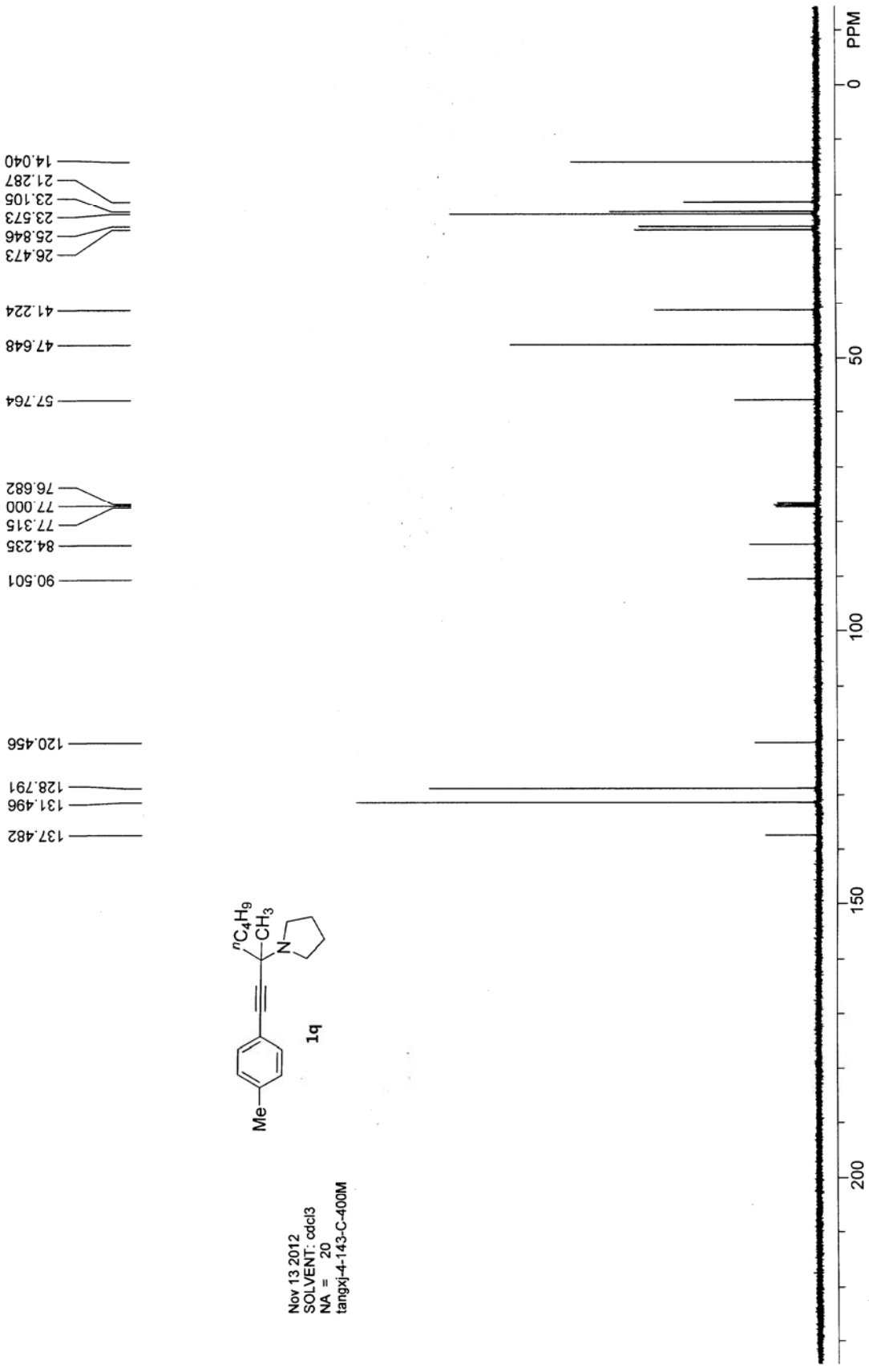
0
PPM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90.501
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100
150
200
Me
1q
Nov 13 2012
SOLVENT: cdcl3
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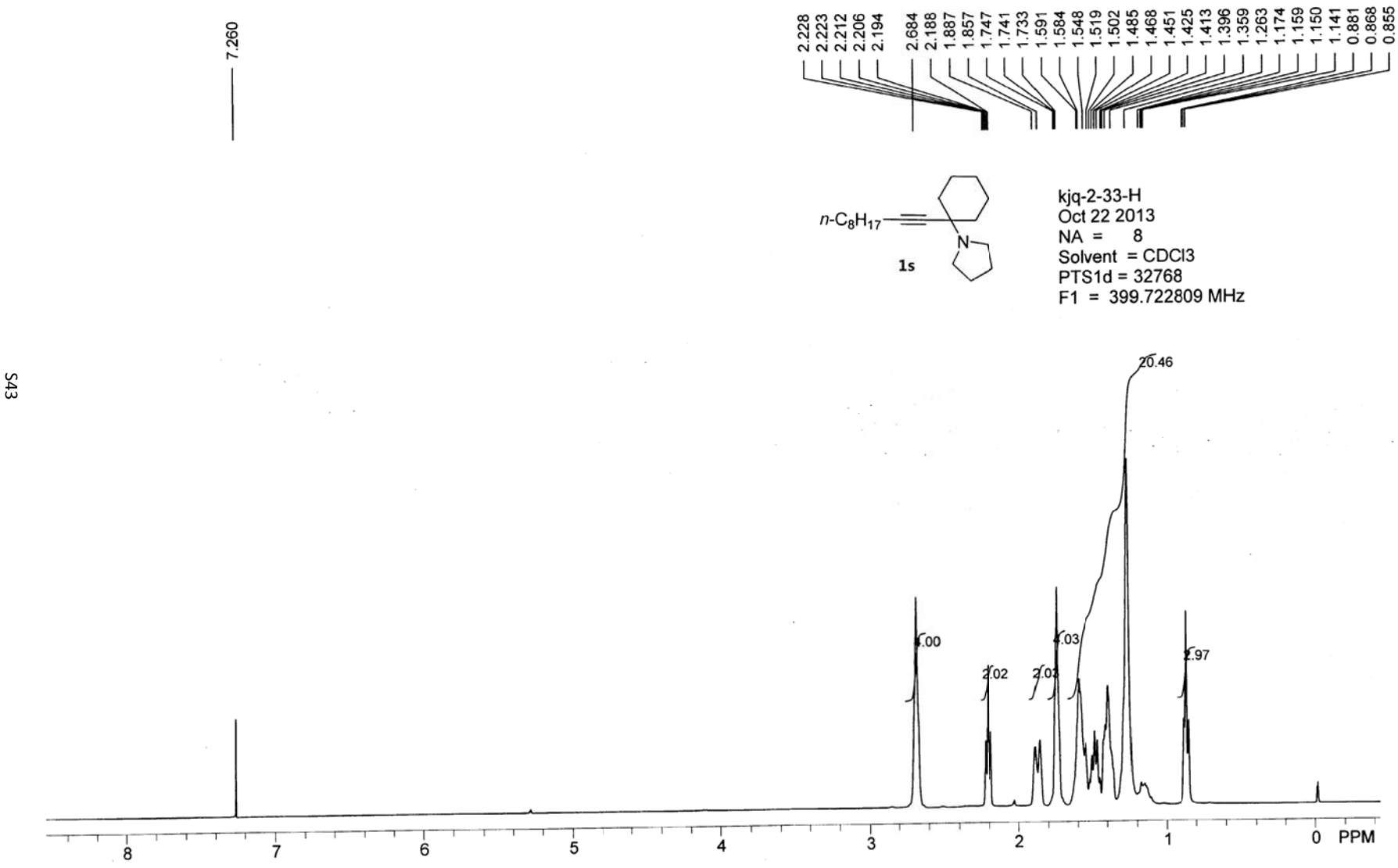
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1.733
1.591
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1.548
1.519
1.502
1.485
1.468
1.451
1.425
1.413
1.396
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1.174
1.159
1.150
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n-C8H17
N
1s
kjq-2-33-H
Oct 22 2013
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Solvent = CDCl3
PTS1d = 32768
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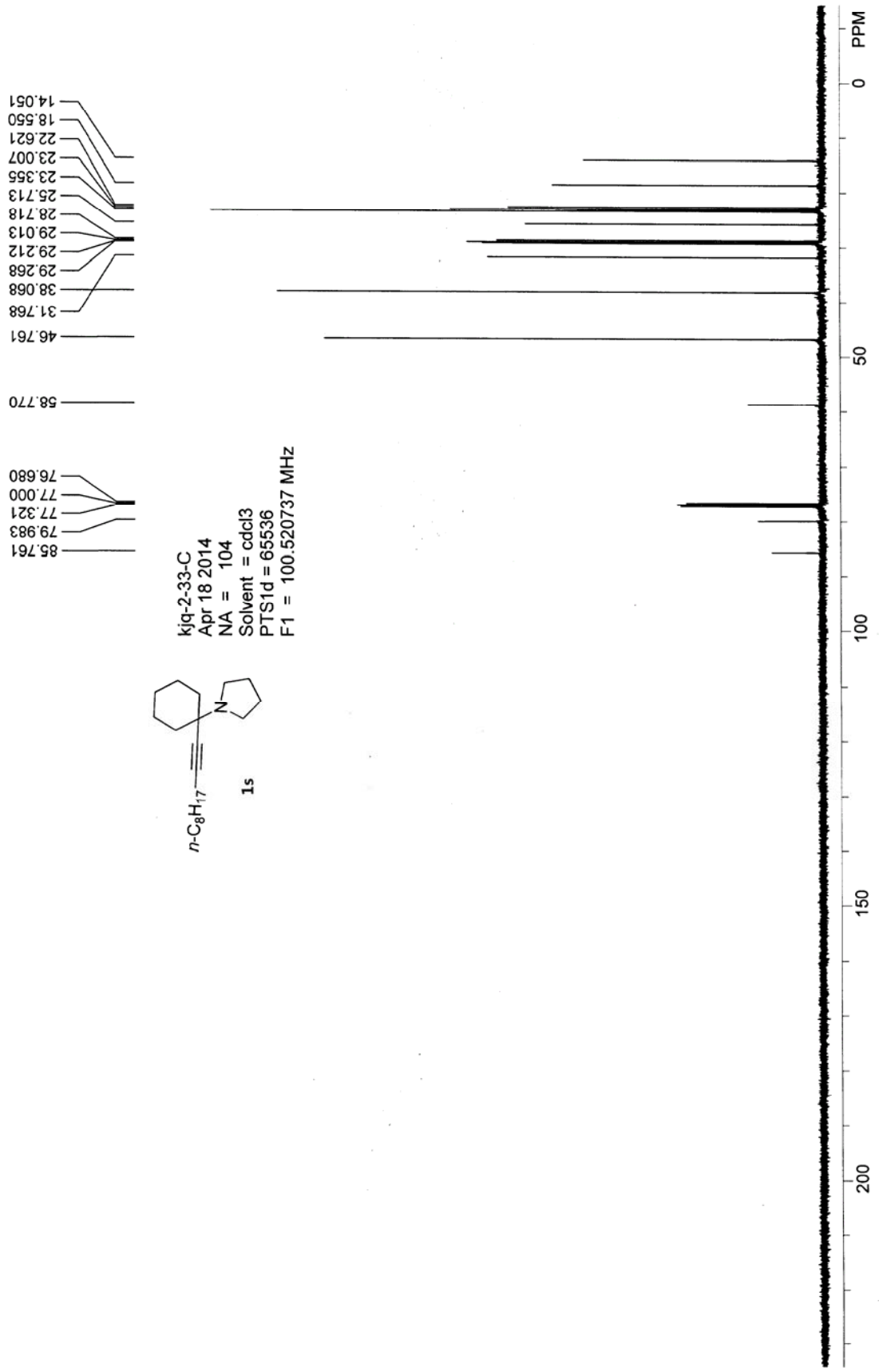
8
7
6
5
4
3
2
1
0
PPM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58.770
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77.000
77.321
79.983
85.761
kjq-2-33-C
Apr 18 2014
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F1 = 100.520737 MHz
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1s
PPM
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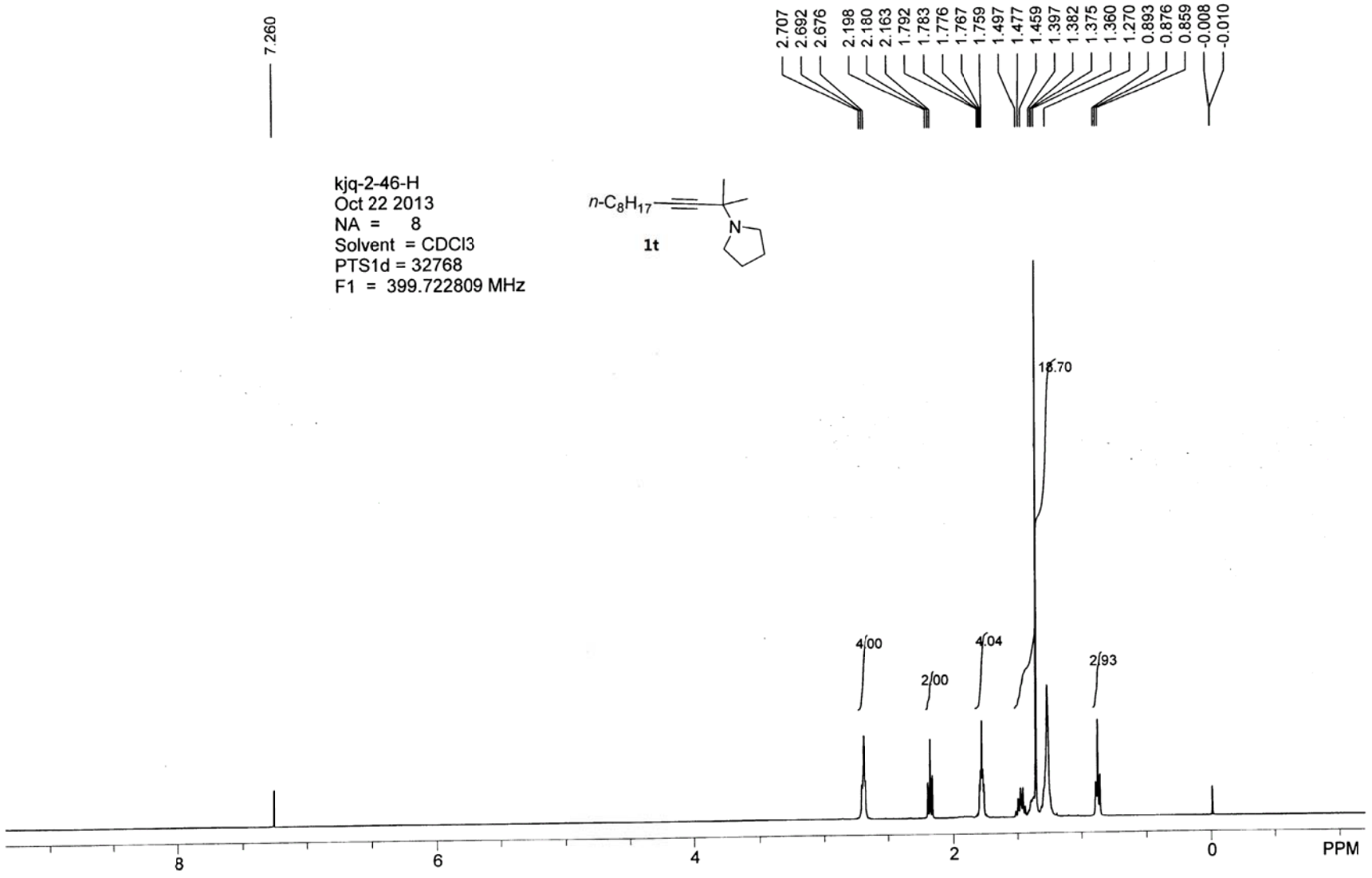
100
150
200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1.497
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1.459
1.397
1.382
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1.360
1.270
0.893
0.876
0.859
-0.008
-0.010
kjq-2-46-H
Oct 22 2013
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PTS1d = 32768
F1 = 399.722809 MHz
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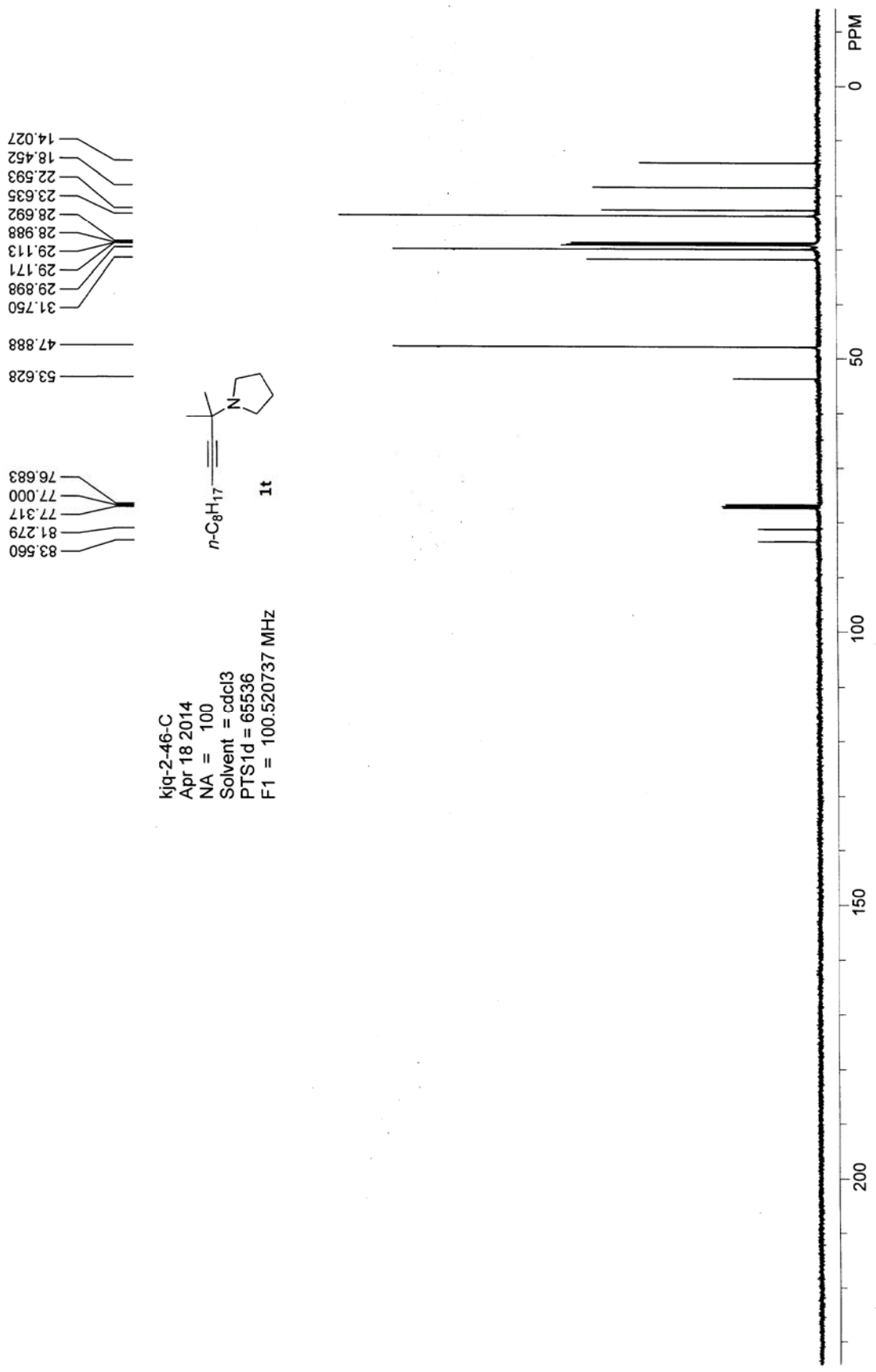
6
4
2
0
PPM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47.888
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77.000
77.317
81.279
83.560
kjq-2-46-C
Apr 18 2014
NA = 100
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1t
PPM
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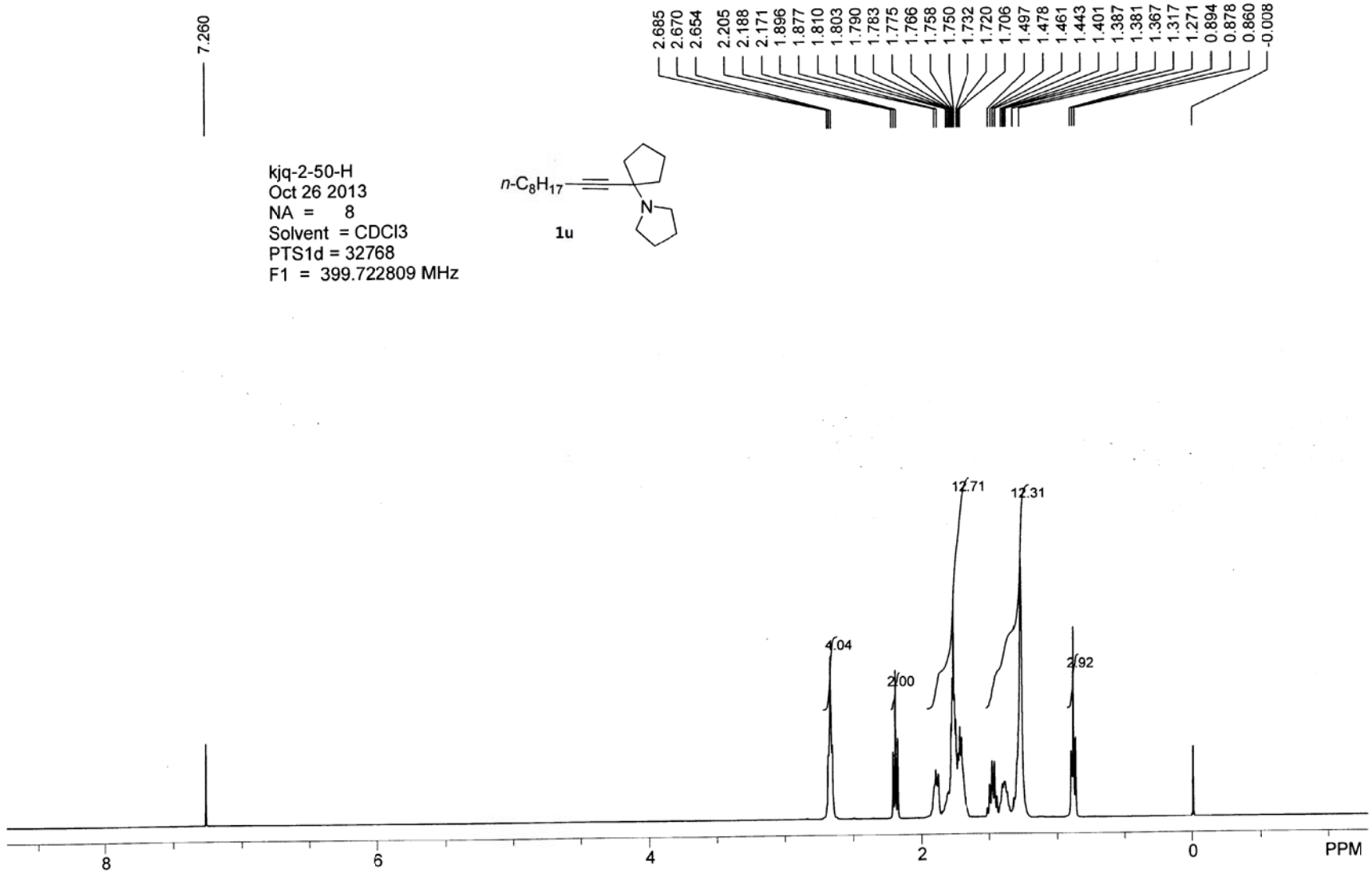
50
100
150
200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1.810
1.803
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1.783
1.775
1.766
1.758
1.750
1.732
1.720
1.706
1.497
1.478
1.461
1.443
1.401
1.387
1.381
1.367
1.317
1.271
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0.878
0.860
-0.008
kjq-2-50-H
Oct 26 2013
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F1 = 399.722809 MHz
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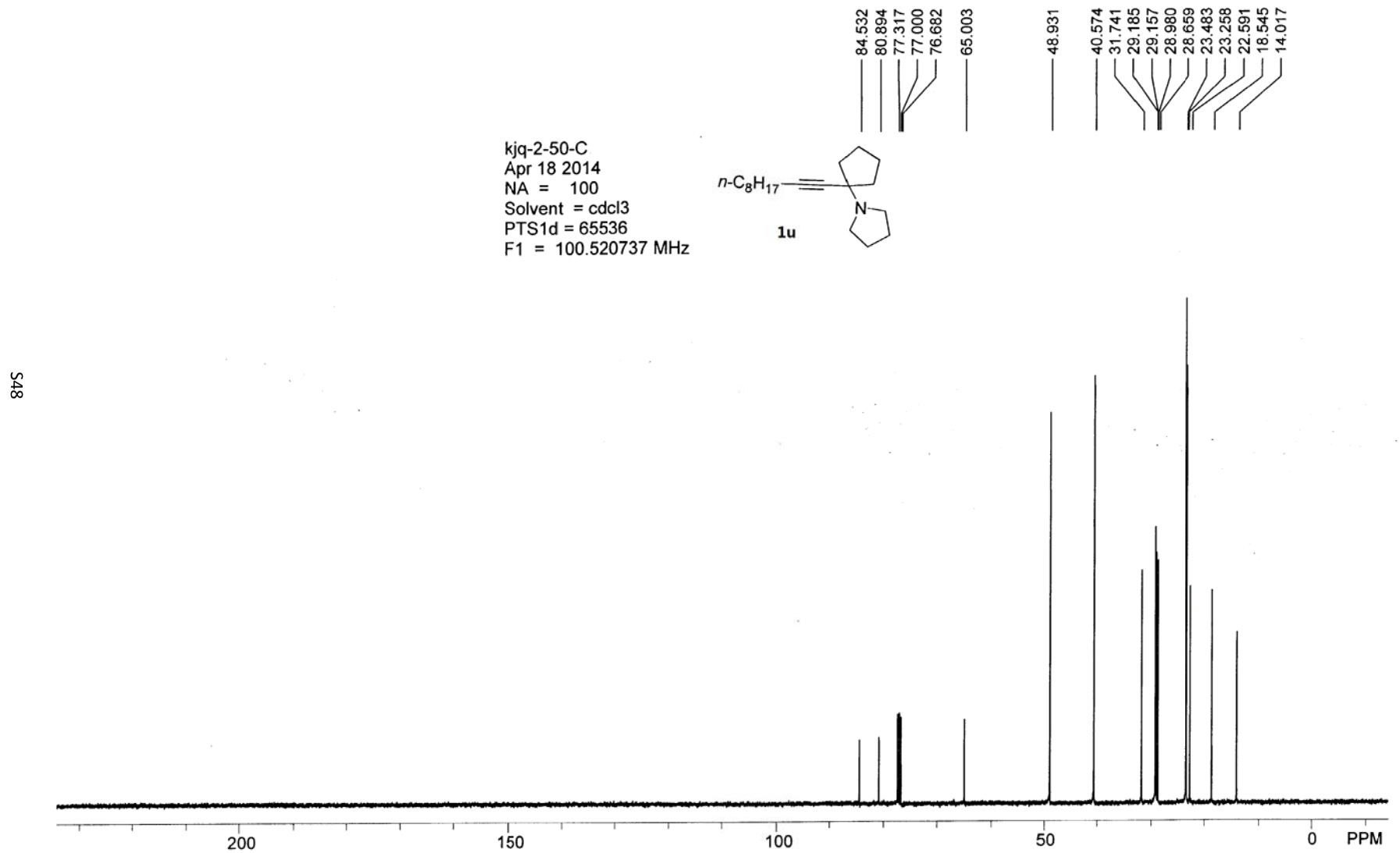
6
4
2
0
PPM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29.185
29.157
28.980
28.659
23.483
23.258
22.591
18.545
14.017
kjq-2-50-C
Apr 18 2014
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1u
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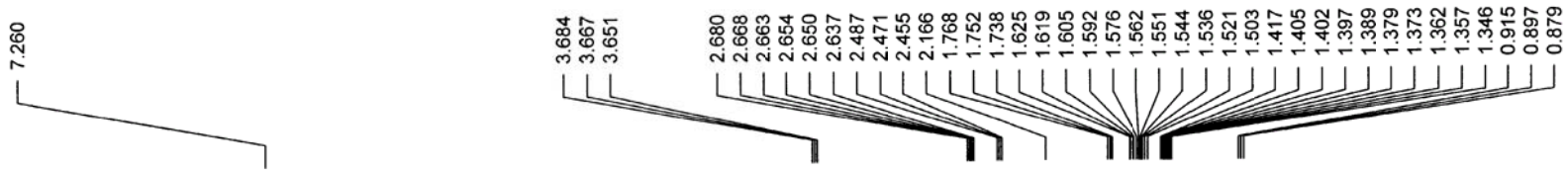
150
100
50
0
PPM



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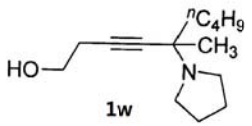

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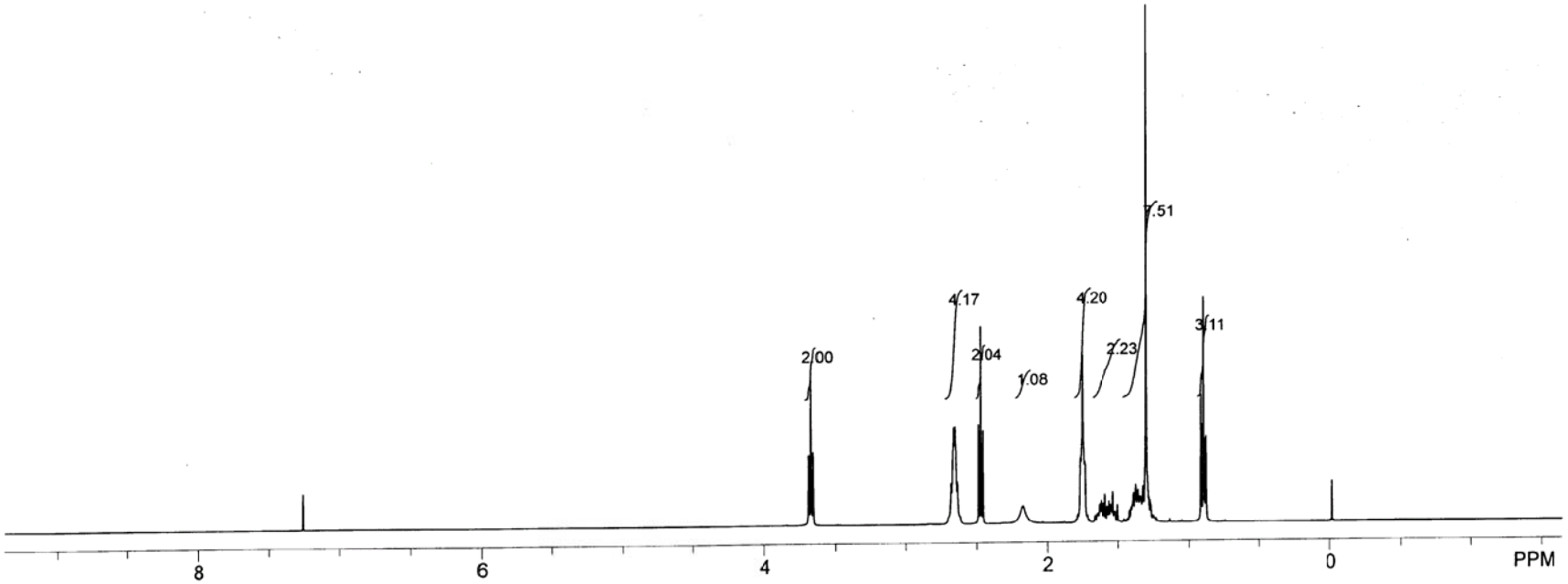

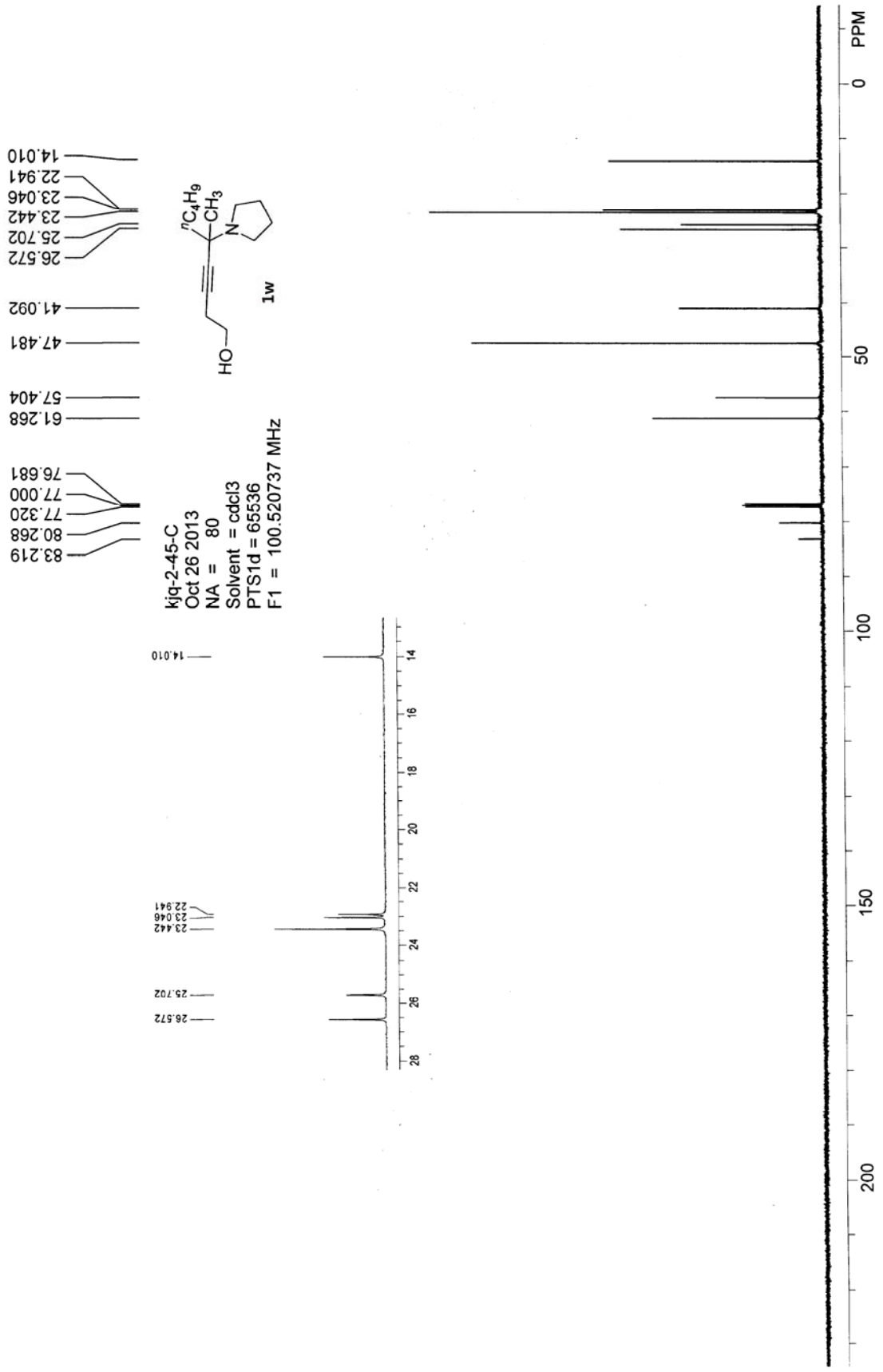
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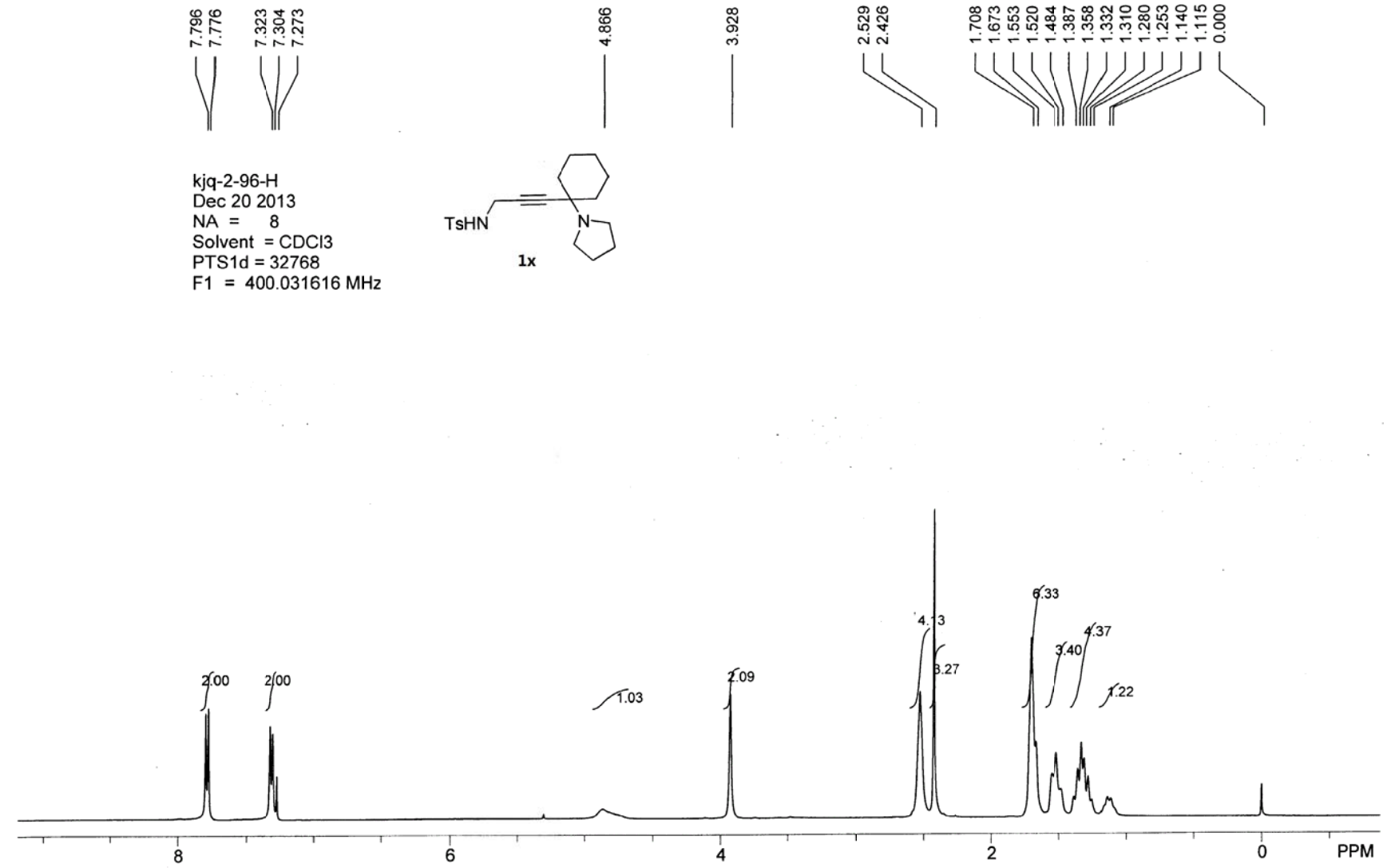
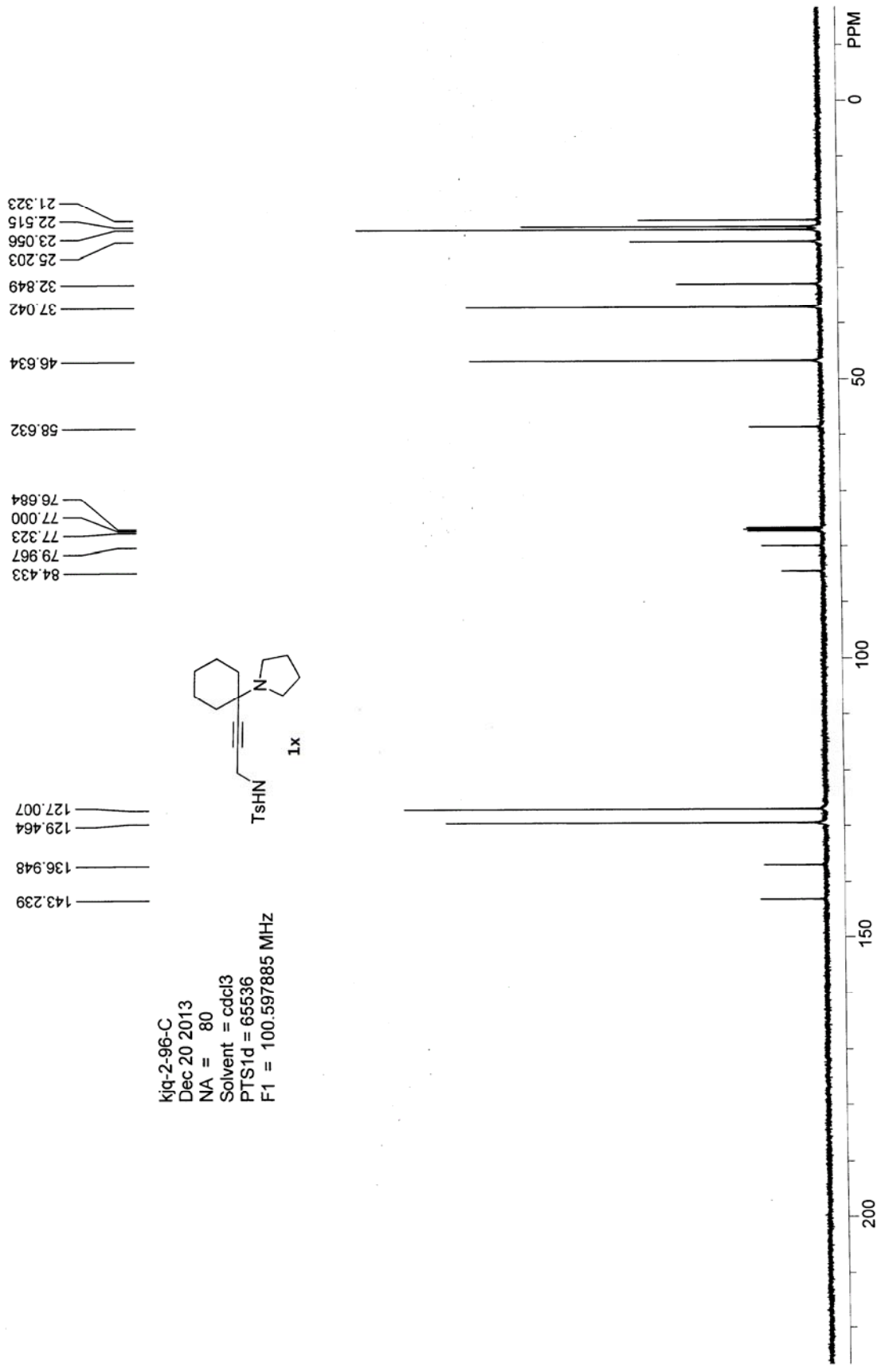
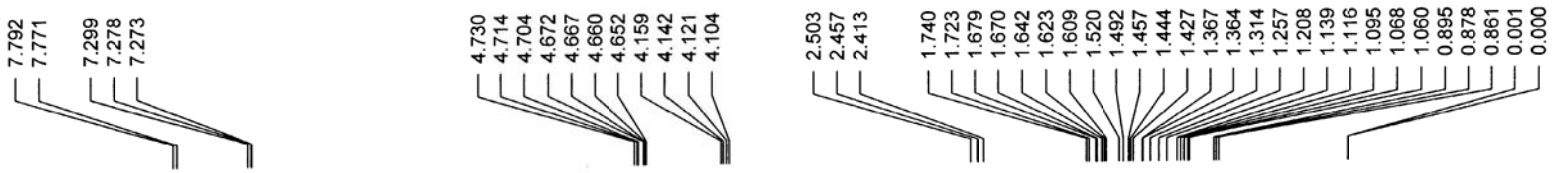
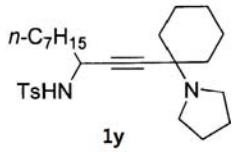
23.442
25.702
26.572
41.092
47.481
57.404
61.268
76.681
77.000
77.320
80.268
83.219
kjq-2-45-C
Oct 26 2013
NA = 80
Solvent = cdcl3
PTS1d = 65536
F1 = 100.520737 MHz
1w
HO
N
CH3
nC4H9
PPM
0
50
100
150
200


7.796
7.776
7.323
7.304
7.273
4.866
3.928
2.529
2.426
1.708
1.673
1.553
1.520
1.484
1.387
1.358
1.332
1.310
1.280
1.253
1.140
1.115
0.000
kjq-2-96-H
Dec 20 2013
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 400.031616 MHz
TsHN
N
1x
2.00
2.00
1.03
2.09
4.13
3.27
6.33
3.40
4.37
1.22
8
6
4
2
0
PPM

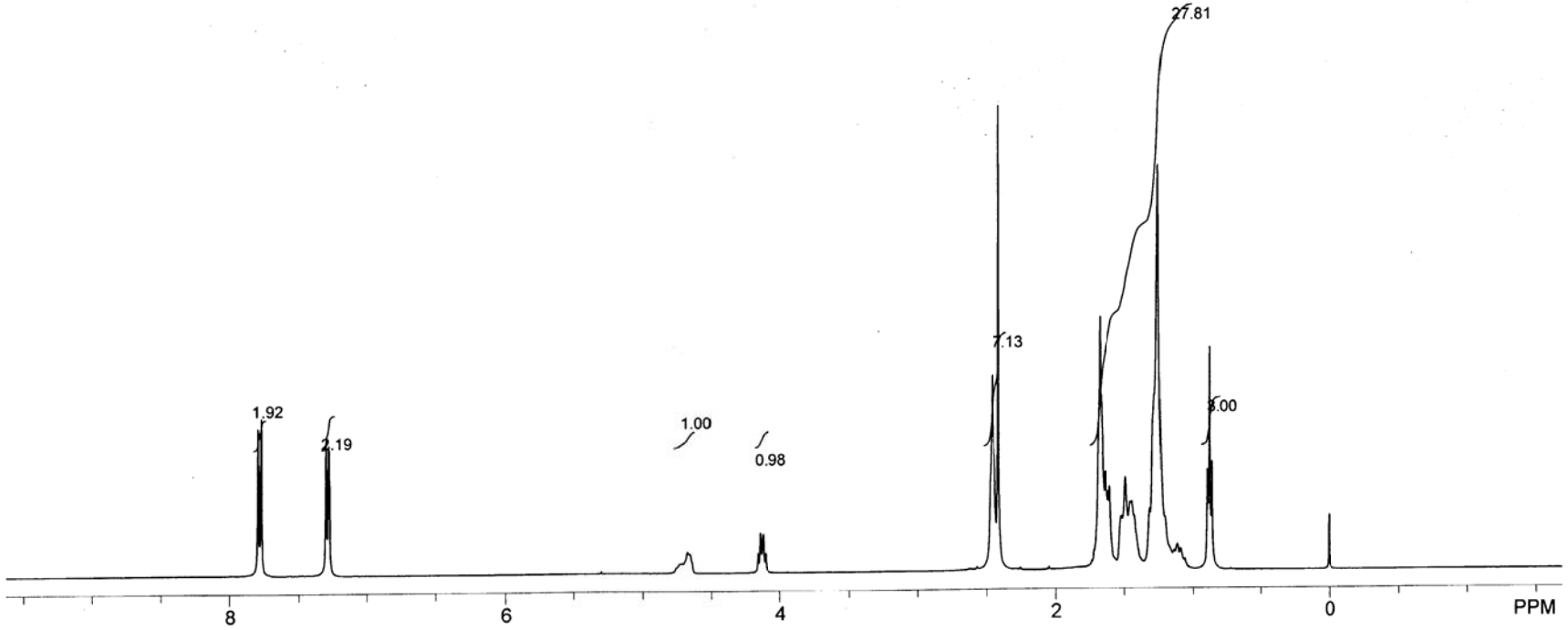

21.323
22.515
23.056
25.203
32.849
37.042
46.634
58.632
76.684
77.000
77.323
79.967
84.433
127.007
129.464
136.948
143.239
kjq-2-96-C
Dec 20 2013
NA = 80
Solvent = cdcl3
PTS1d = 65536
F1 = 100.597885 MHz
TsHN
N
1x
PPM
0
50
100
150
200

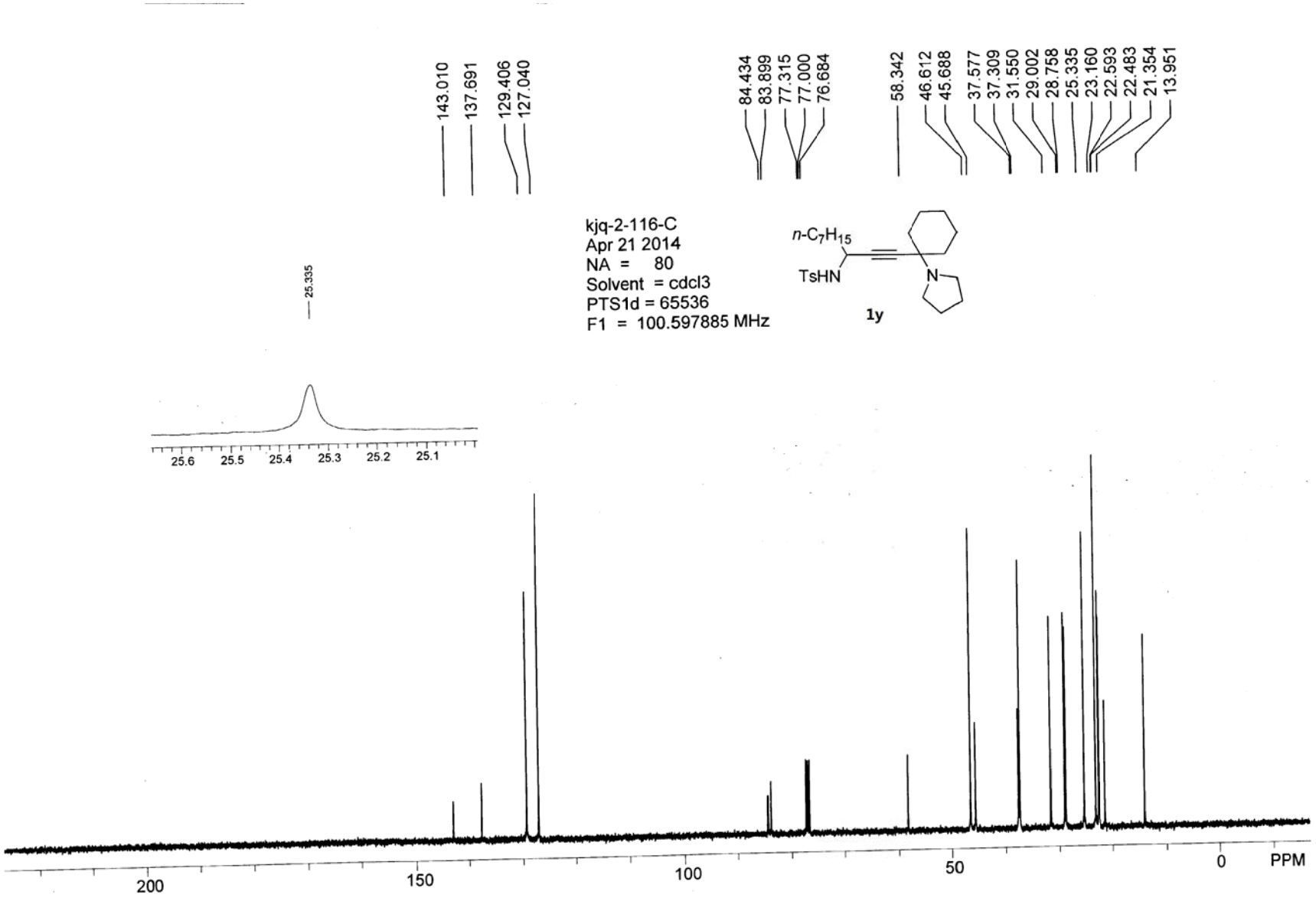


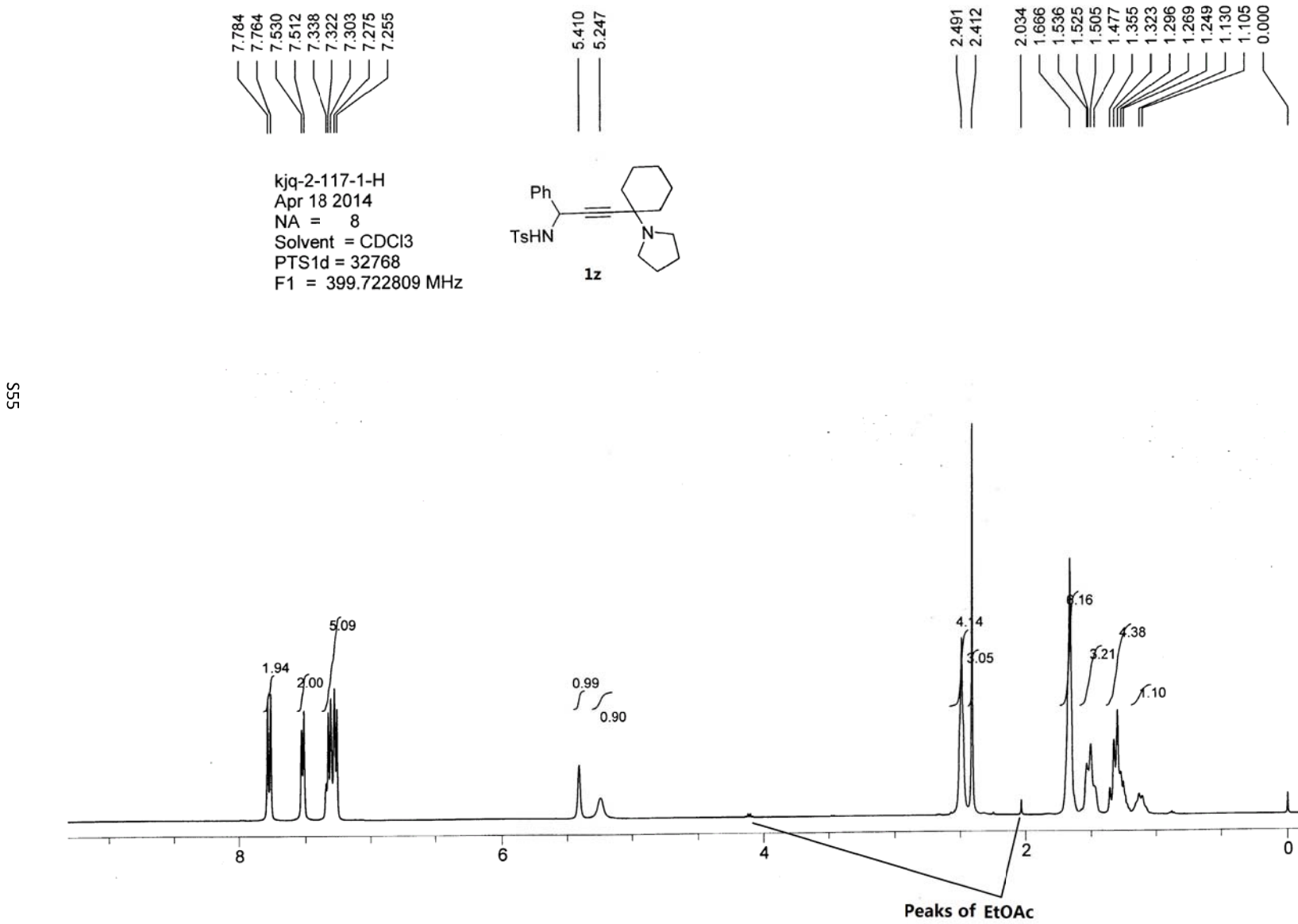
kjq-2-116-H
Jan 7 2014
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 400.031586 MHz

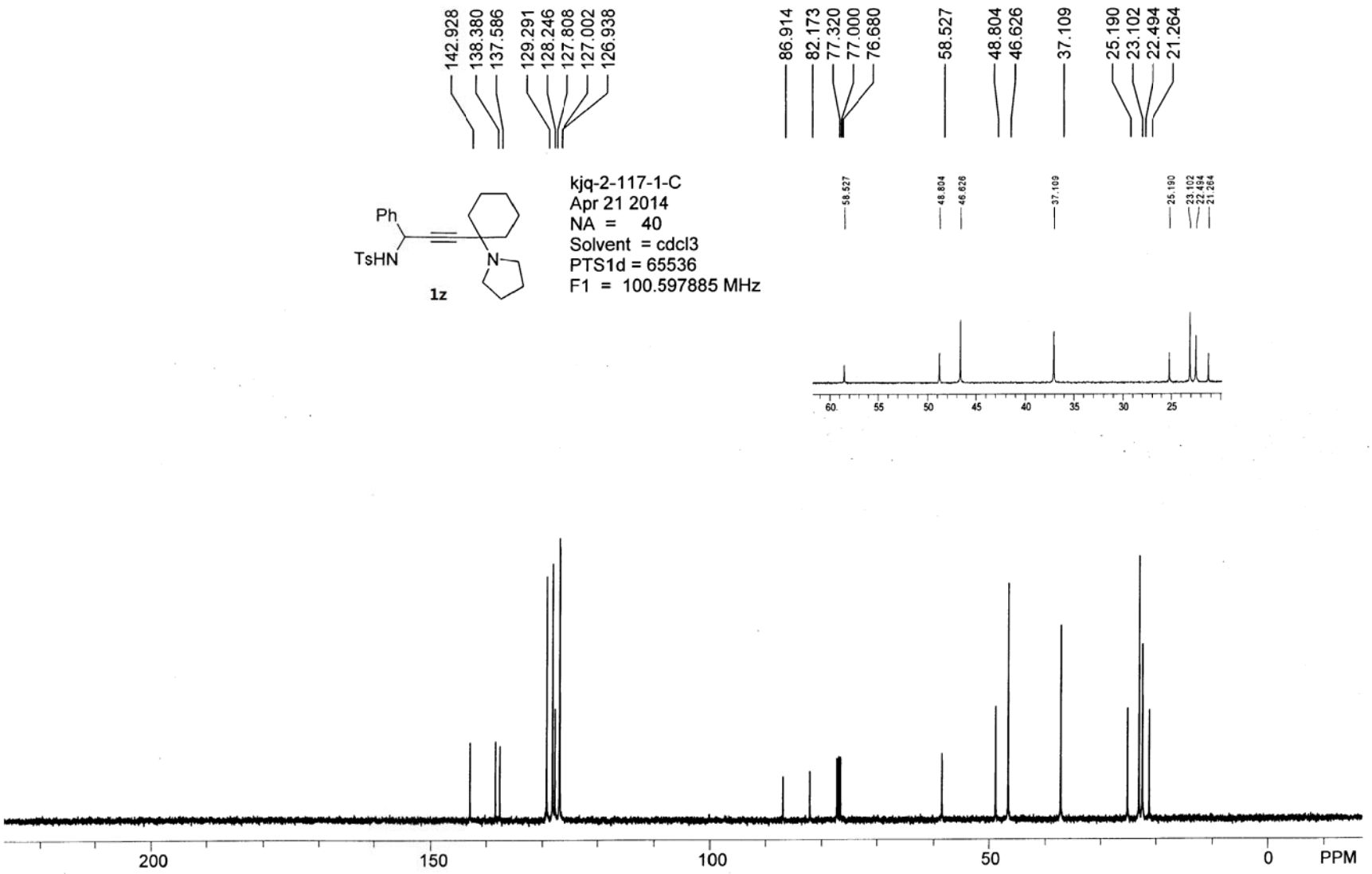


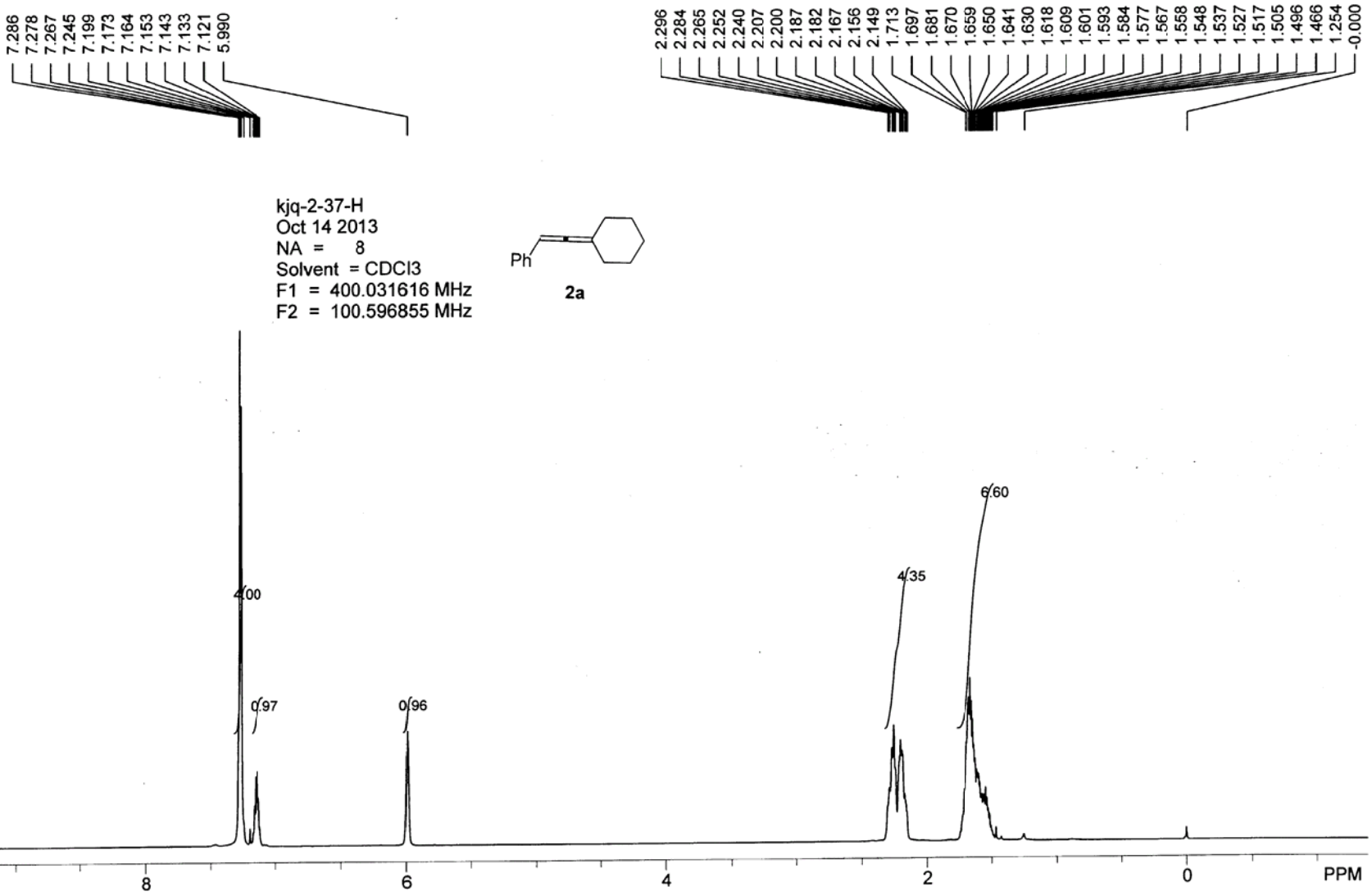
1y

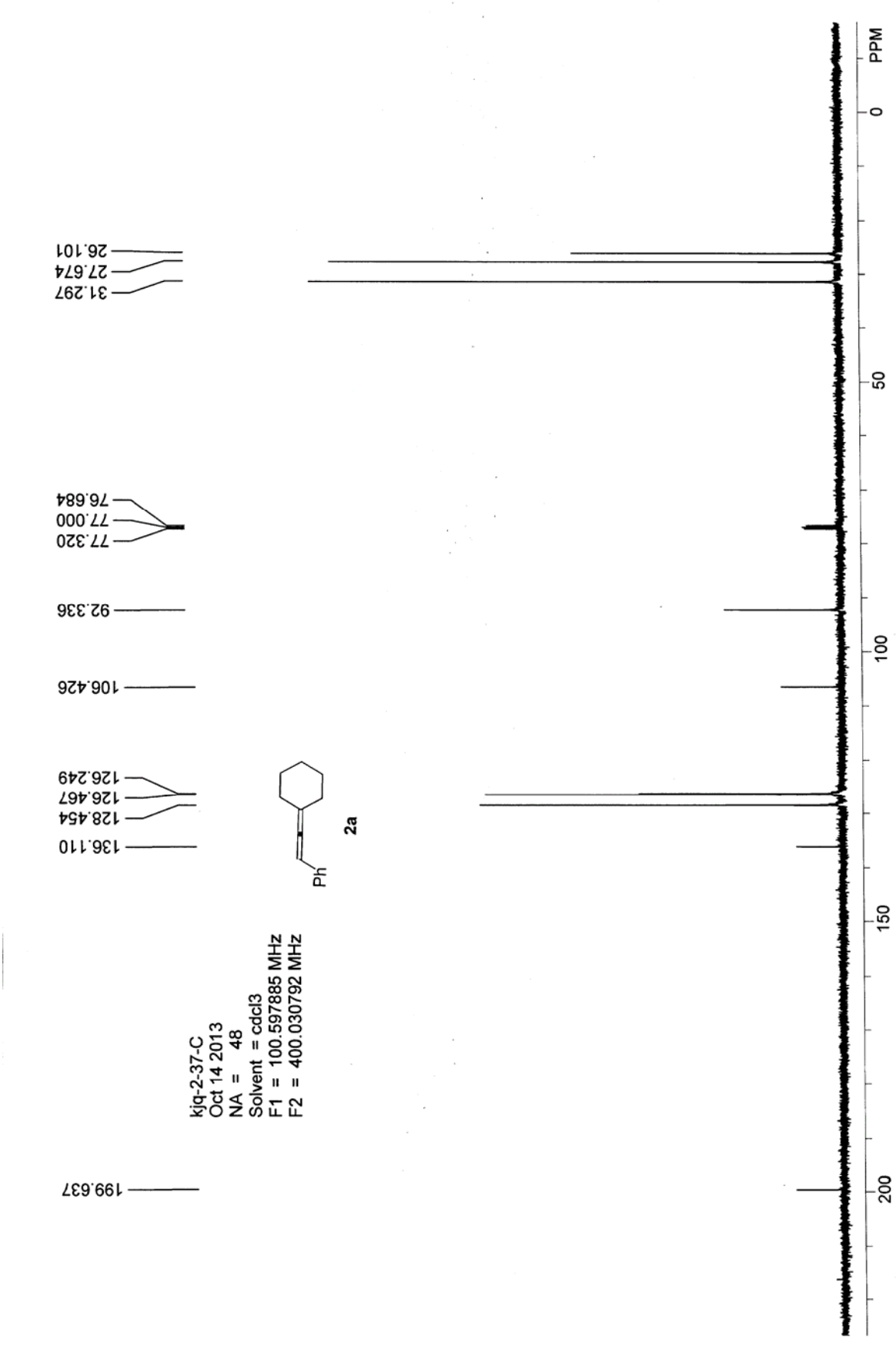


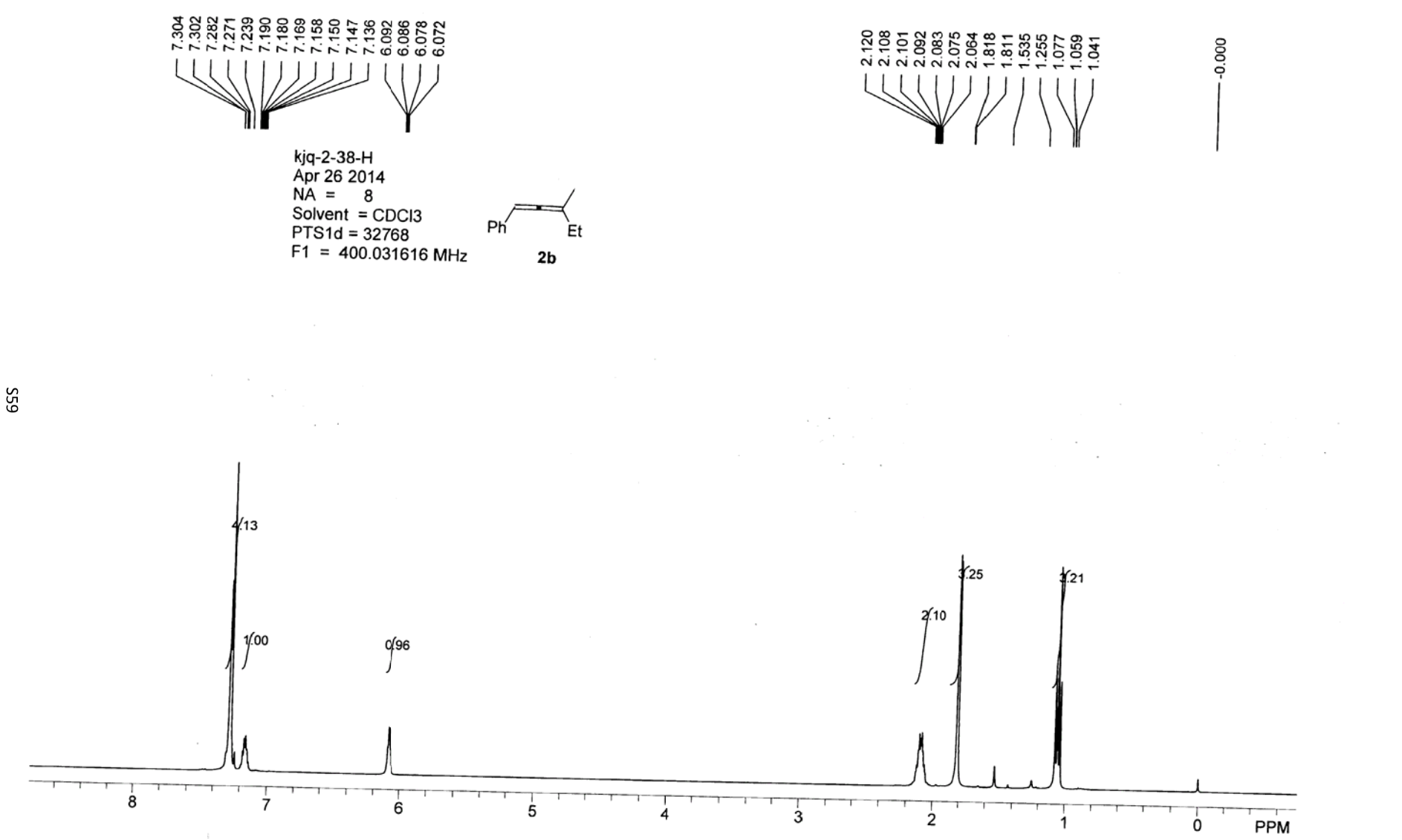

143.010
137.691
129.406
127.040
84.434
83.899
77.315
77.000
76.684
58.342
46.612
45.688
37.577
37.309
31.550
29.002
28.758
25.335
23.160
22.593
22.483
21.354
13.951
kjq-2-116-C
Apr 21 2014
NA = 80
Solvent = cdcl3
PTS1d = 65536
F1 = 100.597885 MHz
n-C7H15
TsHN
N
1y
25.335
25.6
25.5
25.4
25.3
25.2
25.1
200
150
100
50
0
PPM

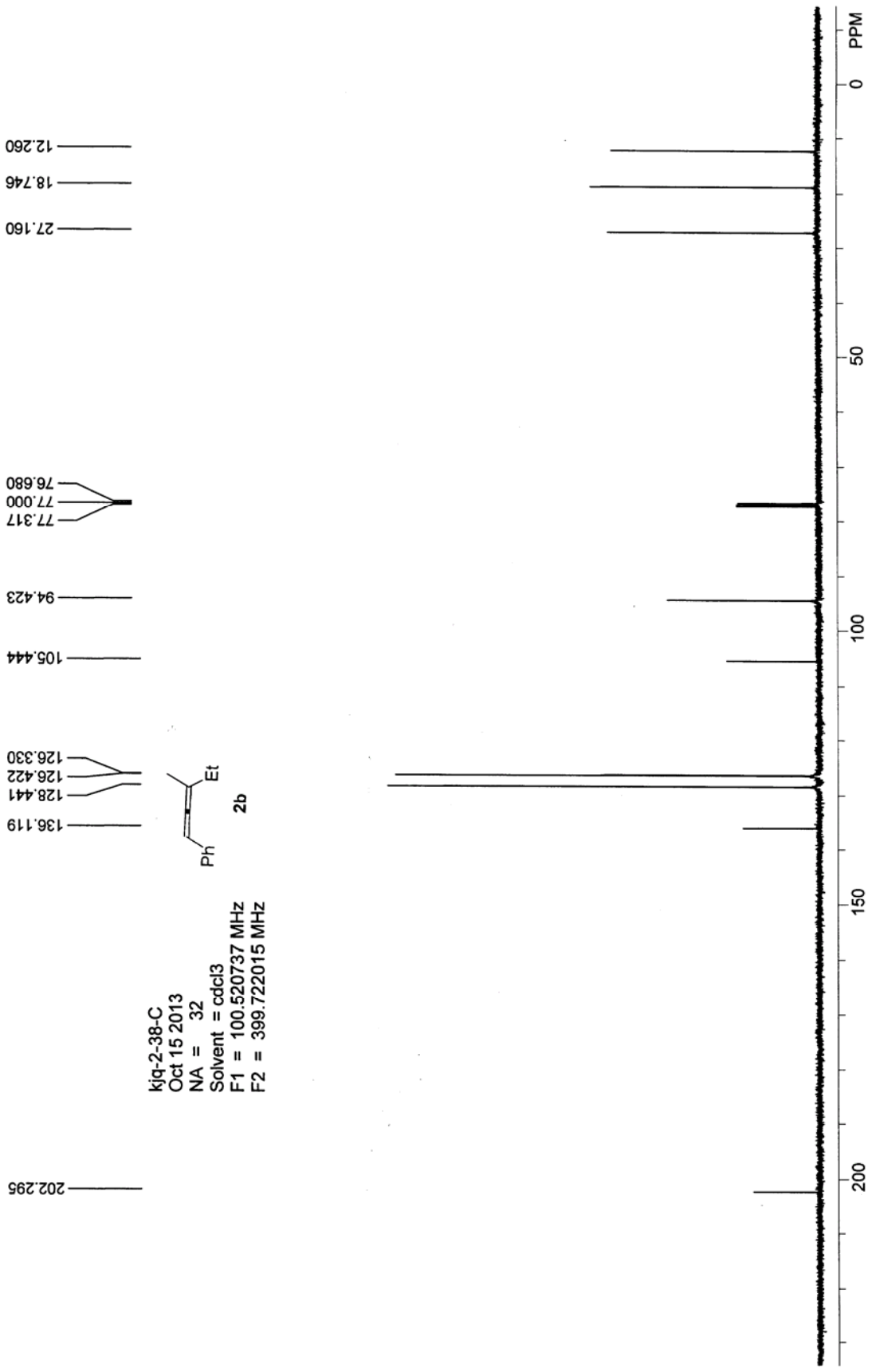

7.784
7.764
7.530
7.512
7.338
7.322
7.303
7.275
7.255
5.410
5.247
2.491
2.412
2.034
1.666
1.536
1.525
1.505
1.477
1.355
1.323
1.296
1.269
1.249
1.130
1.105
0.000
kjq-2-117-1-H
Apr 18 2014
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 399.722809 MHz
Ph
TsHN
N
1z
1.94
2.00
5.09
0.99
0.90
4.14
3.05
6.16
3.21
4.38
1.10
8
6
4
2
0
PPM
Peaks of EtOAc

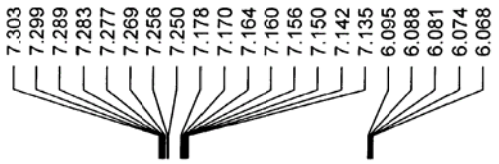

142.928
138.380
137.586
129.291
128.246
127.808
127.002
126.938
86.914
82.173
77.320
77.000
76.680
58.527
48.804
46.626
37.109
25.190
23.102
22.494
21.264
kjq-2-117-1-C
Apr 21 2014
NA = 40
Solvent = cdcl3
PTS1d = 65536
F1 = 100.597885 MHz
Ph
TsHN
N
1z
200
150
100
50
0
PPM

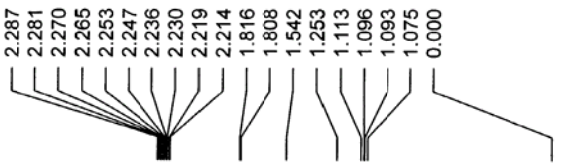

7.286
7.278
7.267
7.245
7.199
7.173
7.164
7.153
7.143
7.133
7.121
5.990
2.296
2.284
2.265
2.252
2.240
2.207
2.200
2.187
2.182
2.167
2.156
2.149
1.713
1.697
1.681
1.670
1.659
1.650
1.641
1.630
1.618
1.609
1.601
1.593
1.584
1.577
1.567
1.558
1.548
1.537
1.527
1.517
1.505
1.496
1.466
1.254
-0.000
kjq-2-37-H
Oct 14 2013
NA = 8
Solvent = CDCl3
F1 = 400.031616 MHz
F2 = 100.596855 MHz
Ph
2a
4.00
0.97
0.96
4.35
6.60
8
6
4
2
0
PPM


199.637
136.110
128.454
126.467
126.249
106.426
92.336
77.320
77.000
76.684
31.297
27.674
26.101
kjq-2-37-C
Oct 14 2013
NA = 48
Solvent = cdcl3
F1 = 100.597885 MHz
F2 = 400.030792 MHz
Ph
2a
PPM
0
50
100
150
200

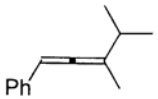

7.304
7.302
7.282
7.271
7.239
7.190
7.180
7.169
7.158
7.150
7.147
7.136
6.092
6.086
6.078
6.072
2.120
2.108
2.101
2.092
2.083
2.075
2.064
1.818
1.811
1.535
1.255
1.077
1.059
1.041
-0.000
kjq-2-38-H
Apr 26 2014
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 400.031616 MHz
Ph
Et
2b
4.13
1.00
0.96
2.10
3.25
3.21
8
7
6
5
4
3
2
1
0
PPM


12.260
18.746
27.160
76.680
77.000
77.317
94.423
105.444
126.330
126.422
128.441
136.119
202.295
Et
Ph
2b
kjq-2-38-C
Oct 15 2013
NA = 32
Solvent = cdcl3
F1 = 100.520737 MHz
F2 = 399.722015 MHz
PPM
0
50
100
150
200

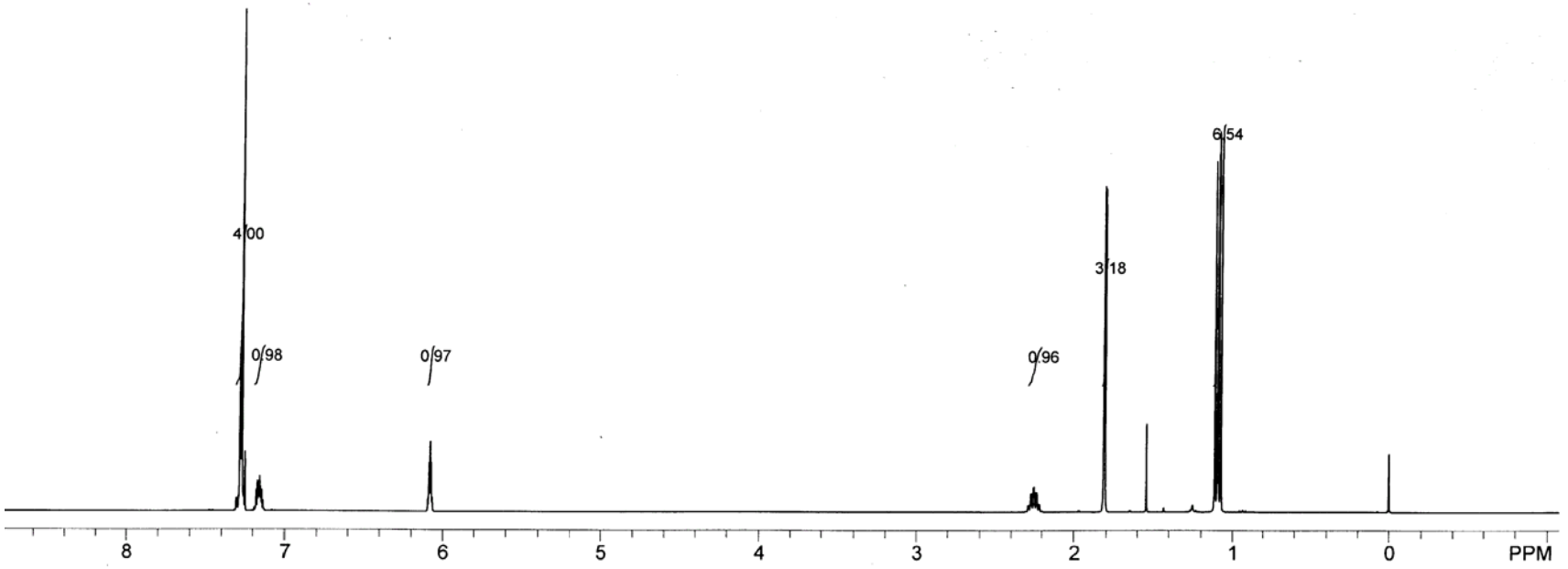


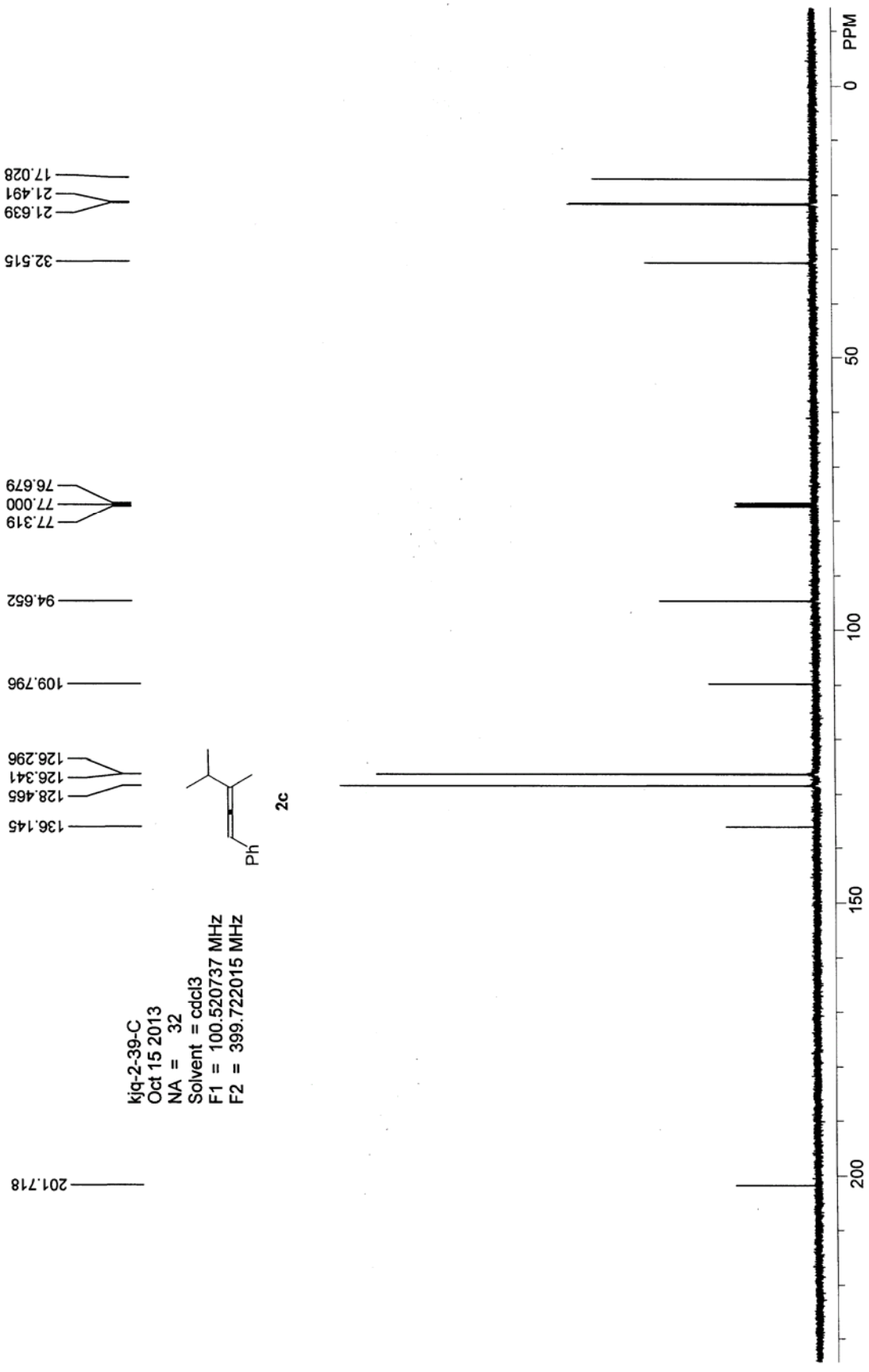


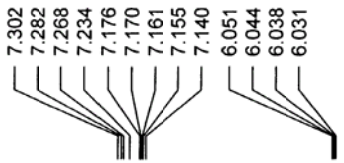
kjq-2-39-H
Apr 27 2014
NA = 12
Solvent = CDCl3
PTS1d = 32768
F1 = 399.722809 MHz

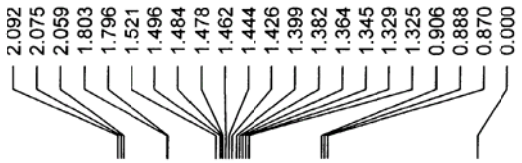


2c

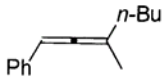



kjq-2-39-C
Oct 15 2013
NA = 32
Solvent = cdcl3
F1 = 100.520737 MHz
F2 = 399.722015 MHz
2c
Ph
201.718
136.145
128.465
126.341
126.296
109.796
94.652
77.319
77.000
76.679
32.515
21.639
21.491
17.028
PPM
0
50
100
150
200

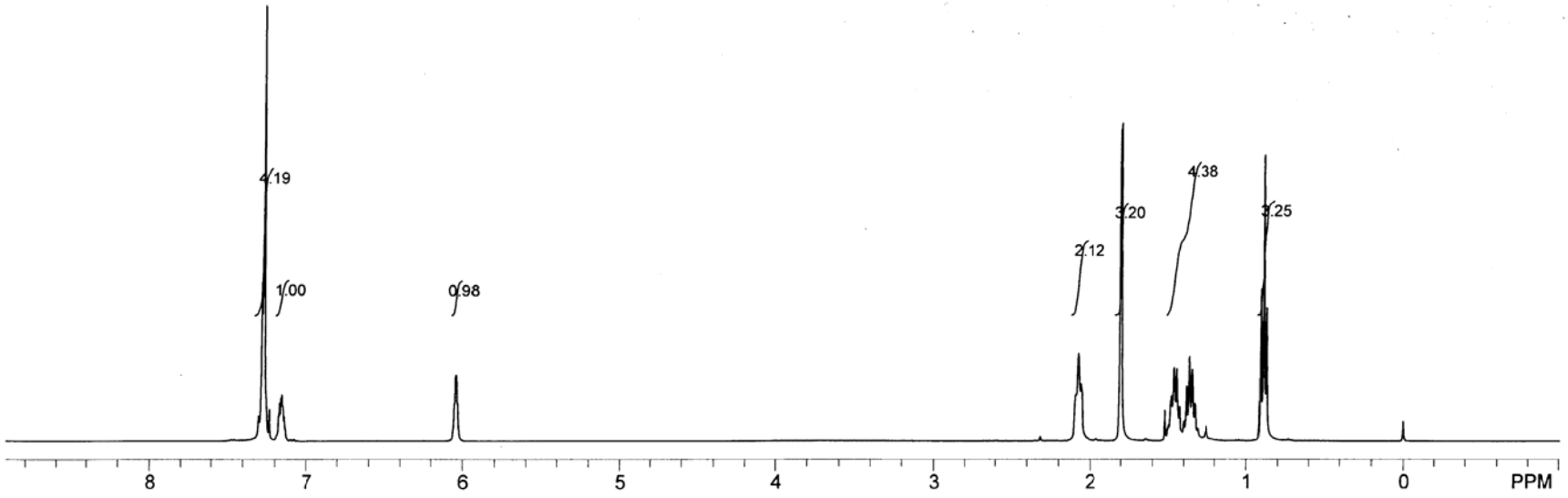


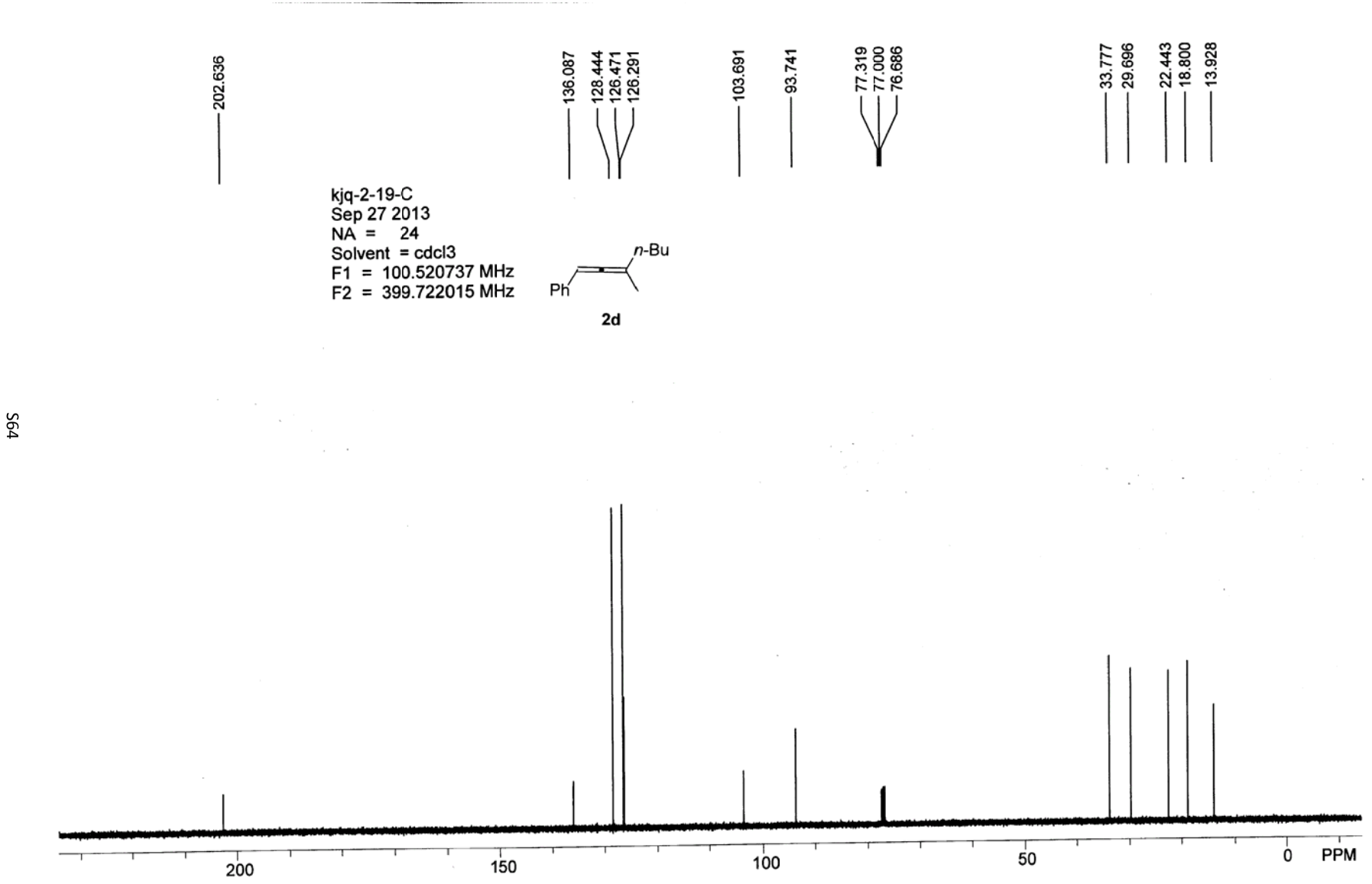


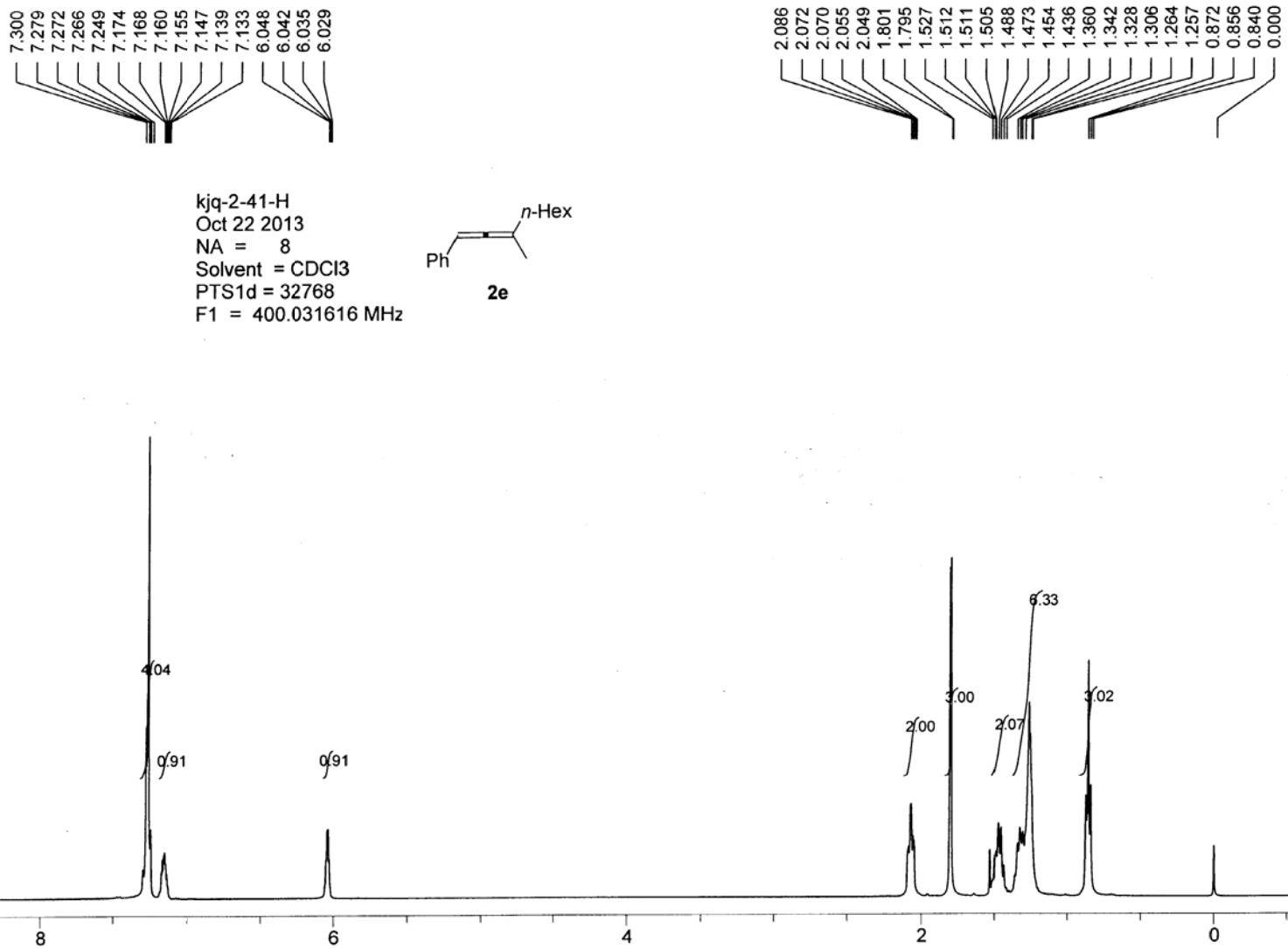
kjq-2-19-H
Sep 27 2013
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 400.031616 MHz

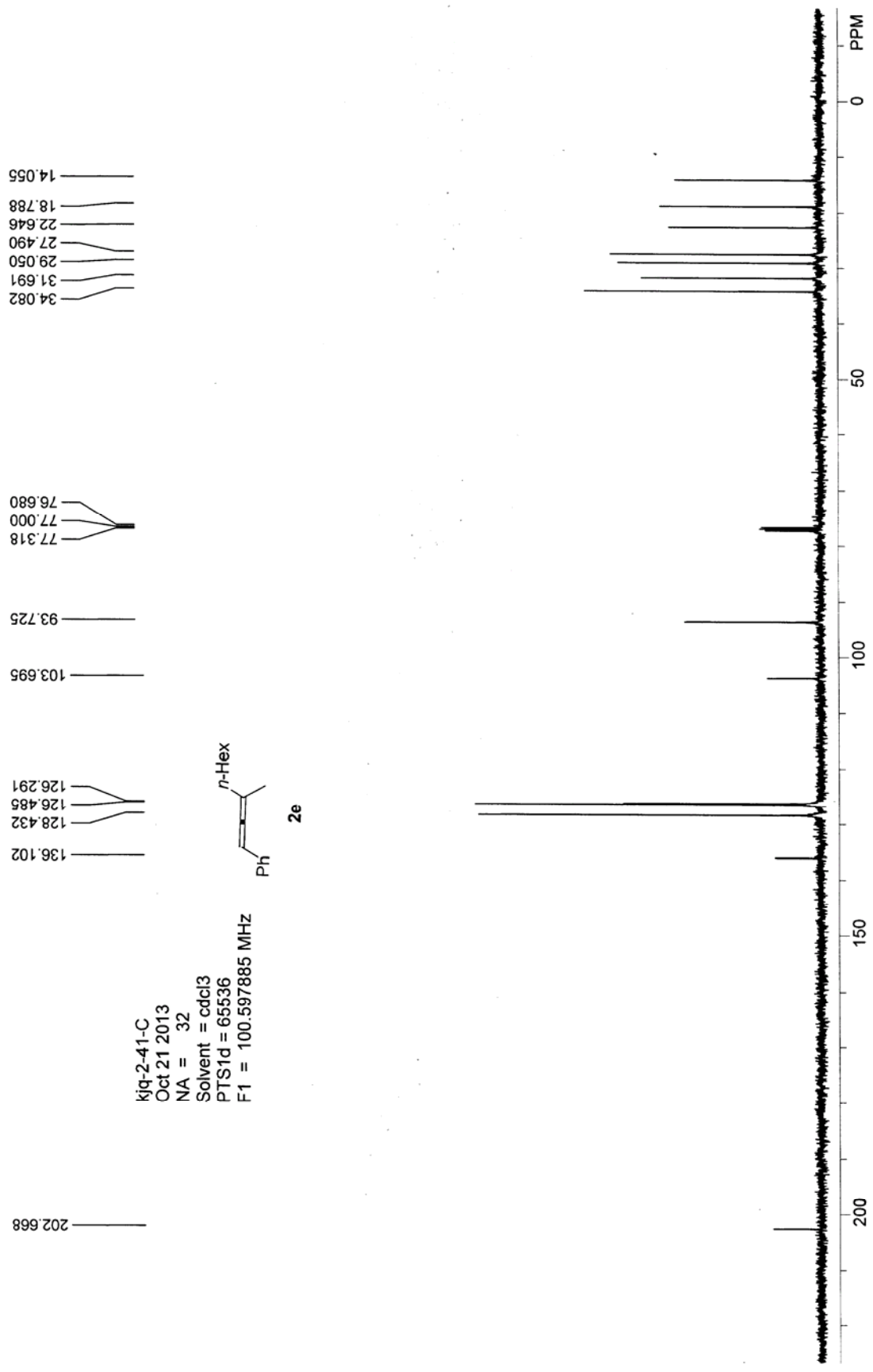


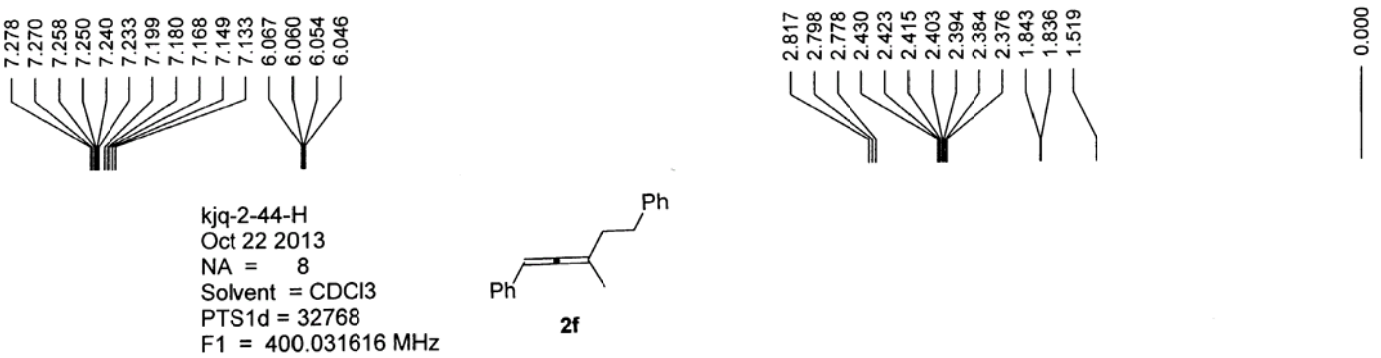
2d

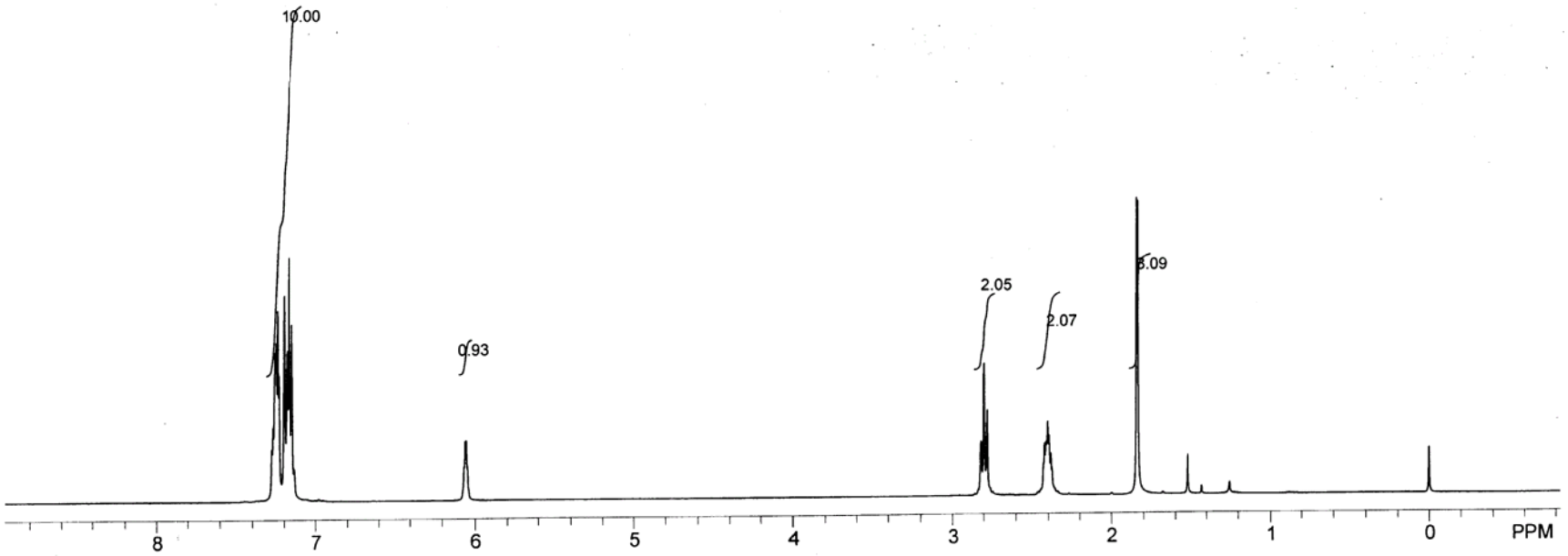


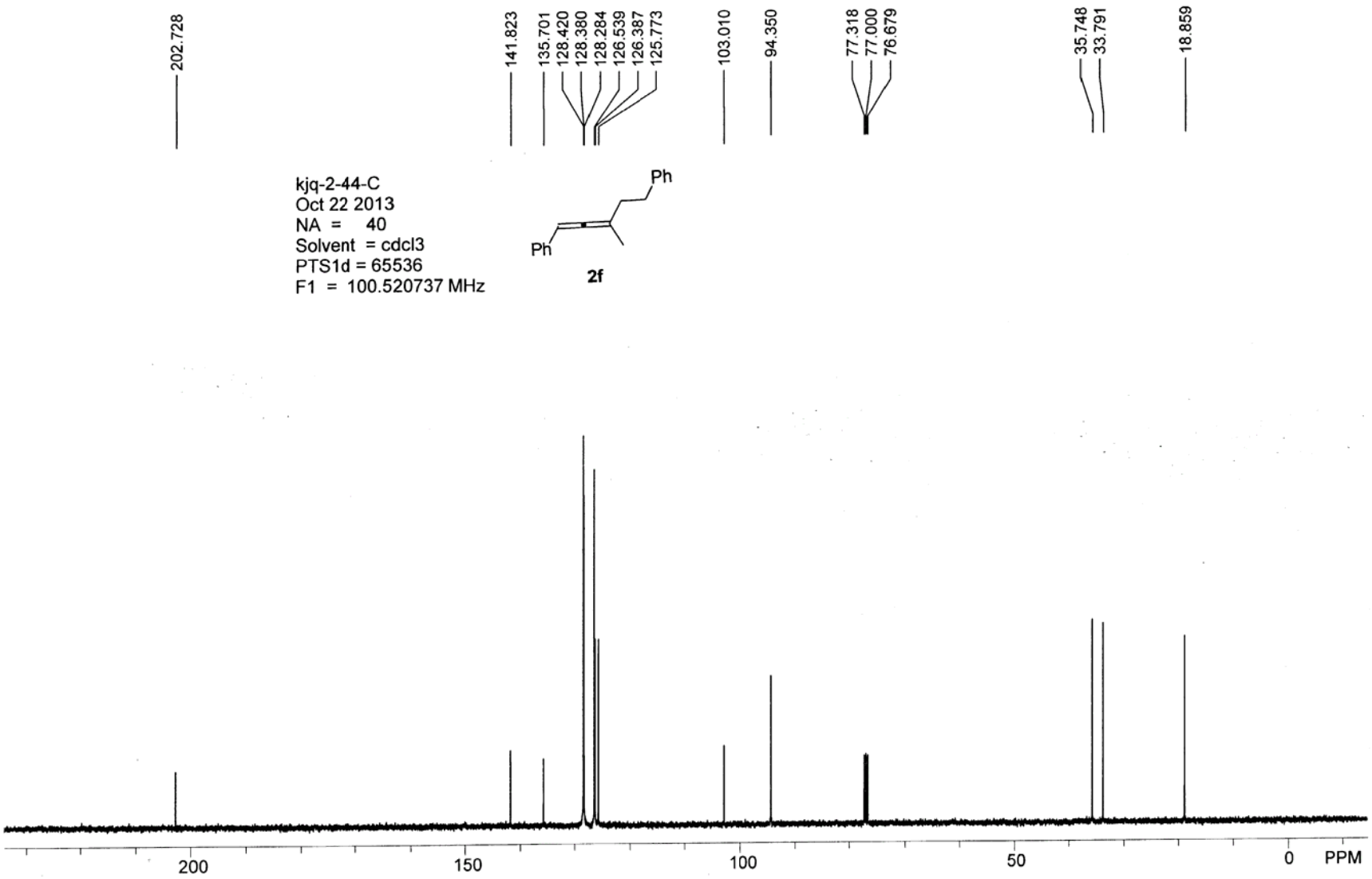

202.636
136.087
128.444
126.471
126.291
103.691
93.741
77.319
77.000
76.686
33.777
29.696
22.443
18.800
13.928
kjq-2-19-C
Sep 27 2013
NA = 24
Solvent = cdcl3
F1 = 100.520737 MHz
F2 = 399.722015 MHz
n-Bu
Ph
2d
200
150
100
50
0
PPM

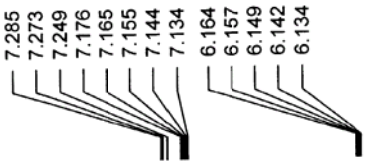

7.300
7.279
7.272
7.266
7.249
7.174
7.168
7.160
7.155
7.147
7.139
7.133
6.048
6.042
6.035
6.029
2.086
2.072
2.070
2.055
2.049
1.801
1.795
1.527
1.512
1.511
1.505
1.488
1.473
1.454
1.436
1.360
1.342
1.328
1.306
1.264
1.257
0.872
0.856
0.840
0.000
kjq-2-41-H
Oct 22 2013
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 400.031616 MHz
n-Hex
Ph
2e
4.04
0.91
0.91
2.00
3.00
2.07
6.33
3.02
8
6
4
2
0
PPM

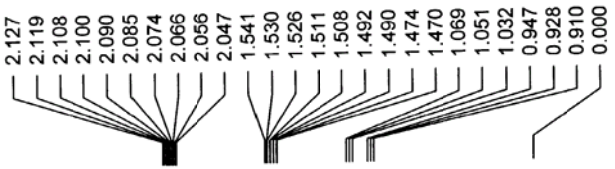

14.055
18.788
22.646
27.490
29.050
31.691
34.082
76.680
77.000
77.318
93.725
103.695
126.291
126.485
128.432
136.102
202.668
n-Hex
Ph
2e
kjq-2-41-C
Oct 21 2013
NA = 32
Solvent = cdcl3
PTS1d = 65536
F1 = 100.597885 MHz
PPM
0
50
100
150
200


7.278
7.270
7.258
7.250
7.240
7.233
7.199
7.180
7.168
7.149
7.133
6.067
6.060
6.054
6.046
2.817
2.798
2.778
2.430
2.423
2.415
2.403
2.394
2.384
2.376
1.843
1.836
1.519
0.000
kjq-2-44-H
Oct 22 2013
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 400.031616 MHz
Ph
Ph
2f

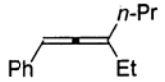

10.00
0.93
2.05
2.07
3.09
8
7
6
5
4
3
2
1
0
PPM


202.728
141.823
135.701
128.420
128.380
128.284
126.539
126.387
125.773
103.010
94.350
77.318
77.000
76.679
35.748
33.791
18.859
kjq-2-44-C
Oct 22 2013
NA = 40
Solvent = cdcl3
PTS1d = 65536
F1 = 100.520737 MHz
Ph
Ph
2f
200
150
100
50
0
PPM

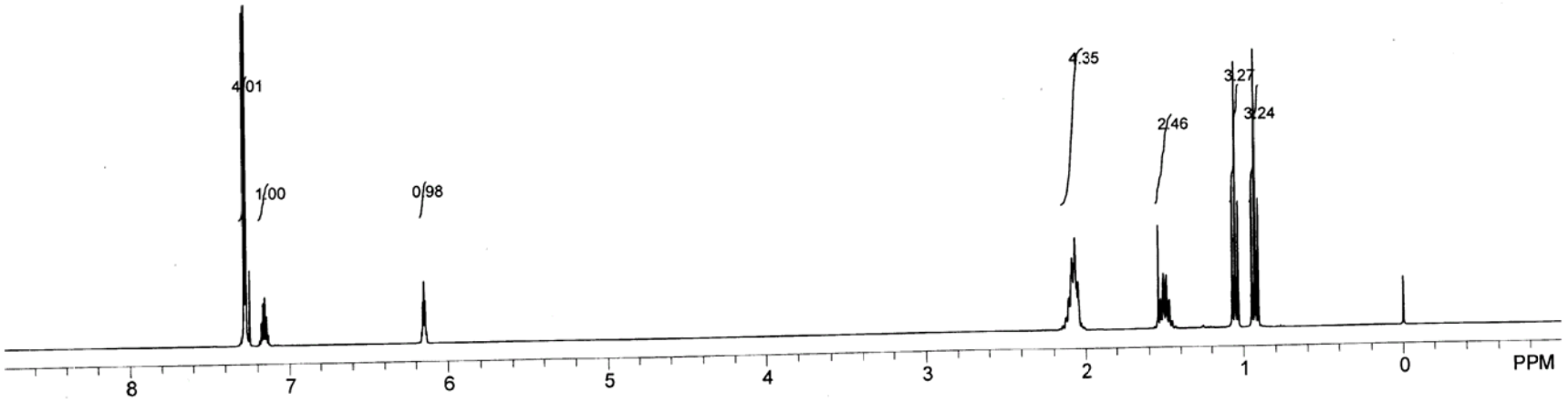


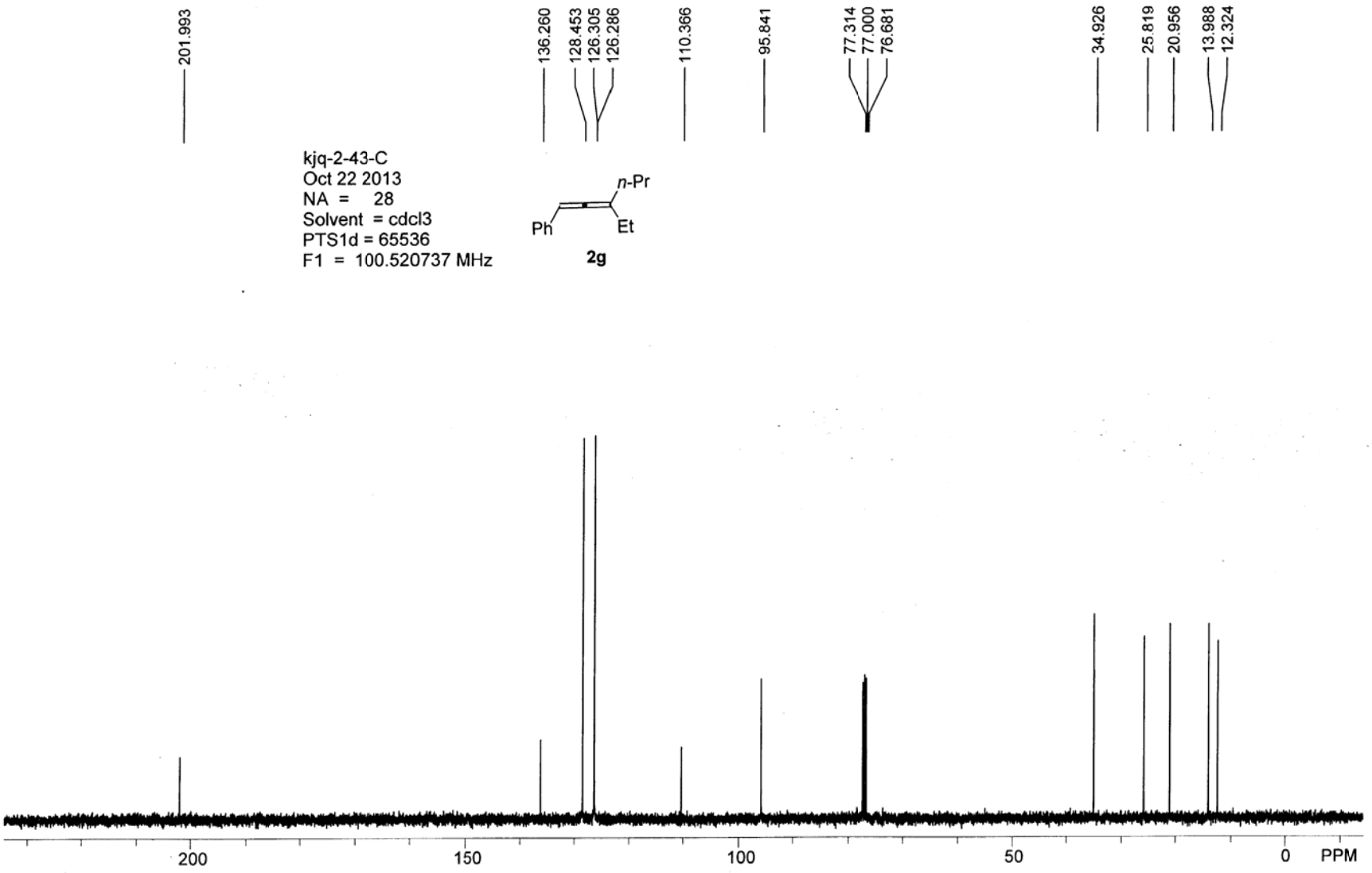


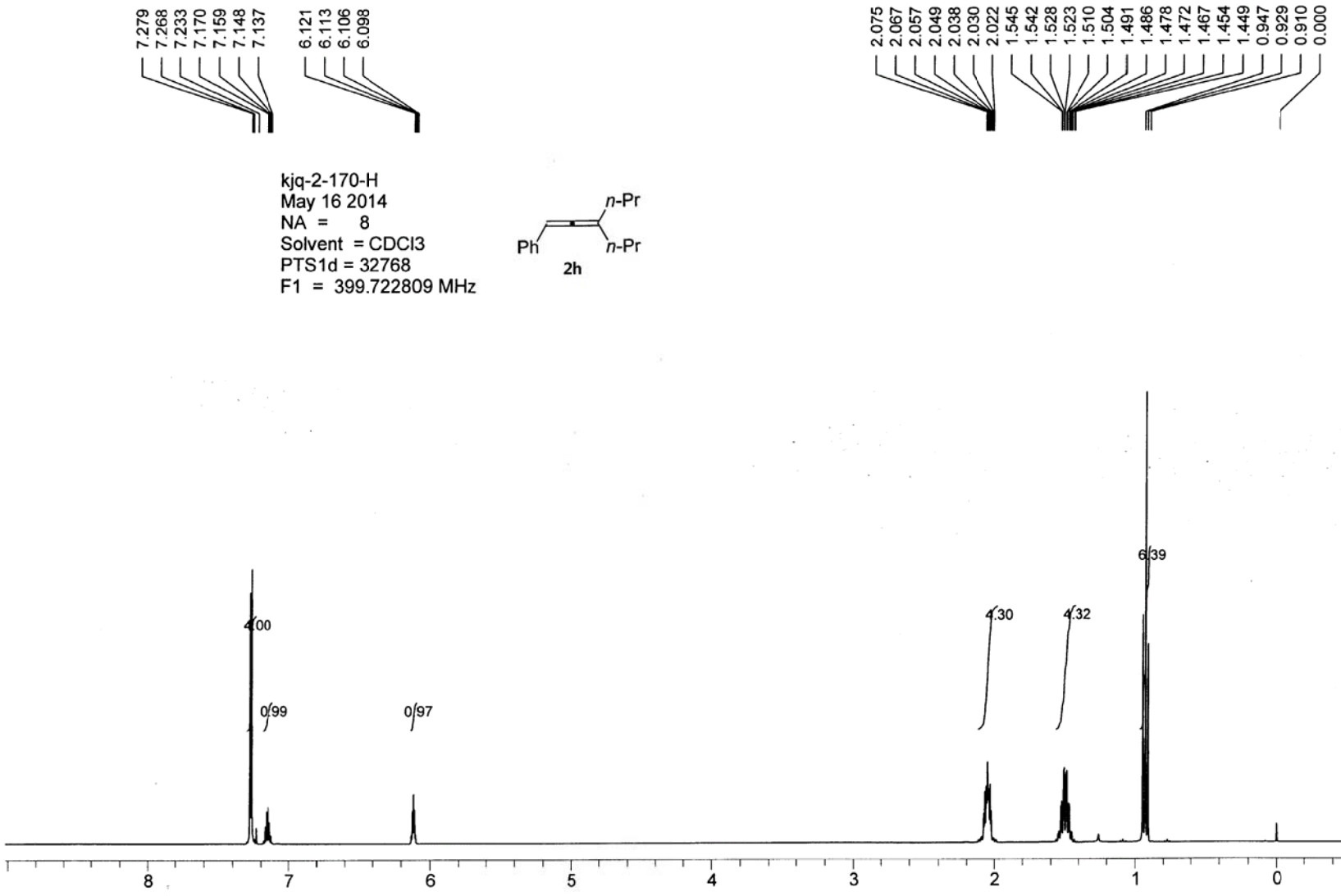
kjq-2-43-H
Apr 27 2014
NA = 12
Solvent = CDCl3
PTS1d = 32768
F1 = 399.722809 MHz

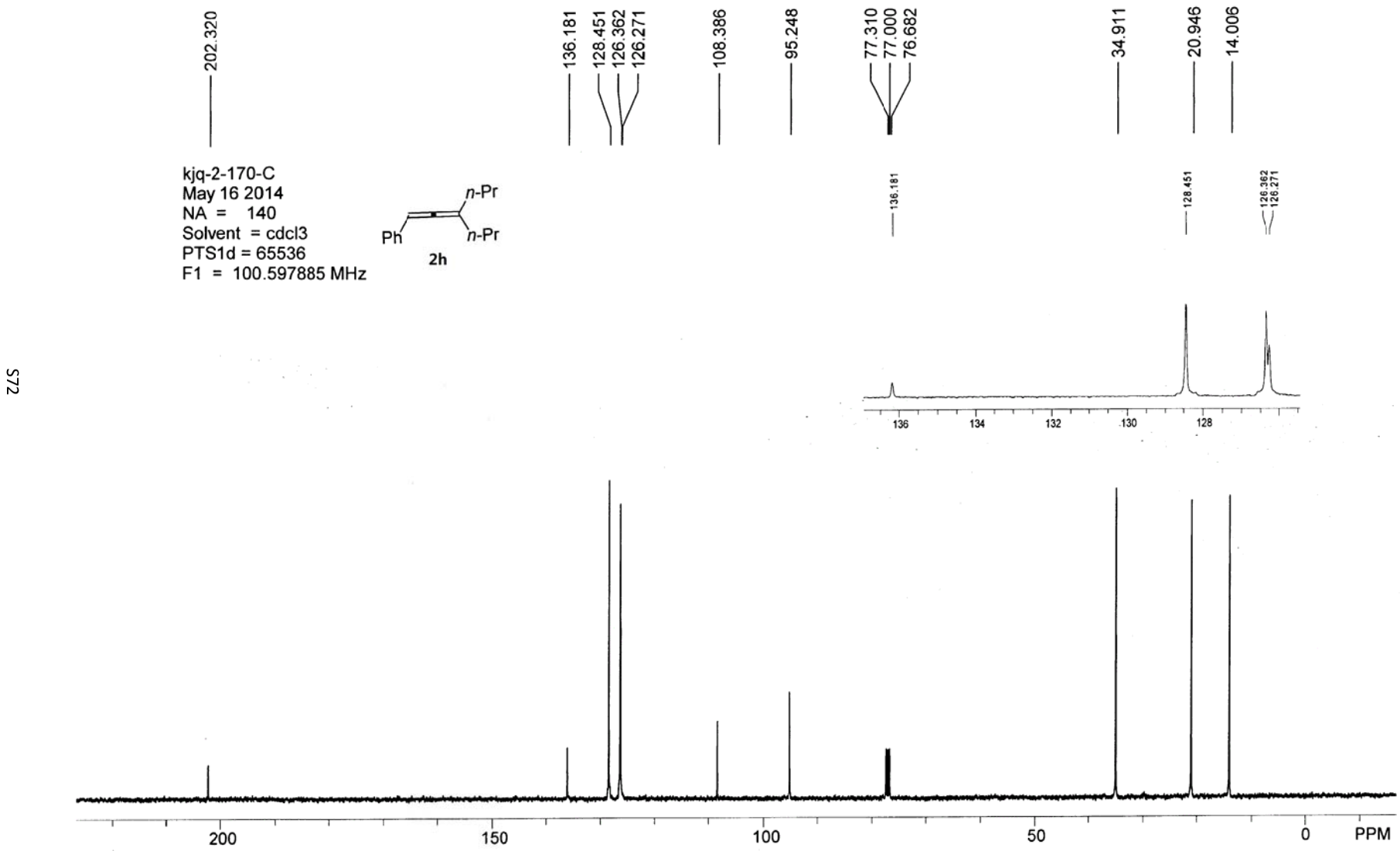


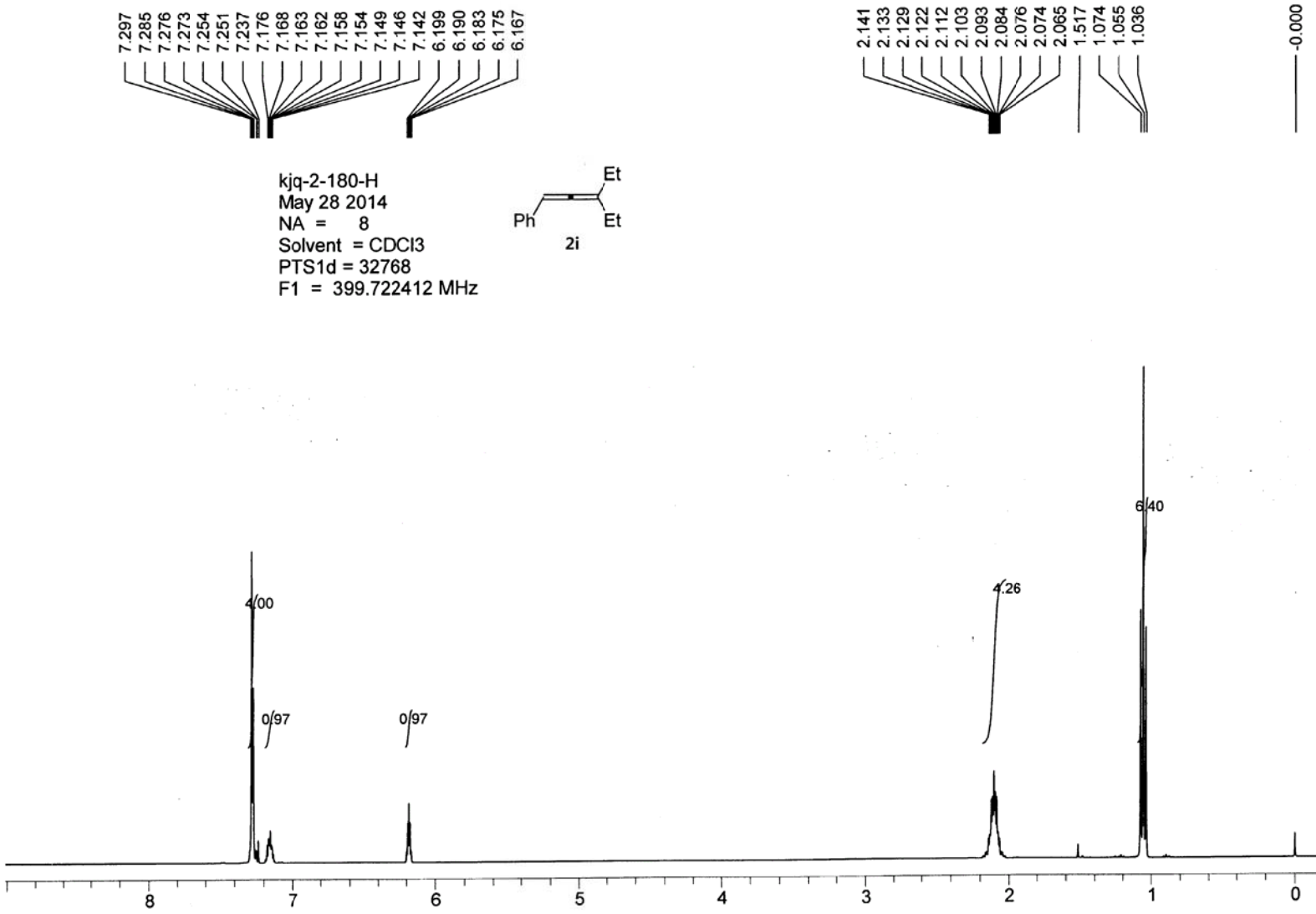
2g

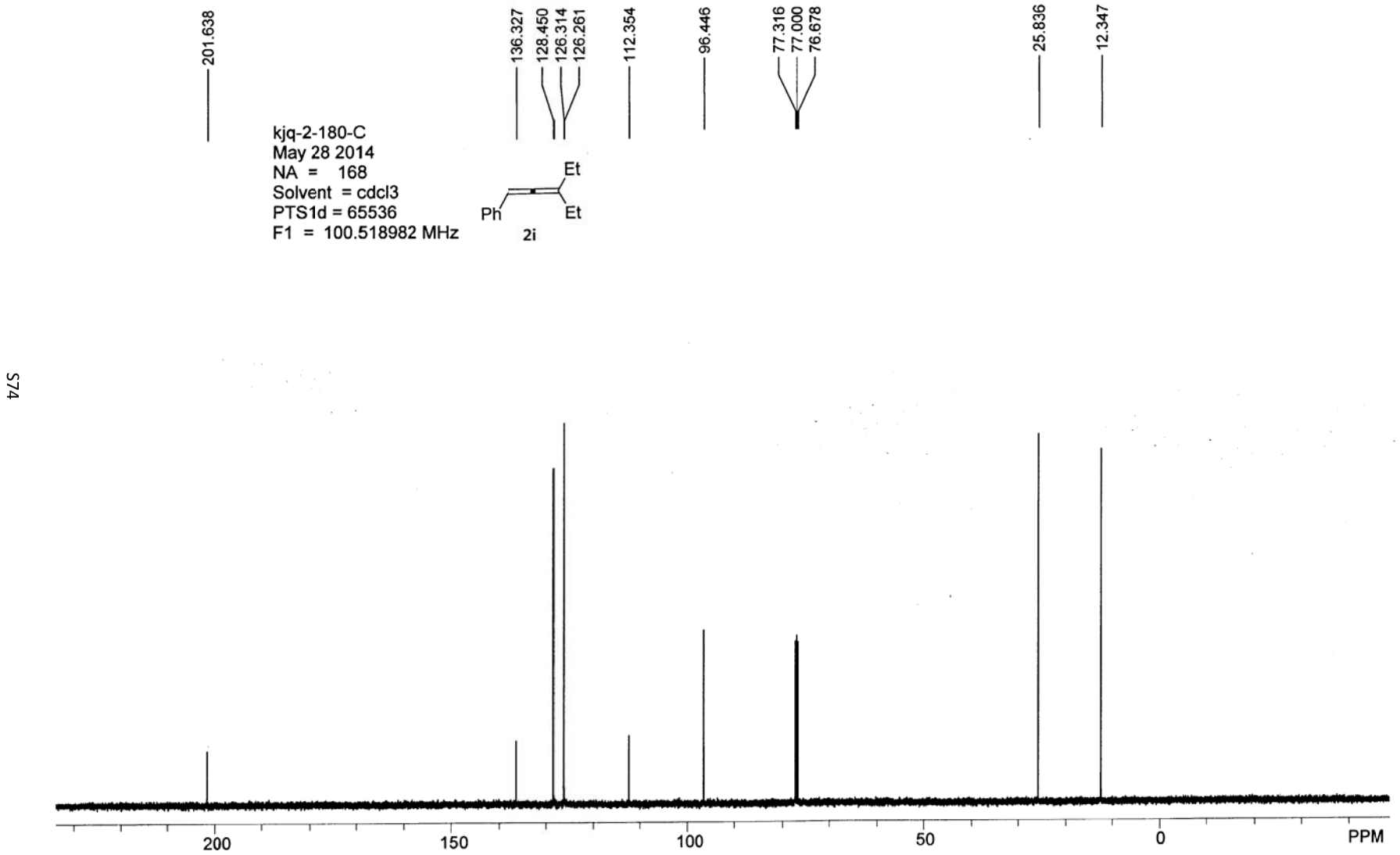


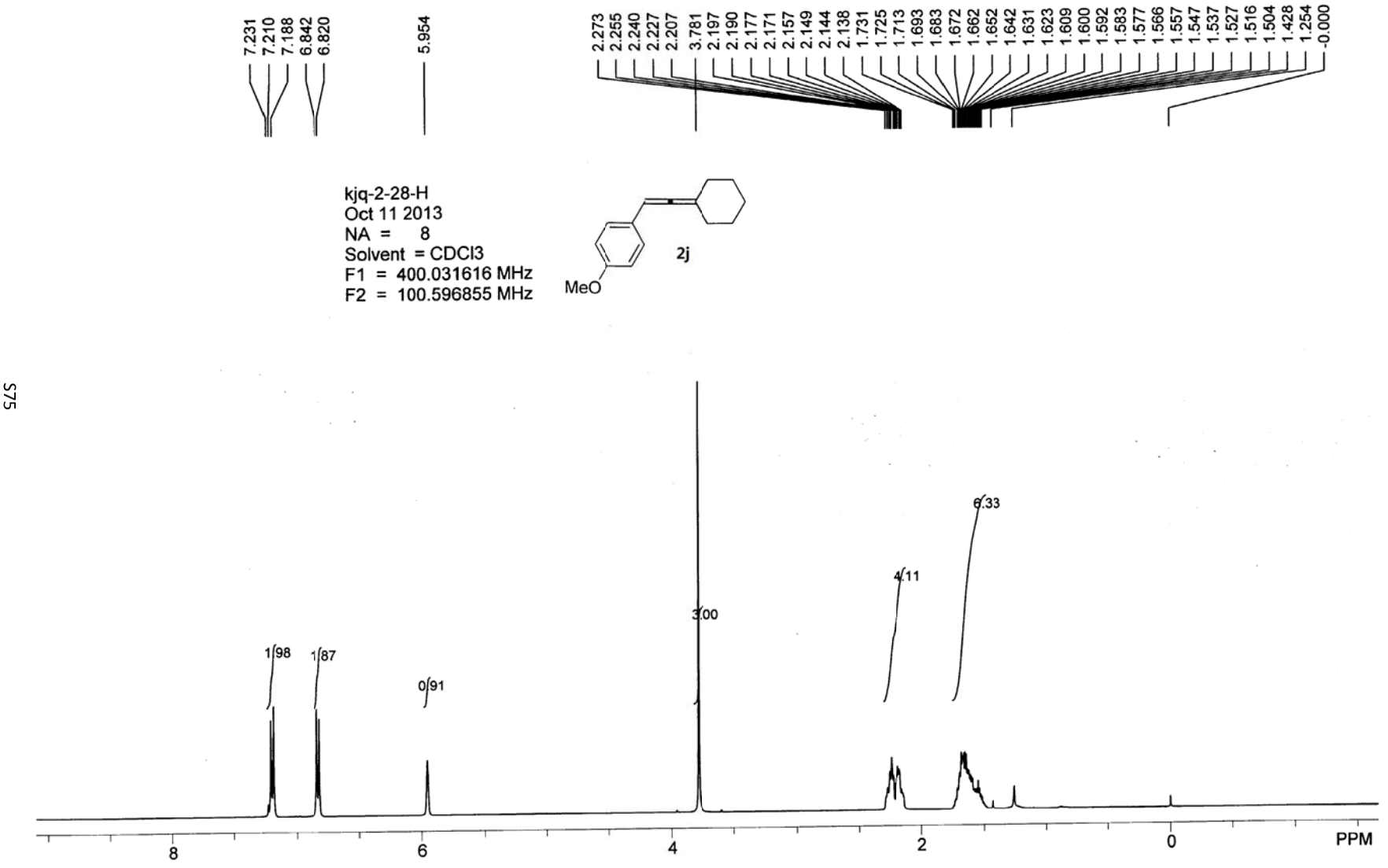

201.993
136.260
128.453
126.305
126.286
110.366
95.841
77.314
77.000
76.681
34.926
25.819
20.956
13.988
12.324
kjq-2-43-C
Oct 22 2013
NA = 28
Solvent = cdcl3
PTS1d = 65536
F1 = 100.520737 MHz
n-Pr
Ph
Et
2g
200
150
100
50
0
PPM

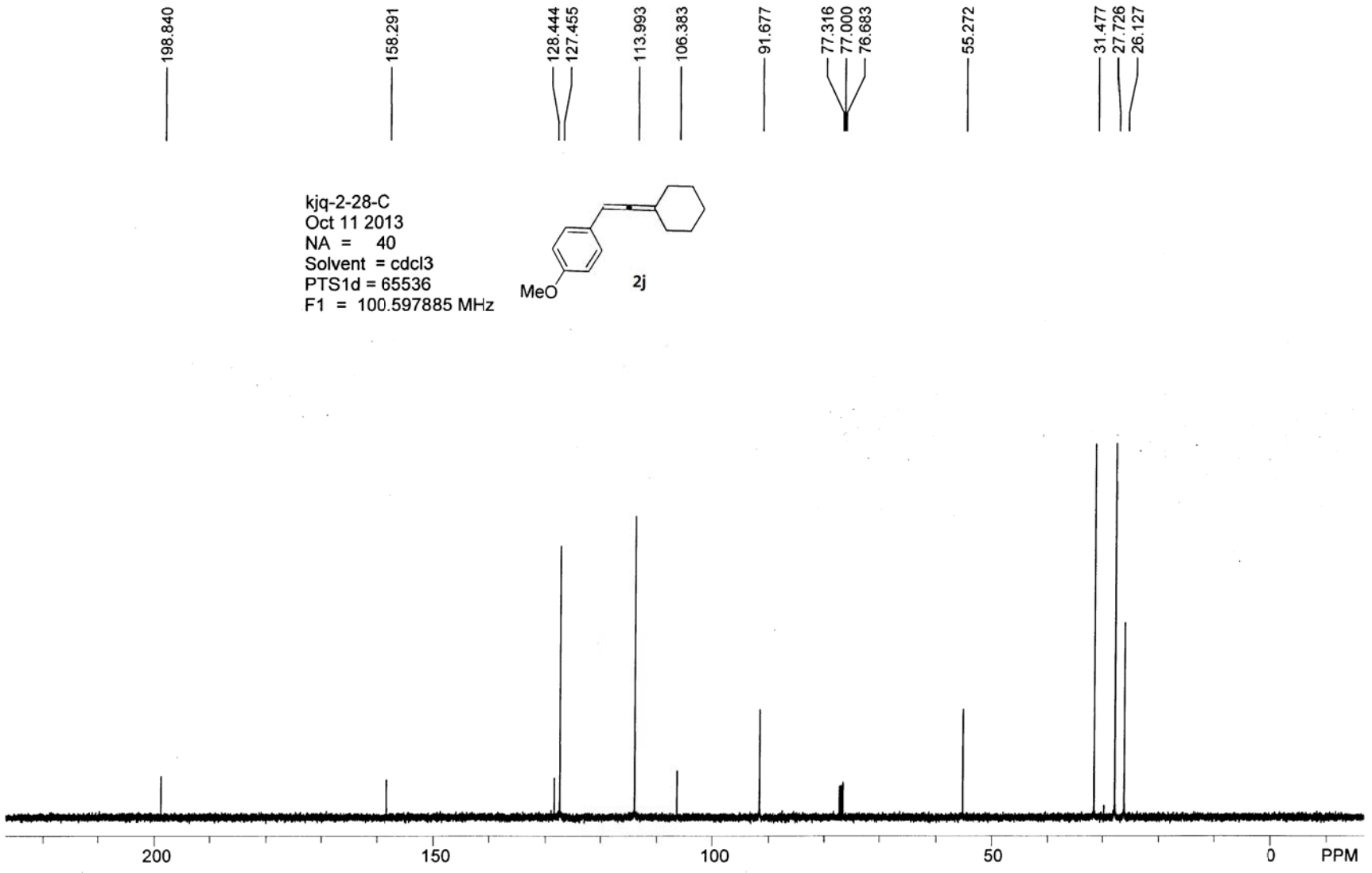

7.279
7.268
7.233
7.170
7.159
7.148
7.137
6.121
6.113
6.106
6.098
2.075
2.067
2.057
2.049
2.038
2.030
2.022
1.545
1.542
1.528
1.523
1.510
1.504
1.491
1.486
1.478
1.472
1.467
1.454
1.449
0.947
0.929
0.910
0.000
kjq-2-170-H
May 16 2014
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 399.722809 MHz
n-Pr
Ph
n-Pr
2h
4.00
0.99
0.97
4.30
4.32
6.39
8
7
6
5
4
3
2
1
0
PPM

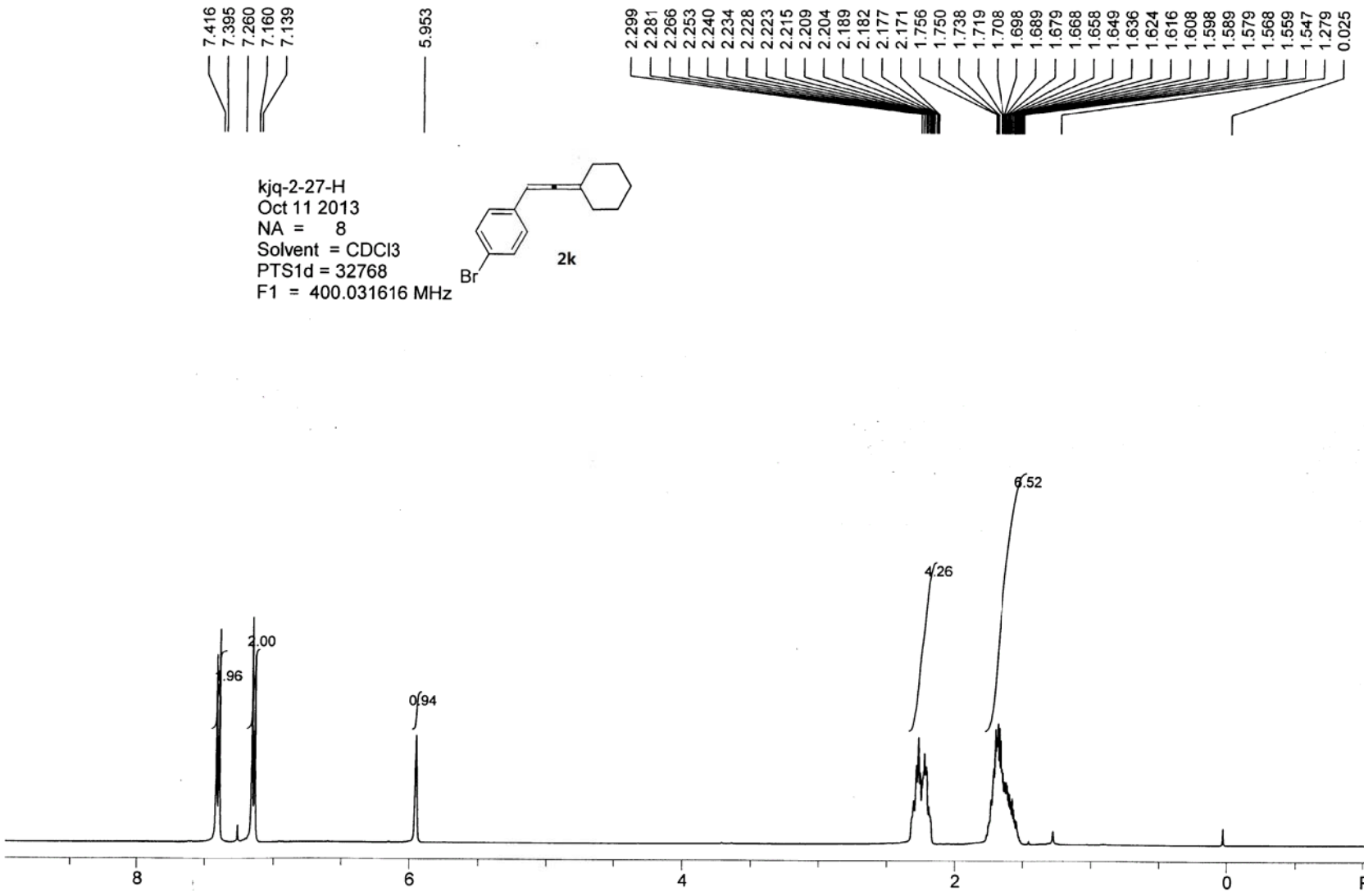

202.320
136.181
128.451
126.362
126.271
108.386
95.248
77.310
77.000
76.682
34.911
20.946
14.006
kjq-2-170-C
May 16 2014
NA = 140
Solvent = cdcl3
PTS1d = 65536
F1 = 100.597885 MHz
n-Pr
Ph
n-Pr
2h
136.181
128.451
126.362
126.271
136
134
132
130
128
200
150
100
50
0
PPM

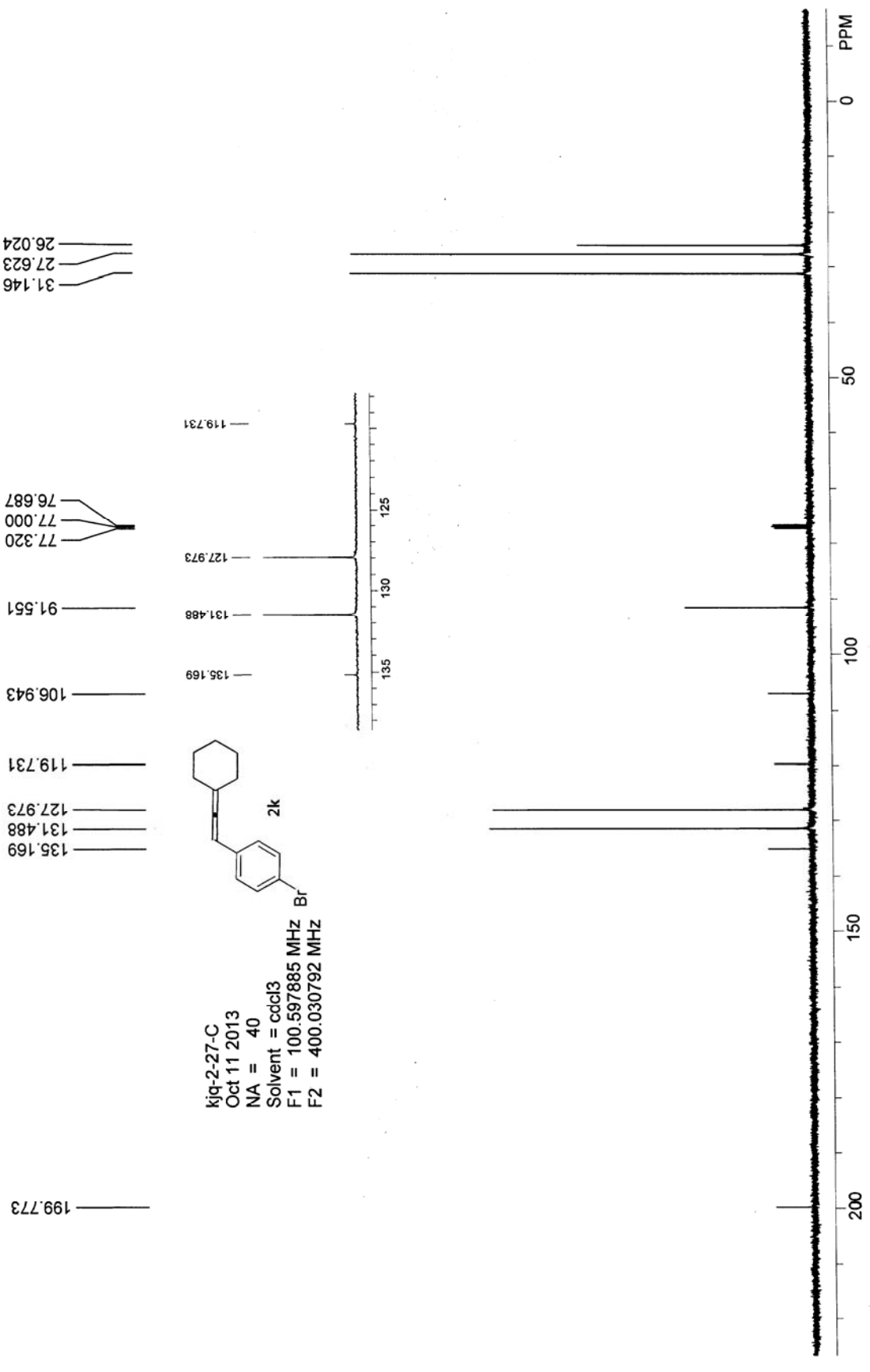

7.297
7.285
7.276
7.273
7.254
7.251
7.237
7.176
7.168
7.163
7.162
7.158
7.154
7.149
7.146
7.142
6.199
6.190
6.183
6.175
6.167
2.141
2.133
2.129
2.122
2.112
2.103
2.093
2.084
2.076
2.074
2.065
1.517
1.074
1.055
1.036
-0.000
kjq-2-180-H
May 28 2014
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 399.722412 MHz
Et
Ph
Et
2i
4.00
0.97
0.97
4.26
6.40
8
7
6
5
4
3
2
1
0
PPM

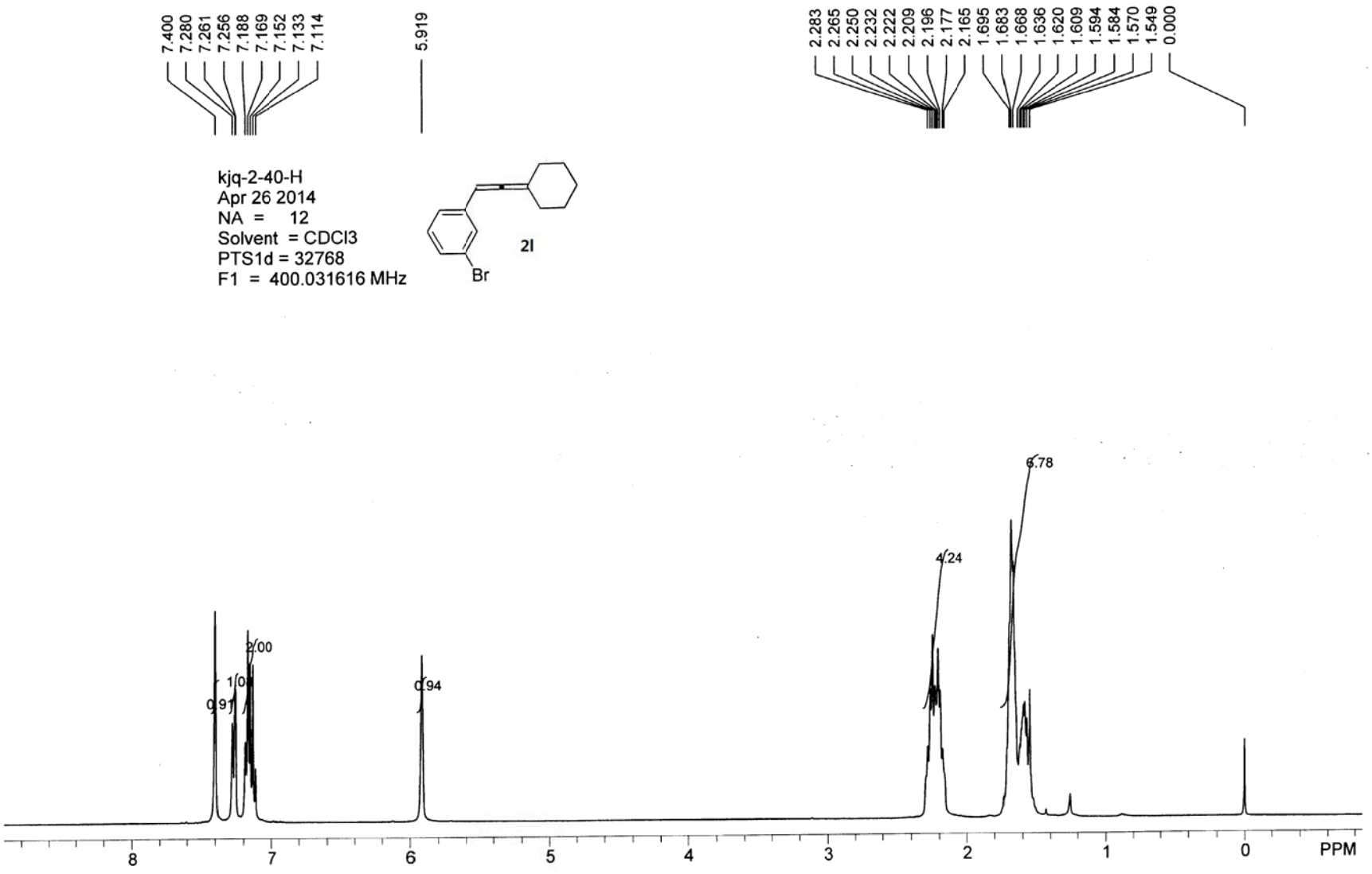

201.638
136.327
128.450
126.314
126.261
112.354
96.446
77.316
77.000
76.678
25.836
12.347
kjq-2-180-C
May 28 2014
NA = 168
Solvent = cdcl3
PTS1d = 65536
F1 = 100.518982 MHz
Et
Ph
Et
2i
200
150
100
50
0
PPM

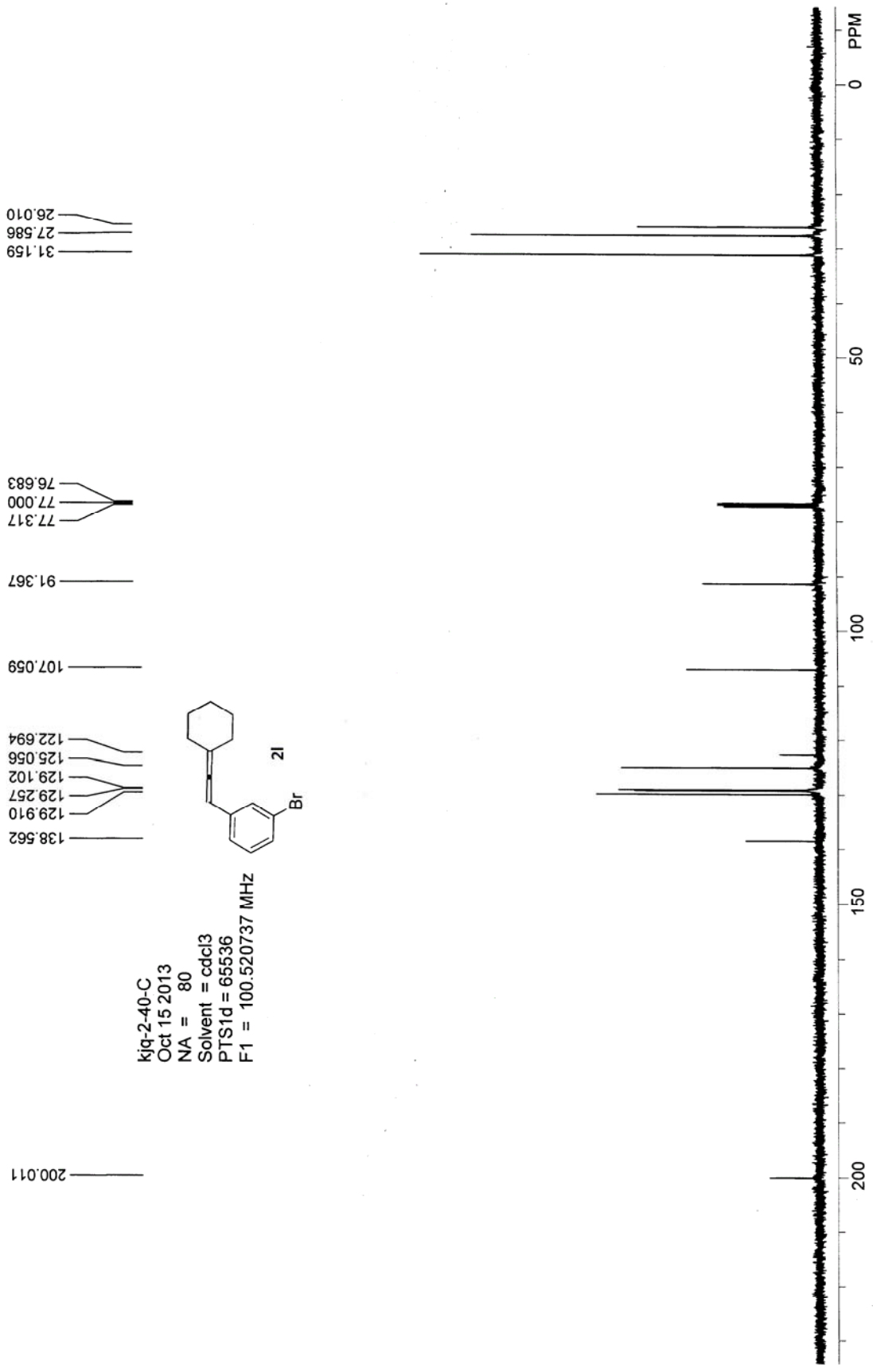

7.231
7.210
7.188
6.842
6.820
5.954
2.273
2.255
2.240
2.227
2.207
3.781
2.197
2.190
2.177
2.171
2.157
2.149
2.144
2.138
1.731
1.725
1.713
1.693
1.683
1.672
1.662
1.652
1.642
1.631
1.623
1.609
1.600
1.592
1.583
1.577
1.566
1.557
1.547
1.537
1.527
1.516
1.504
1.428
1.254
-0.000
kjq-2-28-H
Oct 11 2013
NA = 8
Solvent = CDCl3
F1 = 400.031616 MHz
F2 = 100.596855 MHz
MeO
2j
1.98
1.87
0.91
3.00
4.11
6.33
8
6
4
2
0
PPM

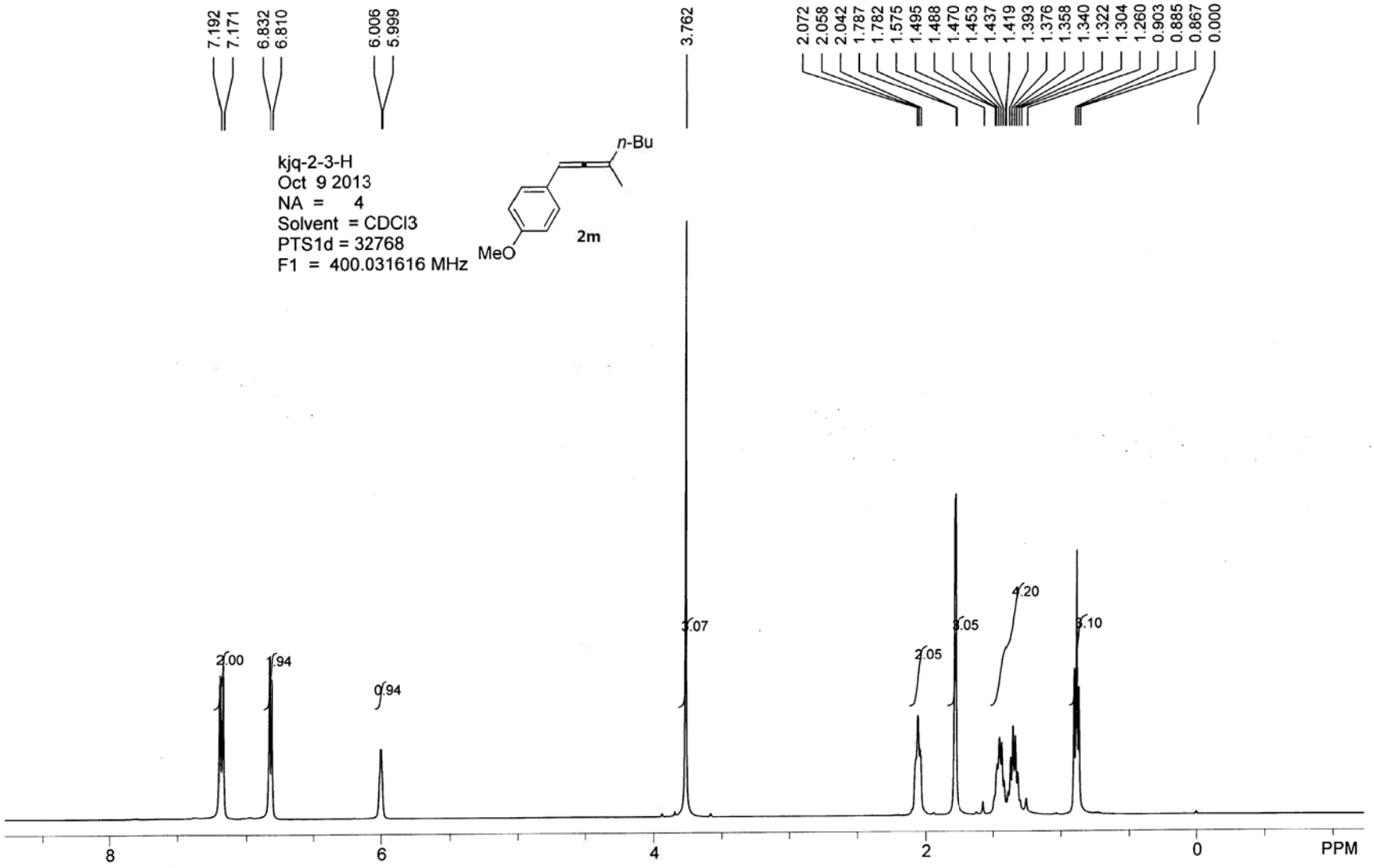

198.840
158.291
128.444
127.455
113.993
106.383
91.677
77.316
77.000
76.683
55.272
31.477
27.726
26.127
kjq-2-28-C
Oct 11 2013
NA = 40
Solvent = cdcl3
PTS1d = 65536
F1 = 100.597885 MHz
MeO
2j
200
150
100
50
0
PPM

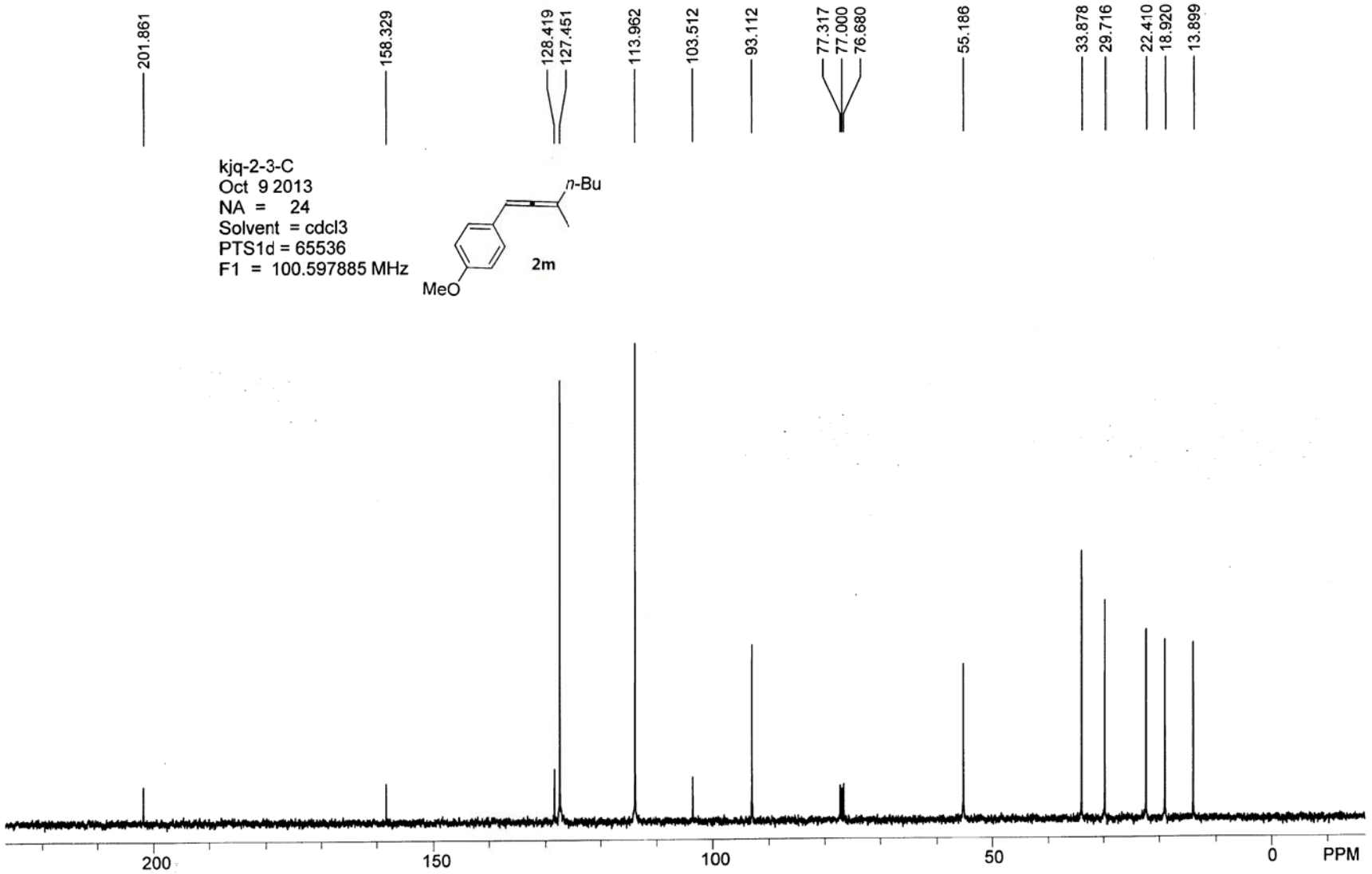

7.416
7.395
7.260
7.160
7.139
5.953
2.299
2.281
2.266
2.253
2.240
2.234
2.228
2.223
2.215
2.209
2.204
2.189
2.182
2.177
2.171
1.756
1.750
1.738
1.719
1.708
1.698
1.689
1.679
1.668
1.658
1.649
1.636
1.624
1.616
1.608
1.598
1.589
1.579
1.568
1.559
1.547
1.279
0.025
kjq-2-27-H
Oct 11 2013
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 400.031616 MHz
Br
2k
1.96
2.00
0.94
4.26
6.52
8
6
4
2
0
PPM

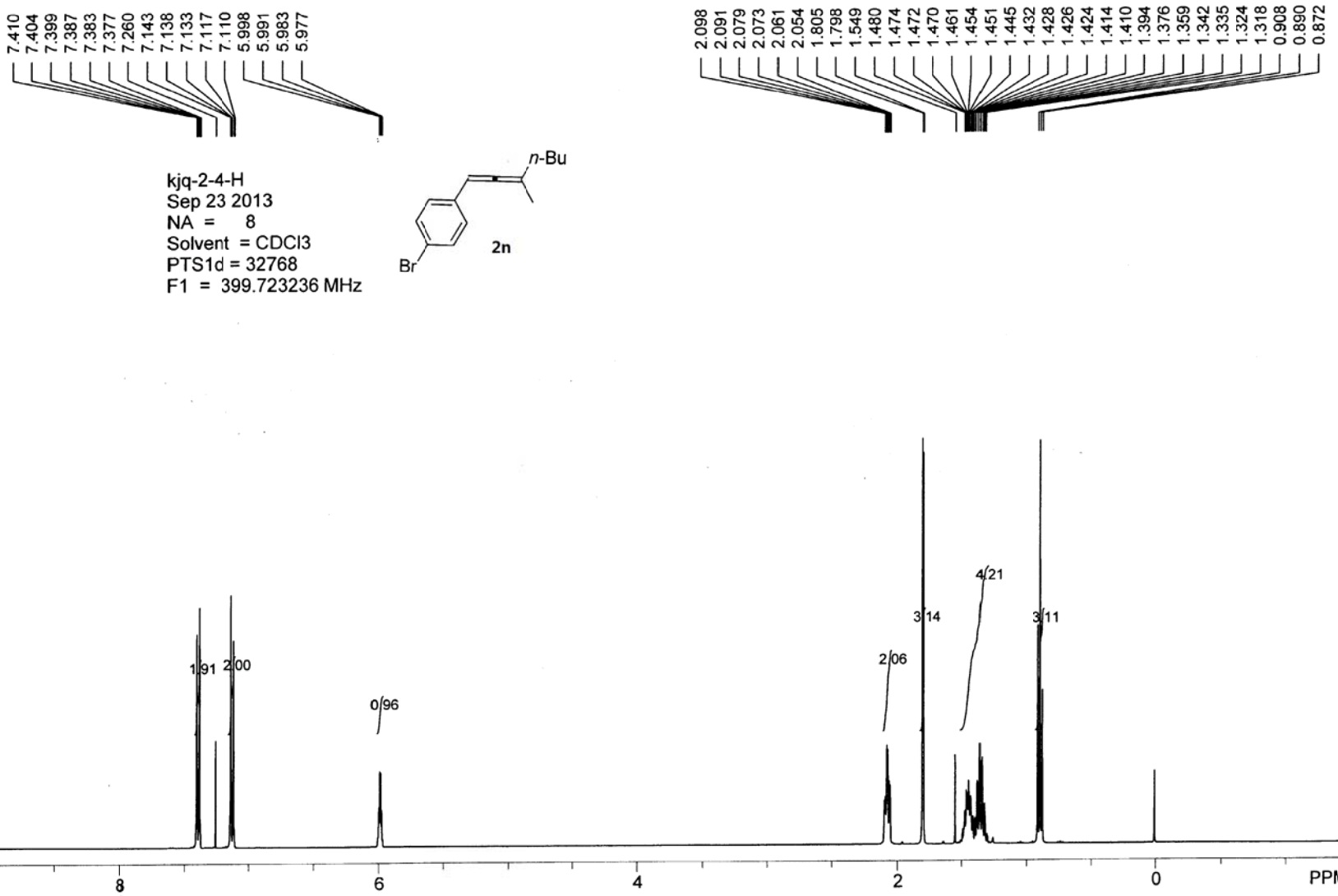

199.773
135.169
131.488
127.973
119.731
106.943
91.551
77.320
77.000
76.687
31.146
27.623
26.024
kjq-2-27-C
Oct 11 2013
NA = 40
Solvent = cdcl3
F1 = 100.597885 MHz
F2 = 400.030792 MHz
2k
Br
PPM

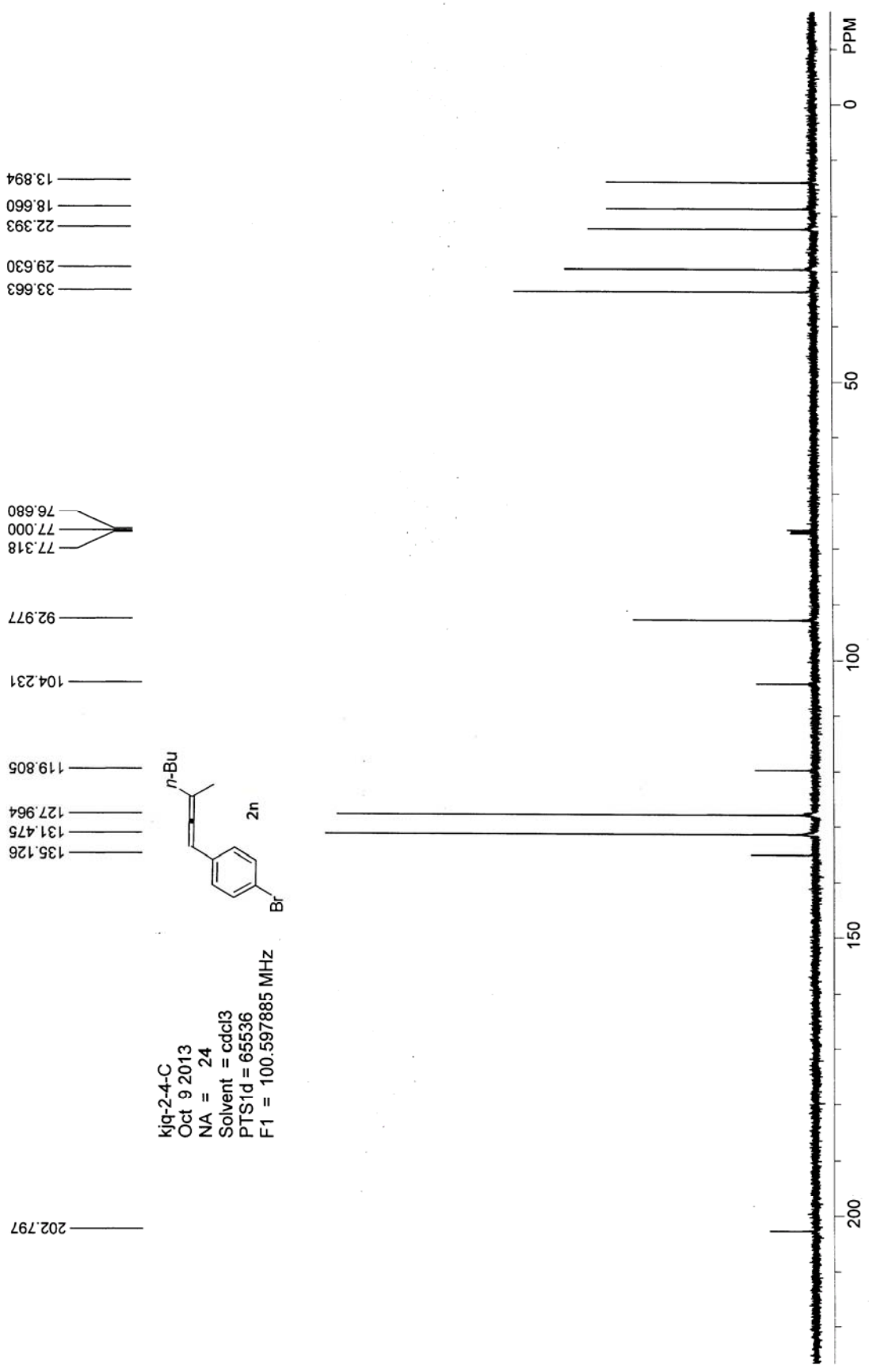

7.400
7.280
7.261
7.256
7.188
7.169
7.152
7.133
7.114
5.919
2.283
2.265
2.250
2.232
2.222
2.209
2.196
2.177
2.165
1.695
1.683
1.668
1.636
1.620
1.609
1.594
1.584
1.570
1.549
0.000
kjq-2-40-H
Apr 26 2014
NA = 12
Solvent = CDCl3
PTS1d = 32768
F1 = 400.031616 MHz
2l
Br
6.78
4.24
2.00
1.00
0.91
0.94
8
7
6
5
4
3
2
1
0
PPM

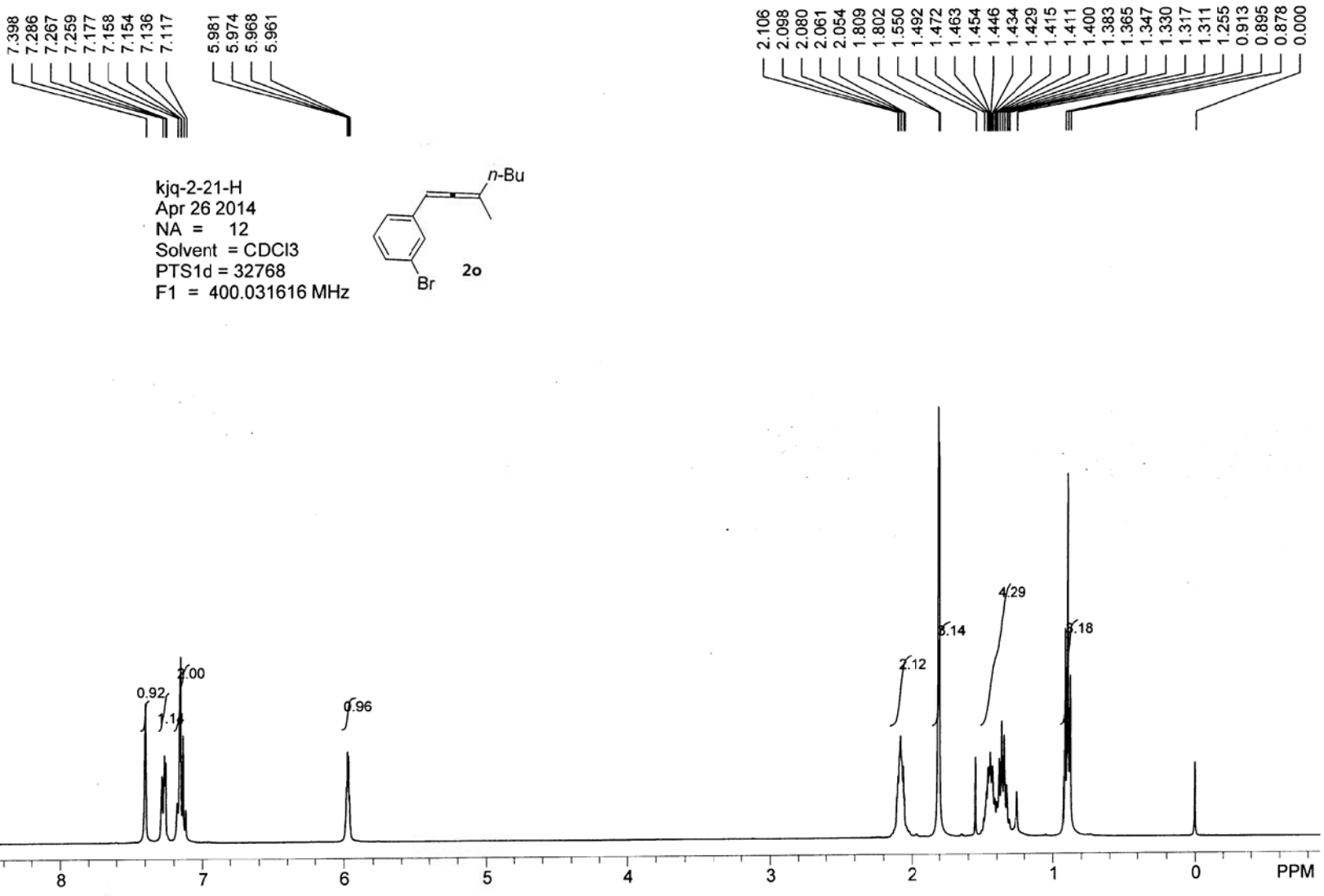

kjq-2-40-C
Oct 15 2013
NA = 80
Solvent = cdcl3
PTS1d = 65536
F1 = 100.520737 MHz
2l
Br
200.011
138.562
129.910
129.257
129.102
125.056
122.694
107.059
91.367
77.317
77.000
76.683
31.159
27.586
26.010
PPM
0
50
100
150
200


7.192
7.171
6.832
6.810
6.006
5.999
3.762
2.072
2.058
2.042
1.787
1.782
1.575
1.495
1.488
1.470
1.453
1.437
1.419
1.393
1.376
1.358
1.340
1.322
1.304
1.260
0.903
0.885
0.867
0.000
kjq-2-3-H
Oct 9 2013
NA = 4
Solvent = CDCl3
PTS1d = 32768
F1 = 400.031616 MHz
n-Bu
MeO
2m
2.00
1.94
0.94
3.07
2.05
3.05
4.20
3.10
8
6
4
2
0
PPM

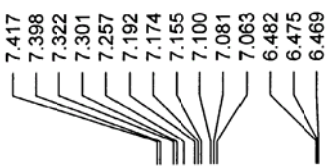

201.861
158.329
128.419
127.451
113.962
103.512
93.112
77.317
77.000
76.680
55.186
33.878
29.716
22.410
18.920
13.899
kjq-2-3-C
Oct 9 2013
NA = 24
Solvent = cdcl3
PTS1d = 65536
F1 = 100.597885 MHz
n-Bu
MeO
2m
200
150
100
50
0
PPM

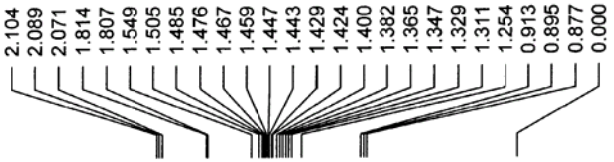

7.410
7.404
7.399
7.387
7.383
7.377
7.260
7.143
7.138
7.133
7.117
7.110
5.998
5.991
5.983
5.977
2.098
2.091
2.079
2.073
2.061
2.054
1.805
1.798
1.549
1.480
1.474
1.472
1.470
1.461
1.454
1.451
1.445
1.432
1.428
1.426
1.424
1.414
1.410
1.394
1.376
1.359
1.342
1.335
1.324
1.318
0.908
0.890
0.872
kjq-2-4-H
Sep 23 2013
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 399.723236 MHz
n-Bu
Br
2n
1.91
2.00
0.96
2.06
3.14
4.21
3.11
8
6
4
2
0
PPM


kjq-2-4-C
Oct 9 2013
NA = 24
Solvent = cdcl3
PTS1d = 65536
F1 = 100.597885 MHz
n-Bu
2n
Br
202.797
135.126
131.475
127.964
119.805
104.231
92.977
77.318
77.000
76.680
33.663
29.630
22.393
18.660
13.894
PPM
0
50
100
150
200

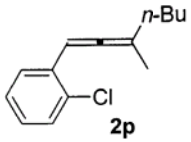

7.398
7.286
7.267
7.259
7.177
7.158
7.154
7.136
7.117
5.981
5.974
5.968
5.961
2.106
2.098
2.080
2.061
2.054
1.809
1.802
1.550
1.492
1.472
1.463
1.454
1.446
1.434
1.429
1.415
1.411
1.400
1.383
1.365
1.347
1.330
1.317
1.311
1.255
0.913
0.895
0.878
0.000
kjq-2-21-H
Apr 26 2014
NA = 12
Solvent = CDCl3
PTS1d = 32768
F1 = 400.031616 MHz
n-Bu
Br
2o
0.92
1.1
2.00
0.96
2.12
3.14
4.29
3.18
8
7
6
5
4
3
2
1
0
PPM

13.900
18.727
22.398
29.583
33.662
76.682
77.000
77.316
92.771
104.429
122.705
125.071
129.165
129.226
129.911
138.530
203.018
n-Bu
Br
2o
kjq-2-21-C
Oct 11 2013
NA = 80
Solvent = cdcl3
F1 = 100.520737 MHz
F2 = 399.722015 MHz
PPM
0
50
100
150
200

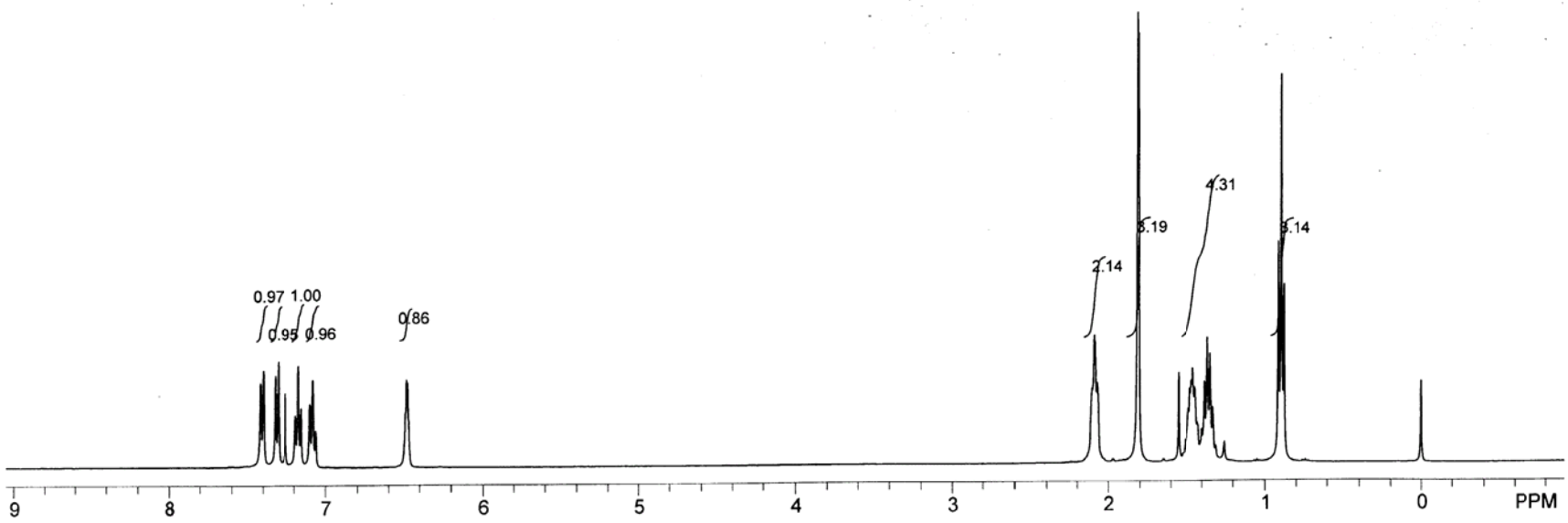


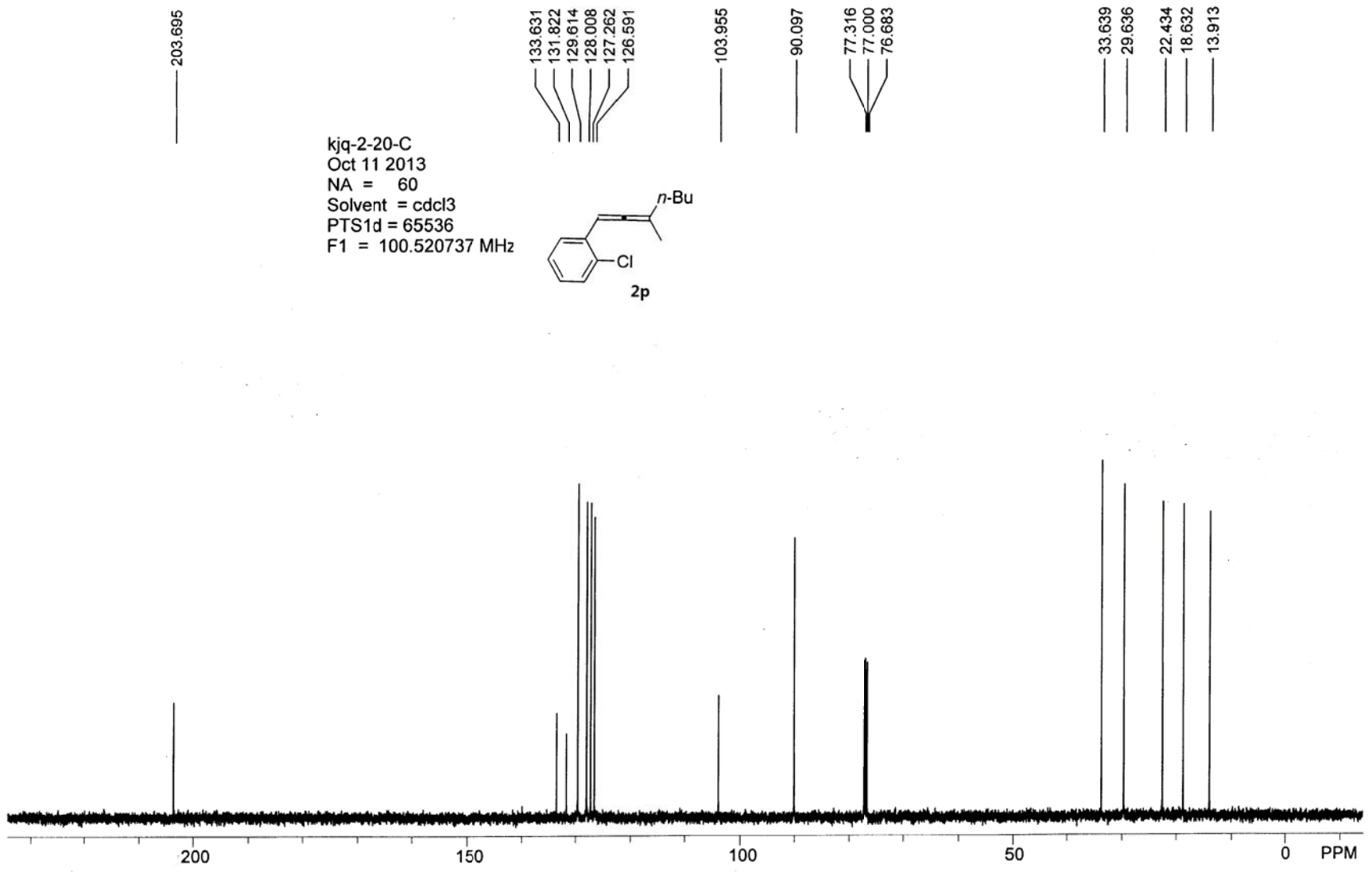


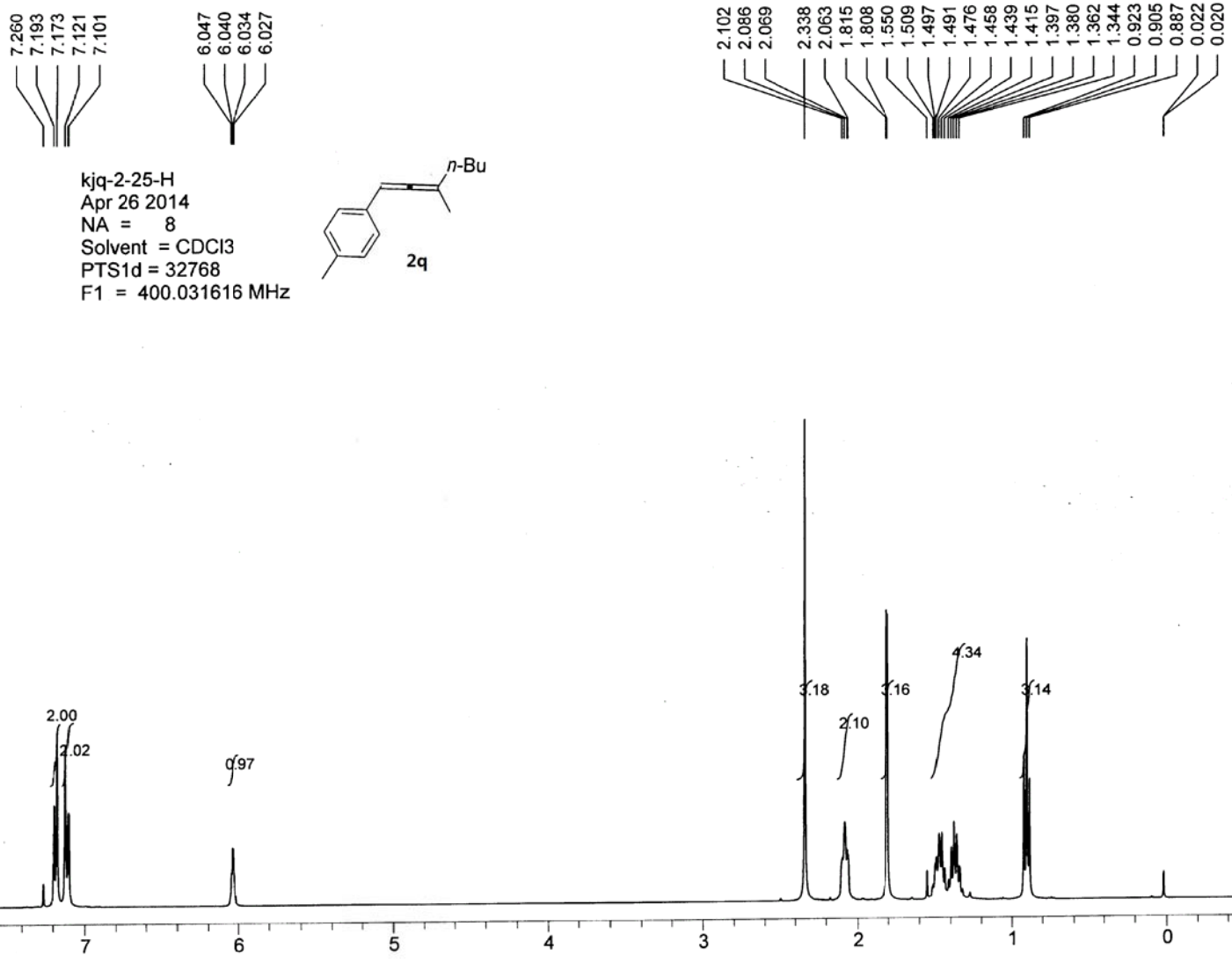
kjq-2-20-H
Apr 26 2014
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 400.031616 MHz

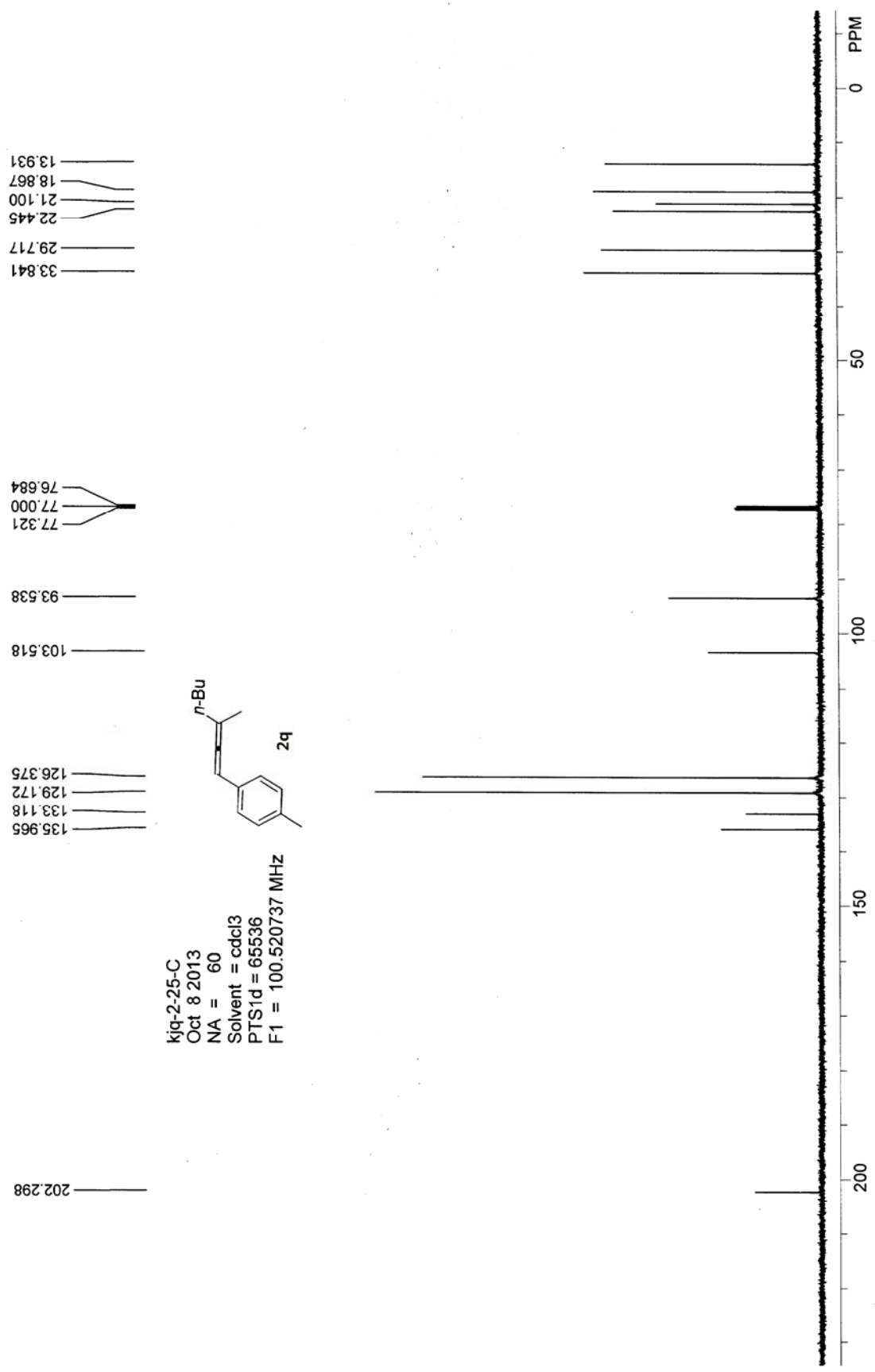


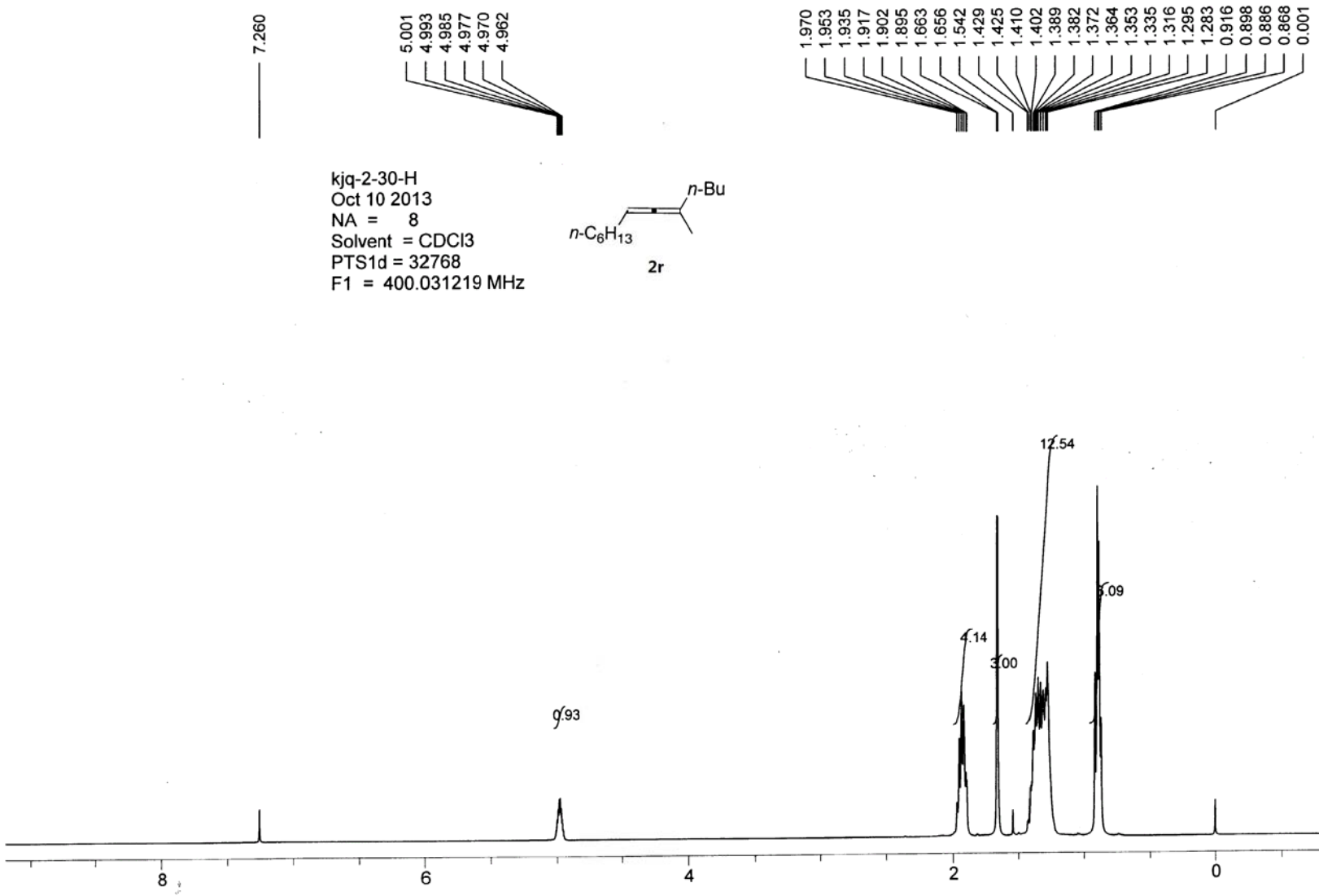
2p

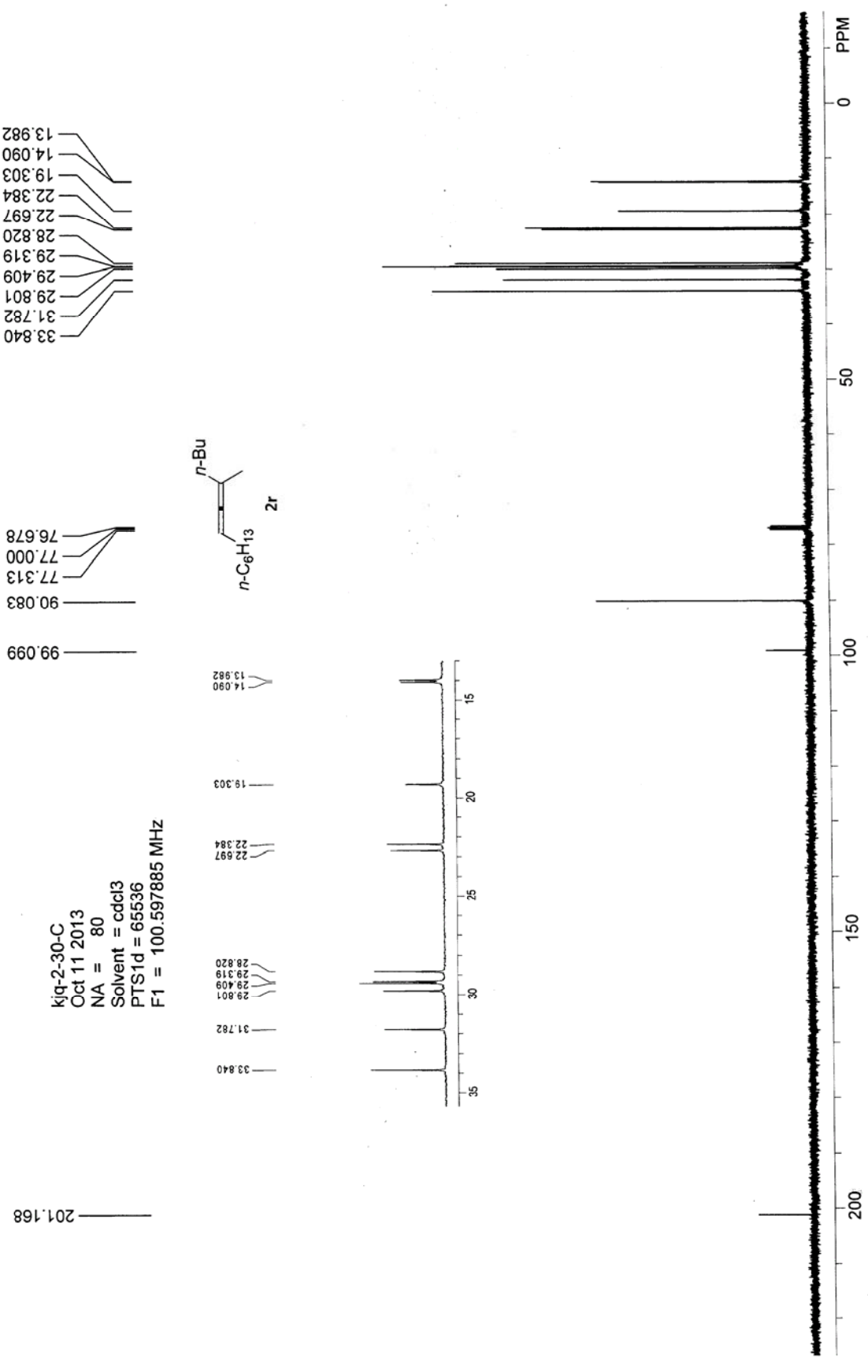


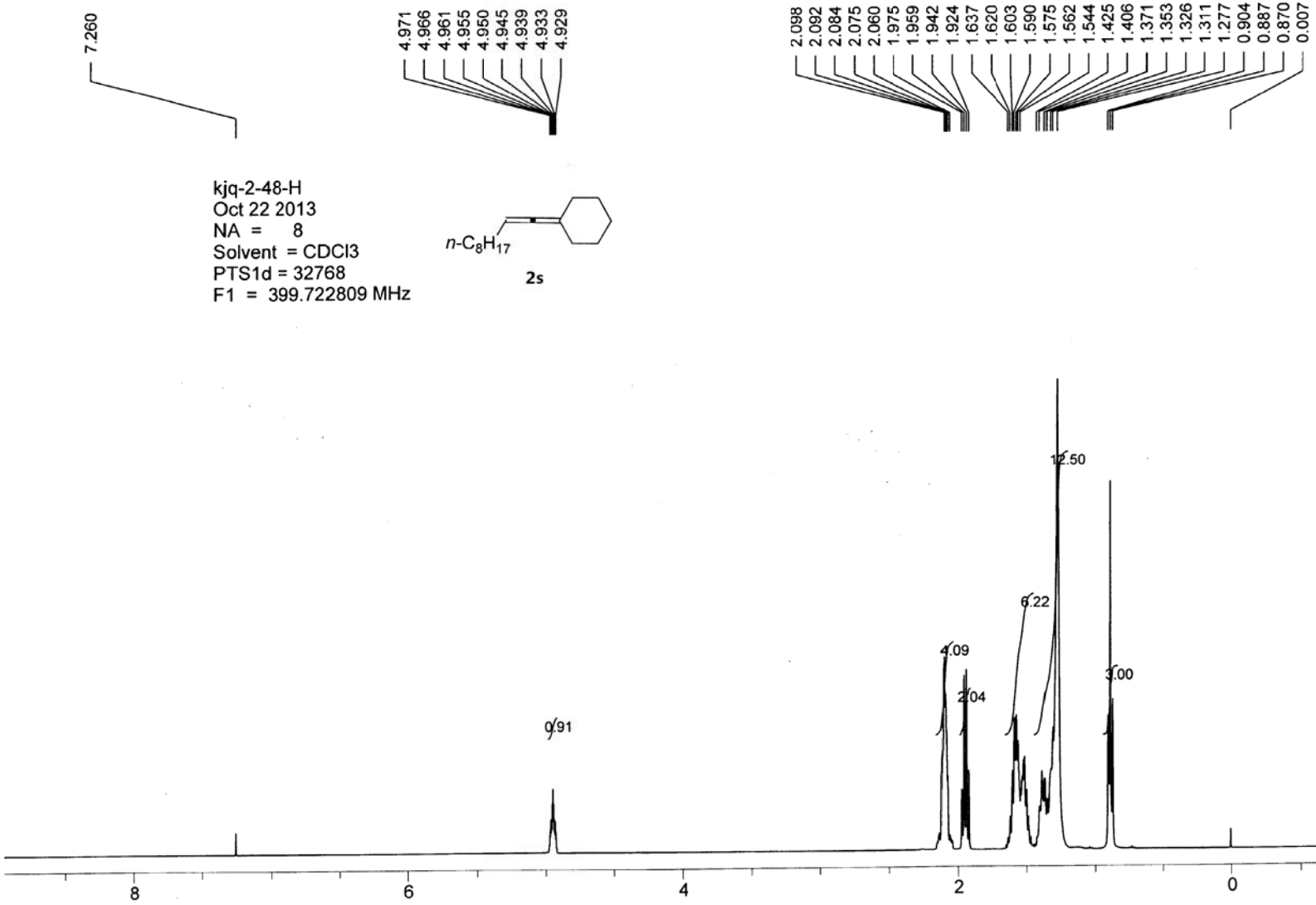

203.695
133.631
131.822
129.614
128.008
127.262
126.591
103.955
90.097
77.316
77.000
76.683
33.639
29.636
22.434
18.632
13.913
kjq-2-20-C
Oct 11 2013
NA = 60
Solvent = cdcl3
PTS1d = 65536
F1 = 100.520737 MHz
n-Bu
Cl
2p
200
150
100
50
0
PPM

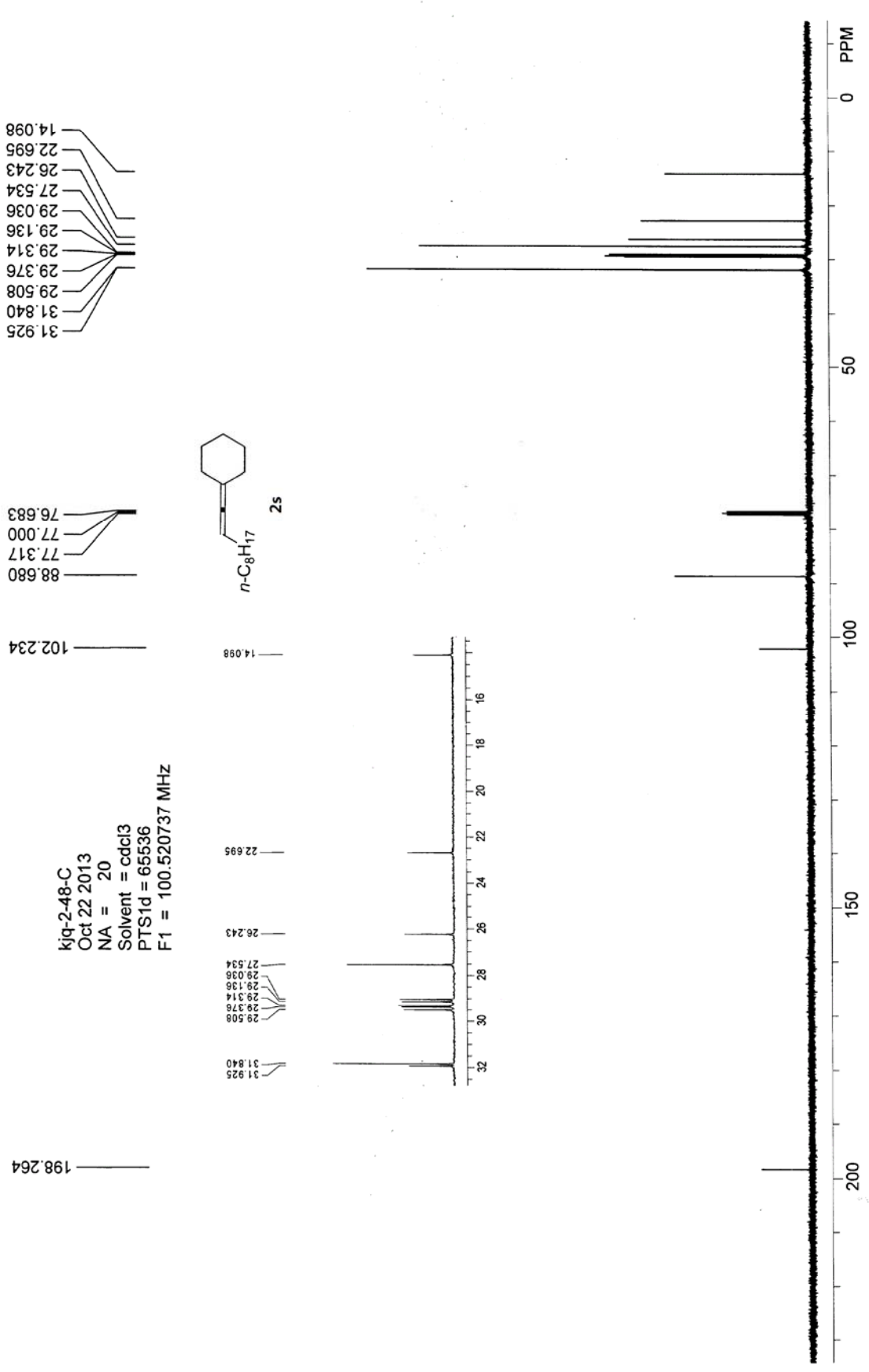

7.260
7.193
7.173
7.121
7.101
6.047
6.040
6.034
6.027
2.102
2.086
2.069
2.338
2.063
1.815
1.808
1.550
1.509
1.497
1.491
1.476
1.458
1.439
1.415
1.397
1.380
1.362
1.344
0.923
0.905
0.887
0.022
0.020
kjq-2-25-H
Apr 26 2014
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 400.031616 MHz
n-Bu
2q
2.00
2.02
0.97
3.18
2.10
3.16
4.34
3.14
8
7
6
5
4
3
2
1
0
PPM

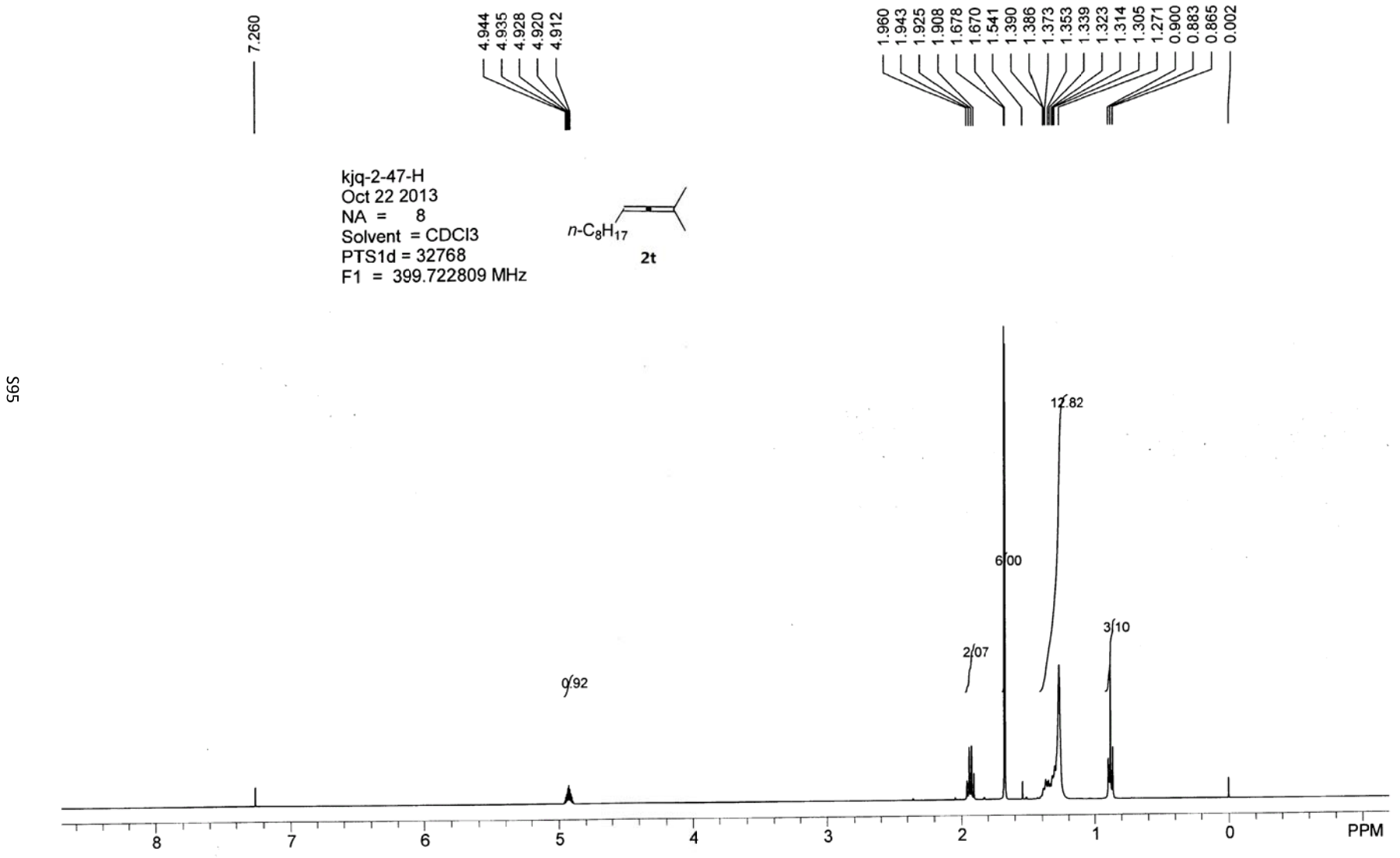

202.298
135.965
133.118
129.172
126.375
103.518
93.538
77.321
77.000
76.684
33.841
29.717
22.445
21.100
18.867
13.931
n-Bu
2q
kjq-2-25-C
Oct 8 2013
NA = 60
Solvent = cdcl3
PTS1d = 65536
F1 = 100.520737 MHz
PPM
0
50
100
150
200

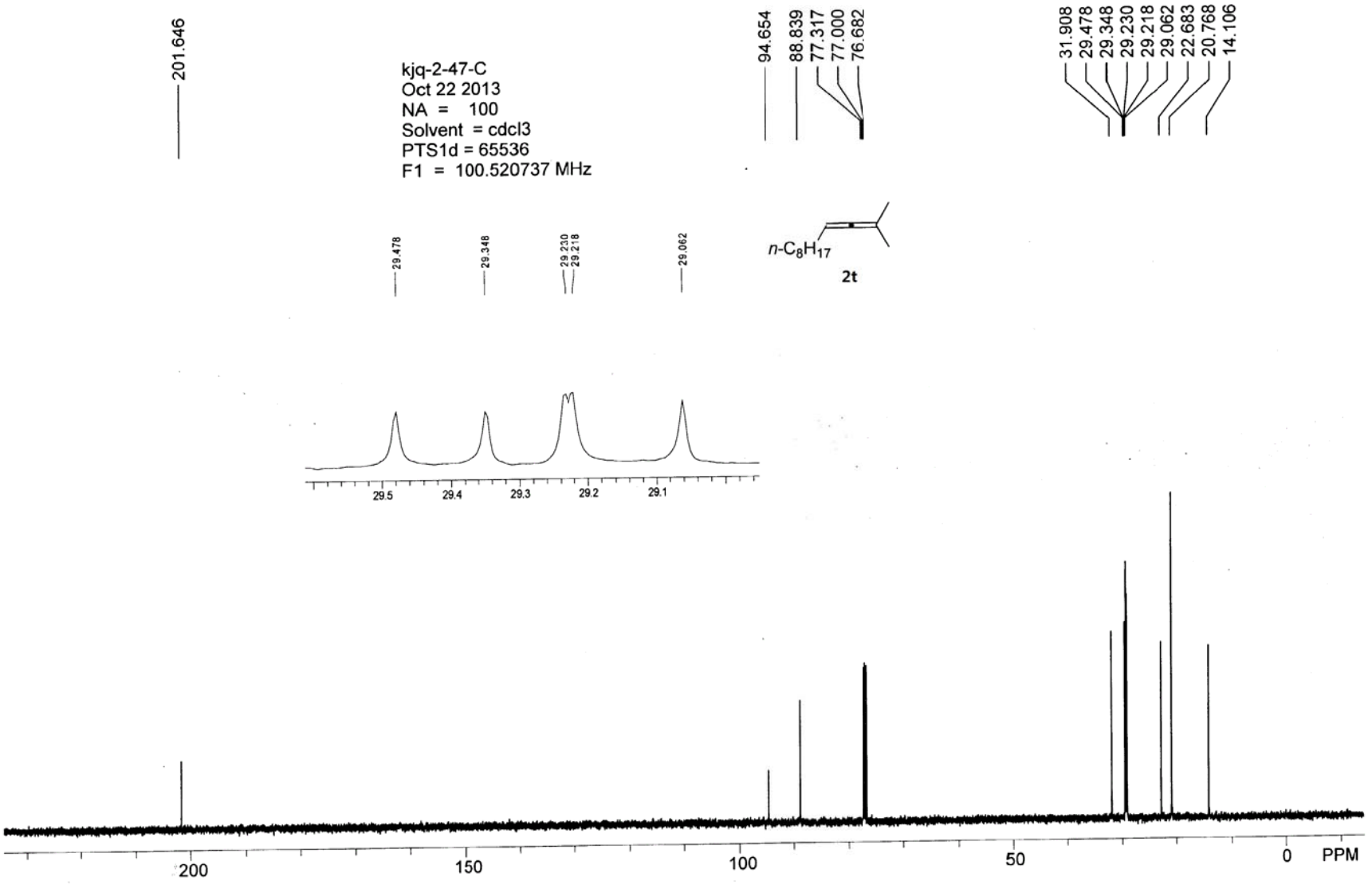

7.260
5.001
4.993
4.985
4.977
4.970
4.962
1.970
1.953
1.935
1.917
1.902
1.895
1.663
1.656
1.542
1.429
1.425
1.410
1.402
1.389
1.382
1.372
1.364
1.353
1.335
1.316
1.295
1.283
0.916
0.898
0.886
0.868
0.001
kjq-2-30-H
Oct 10 2013
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 400.031219 MHz
n-Bu
n-C6H13
2r
12.54
6.09
4.14
3.00
0.93
8
6
4
2
0
PPM

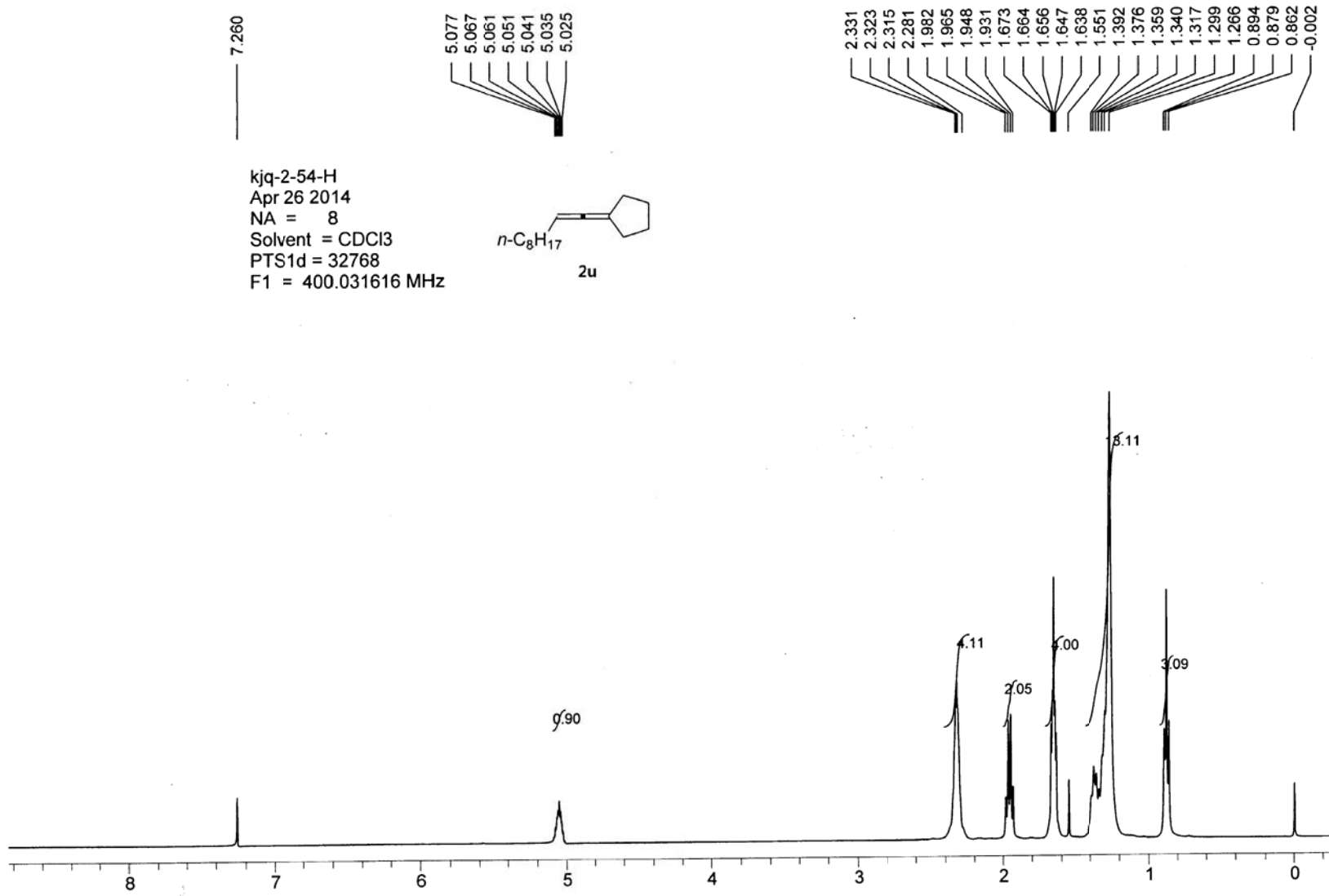

kjq-2-30-C
Oct 11 2013
NA = 80
Solvent = cdcl3
PTS1d = 65536
F1 = 100.597885 MHz
n-Bu
n-C6H13
2r
PPM

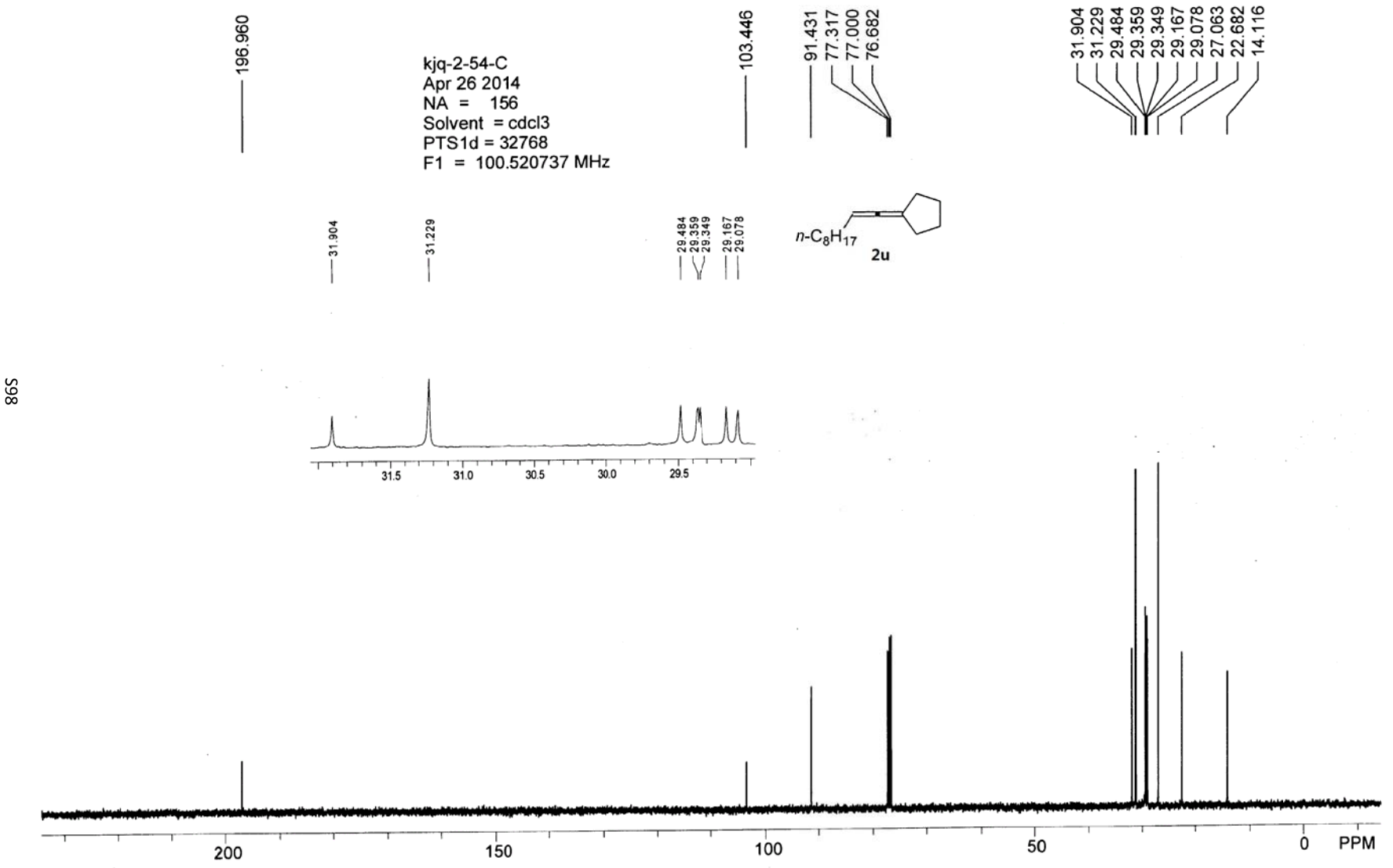

7.260
4.971
4.966
4.961
4.955
4.950
4.945
4.939
4.933
4.929
2.098
2.092
2.084
2.075
2.060
1.975
1.959
1.942
1.924
1.637
1.620
1.603
1.590
1.575
1.562
1.544
1.425
1.406
1.371
1.353
1.326
1.311
1.277
0.904
0.887
0.870
0.007
kjq-2-48-H
Oct 22 2013
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 399.722809 MHz
n-C8H17
2s
0.91
4.09
2.04
6.22
12.50
3.00
8
6
4
2
0
PPM

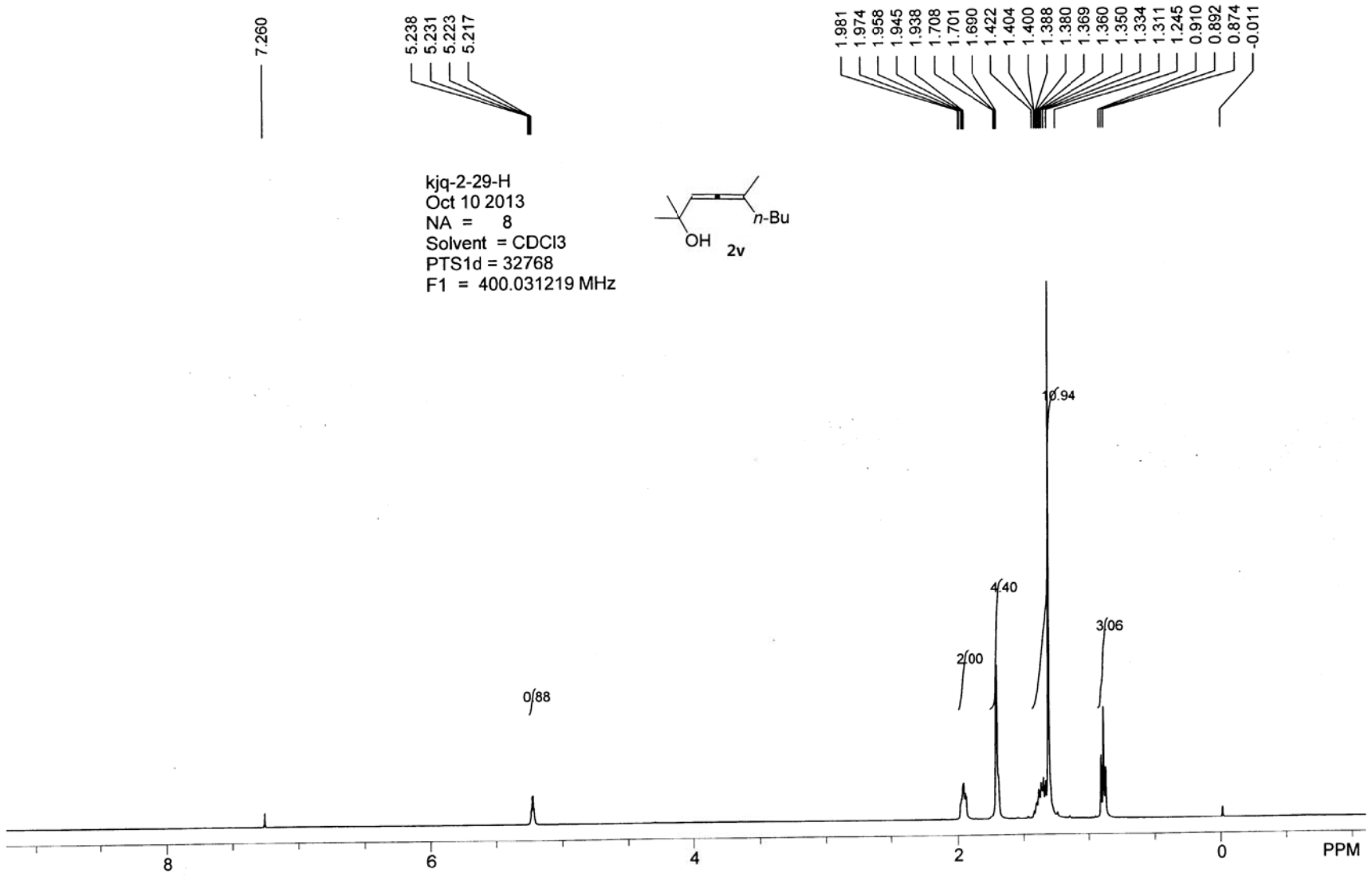

kjq-2-48-C
Oct 22 2013
NA = 20
Solvent = cdcl3
PTS1d = 65536
F1 = 100.520737 MHz
198.264
102.234
88.680
77.317
77.000
76.683
31.925
31.840
29.508
29.376
29.314
29.136
29.036
27.534
26.243
22.695
14.098
2s
n-C8H17
PPM

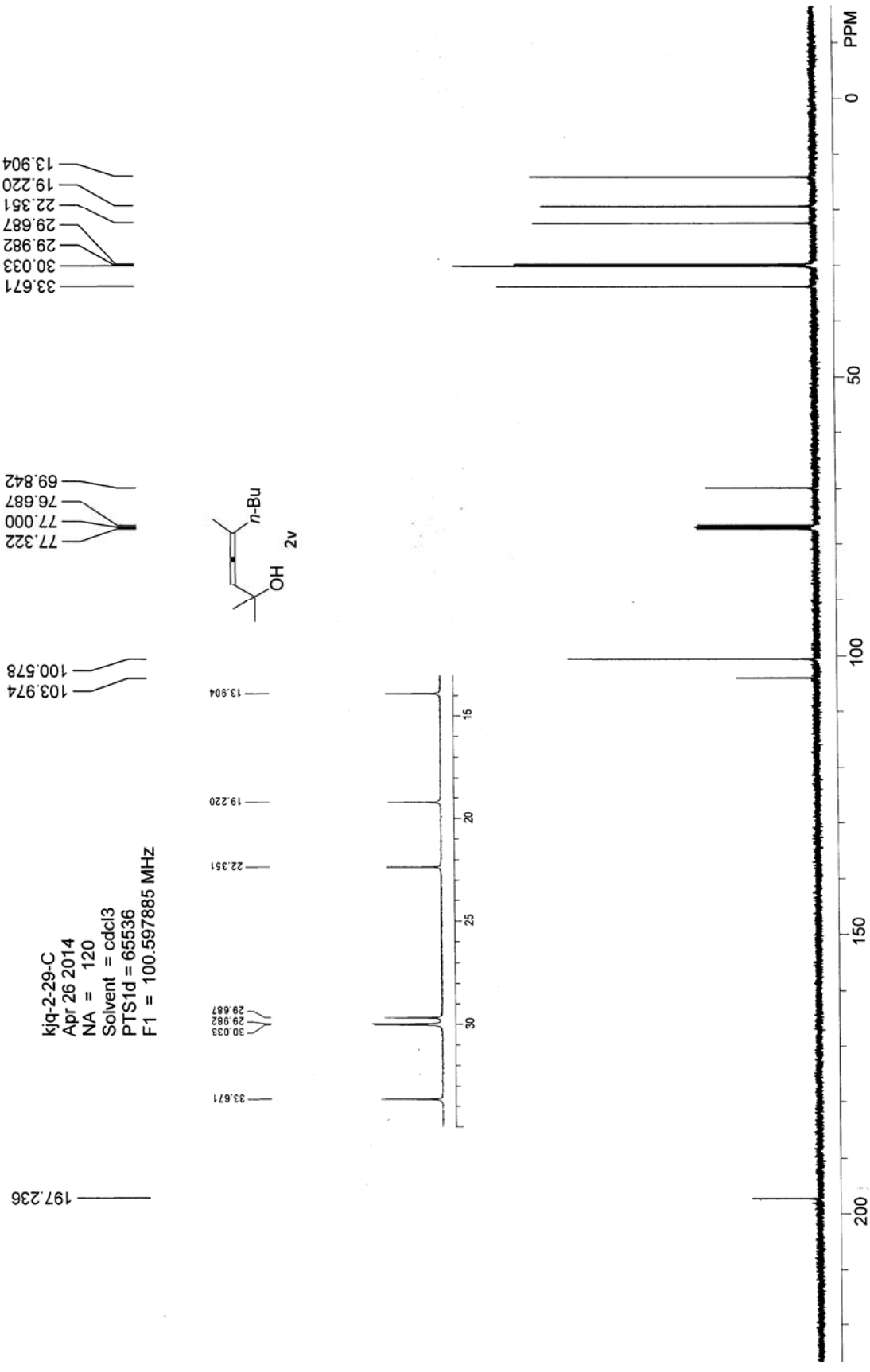

7.260
4.944
4.935
4.928
4.920
4.912
1.960
1.943
1.925
1.908
1.678
1.670
1.541
1.390
1.386
1.373
1.353
1.339
1.323
1.314
1.305
1.271
0.900
0.883
0.865
0.002
kjq-2-47-H
Oct 22 2013
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 399.722809 MHz
n-C8H17
2t
0.92
2.07
6.00
12.82
3.10
8
7
6
5
4
3
2
1
0
PPM

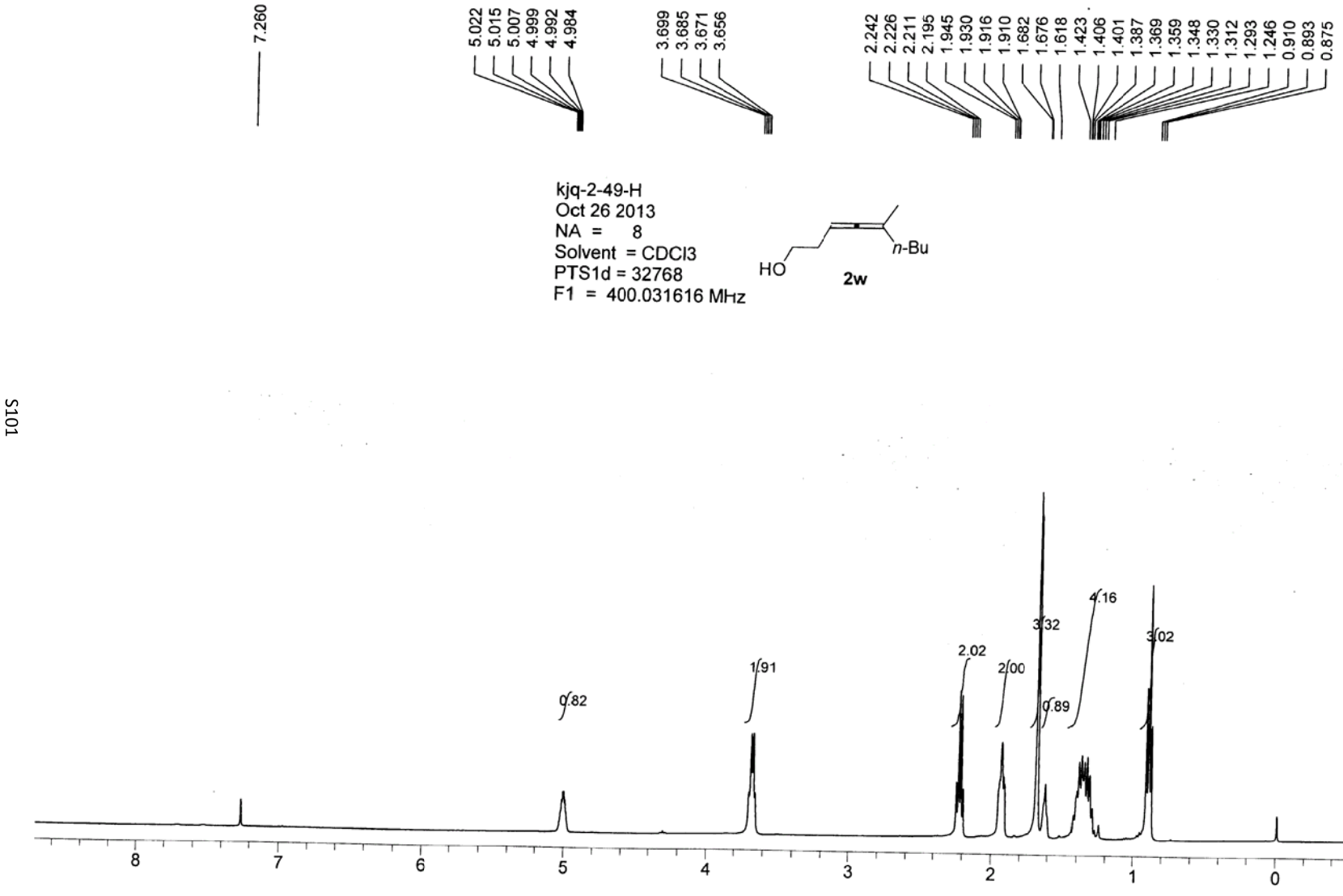

201.646
kjq-2-47-C
Oct 22 2013
NA = 100
Solvent = cdcl3
PTS1d = 65536
F1 = 100.520737 MHz
94.654
88.839
77.317
77.000
76.682
31.908
29.478
29.348
29.230
29.218
29.062
22.683
20.768
14.106
29.478
29.348
29.230
29.218
29.062
n-C8H17
2t
29.5
29.4
29.3
29.2
29.1
200
150
100
50
0
PPM

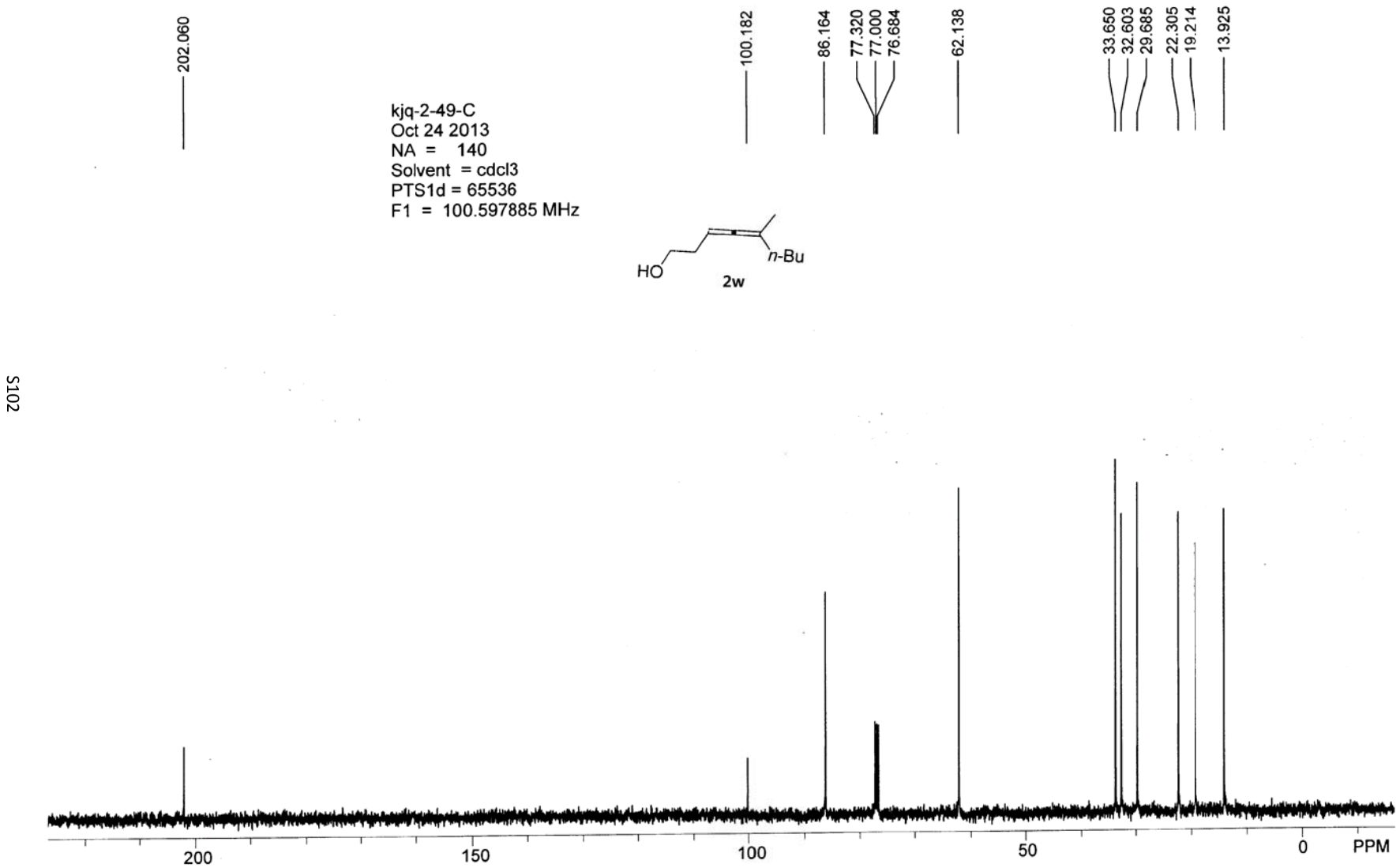

7.260
5.077
5.067
5.061
5.051
5.041
5.035
5.025
2.331
2.323
2.315
2.281
1.982
1.965
1.948
1.931
1.673
1.664
1.656
1.647
1.638
1.551
1.392
1.376
1.359
1.340
1.317
1.299
1.266
0.894
0.879
0.862
-0.002
kjq-2-54-H
Apr 26 2014
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 400.031616 MHz
n-C8H17
2u
0.90
4.11
2.05
4.00
8.11
3.09
8
7
6
5
4
3
2
1
0
PPM

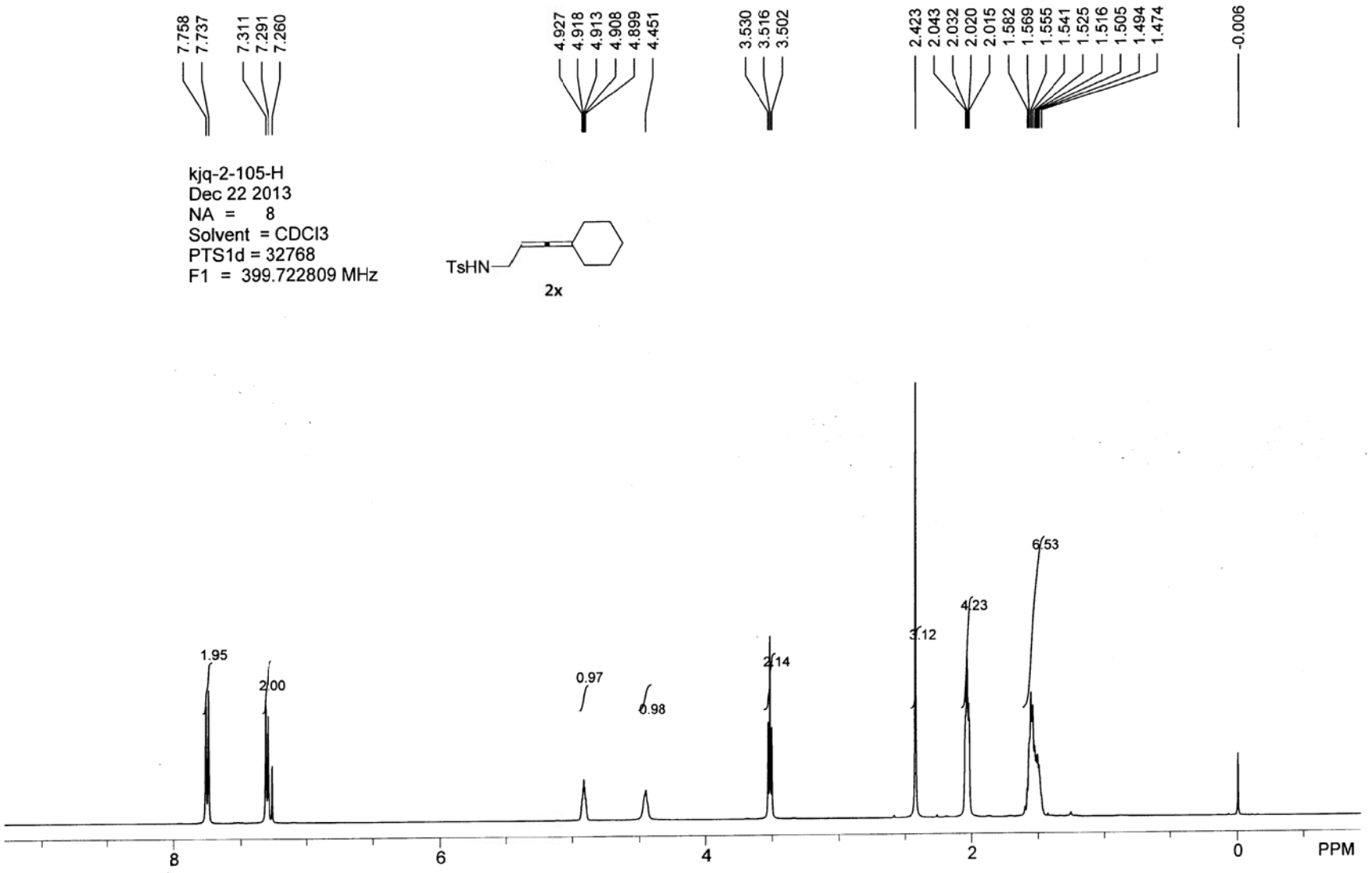

196.960
103.446
91.431
77.317
77.000
76.682
31.904
31.229
29.484
29.359
29.349
29.167
29.078
27.063
22.682
14.116
kjq-2-54-C
Apr 26 2014
NA = 156
Solvent = cdcl3
PTS1d = 32768
F1 = 100.520737 MHz
n-C8H17
2u
200
150
100
50
0
PPM

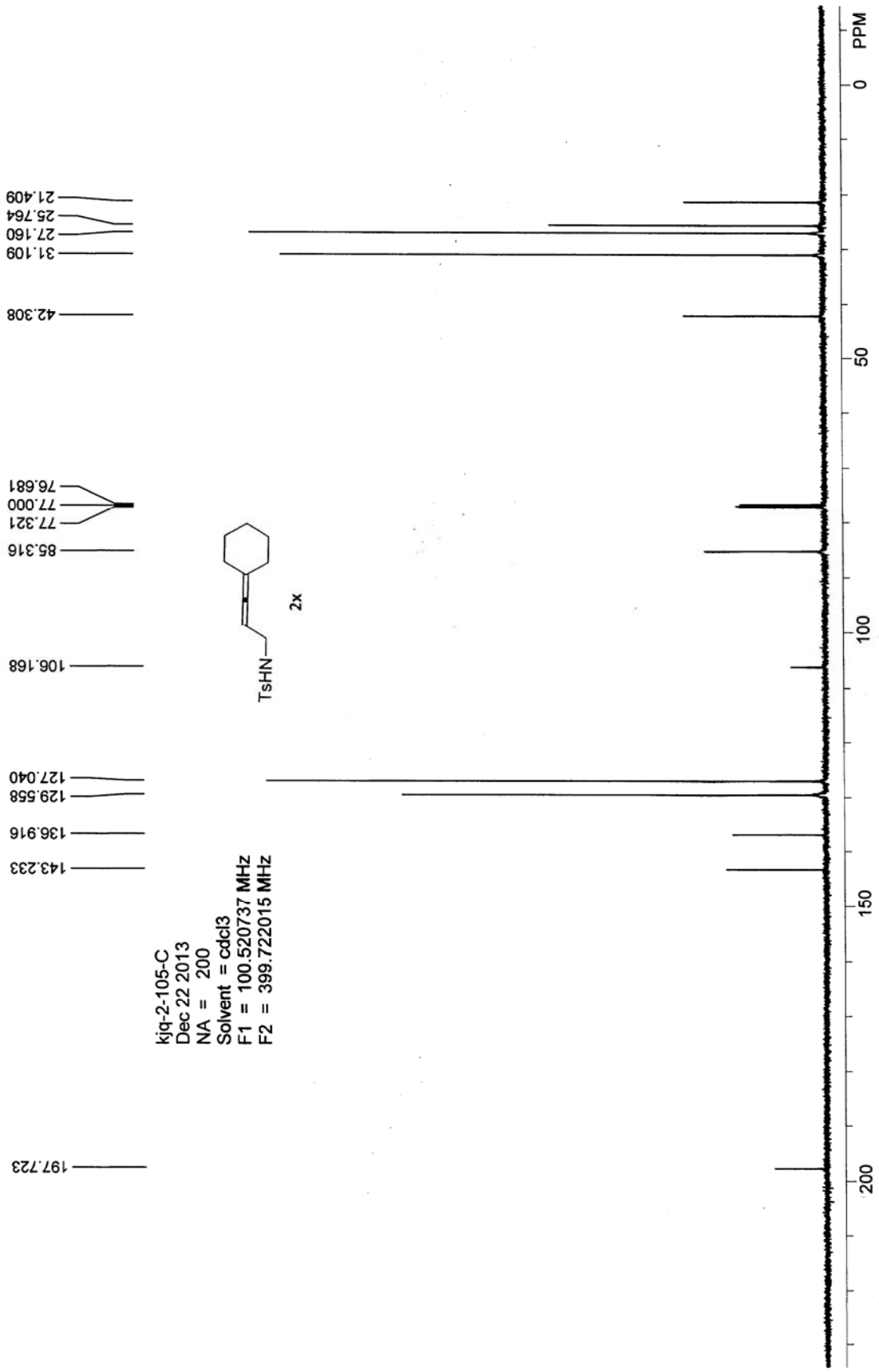

7.260
5.238
5.231
5.223
5.217
1.981
1.974
1.958
1.945
1.938
1.708
1.701
1.690
1.422
1.404
1.400
1.388
1.380
1.369
1.360
1.350
1.334
1.311
1.245
0.910
0.892
0.874
-0.011
kjq-2-29-H
Oct 10 2013
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 400.031219 MHz
n-Bu
OH
2v
0.88
2.00
4.40
10.94
3.06
8
6
4
2
0
PPM

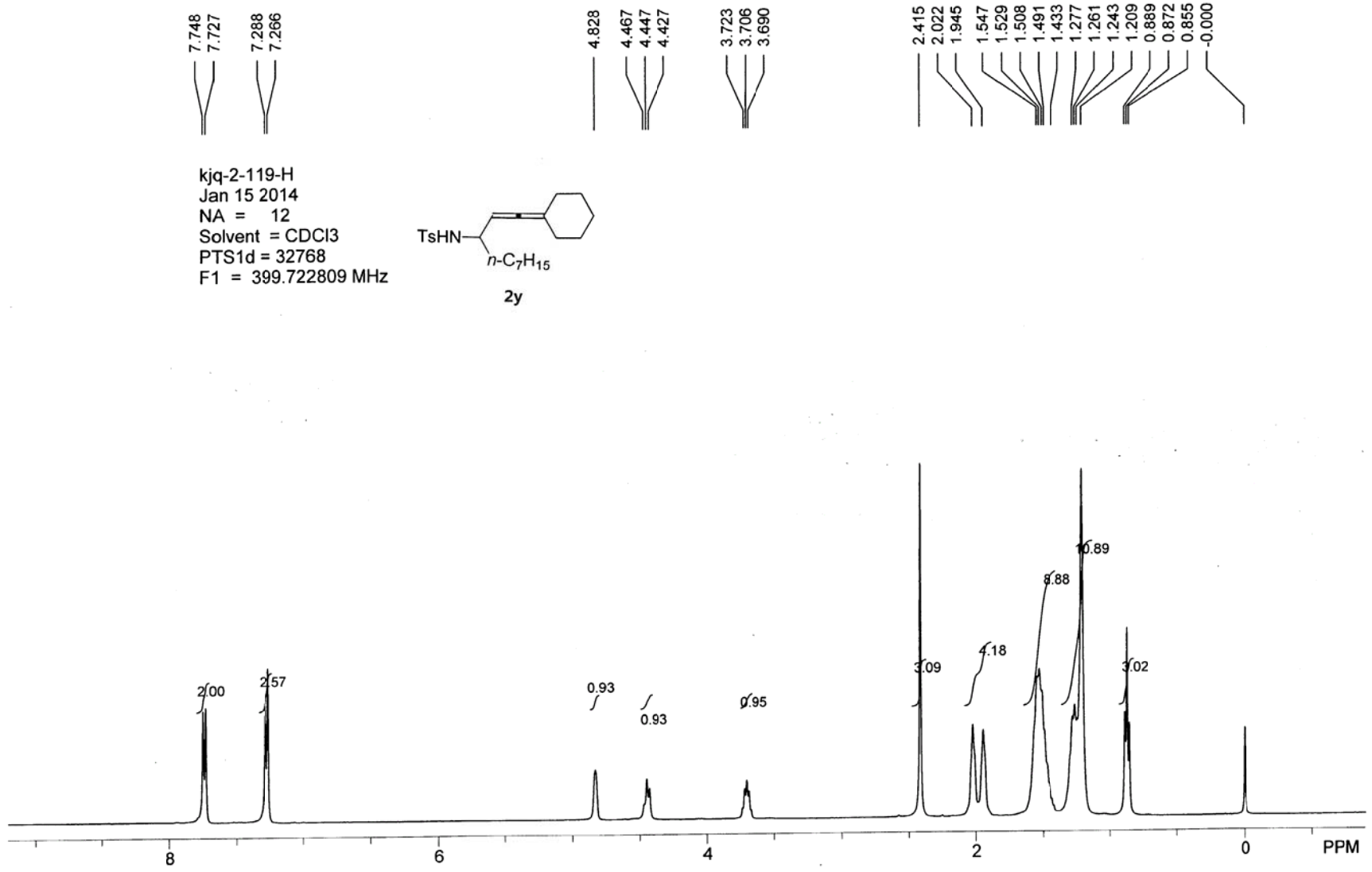

kjq-2-29-C
Apr 26 2014
NA = 120
Solvent = cdcl3
PTS1d = 65536
F1 = 100.597885 MHz
n-Bu
OH
2v
13.904
19.220
22.351
29.687
29.982
30.033
33.671
69.842
76.687
77.000
77.322
100.578
103.974
197.236
PPM

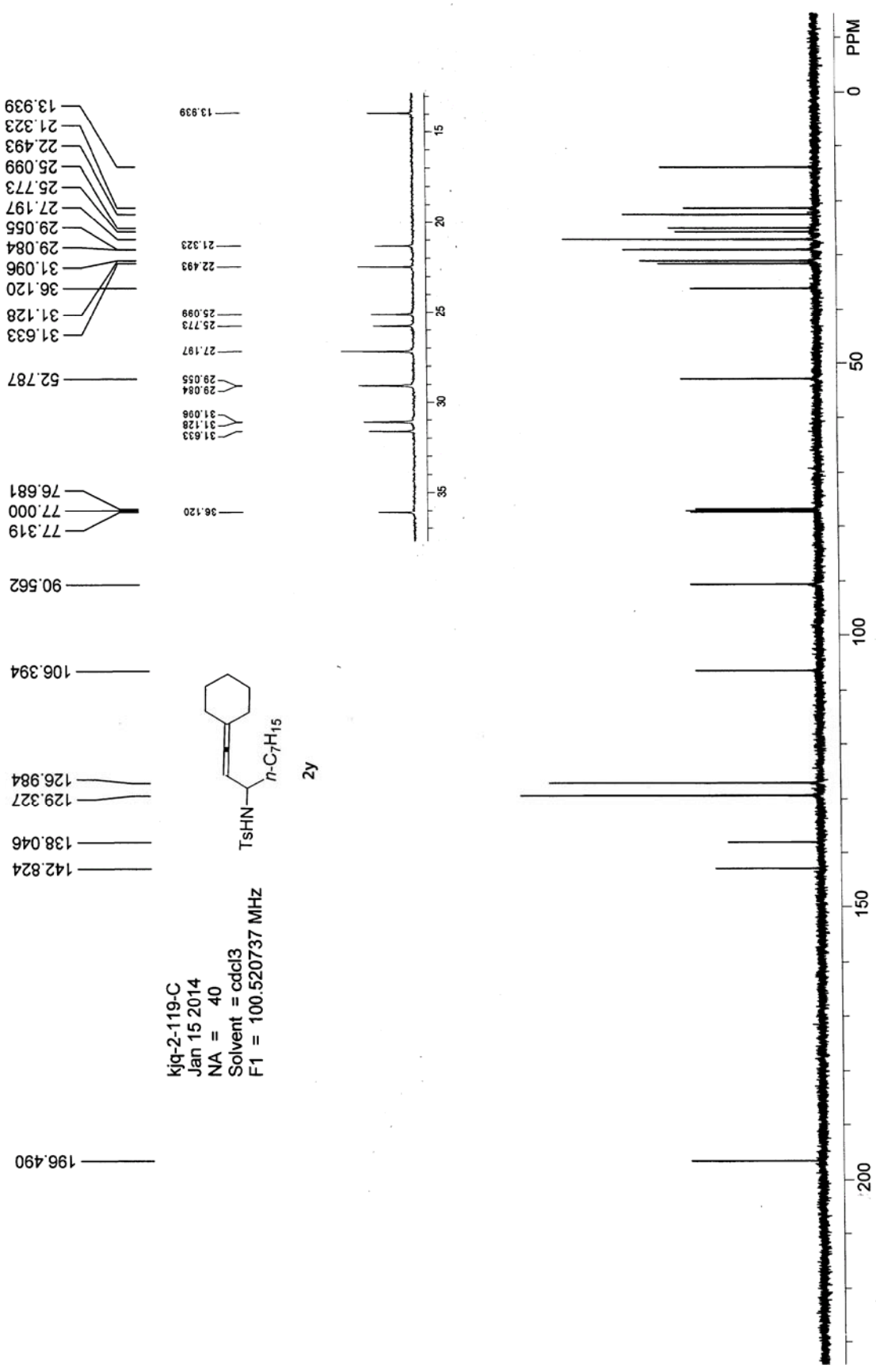

7.260
5.022
5.015
5.007
4.999
4.992
4.984
3.699
3.685
3.671
3.656
2.242
2.226
2.211
2.195
1.945
1.930
1.916
1.910
1.682
1.676
1.618
1.423
1.406
1.401
1.387
1.369
1.359
1.348
1.330
1.312
1.293
1.246
0.910
0.893
0.875
kjq-2-49-H
Oct 26 2013
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 400.031616 MHz
HO
n-Bu
2w
0.82
1.91
2.02
2.00
3.32
0.89
4.16
3.02
8
7
6
5
4
3
2
1
0
PPM

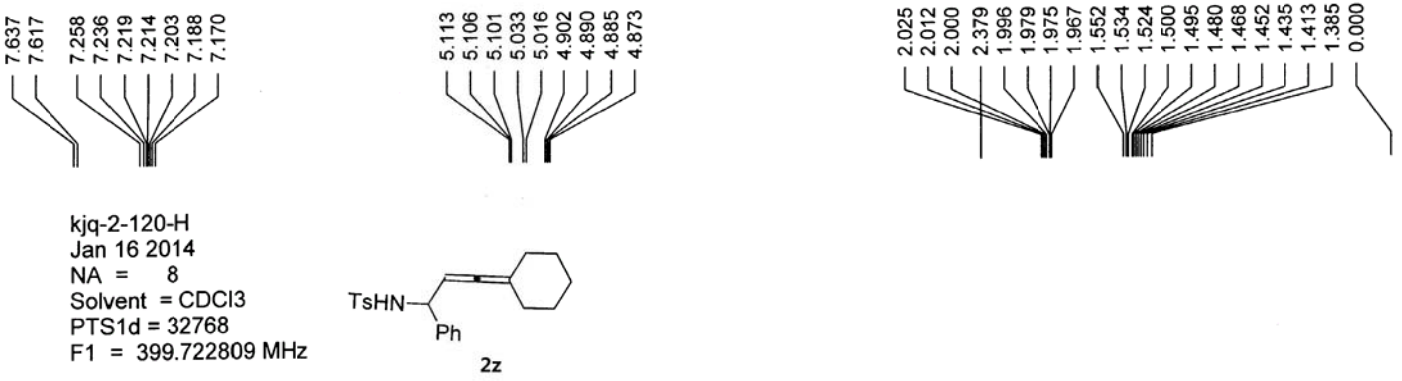

202.060
100.182
86.164
77.320
77.000
76.684
62.138
33.650
32.603
29.685
22.305
19.214
13.925
kjq-2-49-C
Oct 24 2013
NA = 140
Solvent = cdcl3
PTS1d = 65536
F1 = 100.597885 MHz
HO
n-Bu
2w
200
150
100
50
0
PPM

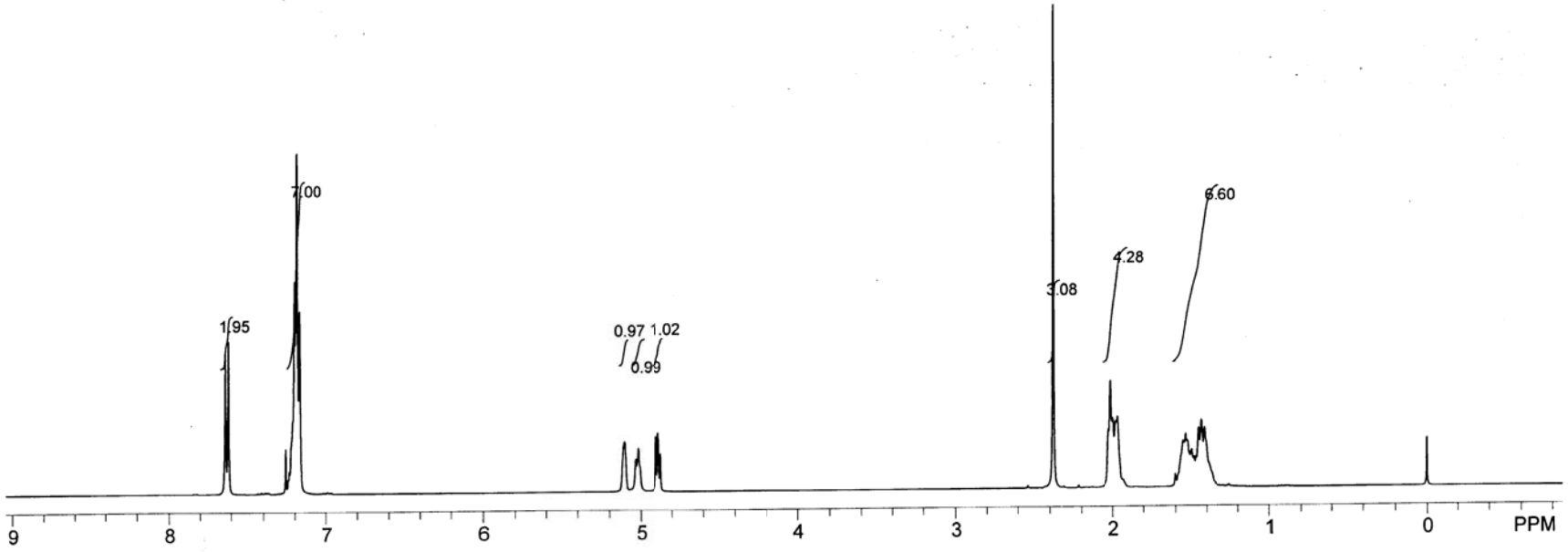

7.758
7.737
7.311
7.291
7.260
4.927
4.918
4.913
4.908
4.899
4.451
3.530
3.516
3.502
2.423
2.043
2.032
2.020
2.015
1.582
1.569
1.555
1.541
1.525
1.516
1.505
1.494
1.474
-0.006
kjq-2-105-H
Dec 22 2013
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 399.722809 MHz
TsHN
2x
1.95
2.00
0.97
0.98
2.14
3.12
4.23
6.53
8
6
4
2
0
PPM

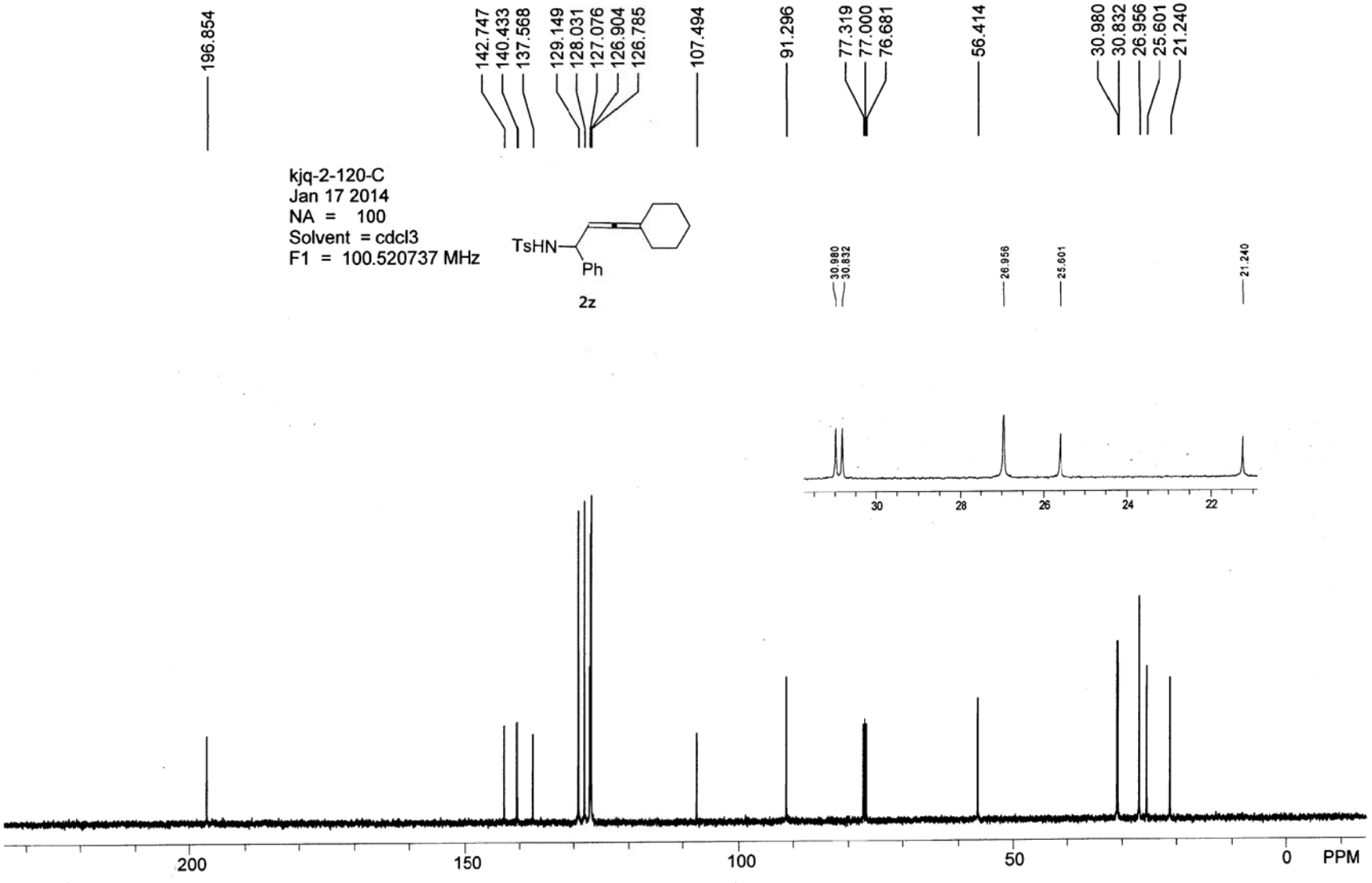

kjq-2-105-C
Dec 22 2013
NA = 200
Solvent = cdcl3
F1 = 100.520737 MHz
F2 = 399.722015 MHz
TsHN
2x
197.723
143.233
136.916
129.558
127.040
106.168
85.316
77.321
77.000
76.681
42.308
31.109
27.160
25.764
21.409
PPM
0
50
100
150
200

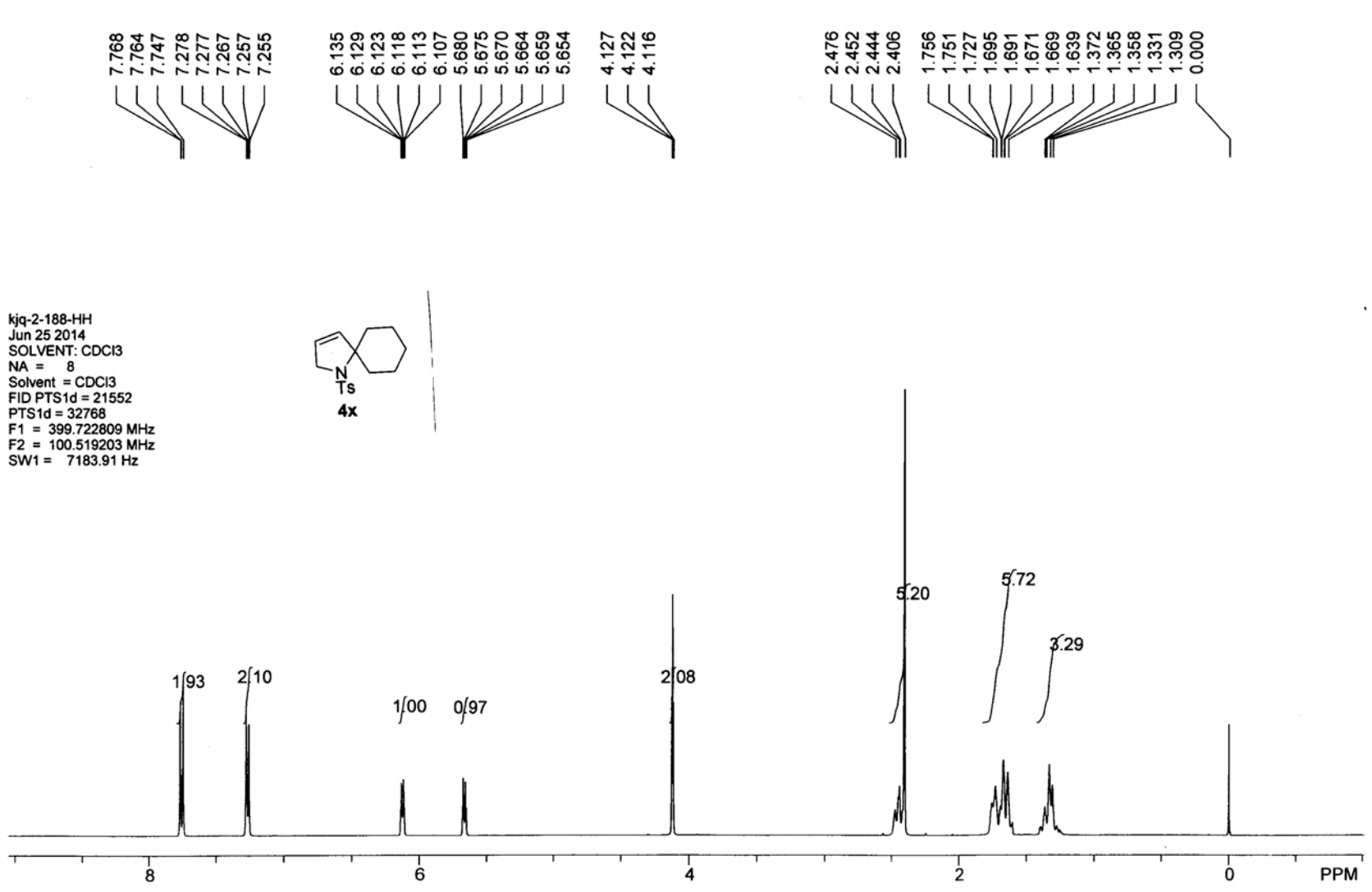

7.748
7.727
7.288
7.266
4.828
4.467
4.447
4.427
3.723
3.706
3.690
2.415
2.022
1.945
1.547
1.529
1.508
1.491
1.433
1.277
1.261
1.243
1.209
0.889
0.872
0.855
-0.000
kjq-2-119-H
Jan 15 2014
NA = 12
Solvent = CDCl3
PTS1d = 32768
F1 = 399.722809 MHz
TsHN
n-C7H15
2y
2.00
2.57
0.93
0.93
0.95
3.09
4.18
6.88
6.89
3.02
8
6
4
2
0
PPM

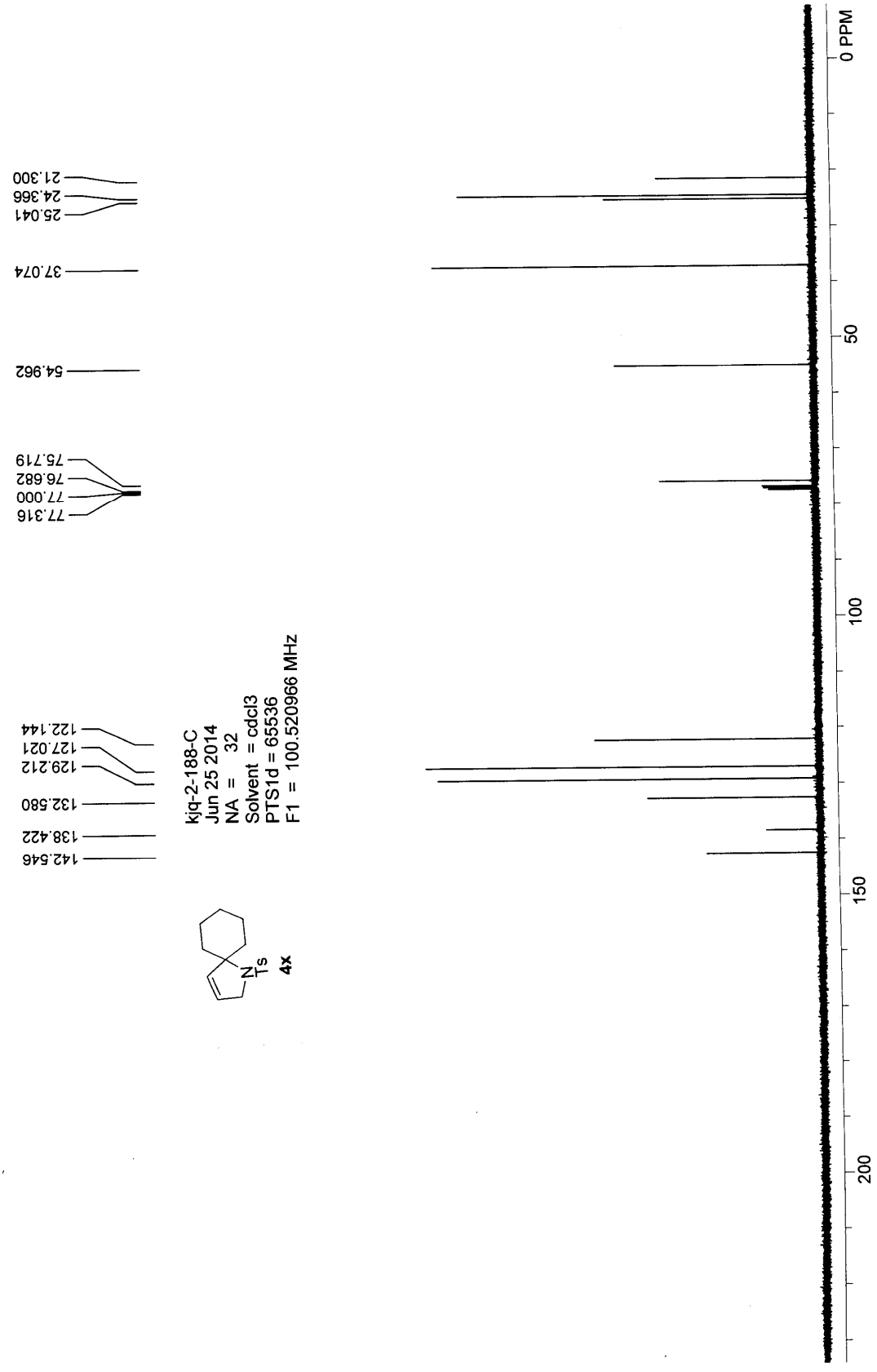

196.490
142.824
138.046
129.327
126.984
106.394
90.562
77.319
77.000
76.681
52.787
31.633
31.128
36.120
31.096
29.084
29.055
27.197
25.773
25.099
22.493
21.323
13.939
kjq-2-119-C
Jan 15 2014
NA = 40
Solvent = cdcl3
F1 = 100.520737 MHz
TsHN
n-C7H15
2y
PPM

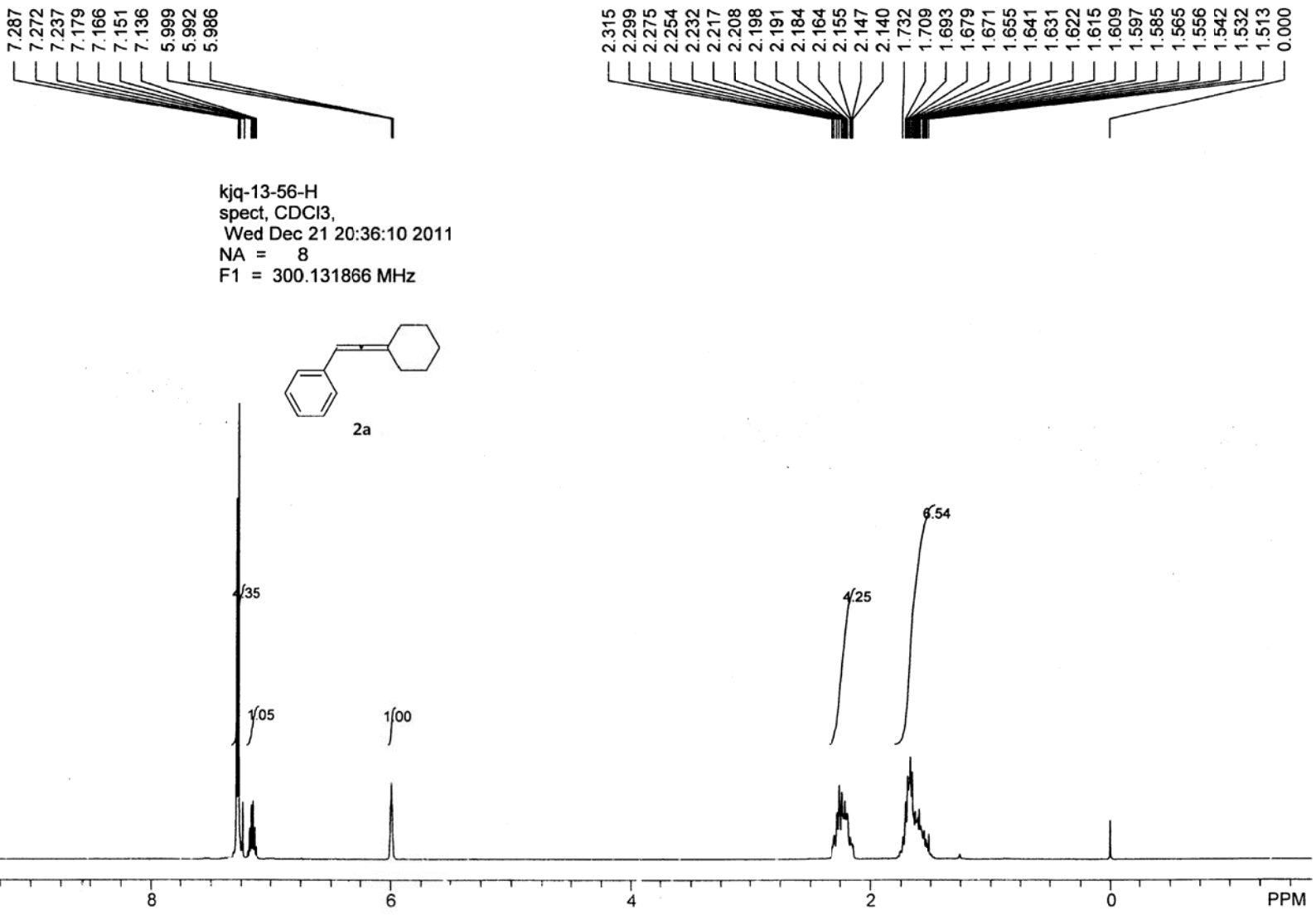

7.637
7.617
7.258
7.236
7.219
7.214
7.203
7.188
7.170
5.113
5.106
5.101
5.033
5.016
4.902
4.890
4.885
4.873
2.025
2.012
2.000
2.379
1.996
1.979
1.975
1.967
1.552
1.534
1.524
1.500
1.495
1.480
1.468
1.452
1.435
1.413
1.385
0.000
kjq-2-120-H
Jan 16 2014
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 399.722809 MHz
TsHN
Ph
2z

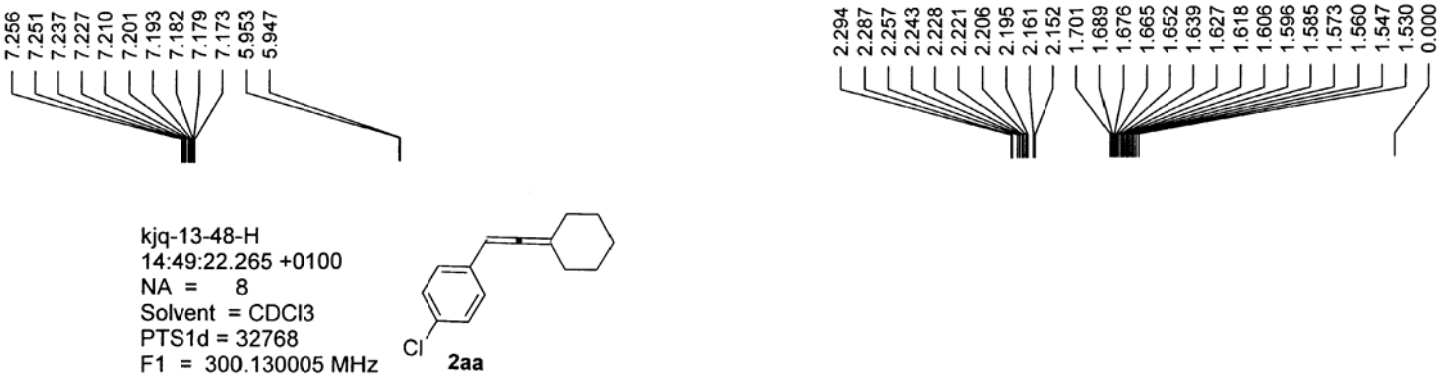

1.95
7.00
0.97
1.02
0.99
3.08
4.28
6.60
9
8
7
6
5
4
3
2
1
0
PPM

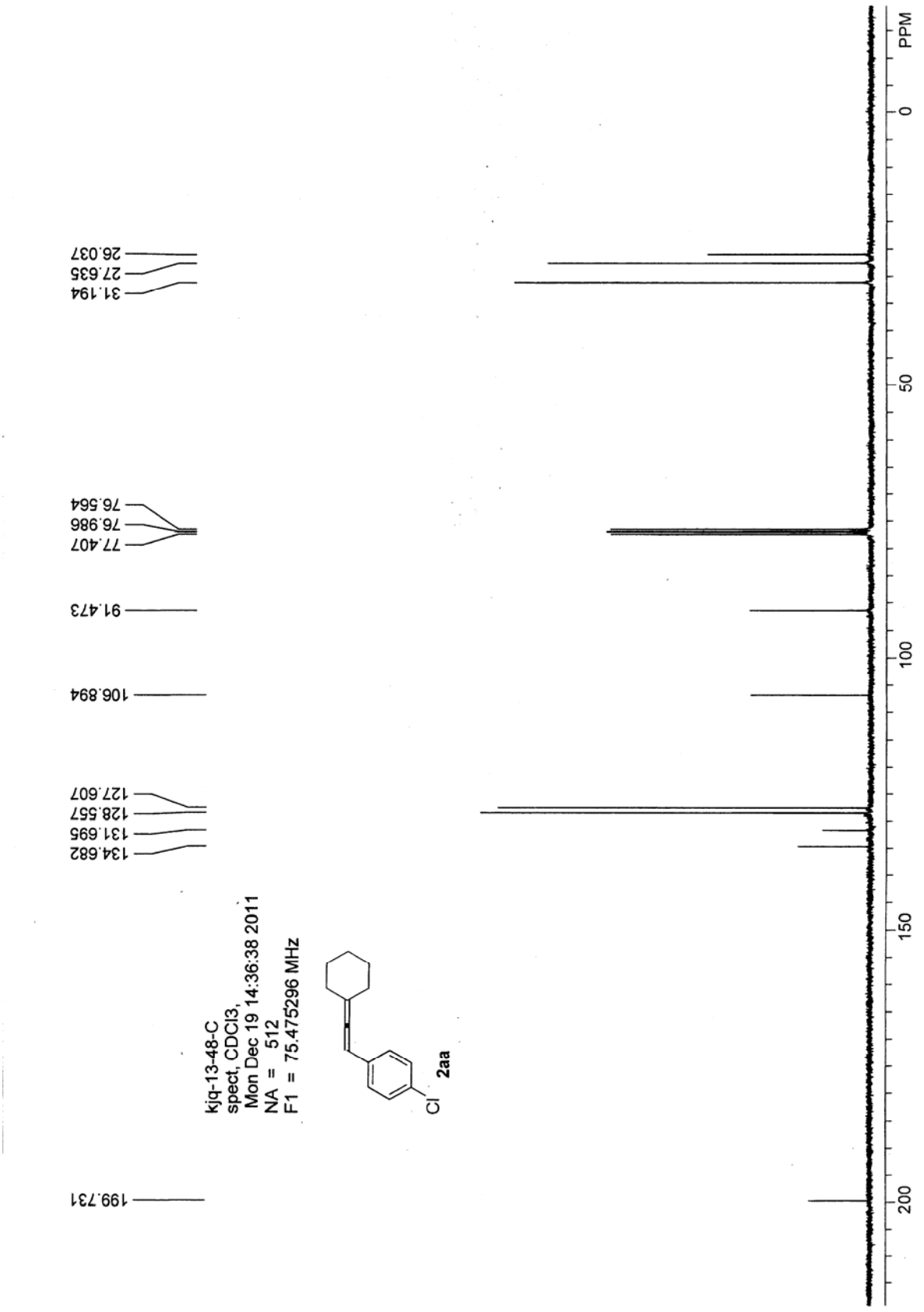

196.854
142.747
140.433
137.568
129.149
128.031
127.076
126.904
126.785
107.494
91.296
77.319
77.000
76.681
56.414
30.980
30.832
26.956
25.601
21.240
kjq-2-120-C
Jan 17 2014
NA = 100
Solvent = cdcl3
F1 = 100.520737 MHz
TsHN
Ph
2z
200
150
100
50
0
PPM
30
28
26
24
22

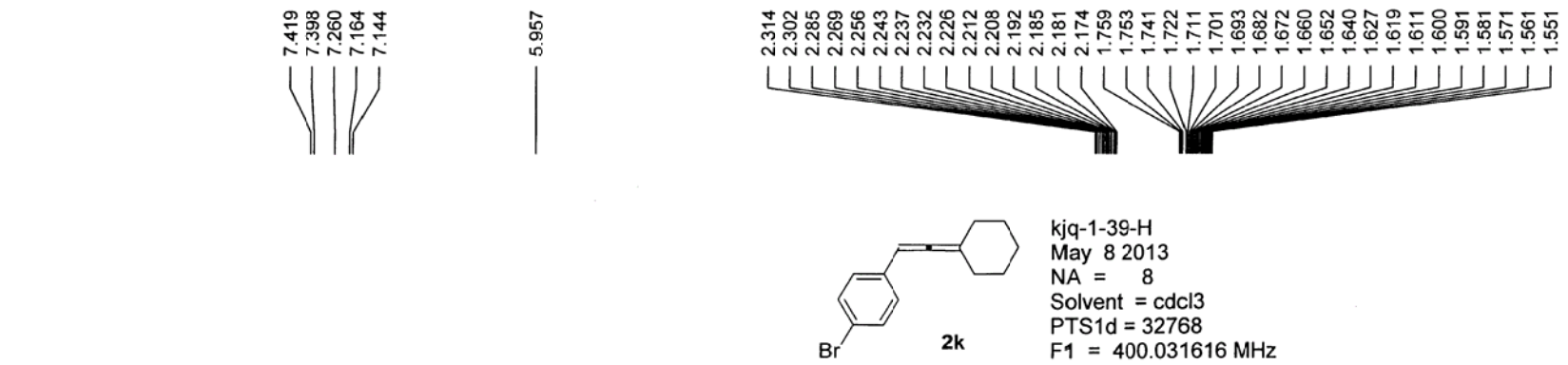

7.768
7.764
7.747
7.278
7.277
7.267
7.257
7.255
6.135
6.129
6.123
6.118
6.113
6.107
5.680
5.675
5.670
5.664
5.659
5.654
4.127
4.122
4.116
2.476
2.452
2.444
2.406
1.756
1.751
1.727
1.695
1.691
1.671
1.669
1.639
1.372
1.365
1.358
1.331
1.309
0.000
kjq-2-188-HH
Jun 25 2014
SOLVENT: CDCl3
NA = 8
Solvent = CDCl3
FID PTS1d = 21552
PTS1d = 32768
F1 = 399.722809 MHz
F2 = 100.519203 MHz
SW1 = 7183.91 Hz
N
Ts
4x
1.93
2.10
1.00
0.97
2.08
5.20
5.72
3.29
8
6
4
2
0
PPM

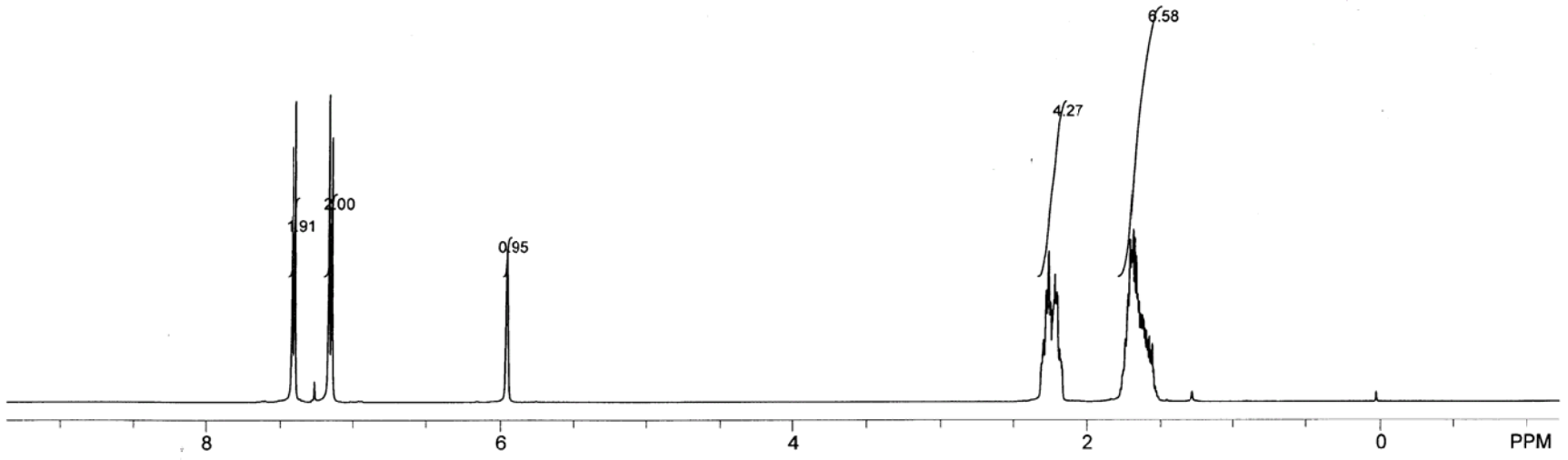

21.300
24.366
25.041
37.074
54.962
75.719
76.682
77.000
77.316
122.144
127.021
129.212
132.580
138.422
142.546
kjq-2-188-C
Jun 25 2014
NA = 32
Solvent = cdcl3
PTS1d = 65536
F1 = 100.520966 MHz
Ts
N
4x
0 PPM
50
100
150
200

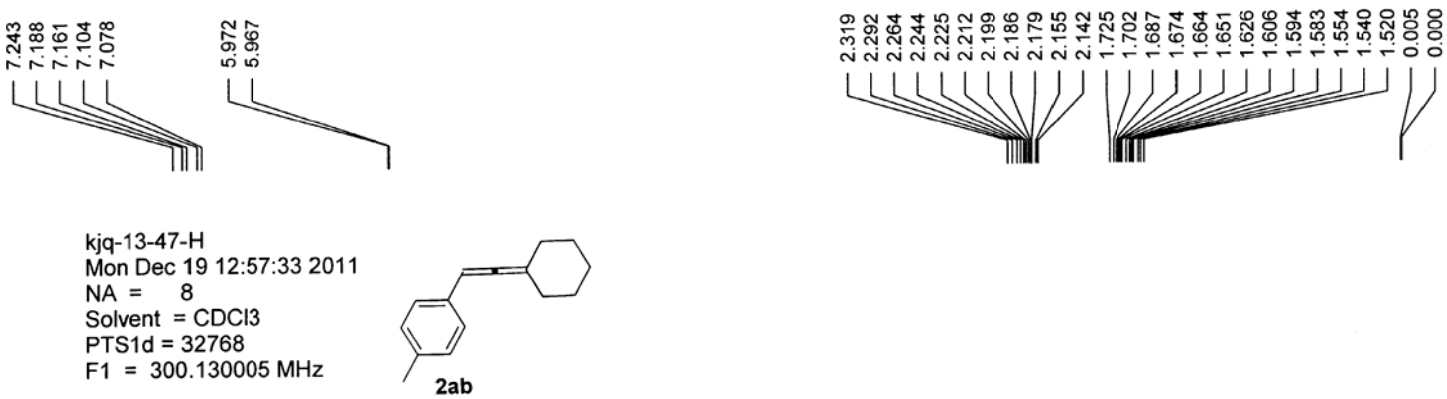

7.287
7.272
7.237
7.179
7.166
7.151
7.136
5.999
5.992
5.986
2.315
2.299
2.275
2.254
2.232
2.217
2.208
2.198
2.191
2.184
2.164
2.155
2.147
2.140
1.732
1.709
1.693
1.679
1.671
1.655
1.641
1.631
1.622
1.615
1.609
1.597
1.585
1.565
1.556
1.542
1.532
1.513
0.000
kjq-13-56-H
spect, CDCl3,
Wed Dec 21 20:36:10 2011
NA = 8
F1 = 300.131866 MHz
2a
4.35
1.05
1.00
4.25
6.54
8
6
4
2
0
PPM

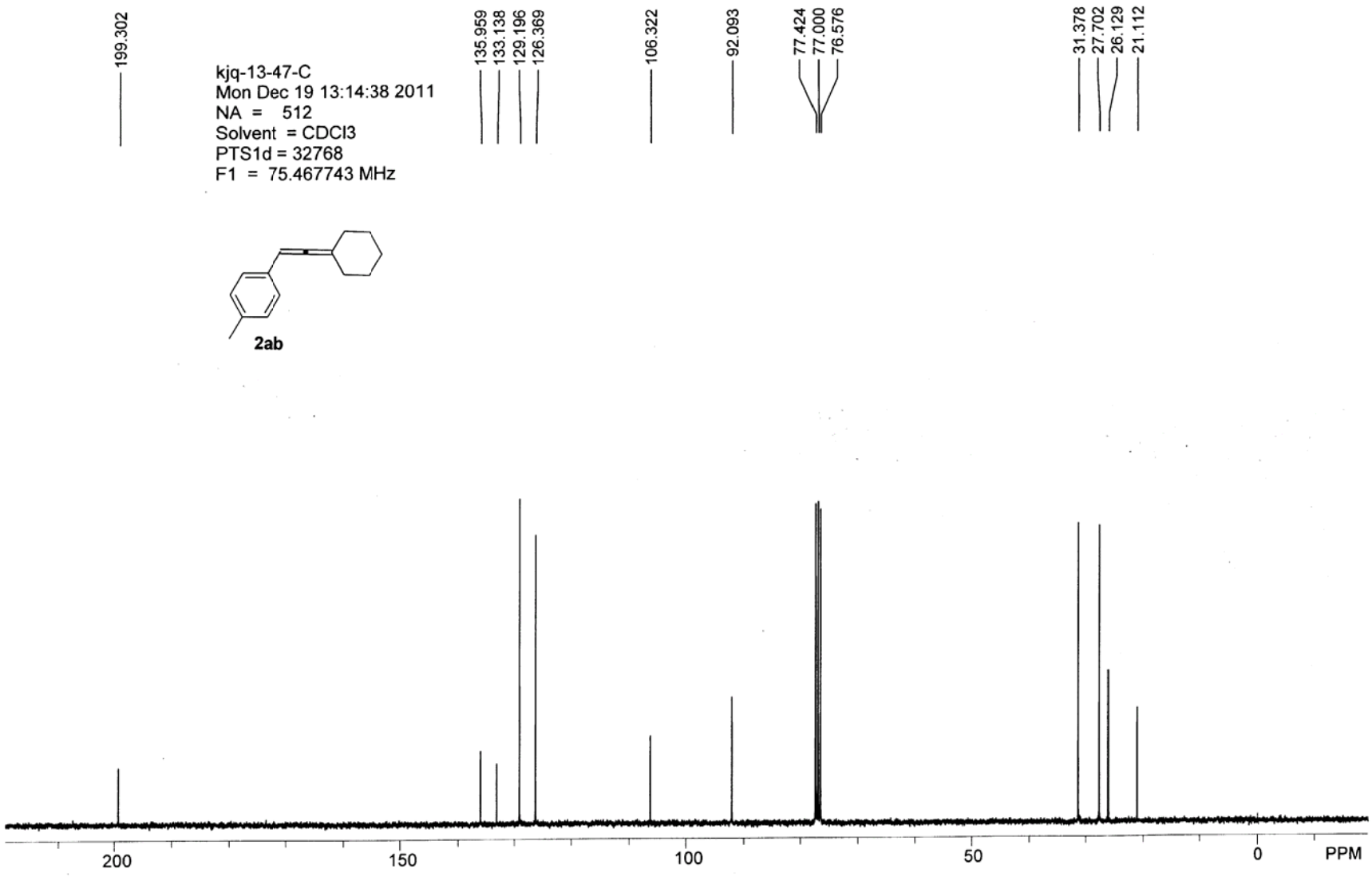

7.256
7.251
7.237
7.227
7.210
7.201
7.193
7.182
7.179
7.173
5.953
5.947
2.294
2.287
2.257
2.243
2.228
2.221
2.206
2.195
2.161
2.152
1.701
1.689
1.676
1.665
1.652
1.639
1.627
1.618
1.606
1.596
1.585
1.573
1.560
1.547
1.530
0.000
kjq-13-48-H
14:49:22.265 +0100
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
Cl
2aa
4.06
0.95
4.00
6.15
8
6
4
2
0
PPM

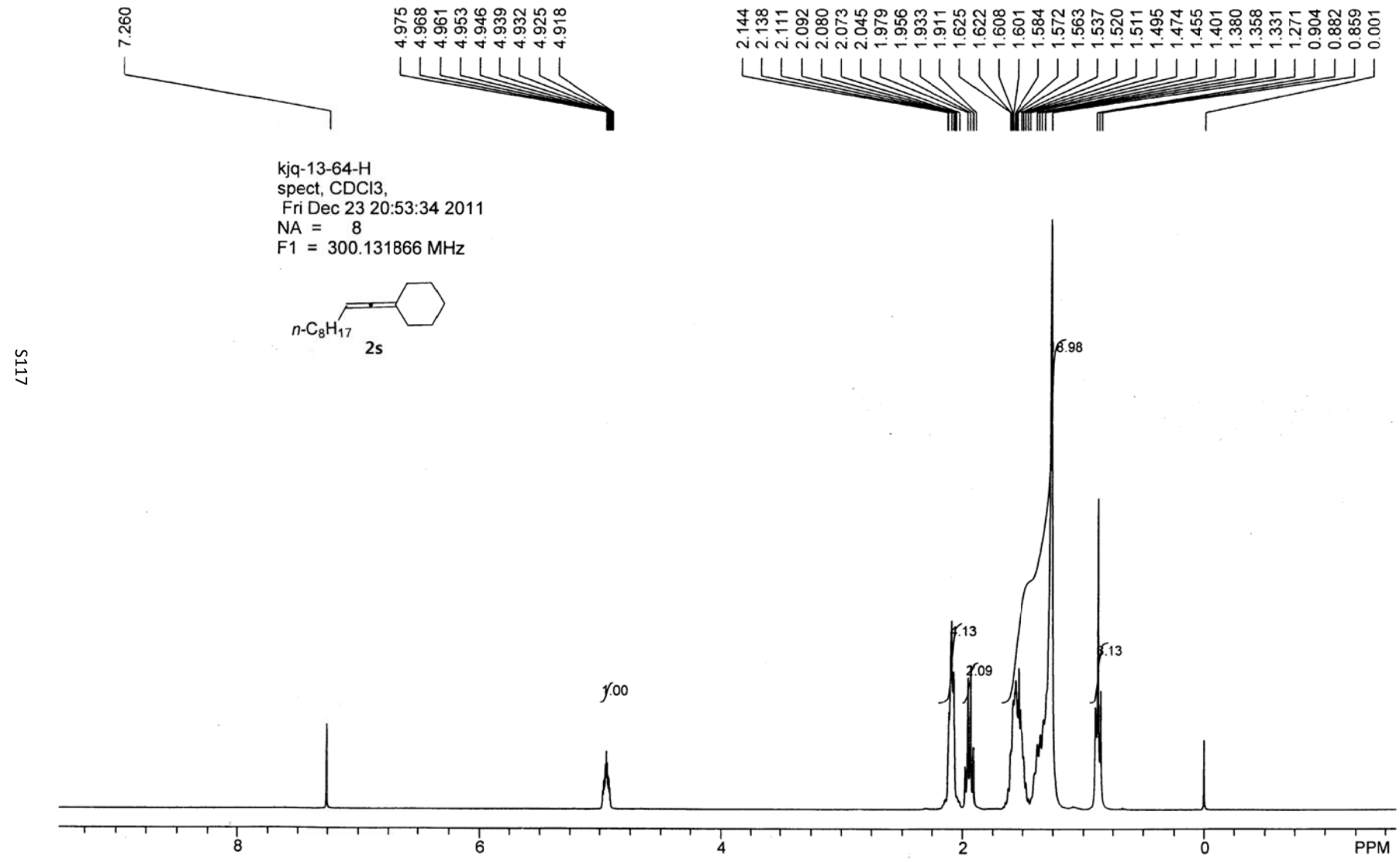

kjq-13-48-C
spect, CDCl3,
Mon Dec 19 14:36:38 2011
NA = 512
F1 = 75.475296 MHz
2aa
Cl
199.731
134.682
131.695
128.557
127.607
106.894
91.473
77.407
76.986
76.564
31.194
27.635
26.037
PPM
200
150
100
50
0

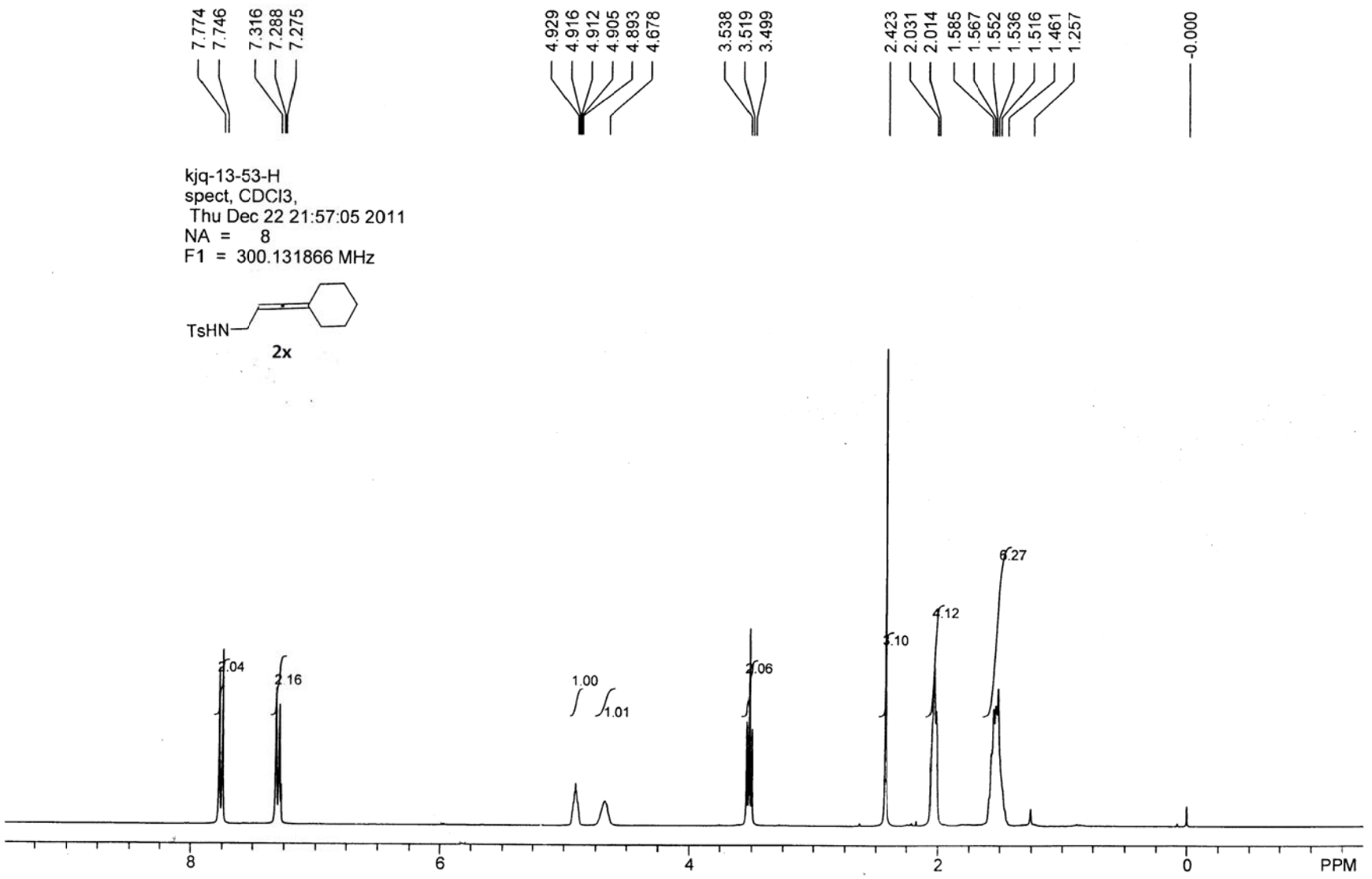

7.419
7.398
7.260
7.164
7.144
5.957
2.314
2.302
2.285
2.269
2.256
2.243
2.237
2.232
2.226
2.212
2.208
2.192
2.185
2.181
2.174
1.759
1.753
1.741
1.722
1.711
1.701
1.693
1.682
1.672
1.660
1.652
1.640
1.627
1.619
1.611
1.600
1.591
1.581
1.571
1.561
1.551
Br
2k
kjq-1-39-H
May 8 2013
NA = 8
Solvent = cdcl3
PTS1d = 32768
F1 = 400.031616 MHz

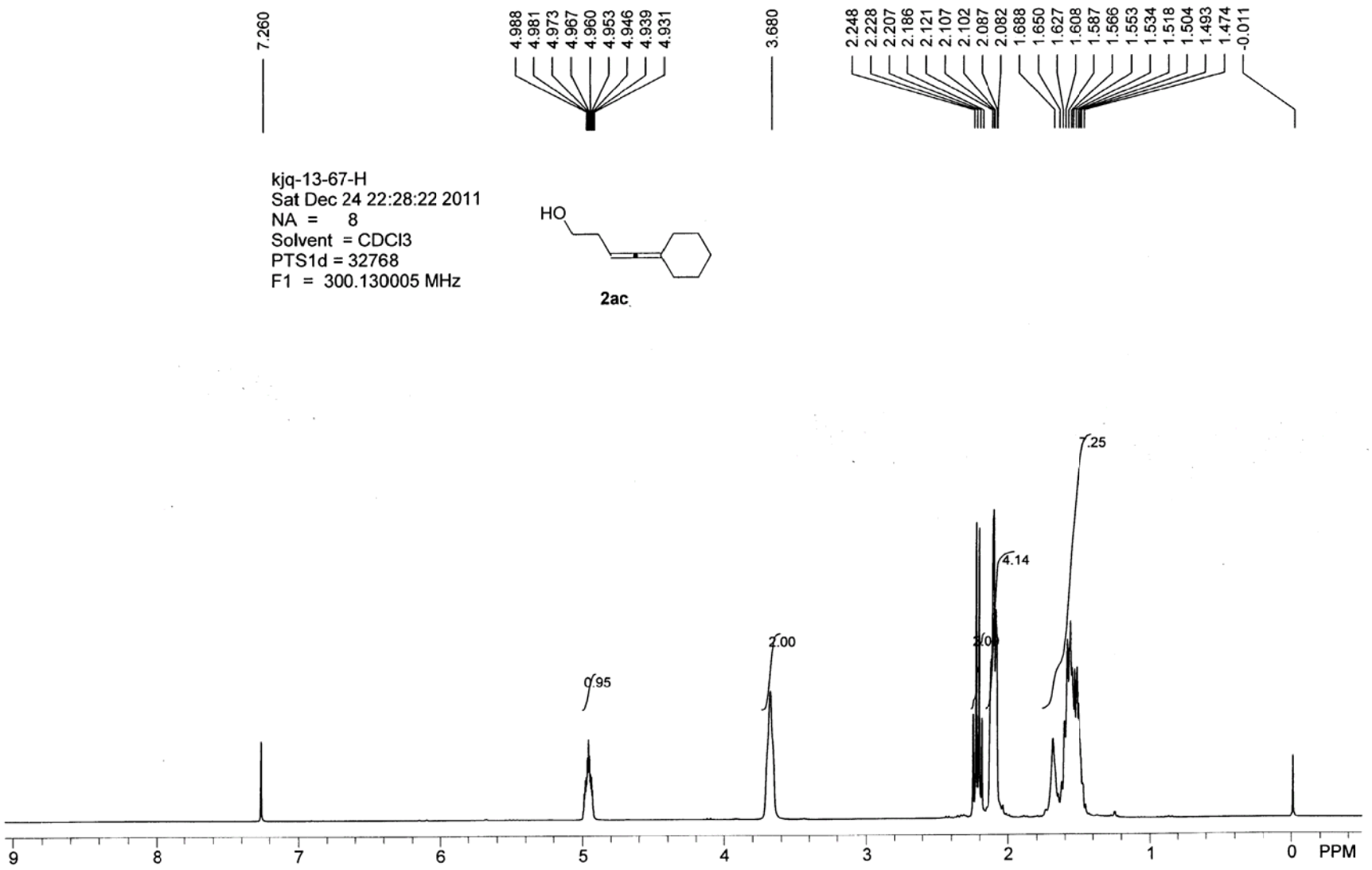

1.91
2.00
0.95
4.27
6.58
8
6
4
2
0
PPM

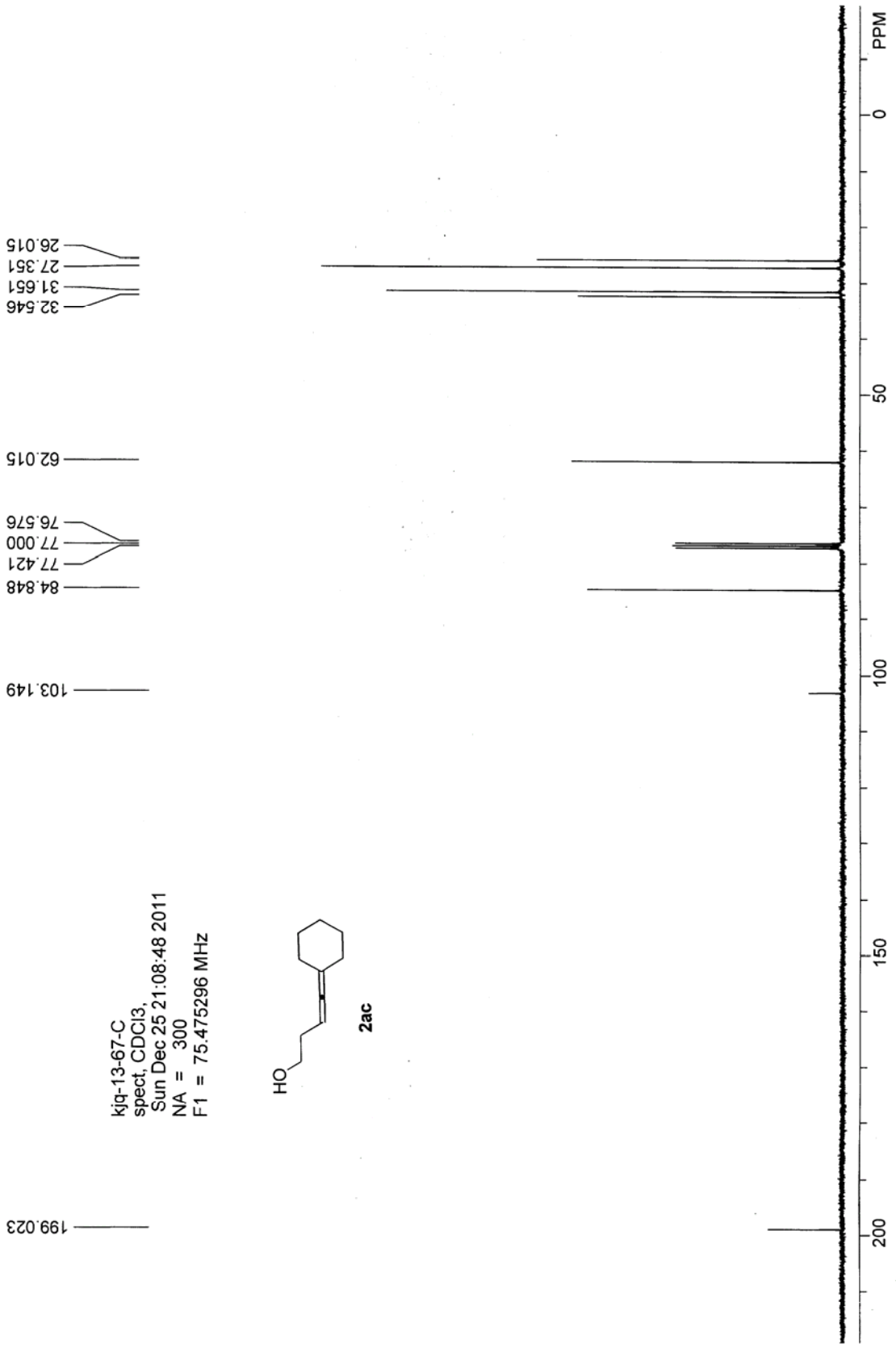

7.243
7.188
7.161
7.104
7.078
5.972
5.967
2.319
2.292
2.264
2.244
2.225
2.212
2.199
2.186
2.179
2.155
2.142
1.725
1.702
1.687
1.674
1.664
1.651
1.626
1.606
1.594
1.583
1.554
1.540
1.520
0.005
0.000
kjq-13-47-H
Mon Dec 19 12:57:33 2011
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
2ab
7.25
6.33
2.00
1.99
0.96
8
6
4
2
0
PPM

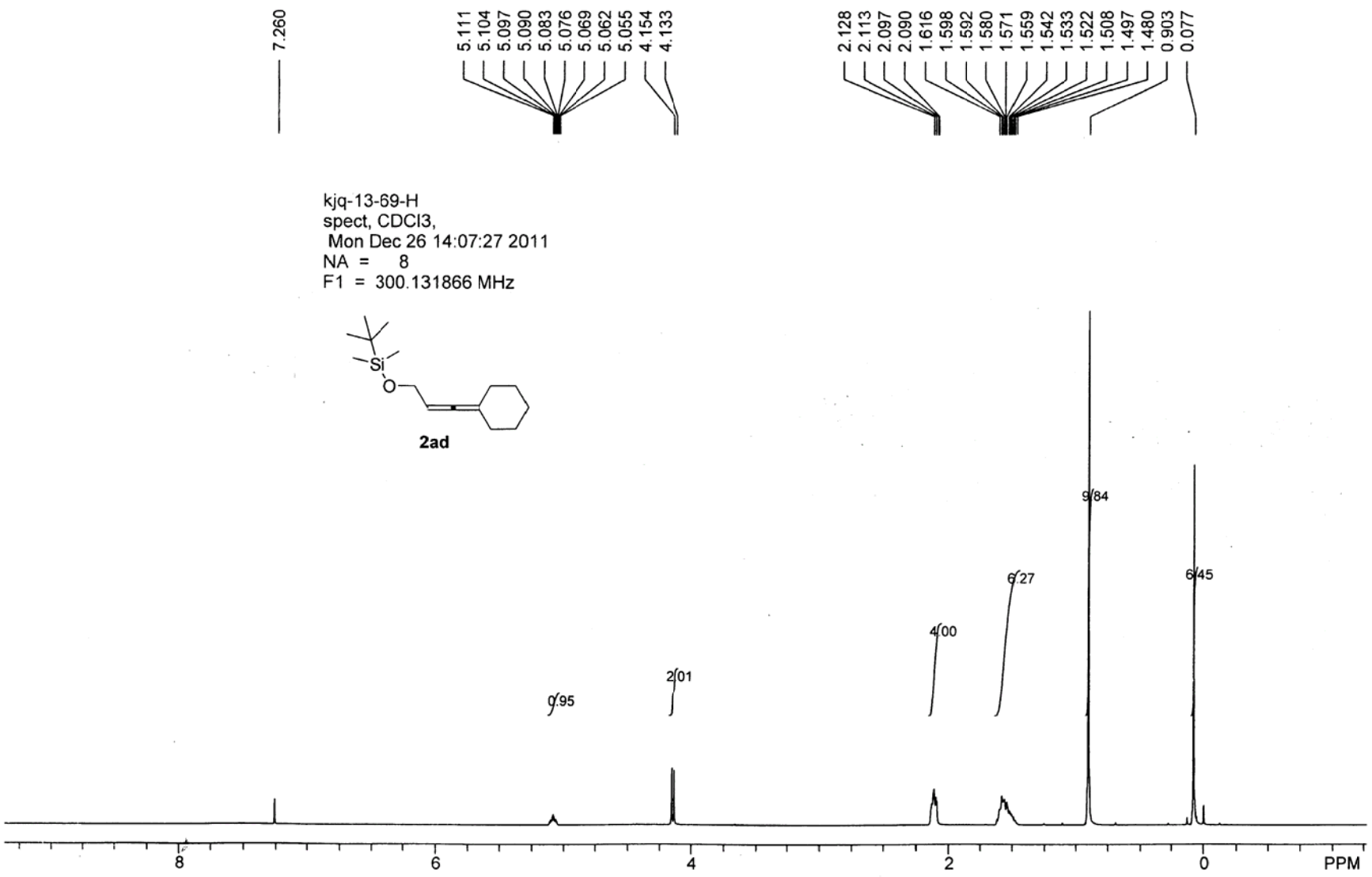

199.302
135.959
133.138
129.196
126.369
106.322
92.093
77.424
77.000
76.576
31.378
27.702
26.129
21.112
kjq-13-47-C
Mon Dec 19 13:14:38 2011
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467743 MHz
2ab
200
150
100
50
0
PPM

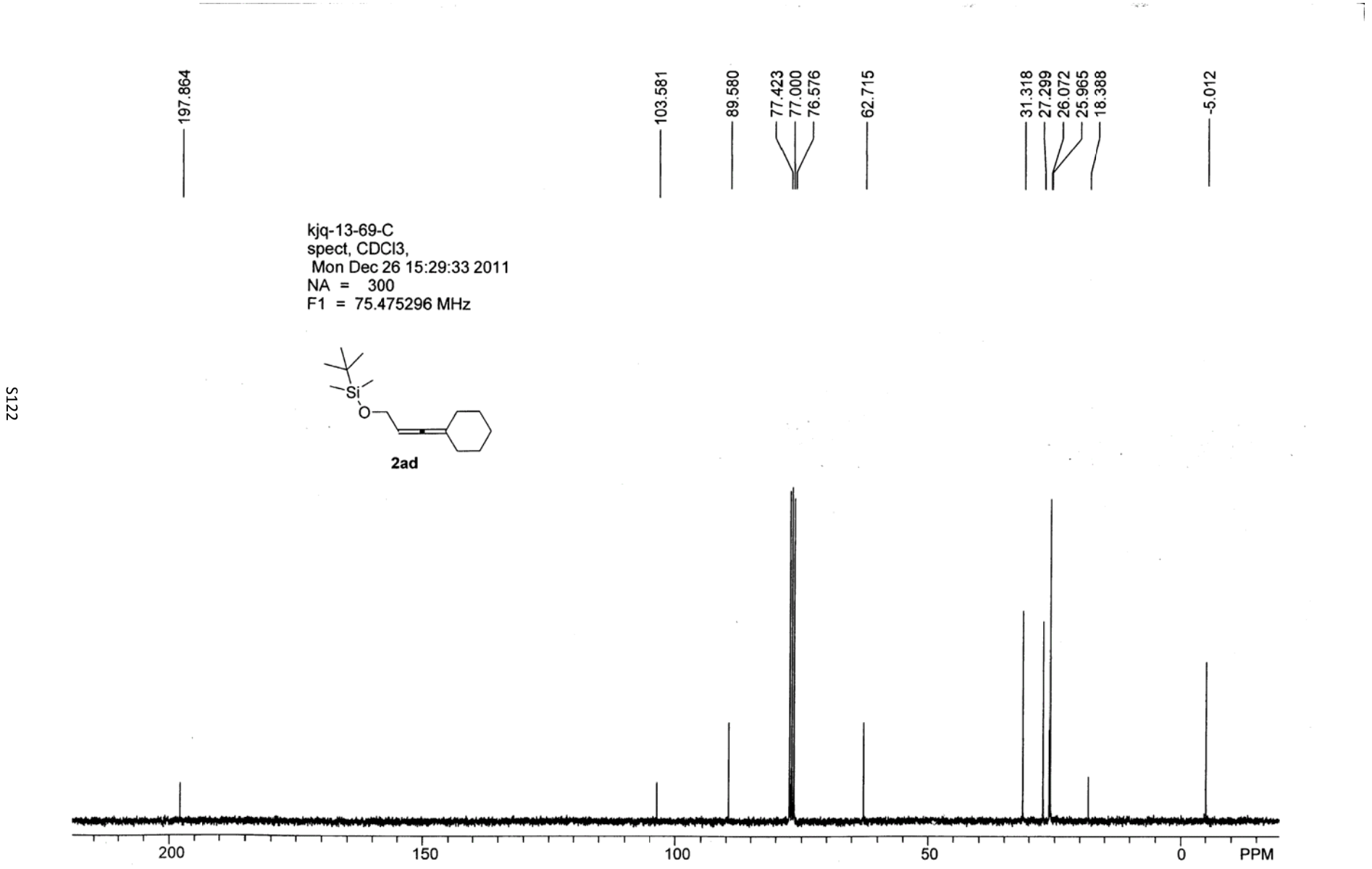

7.260
4.975
4.968
4.961
4.953
4.946
4.939
4.932
4.925
4.918
2.144
2.138
2.111
2.092
2.080
2.073
2.045
1.979
1.956
1.933
1.911
1.625
1.622
1.608
1.601
1.584
1.572
1.563
1.537
1.520
1.511
1.495
1.474
1.455
1.401
1.380
1.358
1.331
1.271
0.904
0.882
0.859
0.001
kjq-13-64-H
spect, CDCl3,
Fri Dec 23 20:53:34 2011
NA = 8
F1 = 300.131866 MHz
n-C8H17
2s
1.00
2.13
2.09
8.98
2.13
8
6
4
2
0
PPM

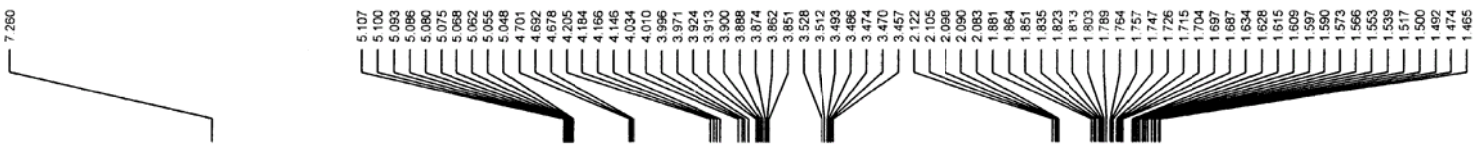

7.774
7.746
7.316
7.288
7.275
4.929
4.916
4.912
4.905
4.893
4.678
3.538
3.519
3.499
2.423
2.031
2.014
1.585
1.567
1.552
1.536
1.516
1.461
1.257
-0.000
kjq-13-53-H
spect, CDCl3,
Thu Dec 22 21:57:05 2011
NA = 8
F1 = 300.131866 MHz
TsHN
2x
2.04
2.16
1.00
1.01
2.06
3.10
4.12
6.27
8
6
4
2
0
PPM


7.260
4.988
4.981
4.973
4.967
4.960
4.953
4.946
4.939
4.931
3.680
2.248
2.228
2.207
2.186
2.121
2.107
2.102
2.087
2.082
1.688
1.650
1.627
1.608
1.587
1.566
1.553
1.534
1.518
1.504
1.493
1.474
-0.011
kjq-13-67-H
Sat Dec 24 22:28:22 2011
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
HO
2ac
0.95
2.00
2.0
4.14
7.25
9
8
7
6
5
4
3
2
1
0
PPM


199.023
103.149
84.848
77.421
77.000
76.576
62.015
32.546
31.651
27.351
26.015
PPM
0
50
100
150
200
kjq-13-67-C
spect, CDCl3,
Sun Dec 25 21:08:48 2011
NA = 300
F1 = 75.475296 MHz
HO
2ac

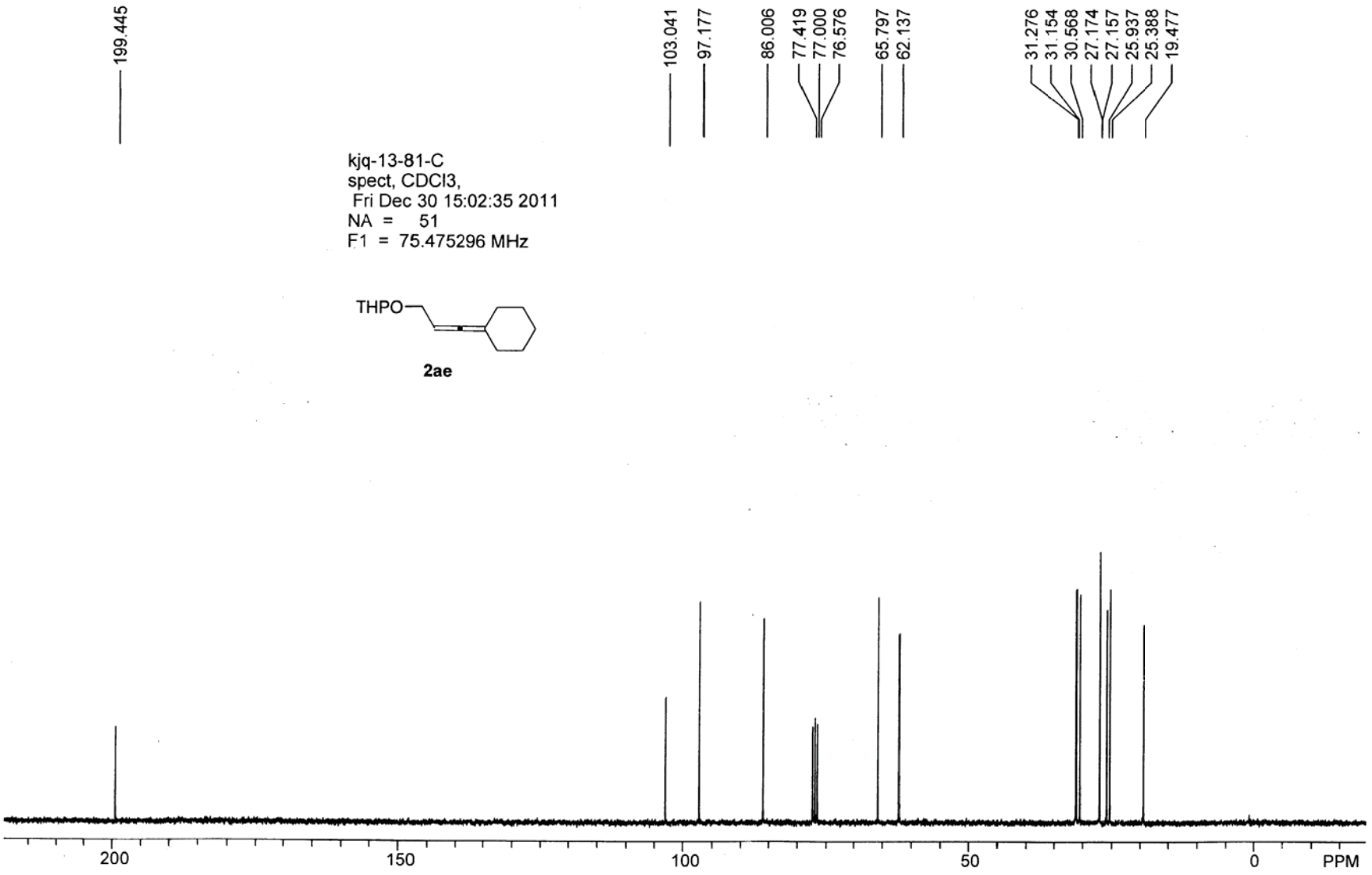

7.260
5.111
5.104
5.097
5.090
5.083
5.076
5.069
5.062
5.055
4.154
4.133
2.128
2.113
2.097
2.090
1.616
1.598
1.592
1.580
1.571
1.559
1.542
1.533
1.522
1.508
1.497
1.480
0.903
0.077
kjq-13-69-H
spect, CDCl3,
Mon Dec 26 14:07:27 2011
NA = 8
F1 = 300.131866 MHz
Si
O
2ad
0.95
2.01
4.00
6.27
9.84
6.45
8
6
4
2
0
PPM

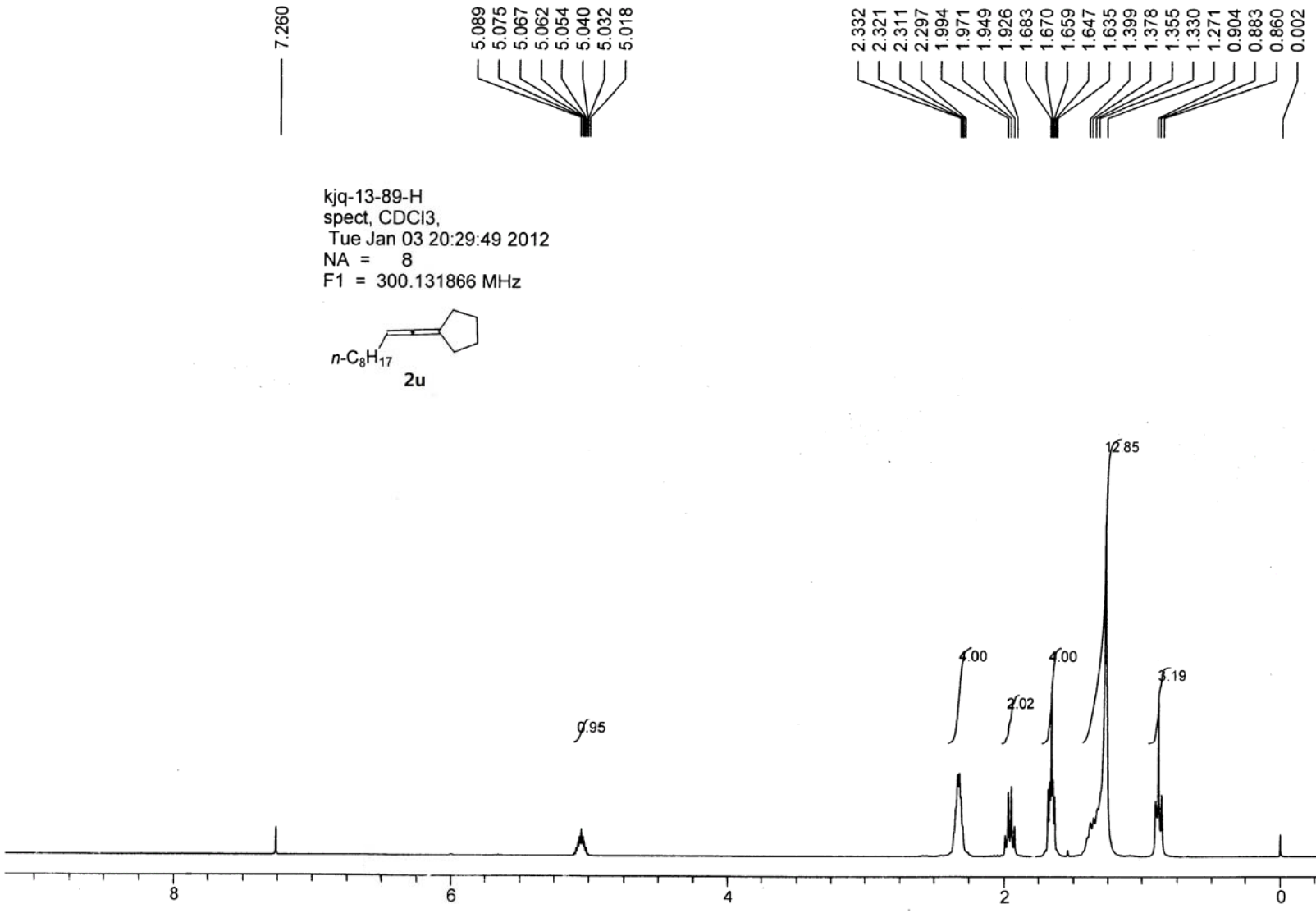

197.864
103.581
89.580
77.423
77.000
76.576
62.715
31.318
27.299
26.072
25.965
18.388
-5.012
kjq-13-69-C
spect, CDCl3,
Mon Dec 26 15:29:33 2011
NA = 300
F1 = 75.475296 MHz
Si
O
2ad
200
150
100
50
0
PPM

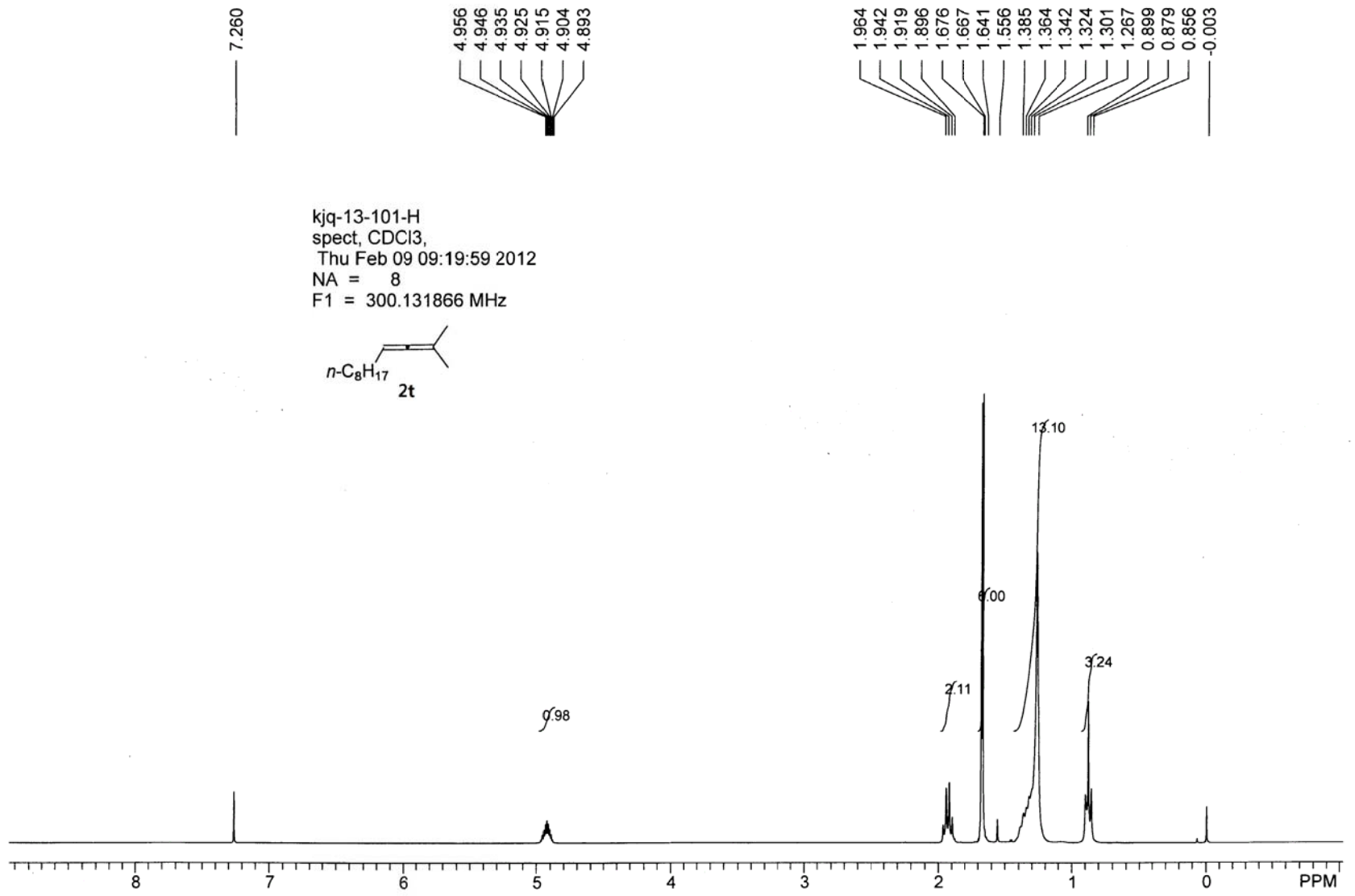


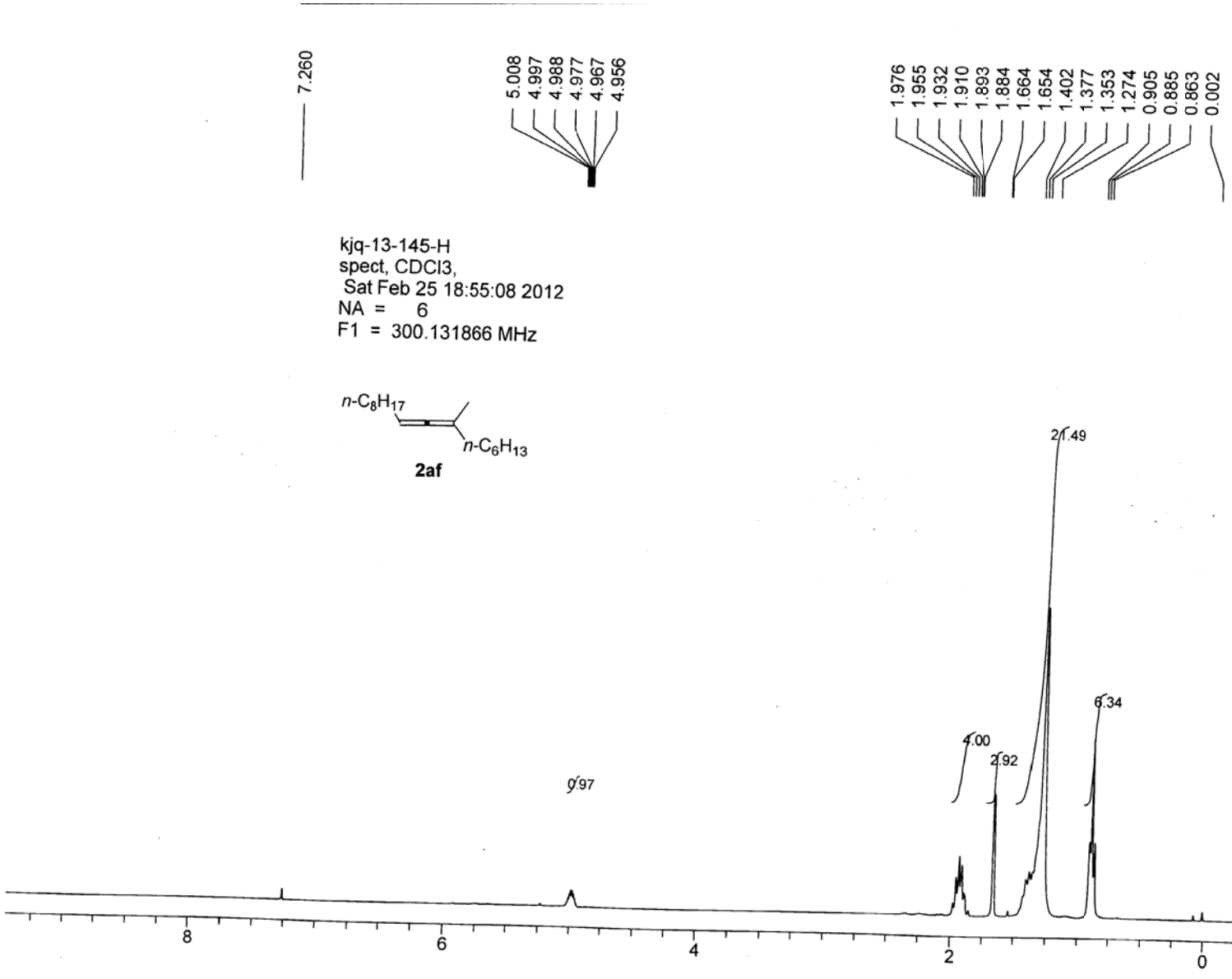
kjq-13-81-H
spect, CDCl3,
Fri Dec 30 14:14:18 2011
NA = 8
F1 = 300.131866 MHz

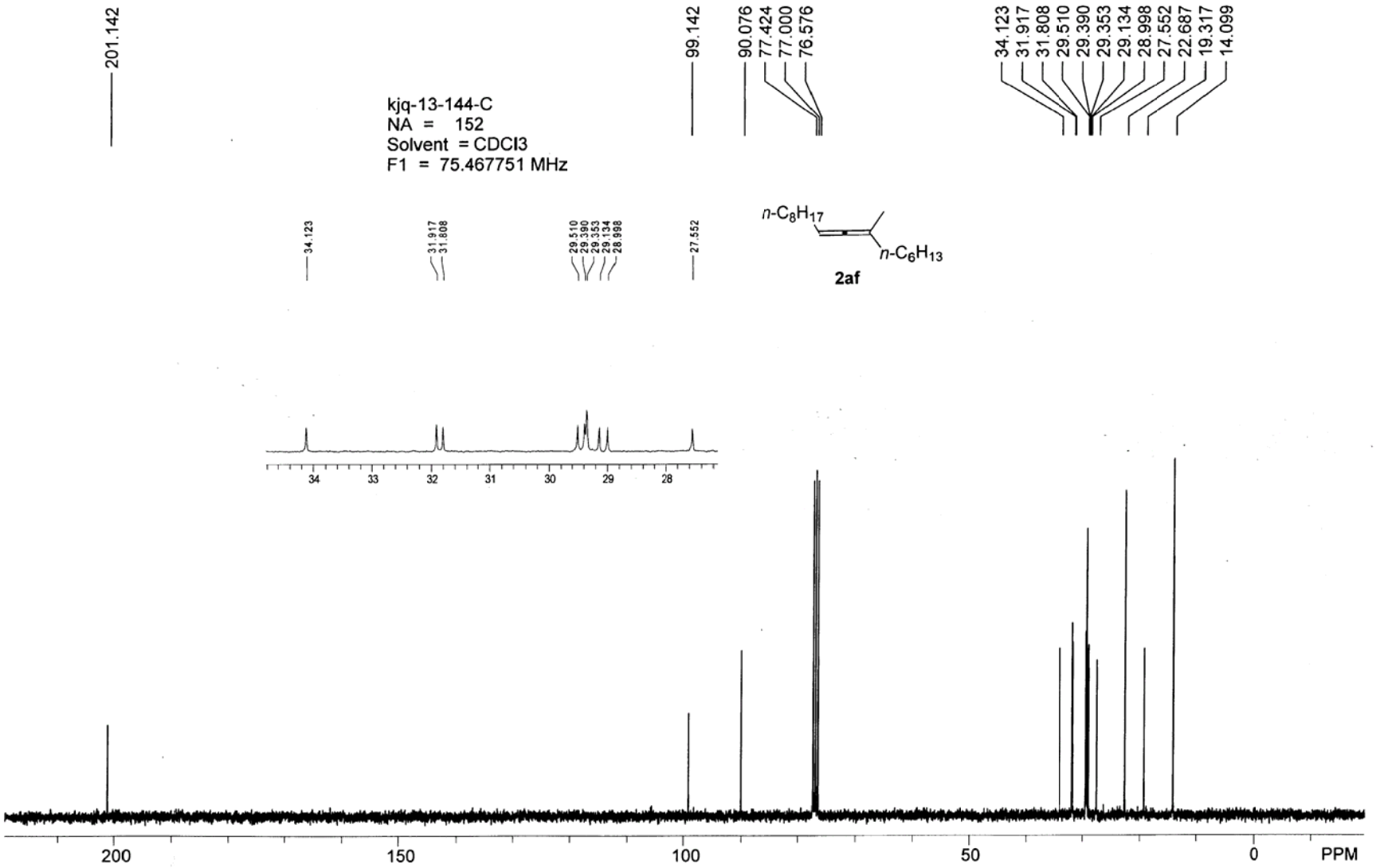
2ae

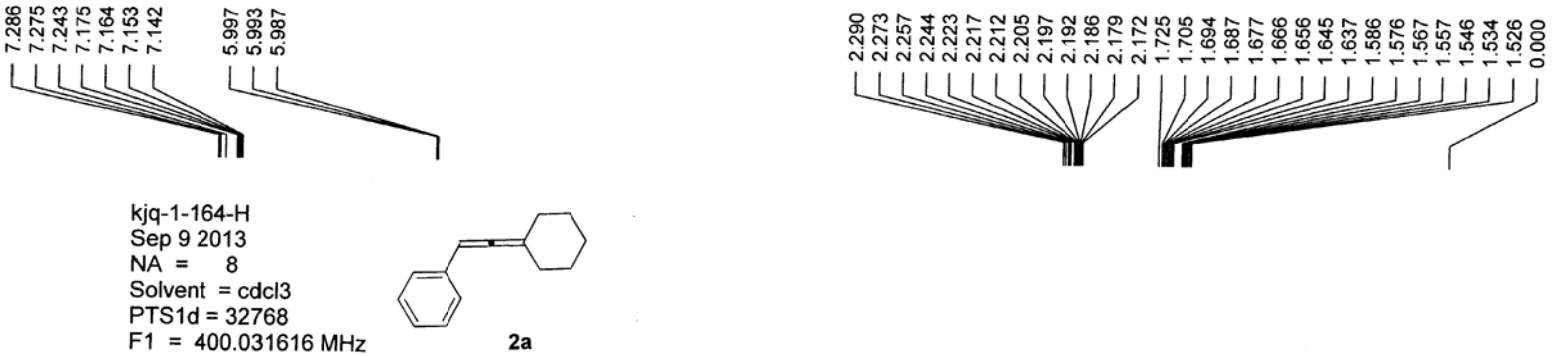

199.445
103.041
97.177
86.006
77.419
77.000
76.576
65.797
62.137
31.276
31.154
30.568
27.174
27.157
25.937
25.388
19.477
kjq-13-81-C
spect, CDCl3,
Fri Dec 30 15:02:35 2011
NA = 51
F1 = 75.475296 MHz
THPO
2ae
200
150
100
50
0
PPM

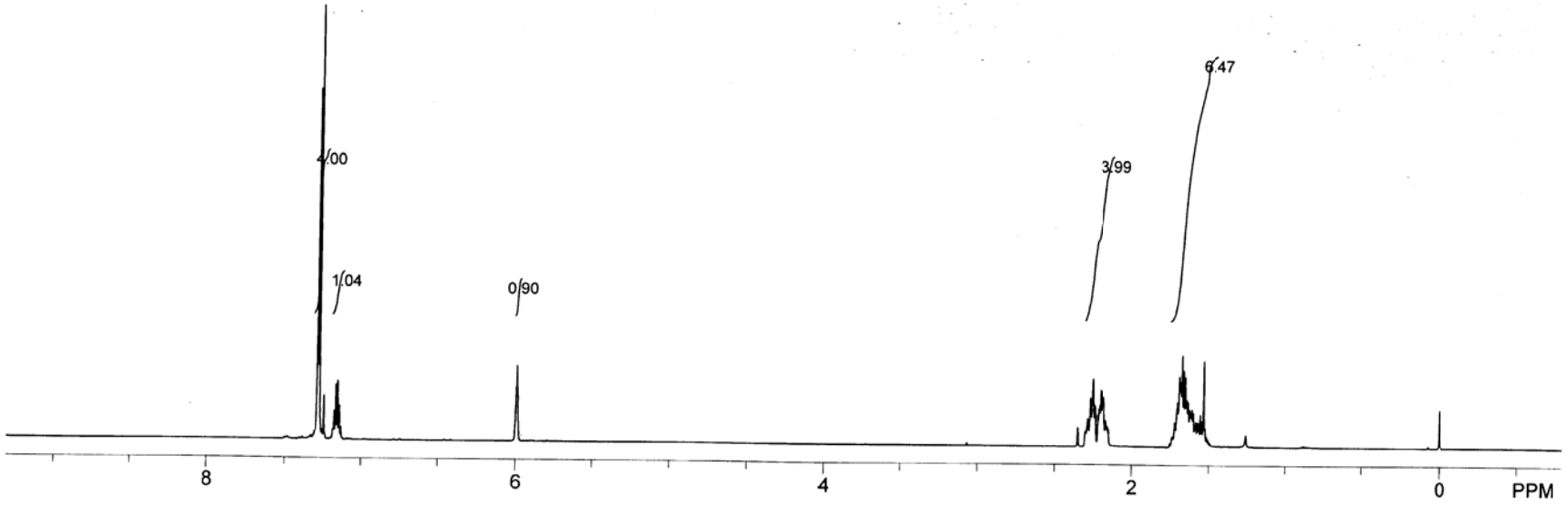

7.260
5.089
5.075
5.067
5.062
5.054
5.040
5.032
5.018
2.332
2.321
2.311
2.297
1.994
1.971
1.949
1.926
1.683
1.670
1.659
1.647
1.635
1.399
1.378
1.355
1.330
1.271
0.904
0.883
0.860
0.002
kjq-13-89-H
spect, CDCl3,
Tue Jan 03 20:29:49 2012
NA = 8
F1 = 300.131866 MHz
n-C8H17
2u
0.95
4.00
2.02
4.00
12.85
3.19
8
6
4
2
0
PPM


7.260
4.956
4.946
4.935
4.925
4.915
4.904
4.893
1.964
1.942
1.919
1.896
1.676
1.667
1.641
1.556
1.385
1.364
1.342
1.324
1.301
1.267
0.899
0.879
0.856
-0.003
kjq-13-101-H
spect, CDCl3,
Thu Feb 09 09:19:59 2012
NA = 8
F1 = 300.131866 MHz
n-C8H17
2t
0.98
2.11
6.00
13.10
3.24
8
7
6
5
4
3
2
1
0
PPM


7.260
5.008
4.997
4.988
4.977
4.967
4.956
1.976
1.955
1.932
1.910
1.893
1.884
1.664
1.654
1.402
1.377
1.353
1.274
0.905
0.885
0.863
0.002
kjq-13-145-H
spect, CDCl3,
Sat Feb 25 18:55:08 2012
NA = 6
F1 = 300.131866 MHz
n-C8H17
n-C6H13
2af
21.49
6.34
4.00
2.92
0.97
8
6
4
2
0
PPM


201.142
99.142
90.076
77.424
77.000
76.576
34.123
31.917
31.808
29.510
29.390
29.353
29.134
28.998
27.552
22.687
19.317
14.099
kjq-13-144-C
NA = 152
Solvent = CDCl3
F1 = 75.467751 MHz
n-C8H17
n-C6H13
2af
200
150
100
50
0
PPM


7.286
7.275
7.243
7.175
7.164
7.153
7.142
5.997
5.993
5.987
2.290
2.273
2.257
2.244
2.223
2.217
2.212
2.205
2.197
2.192
2.186
2.179
2.172
1.725
1.705
1.694
1.687
1.677
1.666
1.656
1.645
1.637
1.586
1.576
1.567
1.557
1.546
1.534
1.526
0.000
kjq-1-164-H
Sep 9 2013
NA = 8
Solvent = cdcl3
PTS1d = 32768
F1 = 400.031616 MHz
2a


4.00
1.04
0.90
3.99
6.47
8
6
4
2
0
PPM