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Synthesis of 2-Substituted Indoles through Cyclization and Demethylation of 2-Alkynyldimethylanilines by Ethanol

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Table of Contents

Experimental procedure for quantification of residual traces of Pd and Cu

Quantification of residual traces of Pd and Cu in S1, S2 and S3 by atomic absorption after mineralization of each samples

The samples S1, S2 and S3 are weighed and solubilized in 4 mL of hydrochloric acid and 4 mL of nitric acid of chromatographic purity. The mineralization takes place in an ANTON PAAR microwave. Cu and Pd residues contained in the 3 samples are dosed by ICP-OES 5100 Agilent. A first analysis was carried out with the PEPure VIII standards from 0.5 ppm to 5 ppm for the determination of Cu and the standard PEMECS4 from 0.2 ppm to 2 ppm for the determination of Pd. Since the values found are inferior to 0.5 ppm for the 3 samples for Cu and Pd, a second analysis was carried out in a lower range of concentrations, from 5 ppb to 50 ppb.

Samples	Cu (initial concentration) ppb	Pd (initial concentration) ppb
S1	0	7
S2	0	3
S3	0	1
Blank	0	0

The results obtained allow us to conclude that the concentration of S1 is inferior to 5 ppb in Cu and less than 10 ppb in Pd and that the concentrations of the samples S2 and S3 are inferior to 5 ppb in Cu and Pd.

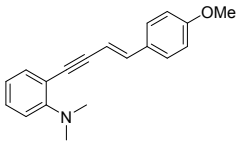
Characterization data for new compounds and synthetic procedures

All glasswares were oven-dried at 140 °C and all reactions were conducted under an argon atmosphere. Solvents: cyclohexane, ethyl acetate (AcOEt), for chromatography, were technical grade. The ^{1}H NMR and ^{13}C NMR and ^{19}F spectra were recorded in $CDCl_3$ on Bruker Avance 300 spectrometer. The chemical shifts of ^{1}H are reported in ppm relative to the solvent residual peak in $CDCl_3$ (δ 7.26) for ^{1}H NMR. For the ^{13}C NMR spectra, the solvent signal of $CDCl_3$ (δ 77.14) was used as the internal standards. The following abreviation are used: m (multiplet), s (singlet), d (doublet), t (triplet), dd (doublet of doublet), td (triplet of doublet), ddd (doublet of doublet of doublet). IR spectra were measured on a Bruker Vector 22 spectrophotometer (neat, cm^{-1}). High-resolution mass spectra were recorded on a Bruker Daltonics micrOTOF-Q instrument. Analytical TLC was performed on Merck precoated silica gel 60 F-254 plates. Merck silica gel 60 (230-400 mesh) was used for column chromatography. The plates were visualized by either UV light (254 nm).

Procedure for the synthesis of diarylenynes 1a, 1c.

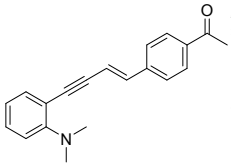
Under argon, chloroenyne **1b** (1.03 g, 5 mmol), the desired boronic acid (1.3 equiv), K_2CO_3 (1.38 g, 2 equiv), and $Pd(PPh_3)_4$ (462.2 mg, 8 mol%) were mixed in toluene (12 mL) and MeOH (6 mL). The reaction mixture was stirred at 90°C in 30 mL sealed tube monitored by TLC (7 h). After the mixture were allowed to cool to room temperature and filtrated through a pad of celite washed by CH_2Cl_2. Solvent was removed under reduced pressure and the crude material was purified by silica gel column chromatography (0 to 30% AcOEt in cyclohexane).

(*E*)-2-(4-(4-Methoxyphenyl)but-3-en-1-yn-1-yl)-*N*,*N*-dimethylaniline (**1a**)



Rf = 0.57 (cyclohexane/AcOEt 10:3), yellow solid, m.p. 54.8 – 55.0 °C, yield 75%, 1.04 g.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.47 (d, *J* = 7.5 Hz, 1H), 7.40 (d, *J* = 8.4 Hz, 2H), 7.26 (t, *J* = 8.4 Hz, 1H), 7.09 – 6.83 (m, 5H), 6.36 (d, *J* = 16.2 Hz, 1H), 3.85 (s, 3H), 3.01 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 160.1 (Cq), 154.6 (Cq), 140.0 (CH), 134.3 (CH), 129.5 (Cq), 127.7 (2 CH), 120.7 (CH), 117.1 (CH), 115.8 (Cq), 114.3 (2 CH), 106.5 (CH), 94.7 (Cq), 90.5 (Cq), 55.4 (CH_3), 43.6 (2 CH_3). IR (neat): 2834, 1603, 1510, 1253 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{19}H_{20}NO$: 278.1545; found: 278.1541.

(*E*)-1-(4-(4-(2-(Dimethylamino)phenyl)but-1-en-3-yn-1-yl)phenyl)ethan-1-one (**1c**)

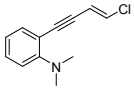


Rf = 0.43 (cyclohexane/AcOEt 10:3), yellow solid, m.p. 123.5 – 123.7 °C, yield 73%, 1.05 g.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.84 (d, *J* = 8.4 Hz, 2H), 7.42 – 7.33 (m, 3H), 7.16 (t, *J* = 7.5 Hz, 1H), 6.94 (d, *J* = 16.2 Hz, 1H), 6.88 – 6.73 (m, 2H), 6.49 (d, *J* = 16.2 Hz, 1H), 2.89 (s, 6H), 2.50 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 197.3 (Cq), 154.9 (Cq), 141.1 (Cq), 138.9 (CH), 136.7 (Cq), 134.5 (CH), 129.6 (CH), 128.9 (2 CH), 126.3 (2 CH), 120.6 (CH), 117.1 (CH), 115.0 (Cq), 111.8 (CH), 93.9 (Cq), 93.2 (Cq), 43.6 (2 CH_3), 26.6 (CH_3). IR (neat): 2833, 2186, 1678, 1598, 1267 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{20}H_{20}NO$: 290.1545; found: 290.1546.

Procedure for the synthesis of (*E*)-chloroenynes 1b, 1d.

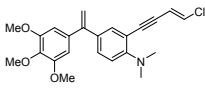
To a solution of 2-ethynyl-*N*,*N*-dimethylaniline (5 mmol) in Et_2O (30 mL) was added successively (*E*)-1,2-dichloroethylene (4 mL, 50 mmol), $PdCl_2(PPh_3)_2$ (175 mg, 0.25 mmol), piperidine (1 mL, 10 mmol) and CuI (95 mg, 0.5 mmol). After complete disappearance of starting material monitored by TLC, the solution was filtered through a pad of celite using AcOEt. The organic layer was washed successively with sat. NH_4Cl, sat. $NaHCO_3$ and HCl (1 M) solutions. After drying over $MgSO_4$ and evaporation *in vacuo*, the crude residue was purified by silica gel column chromatography (0 to 30% AcOEt in cyclohexane).

(*E*)-2-(4-Chlorobut-3-en-1-yn-1-yl)-*N*,*N*-dimethylaniline (**1b**)[1]



Rf = 0.86 (cyclohexane/AcOEt 10:3), pale brown solid, m.p. 36.8 – 37.0 °C, yield 78%, 802 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.52 (dd, *J* = 7.5, 1.2 Hz, 1H), 7.28 (td, *J* = 7.5, 1.2 Hz, 1H), 6.97 – 6.86 (m, 2H), 6.63 (d, *J* = 13.5 Hz, 1H), 6.25 (d, *J* = 13.5 Hz, 1H), 2.95 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 154.9 (Cq), 134.3 (CH), 129.7 (CH), 129.2 (CH), 120.7 (CH), 117.1 (CH), 114.6 (Cq), 114.4 (CH), 91.5 (Cq), 89.7 (Cq), 43.6 (2 CH_3). IR (neat): 2945, 2197, 1595, 1490, 1325 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{12}H_{13}N^{35}Cl$: 206.0737; found: 206.0735.

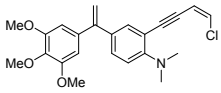
(*E*)-2-(4-Chlorobut-3-en-1-yn-1-yl)-*N*,*N*-dimethyl-4-(1-(3,4,5-trimethoxyphenyl)vinyl)aniline (1**d**)[1]



Rf = 0.60 (cyclohexane/AcOEt 5:5), brown oil, yield 78%, 1.55 g.
^{1}H NMR (300 MHz, Acetone) δ = 7.36 (d, *J* = 2.1 Hz, 1H), 7.26 (dd, *J* = 8.7, Hz, 1H), 6.91 (d, *J* = 8.7 Hz, 1H), 6.82 (d, *J* = 13.5 Hz, 1H), 6.62 (s, 2H), 6.33 (d, *J* = 13.5 Hz, 1H), 5.38 (s, 1H), 5.35 (s, 1H), 3.79 (s, 6H), 3.77 (s, 3H), 2.97 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 159.1 (Cq), 157.9 (2 Cq), 153.1 (Cq), 142.6 (Cq), 141.7 (Cq), 138.3 (CH), 137.1 (Cq), 135.2 (CH), 134.6 (CH), 121.9 (CH), 119.4 (CH), 118.3 (CH_2), 117.3 (Cq), 110.8 (2 CH), 96.7 (Cq), 94.8 (Cq), 65.2 (CH_3), 61.1 (2 CH_3), 47.9 (2 CH_3). IR (neat): 2937, 2196, 1579, 1502, 1347, 1235 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{23}H_{25}NO_3{}^{35}Cl$: 398.1523; found: 398.1523.

Procedure for the synthesis of (*Z*)-chloroenynes 1e.

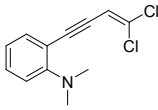
To a solution of 2-ethynyl-*N*,*N*-dimethylaniline (1.69 g, 5 mmol) in Et_2O (30 mL) was added successively (*Z*)-1,2-dichloroethylene (4 mL, 50 mmol), $PdCl_2(PPh_3)_2$ (175 mg, 0.25 mmol), *n*-$BuNH_2$ (1 mL, 10 mmol) and CuI (95 mg, 0.5 mmol). After complete disappearance of starting material monitored by TLC, the solution was filtered through a pad of celite using AcOEt. The organic layer was washed successively with sat. NH_4Cl, sat. $NaHCO_3$ and HCl (1 M) solutions. After drying over $MgSO_4$ and evaporation *in vacuo*, the crude residue was purified by silica gel column chromatography (0 to 30% AcOEt in cyclohexane).
(*Z*)-2-(4-Chlorobut-3-en-1-yn-1-yl)-*N*,*N*-dimethyl-4-(1-(3,4,5-trimethoxyphenyl)vinyl)aniline (**1e**)



Rf = 0.40 (cyclohexane/AcOEt 5:5), brown oil, yield 69%, 1.37 g.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.47 (d, *J* = 2.1 Hz, 1H), 7.22 (dd, *J* = 8.7, 2.4 Hz, 1H), 6.84 (d, *J* = 8.7 Hz, 1H), 6.55 (s, 2H), 6.39 (d, *J* = 7.5 Hz, 1H), 6.13 (d, *J* = 7.5 Hz, 1H), 5.37 (s, 1H), 5.31 (s, 1H), 3.88 (s, 3H), 3.82 (s, 6H), 3.01 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 154.3 (Cq), 153.0 (2 Cq), 149.0 (Cq), 137.9 (Cq), 137.3 (Cq), 134.4 (CH), 133.0 (Cq), 129.9 (CH), 127.4 (CH), 116.5 (CH), 113.6 (Cq), 112.8 (CH_2), 112.6 (CH), 105.7 (2 CH), 96.9 (Cq), 88.7 (Cq), 61.0 (CH_3), 56.3 (2 CH_3), 43.6 (2 CH_3). IR (neat): 2987, 1684, 1579, 1542, 1470, 1376 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{23}H_{25}NO_3{}^{35}Cl$: 398.1523; found: 398.1529.

Procedure for the synthesis of dichloroenyne 1f

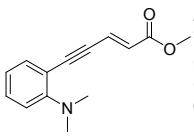
Triethylamine (10 mL) was added to a stirred mixture of 2-ethynyl-*N*,*N*-dimethylaniline (726 mg, 5 mmol), 2-bromo-1,1-dichloroethylene (1.41 g, 8 mmol), $PdCl_2(PPh_3)_2$ (14.1 mg, 0.02mmol), CuI (6.7 mg, 0.035 mmol) and PPh_3 (13.1 mg, 0.05 mmol) under argon atmosphere. The mixture was heated under reflux for 4 h. The precipitated ammonium salt was filtered off, the solvent was evaporated and water (20 mL) was added to the residue. The mixture was extracted with Et_2O (3 x 20 mL), dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (0 to 30% AcOEt in cyclohexane).
2-(4,4-Dichlorobut-3-en-1-yn-1-yl)-*N*,*N*-dimethylaniline (**1f**)



Rf = 0.65 (cyclohexane/AcOEt 10:3), yellow oil, yield 45%, 540.3 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.32 (d, *J* = 7.5 Hz, 1H), 7.16 (td, *J* = 7.8 Hz, 1.5 Hz, 1H), 6.87 – 6.69 (m, 2H), 6.14 (s, 1H), 2.86 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 154.8 (Cq), 134.7 (CH), 130.3 (Cq), 130.1 (CH), 120.5 (CH), 117.1 (CH), 114.1 (Cq), 111.6 (CH), 97.4 (Cq), 88.5 (Cq), 43.7 (2 CH_3). IR (neat): 2786, 2194, 1596, 1491, 1429, 1330 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{12}H_{12}N^{35}Cl_2$: 240.0347; found: 240.0342.

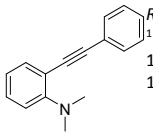
General procedure for the synthesis of functionalized alkynes 1g, 1h, 1i, 1j, 1k, 1l , 1n, 1o, 1p, 1q, 1s, 1t, 1u, 1v, 3.

To a stirred mixture of iodide compound (5 mmol), $PdCl_2(PPh_3)_2$ (0.05 equiv) and CuI (0.1 equiv) in Et_3N (10 mL), terminal alkyne (1.1 equiv) was added dropwise at 0 °C. The mixture was then stirred at room temperature. When the starting material was disappeared (TLC monitoring), the mixture was passed through a pad of Celite to remove the insoluble materials. The filtrate was concentrated in vacuo and the residue was purified by silica gel column chromatography (0 to 10% AcOEt in cyclohexane).
Methyl (*E*)-5-(2-(dimethylamino)phenyl)pent-2-en-4-ynoate (**1g**)



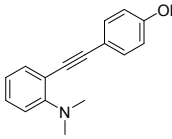
Rf = 0.38 (cyclohexane/AcOEt 10:1), brown oil, yield 73%, 836.9 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.43 (d, *J* = 7.5 Hz, 1H), 7.28 (t, *J* = 7.8 Hz, 1H), 7.08 (d, *J* = 15.9 Hz, 1H), 6.99 – 6.79 (m, 2H), 6.31 (d, *J* = 15.9 Hz, 1H), 3.80 (s, 3H), 2.98 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 166.6 (Cq), 155.3 (Cq), 134.9 (CH), 130.5 (CH), 128.3 (CH), 125.9 (CH), 120.3 (CH), 117.0 (CH), 113.5 (Cq), 98.6 (Cq), 91.8 (Cq), 51.8 (CH_3), 43.5 (2 CH_3). IR (neat): 2788, 2188, 1719, 1615, 1491, 1431, 1318, 1159 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{14}H_{16}NO_2$: 230.1181; found: 230.1177.

N,*N*-Dimethyl-2-(phenylethynyl)aniline (**1h**)[2]



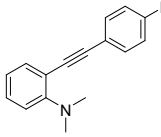
Rf = 0.66 (cyclohexane/AcOEt 10:1), yellow solid, m.p. 52.0 – 52.2 °C, yield 81%, 896.3 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.64 – 7.50 (m, 3H), 7.42 – 7.26 (m, 4H), 7.00 – 6.91 (m, 2H), 3.06 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 154.7 (Cq), 134.5 (CH), 131.4 (2 CH), 129.3 (CH), 128.4 (2 CH), 128.1 (CH), 124.0 (Cq), 120.6 (CH), 117.1 (CH), 115.2 (Cq), 94.8 (Cq), 89.0 (Cq), 43.6 (2 CH_3). IR (neat): 2783, 2211, 1591, 1497, 1329 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{16}H_{16}N$: 222.1283; found: 222.1278.

2-((4-Methoxyphenyl)ethynyl)-*N*,*N*-dimethylaniline (**1i**)[2]



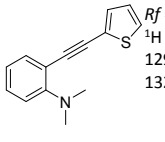
Rf = 0.43 (cyclohexane/AcOEt 10:1), yellow solid, m.p. 65.5 – 65.7 °C, yield 85%, 1.07 g.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.58 – 7.43 (m, 3H), 7.29 – 7.23 (m, 1H), 6.97 – 6.88 (m, 4H), 3.85 (s, 3H), 3.02 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 159.6 (Cq), 154.7 (Cq), 134.3 (CH), 132.8 (2 CH), 129.0 (CH), 120.6 (CH), 117.1 (CH), 116.2 (Cq), 115.7 (Cq), 114.1 (2 CH), 94.9 (Cq), 87.6 (Cq), 55.4 (CH_3), 43.6 (2 CH_3). IR (neat): 2783, 1606, 1510, 1287, 1246 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{17}H_{18}NO$: 252.1388; found: 252.1382.

2-((4-Fluorophenyl)ethynyl)-*N*,*N*-dimethylaniline (**1j**)



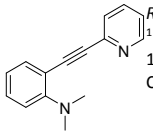
Rf = 0.56 (cyclohexane/AcOEt 10:1), yellow solid, m.p. 40.5 – 40.7 °C, yield 80%, 957.2 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.62 – 7.45 (m, 3H), 7.29 (t, *J* = 7.8 Hz, 1H), 7.07 (t, *J* = 8.4 Hz, 2H), 6.98 – 6.90 (m, 2H), 3.04 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 162.4 (d, *J* = 247.6 Hz, Cq), 154.7 (Cq), 134.3 (CH), 133.1 (d, *J* = 8.3 Hz, 2 CH), 129.3 (CH), 120.5 (CH), 120.1 (d, *J* = 3.3 Hz, Cq), 116.9 (CH), 115.6 (d, *J* = 21.9 Hz, 2 CH), 114.9 (Cq), 93.5 (Cq), 88.6 (Cq), 43.5 (2 CH_3). ^{19}F (188 MHz, $CDCl_3$): -111.394. IR (neat): 2785, 2214, 1590, 1329, 1231 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{16}H_{15}NF$: 240.1189; found: 240.1180.

N,*N*-Dimethyl-2-(thiophen-2-ylethynyl)aniline (**1k**)



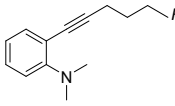
Rf = 0.55 (cyclohexane/AcOEt 10:1), brown oil, yield 82%, 932.0 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.64 – 7.50 (m, 2H), 7.37 – 7.25 (m, 3H), 7.04 – 6.92 (m, 2H), 3.06 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 154.7 (Cq), 134.2 (CH), 129.7 (CH), 129.2 (CH), 127.9 (CH), 125.3 (CH), 122.9 (Cq), 120.5 (CH), 117.0 (CH), 115.1 (Cq), 89.9 (Cq), 88.4 (Cq), 43.5 (2 CH_3). IR (neat): 2782, 1592, 1488, 1326, 1194 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{14}H_{14}NS$: 228.0847; found: 228.0843.

N,*N*-Dimethyl-2-(pyridin-2-ylethynyl)aniline (**1l**)



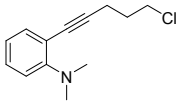
Rf = 0.12 (cyclohexane/AcOEt 10:1), brown oil, yield 65%, 722.5 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 8.63 (d, *J* = 4.8 Hz, 1H), 7.70 – 7.50 (m, 3H), 7.31 – 7.18 (m, 2H), 6.96 – 6.86 (m, 2H), 3.04 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 155.3 (Cq), 150.1 (CH), 144.1 (Cq), 136.1 (CH), 134.9 (CH), 130.0 (CH), 126.9 (CH), 122.4 (CH), 120.3 (CH), 116.9 (CH), 113.8 (Cq), 94.0 (Cq), 89.2 (Cq), 43.6 (2 CH_3). IR (neat): 2786, 2213, 1580, 1493, 1461, 1426, 1322 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{15}H_{15}N_2$: 223.1235; found: 223.1232.

2-(Hex-1-yn-1-yl)-*N*,*N*-dimethylaniline (**1n**)[3]



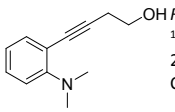
Rf = 0.64 (cyclohexane/AcOEt 10:1), colorless oil, yield 83%, 835.5 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.37 (dd, *J* = 7.5, 1.2 Hz, 1H), 7.19 (td, *J* = 7.5, 1.2 Hz, 1H), 6.93 – 6.81 (m, 2H), 2.92 (s, 6H), 2.49 (t, *J* = 7.2 Hz, 2H), 1.69 – 1.44 (m, 4H), 0.96 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 154.7 (Cq), 134.4 (CH), 128.4 (CH), 120.8 (CH), 117.1 (CH), 116.7 (Cq), 95.9 (Cq), 79.7 (Cq), 43.7 (2 CH_3), 31.0 (CH_2), 22.2 (CH_2), 19.7 (CH_2), 13.8 (CH_3). IR (neat): 2932, 2360, 1593, 1492, 1429, 1328 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{14}H_{20}N$: 202.1596; found: 202.1590.

2-(5-Chloropent-1-yn-1-yl)-*N*,*N*-dimethylaniline (**1o**)[4]



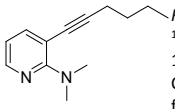
Rf = 0.58 (cyclohexane/AcOEt 10:1), colorless oil, yield 86%, 953.4 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.39 (d, *J* = 7.5 Hz, 1H), 7.24 (t, *J* = 7.2 Hz, 1H), 6.95 – 6.86 (m, 2H), 3.76 (t, *J* = 6.3 Hz, 2H), 2.94 (s, 6H), 2.71 (t, *J* = 6.4 Hz, 2H), 2.16 – 2.06 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 154.8 (Cq), 134.4 (CH), 128.8 (CH), 120.8 (CH), 117.1 (CH), 116.1 (Cq), 93.4 (Cq), 80.8 (Cq), 43.9 (CH_2), 43.7 (2 CH_3), 31.7 (CH_2), 17.5 (CH_2). IR (neat): 2782, 1592, 1492, 1429, 1355 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{13}H_{17}N^{35}Cl$: 222.1050; found: 222.1042.

4-(2-(Dimethylamino)phenyl)but-3-yn-1-ol (**1p**)



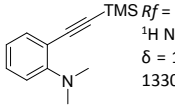
Rf = 0.10 (cyclohexane/AcOEt 10:3), yellow oil, yield 85%, 804.3 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.39 (d, *J* = 7.5 Hz, 1H), 7.25 (t, *J* = 7.8 Hz, 1H), 6.99 – 6.87 (m, 2H), 3.84 (t, *J* = 6.3 Hz, 2H), 2.92 (s, 6H), 2.76 (t, *J* = 6.3 Hz, 2H), 2.56 (br, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 154.9 (Cq), 134.1 (CH), 128.9 (CH), 121.4 (CH), 117.5 (CH), 116.4 (Cq), 92.1 (Cq), 81.7 (Cq), 61.2 (CH_2), 43.9 (2 CH_3), 24.4 (CH_2). IR (neat): 2952, 2363, 1593, 1492, 1450, 1318 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{12}H_{16}NO$: 190.1232; found: 190.1230.

3-(Hex-1-yn-1-yl)-*N*,*N*-dimethylpyridin-2-amine (**1q**)



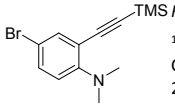
Rf = 0.46 (cyclohexane/AcOEt 10:1), yellow oil, yield 71%, 718.2 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 8.09 (dd, *J* = 4.8 Hz, 1.5 Hz, 1H), 7.53 (dd, *J* = 7.5, 1.5 Hz, 1H), 6.64 – 6.59 (m, 1H), 3.13 (s, 6H), 2.45 (t, *J* = 7.2 Hz, 2H), 1.65 – 1.43 (m, 4H), 0.94 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 161.9 (Cq), 146.3 (CH), 142.7 (CH), 113.9 (CH), 107.7 (Cq), 96.9 (Cq), 78.7 (Cq), 41.1 (2 CH_3), 30.9 (CH_2), 22.2 (CH_2), 19.6 (CH_2), 13.7 (CH_3). IR (neat): 2932, 1579, 1492, 1401, 1233 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{13}H_{19}N_2$: 203.1548; found: 203.1547.

N,*N*-Dimethyl-2-((trimethylsilyl)ethynyl)aniline (**1s**)[2]



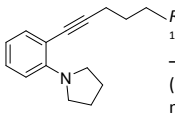
Rf = 0.71 (cyclohexane/AcOEt 10:1), brown oil, yield 85%, 923.9 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.22 (dd, *J* = 7.5, 1.5 Hz, 1H), 7.01 (td, *J* = 7.5, 1.5 Hz,, 1H), 6.68 – 6.59 (m, 2H), 2.76 (s, 6H), 0.07 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 155.2 (Cq), 134.9 (CH), 129.5 (CH), 120.1 (CH), 116.8 (CH), 114.7 (Cq), 104.8 (Cq), 99.6 (Cq), 43.4 (2 CH_3), 0.1 (3 CH_3). IR (neat): 2958, 2149, 1594, 1492, 1330, 1248 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{13}H_{20}NSi$: 218.1365; found: 218.1362.

4-Bromo-*N*,*N*-dimethyl-2-((trimethylsilyl)ethynyl) aniline (**1t**)[1]



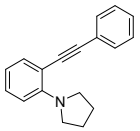
Rf = 0.80 (cyclohexane/AcOEt 10:1), colorless oil, yield 84%, 1.25 g.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.29 (d, *J* = 2.4 Hz, 1H), 7.06 (dd, *J* = 9.0, 2.5 Hz, 1H), 6.48 (d, *J* = 9.0 Hz, 1H), 2.70 (s, 6H), 0.03 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 154.2 (Cq), 137.0 (CH), 132.3 (CH), 118.3 (CH), 116.4 (Cq), 111.8 (Cq), 103.2 (Cq), 101.2 (Cq), 43.3 (2 CH_3), 0.0 (3 CH_3). IR (neat): 2958, 2838, 2151, 1488, 1386, 838 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{13}H_{19}N^{79}BrSi$: 296.0470; found: 296.0469.

1-(2-(Hex-1-yn-1-yl)phenyl)pyrrolidine (**1u**)



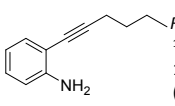
Rf = 0.77 (cyclohexane/AcOEt 10:1), brown oil, yield 83%, 943.5 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.23 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.02 (td, *J* = 7.8, 1.5 Hz, 1H), 6.59 - 6.53 (m, 2H), 3.45 – 3.40 (m, 4H), 2.33 (t, *J* = 6.9 Hz, 2H), 1.92 – 1.77 (m, 4H), 1.55 – 1.32 (m, 4H), 0.85 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 150.5 (Cq), 135.2 (CH), 128.4 (CH), 117.4 (CH), 114.1 (CH), 110.7 (Cq), 93.6 (Cq), 81.3 (Cq), 50.4 (2 CH_2), 30.9 (CH_2), 25.6 (2 CH_2), 22.2 (CH_2), 19.6 (CH_2), 13.7 (CH_3). IR (neat): 2957, 1593, 1481, 1441, 1356 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{16}H_{22}N$: 228.1752; found: 228.1748.

1-(2-(Phenylethynyl)phenyl)pyrrolidine (**1v**)



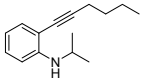
Rf = 0.64 (cyclohexane/AcOEt 10:1), colorless oil, yield 87%, 1.08 g.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.66 – 7.45 (m, 3H), 7.43 – 7.35 (m, 3H), 7.25 (td, *J* = 7.5, 1.0 Hz, 1H), 6.76 (t, *J* = 8.4 Hz, 2H), 3.73 – 3.68 (m, 4H), 2.07 – 1.99 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 150.4 (Cq), 135.3 (CH), 130.8 (2 CH), 129.4 (CH), 128.4 (2 CH), 127.7 (CH), 124.4 (Cq), 116.9 (CH), 114.0 (CH), 108.8 (Cq), 92.4 (Cq), 91.2 (Cq), 50.5 (2 CH_2), 25.8 (2 CH_2). IR (neat): 3059, 2870, 2205, 1592, 1497, 1477, 1441, 1355 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{18}H_{18}N$: 248.1439; found: 248.1430.

2-(Hex-1-yn-1-yl)aniline (**3**)



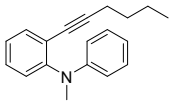
Rf = 0.64 (cyclohexane/AcOEt 10:3), brown oil, yield 80%, 693.0 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.31 (dd, *J* = 7.5, 1.5 Hz, 1H), 7.12 (td, *J* = 7.5, 1.5 Hz, 1H), 6.81 – 6.63 (m, 2H), 4.21 (br, 2H), 2.53 (t, *J* = 7.2 Hz, 2H), 1.72 – 1.49 (m, 4H), 1.02 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 147.7 (Cq), 132.0 (CH), 128.8 (CH), 117.8 (CH), 114.2 (CH), 109.0 (Cq), 95.7 (Cq), 77.14, (Cq), 31.1 (CH_2), 22.1 (CH_2), 19.4 (CH_2), 13.7 (CH_3). IR (neat): 3478, 2958, 2360, 1613, 1493, 1455, 1316 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{12}H_{16}N$: 174.1283; found: 174.1281.

2-(hex-1-yn-1-yl)-*N*-isopropylaniline



Rf = 0.33 (cyclohexane/AcOEt 10:1), yellow oil, yield 81%, 872.1 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.33 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.22 (td, *J* = 8.1, 1.5 Hz, 1H), 6.68 – 6.62 (m, 2H), 4.53 (br, 1H), 3.76 – 3.69 (m, 1H), 2.57 (t, *J* = 6.6 Hz, 2H), 1.74 – 1.57 (m, 4H), 1.33 (d, *J* = 6.3 Hz, 6H), 1.06 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 148.1 (Cq), 132.0 (CH), 129.0 (CH), 115.7 (CH), 109.9 (CH), 108.6 (Cq), 95.9 (Cq), 77.4 (Cq), 44.0 (CH), 31.1 (CH_2), 23.0 (2 CH_3), 22.0 (CH_2), 19.3 (CH_2), 13.6 (CH_3). IR (neat): 3400, 2960, 1600, 1506, 1546, 1321, 1281, 1176 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{15}H_{22}N$: 216.1752; found: 216.1754.

2-(hex-1-yn-1-yl)-*N*-methyl-*N*-phenylaniline (**7**)

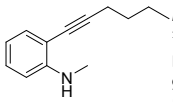


Rf = 0.47 (cyclohexane/AcOEt 10:1), colorless oil, yield 68%, 895.5 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.49 (d, *J* = 7.5 Hz, 1H), 7.34 – 7.14 (m, 5H), 6.89 – 6.61 (m, 3H), 3.34 (s, 3H), 2.29 (t, *J* = 6.6 Hz, 2H), 1.45 – 1.29 (m, 4H), 0.89 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 150.2 (Cq), 149.4 (Cq), 134.0 (CH), 128.8 (3 CH), 127.8 (CH), 125.3 (CH), 122.9 (Cq), 117.7 (CH), 114.5 (2 CH), 95.7 (Cq), 78.2 (Cq), 39.6 (CH_3), 30.7 (CH_2), 21.9 (CH_2), 19.3 (CH_2), 13.7 (CH_3). IR (neat): 2931, 1590, 1500, 1445, 1344 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{19}H_{22}N$: 264.1752; found: 264.1744.

Procedure for the synthesis of secondary aniline 4.

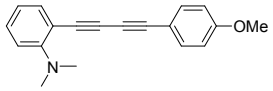
To a solution of **3** (5 mmol, 866 mg) in DMF (30 mL) was added K_2CO_3 (1.5 equiv) and iodomethane (1.1 equiv). The reaction was stirred at room temperature for 3 h then quenched with water (30 mL). The aqueous layer was extracted with CH_2Cl_2 three times and the combined organic extracts were dried with anhydrous $MgSO_4$, filtered and concentrated. The crude mixture was purified by silica gel column chromatography (0 to 30% AcOEt in cyclohexane)**.**

2-(Hex-1-yn-1-yl)-*N*-methylaniline (**4**)



Rf = 0.78 (cyclohexane/AcOEt 10:3), brown oil, yield 63%, 589.9 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.37 (dd, *J* = 7.5 Hz, 0.9 Hz, 1H), 7.28 (td, *J* = 7.8 Hz, 1.2 Hz , 1H), 6.80 – 6.60 (m, 2H), 4.70 (br, 1H), 2.97 (s, 3H), 2.58 (t, *J* = 7.2 Hz, 2H), 1.78 – 1.55 (m, 4H), 1.08 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 149.7 (Cq), 131.8 (CH), 129.1 (CH), 116.0 (CH), 108.7 (CH), 108.4 (Cq), 95.8 (Cq), 77.14 (Cq), 31.1 (CH_2), 30.2 (CH_3), 22.1 (CH_2), 19.4 (CH_2), 13.6 (CH_3). IR (neat): 3420, 2931, 1601, 1510, 1461, 1320, 1168 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{13}H_{18}N$: 188.1439; found: 188.1438.

Procedure for the synthesis of diyne 1m.
To a stirred mixture of 1-(bromorthynyl)-4-methoxybenzene (1.06 g, 5 mmol), $PdCl_2(PPh_3)_2$ (175.5 mg, 0.05 equiv) and CuI (95.3, 0.1 equiv) in pyrrolidine (10 mL), 2-ethynyl-*N*,*N*-dimethylaniline (798.6 mg, 1.1 equiv) was added dropwise at 0 °C. The mixture was then stirred at room temperature. When the starting material was disappeared (TLC monitoring), the mixture was passed through a pad of Celite to remove the insoluble materials. The filtrate was concentrated in vacuo and the residue was purified by silica gel column chromatography (0 to 10% AcOEt in cyclohexane).
2-((4-Methoxyphenyl)buta-1,3-diyn-1-yl)-*N*,*N*-dimethylaniline (**1m**)

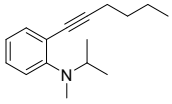


Rf = 0.40 (cyclohexane/AcOEt 10:1), yellow oil, yield 46%, 633.3 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.39 – 7.35 (m, 3H), 7.16 (t, *J* = 7.5 Hz, 1H), 6.83 – 6.69 (m, 4H), 3.72 (s, 3H), 2.89 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 160.3 (Cq), 156.2 (Cq), 135.4 (CH), 134.1 (2 CH), 130.0 (CH), 120.3 (CH), 116.9 (CH), 114.2 (2 CH), 113.4 (Cq), 82.9 (Cq), 81.3 (Cq), 80.5 (Cq), 79.3 (Cq), 73.3 (Cq), 55.4 (CH_3), 43.6 (2 CH_3). IR (neat): 2836, 2787, 2136, 1602, 1506, 1464, 1344 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{19}H_{18}NO$: 276.1388; found: 276.1387.

Procedure for the synthesis of 2-(hex-1-yn-1-yl)-*N*-isopropyl-*N*-methylaniline (8).

To a solution of 2-(hex-1-yn-1-yl)-*N*-isopropylaniline (2 mmol, 430.6 mg) in THF (10 mL) was added NaH (2 equiv) and iodomethane (2 equiv). The reaction was stirred at 70 °C in a sealed tube for 3 h then quenched with water (10 mL). The aqueous layer was extracted with CH_2Cl_2 three times and the combined organic extracts were dried with anhydrous $MgSO_4$, filtered and concentrated. The crude mixture was purified by silica gel column chromatography (0 to 10% AcOEt in cyclohexane)**.**

2-(hex-1-yn-1-yl)-*N*-isopropyl-*N*-methylaniline (**8**)



Rf = 0.35 (cyclohexane/AcOEt 10:1), yellow oil, yield 69%, 316.5 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.27 (d, *J* = 7.5 Hz, 1H), 7.07 (t, *J* = 7.5 Hz, 1H), 6.83 – 6.68 (m, 2H), 4.17 – 4.07 (m, 1H), 2.58 (s, 3H), 2.39 (t, *J* = 7.2 Hz, 2H), 1.58 – 1.33 (m, 4H), 1.03 (d, *J* = 6.6 Hz, 6H), 0.86 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 154.5 (Cq), 134.3 (CH), 128.1 (CH), 120.5 (CH), 118.9 (CH), 117.4 (Cq), 95.1 (Cq), 79.7 (Cq), 53.1 (CH), 31.1 (CH_2), 31.0 (CH_3), 22.2 (CH_2), 19.6 (CH_2), 18.7 (2 CH_3), 13.7 (CH_3). IR (neat): 2957, 1591, 1486, 1444, 1388, 1328, 1198 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{16}H_{24}N$: 230.1909; found: 230.1907.

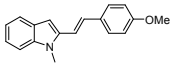
Procedure for the synthesis of terminal alkyne 1r.

To an oven dried 100 mL round-bottomed flask was added *N*,*N*-dimethyl-2-((trimethylsilyl)ethynyl)aniline (1.09 g, 5 mmol), MeOH (30 mL), and oven dried K_2CO_3 (691 mg, 1 equiv). The flask was stirred under argon atmosphere at room temperature. When TLC show the disappearance of starting material, H_2O (30 mL) was added to the flask. After extracted with CH_2Cl_2 (3 × 30 mL), the organic layer was dried over anhydrous $MgSO_4$, and concentrated in vacuo. The crude oil was then purified by column chromatography on silica gel (0 to 10% AcOEt in cyclohexane).
2-Ethynyl-*N*,*N*-dimethylaniline (**1r**)[2]

Rf = 0.35 (cyclohexane/AcOEt 10:1), brown oil, yield 95%, 689.7 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.34 (dd, *J* = 7.5, 1.5 Hz, 1H), 7.12 (t, *J* = 7.5 Hz, 1H), 6.85 – 6.68 (m, 2H), 3.30 (s, 1H), 2.80 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 155.5 (Cq), 134.8 (CH), 129.6 (CH), 120.6 (CH), 117.0 (CH), 114.3 (Cq), 82.9 (Cq), 82.4 (CH), 43.5 (2 CH_3). IR (neat): 2956, 2360, 1650, 1561, 1431 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{10}H_{12}N$: 146.0970; found: 146.0967.

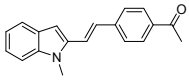
General procedure for the synthesis of indoles 2a-v, 5, 6.
A solution of alkyne (0.5 mmol) in EtOH (3 mL) was stirred under microwave irradiation (for time and temperature see for each compound). When the starting material disappeared (TLC monitoring), the mixture was concentrated *in vacuo*, the crude mixture purified by silica gel column chromatography (0 to 30% AcOEt in cyclohexane).

(*E*)-2-(4-Methoxystyryl)-1-methyl-1*H*-indole (**2a**)[1]



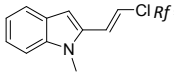
Rf = 0.48 (cyclohexane/AcOEt 10:3), yellow solid, m.p. 141.4 – 141.8 °C, yield 92%, 55.8 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.63 (d, *J* = 7.5 Hz, 1H), 7.51 (d, *J* = 8.4 Hz, 2H), 7.34 – 7.12 (m, 4H), 7.05 (d, *J* = 15.9 Hz, 1H), 6.96 (d, *J* = 8.4 Hz, 2H), 6.80 (s, 1H), 3.87 (s, 3H), 3.83 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 159.5 (Cq), 138.8 (Cq), 138.1 (Cq), 130.6 (CH), 130.0 (Cq), 128.1 (Cq), 127.7 (2 CH), 121.5 (CH), 120.3 (CH), 119.8 (CH), 114.9 (CH), 114.2 (2 CH), 109.1 (CH), 98.4 (CH), 55.4 (CH_3), 29.9 (CH_3). IR (neat): 1573, 1463, 1395, 1247 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{18}H_{18}NO$: 264.1388; found: 264.1387.

(*E*)-1-(4-(2-(1-Methyl-1*H*-indol-2-yl)vinyl)phenyl)ethan-1-one (**2c**)[1]



Rf = 0.38 (cyclohexane/AcOEt 10:3), yellow solid, m.p. 188.5 – 188.9 °C, yield 90%, 123.9 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.99 (d, *J* = 8.4 Hz, 2H), 7.65 – 7.60 (m, 3H), 7.36 – 7.11 (m, 5H), 6.90 (s, 1H), 3.86 (s, 3H), 2.64 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 197.5 (Cq), 141.9 (Cq), 138.5 (Cq), 137.8 (Cq), 136.1 (Cq), 129.3 (CH), 129.1 (2 CH), 128.0 (Cq), 126.5 (2 CH), 122.4 (CH), 120.8 (CH), 120.2 (CH), 119.7 (CH), 109.4 (CH), 100.2 (CH), 30.1 (CH_3), 26.7 (CH_3). IR (neat): 3054, 1675, 1597, 1348, 1274 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{19}H_{18}NO$: 276.1388; found: 276.1378.

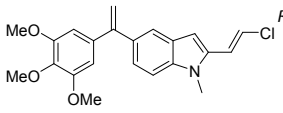
(*E*)-2-(2-Chlorovinyl)-1-methyl-1*H*-indole (**2b**)



Rf = 0.84 (cyclohexane/AcOEt 10:1), brown solid, m.p. 65.2 – 65.5 °C, yield 75%, 71.9 mg.

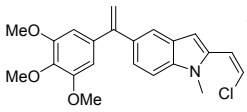
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.49 (d, *J* = 7.8 Hz, 1H), 7.22 – 7.11 (m, 2H), 7.02 (t, *J* = 7.5 Hz, 1H), 6.84 (d, *J* = 13.5 Hz, 1H), 6.62 (d, *J* = 13.5 Hz, 1H), 6.52 (s, 1H), 3.65 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 138.0 (Cq), 135.0 (Cq), 127.7 (Cq), 123.0 (CH), 122.3 (CH), 120.7 (CH), 120.4 (CH), 120.2 (CH), 109.4 (CH), 99.8 (CH), 30.1 (CH_3). IR (neat): 2940, 1463, 1343, 1319, 1166 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{11}H_{11}{}^{35}ClN$: 192.0580; found: 192.0571.

(*E*)-2-(2-Chlorovinyl)-1-methyl-5-(1-(3,4,5-trimethoxyphenyl)vinyl)-1*H*-indole (**2d**)



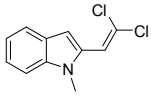
Rf = 0.72 (cyclohexane/AcOEt 5:5), brown oil, yield 56%, 107.5 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.48 (s, 1H), 7.18 – 7.15 (m, 2H), 6.84 (d, *J* = 13.5 Hz, 1H), 6.62 (d, *J* = 13.5 Hz, 1H), 6.53 – 6.51 (m, 3H), 5.35 (s, 1H), 5.29 (s, 1H), 3.81 (s, 3H), 3.72 (s, 6H), 3.66 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 152.9 (2 Cq), 151.0 (Cq), 138.2 (Cq), 137.9 (Cq), 137.8 (Cq), 135.6 (Cq), 133.6 (Cq), 127.6 (Cq), 123.2 (CH), 122.8 (CH), 120.7 (CH), 120.6 (CH), 112.7 (CH_2), 108.9 (CH), 106.0 (2 CH), 100.1 (CH), 61.0 (CH_3), 56.3 (2 CH_3), 30.2 (CH_3). IR (neat): 2935, 1579, 1504, 1450, 1348, 1237, 1127 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{22}H_{23}NO_3{}^{35}Cl$: 384.1366; found: 384.1366.

(*Z*)-2-(2-Chlorovinyl)-1-methyl-5-(1-(3,4,5-trimethoxyphenyl)vinyl)-1*H*-indole (**2e**)



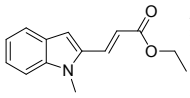
Rf = 0.31 (cyclohexane/AcOEt 5:5), brown oil, yield 42%, 80.6 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.66 (s, 1H), 7.29 – 7.27 (m, 3H), 6.79 (d, *J* = 8.1 Hz, 1H), 6.63 (s, 2H), 6.42 (d, *J* = 8.1 Hz, 1H), 5.46 (s, 1H), 5.40 (s, 1H), 3.91 (s, 3H), 3.82 (s, 6H), 3.78 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 152.8 (2 Cq), 150.9 (Cq), 138.1 (Cq), 137.8 (Cq), 136.9 (Cq), 133.4 (Cq), 133.3 (Cq), 127.5 (Cq), 123.5 (CH), 121.0 (CH), 119.2 (CH), 118.2 (CH), 112.5 (CH_2), 108.7 (CH), 105.9 (2 CH), 105.1 (CH), 60.9 (CH_3), 56.1 (2 CH_3), 29.9 (CH_3). IR (neat): 2987, 1684, 1579, 1542, 1470, 1376 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{22}H_{23}NO_3{}^{35}Cl$: 384.1366; found: 384.1359.

2-(2,2-Dichlorovinyl)-1-methyl-1*H*-indole (**2f**)



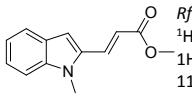
Rf = 0.67 (cyclohexane/AcOEt 10:1), yellow oil, yield 85%, 96.1 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.55 (d, *J* = 8.1 Hz, 1H), 7.23 – 7.14 (m, 2H), 7.12 – 6.95 (m, 2H), 6.85 (s, 1H), 3.64 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 137.4 (Cq), 132.2 (Cq), 127.6 (Cq), 123.1 (CH), 122.1 (Cq), 121.3 (CH), 120.2 (CH), 118.2 (CH), 109.4 (CH), 104.3 (CH), 29.9 (CH_3). IR (neat): 2928, 1462, 2365, 1289 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{11}H_{10}{}^{35}Cl_2N$: 226.0185; found: 226.0180.

Ethyl (*E*)-3-(1-methyl-1*H*-indol-2-yl)acrylate (**2g**)



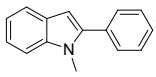
Rf = 0.24 (cyclohexane/AcOEt 10:1), yellow solid, m.p. 86.4 – 86.6 °C, yield 90%, 103.2 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.69 (d, *J* = 15.6 Hz, 1H), 7.50 (d, *J* = 7.8 Hz, 1H), 7.21 – 7.12 (m, 2H), 7.01 (t, *J* = 7.5 Hz, 1H), 6.84 (s, 1H), 6.38 (d, *J* = 15.6 Hz, 1H), 4.19 (q, *J* = 7.2 Hz, 2H), 3.69 (s, 3H), 1.26 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 167.1 (Cq), 139.1 (Cq), 135.0 (Cq), 132.7 (CH), 127.5 (Cq), 123.6 (CH), 121.4 (CH), 120.5 (CH), 118.3 (CH), 109.7 (CH), 103.8 (CH), 60.6 (CH_2), 30.1 (CH_3), 14.4 (CH_3). IR (neat): 2979, 1701, 1628, 1462, 1304, 1148 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{14}H_{16}NO_2$: 230.1181; found: 230.1175.

Methyl (*E*)-3-(1-methyl-1*H*-indol-2-yl)acrylate (**2h**)



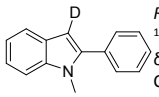
Rf = 0.26 (cyclohexane/AcOEt 10:1), yellow solid, m.p. 94.9 – 95.2 °C, yield 88%, 94.7 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.81 (d, *J* = 15.9 Hz, 1H), 7.62 (d, *J* = 7.8 Hz, 1H), 7.33 – 7.24 (m, 2H), 7.13 (t, *J* = 7.5 Hz, 1H), 6.96 (s, 1H), 6.49 (d, *J* = 15.9 Hz, 1H), 3.84 (s, 3H), 3.80 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 167.5 (Cq), 139.1 (Cq), 134.9 (Cq), 132.9 (CH), 127.5 (Cq), 123.7 (CH), 121.5 (CH), 120.5 (CH), 117.8 (CH), 109.7 (CH), 103.9 (CH), 51.8 (CH_3), 30.1 (CH_3). IR (neat): 2949, 1716, 1520, 1282, 1164 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{13}H_{14}NO_2$: 216.1025; found: 216.1029.

1-Methyl-2-phenyl-1*H*-indole (**2i**)[5]



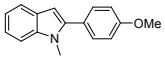
Rf = 0.58 (cyclohexane/AcOEt 10:1), yellow solid, m.p. 101.7 – 101.9 °C, yield 93%, 96.4 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.55 (d, *J* = 7.8 Hz, 1H), 7.49 – 7.21 (m, 6H), 7.16 (t, *J* = 7.8 Hz, 1H), 7.05 (t, *J* = 7.5 Hz, 1H), 6.48 (s, 1H), 3.65 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 141.6 (Cq), 138.4 (Cq), 132.9 (Cq), 129.4 (2 CH), 128.5 (2 CH), 128.0 (Cq), 127.9 (CH), 121.7 (CH), 120.5 (CH), 119.9 (CH), 109.6 (CH), 101.7 (CH), 31.2 (CH_3). IR (neat): 3057, 1604, 1467, 1386, 1340, 1319 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{15}H_{14}N$: 208.1126; found: 208.1126.

1-Methyl-2-phenyl-1*H*-indole-3-d ((D)-**2i**)



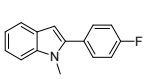
Rf = 0.58 (cyclohexane/AcOEt 10:1), yellow solid, m.p. 99.5 – 99.8 °C, yield 90%, 93.7 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.80 (d, *J* = 7.8 Hz, 1H), 7.69 – 7.47 (m, 6H), 7.40 (t, *J* = 7.5 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 1H), 3.85 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 141.6 (Cq), 138.5 (Cq), 132.9 (Cq), 129.4 (2 CH), 128.6 (2 CH), 128.0 (Cq), 127.9 (CH), 121.7 (CH), 120.5 (CH), 119.9 (CH), 109.7 (CH), 101.5 (t, *J* = 26.3 Hz, Cq), 31.2 (CH_3). IR (neat): 3054, 1603, 1465, 1380, 1336, 1302 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{15}H_{13}{}^2HN$: 209.1189; found: 209.1186.

2-(4-Methoxyphenyl)-1-methyl-1*H*-indole (**2j**)[6]



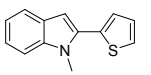
Rf = 0.37 (cyclohexane/AcOEt 10:1), yellow solid, m.p. 126.5 – 126.7 °C, yield 91%, 108.0 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.53 (d, *J* = 7.8 Hz, 1H), 7.34 (d, *J* = 8.1 Hz, 2H), 7.26 (d, *J* = 8.1 Hz, 1H), 7.14 (t, *J* = 7.5 Hz, 1H), 7.04 (t, *J* = 7.5 Hz, 1H), 6.91 (d, *J* = 8.1 Hz, 2H), 6.42 (s, 1H), 3.78 (s, 3H), 3.63 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 159.6 (Cq), 141.5 (Cq), 138.3 (Cq), 130.7 (2 CH), 128.2 (Cq), 125.4 (Cq), 121.5 (CH), 120.4 (CH), 119.9 (CH), 114.1 (2 CH), 109.6 (CH), 101.2 (CH), 55.5 (CH_3), 31.1 (CH_3). IR (neat): 2836, 1611, 1497, 1466, 1285, 1250 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{16}H_{26}NO$: 238.1232; found: 238.1227.

2-(4-Fluorophenyl)-1-methyl-1*H*-indole (**2k**)[6]



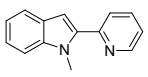
Rf = 0.47 (cyclohexane/AcOEt 10:1), yellow solid, m.p. 116.7 – 117.3 °C, yield 94%, 105.9 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.53 (d, *J* = 7.8 Hz, 1H), 7.38 – 7.32 (m, 2H), 7.24 (d, *J* = 8.1 Hz, 1H), 7.15 (t, *J* = 7.5 Hz, 1H), 7.08 – 7.01 (m, 3H), 6.42 (s, 1H), 3.59 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 162.7 (d, *J* = 246.2 Hz, Cq), 140.5 (Cq), 138.4 (Cq), 131.1 (d, *J* = 8.0 Hz, 2 CH), 129.1 (d, *J* = 3.0 Hz, Cq), 128.0 (Cq), 121.9 (CH), 120.6 (CH), 120.1 (CH), 115.6 (d, *J* = 21.5 Hz, 2 CH), 109.7 (CH), 101.8 (CH), 31.1 (CH_3). ^{19}F (188 MHz, $CDCl_3$): -113.776. IR (neat): 3066, 1603, 1494, 1432, 1219 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{15}H_{13}NF$: 226.1032; found: 226.1030.

1-Methyl-2-(thiophen-2-yl)-1*H*-indole (**2l**)[7]



Rf = 0.50 (cyclohexane/AcOEt 10:1), yellow solid, m.p. 82.4 – 82.6 °C, yield 95%, 101.3 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.52 (d, *J* = 7.8 Hz, 1H), 7.32 – 7.11 (m, 5H), 7.03 (t, *J* = 7.5 Hz, 1H), 6.49 (s, 1H), 3.67 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 138.2 (Cq), 136.5 (Cq), 133.5 (Cq), 128.5 (CH), 127.9 (Cq), 125.9 (CH), 123.3 (CH), 121.8 (CH), 120.5(CH), 119.9 (CH), 109.6 (CH), 101.6 (CH), 31.2 (CH_3). IR (neat): 2944, 1465, 1405, 1334, 1310 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{13}H_{12}NS$: 214.0690; found: 214.0689.

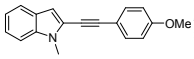
1-Methyl-2-(pyridin-2-yl)-1*H*-indole (**2m**)



Rf = 0.29 (cyclohexane/AcOEt 10:1), yellow solid, m.p. 88.8 – 89.0 °C, yield 95%, 98.9 mg.

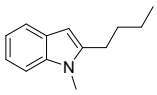
^{1}H NMR (300 MHz, $CDCl_3$) δ = 8.59 (d, *J* = 4.5 Hz, 1H), 7.73 – 7.48 (m, 3H), 7.31 (d, *J* = 8.4 Hz, 1H), 7.21 – 7.02 (m, 3H), 6.76 (s, 1H), 3.98 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 152.7 (Cq), 149.1 (CH), 139.4 (Cq), 139.1 (Cq), 136.6 (CH), 127.6 (Cq), 123.6 (CH), 122.6 (CH), 121.8 (CH), 121.0 (CH), 120.0 (CH), 109.9 (CH), 103.6 (CH), 32.0 (CH_3). IR (neat): 3052, 2360, 1613, 1453, 1320 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{14}H_{13}N_2$: 209.1079; found: 209.1073.

2-((4-Methoxyphenyl)ethynyl)-1-methyl-1*H*-indole (**2n**)



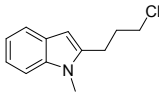
Rf = 0.40 (cyclohexane/AcOEt 10:1), white solid, m.p. 135.7 – 135.9 °C, yield 74%, 96.7 mg.
^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.59 (d, *J* = 8.0 Hz, 1H), 7.54 – 7.49 (m, 2H), 7.32 – 7.23 (m, 2H), 7.14 – 7.09 (m, 1H), 6.93 – 6.89 (m, 2H), 6.80 (s, 1H), 3.86 (s, 3H), 3.85 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ = 160.1 (Cq), 137.4 (Cq), 133.1 (2 CH), 127.5 (Cq), 122.9 (CH), 122.6 (Cq), 120.9 (CH), 120.1 (CH), 114.9 (Cq), 114.2 (2 CH), 109.4 (CH), 107.0 (CH), 95.3 (Cq), 79.9 (Cq), 55.5 (CH_3), 30.7 (CH_3). IR (neat): 3009, 2360, 2210, 1602, 1537, 1502, 1441, 1250 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{18}H_{16}NO$: 262.1232; found: 262.1233.

2-Butyl-1-methyl-1*H*-indole (**2o**)



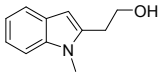
Rf = 0.55 (cyclohexane/AcOEt 10:1), brown oil, yield 95%, 89.0 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.56 (d, *J* = 7.8 Hz, 1H), 7.28 (d, *J* = 8.1 Hz, 1H), 7.17 (t, *J* = 7.5 Hz, 1H), 7.08 (t, *J* = 7.5 Hz, 1H), 6.28 (s, 1H), 3.68 (s, 3H), 2.76 (t, *J* = 7.5 Hz, 2H), 1.80 – 1.69 (m, 2H), 1.55 – 1.44 (m, 2H), 1.01 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 141.5 (Cq), 137.5 (Cq), 128.1 (Cq), 120.5 (CH), 119.8 (CH), 119.3 (CH), 108.8 (CH), 98.8 (CH), 30.9 (CH_2), 29.5 (CH_3), 26.7 (CH_2), 22.6 (CH_2), 14.0 (CH_3). IR (neat): 2929, 2359, 1546, 1467, 1401, 1314, 1252 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{13}H_{18}N$: 188.1439; found: 188.1445.

2-(3-Chloropropyl)-1-methyl-1*H*-indole (**2p**)



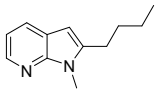
Rf = 0.59 (cyclohexane/AcOEt 10:1), colorless oil, yield 80%, 83.1 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.56 (d, *J* = 7.8 Hz, 1H), 7.29 (d, *J* = 8.1 Hz, 1H), 7.19 (t, *J* = 7.5 Hz, 1H), 7.09 (t, *J* = 7.5 Hz, 1H), 6.30 (s, 1H), 3.73 – 3.58 (m, 5H), 2.95 (t, *J* = 7.5 Hz, 2H), 2.54 – 2.16 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 139.3 (Cq), 137.5 (Cq), 127.9 (Cq), 120.9 (CH), 119.9 (CH), 119.5 (CH), 108.9 (CH), 99.4 (CH), 44.3 (CH_2), 31.5 (CH_2), 29.5 (CH_3), 23.9 (CH_2). IR (neat): 2958, 1546, 1468, 1337, 1234 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{12}H_{15}N^{35}Cl$: 208.0893; found: 208.0886.

2-(1-Methyl-1*H*-indol-2-yl)ethan-1-ol (**2q**)



Rf = 0.15 (cyclohexane/AcOEt 10:3), yellow solid, m.p. 63.3 – 63.5 °C, yield 79%, 69.2 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.57 (d, *J* = 7.8 Hz, 1H), 7.30 (d, *J* = 8.1 Hz, 1H), 7.21 (t, *J* = 7.5 Hz, 1H), 7.11 (t, *J* = 7.5 Hz, 1H), 6.35 (s, 1H), 3.94 (t, *J* = 6.3 Hz, 2H), 3.70 (s, 3H), 3.04 (t, *J* = 6.3 Hz, 2H), 1.81 (br, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 137.7 (Cq), 137.2 (Cq), 127.9 (Cq), 121.1 (CH), 120.0 (CH), 119.6 (CH), 109.1 (CH), 100.0 (CH), 61.4 (CH_2), 30.2 (CH_2), 29.7 (CH_3). IR (neat): 2946, 1468, 1341, 1234, 1044 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{11}H_{14}NO$: 176.1075; found: 176.1081.

2-Butyl-1-methyl-1*H*-pyrrolo[2,3-b]pyridine (**2r**)

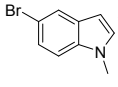


Rf = 0.18 (cyclohexane/AcOEt 10:1), yellow oil, yield 90%, 84.7 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 8.24 (d, *J* = 4.5 Hz, 1H), 7.78 (d, *J* = 7.8 Hz, 1H), 7.02 – 6.97 (m, 1H), 6.19 (s, 1H), 3.79 (s, 3H), 2.76 (t, *J* = 7.5 Hz, 2H), 1.78 – 1.67 (m, 2H), 1.54 – 1.43 (m, 2H), 0.99 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 148.7 (Cq), 142.3 (Cq), 141.5 (CH), 127.2 (CH), 120.7 (Cq), 115.5 (CH), 96.7 (CH), 30.5 (CH_2), 28.0 (CH_3), 26.8 (CH_2), 22.6 (CH_2), 13.9 (CH_3). IR (neat): 2930, 1594, 1541, 1454, 1407, 1310 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{12}H_{17}N_2$: 189.1392; found: 189.1389.

1-Methyl-1*H*-indole (**2s**)[6]

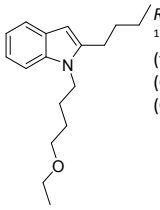
Rf = 0.80 (cyclohexane/AcOEt 10:1), yellow oil, for yields see the text.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.54 (d, *J* = 7.8 Hz, 1H), 7.22 (d, *J* = 8.1 Hz, 1H), 7.13 (t, *J* = 7.5 Hz, 1H), 7.01 (t, *J* = 7.5 Hz, 1H), 6.92 (d, *J* = 3.0 Hz, 1H), 6.39 (d, *J* = 3.0 Hz, 1H), 3.66 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 136.8 (Cq), 128.8 (CH), 128.6 (Cq), 121.5 (CH), 120.9 (CH), 119.3 (CH), 109.2 (CH), 101.0 (CH), 32.8 (CH_3). IR (neat): 3055, 1513, 1462, 1316, 1242 cm^{-1}.

5-Bromo-1-methyl-1*H*-indole (**2t**)[8]



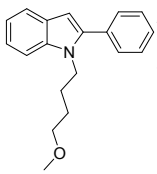
Rf = 0.46 (cyclohexane/AcOEt 10:1), yellow solid, m.p. 44.2 – 44.5 °C, yield 88%, 92.5 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.79 (d, *J* = 1.5 Hz, 1H), 7.34 (dd, *J* = 8.7, 1.5 Hz, 1H), 7.21 (d, *J* = 8.7 Hz, 1H), 7.07 (d, *J* = 3.0 Hz, 1H), 6.46 (d, *J* = 3.0 Hz, 1H), 3.79 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 135.5 (Cq), 130.2 (Cq), 130.0 (CH), 124.4 (CH), 123.4 (CH), 112.7 (Cq), 110.7 (CH), 100.6 (CH), 33.0 (CH_3). IR (neat): 2915, 1608, 1562, 1473, 1330, 1276, 1241 cm^{-1}.

2-Butyl-1-(4-ethoxybutyl)-1*H*-indole (**2u**)



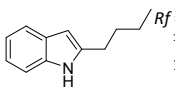
Rf = 0.53 (cyclohexane/AcOEt 10:1), yellow oil, yield 85%, 116.2 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.53 (d, *J* = 7.5 Hz, 1H), 7.28 (d, *J* = 8.1 Hz, 1H), 7.16 – 7.02 (m, 2H), 6.24 (s, 1H), 4.11 (t, *J* = 7.5 Hz, 2H), 3.51 – 3.41 (m, 4H), 2.74 (t, *J* = 7.5 Hz, 2H), 1.89 – 1.45 (m, 8H), 1.20 (t, *J* = 7.2 Hz, 3H), 0.99 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 141.1 (Cq), 136.7 (Cq), 128.2 (Cq), 120.4 (CH), 119.8 (CH), 119.2 (CH), 109.2 (CH), 98.8 (CH), 70.2 (CH_2), 66.3 (CH_2), 43.1 (CH_2), 30.8 (CH_2), 27.4 (CH_2), 27.3 (CH_2), 26.5 (CH_2), 22.7 (CH_2), 15.3 (CH_3), 14.1 (CH_3). IR (neat): 2956, 2870, 1545, 1463, 1410, 1377, 1352, 1117 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{18}H_{28}NO$: 274.2171; found: 274.2175.

1-(4-Methoxybutyl)-2-phenyl-1H-indole (**2v**)



Rf = 0.62 (cyclohexane/AcOEt 10:3), colorless oil, yield 71%, 99.2 mg.
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.54 (d, *J* = 7.8 Hz, 1H), 7.49 – 7.21 (m, 6H), 7.14 (t, *J* = 7.5 Hz, 1H), 7.04 (t, *J* = 7.5 Hz, 1H), 6.43 (s, 1H), 4.10 (t, *J* = 7.5 Hz, 2H), 3.29 – 2.94 (m, 5H), 1.73 -1.62 (m, 2H), 1.38 – 1.27 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 141.4 (Cq), 137.5 (Cq), 133.4 (Cq), 129.5 (2 CH), 128.6 (2 CH), 128.4 (Cq), 128.0 (CH), 121.6 (CH), 120.7 (CH), 119.8 (CH), 110.2 (CH), 102.3 (CH), 72.1 (CH_2), 58.6 (CH_3), 43.8 (CH_2), 26.9 (CH_2), 26.8 (CH_2). IR (neat): 2942, 1444, 1393, 1347, 1314, 1115 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{19}H_{22}NO$: 280.1701; found: 280.1698.

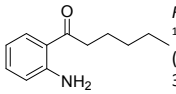
2-Butyl-1*H*-indole (**5**)



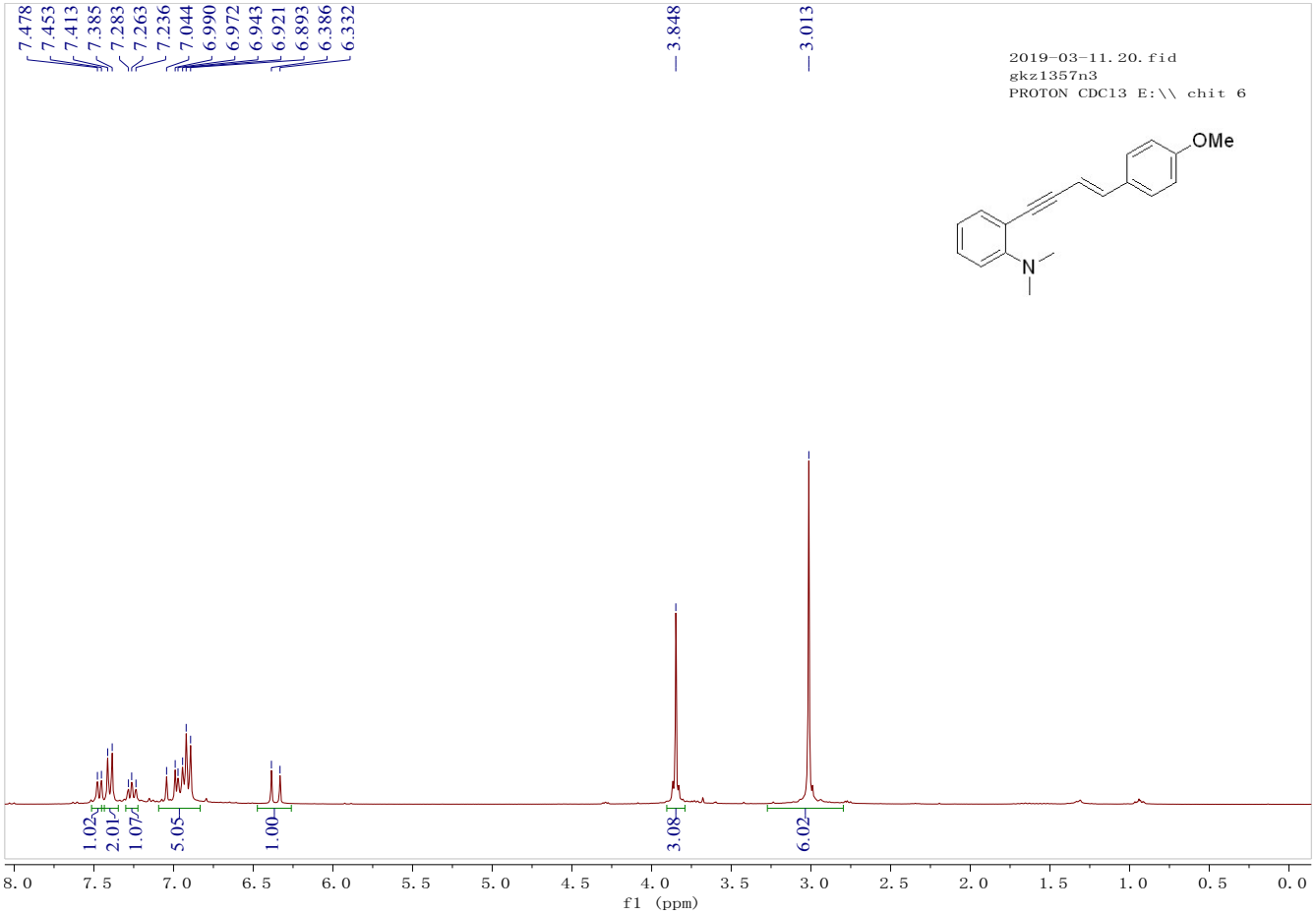
Rf = 0.60 (cyclohexane/AcOEt 10:1), yellow oil, yield 45%, 39.0 mg.
^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.84 (br, 1H), 7.53 (d, *J* = 7.6 Hz, 1H), 7.29 (d, *J* = 7.6 Hz, 1H), 7.19 – 7.00 (m, 2H), 6.25 (s, 1H), 2.76 (t, *J* = 7.6 Hz, 2H), 1.76 – 1.68 (m, 2H), 1.48 – 1.38 (m, 2H), 0.96 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ = 140.1 (Cq), 135.9 (Cq), 129.0 (Cq), 121.0 (CH), 119.8 (CH), 119.7

(CH), 110.3 (CH), 99.6 (CH), 31.4 (CH_2), 28.1 (CH_2), 22.5 (CH_2), 14.0 (CH_3). IR (neat): 3480, 2957, 1686, 1617, 1457, 1261 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{12}H_{16}N$: 174.1283; found: 174.1283.

1-(2-Aminophenyl)hexan-1-one (**6**)



Rf = 0.47 (cyclohexane/AcOEt 10:1), yellow oil, yield 43%, 41.2 mg.
^1H NMR (400 MHz, $CDCl_3$) δ = 7.74 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.28 – 7.23 (m, 1H), 6.72 – 6.59 (m, 2H), 2.92 (t, *J* = 7.2 Hz, 2H), 1.77 – 1.68 (m, 2H), 1.40 – 1.34 (m, 4H), 0.95 – 0.87 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ = 203.3 (Cq), 150.4 (Cq), 134.2 (CH), 131.3 (CH), 118.2 (Cq), 117.5 (CH), 115.8 (CH), 39.4 (CH_2), 31.8 (CH_2), 24.8 (CH_2), 22.7 (CH_2), 14.1 (CH_3). IR (neat): 3341, 2988, 1649, 1615, 1582, 1548, 1452 cm^{-1}. HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{12}H_{18}NO$: 192.1388; found: 192.1394.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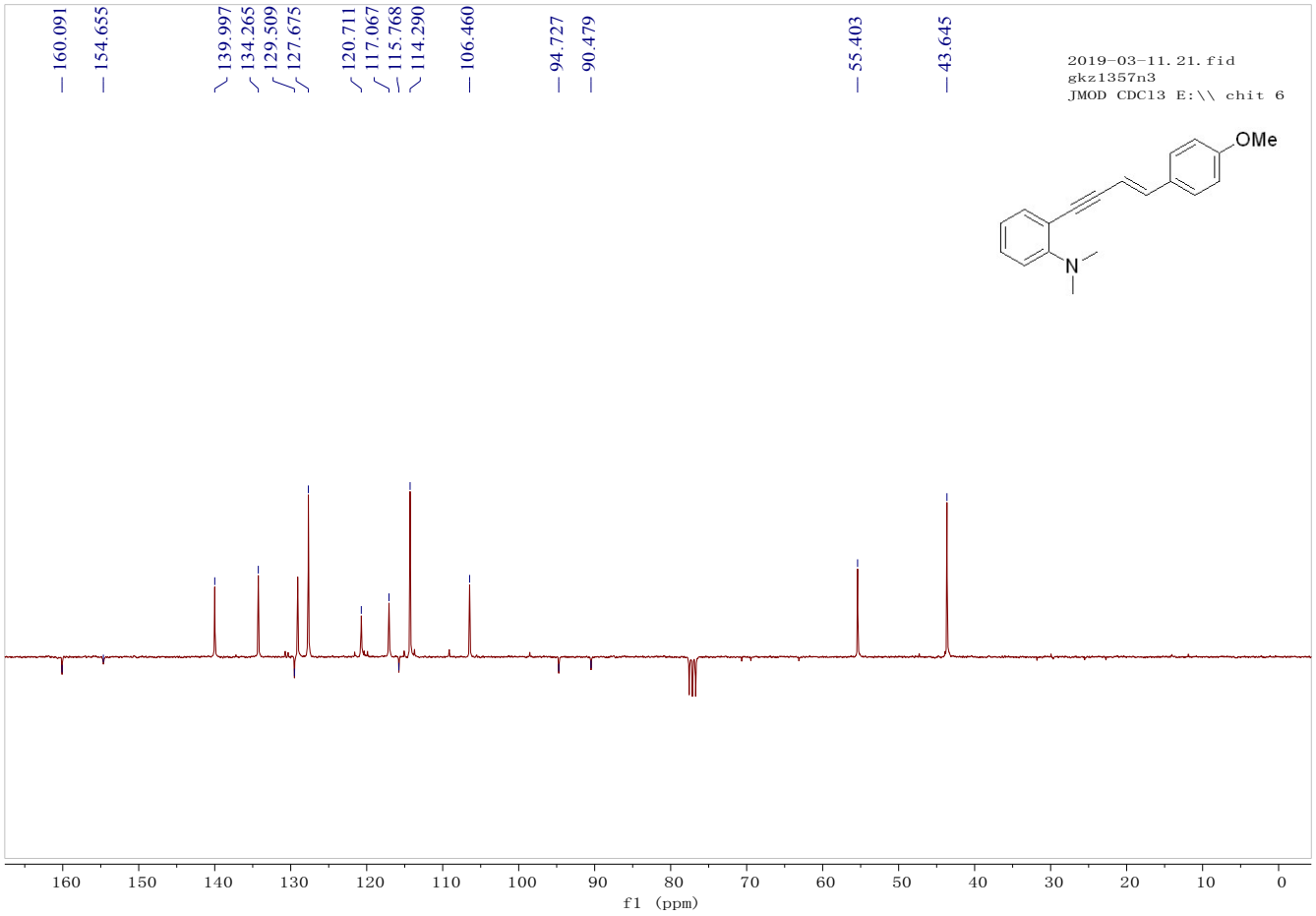
7.453
7.413
7.385
7.283
7.263
7.236
7.044
6.990
6.972
6.943
6.921
6.893
6.386
6.332
3.848
3.013
2019-03-11.20.fid
gkz1357n3
PROTON CDCl3 E:\\ chit 6
OMe
N
1.02
2.01
1.07
5.05
1.00
3.08
6.02
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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139.997
134.265
129.509
127.675
120.711
117.067
115.768
114.290
106.460
94.727
90.479
55.403
43.645
2019-03-11.21.fid
gkz1357n3
JMOD CDCl3 E:\\ chit 6
OMe
N
160
150
140
130
120
110
100
90
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50
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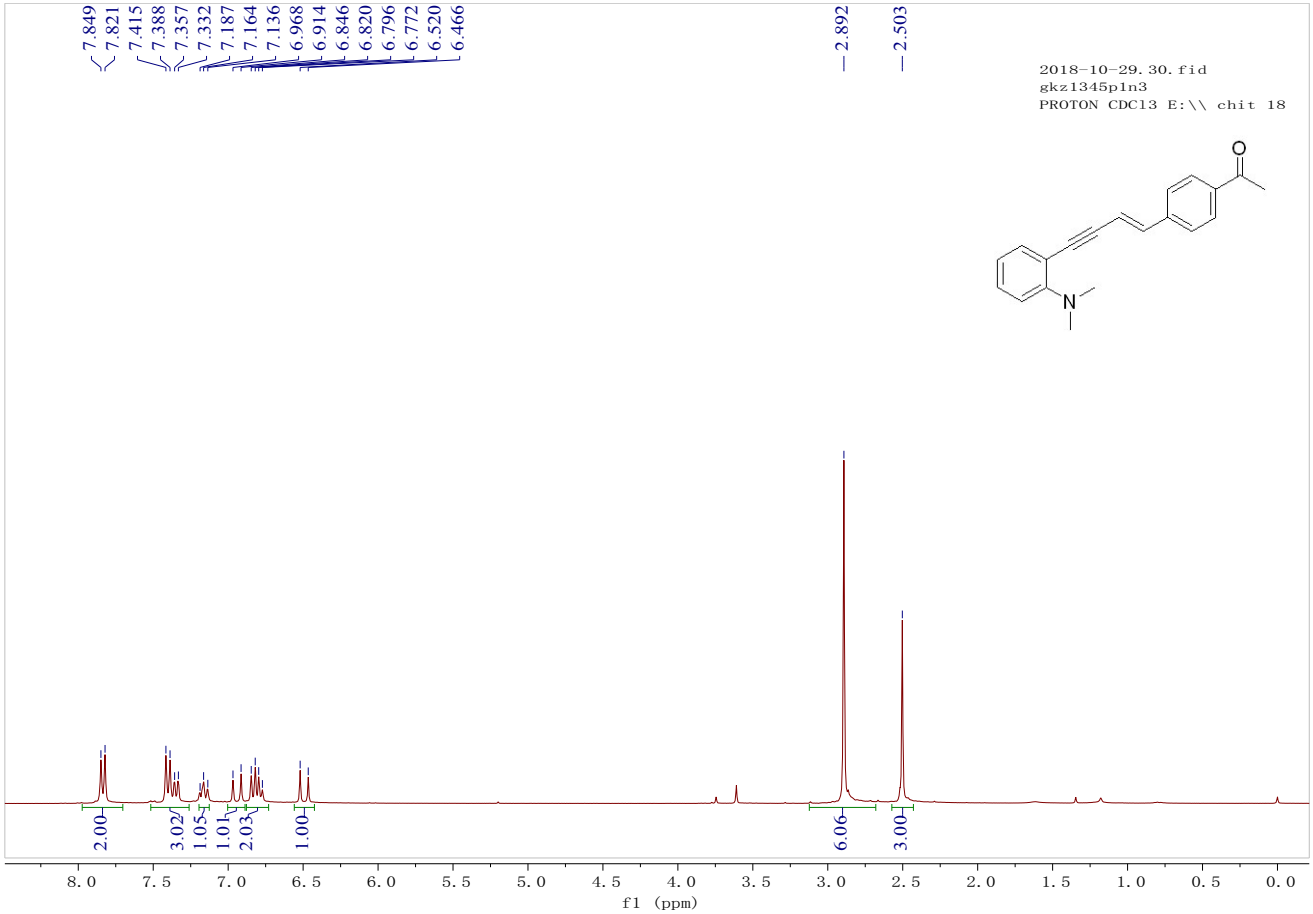
20
10
0
f1 (ppm)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6.968
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6.846
6.820
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6.520
6.466
2.892
2.503
2018-10-29.30.fid
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PROTON CDCl3 E:\\ chit 18
O
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2.00
3.02
1.05
1.01
2.03
1.00
6.06
3.00
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
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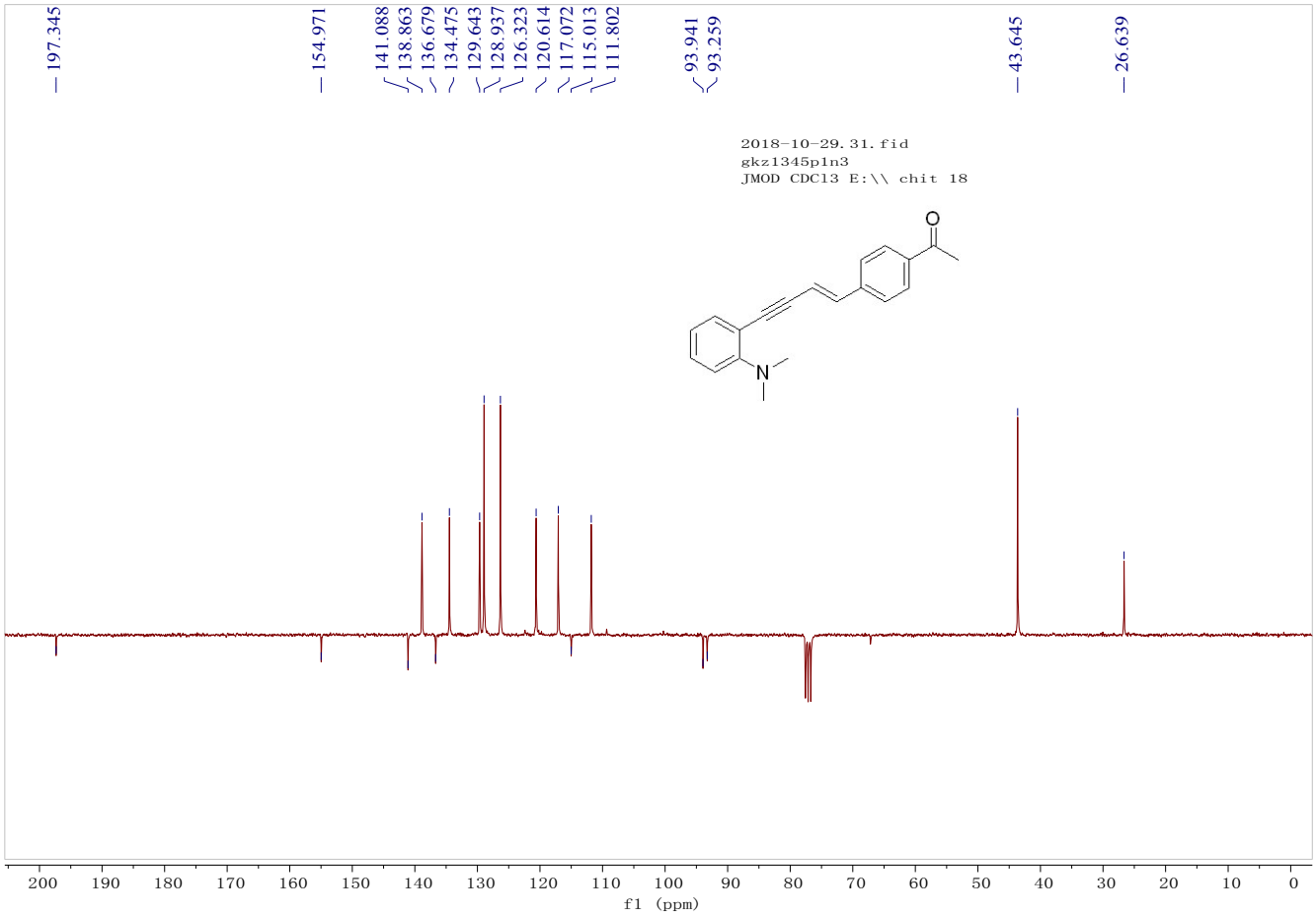
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


197.345
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141.088
138.863
136.679
134.475
129.643
128.937
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120.614
117.072
115.013
111.802
93.941
93.259
43.645
26.639
2018-10-29.31.fid
gkz1345p1n3
JMOD CDCl3 E:\\ chit 18
O
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200
190
180
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160
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110
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90
80
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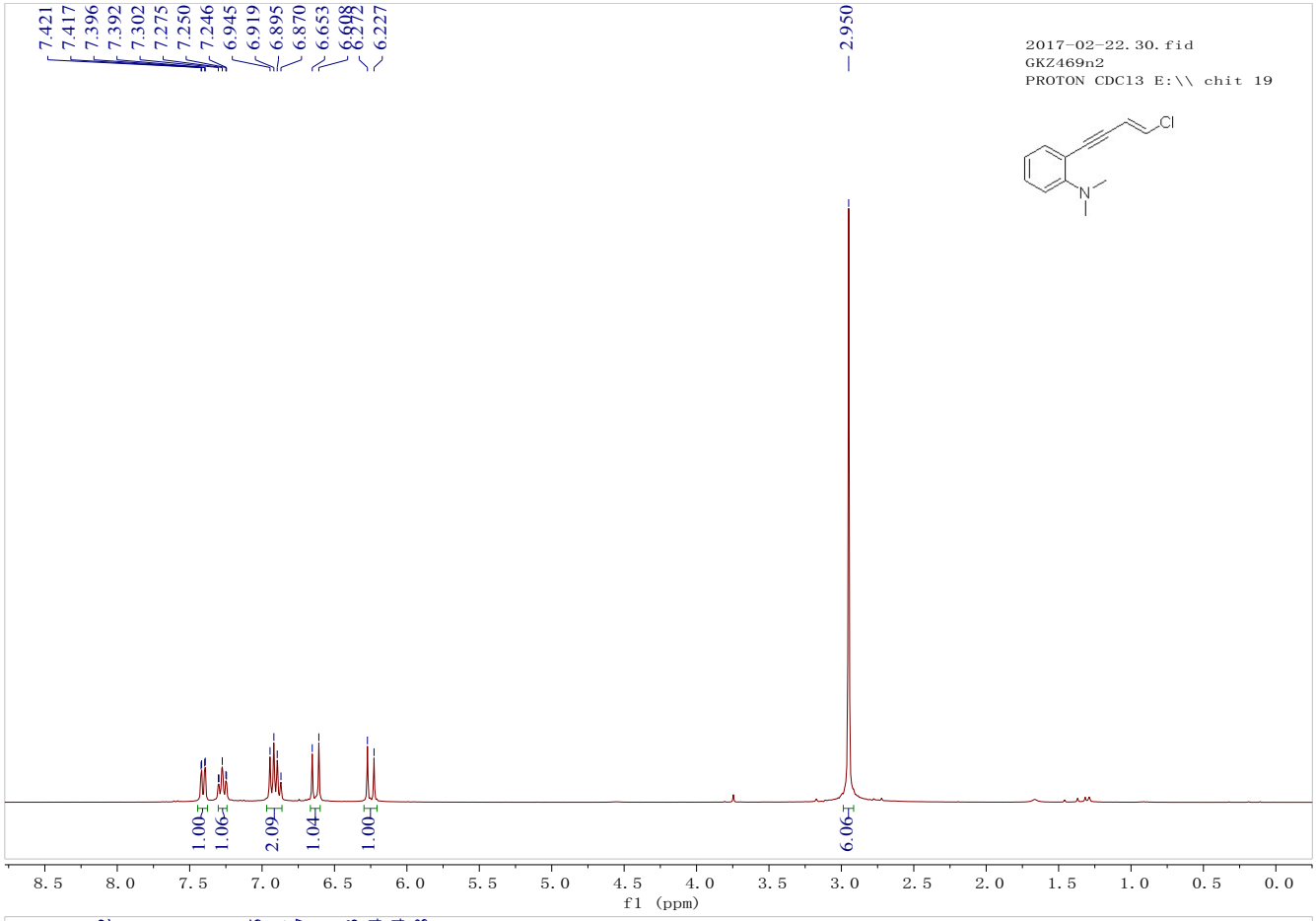
50
40
30
20
10
0
f1 (ppm)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7.275
7.250
7.246
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6.895
6.870
6.653
6.608
6.227
2.950
2017-02-22.30.fid
GKZ469n2
PROTON CDCl3 E:\\ chit 19
Cl
N
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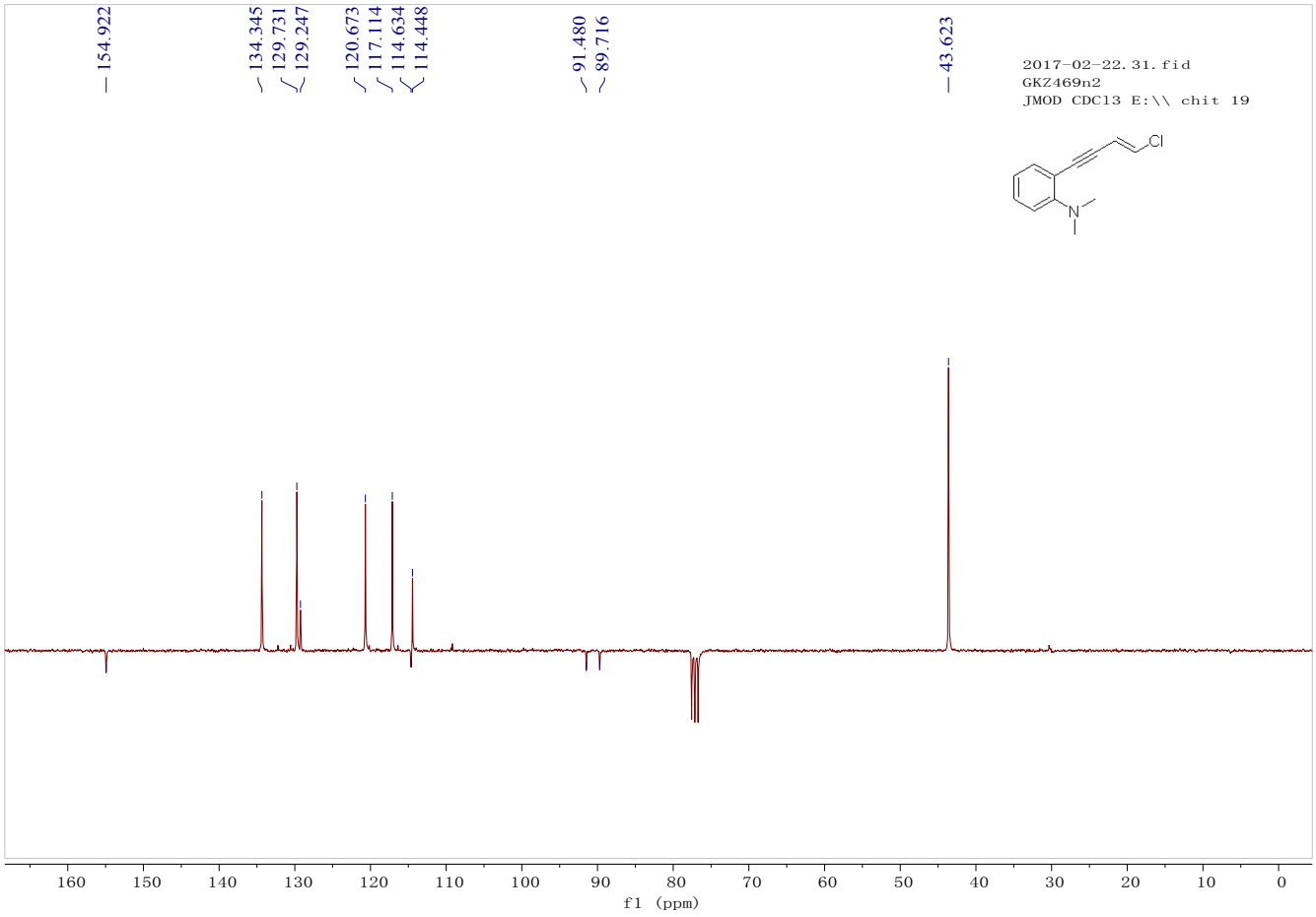
1.06
2.09
1.04
1.00
6.06
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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114.634
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JMOD CDCl3 E:\\ chit 19
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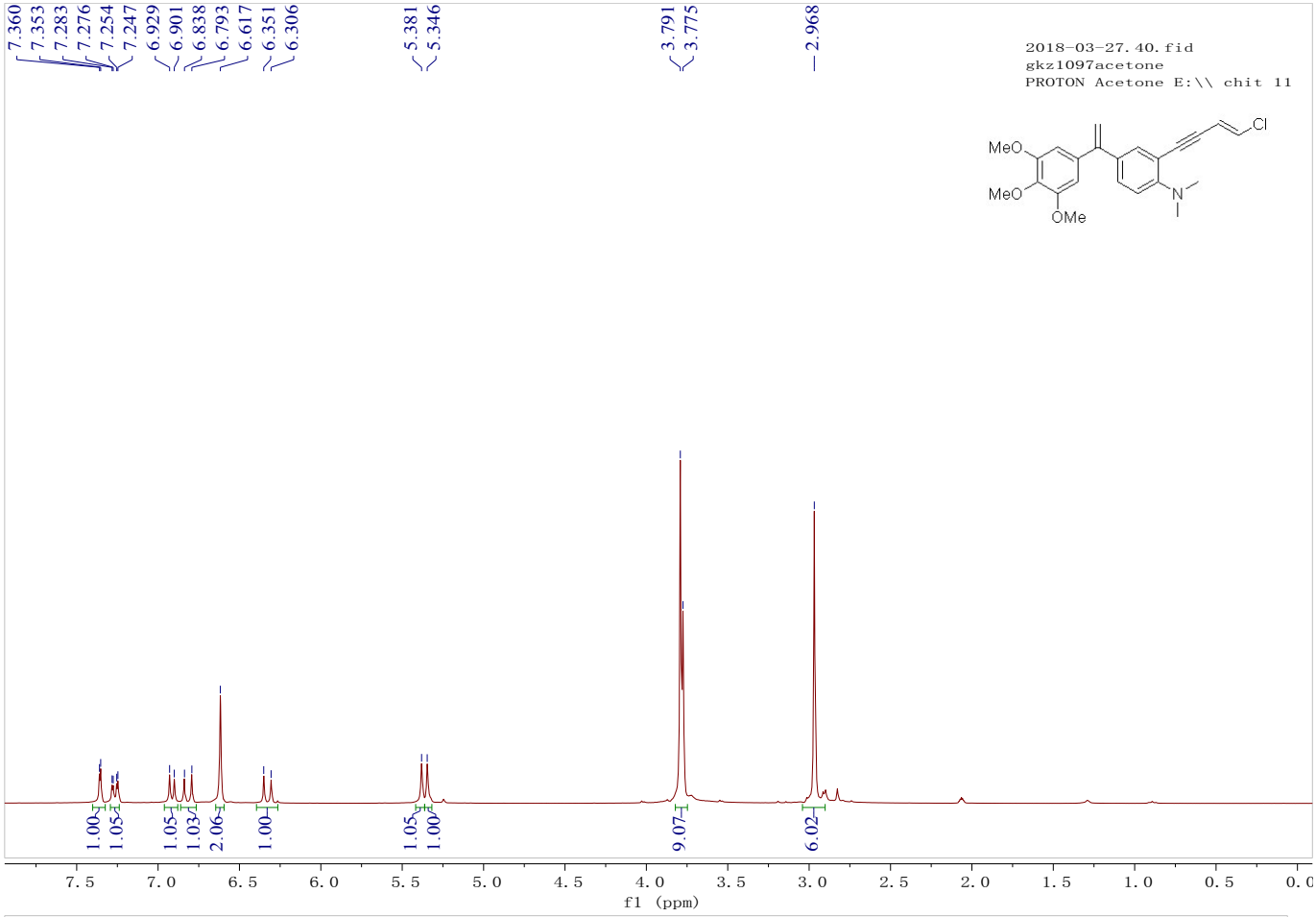
f1 (ppm)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7.254
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6.793
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6.306
5.381
5.346
3.791
3.775
2.968
2018-03-27.40.fid
gkz1097acetone
PROTON Acetone E:\\ chit 11
MeO
MeO
OMe
N
Cl
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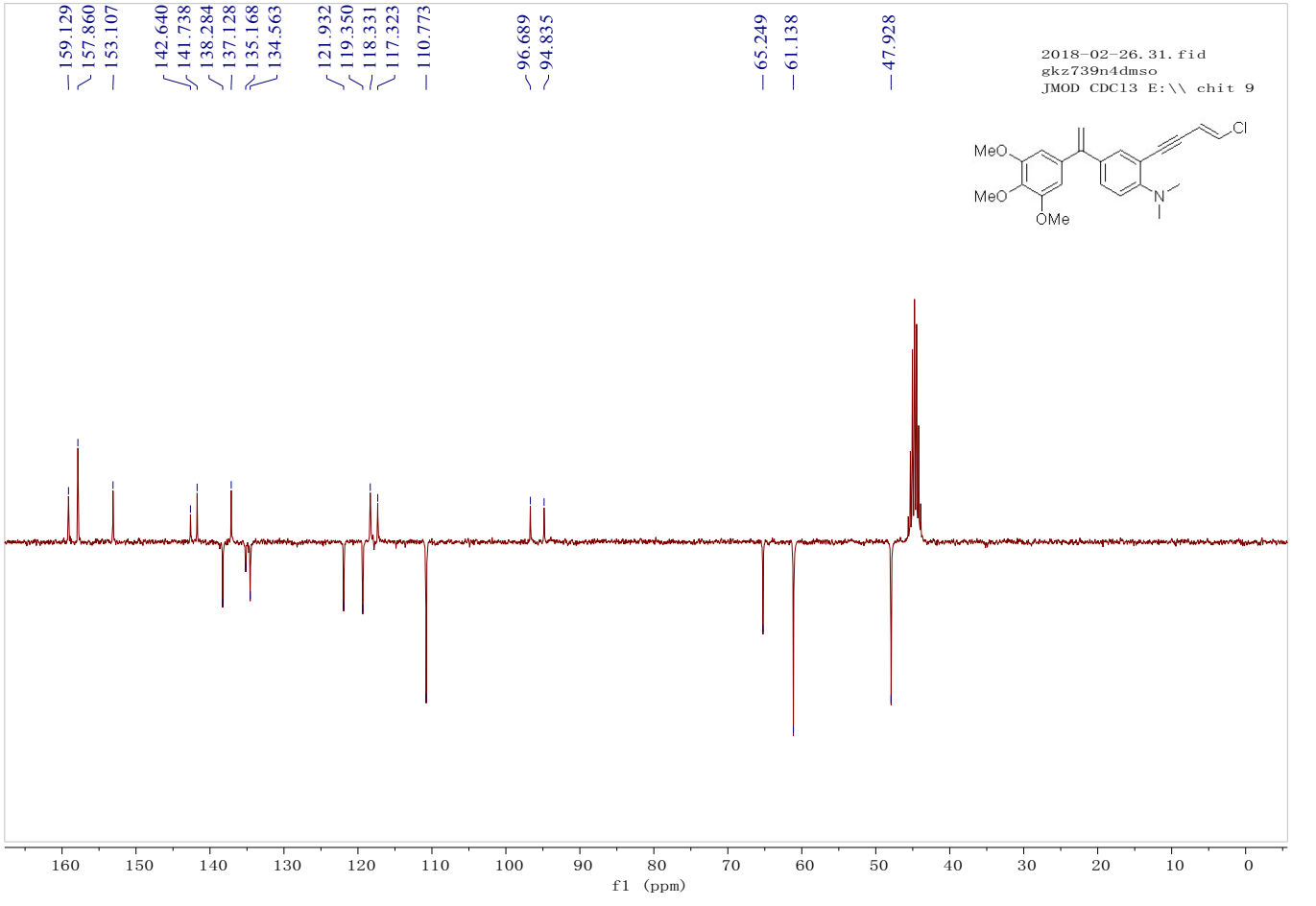
1.05
1.05
1.03
2.06
1.00
1.05
1.00
9.07
6.02
f1 (ppm)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153.107
142.640
141.738
138.284
137.128
135.168
134.563
121.932
119.350
118.331
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110.773
96.689
94.835
65.249
61.138
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2018-02-26.31.fid
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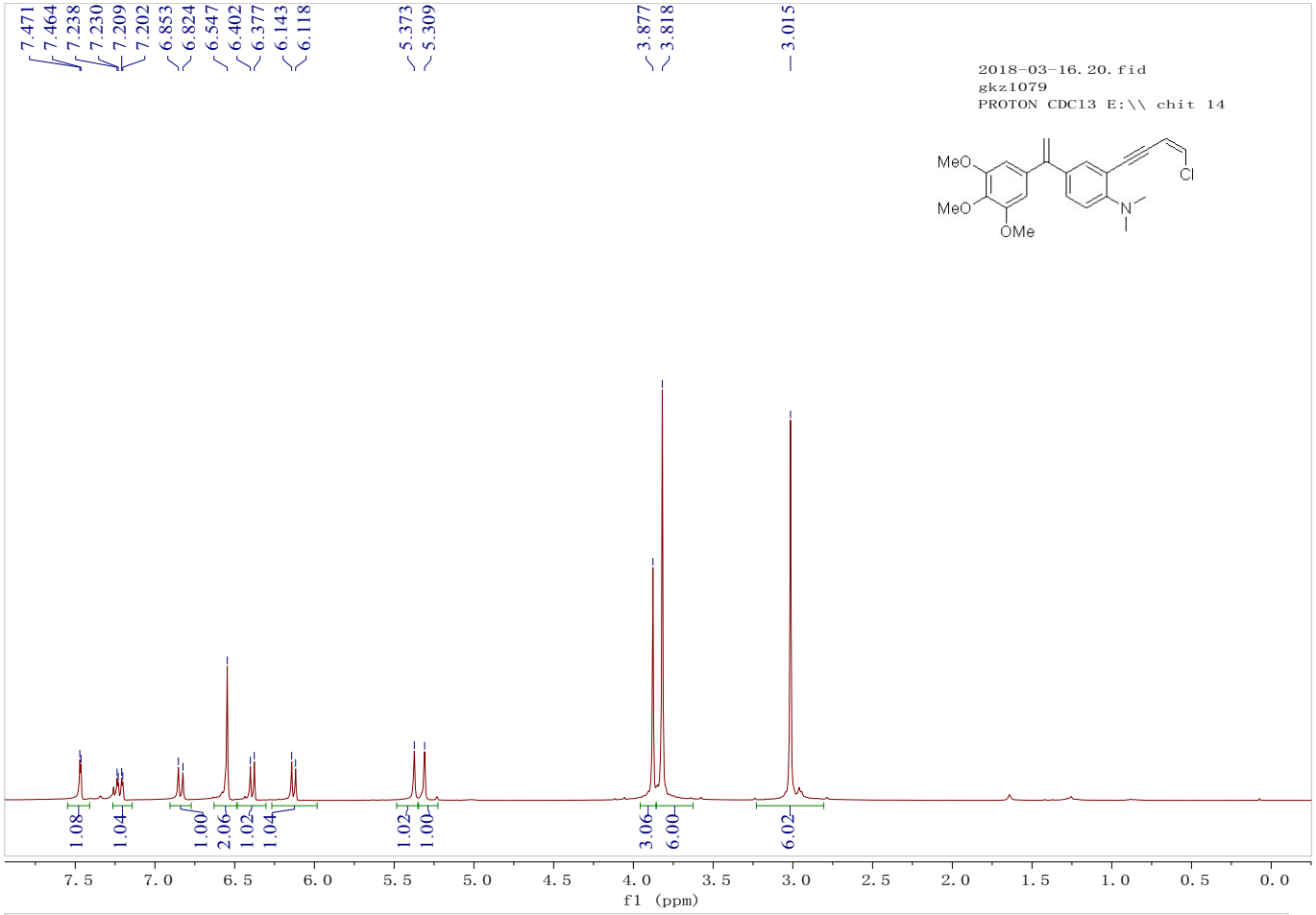
MeO
OMe
N
Cl
f1 (ppm)


7.471
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7.230
7.209
7.202
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6.824
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6.402
6.377
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6.118
5.373
5.309
3.877
3.818
3.015
2018-03-16.20.fid
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PROTON CDC13 E:\\ chit 14
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MeO
OMe
N
Cl
1.08
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1.04
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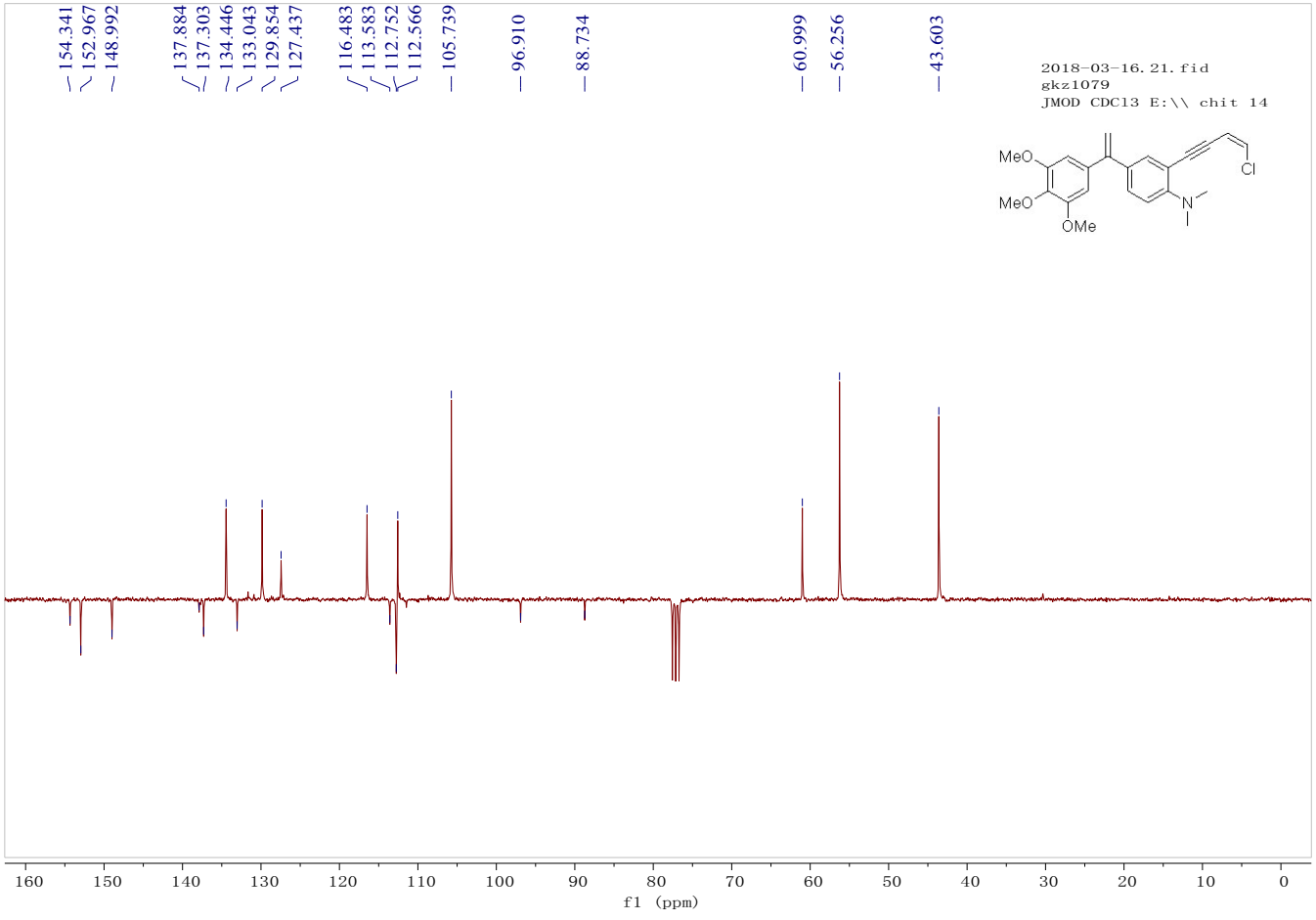
1.00
3.06
6.00
6.02
f1 (ppm)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105.739
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60.999
56.256
43.603
2018-03-16.21.fid
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MeO
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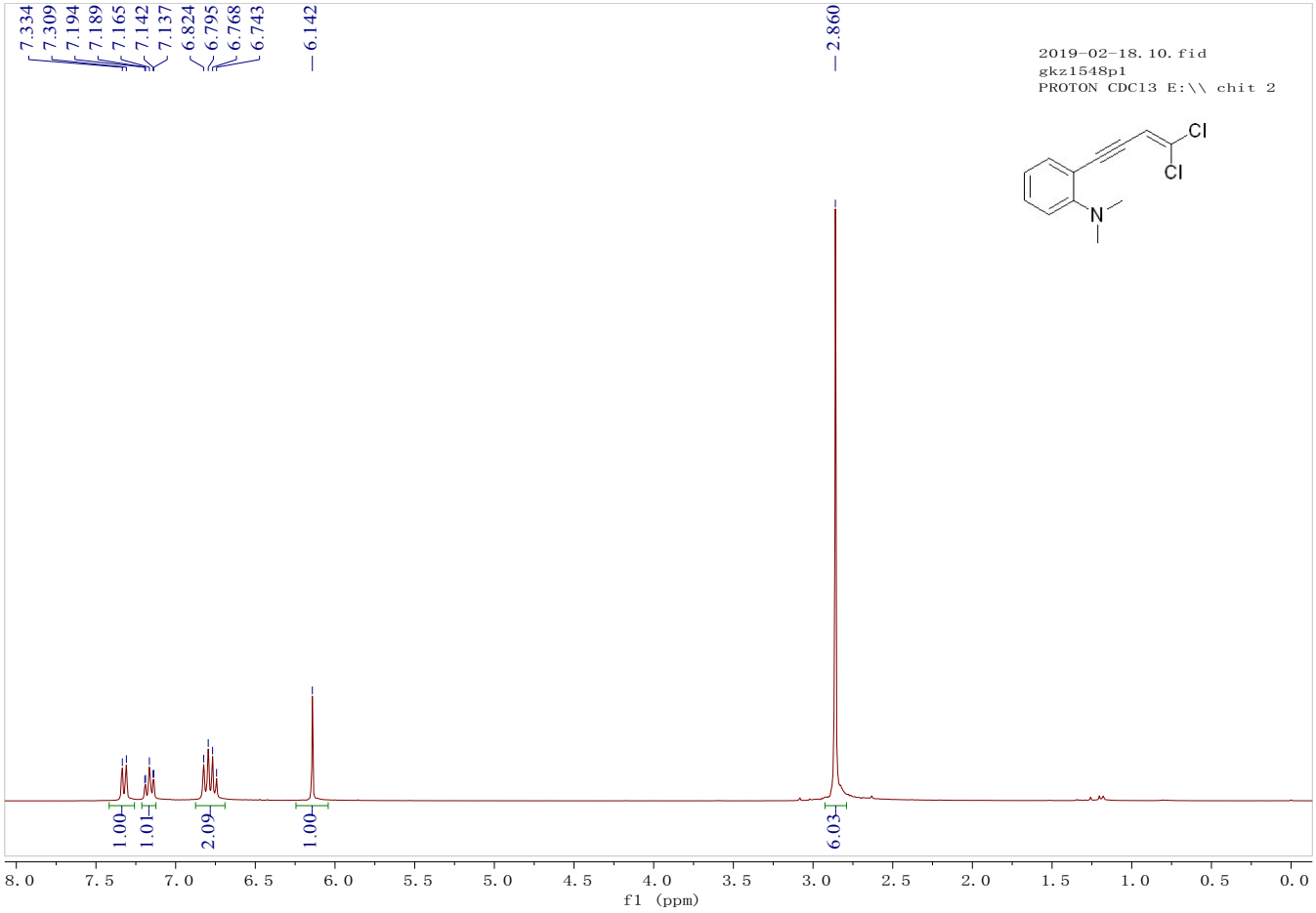
N
Cl
f1 (ppm)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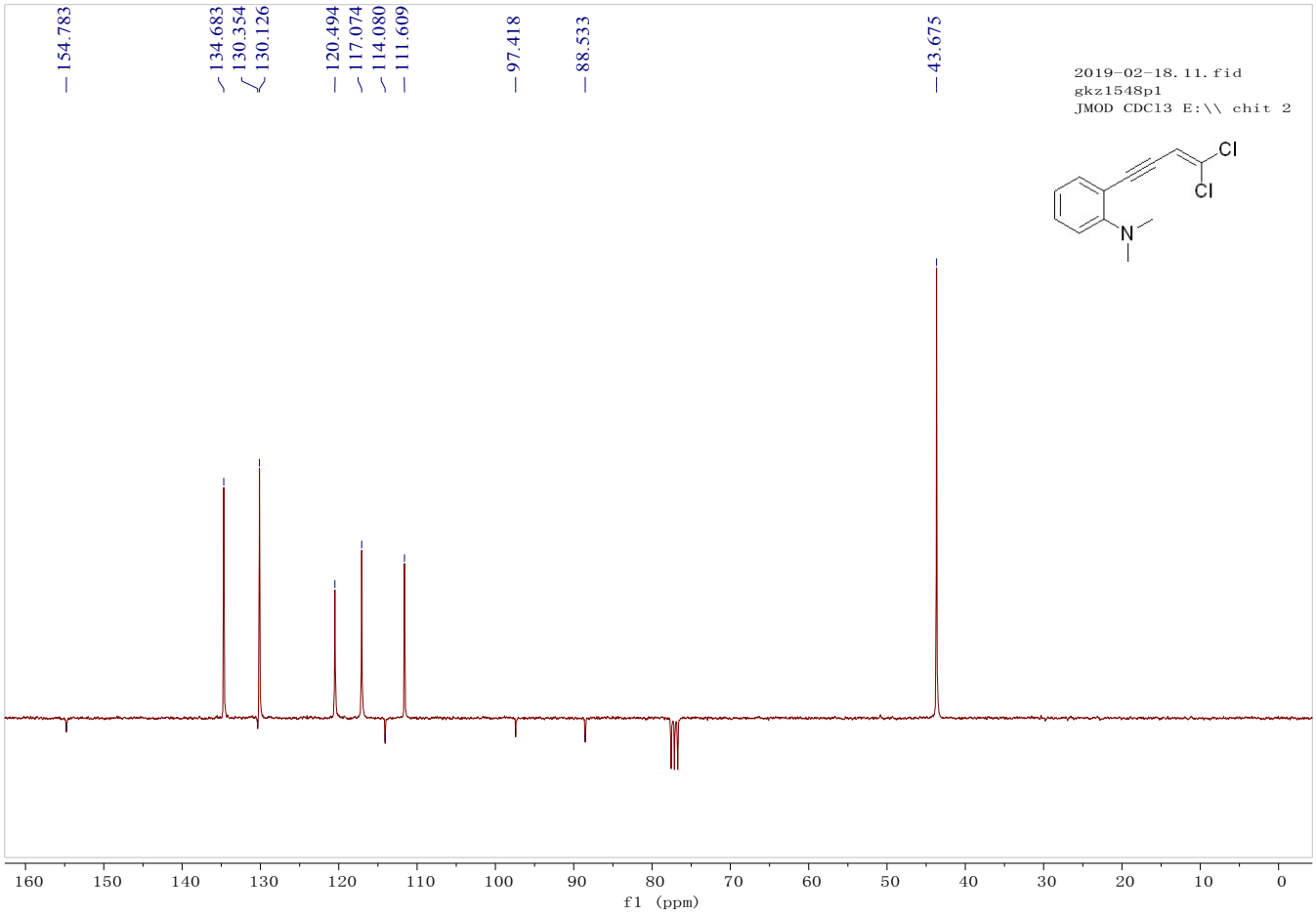
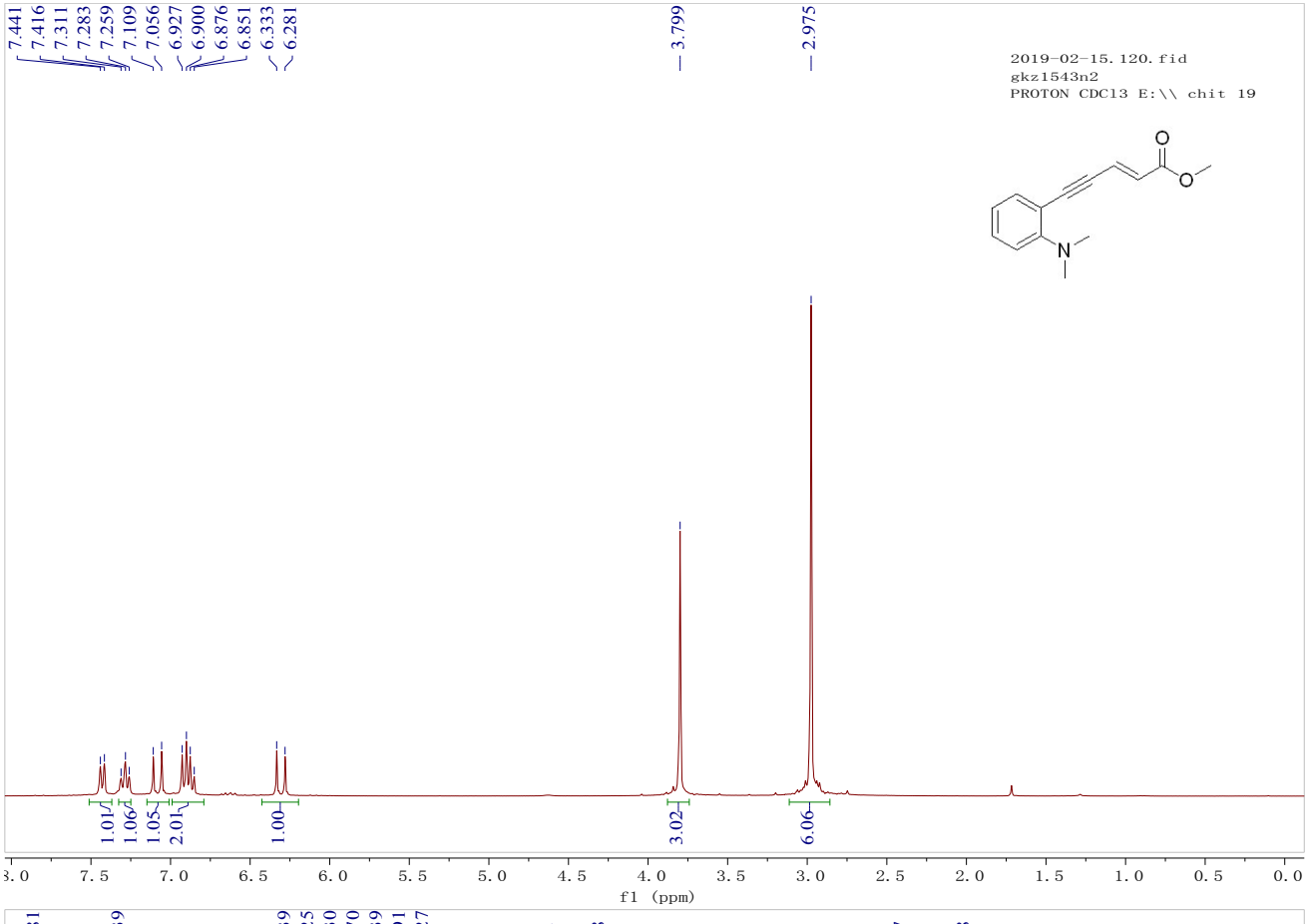
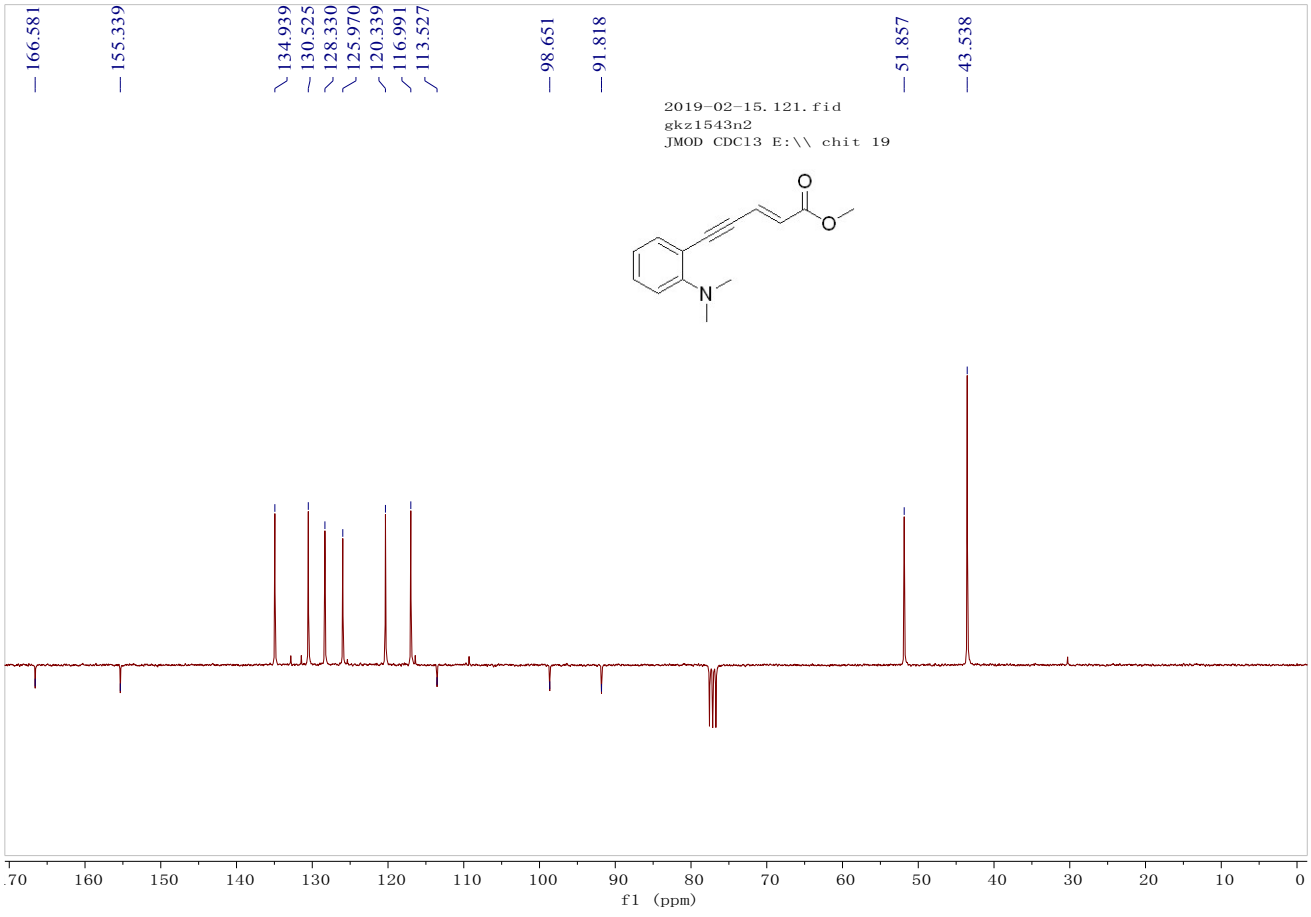
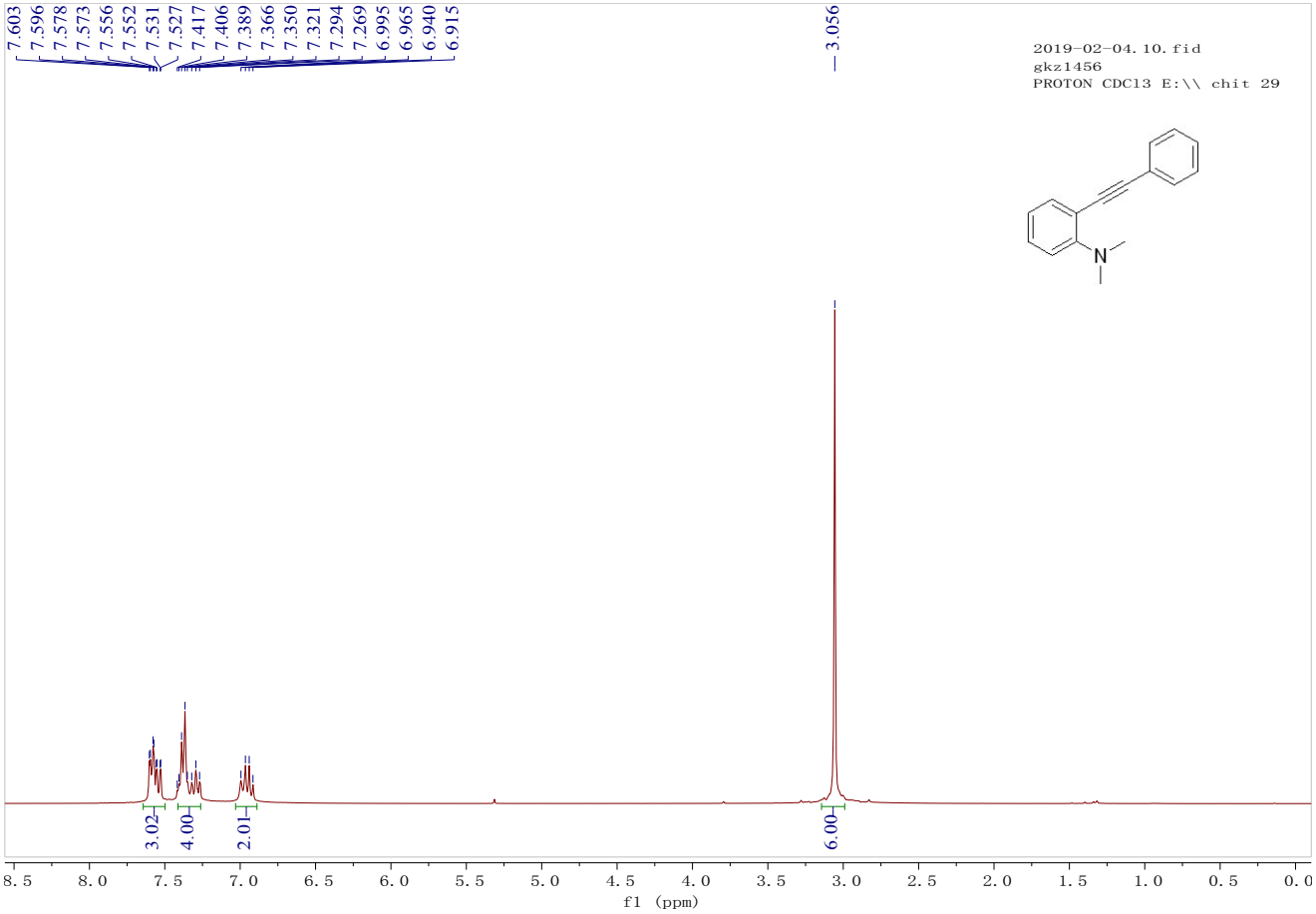
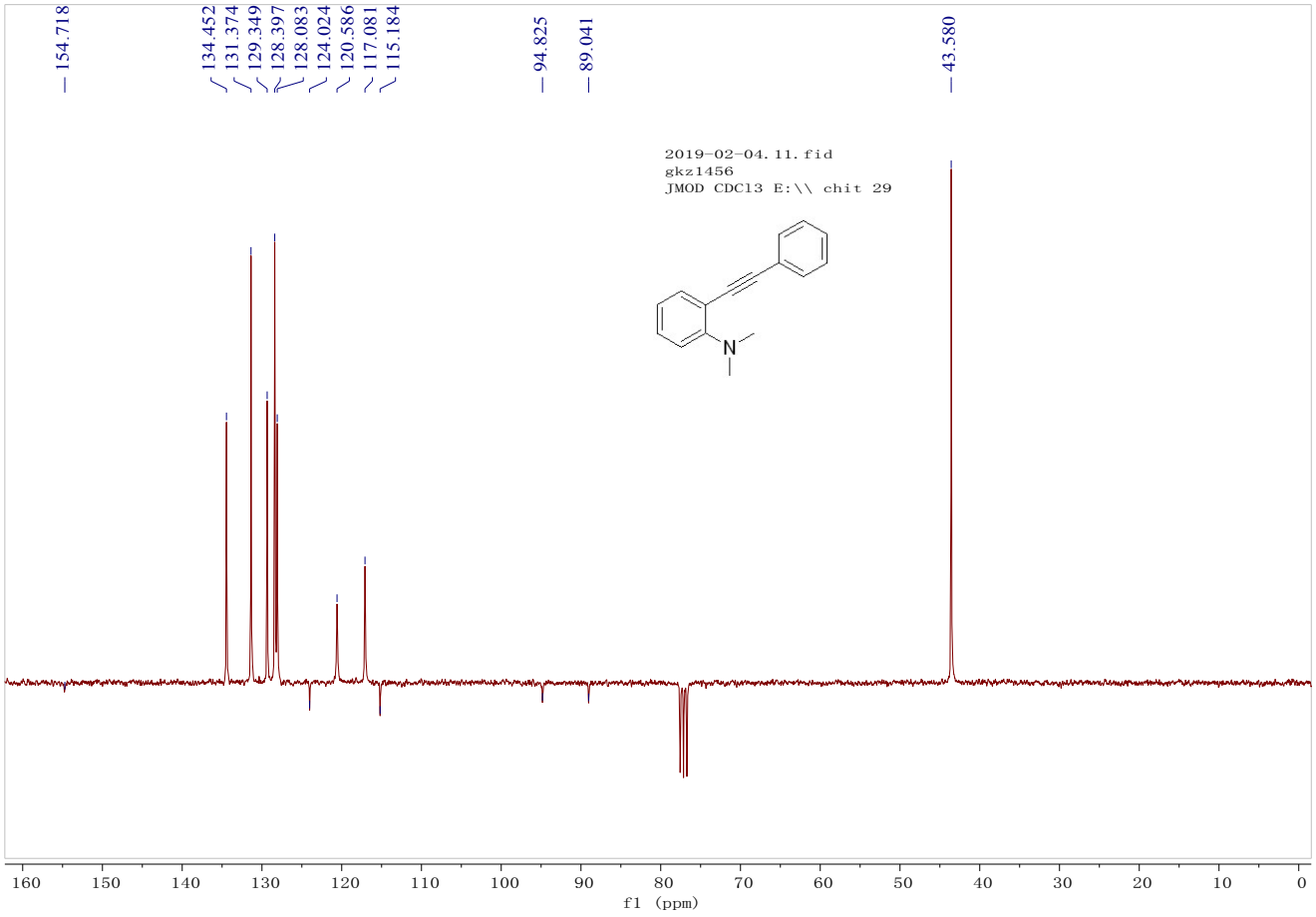
7.189
7.165
7.142
7.137
6.824
6.795
6.768
6.743
6.142
2.860
2019-02-18.10.fid
gkz1548p1
PROTON CDCl3 E:\\ chit 2
Cl
Cl
N
1.00
1.01
2.09
1.00
6.03
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

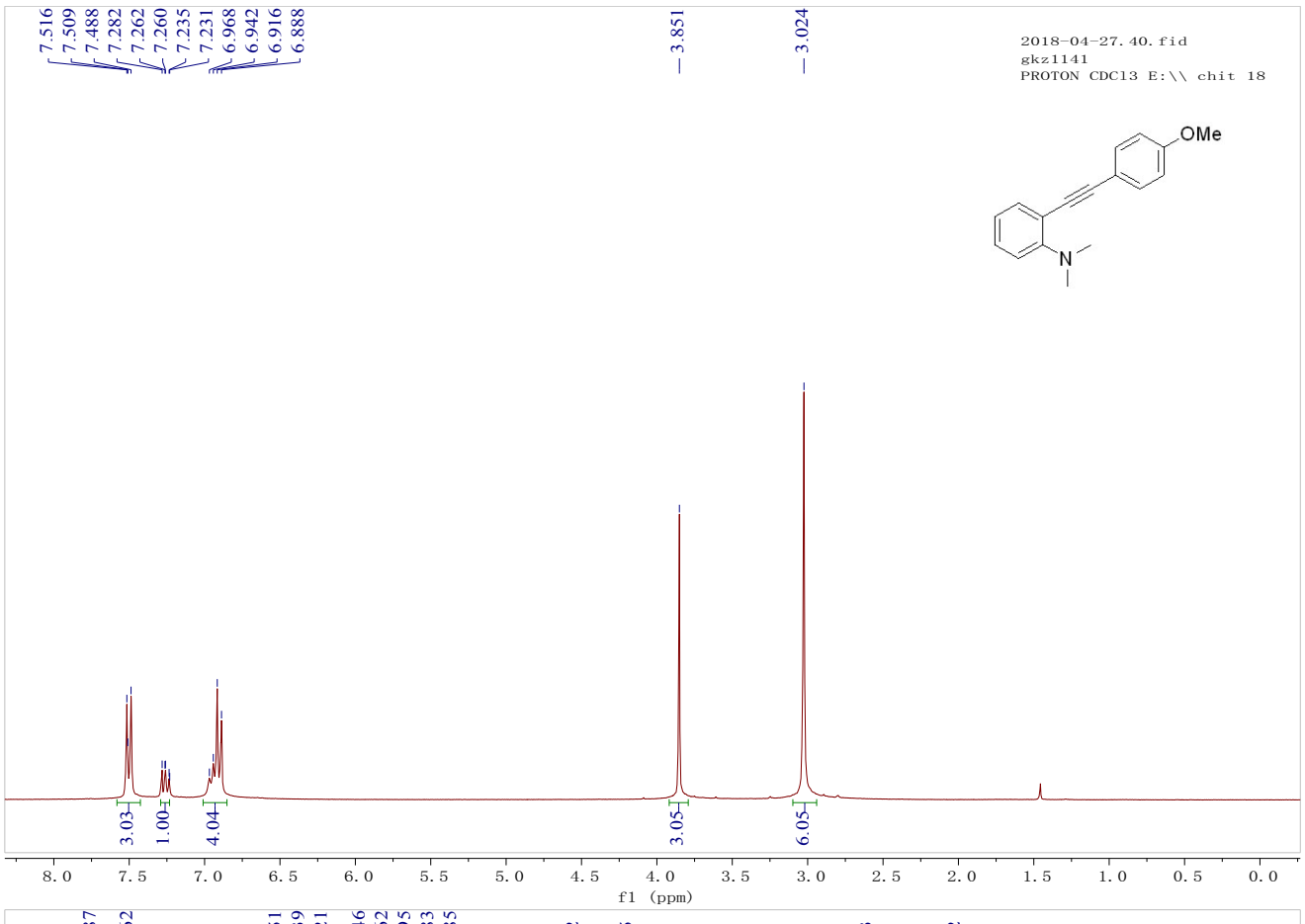

154.783
134.683
130.354
130.126
120.494
117.074
114.080
111.609
97.418
88.533
43.675
2019-02-18.11.fid
gkz1548p1
JMOD CDCl3 E:\\ chit 2
Cl
Cl
N
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

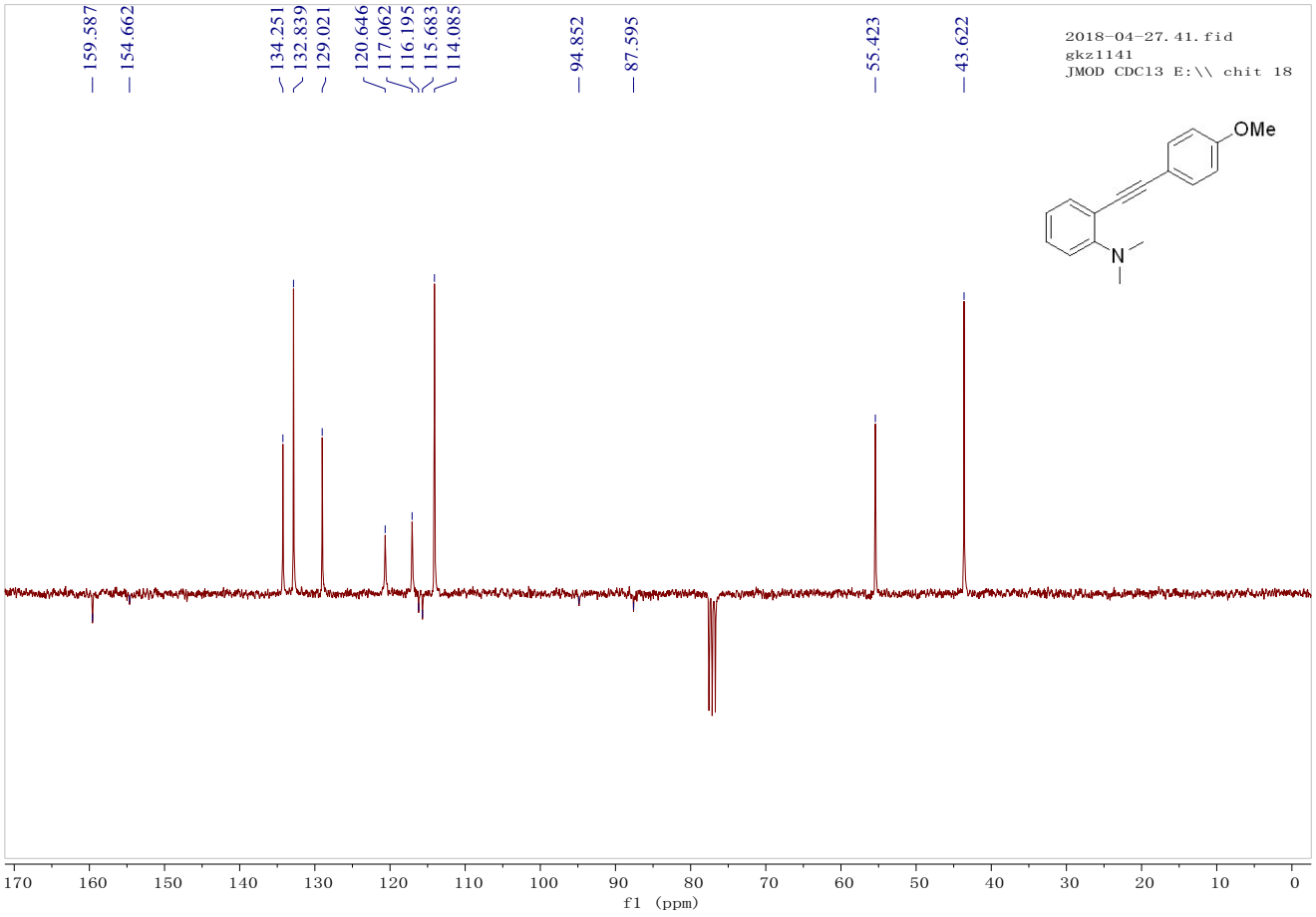

7.441
7.416
7.311
7.283
7.259
7.109
7.056
6.927
6.900
6.876
6.851
6.333
6.281
3.799
2.975
2019-02-15.120.fid
gkz1543n2
PROTON CDCl3 E:\\ chit 19
O
O
N
1.01
1.06
1.05
2.01
1.00
3.02
6.06
f1 (ppm)

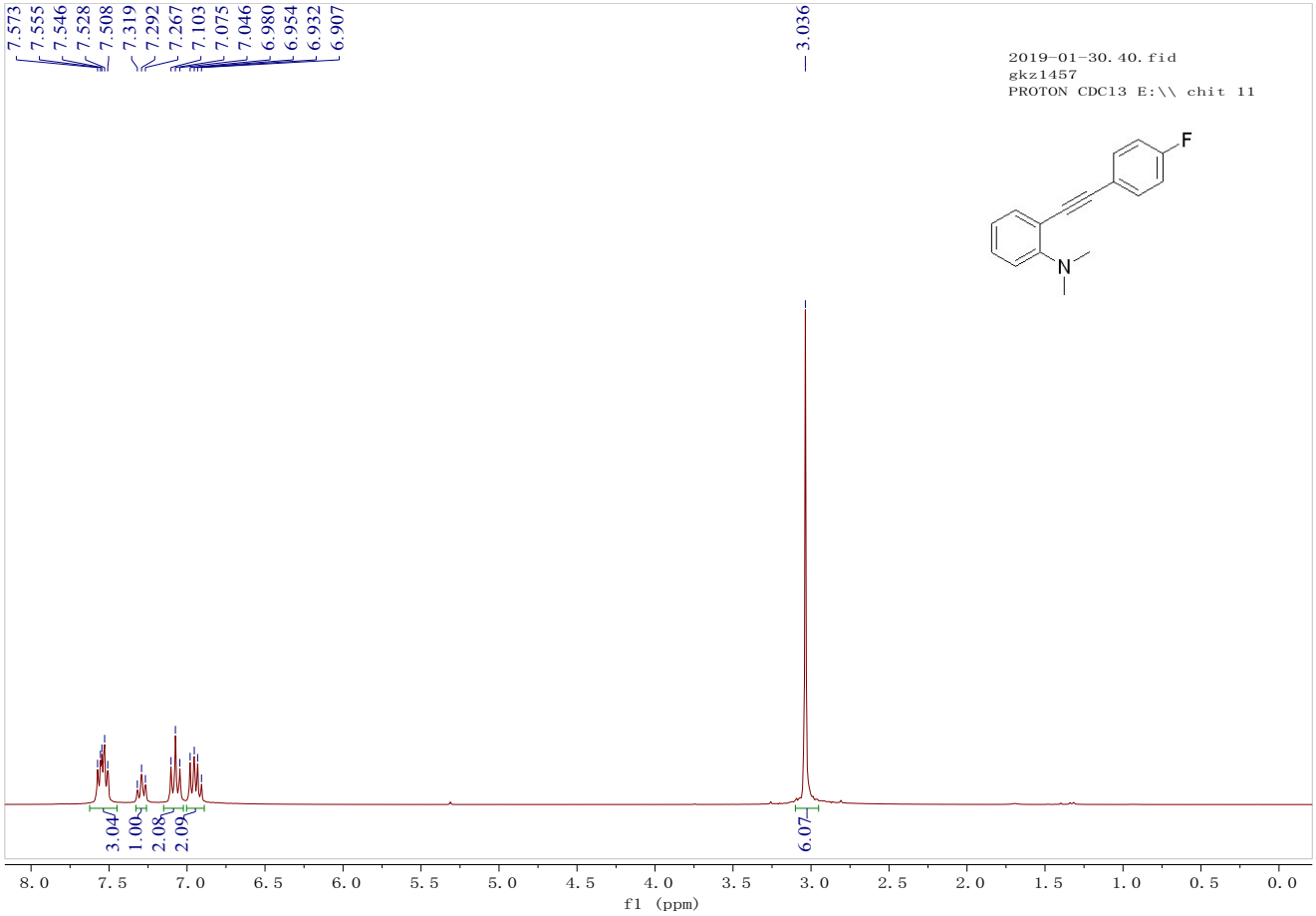

166.581
155.339
134.939
130.525
128.330
125.970
120.339
116.991
113.527
98.651
91.818
51.857
43.538
2019-02-15.121.fid
gkz1543n2
JMOD CDCl3 E:\\ chit 19
O
O
N
f1 (ppm)

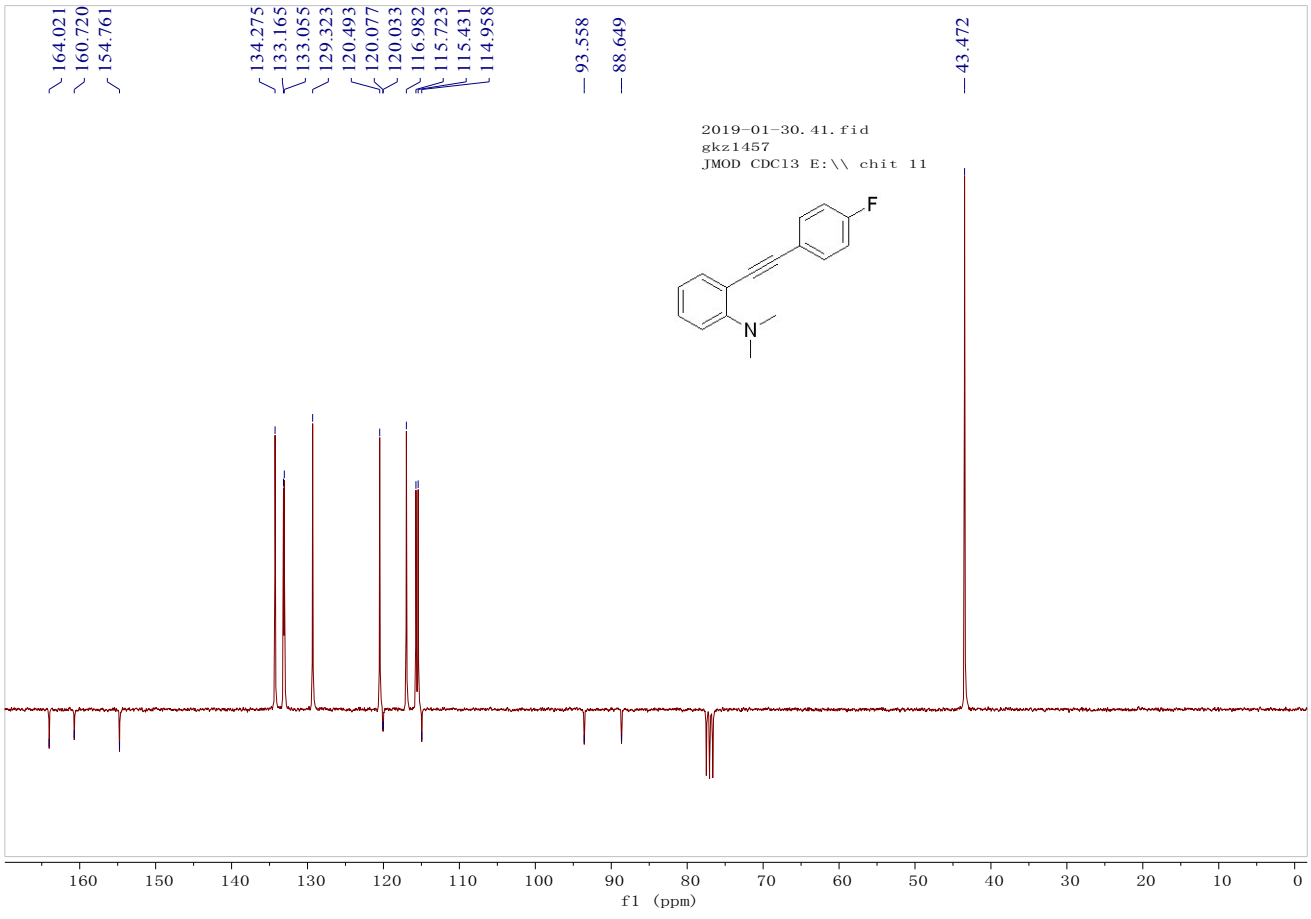

7.603
7.596
7.578
7.573
7.556
7.552
7.531
7.527
7.417
7.406
7.389
7.366
7.350
7.321
7.294
7.269
6.995
6.965
6.940
6.915
3.056
2019-02-04.10.fid
gkz1456
PROTON CDC13 E:\\ chit 29
3.02
4.00
2.01
6.00
f1 (ppm)

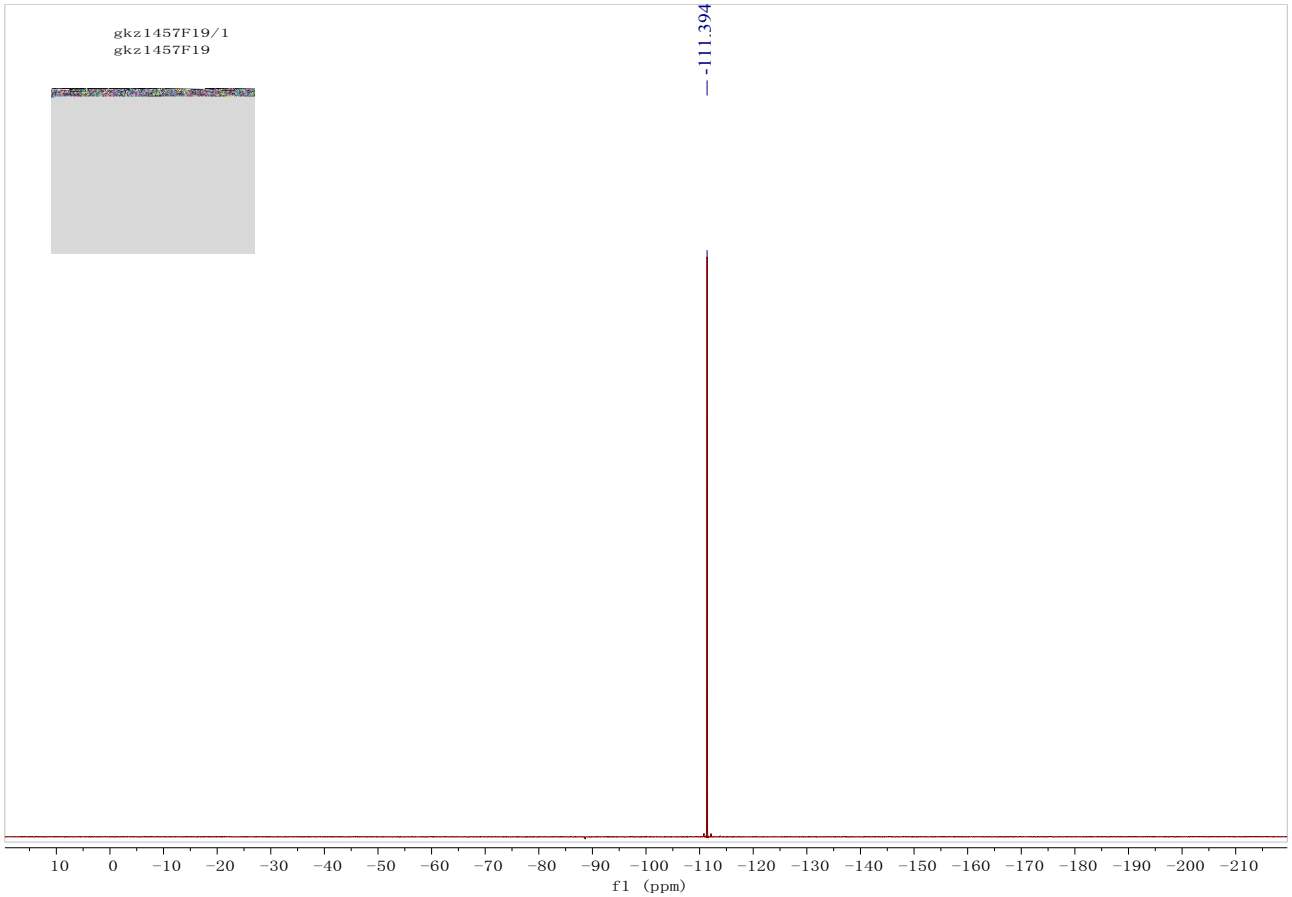

154.718
134.452
131.374
129.349
128.397
128.083
124.024
120.586
117.081
115.184
94.825
89.041
43.580
2019-02-04.11.fid
gkz1456
JMOD CDC13 E:\\ chit 29
f1 (ppm)

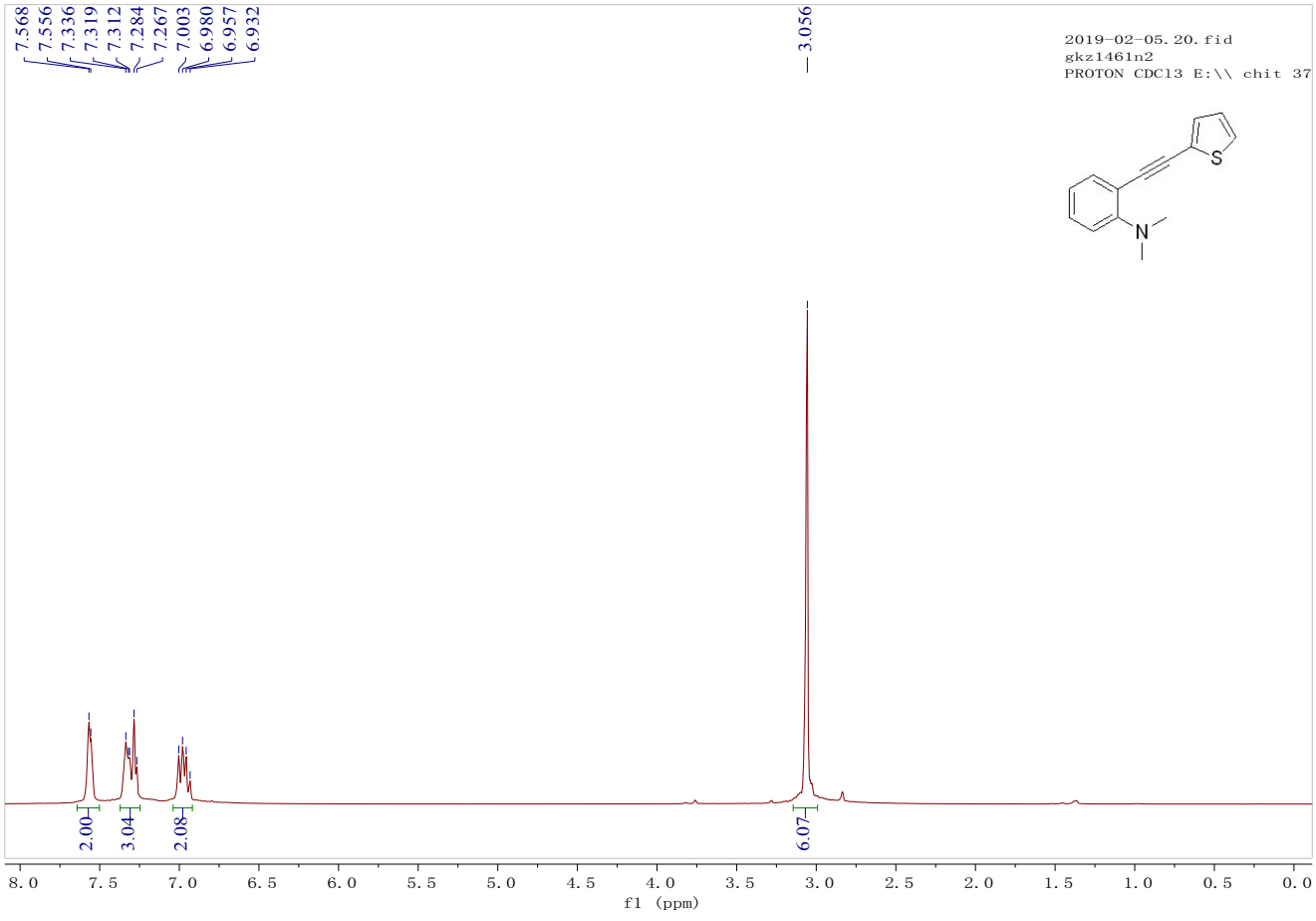

7.516
7.509
7.488
7.282
7.262
7.260
7.235
7.231
6.968
6.942
6.916
6.888
3.851
3.024
2018-04-27.40.fid
gkz1141
PROTON CDCl3 E:\\ chit 18
OMe
N
3.03
1.00
4.04
3.05
6.05
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

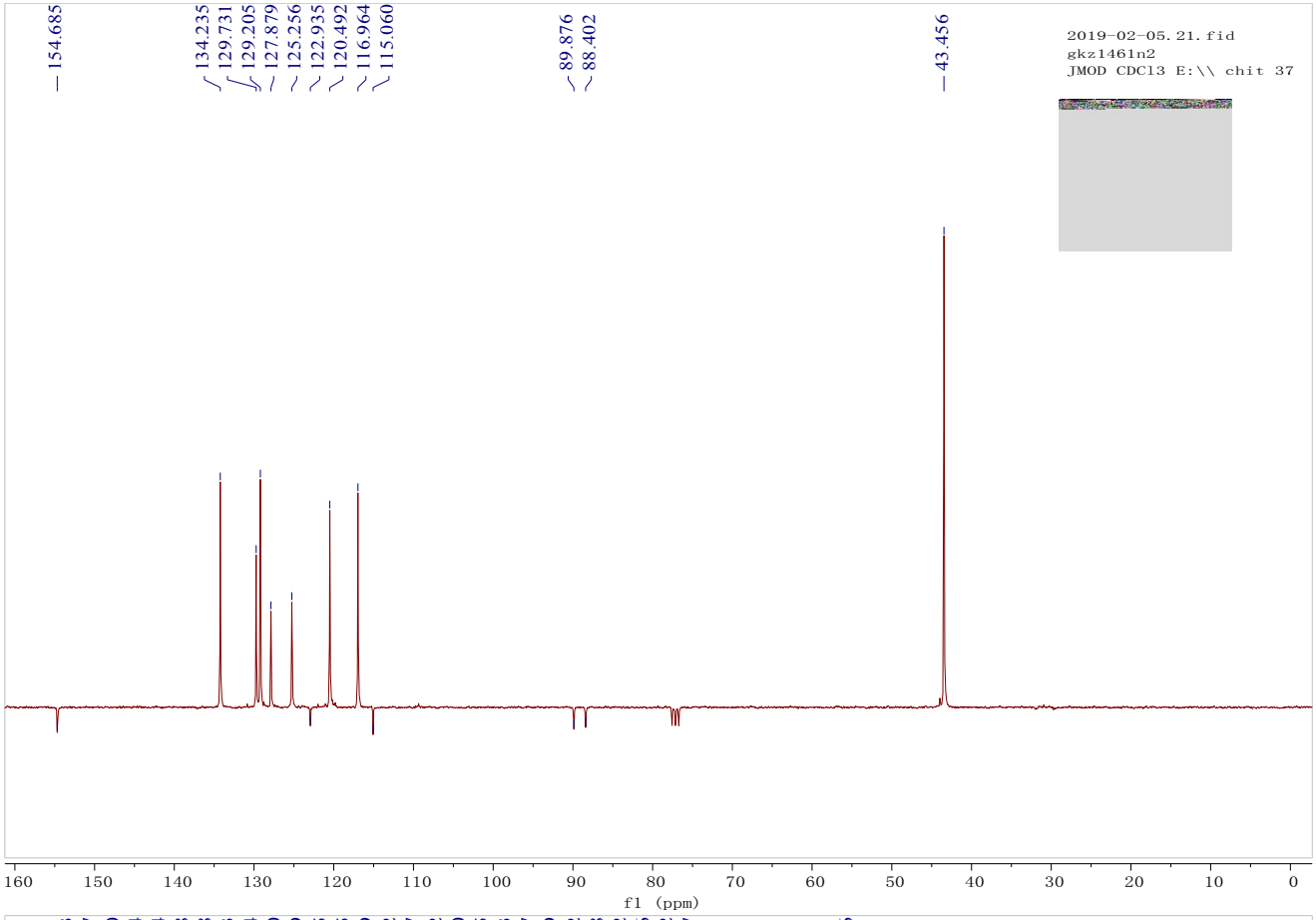

159.587
154.662
134.251
132.839
129.021
120.646
117.062
116.195
115.683
114.085
94.852
87.595
55.423
43.622
2018-04-27.41.fid
gkz1141
JMOD CDCl3 E:\\ chit 18
OMe
N
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

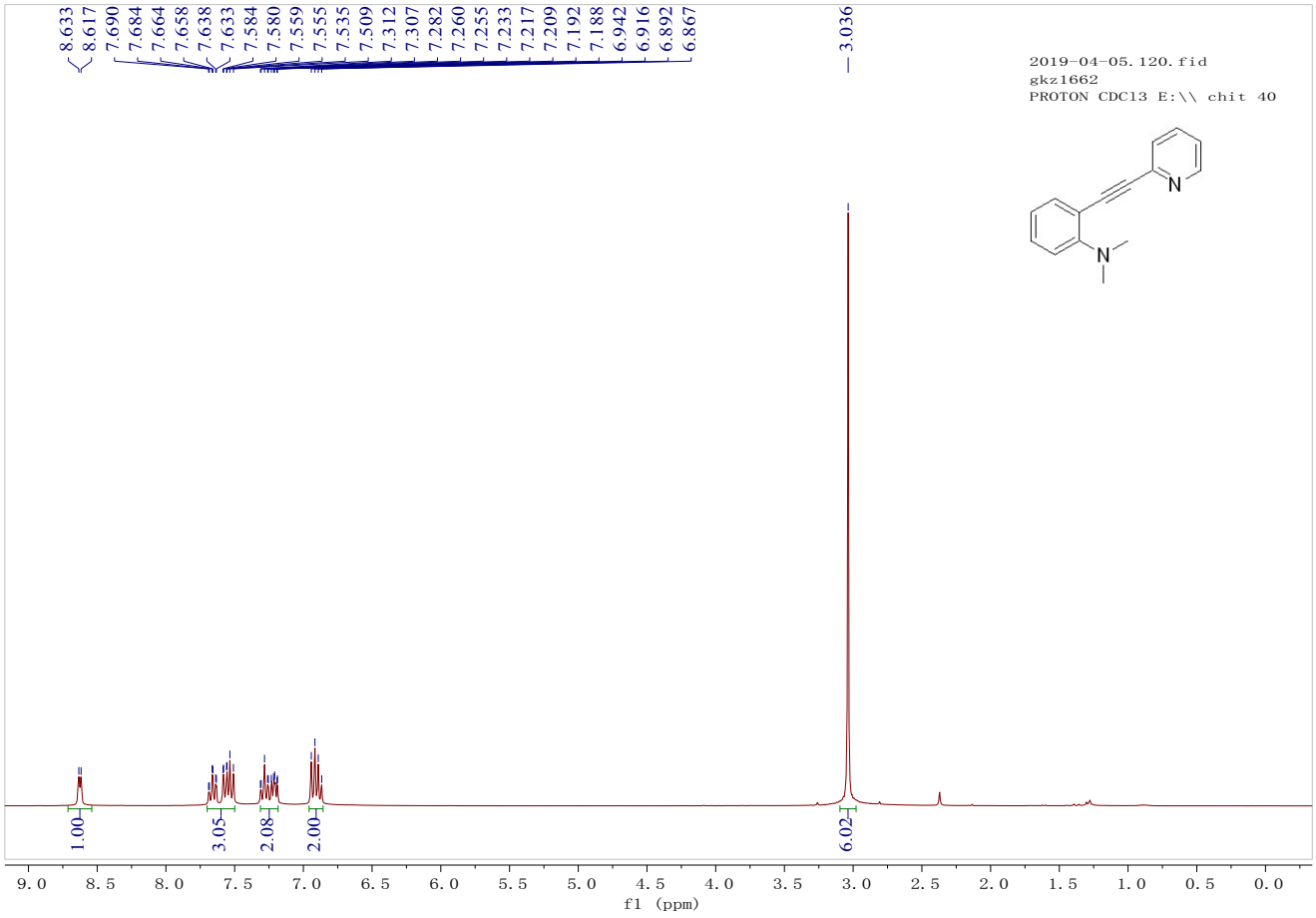

7.573
7.555
7.546
7.528
7.508
7.319
7.292
7.267
7.103
7.075
7.046
6.980
6.954
6.932
6.907
3.036
2019-01-30.40.fid
gkz1457
PROTON CDCl3 E:\\ chit 11
F
N
3.04
1.00
2.08
2.09
6.07
f1 (ppm)

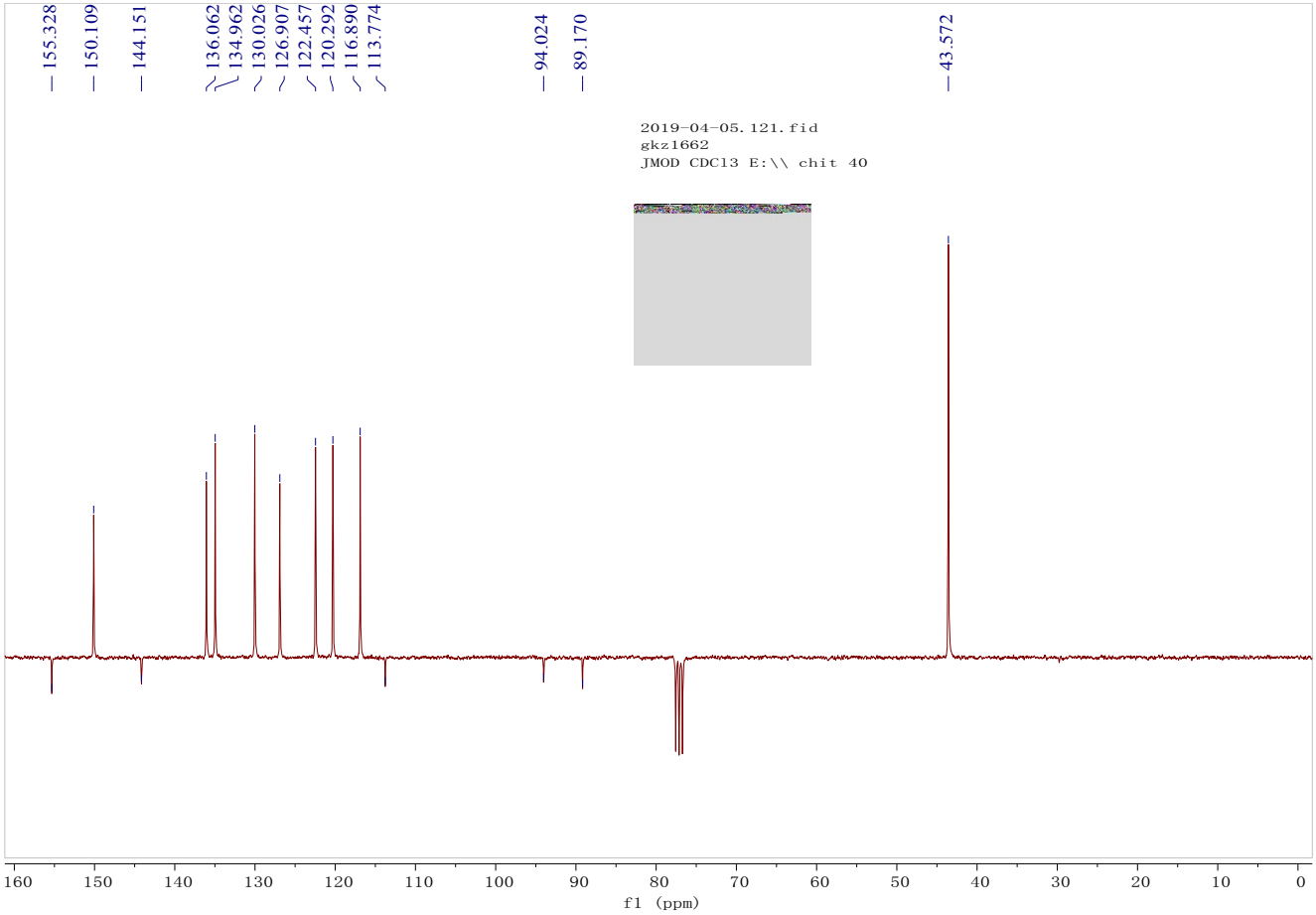

164.021
160.720
154.761
134.275
133.165
133.055
129.323
120.493
120.077
120.033
116.982
115.723
115.431
114.958
93.558
88.649
43.472
2019-01-30.41.fid
gkz1457
JMOD CDCl3 E:\\ chit 11
F
N
f1 (ppm)

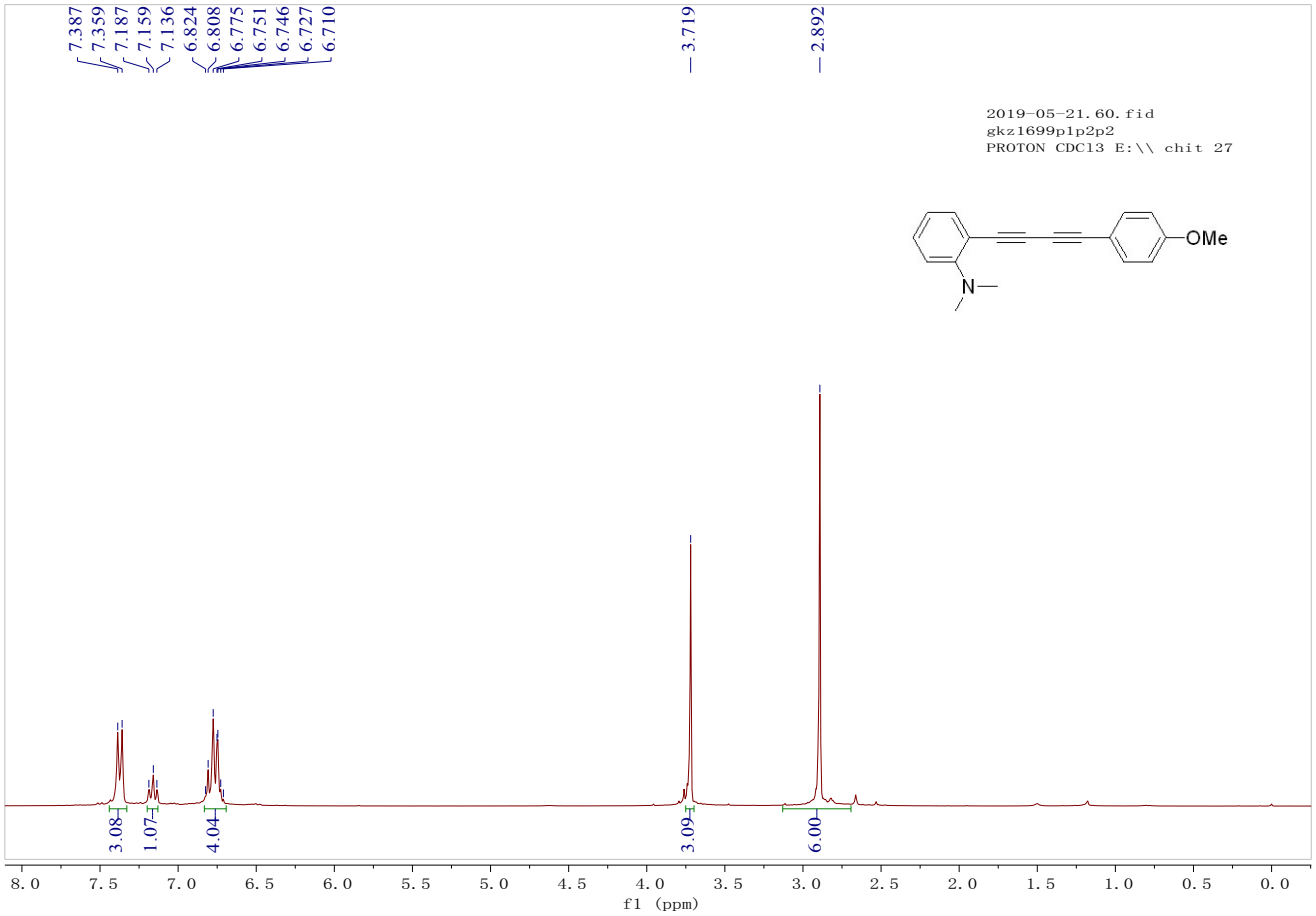

gkz1457F19/1
gkz1457F19
-111.394
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

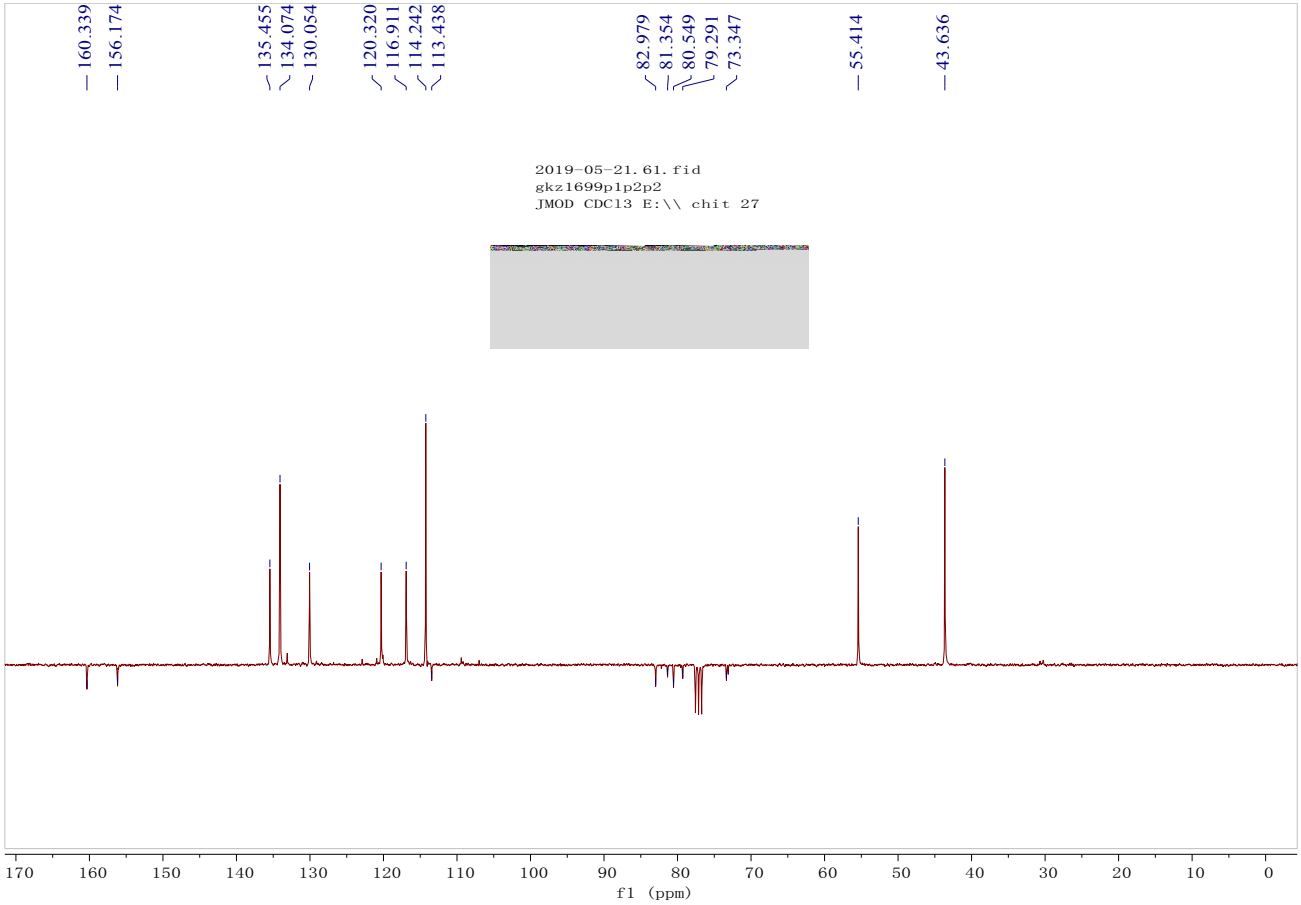

7.568
7.556
7.336
7.319
7.312
7.284
7.267
7.003
6.980
6.957
6.932
3.056
2019-02-05.20.fid
gkz1461n2
PROTON CDCl3 E:\\ chit 37
S
N
2.00
3.04
2.08
6.07
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

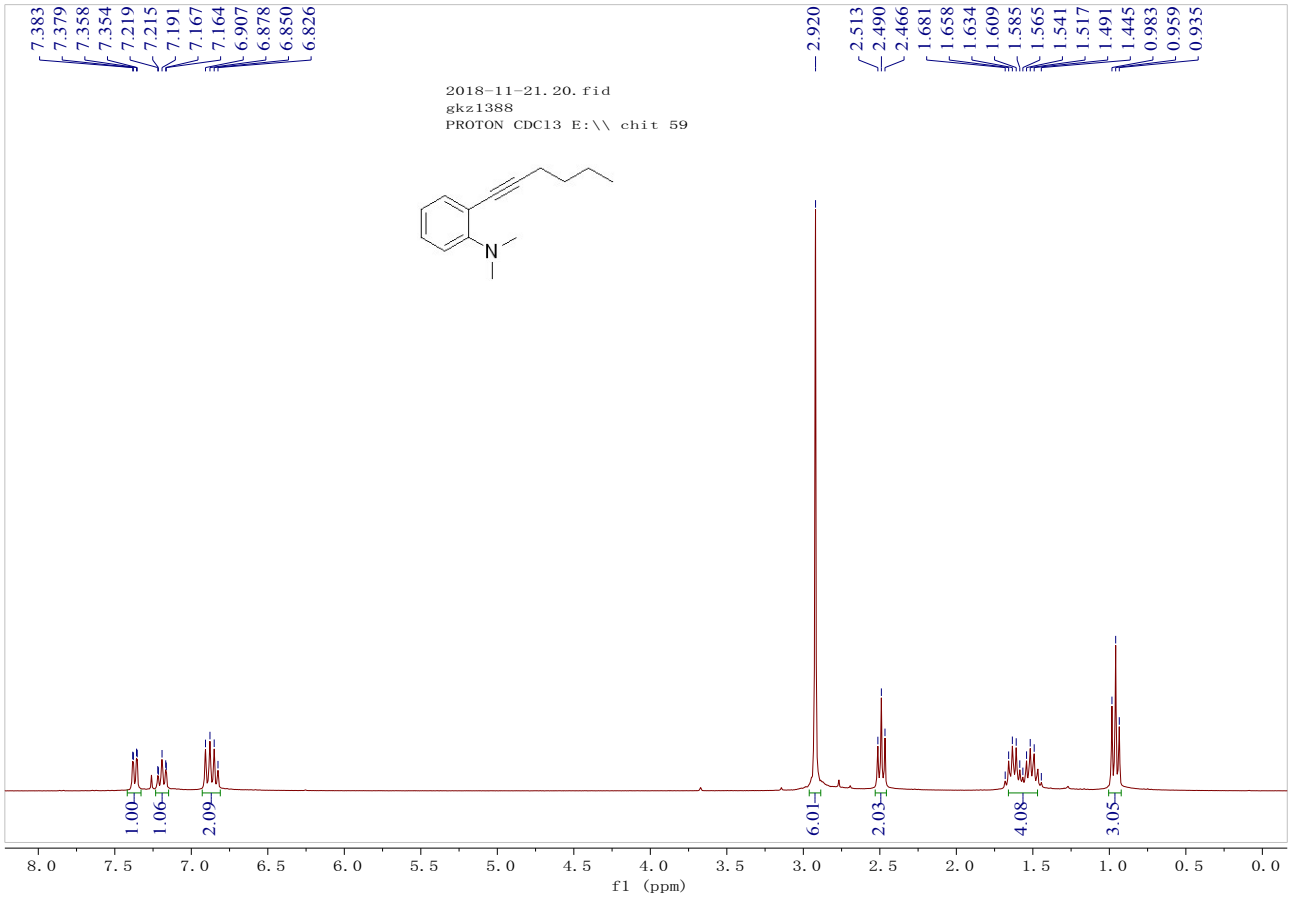

154.685
134.235
129.731
129.205
127.879
125.256
122.935
120.492
116.964
115.060
89.876
88.402
43.456
2019-02-05.21.fid
gkz1461n2
JMOD CDCl3 E:\\ chit 37
f1 (ppm)

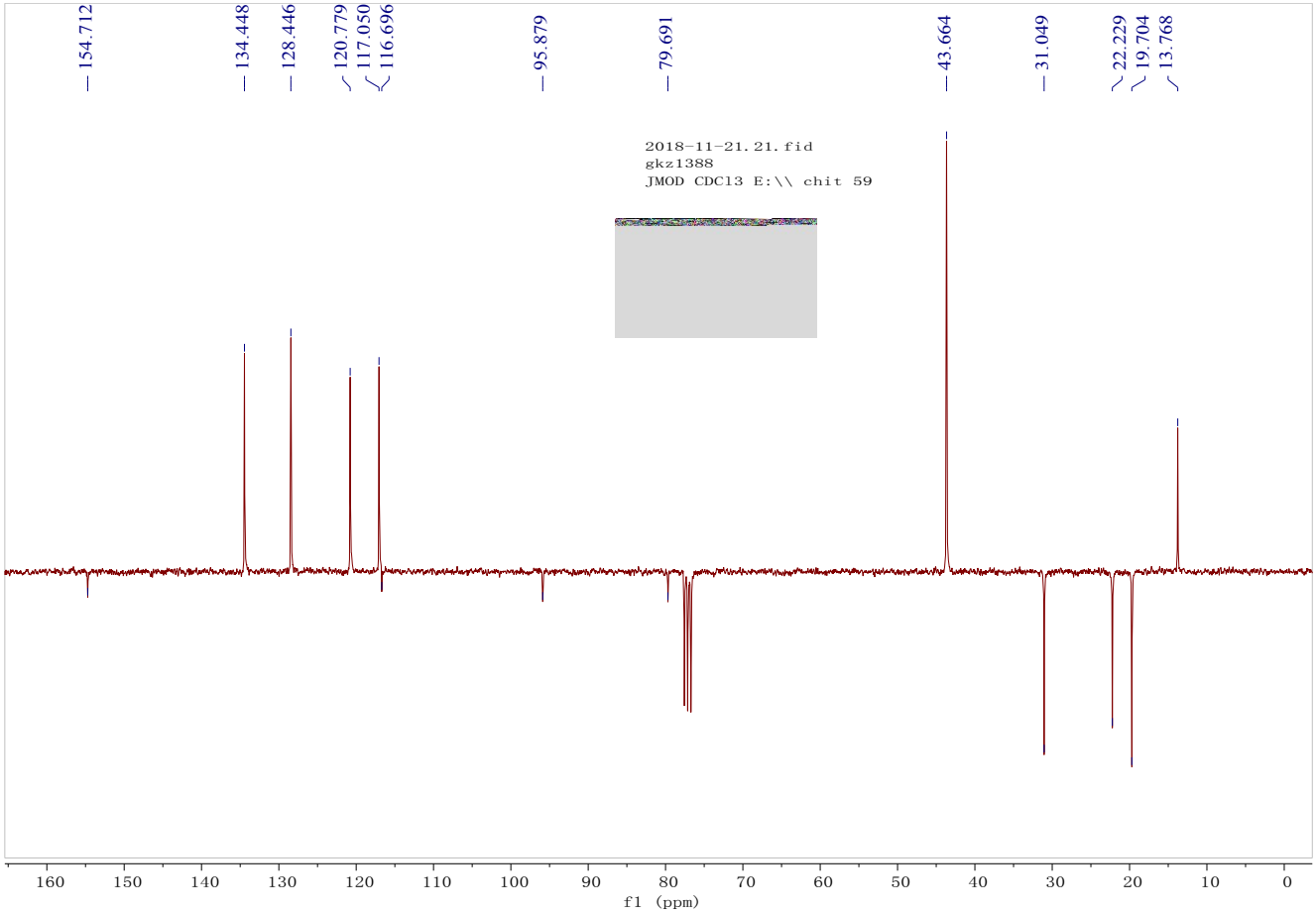

8.633
8.617
7.690
7.684
7.664
7.658
7.638
7.633
7.584
7.580
7.559
7.555
7.535
7.509
7.312
7.307
7.282
7.260
7.255
7.233
7.217
7.209
7.192
7.188
6.942
6.916
6.892
6.867
3.036
2019-04-05.120.fid
gkz1662
PROTON CDCl3 E:\\ chit 40
N
N
1.00
3.05
2.08
2.00
6.02
f1 (ppm)

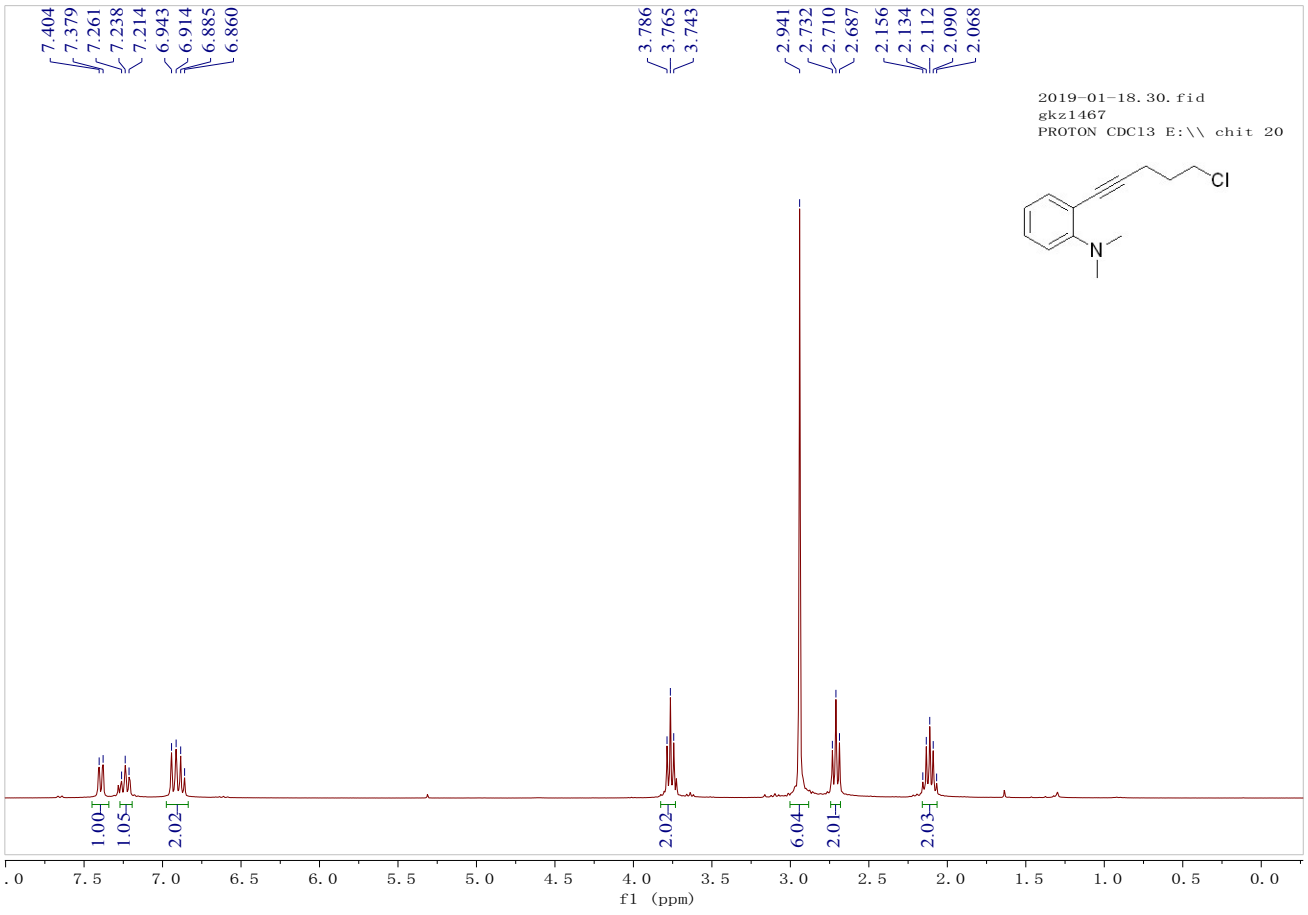

155.328
150.109
144.151
136.062
134.962
130.026
126.907
122.457
120.292
116.890
113.774
94.024
89.170
43.572
2019-04-05.121.fid
gkz1662
JMOD CDCl3 E:\\ chit 40
f1 (ppm)

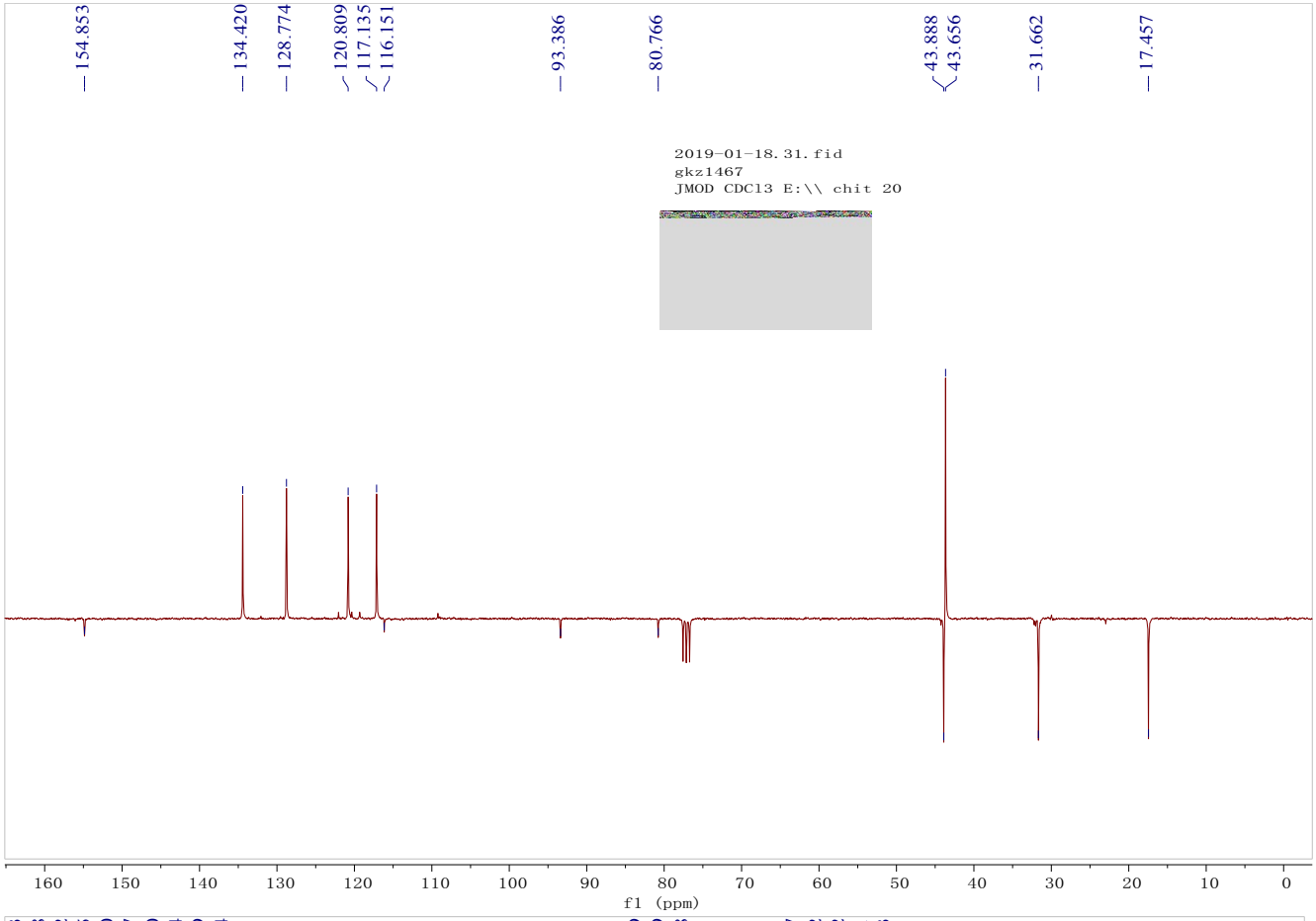

7.387
7.359
7.187
7.159
7.136
6.824
6.808
6.775
6.751
6.746
6.727
6.710
3.719
2.892
2019-05-21.60.fid
gkz1699p1p2p2
PROTON CDCl3 E:\\ chit 27
OMe
N
3.08
1.07
4.04
3.09
6.00
f1 (ppm)

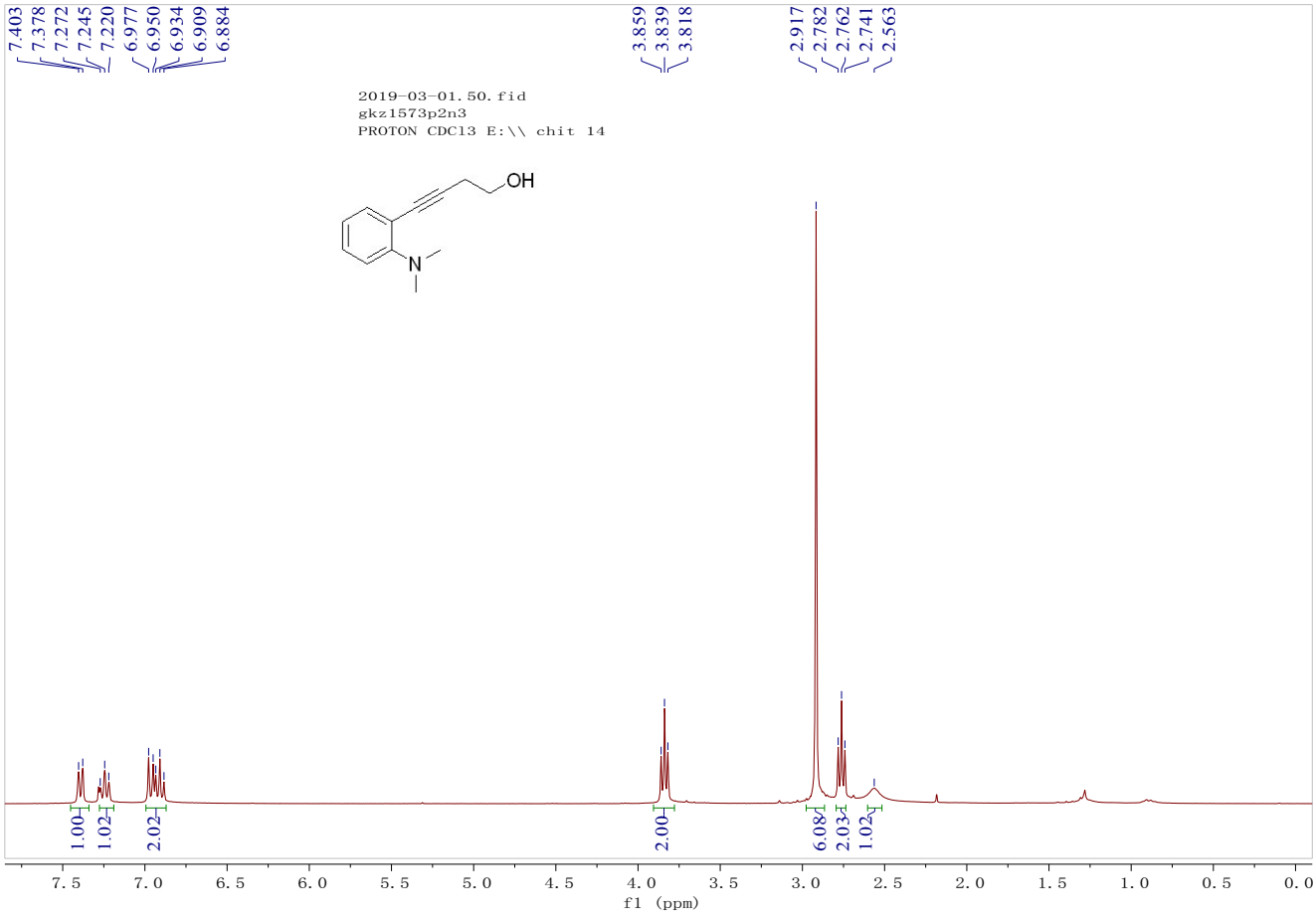

160.339
156.174
135.455
134.074
130.054
120.320
116.911
114.242
113.438
82.979
81.354
80.549
79.291
73.347
55.414
43.636
2019-05-21.61.fid
gkz1699p1p2p2
JMOD CDC13 E:\\ chit 27
f1 (ppm)

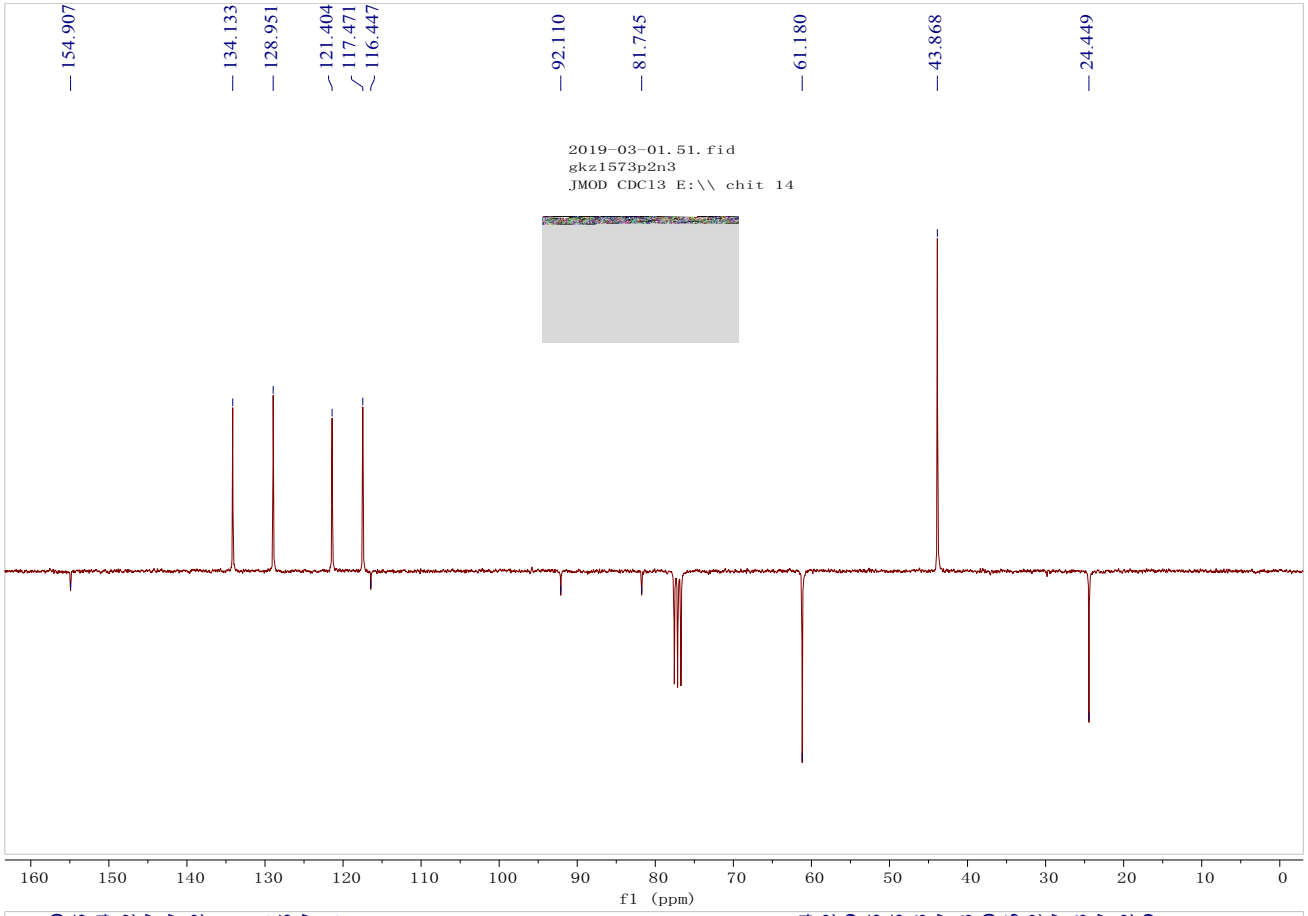

7.383
7.379
7.358
7.354
7.219
7.215
7.191
7.167
7.164
6.907
6.878
6.850
6.826
2.920
2.513
2.490
2.466
1.681
1.658
1.634
1.609
1.585
1.565
1.541
1.517
1.491
1.445
0.983
0.959
0.935
2018-11-21.20.fid
gkz1388
PROTON CDC13 E:\\ chit 59
N
1.00
1.06
2.09
6.01
2.03
4.08
3.05
f1 (ppm)

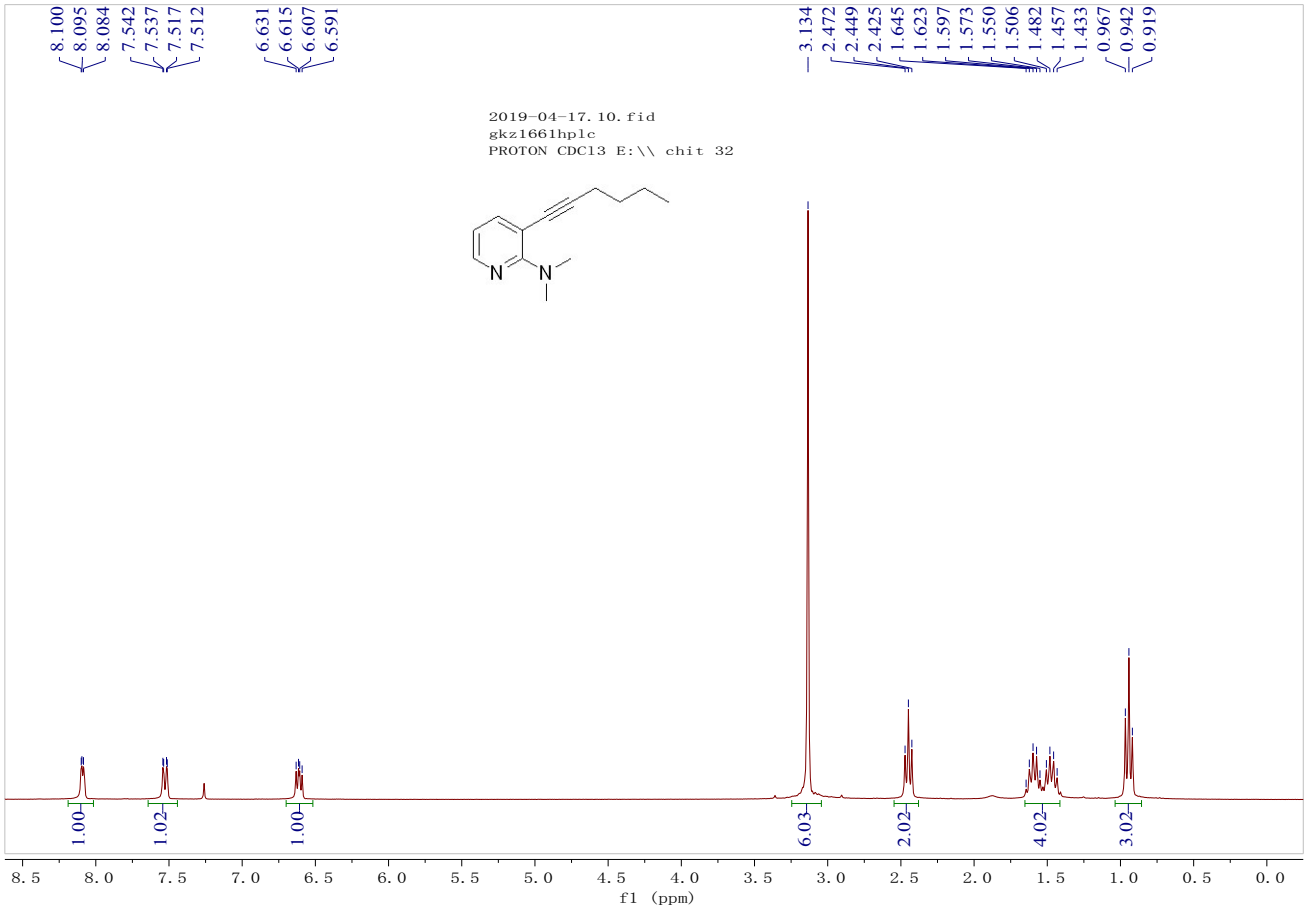

154.712
134.448
128.446
120.779
117.050
116.696
95.879
79.691
43.664
31.049
22.229
19.704
13.768
2018-11-21.21.fid
gkz1388
JMOD CDC13 E:\\ chit 59
f1 (ppm)

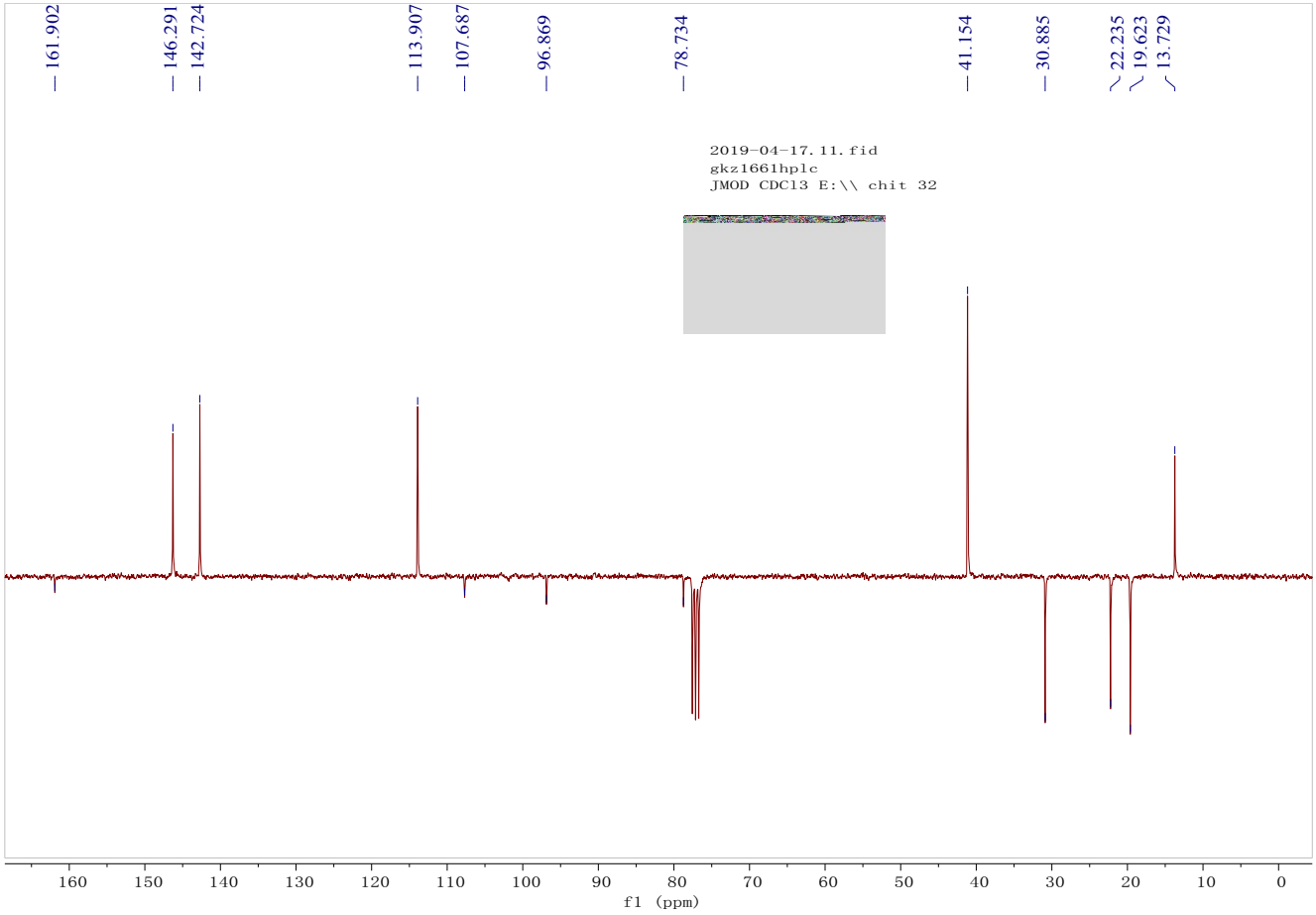

7.404
7.379
7.261
7.238
7.214
6.943
6.914
6.885
6.860
3.786
3.765
3.743
2.941
2.732
2.710
2.687
2.156
2.134
2.112
2.090
2.068
2019-01-18.30.fid
gkz1467
PROTON CDC13 E:\\ chit 20
Cl
N
1.00
1.05
2.02
2.02
6.04
2.01
2.03
f1 (ppm)

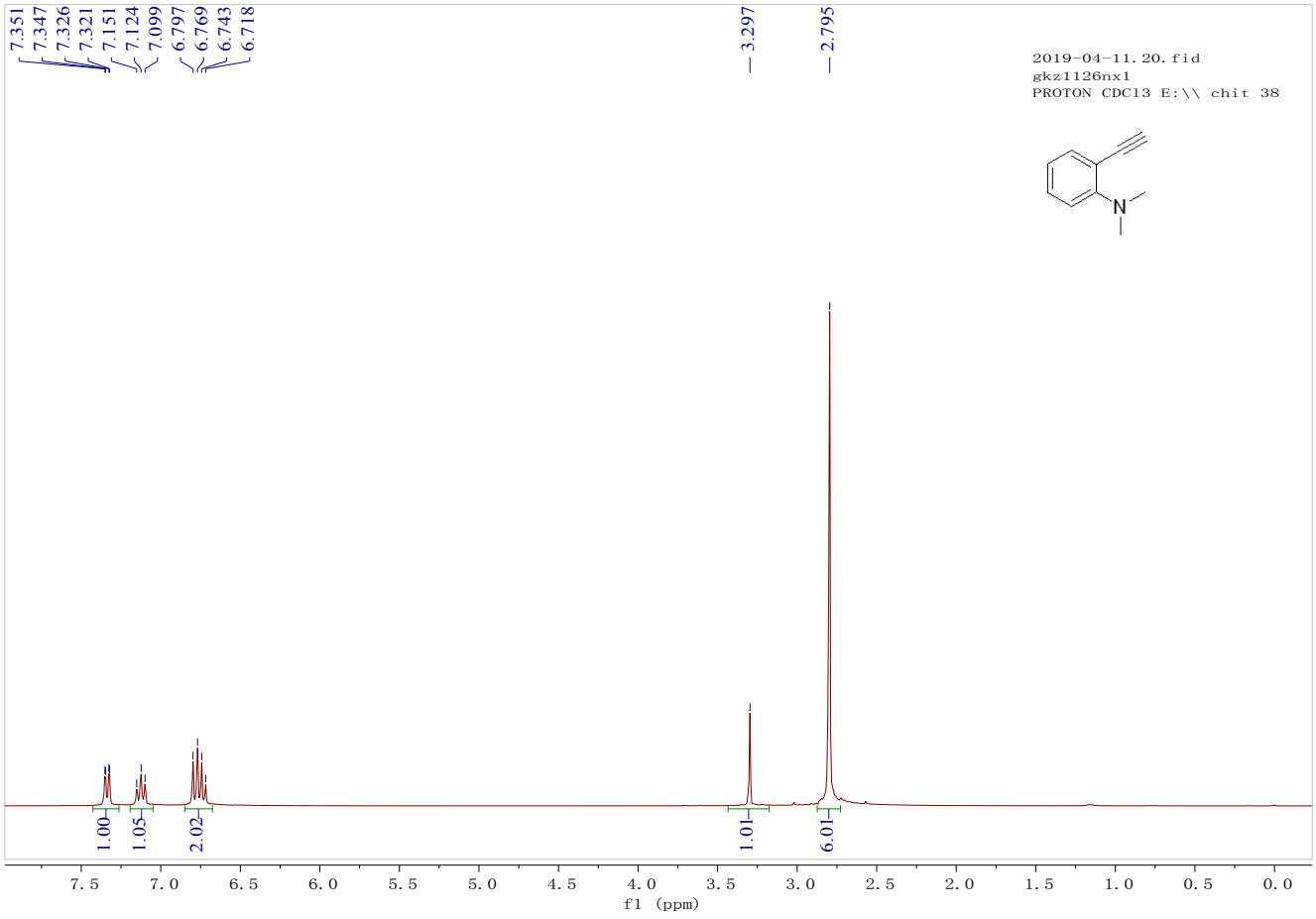

154.853
134.420
128.774
120.809
117.135
116.151
93.386
80.766
43.888
43.656
31.662
17.457
2019-01-18.31.fid
gkz1467
JMOD CDCl3 E:\\ chit 20
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

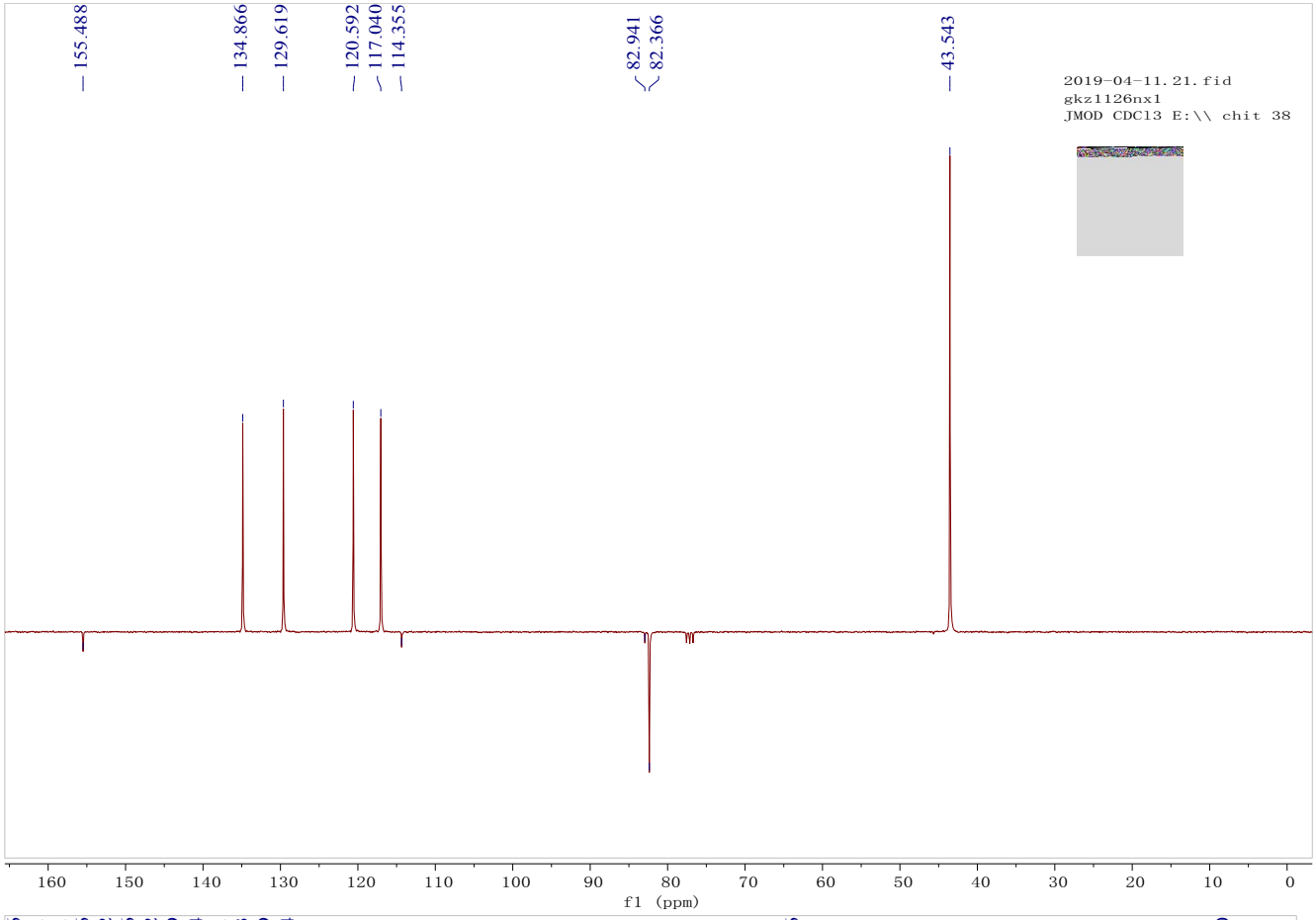

7.403
7.378
7.272
7.245
7.220
6.977
6.950
6.934
6.909
6.884
3.859
3.839
3.818
2.917
2.782
2.762
2.741
2.563
2019-03-01.50.fid
gkz1573p2n3
PROTON CDCl3 E:\\ chit 14
OH
N
1.00
1.02
2.02
2.00
6.08
2.03
1.02
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

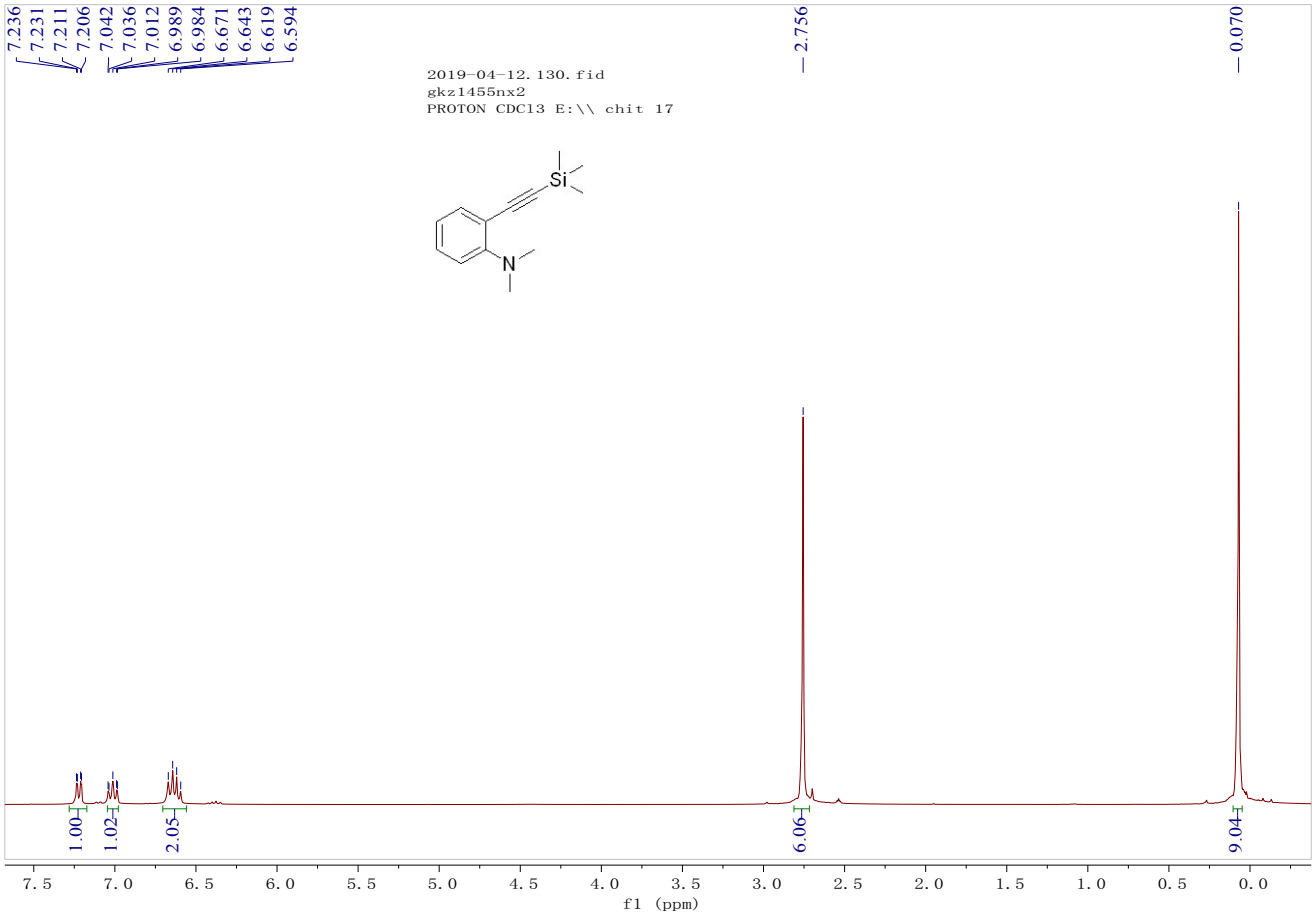

154.907
134.133
128.951
121.404
117.471
116.447
92.110
81.745
61.180
43.868
24.449
2019-03-01.51.fid
gkz1573p2n3
JMOD CDC13 E:\\ chit 14
f1 (ppm)

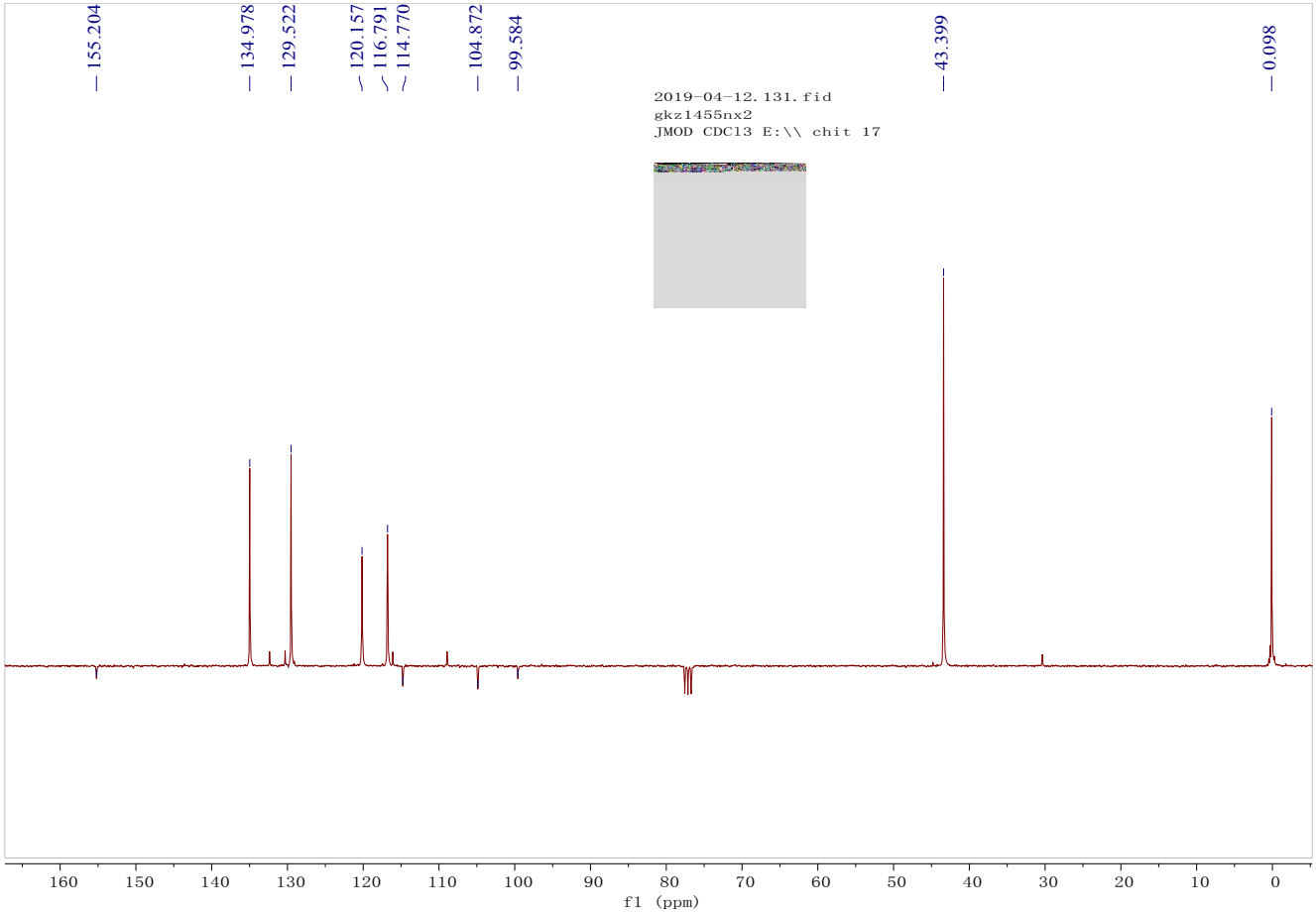

8.100
8.095
8.084
7.542
7.537
7.517
7.512
6.631
6.615
6.607
6.591
3.134
2.472
2.449
2.425
1.645
1.623
1.597
1.573
1.550
1.506
1.482
1.457
1.433
0.967
0.942
0.919
2019-04-17.10.fid
gkz1661hplc
PROTON CDC13 E:\\ chit 32
N
N
1.00
1.02
1.00
6.03
2.02
4.02
3.02
f1 (ppm)

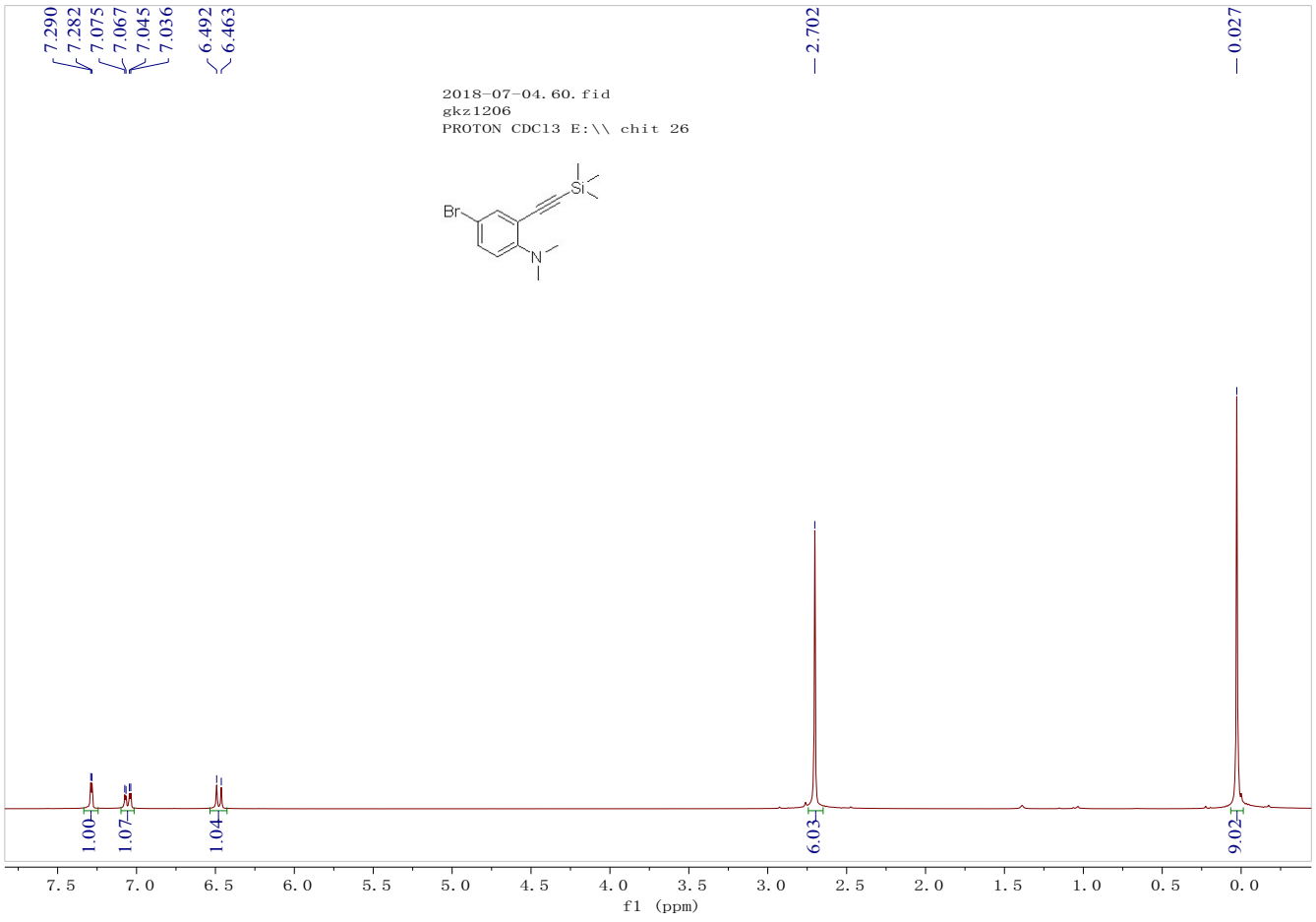

161.902
146.291
142.724
113.907
107.687
96.869
78.734
41.154
30.885
22.235
19.623
13.729
2019-04-17.11.fid
gkz1661hplc
JMOD CDCl3 E:\\ chit 32
f1 (ppm)

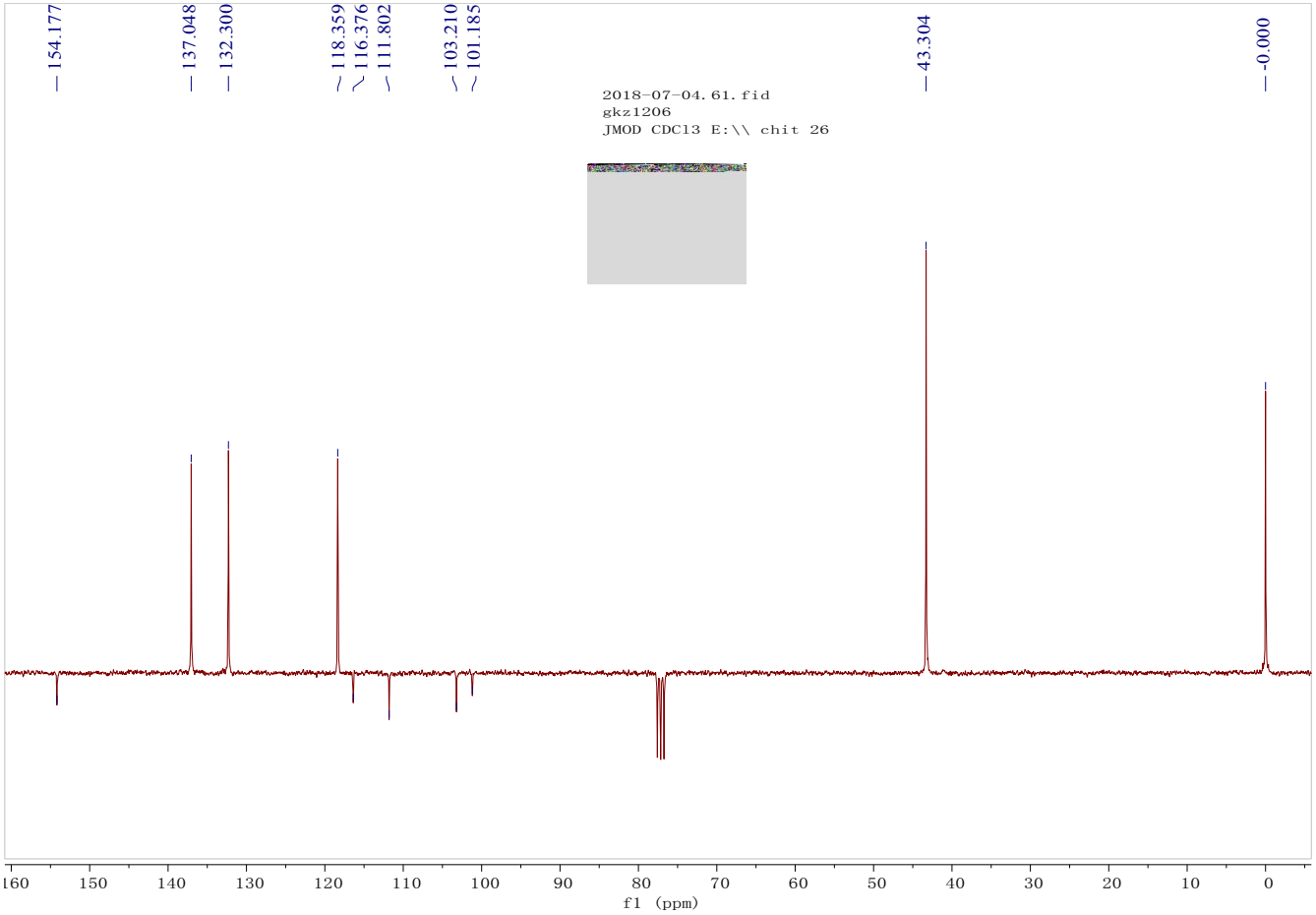

7.351
7.347
7.326
7.321
7.151
7.124
7.099
6.797
6.769
6.743
6.718
3.297
2.795
2019-04-11.20.fid
gkz1126nx1
PROTON CDCl3 E:\\ chit 38
N
1.00
1.05
2.02
1.01
6.01
f1 (ppm)

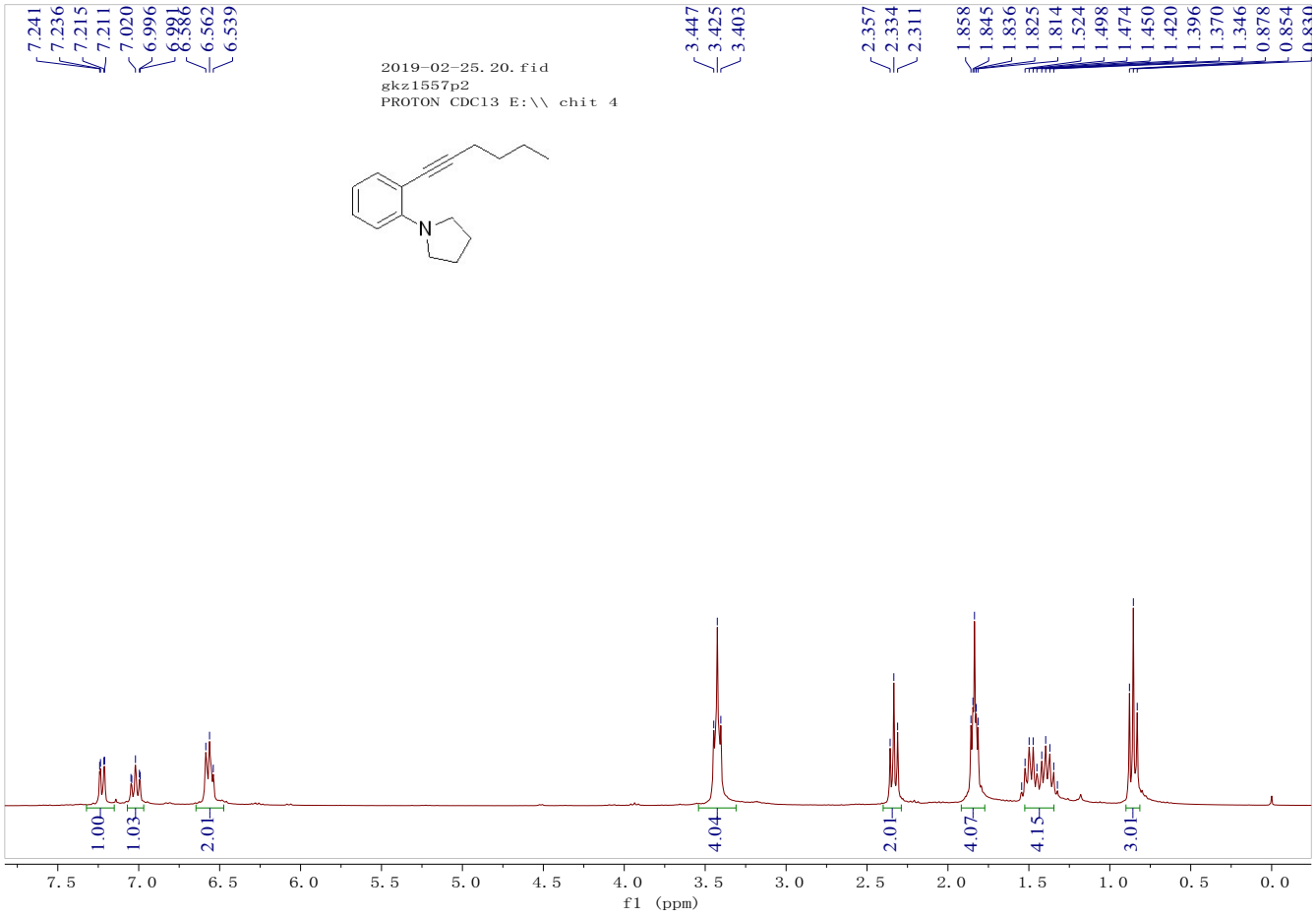

155.488
134.866
129.619
120.592
117.040
114.355
82.941
82.366
43.543
2019-04-11.21.fid
gkz1126nx1
JMOD CDCl3 E:\\ chit 38
f1 (ppm)

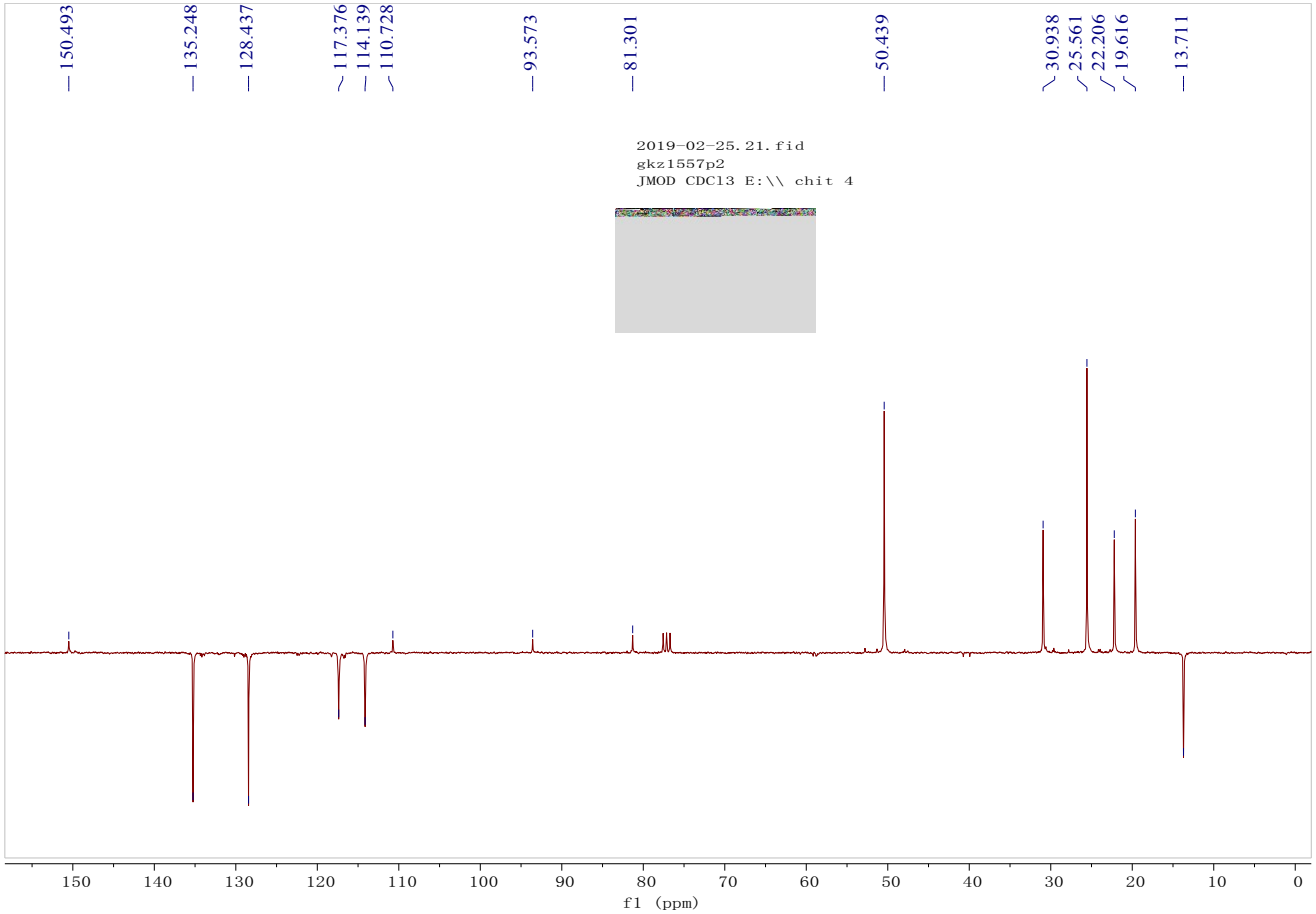

7.236
7.231
7.211
7.206
7.042
7.036
7.012
6.989
6.984
6.671
6.643
6.619
6.594
2.756
0.070
2019-04-12.130.fid
gkz1455nx2
PROTON CDCl3 E:\\ chit 17
Si
N
1.00
1.02
2.05
6.06
9.04
f1 (ppm)

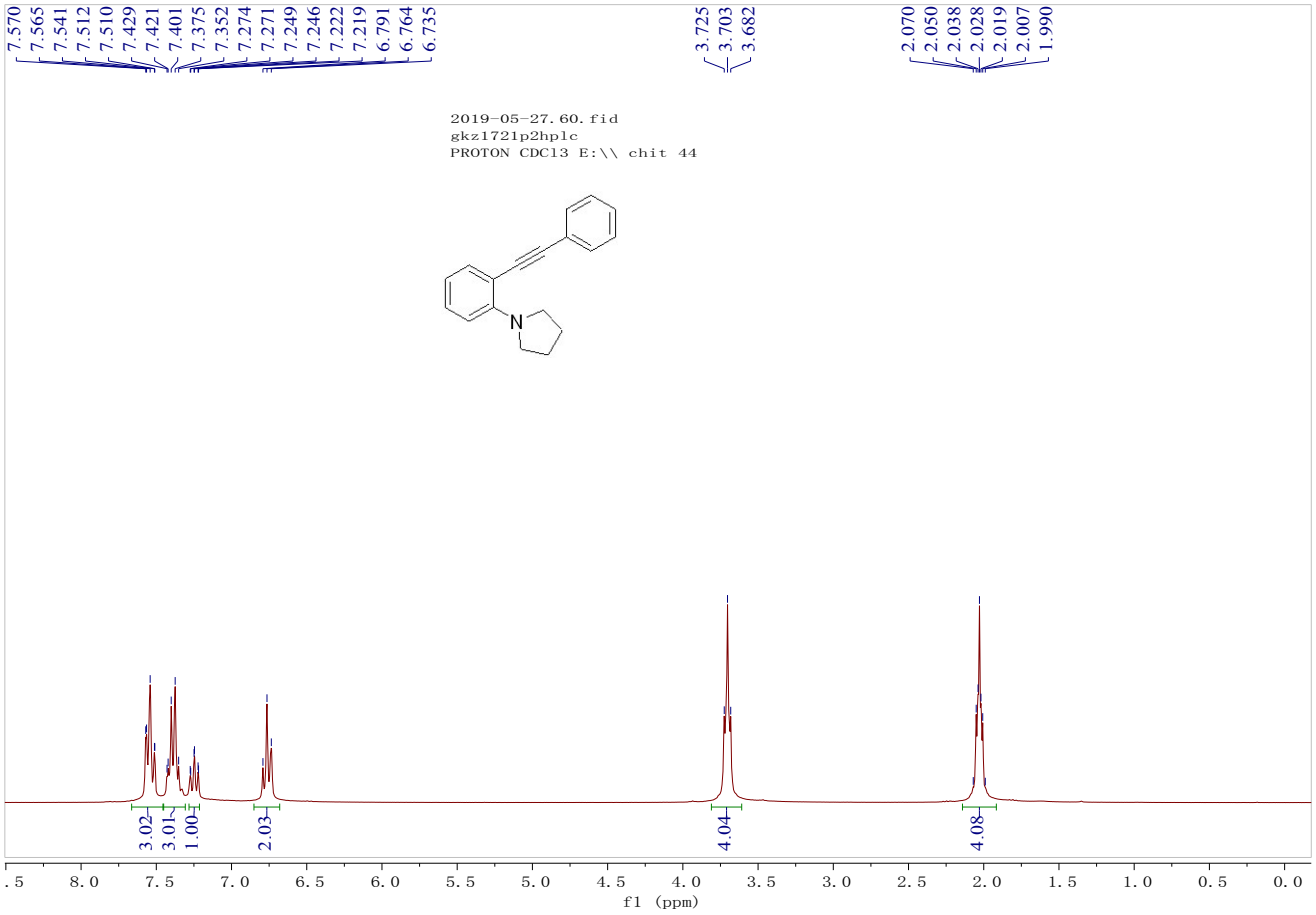

155.204
134.978
129.522
120.157
116.791
114.770
104.872
99.584
43.399
0.098
2019-04-12.131.fid
gkz1455nx2
JMOD CDCl3 E:\\ chit 17
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

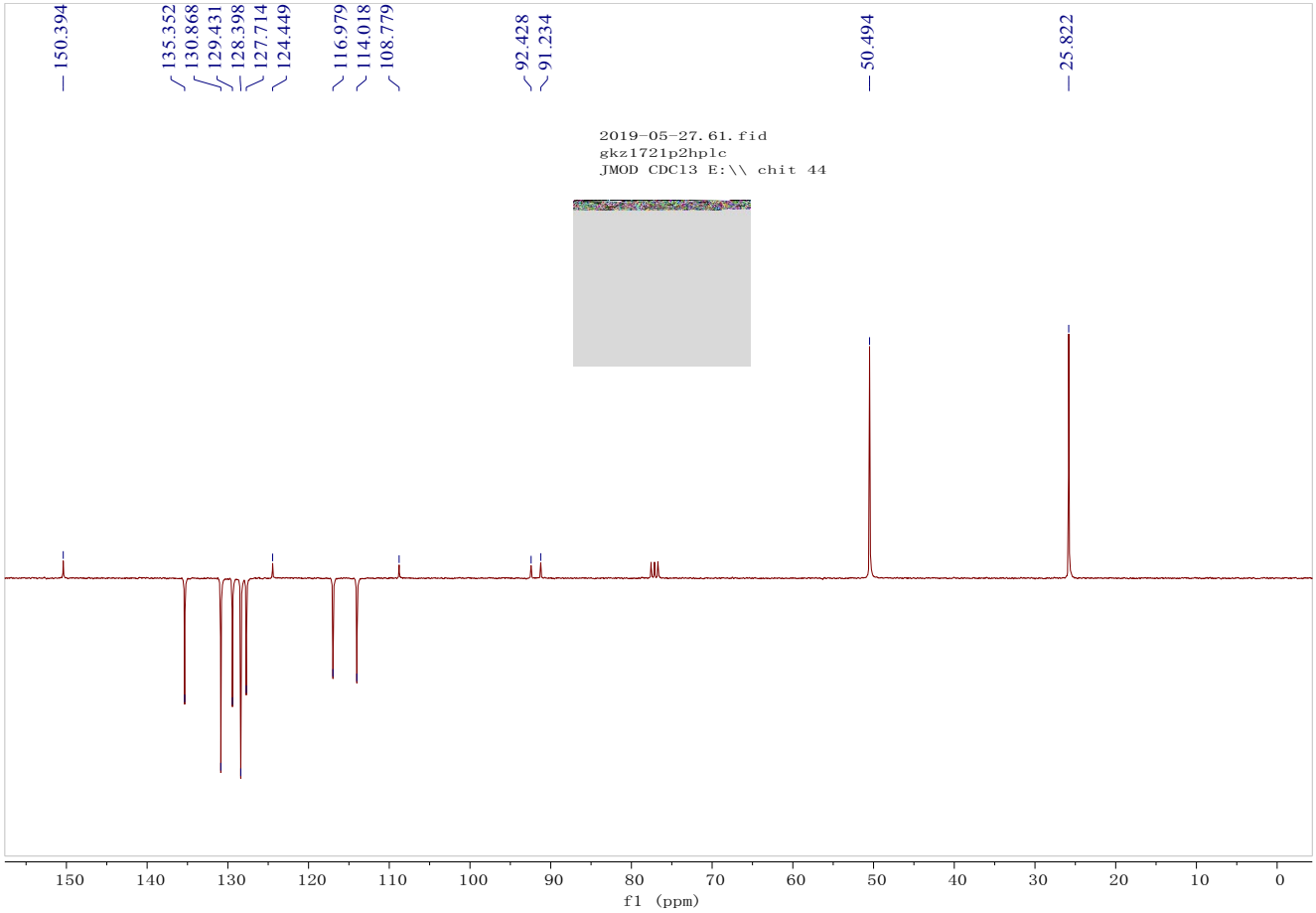

7.290
7.282
7.075
7.067
7.045
7.036
6.492
6.463
2.702
0.027
2018-07-04.60.fid
gkz1206
PROTON CDCl3 E:\\ chit 26
Br
Si
N
1.00
1.07
1.04
6.03
9.02
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

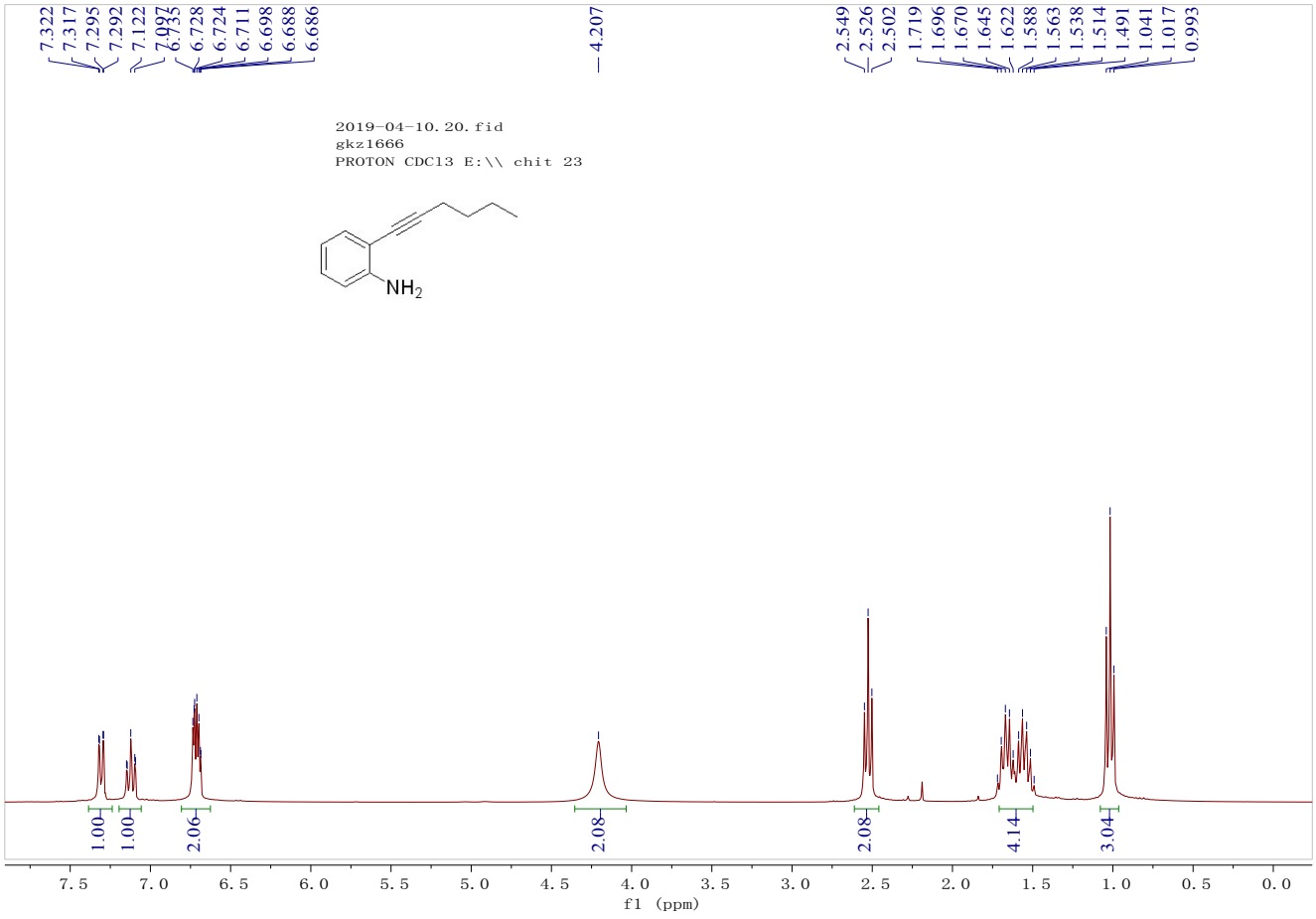

154.177
137.048
132.300
118.359
116.376
111.802
103.210
101.185
43.304
-0.000
2018-07-04.61.fid
gkz1206
JMOD CDCl3 E:\\ chit 26
f1 (ppm)

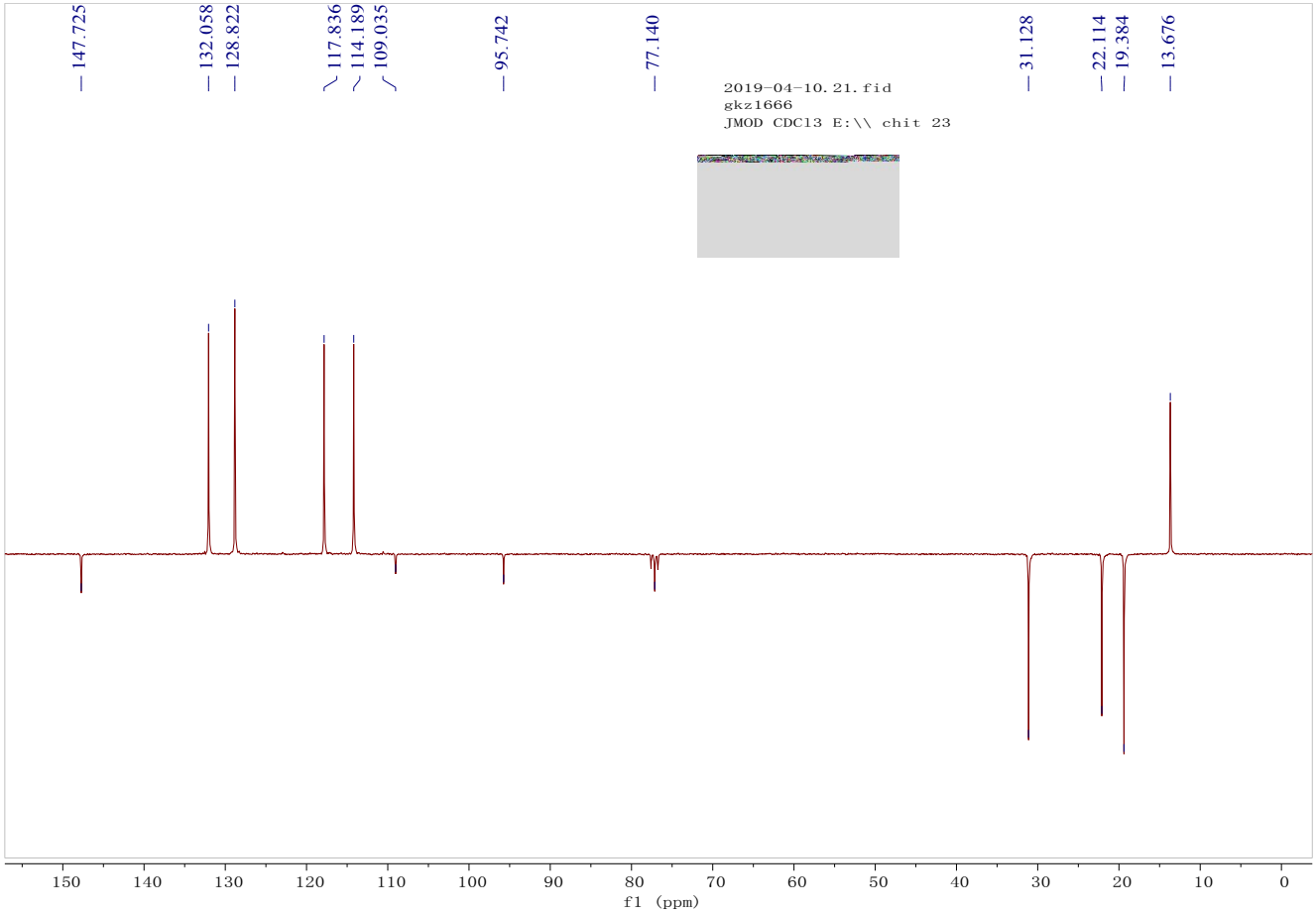

7.241
7.236
7.215
7.211
7.020
6.996
6.991
6.586
6.562
6.539
3.447
3.425
3.403
2.357
2.334
2.311
1.858
1.845
1.836
1.825
1.814
1.524
1.498
1.474
1.450
1.420
1.396
1.370
1.346
0.878
0.854
2019-02-25.20.fid
gkz1557p2
PROTON CDCl3 E:\\ chit 4
N
1.00
1.03
2.01
4.04
2.01
4.07
4.15
3.01
f1 (ppm)

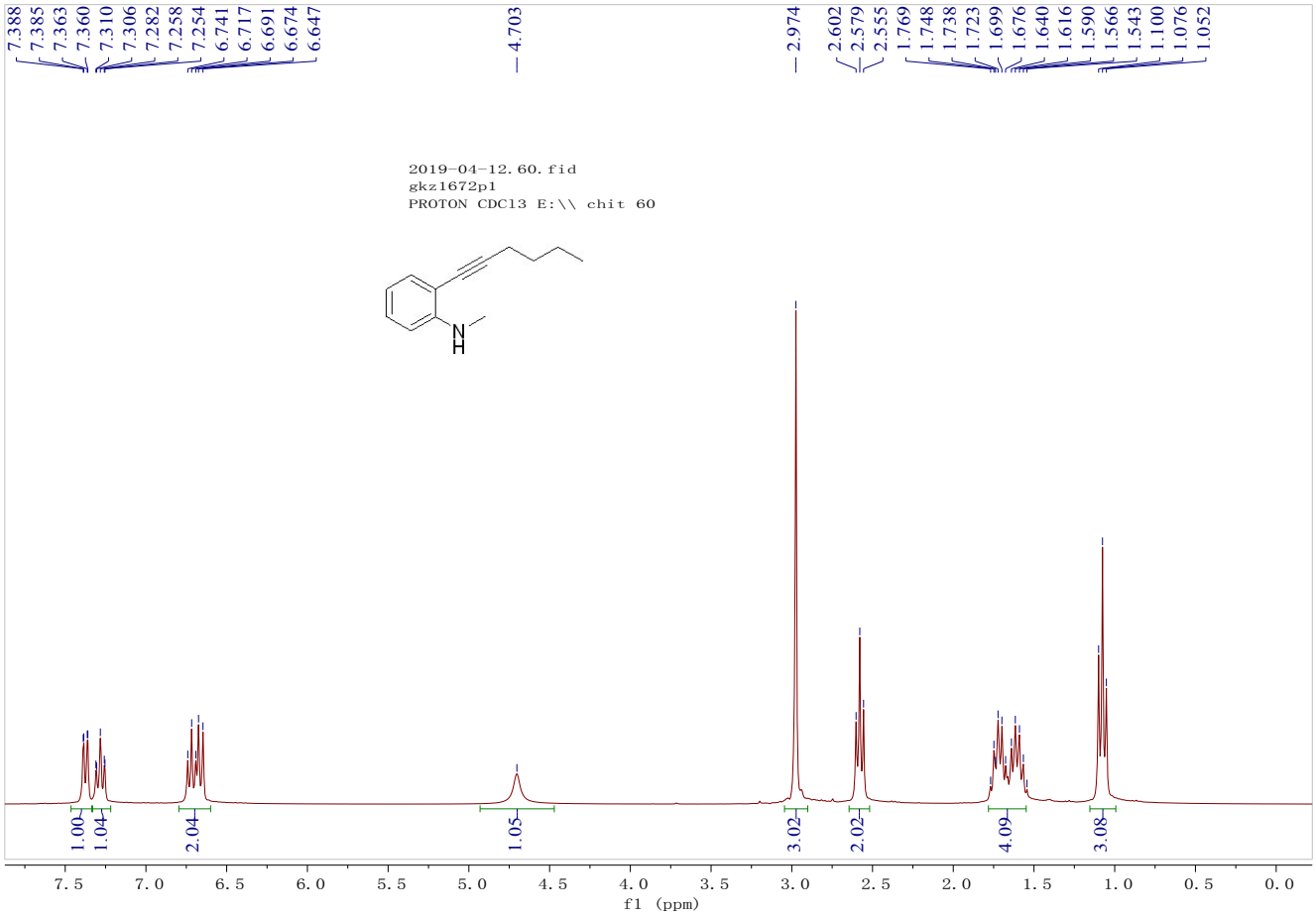

150.493
135.248
128.437
117.376
114.139
110.728
93.573
81.301
50.439
30.938
25.561
22.206
19.616
13.711
2019-02-25.21.fid
gkz1557p2
JMOD CDC13 E:\\ chit 4
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

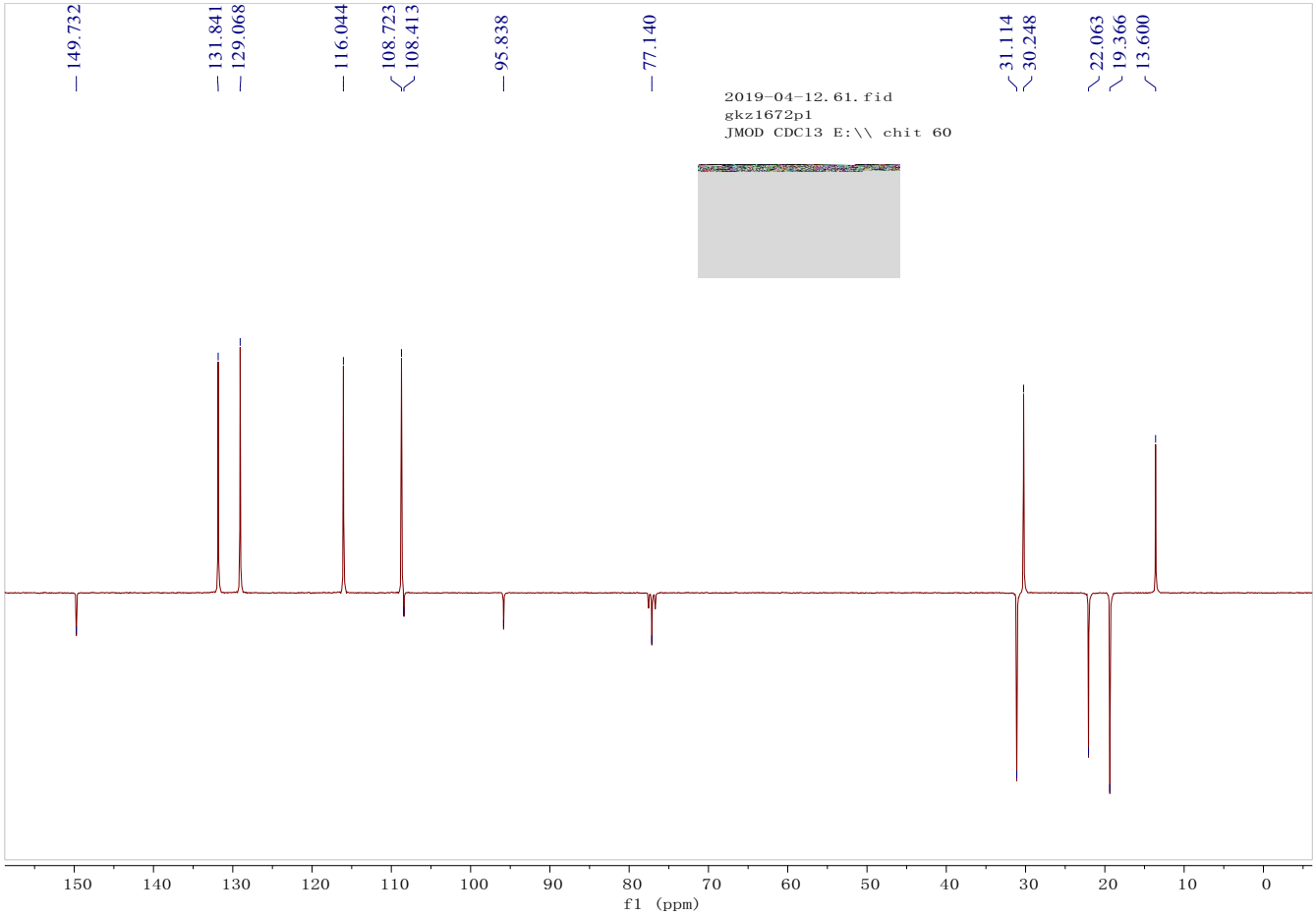

7.570
7.565
7.541
7.512
7.510
7.429
7.421
7.401
7.375
7.352
7.274
7.271
7.249
7.246
7.222
7.219
6.791
6.764
6.735
3.725
3.703
3.682
2.070
2.050
2.038
2.028
2.019
2.007
1.990
2019-05-27.60.fid
gkz1721p2hplc
PROTON CDC13 E:\\ chit 44
N
3.02
3.01
1.00
2.03
4.04
4.08
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

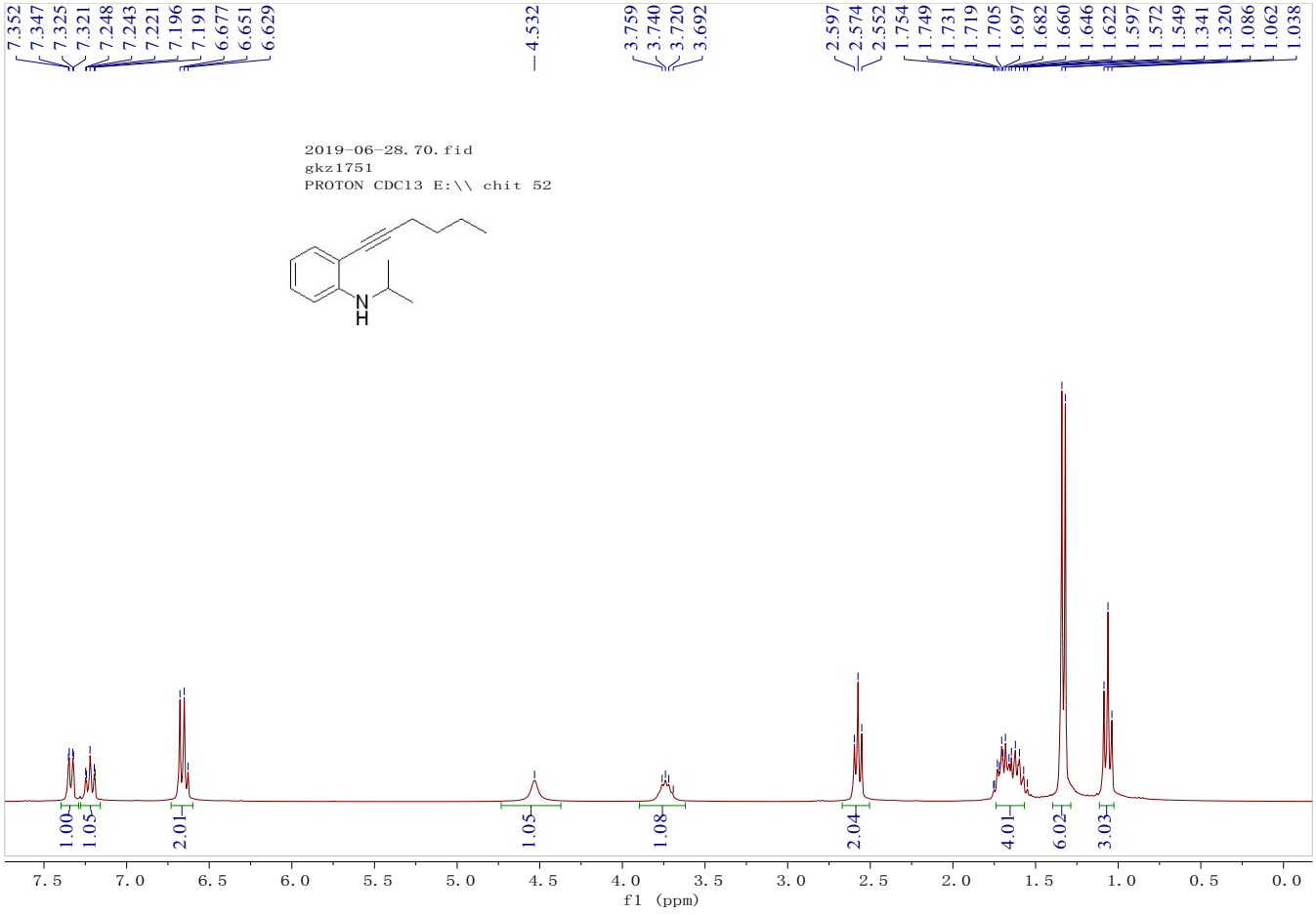

150.394
135.352
130.868
129.431
128.398
127.714
124.449
116.979
114.018
108.779
92.428
91.234
50.494
25.822
2019-05-27.61.fid
gkz1721p2hplc
JMOD CDCl3 E:\\ chit 44
f1 (ppm)

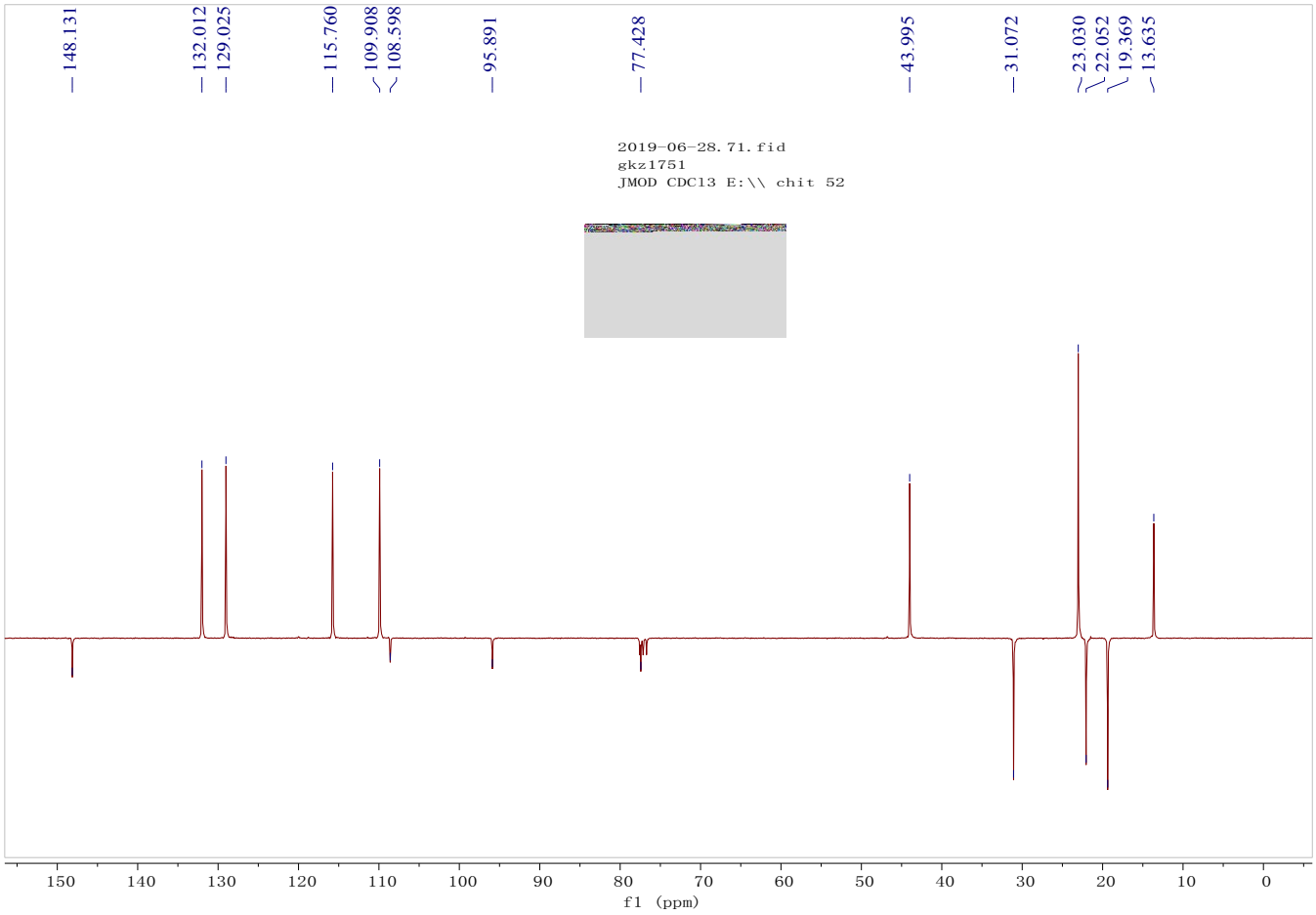

7.322
7.317
7.295
7.292
7.122
7.097
6.735
6.728
6.724
6.711
6.698
6.688
6.686
4.207
2.549
2.526
2.502
1.719
1.696
1.670
1.645
1.622
1.588
1.563
1.538
1.514
1.491
1.041
1.017
0.993
2019-04-10.20.fid
gkz1666
PROTON CDCl3 E:\\ chit 23
NH2
1.00
1.00
2.06
2.08
2.08
4.14
3.04
f1 (ppm)

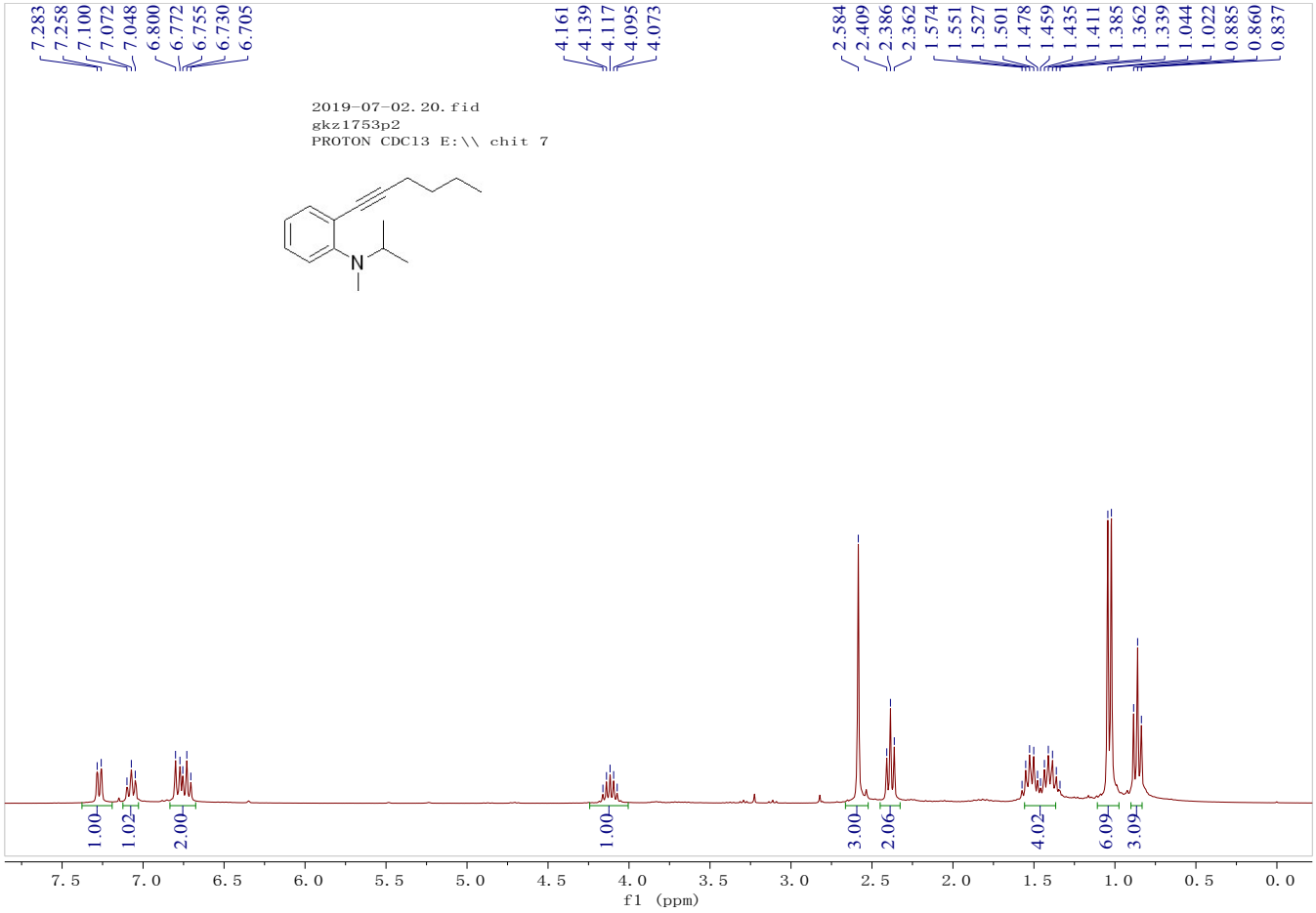

147.725
132.058
128.822
117.836
114.189
109.035
95.742
77.140
31.128
22.114
19.384
13.676
2019-04-10.21.fid
gkz1666
JMOD CDCl3 E:\\ chit 23
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

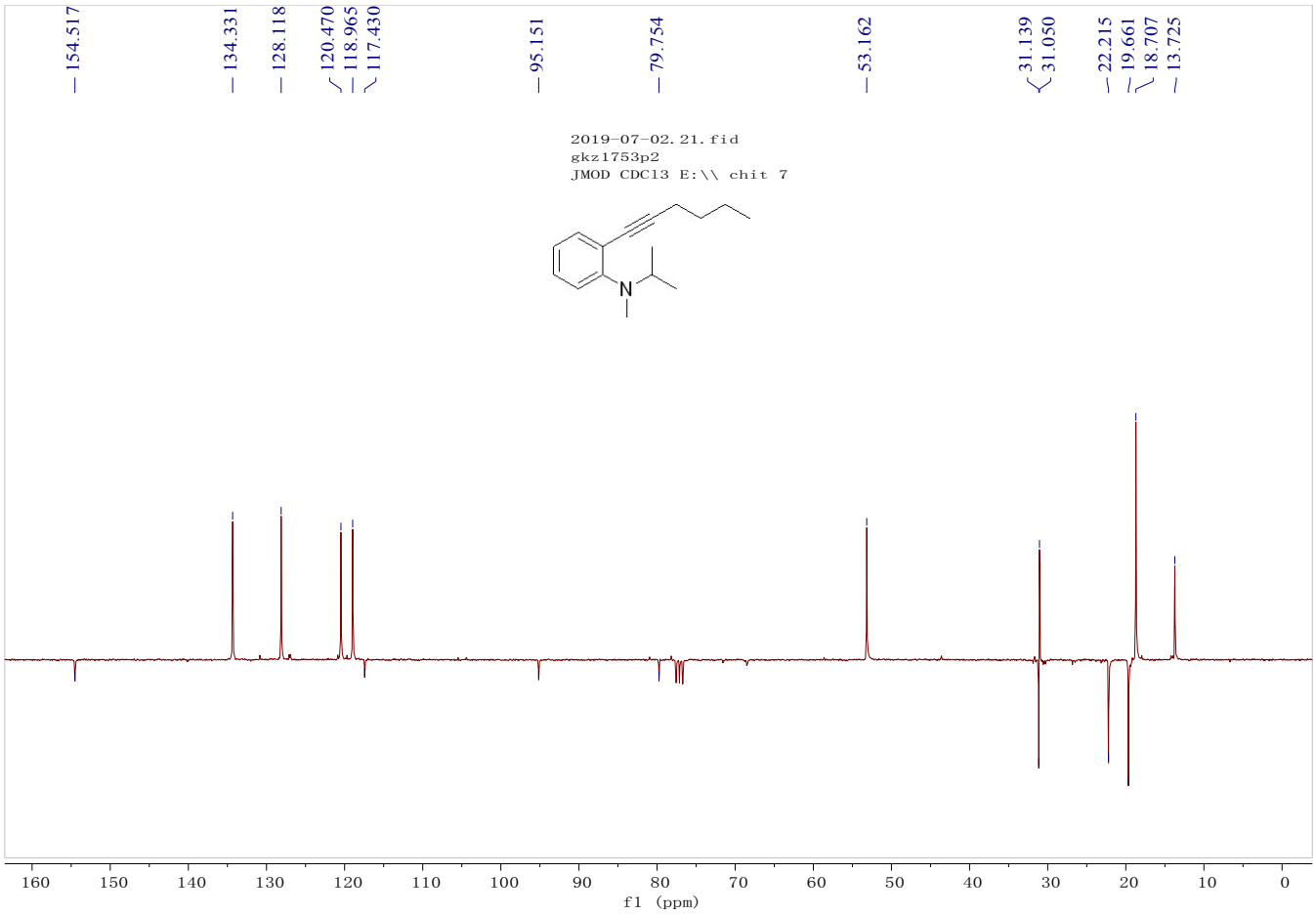

7.388
7.385
7.363
7.360
7.310
7.306
7.282
7.258
7.254
6.741
6.717
6.691
6.674
6.647
4.703
2.974
2.602
2.579
2.555
1.769
1.748
1.738
1.723
1.699
1.676
1.640
1.616
1.590
1.566
1.543
1.100
1.076
1.052
2019-04-12.60.fid
gkz1672p1
PROTON CDCl3 E:\\ chit 60
1.00
1.04
2.04
1.05
3.02
2.02
4.09
3.08
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

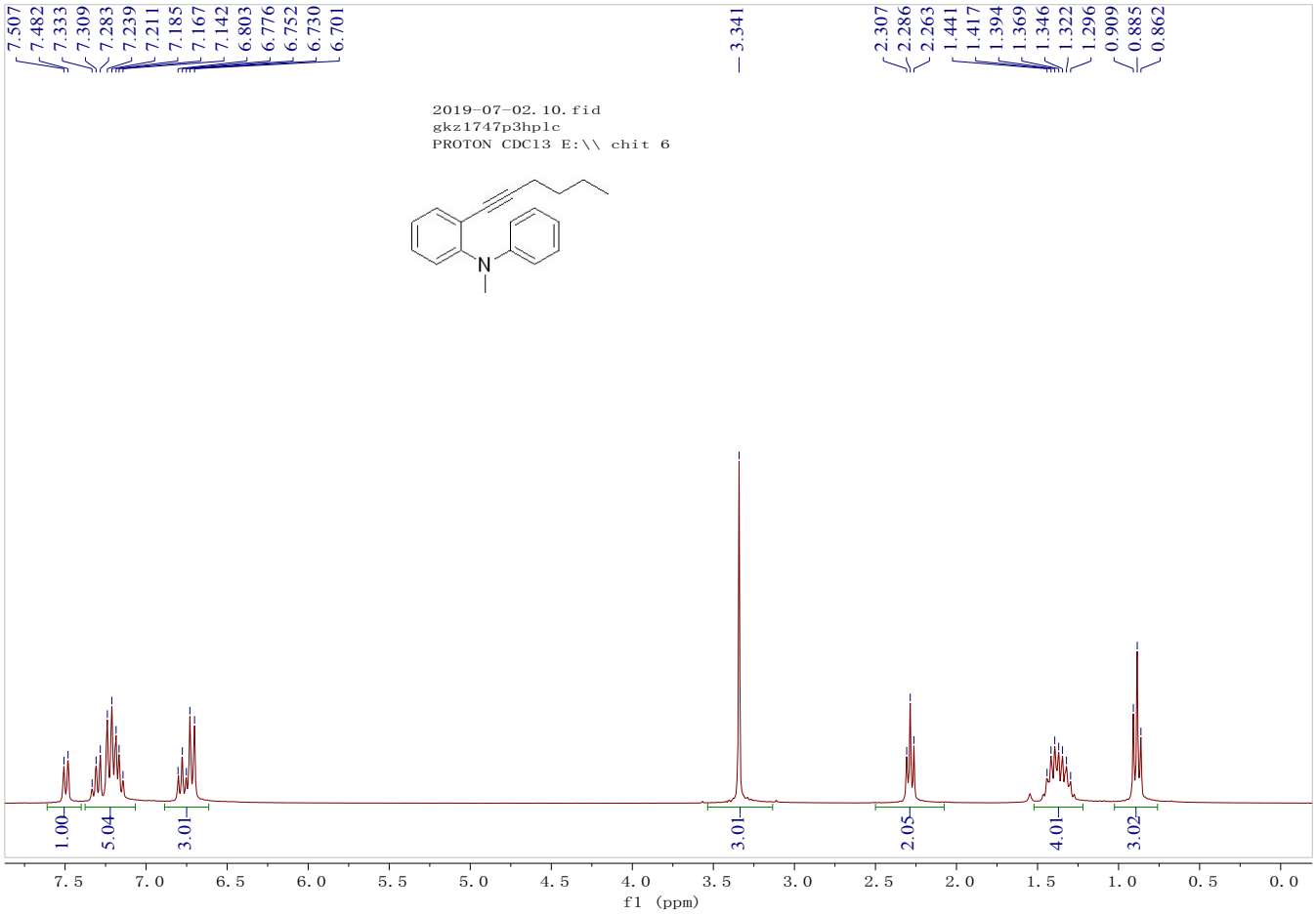

149.732
131.841
129.068
116.044
108.723
108.413
95.838
77.140
31.114
30.248
22.063
19.366
13.600
2019-04-12.61.fid
gkz1672p1
JMOD CDCl3 E:\\ chit 60
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

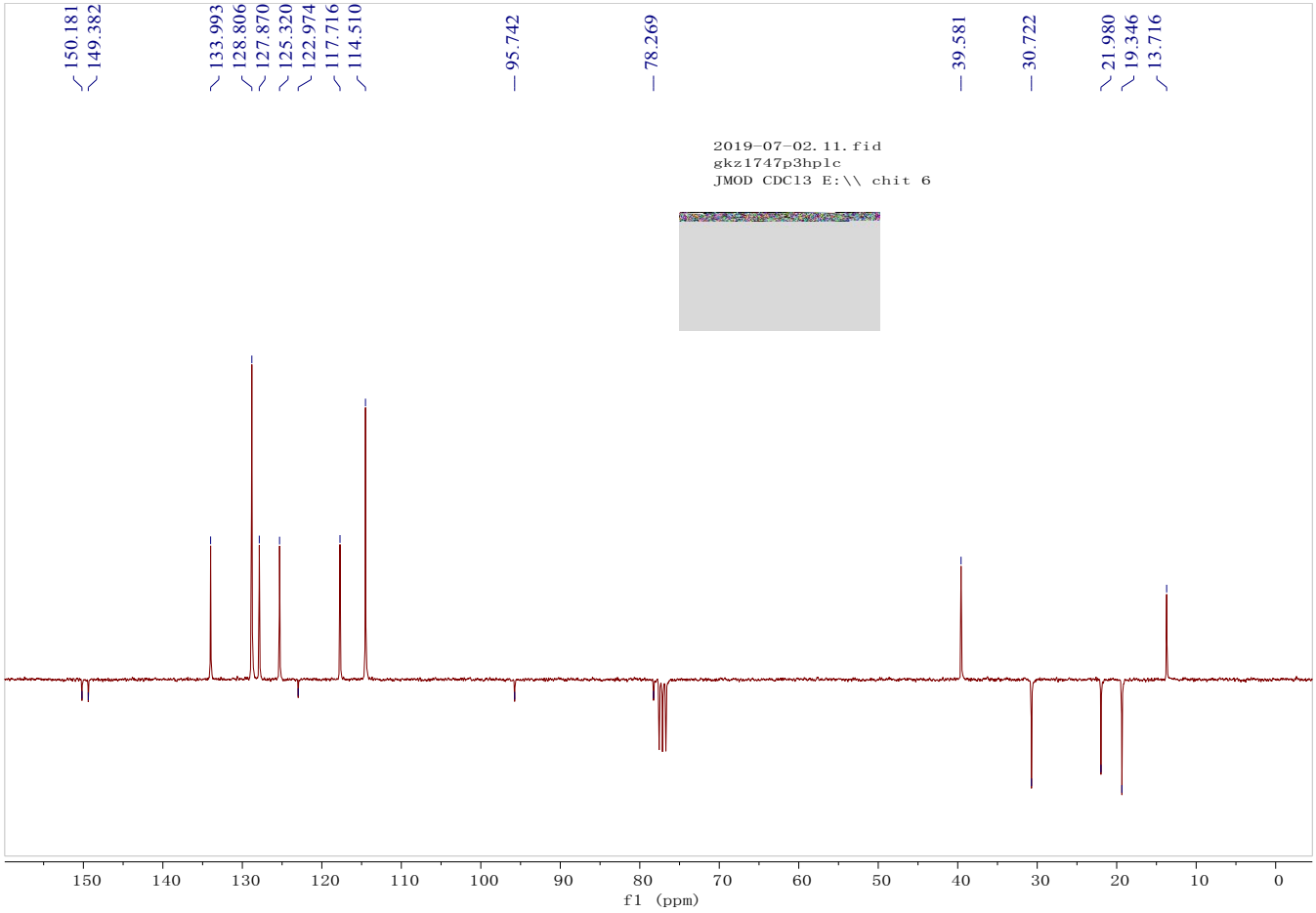

7.352
7.347
7.325
7.321
7.248
7.243
7.221
7.196
7.191
6.677
6.651
6.629
4.532
3.759
3.740
3.720
3.692
2.597
2.574
2.552
1.754
1.749
1.731
1.719
1.705
1.697
1.682
1.660
1.646
1.622
1.597
1.572
1.549
1.341
1.320
1.086
1.062
1.038
2019-06-28.70.fid
gkz1751
PROTON CDCl3 E:\\ chit 52
N
H
1.00
1.05
2.01
1.05
1.08
2.04
4.01
6.02
3.03
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

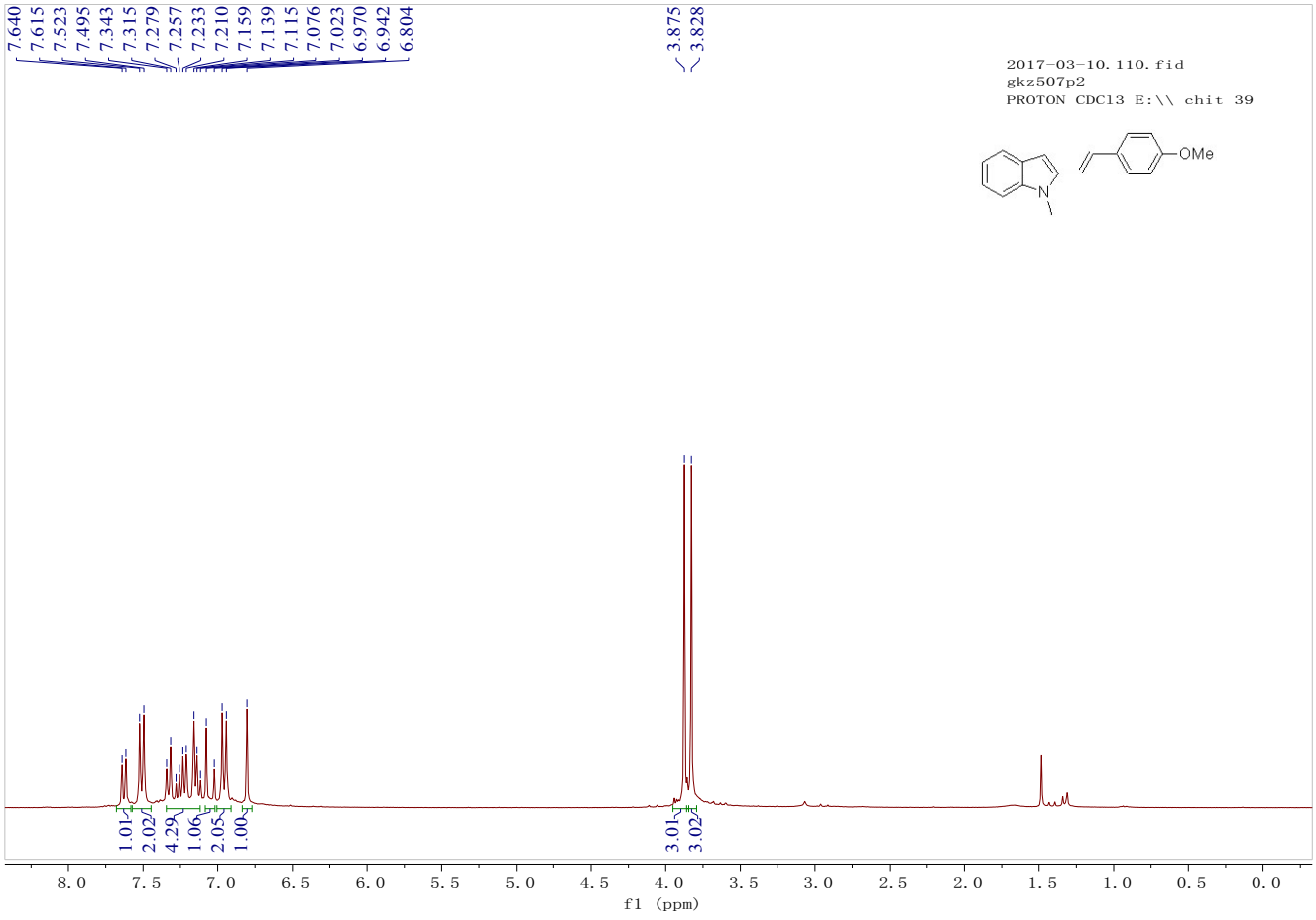

148.131
132.012
129.025
115.760
109.908
108.598
95.891
77.428
43.995
31.072
23.030
22.052
19.369
13.635
2019-06-28.71.fid
gkz1751
JMOD CDCl3 E:\\ chit 52
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

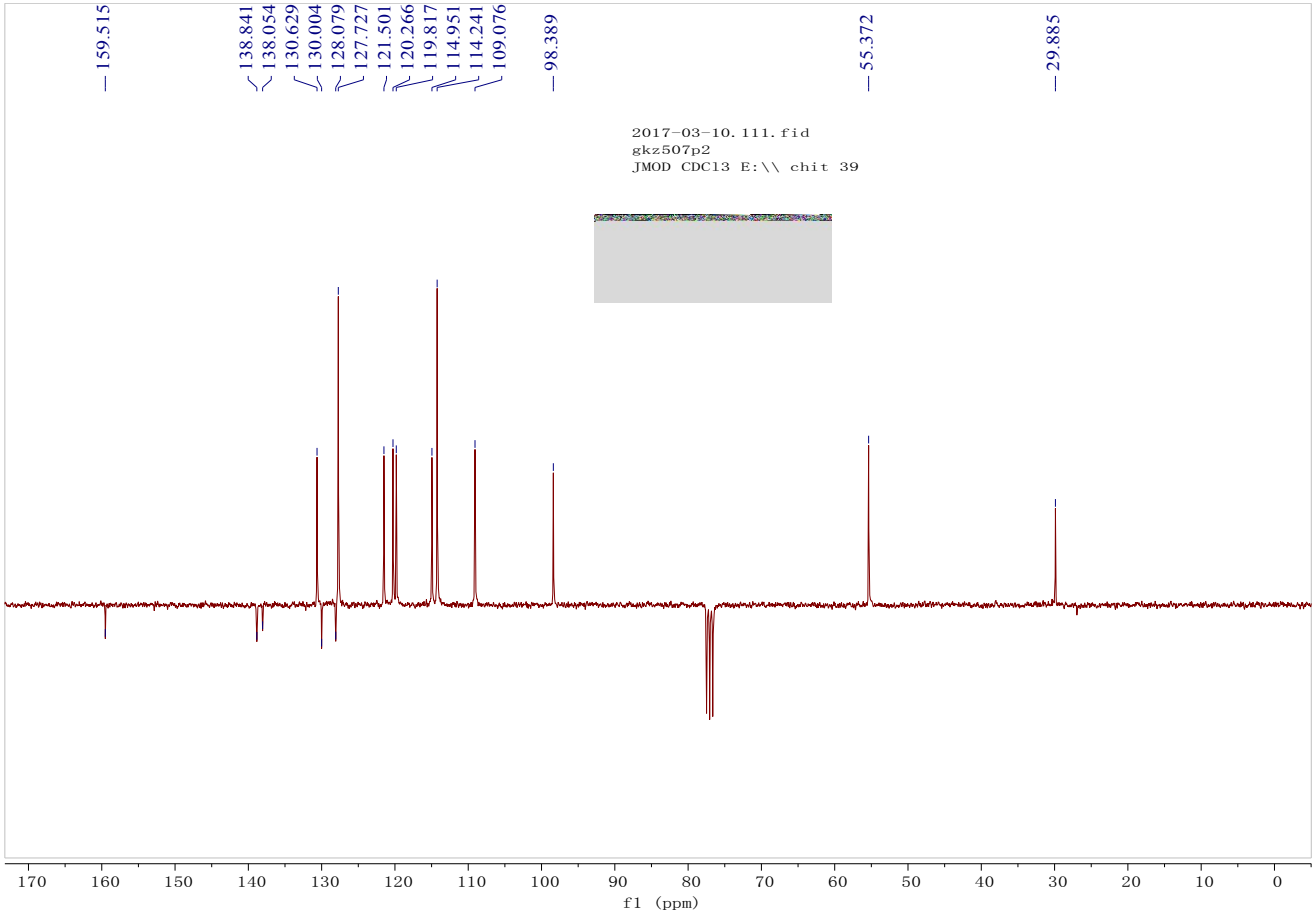

7.283
7.258
7.100
7.072
7.048
6.800
6.772
6.755
6.730
6.705
4.161
4.139
4.117
4.095
4.073
2.584
2.409
2.386
2.362
1.574
1.551
1.527
1.501
1.478
1.459
1.435
1.411
1.385
1.362
1.339
1.044
1.022
0.885
0.860
0.837
2019-07-02.20.fid
gkz1753p2
PROTON CDCl3 E:\\ chit 7
N
1.00
1.02
2.00
1.00
3.00
2.06
4.02
6.09
3.09
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

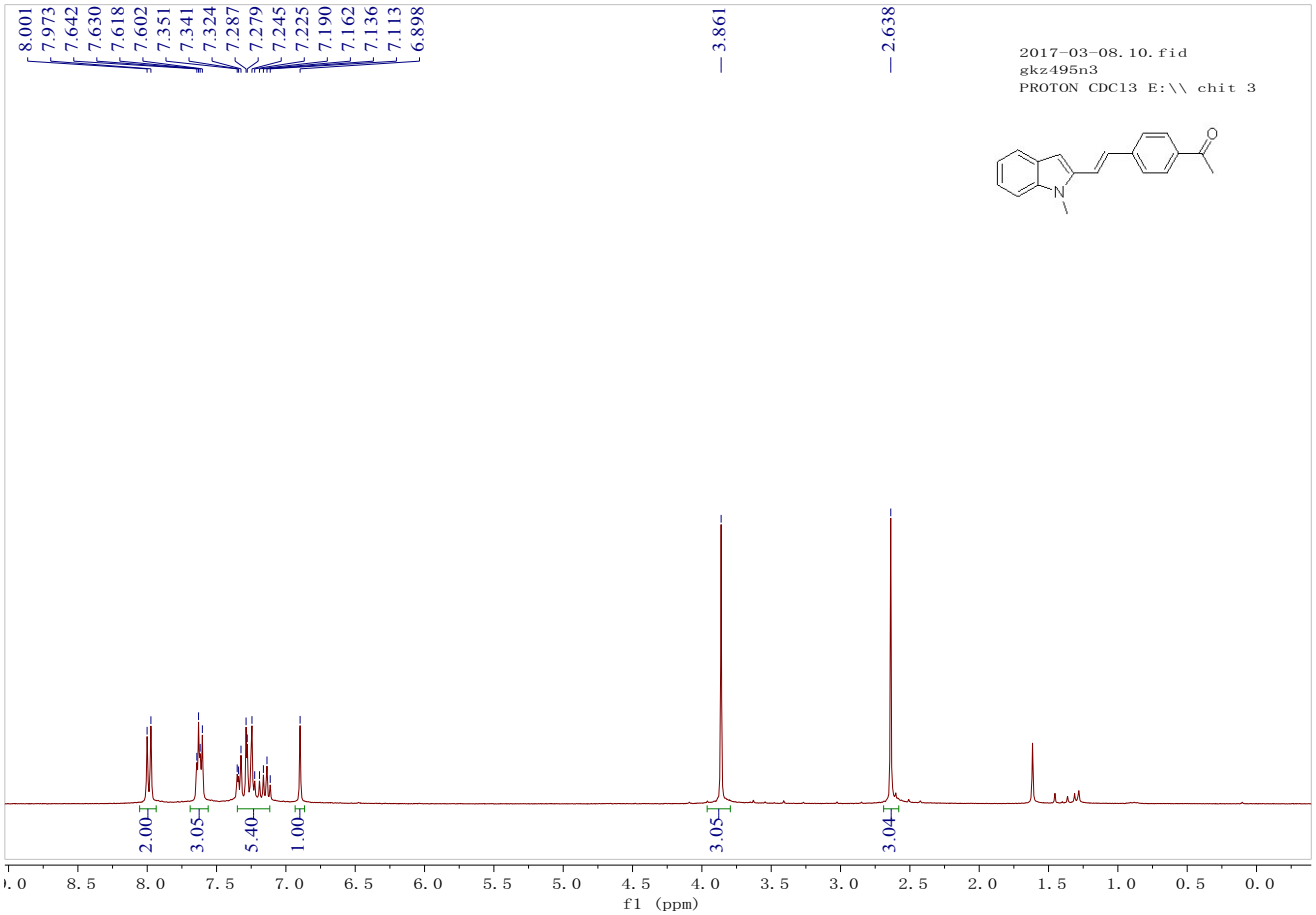

154.517
134.331
128.118
120.470
118.965
117.430
95.151
79.754
53.162
31.139
31.050
22.215
19.661
18.707
13.725
2019-07-02.21.fid
gkz1753p2
JMOD CDCl3 E:\\ chit 7
N
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

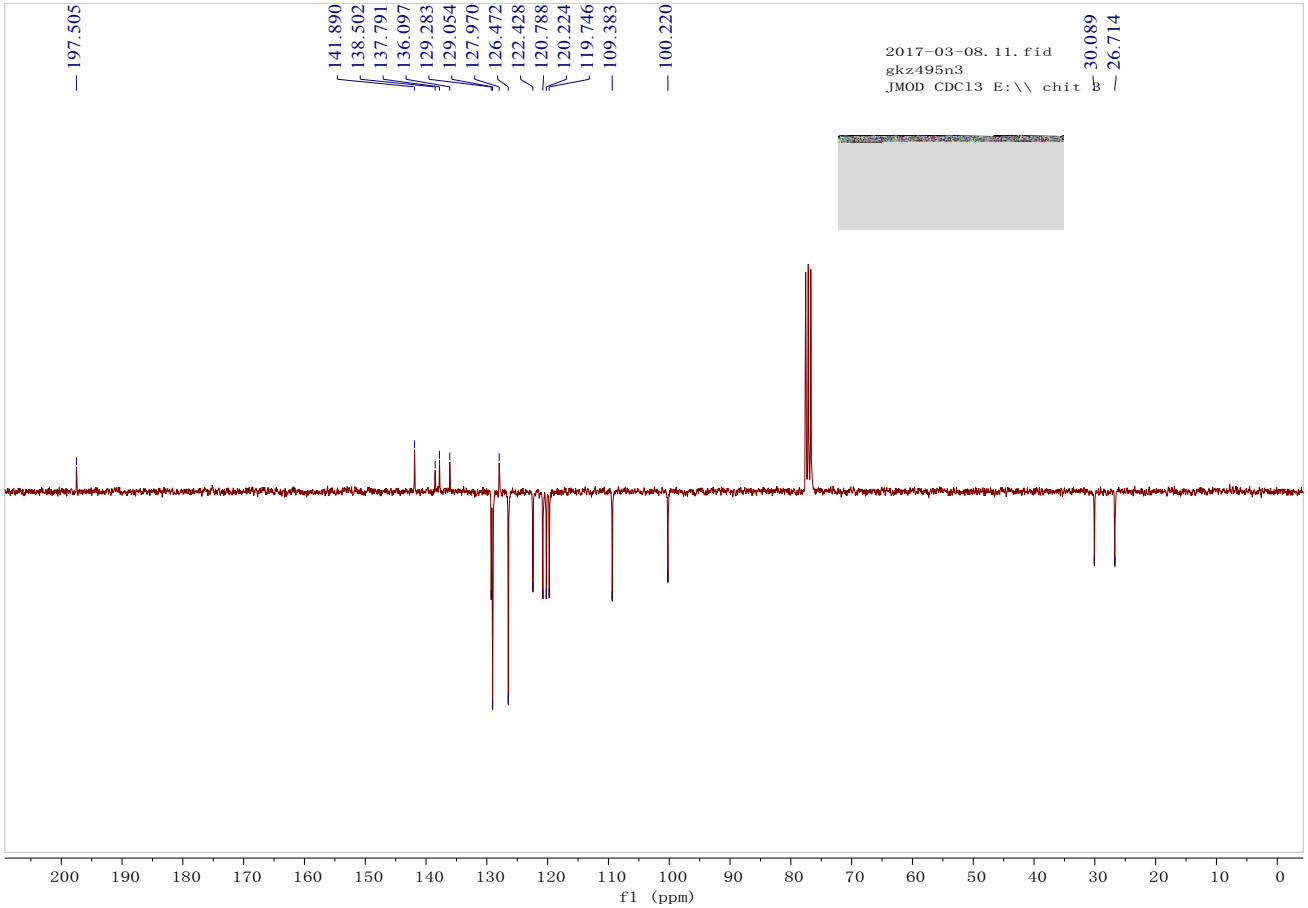

7.507
7.482
7.333
7.309
7.283
7.239
7.211
7.185
7.167
7.142
6.803
6.776
6.752
6.730
6.701
3.341
2.307
2.286
2.263
1.441
1.417
1.394
1.369
1.346
1.322
1.296
0.909
0.885
0.862
2019-07-02.10.fid
gkz1747p3hplc
PROTON CDCl3 E:\\ chit 6
N
1.00
5.04
3.01
3.01
2.05
4.01
3.02
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

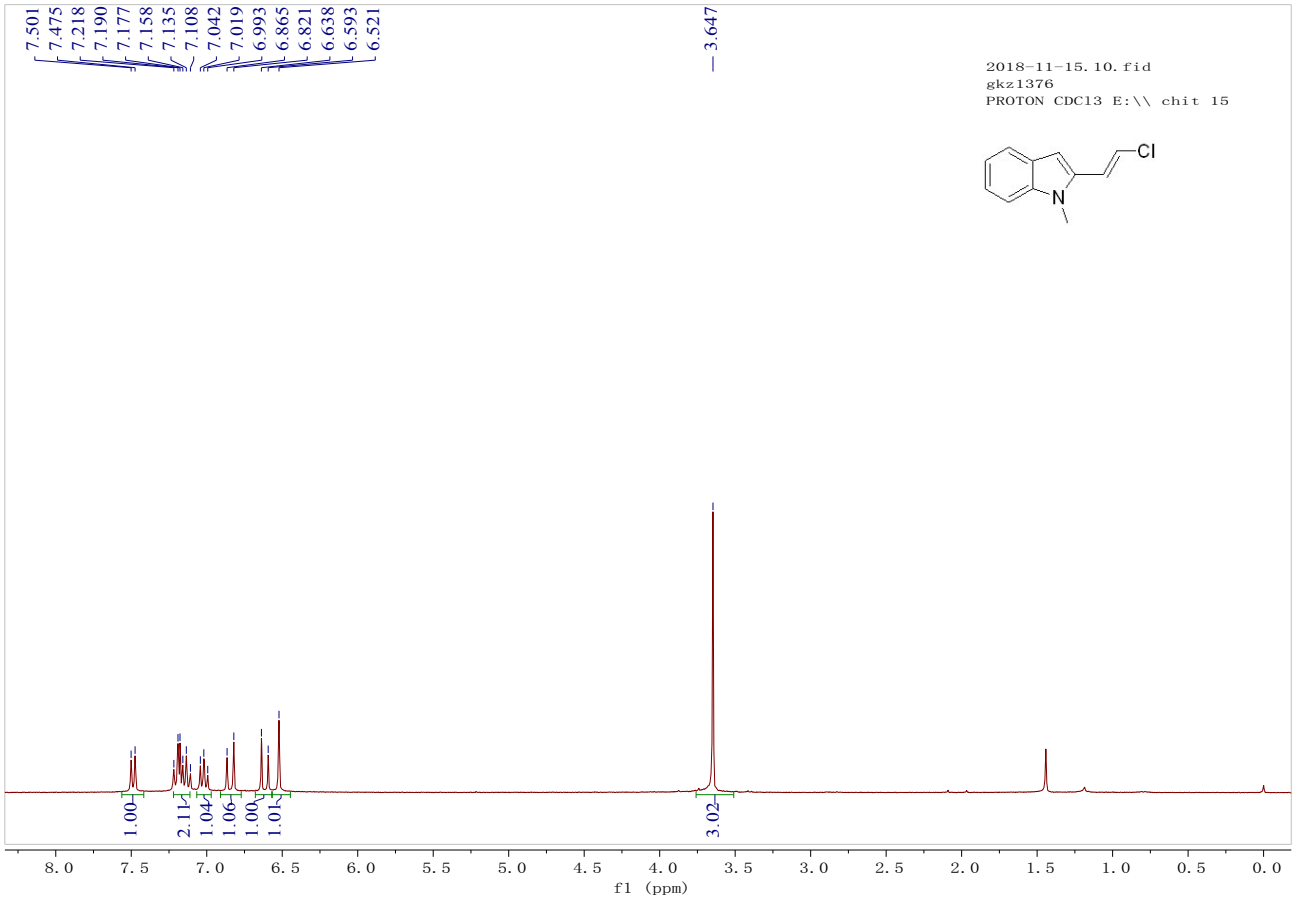

150.181
149.382
133.993
128.806
127.870
125.320
122.974
117.716
114.510
95.742
78.269
39.581
30.722
21.980
19.346
13.716
2019-07-02.11.fid
gkz1747p3hplc
JMOD CDC13 E:\\ chit 6
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

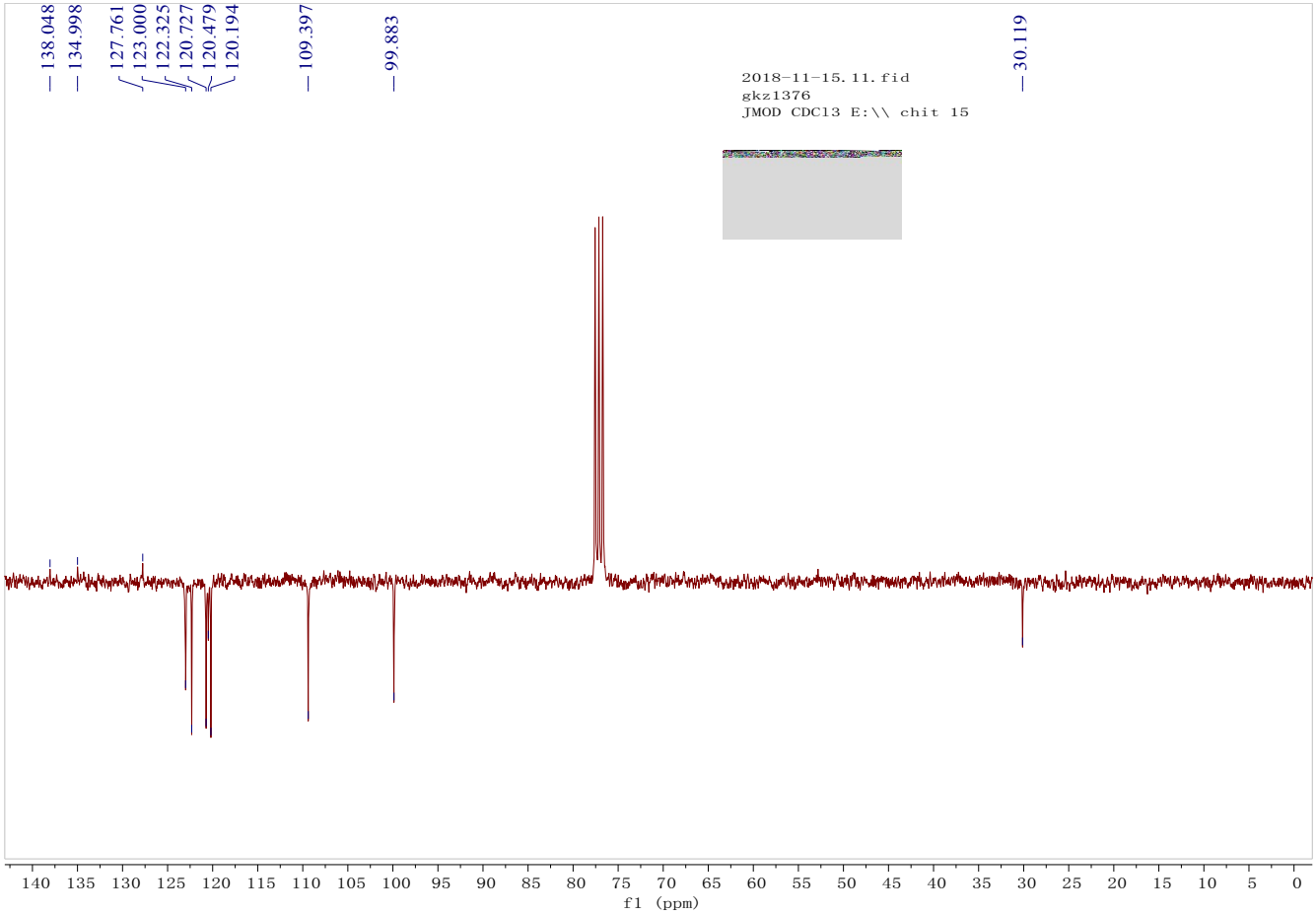

7.640
7.615
7.523
7.495
7.343
7.315
7.279
7.257
7.233
7.210
7.159
7.139
7.115
7.076
7.023
6.970
6.942
6.804
3.875
3.828
2017-03-10.110.fid
gkz507p2
PROTON CDC13 E:\\ chit 39
OMe
N
1.01
2.02
4.29
1.06
2.05
1.00
3.01
3.02
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

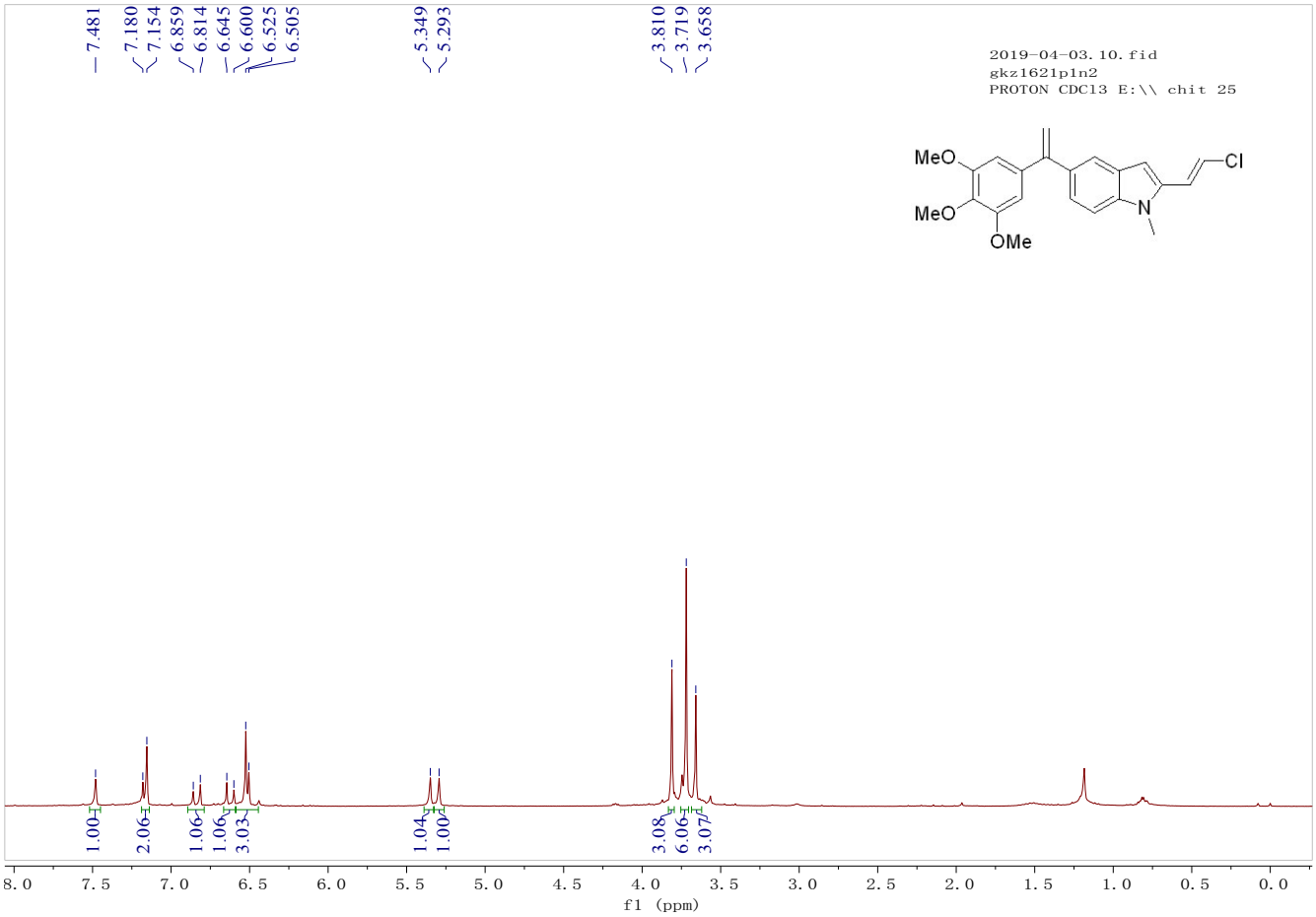

159.515
138.841
138.054
130.629
130.004
128.079
127.727
121.501
120.266
119.817
114.951
114.241
109.076
98.389
55.372
29.885
2017-03-10.111.fid
gkz507p2
JMOD CDCl3 E:\\ chit 39
f1 (ppm)

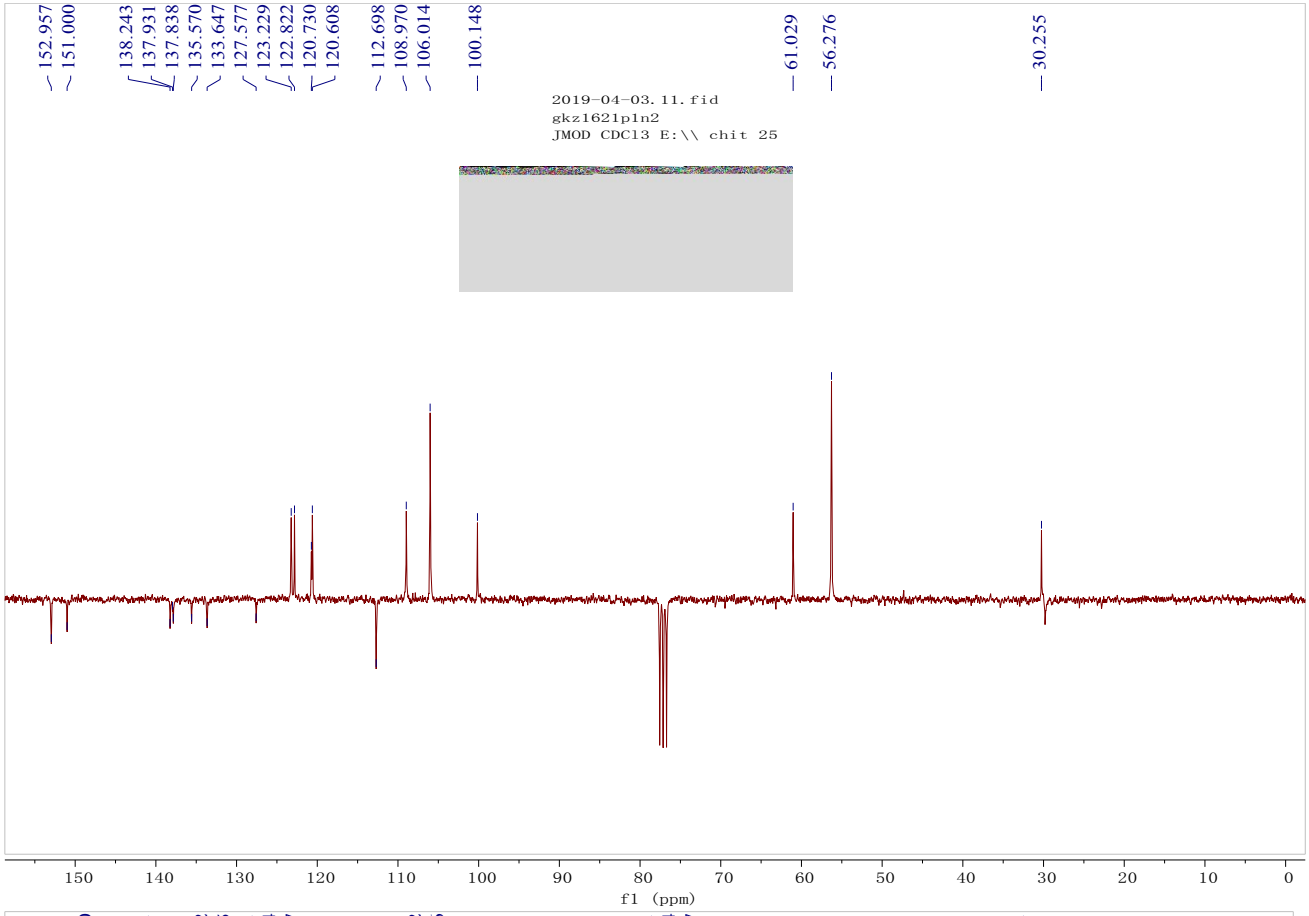

8.001
7.973
7.642
7.630
7.618
7.602
7.351
7.341
7.324
7.287
7.279
7.245
7.225
7.190
7.162
7.136
7.113
6.898
3.861
2.638
2017-03-08.10.fid
gkz495n3
PROTON CDCl3 E:\\ chit 3
2.00
3.05
5.40
1.00
3.05
3.04
f1 (ppm)

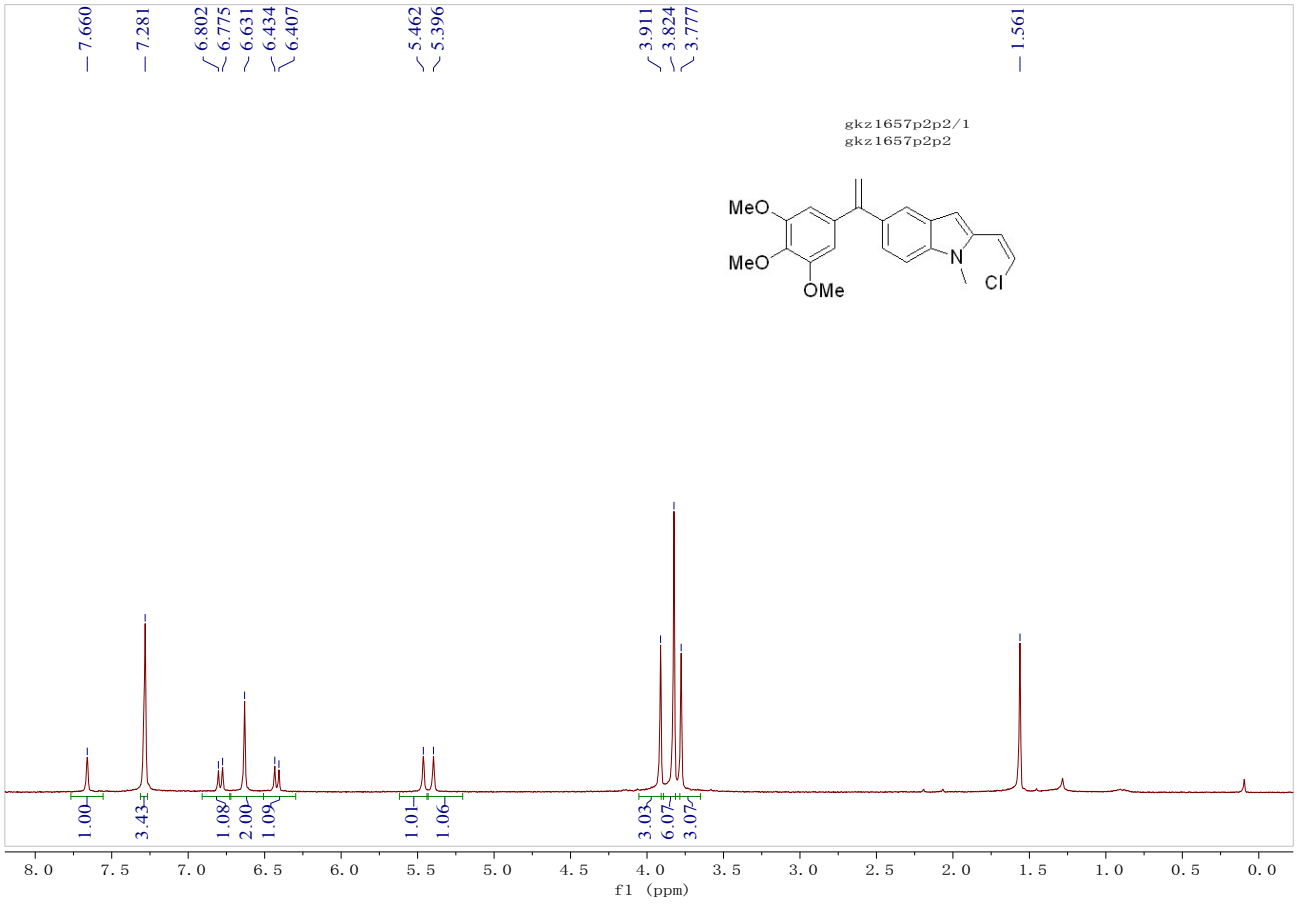

197.505
141.890
138.502
137.791
136.097
129.283
129.054
127.970
126.472
122.428
120.788
120.224
119.746
109.383
100.220
30.089
26.714
2017-03-08.11.fid
gkz495n3
JMOD CDCl3 E:\\ chit 3
f1 (ppm)

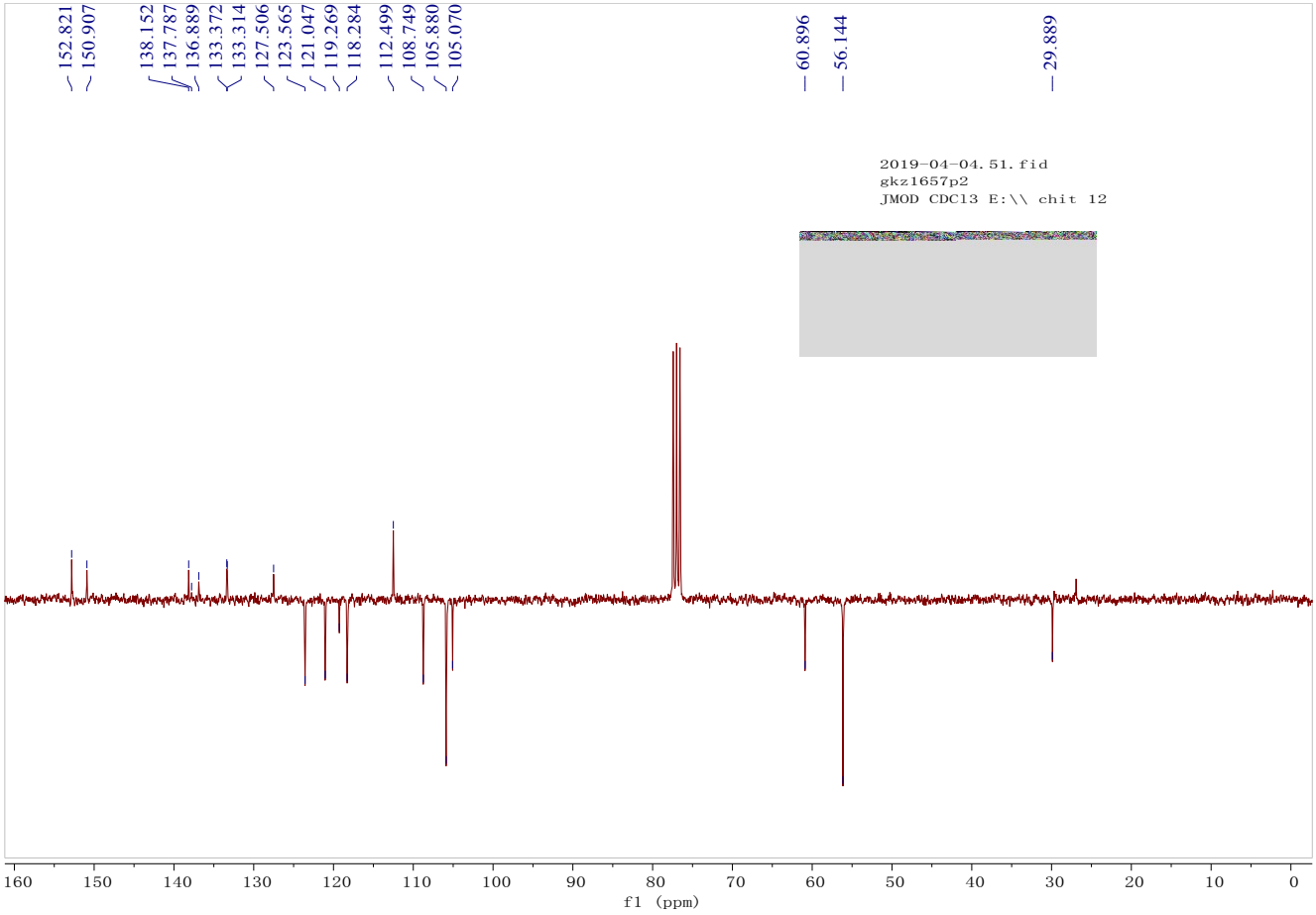

7.501
7.475
7.218
7.190
7.177
7.158
7.135
7.108
7.042
7.019
6.993
6.865
6.821
6.638
6.593
6.521
3.647
2018-11-15.10.fid
gkz1376
PROTON CDCl3 E:\\ chit 15
Cl
N
1.00
2.11
1.04
1.06
1.00
1.01
3.02
f1 (ppm)

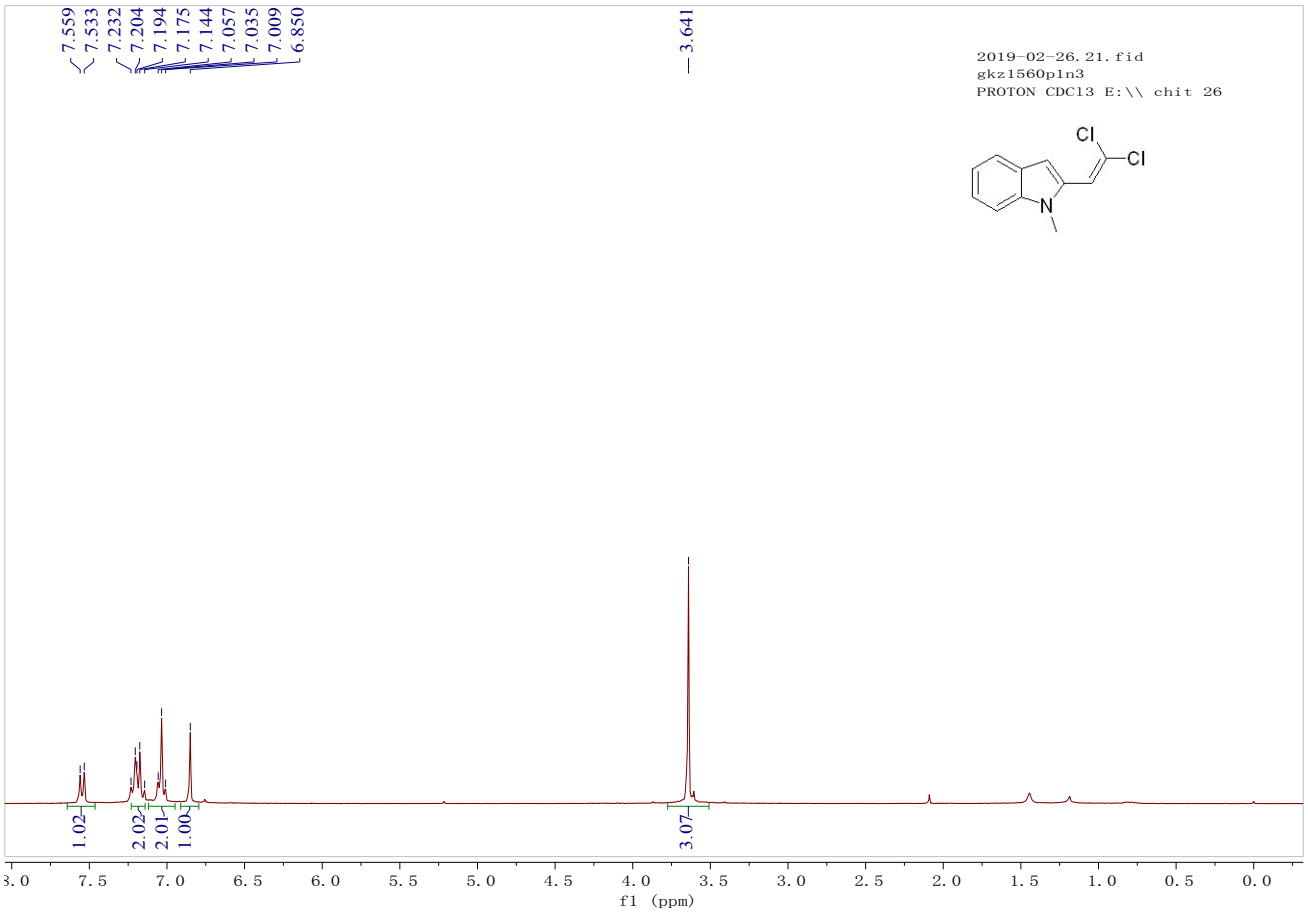

138.048
134.998
127.761
123.000
122.325
120.727
120.479
120.194
109.397
99.883
30.119
2018-11-15.11.fid
gkz1376
JMOD CDCl3 E:\\ chit 15
f1 (ppm)

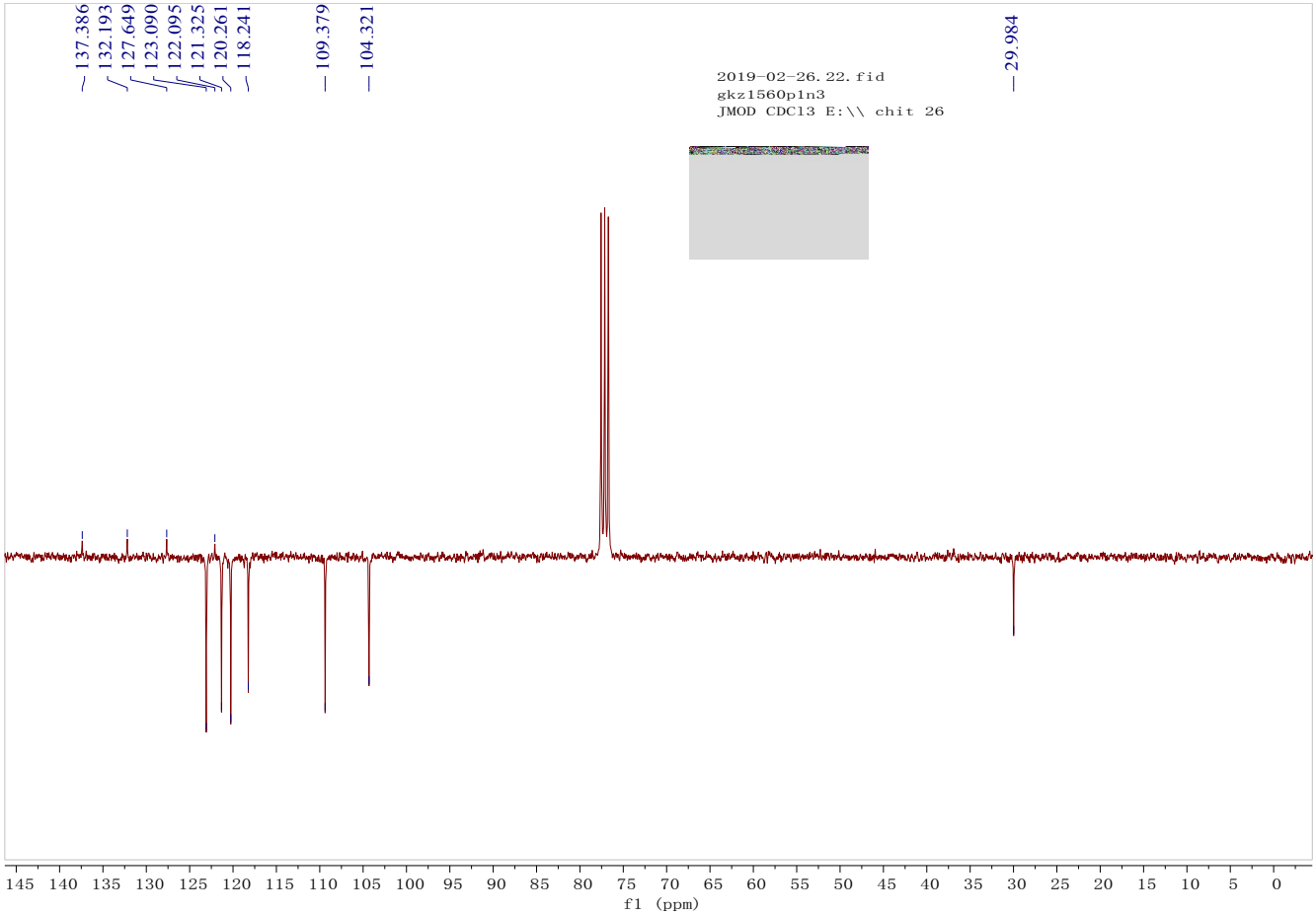

7.481
7.180
7.154
6.859
6.814
6.645
6.600
6.525
6.505
5.349
5.293
3.810
3.719
3.658
2019-04-03.10.fid
gkz1621p1n2
PROTON CDCl3 E:\\ chit 25
MeO
MeO
OMe
N
Cl
1.00
2.06
1.06
1.06
3.03
1.04
1.00
3.08
6.06
3.07
f1 (ppm)

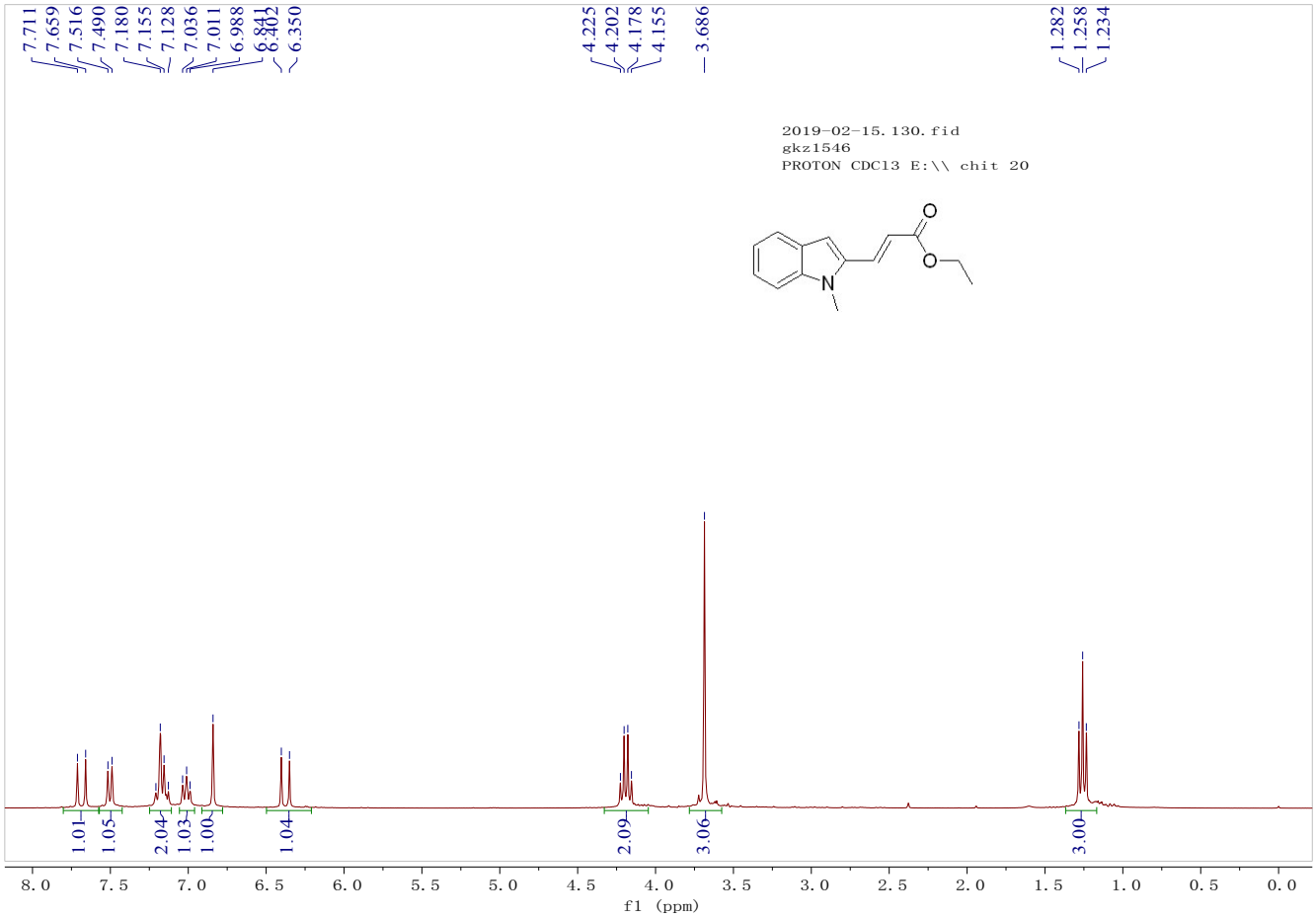

152.957
151.000
138.243
137.931
137.838
135.570
133.647
127.577
123.229
122.822
120.730
120.608
112.698
108.970
106.014
100.148
61.029
56.276
30.255
2019-04-03.11.fid
gkz1621p1n2
JMOD CDCl3 E:\\ chit 25
f1 (ppm)

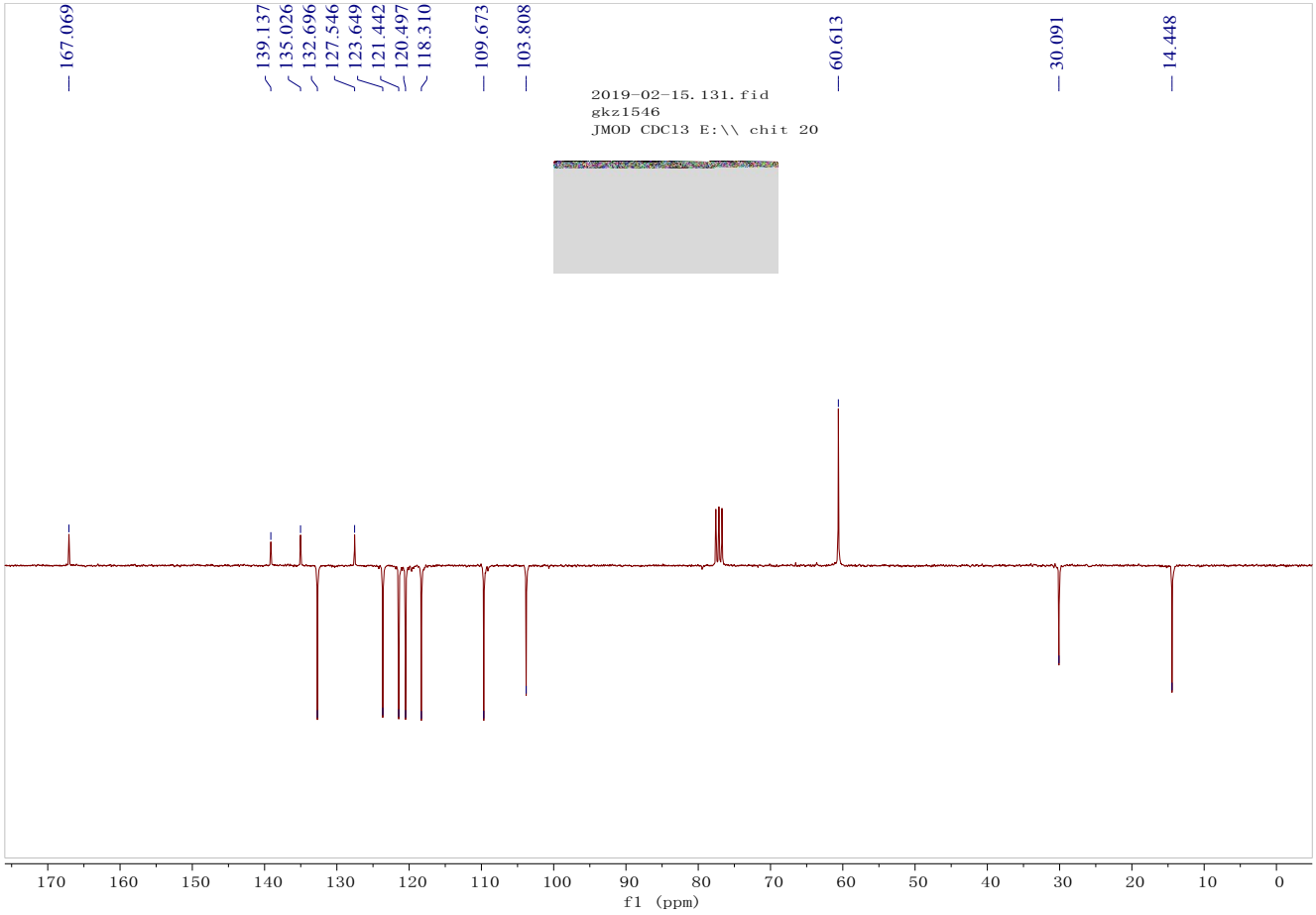

7.660
7.281
6.802
6.775
6.631
6.434
6.407
5.462
5.396
3.911
3.824
3.777
1.561
gkz1657p2p2/1
gkz1657p2p2
MeO
MeO
OMe
N
Cl
1.00
3.43
1.08
2.00
1.09
1.01
1.06
3.03
6.07
3.07
f1 (ppm)

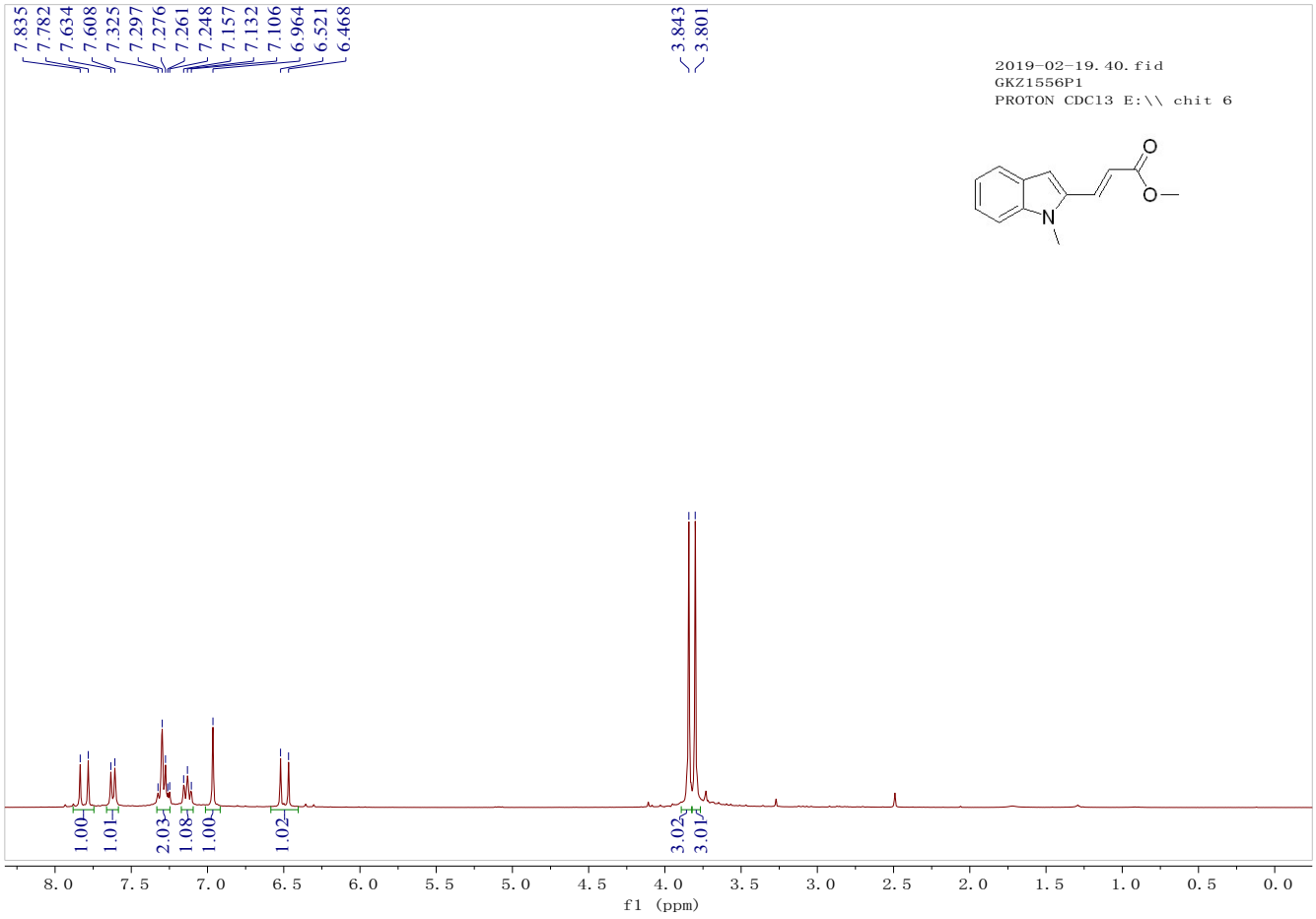

152.821
150.907
138.152
137.787
136.889
133.372
133.314
127.506
123.565
121.047
119.269
118.284
112.499
108.749
105.880
105.070
60.896
56.144
29.889
2019-04-04.51.fid
gkz1657p2
JMOD CDCl3 E:\\ chit 12
f1 (ppm)

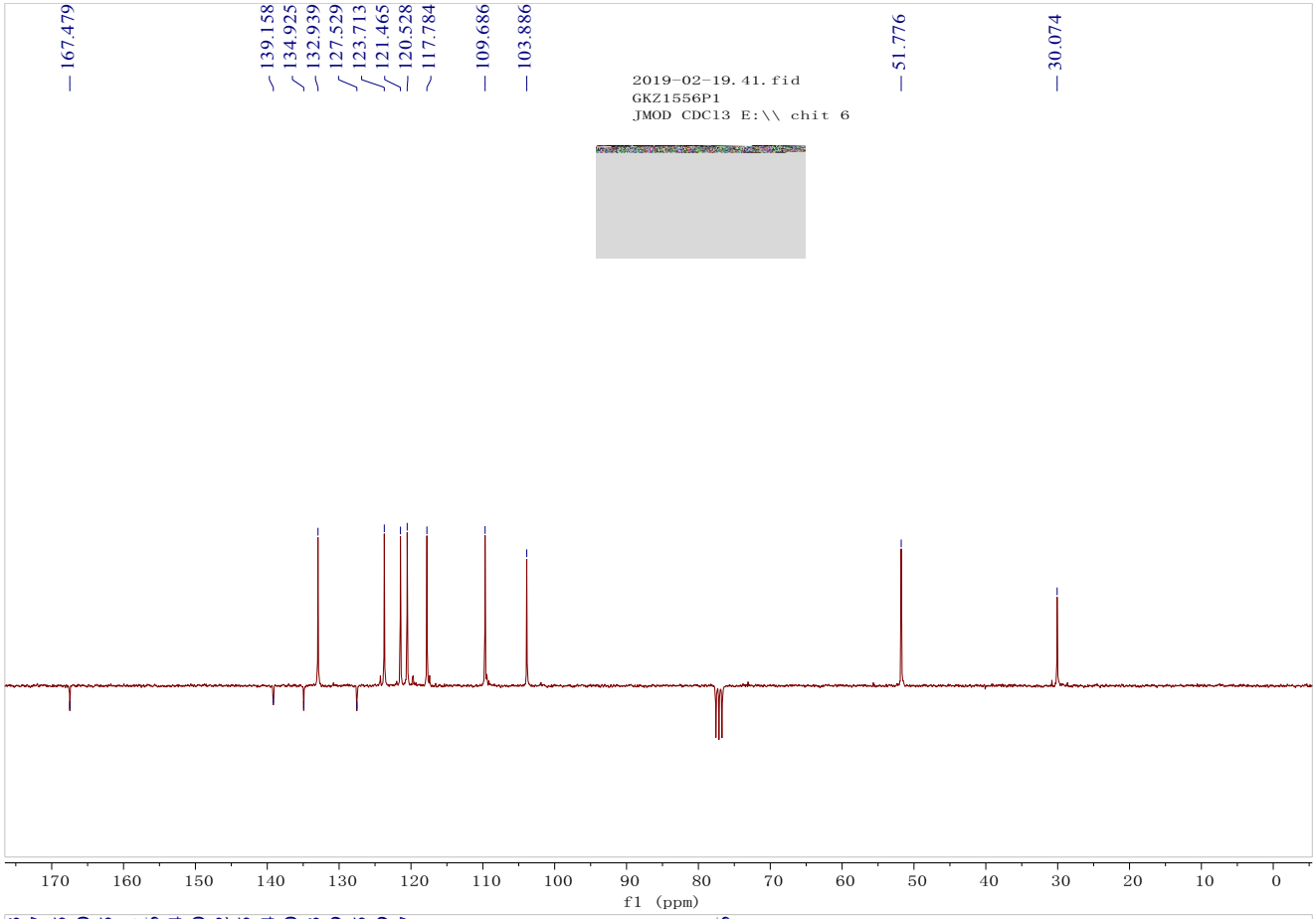

7.559
7.533
7.232
7.204
7.194
7.175
7.144
7.057
7.035
7.009
6.850
3.641
2019-02-26.21.fid
gkz1560p1n3
PROTON CDCl3 E:\\ chit 26
Cl
Cl
N
1.02
2.02
2.01
1.00
3.07
f1 (ppm)

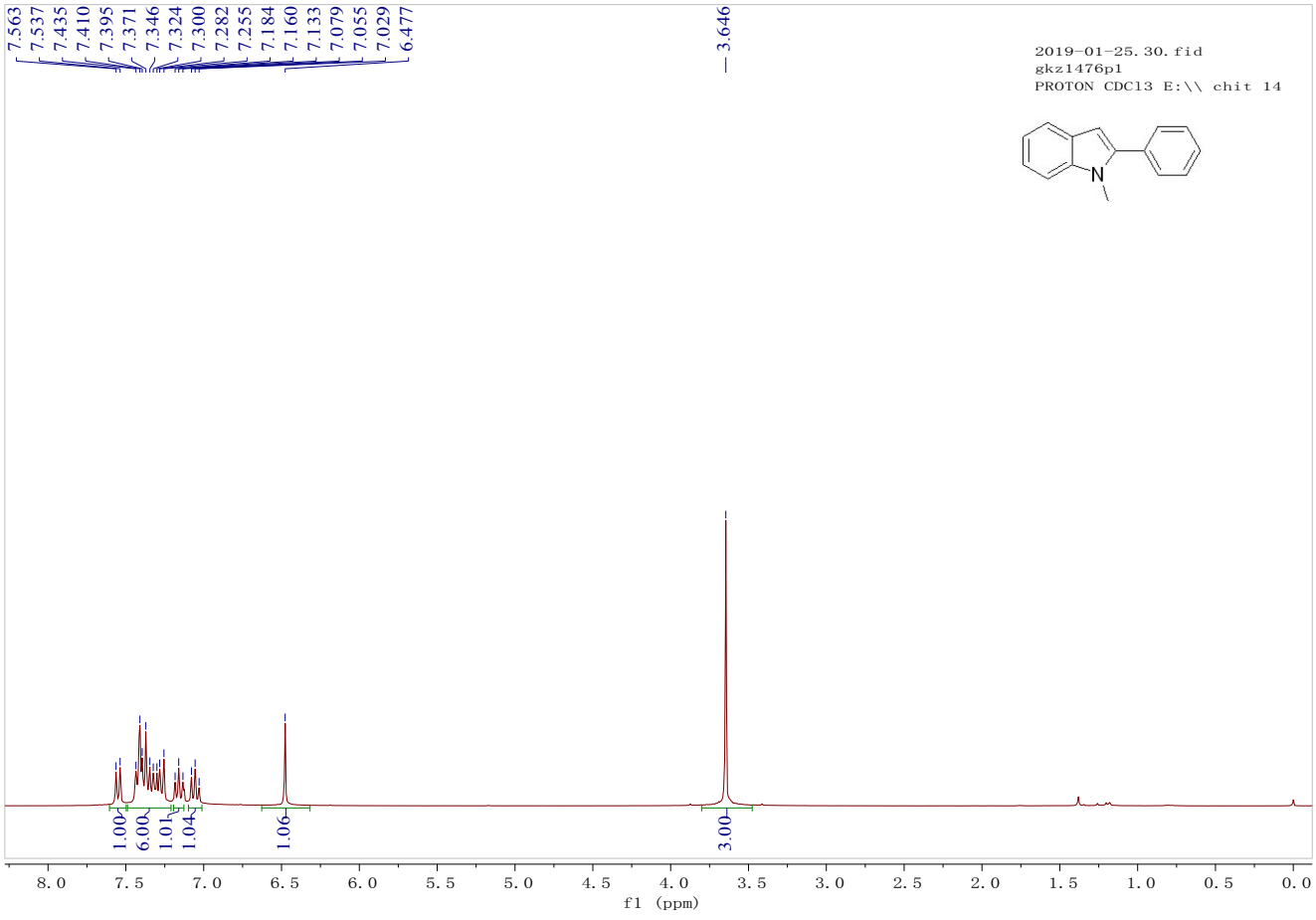

137.386
132.193
127.649
123.090
122.095
121.325
120.261
118.241
109.379
104.321
29.984
2019-02-26.22.fid
gkz1560p1n3
JMOD CDCl3 E:\\ chit 26
f1 (ppm)

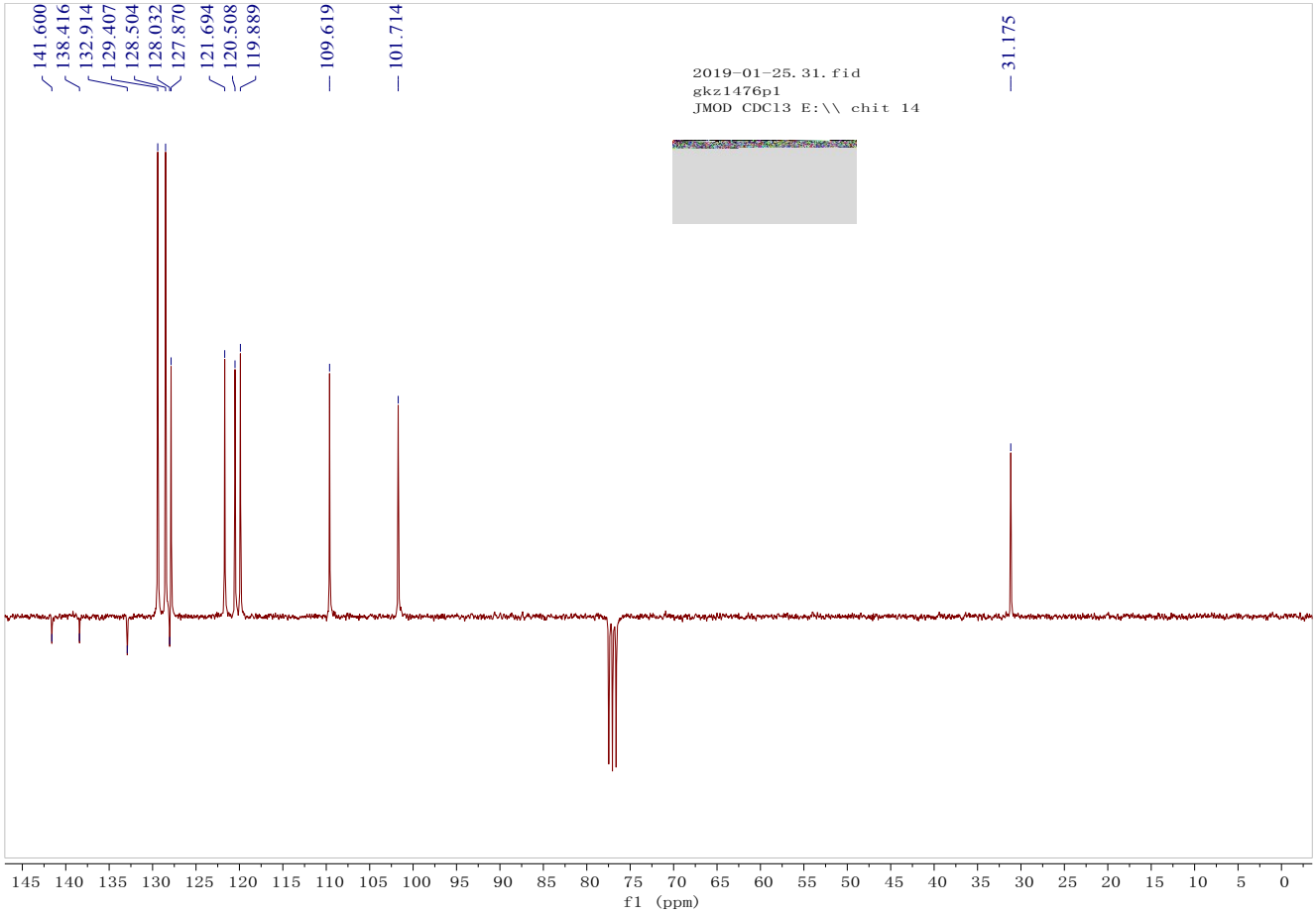

7.711
7.659
7.516
7.490
7.180
7.155
7.128
7.036
7.011
6.988
6.841
6.402
6.350
4.225
4.202
4.178
4.155
3.686
1.282
1.258
1.234
2019-02-15.130.fid
gkz1546
PROTON CDCl3 E:\\ chit 20
O
O
N
1.01
1.05
2.04
1.03
1.00
1.04
2.09
3.06
3.00
f1 (ppm)

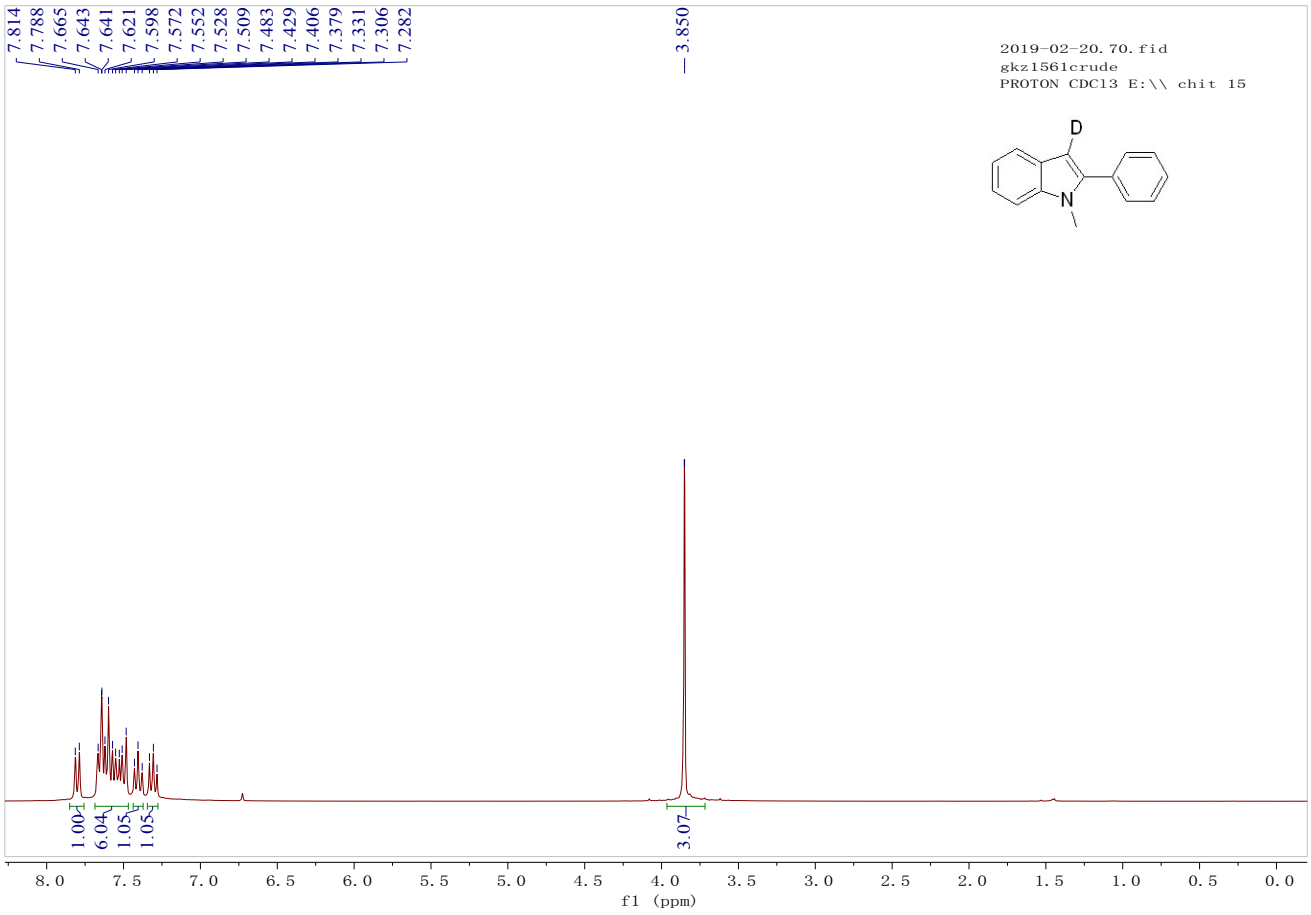

167.069
139.137
135.026
132.696
127.546
123.649
121.442
120.497
118.310
109.673
103.808
60.613
30.091
14.448
2019-02-15.131.fid
gkz1546
JMOD CDCl3 E:\\ chit 20
f1 (ppm)

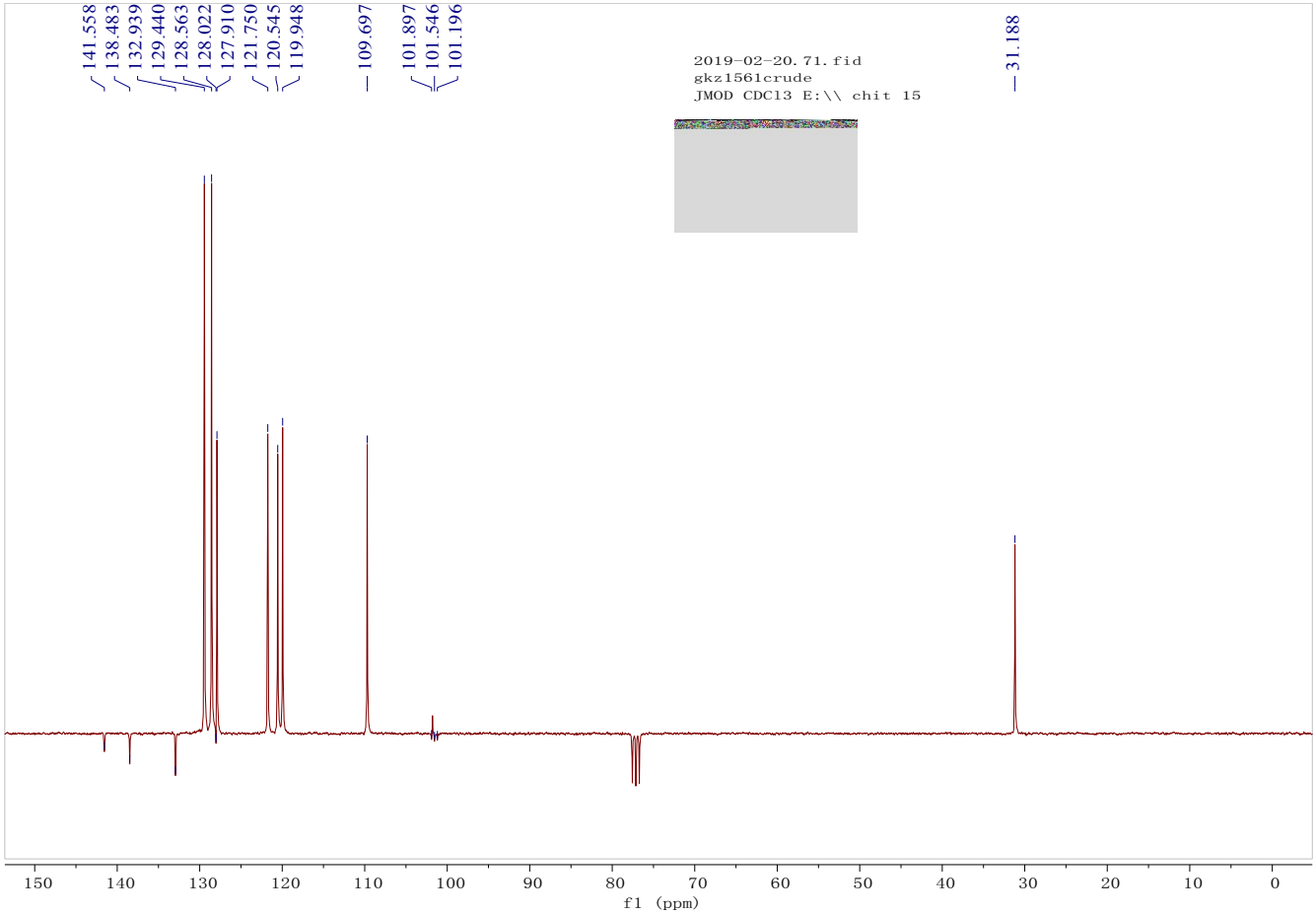

7.835
7.782
7.634
7.608
7.325
7.297
7.276
7.261
7.248
7.157
7.132
7.106
6.964
6.521
6.468
3.843
3.801
2019-02-19.40.fid
GKZ1556P1
PROTON CDCl3 E:\\ chit 6
O
O
N
1.00
1.01
2.03
1.08
1.00
1.02
3.02
3.01
f1 (ppm)

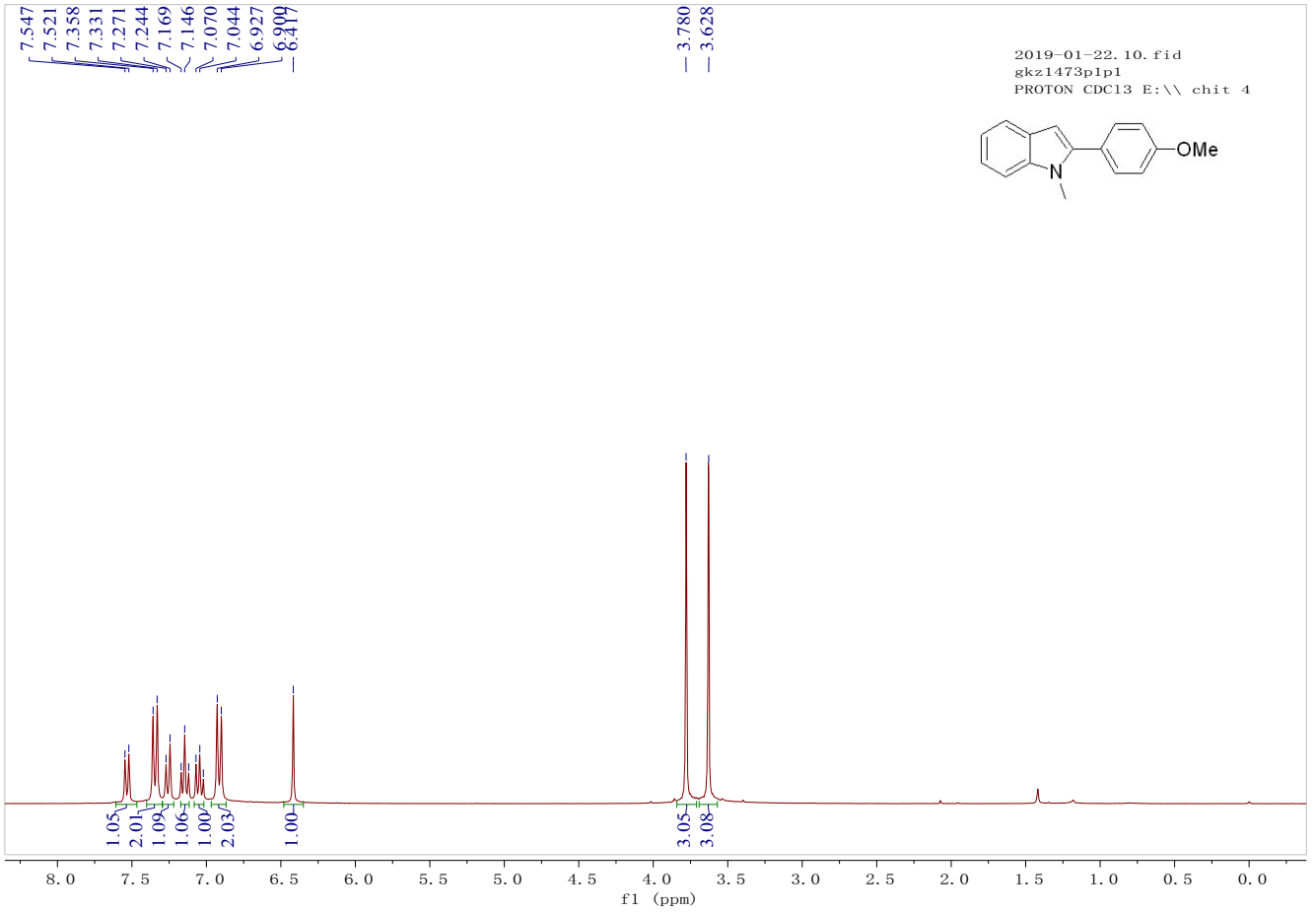

167.479
139.158
134.925
132.939
127.529
123.713
121.465
120.528
117.784
109.686
103.886
51.776
30.074
2019-02-19.41.fid
GKZ1556P1
JMOD CDC13 E:\\ chit 6
f1 (ppm)

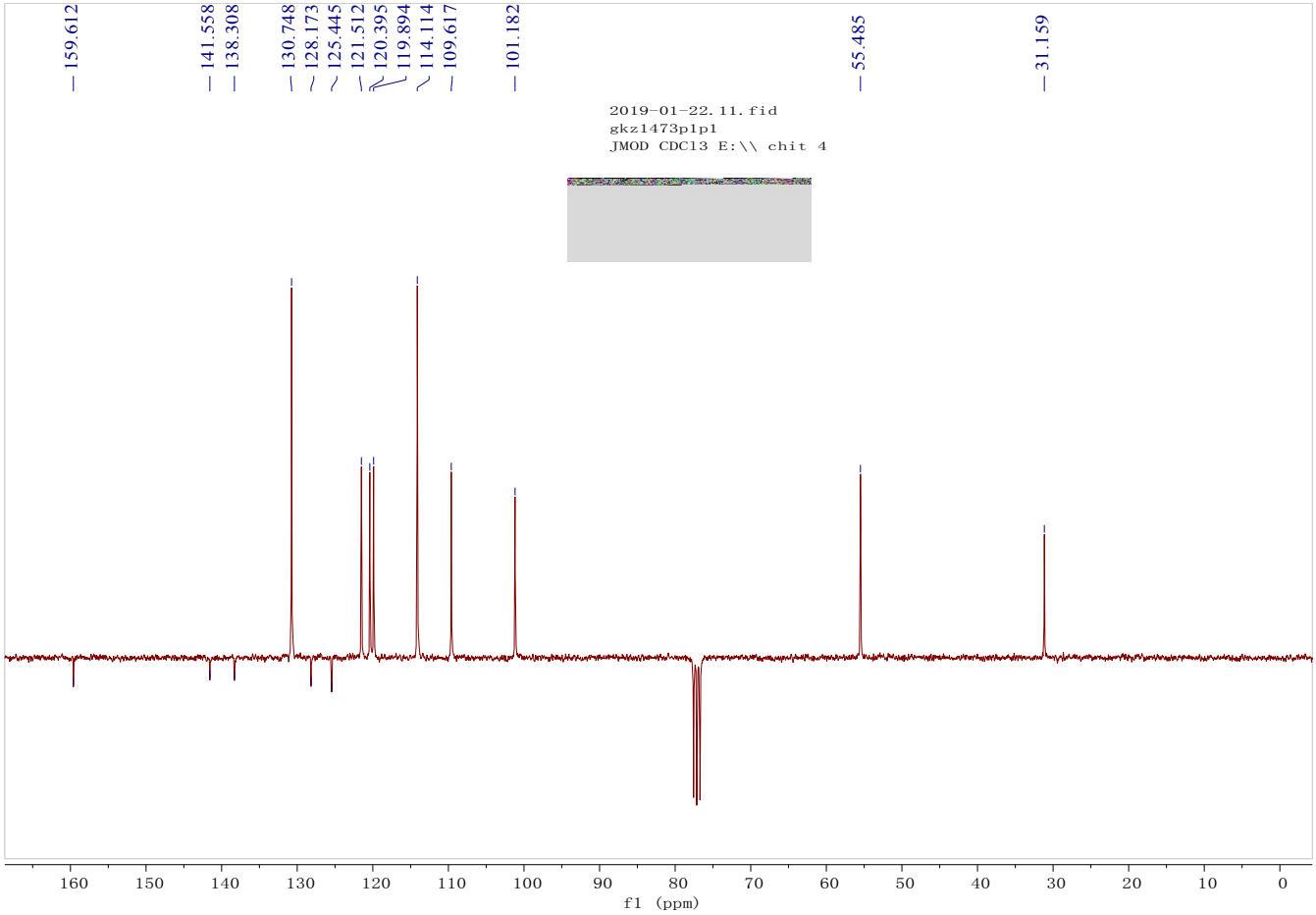

7.563
7.537
7.435
7.410
7.395
7.371
7.346
7.324
7.300
7.282
7.255
7.184
7.160
7.133
7.079
7.055
7.029
6.477
3.646
2019-01-25.30.fid
gkz1476p1
PROTON CDC13 E:\\ chit 14
N
1.00
6.00
1.01
1.04
1.06
3.00
f1 (ppm)

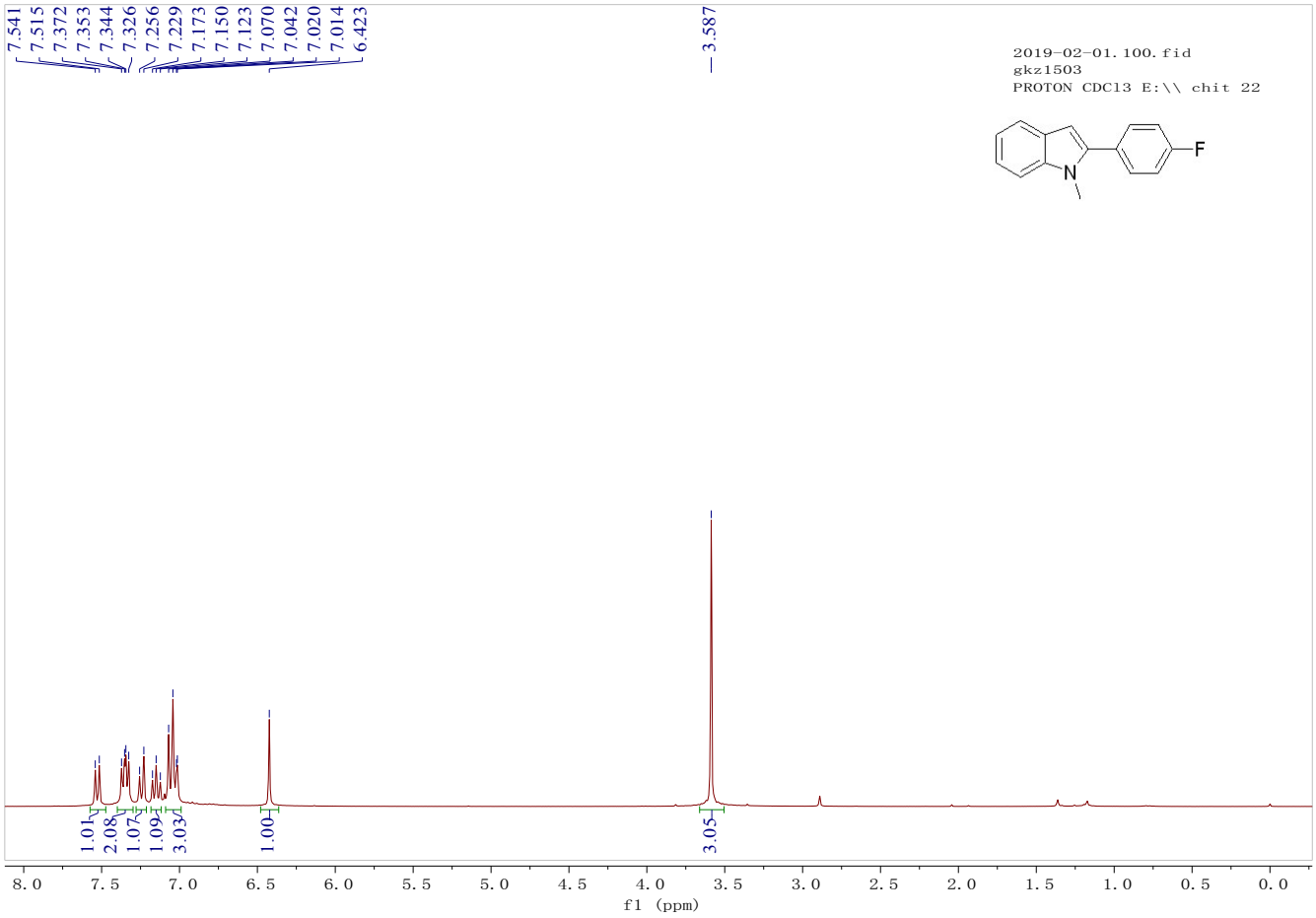

141.600
138.416
132.914
129.407
128.504
128.032
127.870
121.694
120.508
119.889
109.619
101.714
31.175
2019-01-25.31.fid
gkz1476p1
JMOD CDCl3 E:\\ chit 14
f1 (ppm)

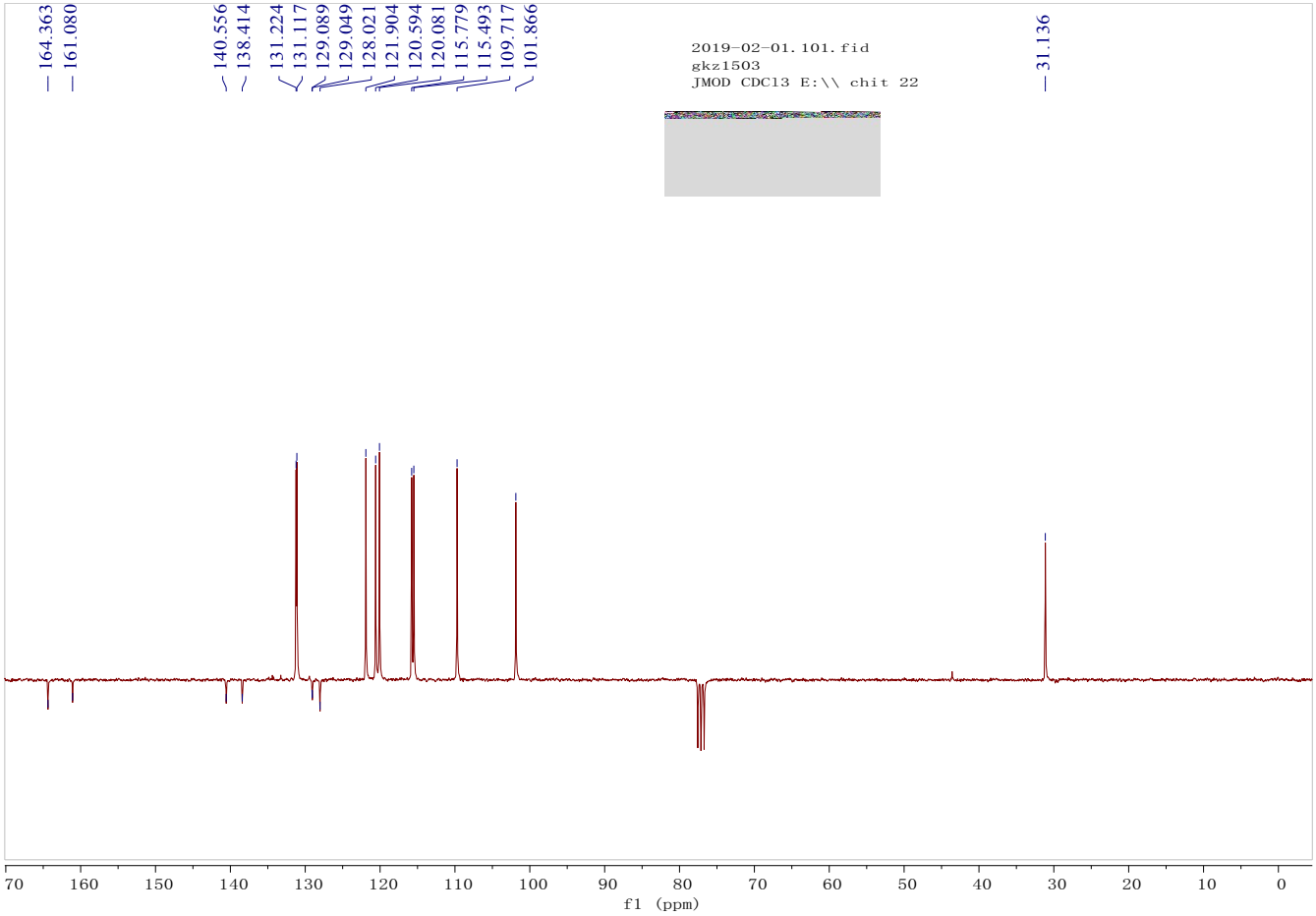

7.814
7.788
7.665
7.643
7.641
7.621
7.598
7.572
7.552
7.528
7.509
7.483
7.429
7.406
7.379
7.331
7.306
7.282
3.850
2019-02-20.70.fid
gkz1561crude
PROTON CDCl3 E:\\ chit 15
D
N
1.00
6.04
1.05
1.05
3.07
f1 (ppm)

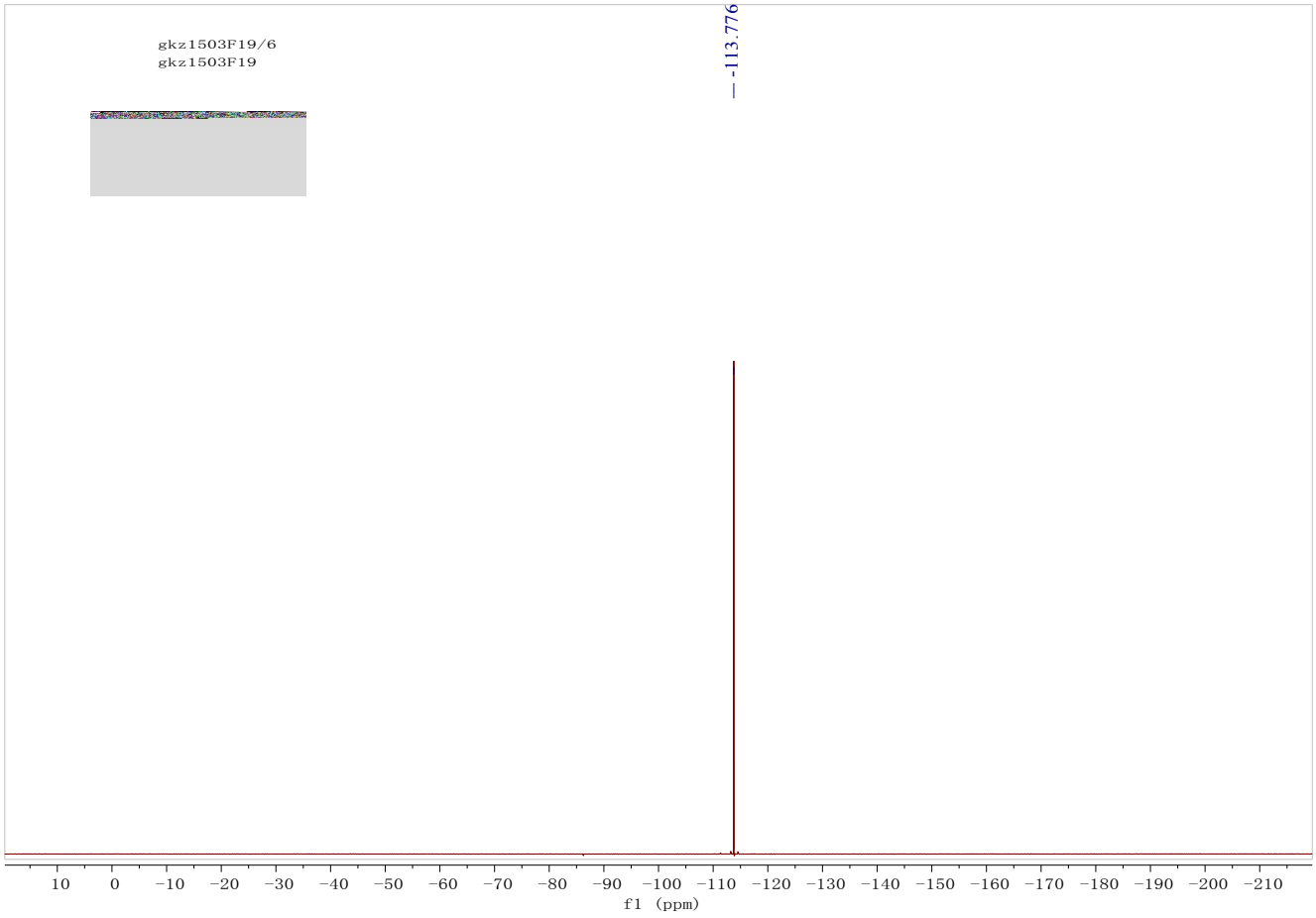

141.558
138.483
132.939
129.440
128.563
128.022
127.910
121.750
120.545
119.948
109.697
101.897
101.546
101.196
31.188
2019-02-20.71.fid
gkz1561crude
JMOD CDCl3 E:\\ chit 15
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

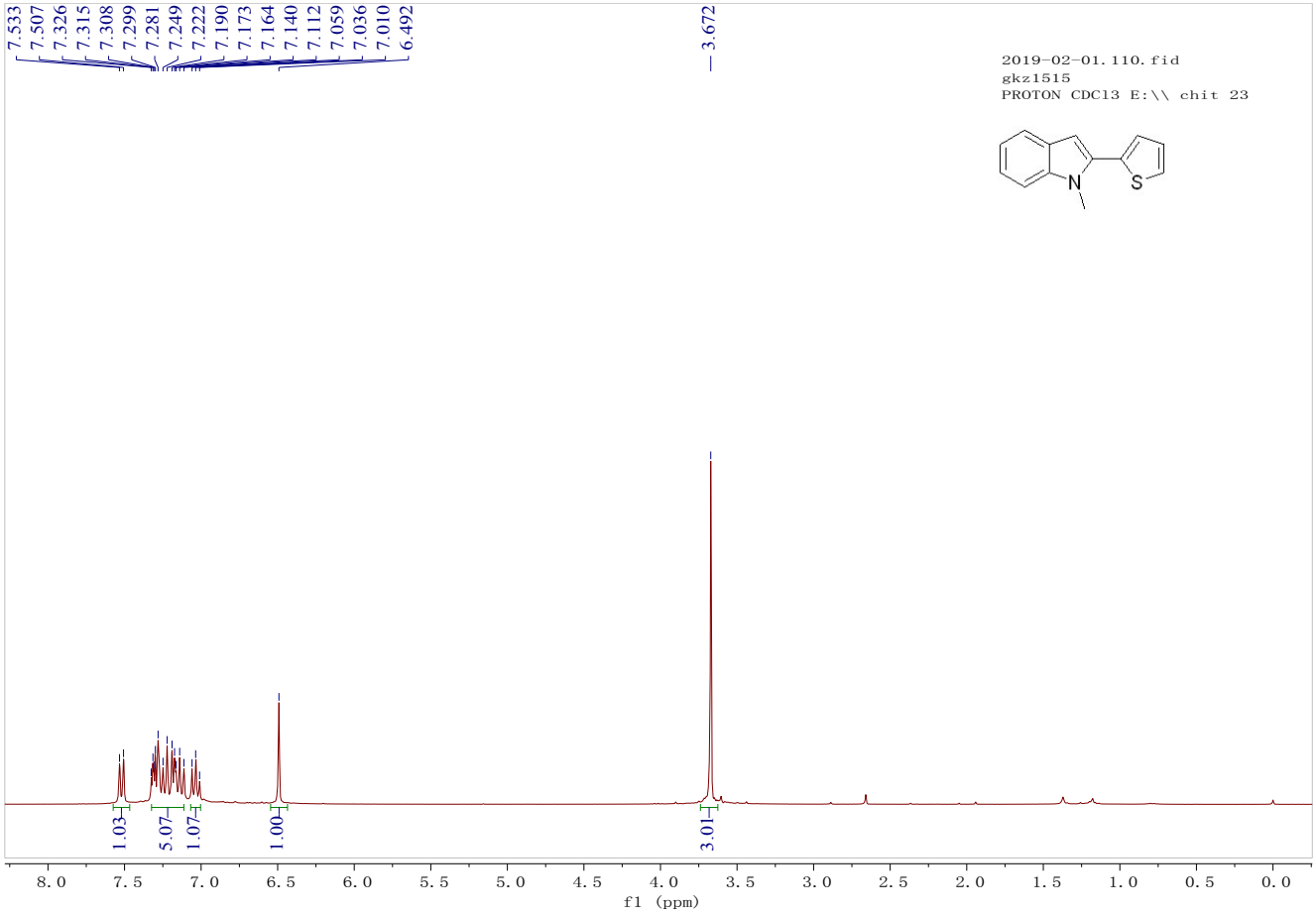

7.547
7.521
7.358
7.331
7.271
7.244
7.169
7.146
7.070
7.044
6.927
6.900
6.417
3.780
3.628
2019-01-22.10.fid
gkz1473p1p1
PROTON CDCl3 E:\\ chit 4
OMe
N
1.05
2.01
1.09
1.06
1.00
2.03
1.00
3.05
3.08
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

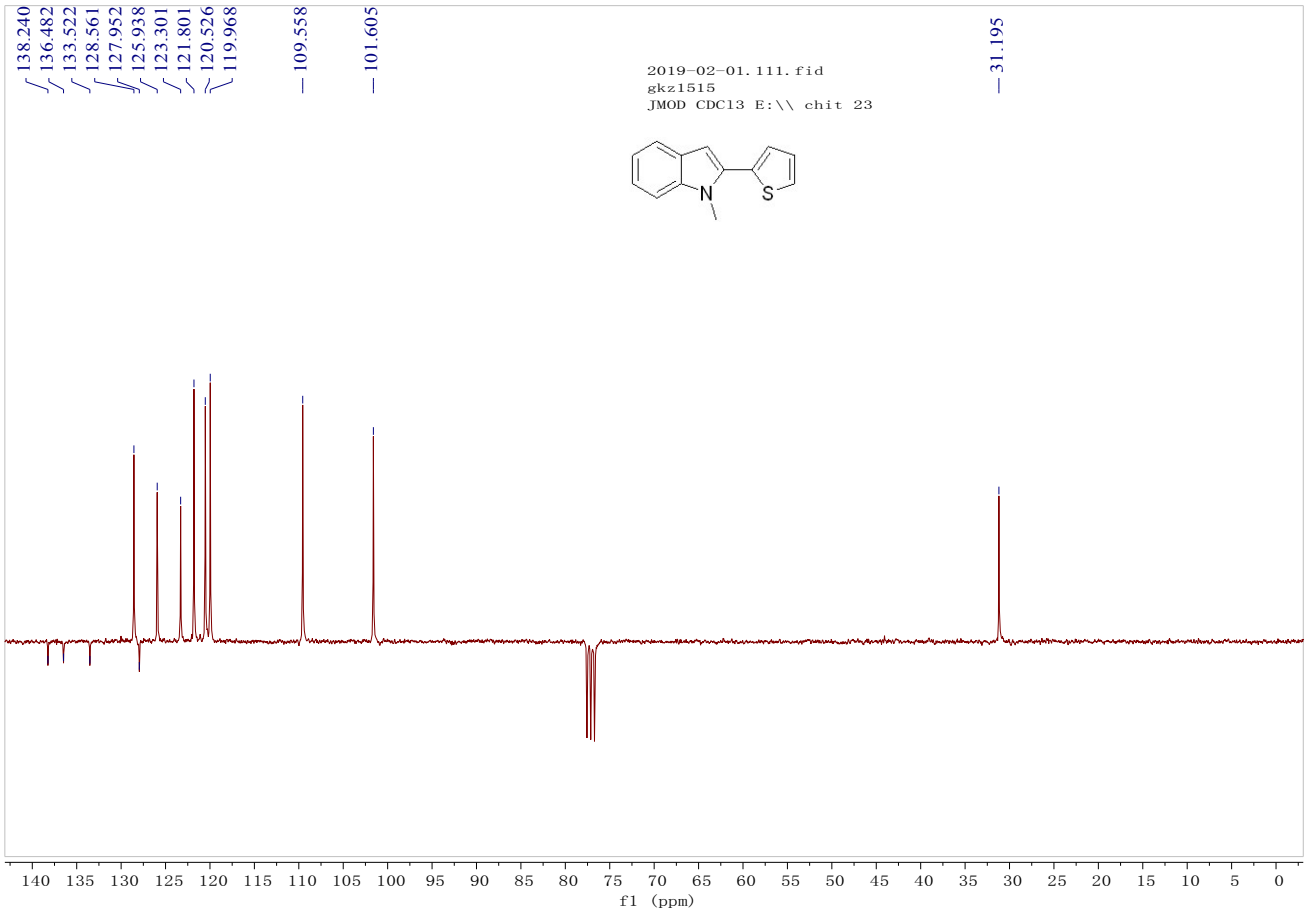

159.612
141.558
138.308
130.748
128.173
125.445
121.512
120.395
119.894
114.114
109.617
101.182
55.485
31.159
2019-01-22.11.fid
gkz1473p1p1
JMOD CDC13 E:\\ chit 4
f1 (ppm)

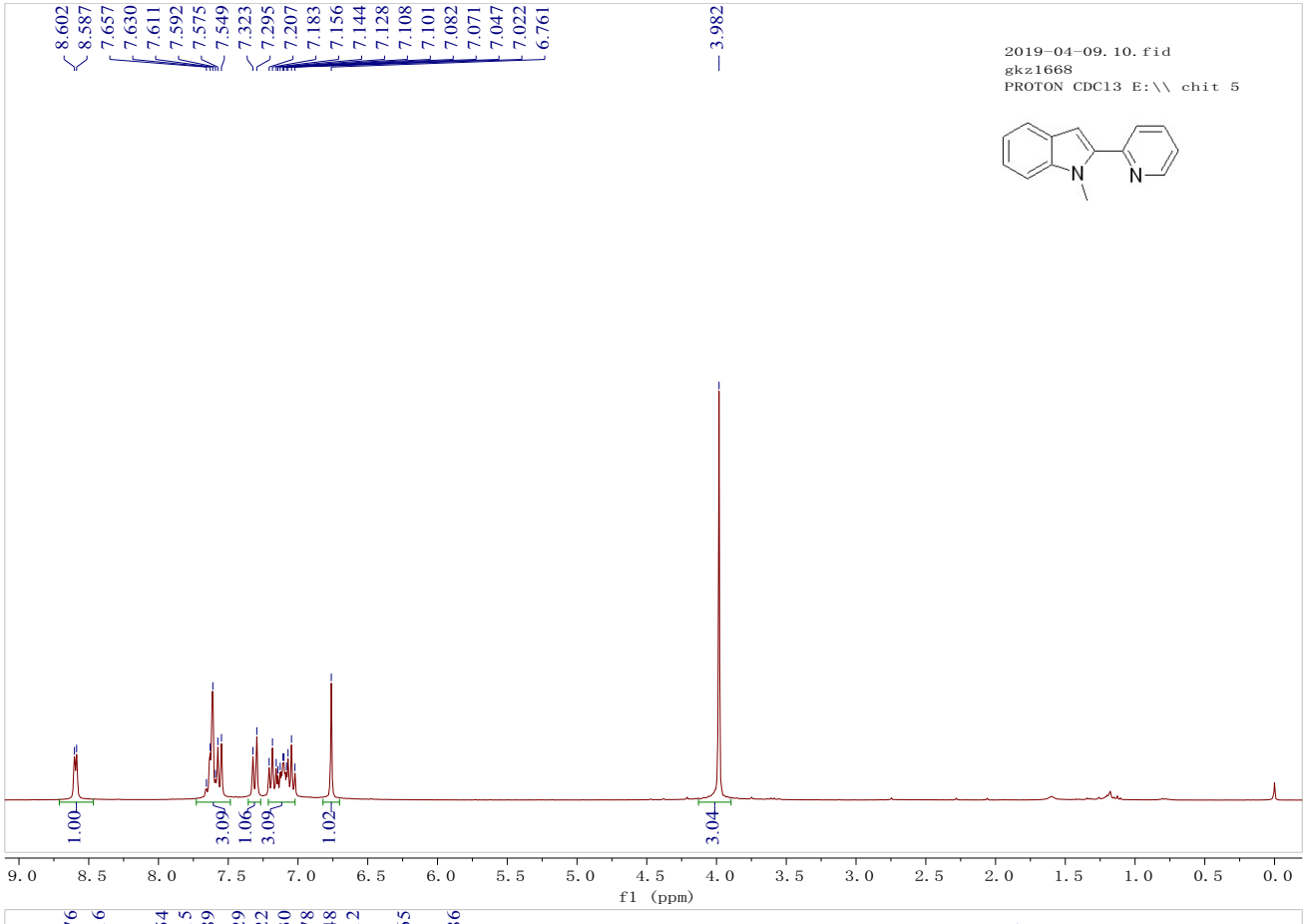

7.541
7.515
7.372
7.353
7.344
7.326
7.256
7.229
7.173
7.150
7.123
7.070
7.042
7.020
7.014
6.423
3.587
2019-02-01.100.fid
gkz1503
PROTON CDC13 E:\\ chit 22
F
N
1.01
2.08
1.07
1.09
3.03
1.00
3.05
f1 (ppm)

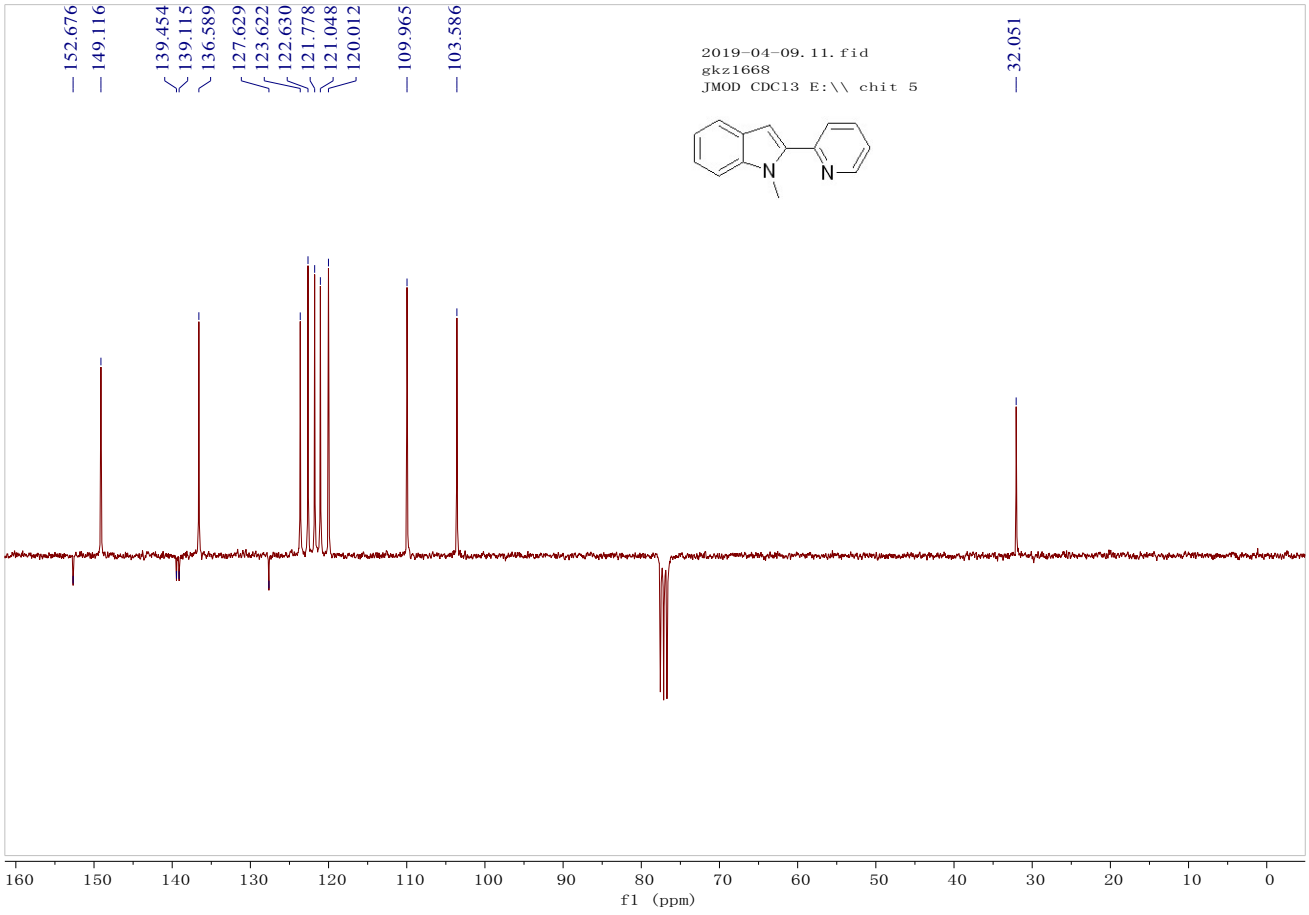

164.363
161.080
140.556
138.414
131.224
131.117
129.089
129.049
128.021
121.904
120.594
120.081
115.779
115.493
109.717
101.866
31.136
2019-02-01.101.fid
gkz1503
JMOD CDC13 E:\\ chit 22
f1 (ppm)

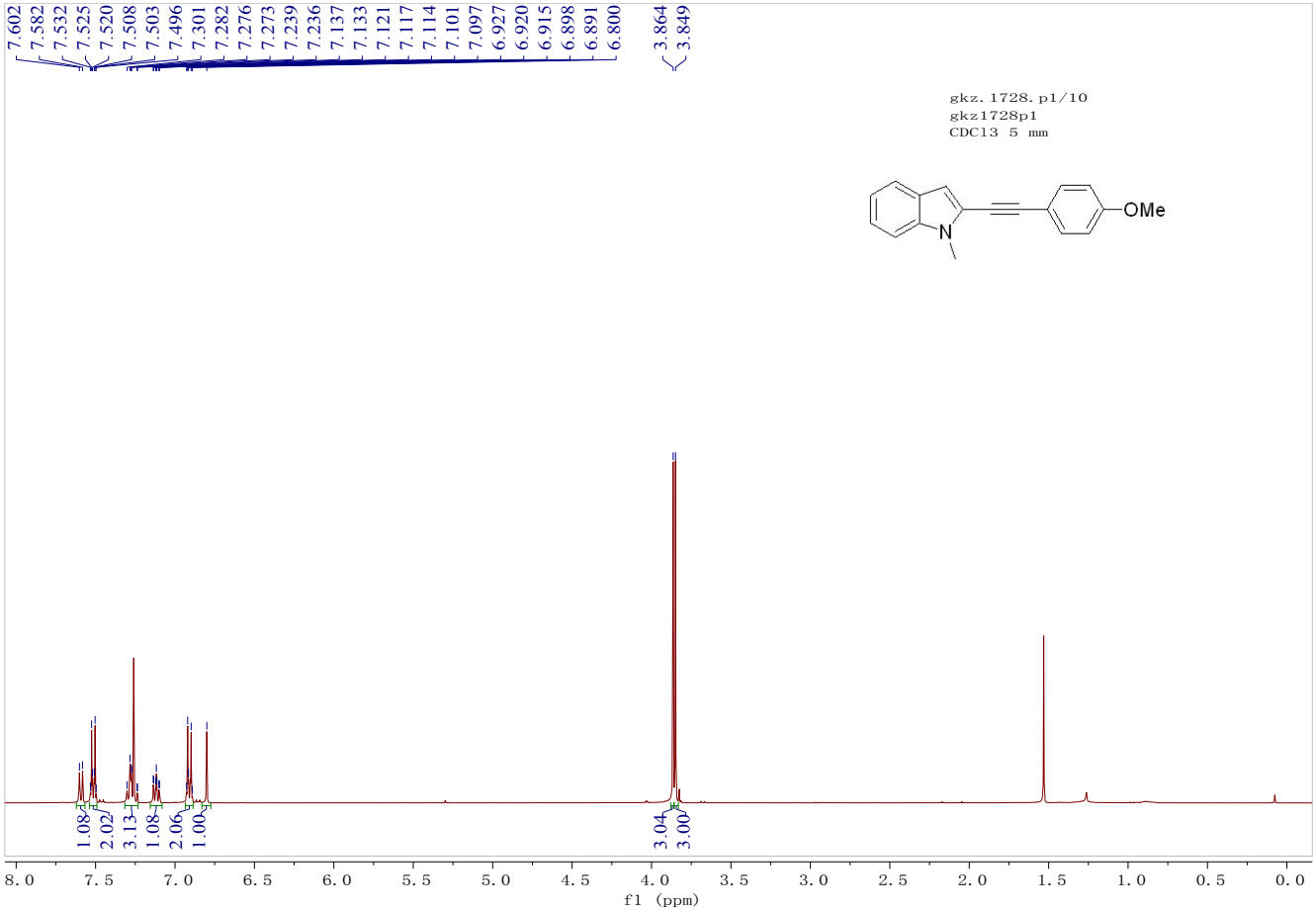

gkz1503F19/6
gkz1503F19
-113.776
f1 (ppm)


7.533
7.507
7.326
7.315
7.308
7.299
7.281
7.249
7.222
7.190
7.173
7.164
7.140
7.112
7.059
7.036
7.010
6.492
3.672
2019-02-01.110.fid
gkz1515
PROTON CDCl3 E:\\ chit 23
N
S
1.03
5.07
1.07
1.00
3.01
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


138.240
136.482
133.522
128.561
127.952
125.938
123.301
121.801
120.526
119.968
109.558
101.605
31.195
2019-02-01.111.fid
gkz1515
JMOD CDCl3 E:\\ chit 23
N
S
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
f1 (ppm)


8.602
8.587
7.657
7.630
7.611
7.592
7.575
7.549
7.323
7.295
7.207
7.183
7.156
7.144
7.128
7.108
7.101
7.082
7.071
7.047
7.022
6.761
3.982
2019-04-09.10.fid
gkz1668
PROTON CDCl3 E:\\ chit 5
1.00
3.09
1.06
3.09
1.02
3.04
f1 (ppm)


152.676
149.116
139.454
139.115
136.589
127.629
123.622
122.630
121.778
121.048
120.012
109.965
103.586
32.051
2019-04-09.11.fid
gkz1668
JMOD CDCl3 E:\\ chit 5
f1 (ppm)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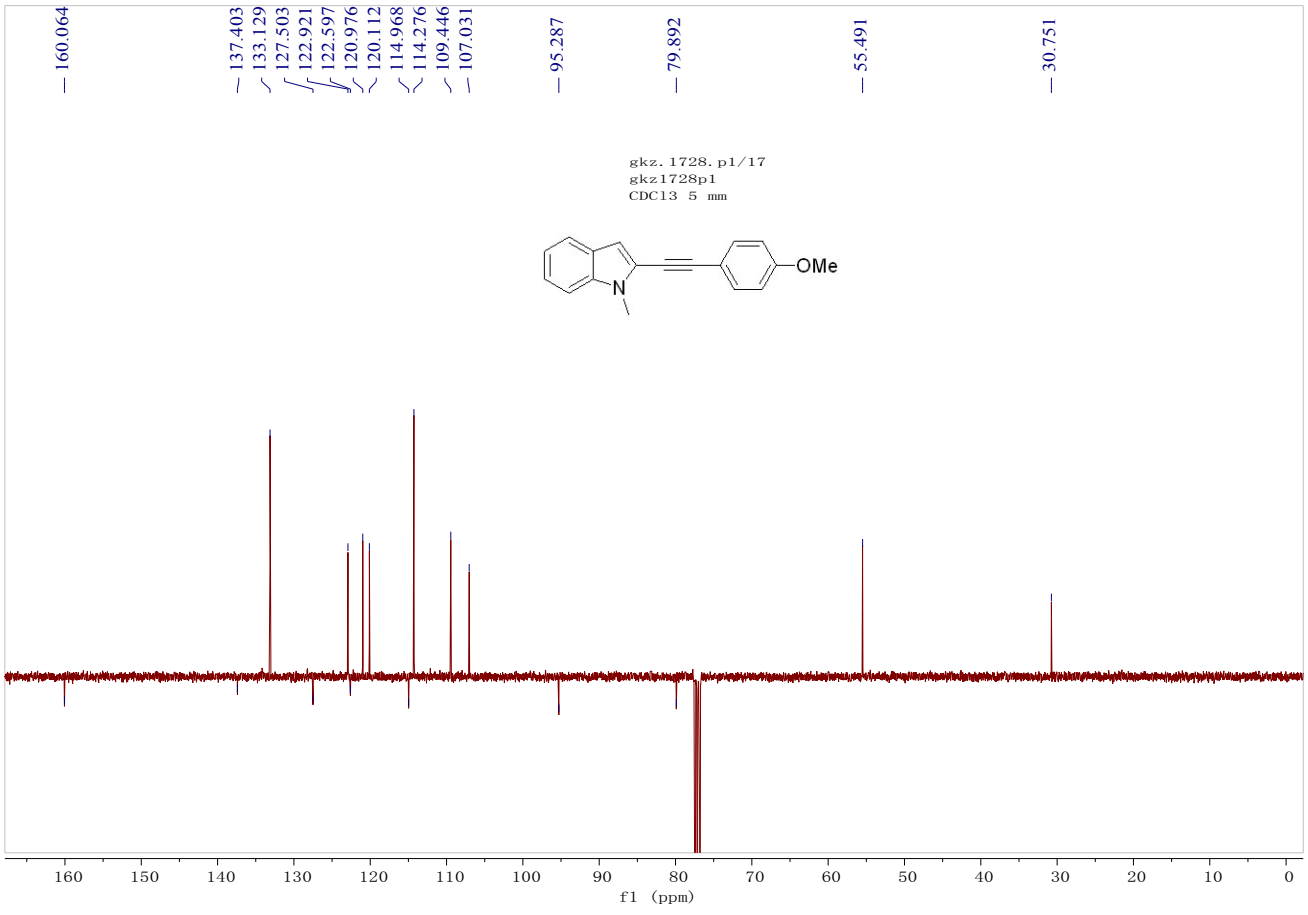
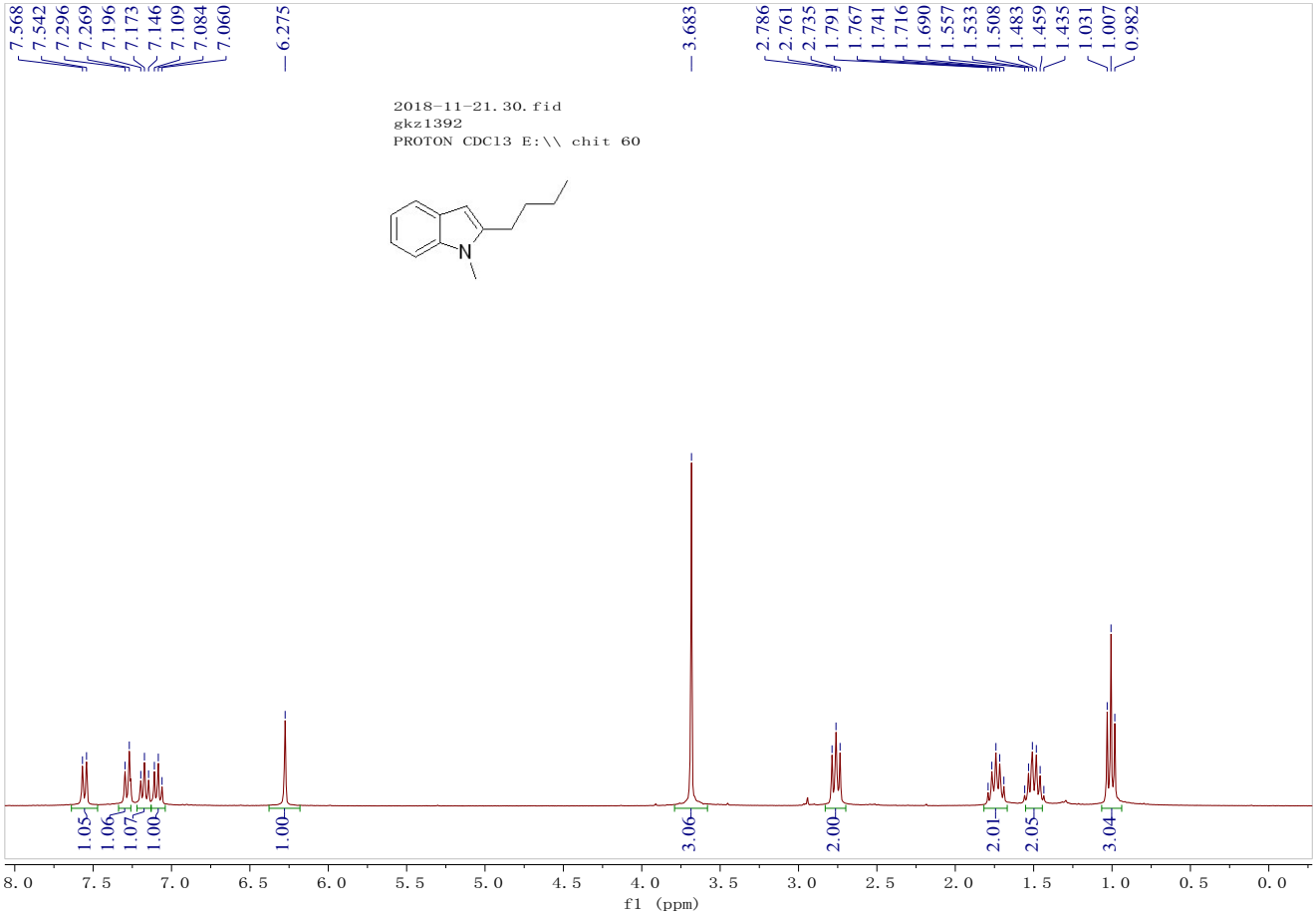
7.525
7.520
7.508
7.503
7.496
7.301
7.282
7.276
7.273
7.239
7.236
7.137
7.133
7.121
7.117
7.114
7.101
7.097
6.927
6.920
6.915
6.898
6.891
6.800
3.864
3.849
gkz. 1728. p1/10
gkz1728p1
CDC13 5 mm
OMe
N
1.08
2.02
3.13
1.08
2.06
1.00
3.04
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


160.064
137.403
133.129
127.503
122.921
122.597
120.976
120.112
114.968
114.276
109.446
107.031
95.287
79.892
55.491
30.751
gkz. 1728. p1/17
gkz1728p1
CDC13 5 mm
OMe
N
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.568
7.542
7.296
7.269
7.196
7.173
7.146
7.109
7.084
7.060
6.275
3.683
2.786
2.761
2.735
1.791
1.767
1.741
1.716
1.690
1.557
1.533
1.508
1.483
1.459
1.435
1.031
1.007
0.982
2018-11-21.30.fid
gkz1392
PROTON CDCl3 E:\\ chit 60
N
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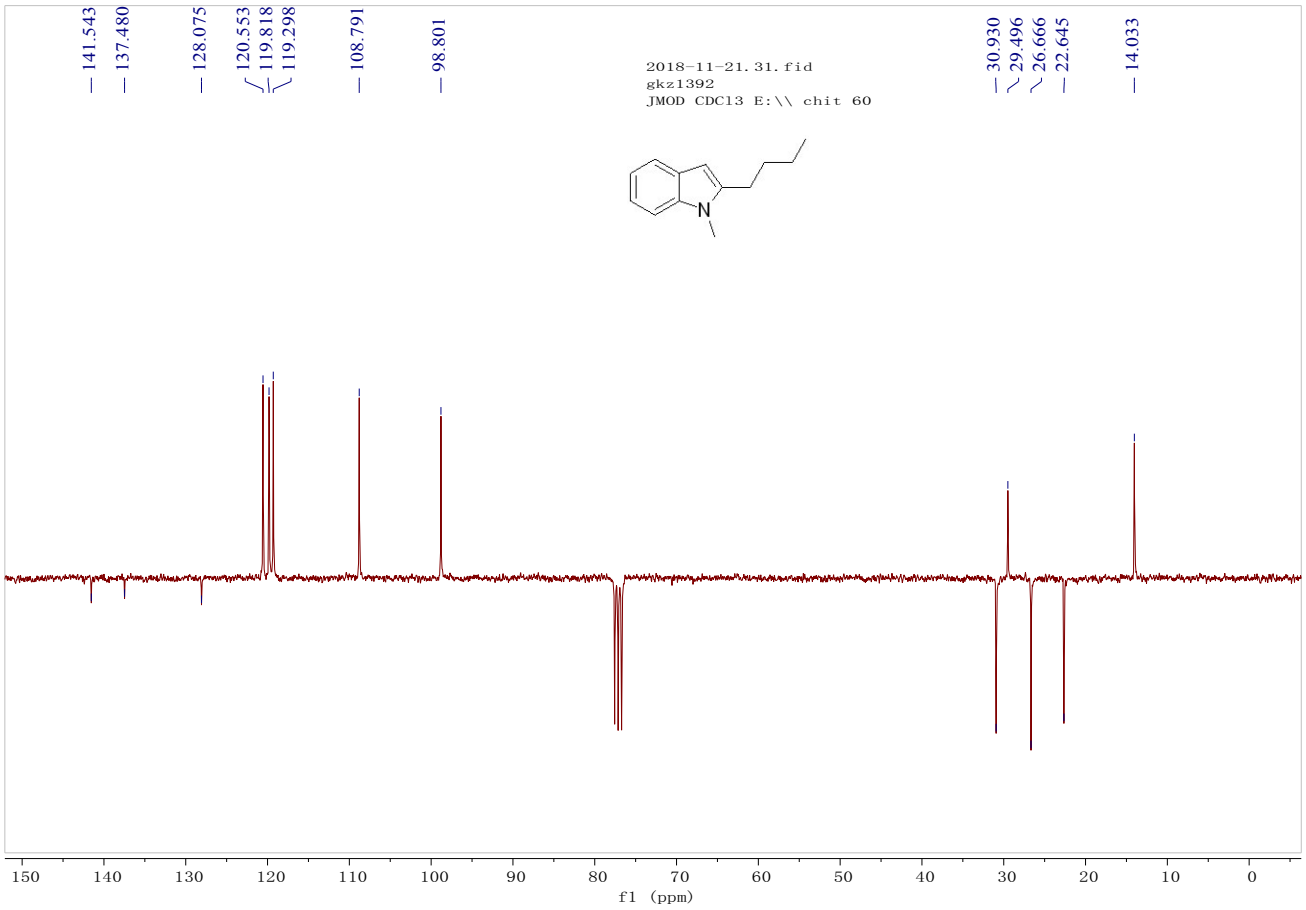
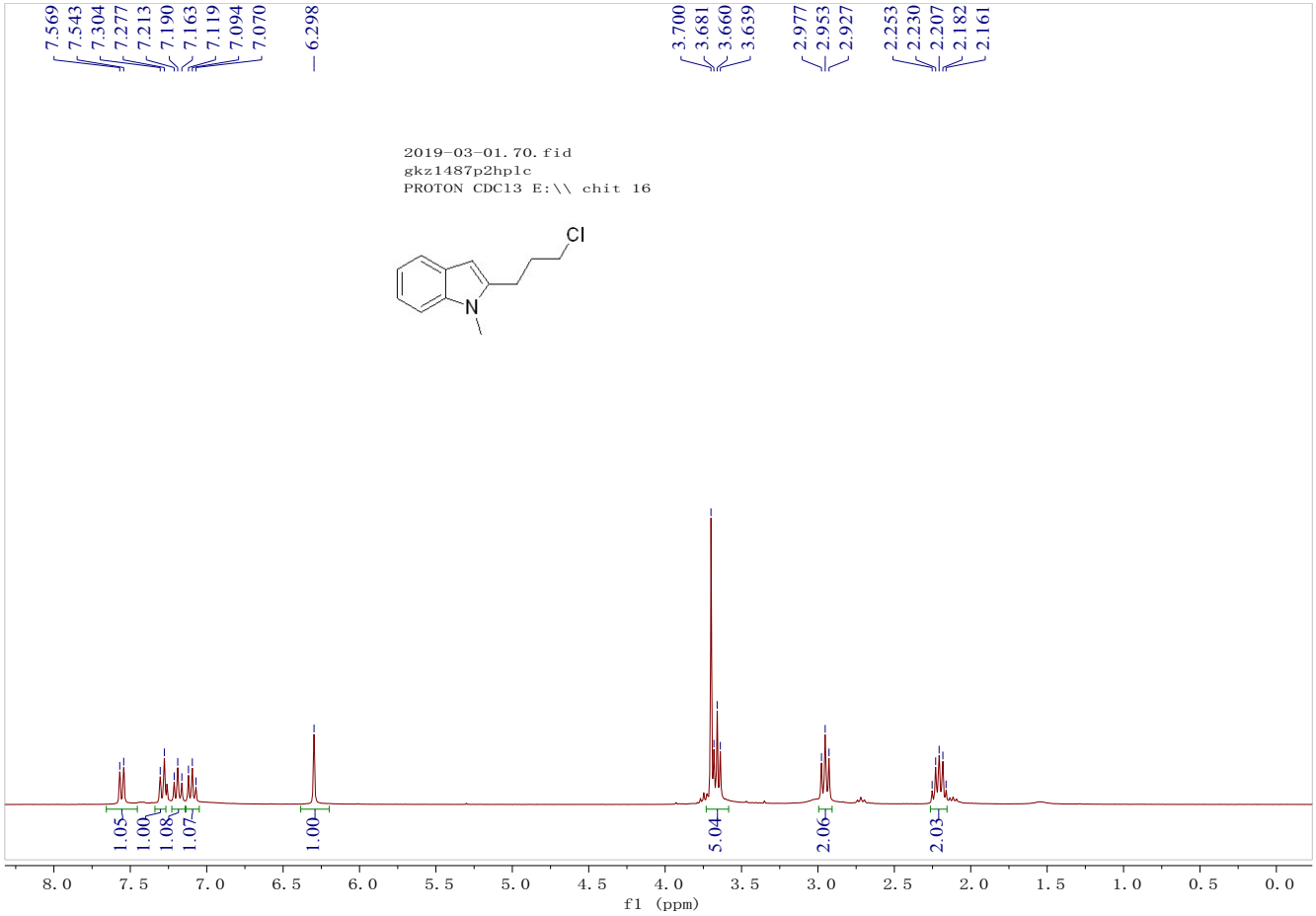
1.06
1.07
1.00
1.00
3.06
2.00
2.01
2.05
3.04
f1 (ppm)


141.543
137.480
128.075
120.553
119.818
119.298
108.791
98.801
30.930
29.496
26.666
22.645
14.033
2018-11-21.31.fid
gkz1392
JMOD CDCl3 E:\\ chit 60
N
f1 (ppm)


7.569
7.543
7.304
7.277
7.213
7.190
7.163
7.119
7.094
7.070
6.298
3.700
3.681
3.660
3.639
2.977
2.953
2.927
2.253
2.230
2.207
2.182
2.161
2019-03-01.70.fid
gkz1487p2hplc
PROTON CDCl3 E:\\ chit 16
Cl
N
1.05
1.00
1.08
1.07
1.00
5.04
2.06
2.03
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


139.297
137.569
127.918
120.955
119.964
119.526
108.942
99.389
44.333
31.531
29.568
23.951
2019-03-01.71.fid
gkz1487p2hplc
JMOD CDCl3 E:\\ chit 16
Cl
N
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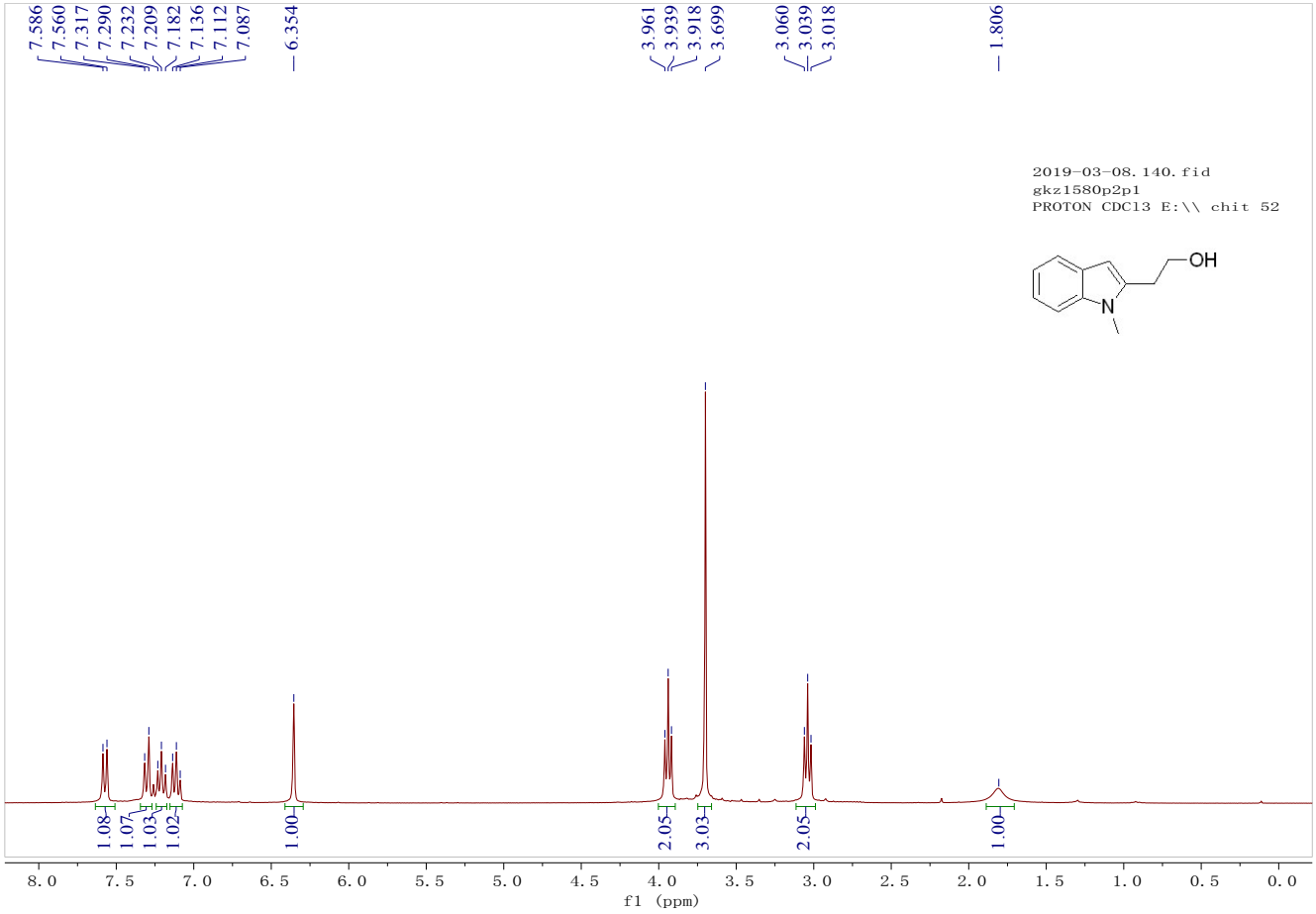
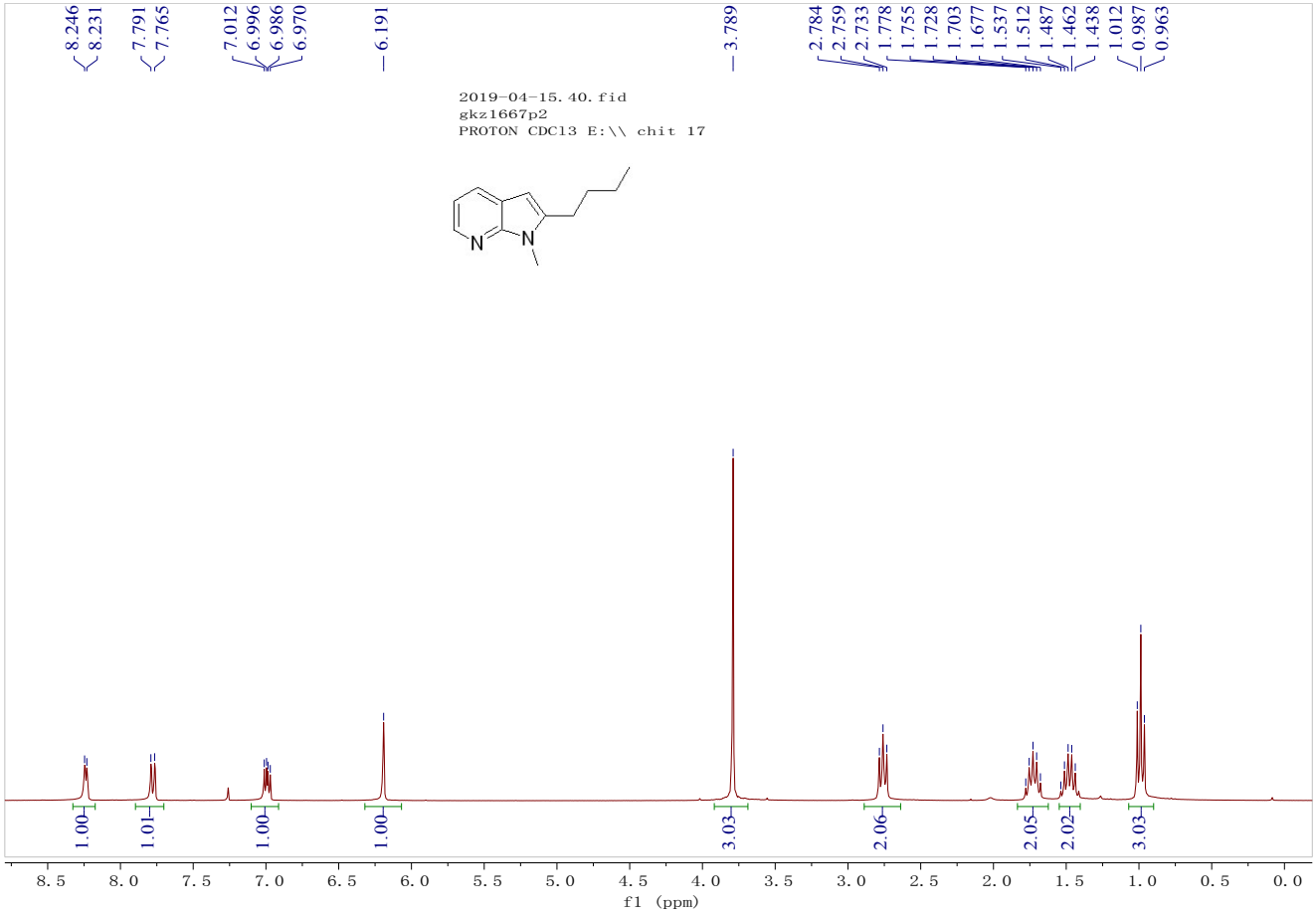
f1 (ppm)


7.586
7.560
7.317
7.290
7.232
7.209
7.182
7.136
7.112
7.087
6.354
3.961
3.939
3.918
3.699
3.060
3.039
3.018
1.806
2019-03-08.140.fid
gkz1580p2p1
PROTON CDCl3 E:\\ chit 52
OH
N
1.08
1.07
1.03
1.02
1.00
2.05
3.03
2.05
1.00
f1 (ppm)


137.668
137.200
127.882
121.109
120.007
119.600
109.065
100.024
61.452
30.225
29.670
2019-03-08.141.fid
gkz1580p2p1
JMOD CDCl3 E:\\ chit 52
OH
N
f1 (ppm)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8.231
7.791
7.765
7.012
6.996
6.986
6.970
6.191
3.789
2.784
2.759
2.733
1.778
1.755
1.728
1.703
1.677
1.537
1.512
1.487
1.462
1.438
1.012
0.987
0.963
2019-04-15.40.fid
gkz1667p2
PROTON CDCl3 E:\\ chit 17
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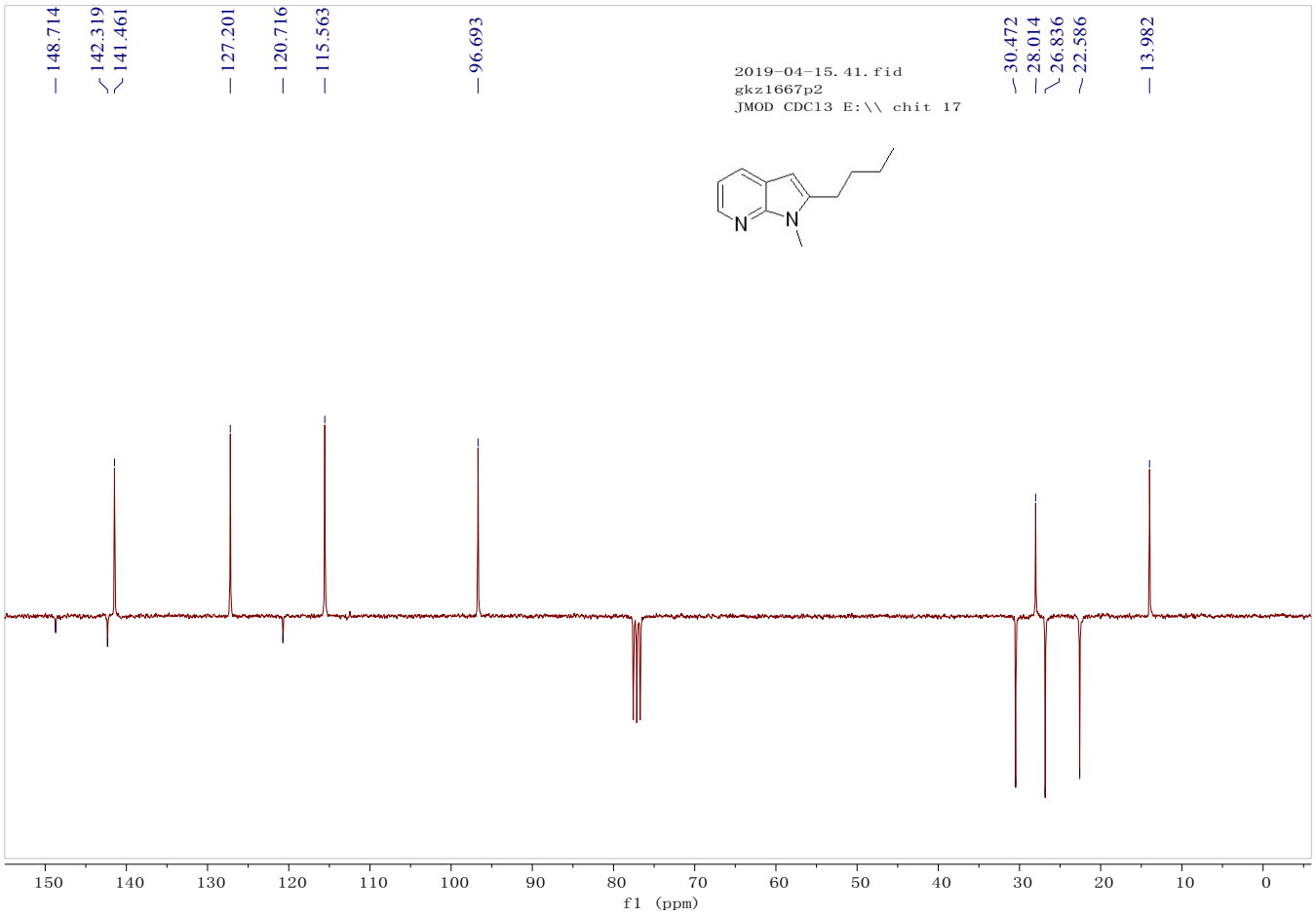
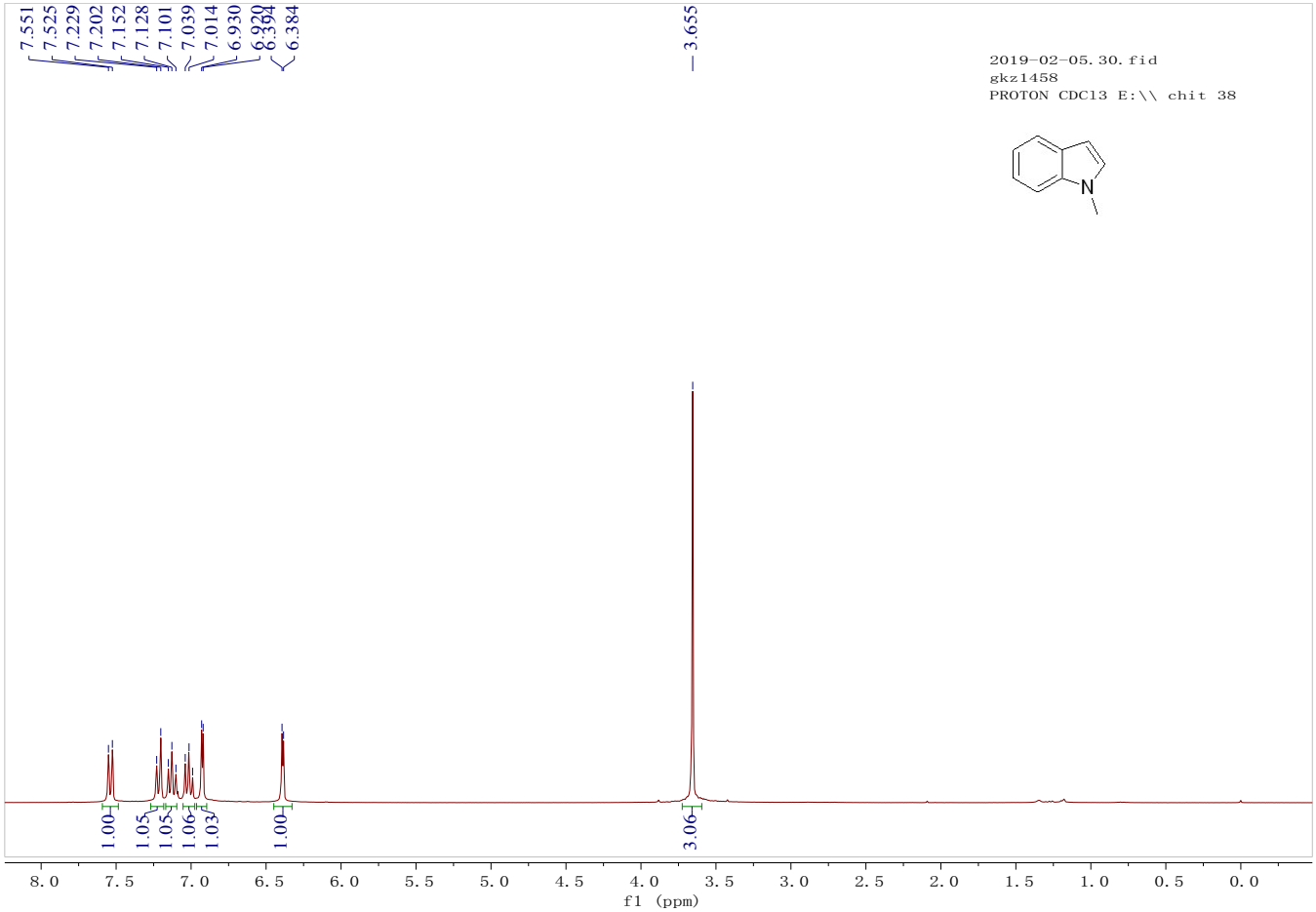
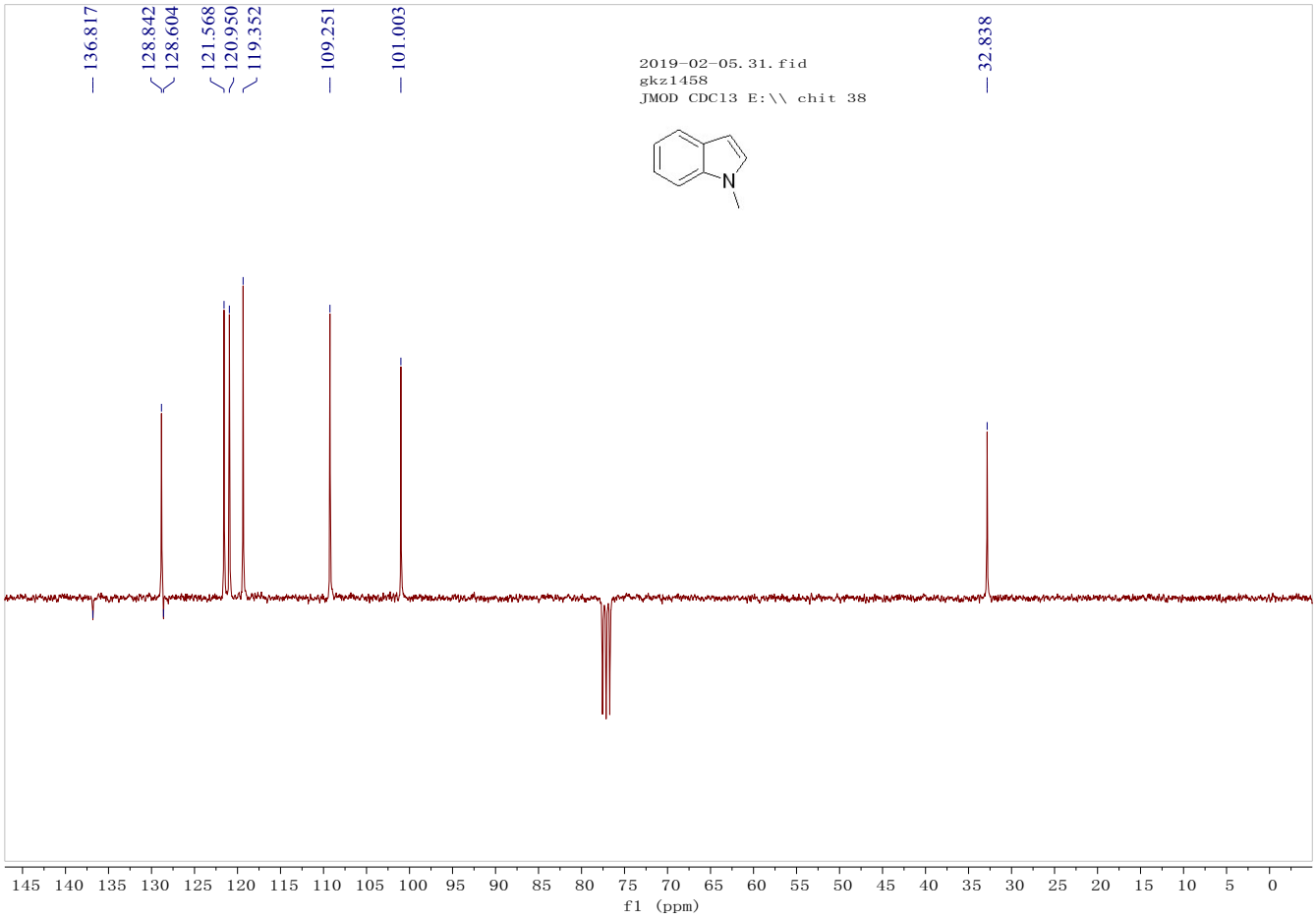
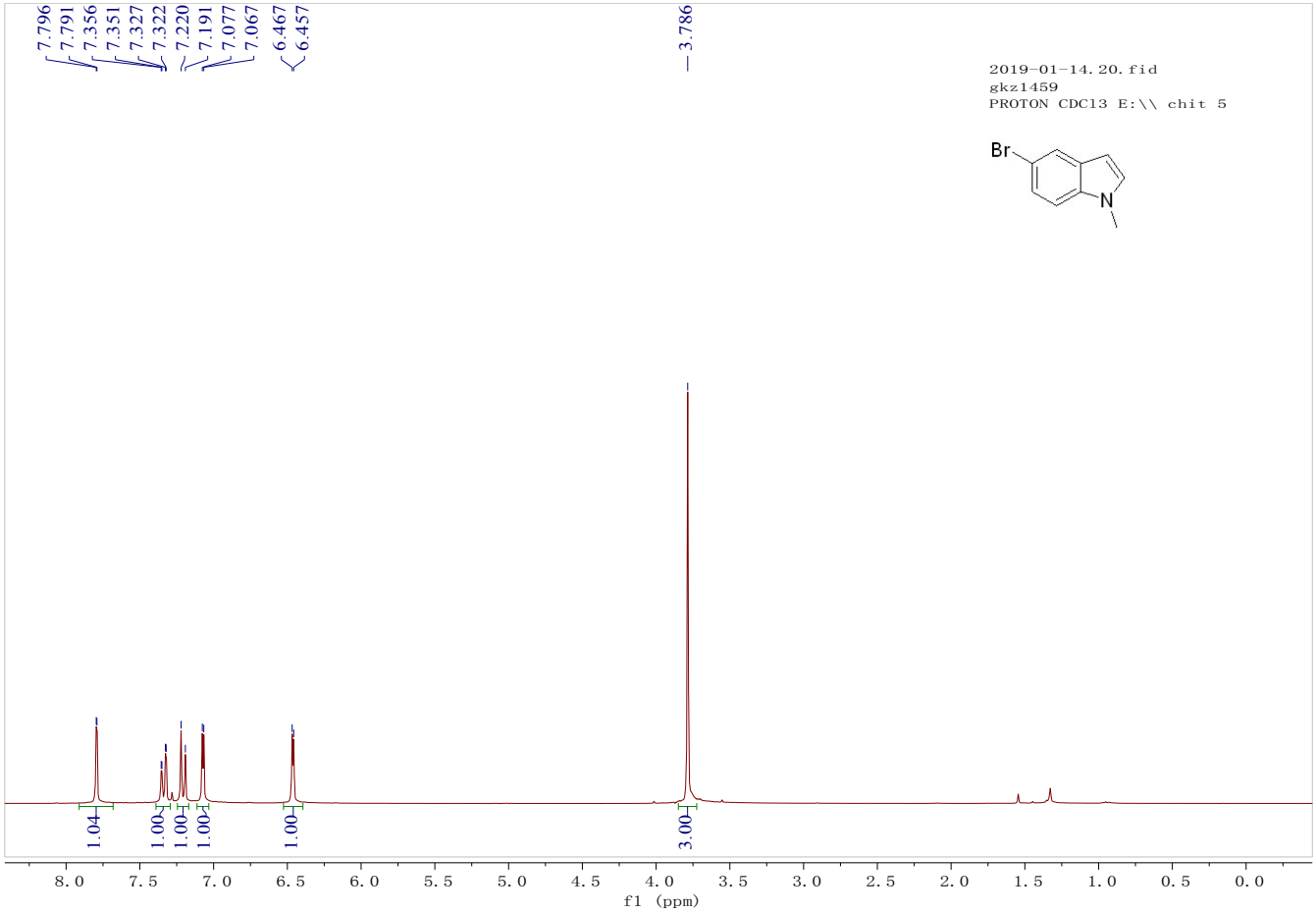
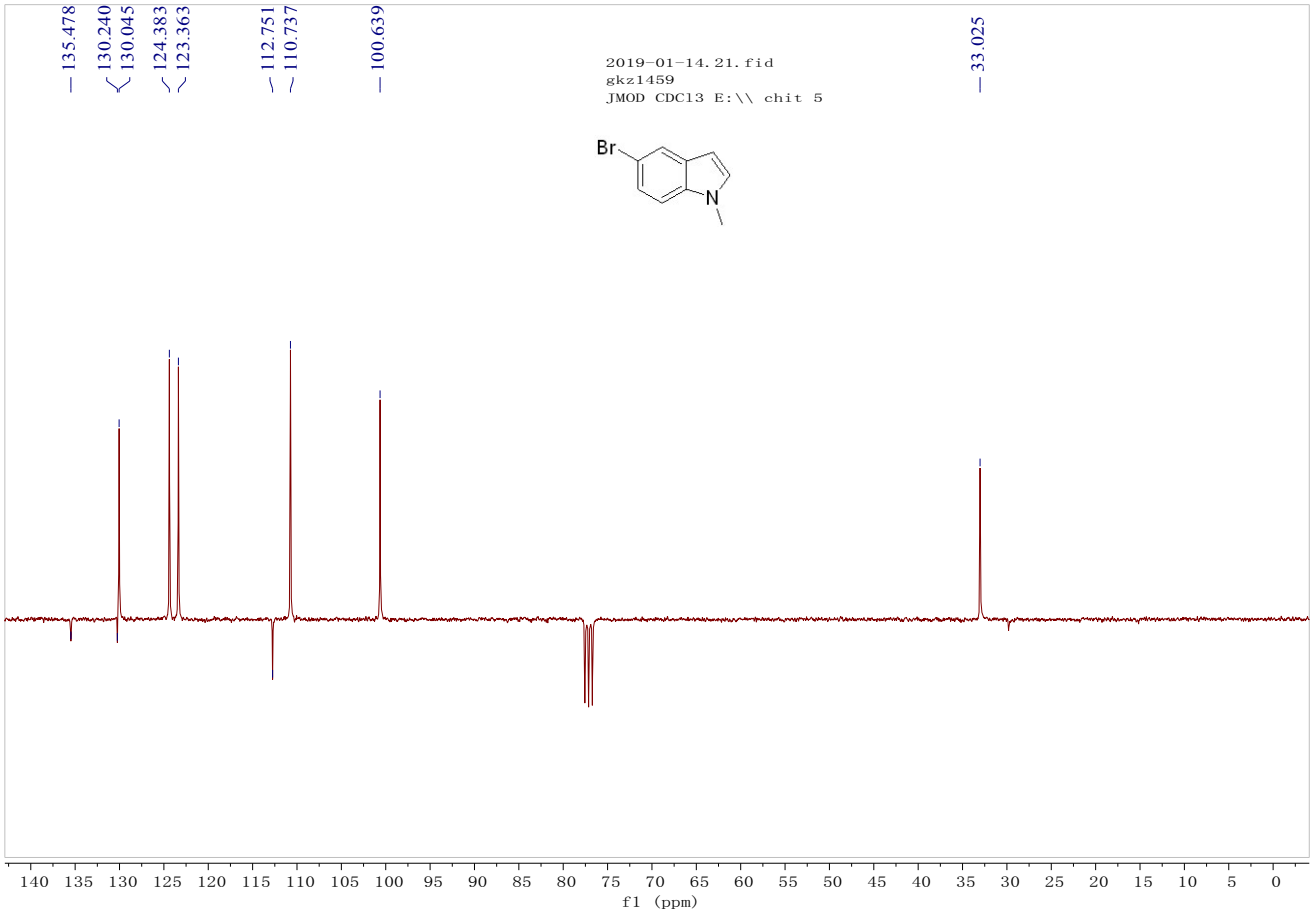
1.01
1.00
1.00
3.03
2.06
2.05
2.02
3.03
f1 (ppm)

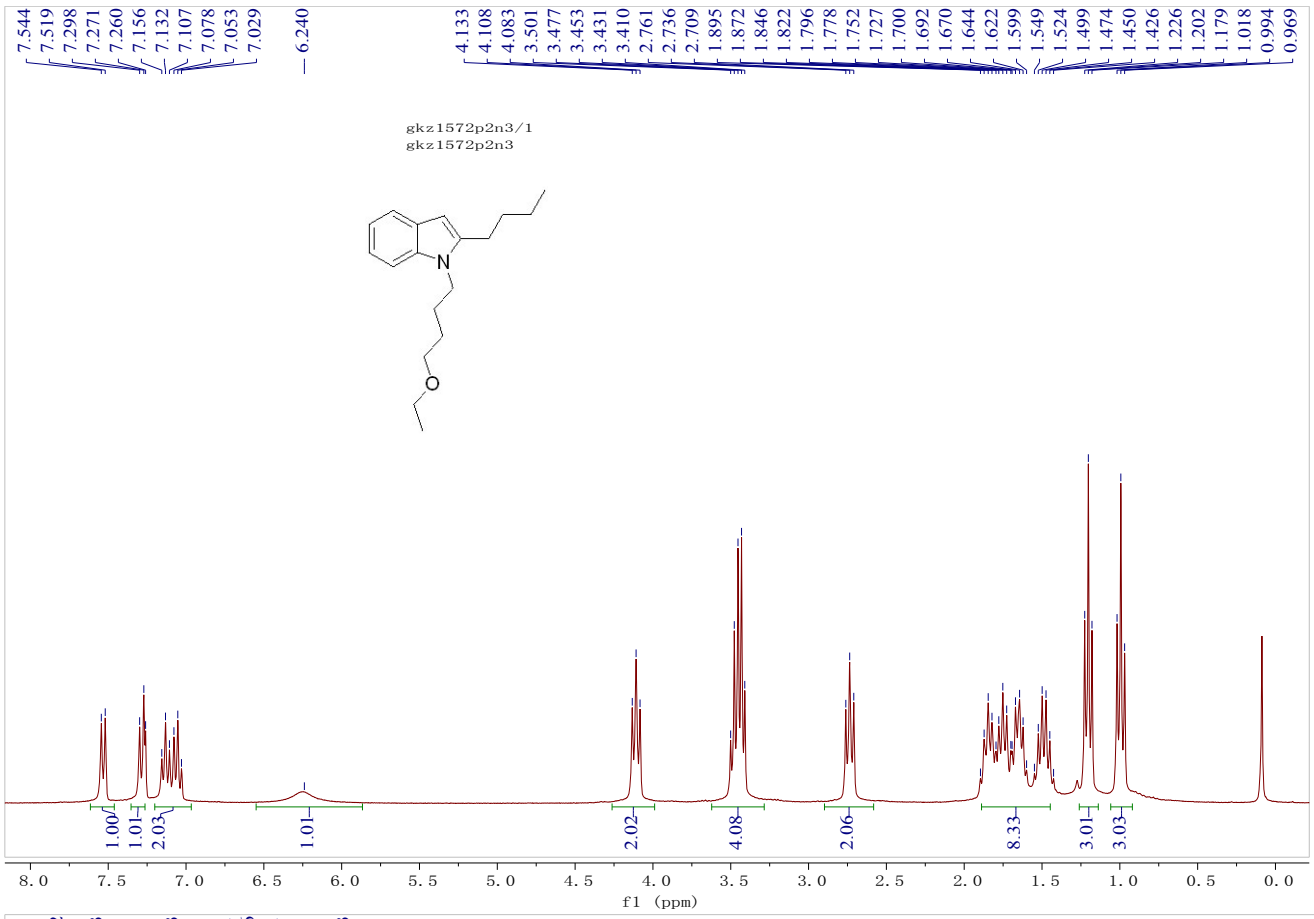

148.714
142.319
141.461
127.201
120.716
115.563
96.693
30.472
28.014
26.836
22.586
13.982
2019-04-15.41.fid
gkz1667p2
JMOD CDCl3 E:\\ chit 17
f1 (ppm)

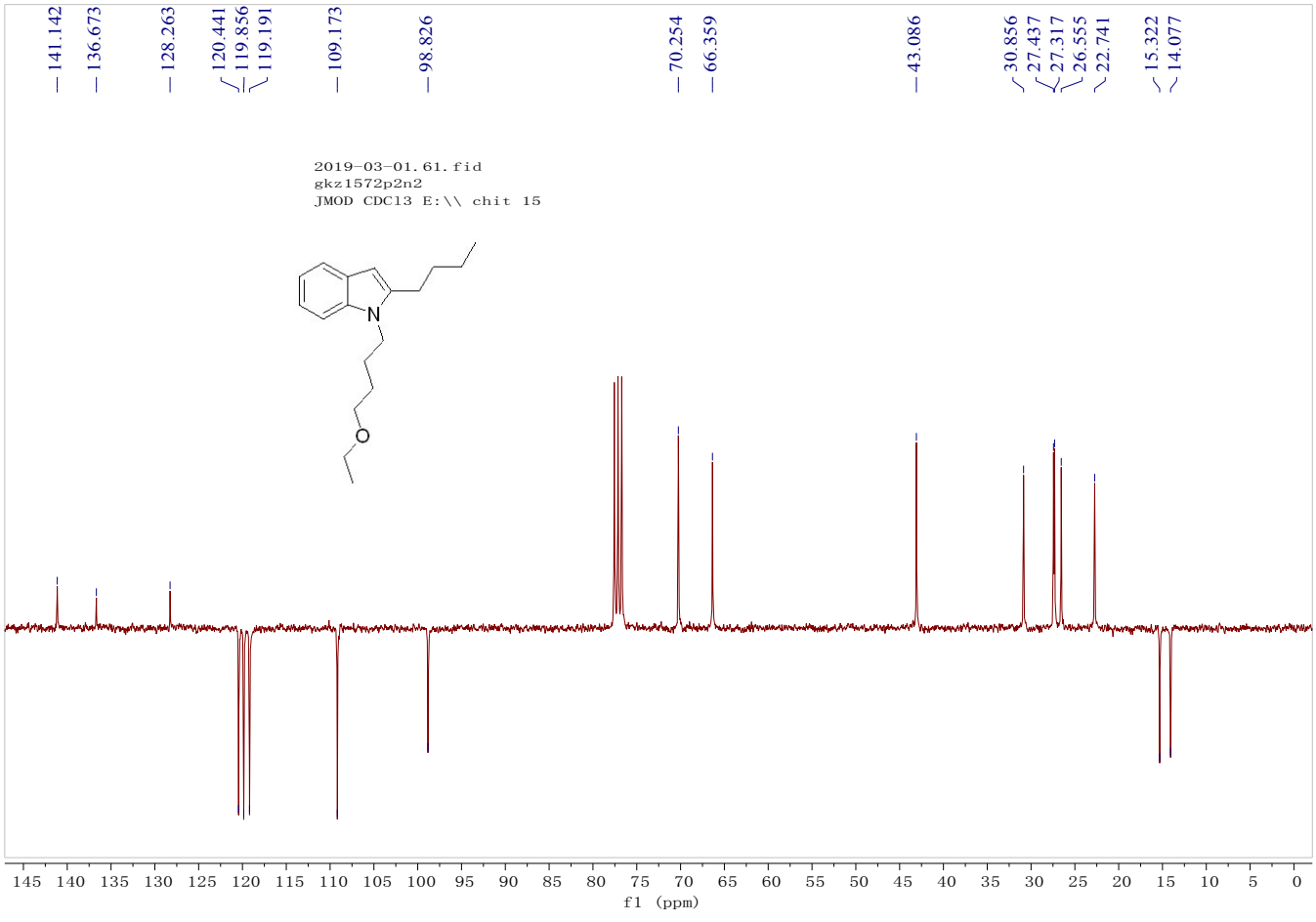

7.551
7.525
7.229
7.202
7.152
7.128
7.101
7.039
7.014
6.930
6.920
6.904
6.384
3.655
2019-02-05.30.fid
gkz1458
PROTON CDCl3 E:\\ chit 38
N
1.00
1.05
1.05
1.06
1.03
1.00
3.06
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

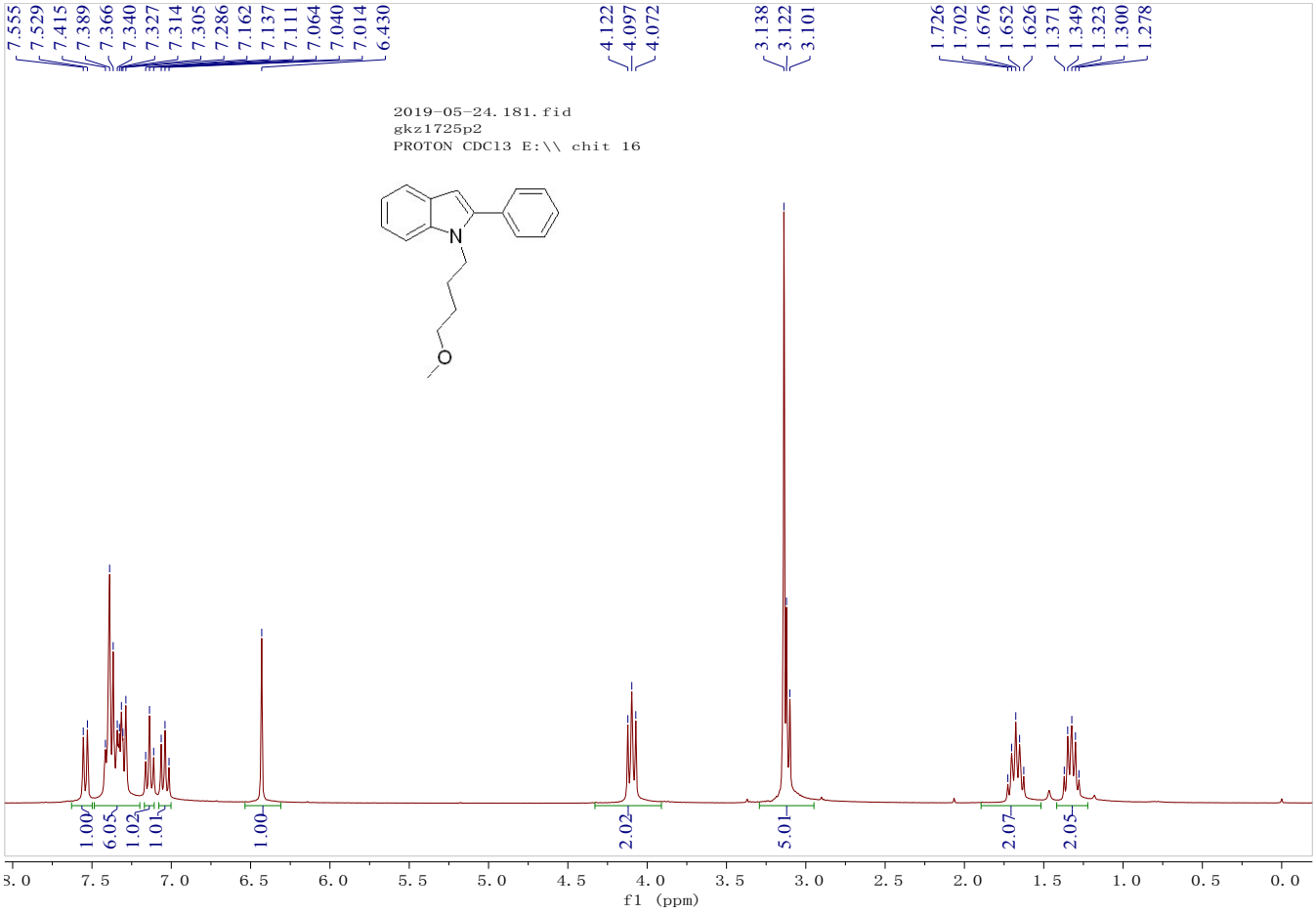

136.817
128.842
128.604
121.568
120.950
119.352
109.251
101.003
32.838
2019-02-05.31.fid
gkz1458
JMOD CDCl3 E:\\ chit 38
N
145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

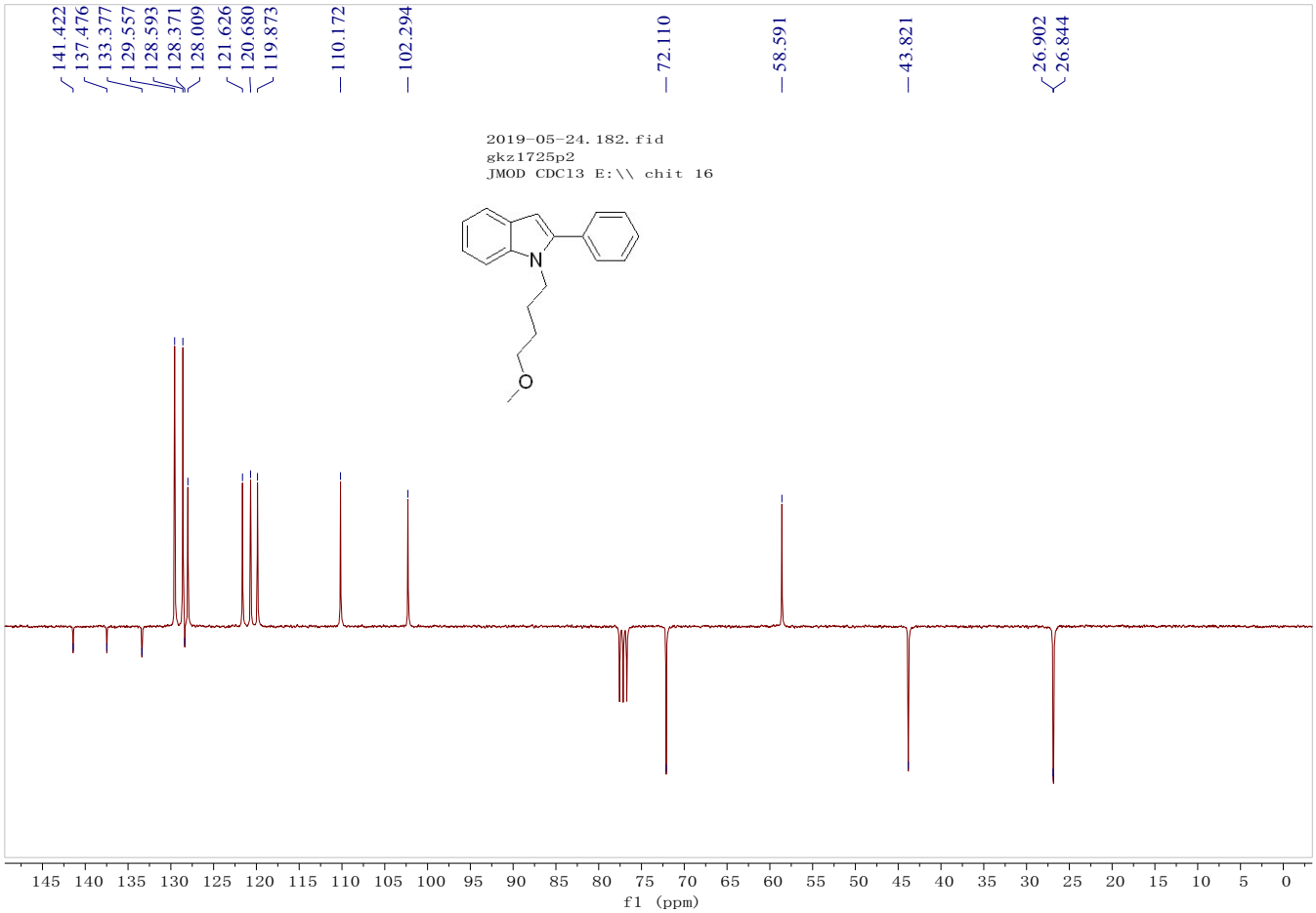

7.796
7.791
7.356
7.351
7.327
7.322
7.220
7.191
7.077
7.067
6.467
6.457
3.786
2019-01-14.20.fid
gkz1459
PROTON CDCl3 E:\\ chit 5
Br
N
1.04
1.00
1.00
1.00
1.00
3.00
f1 (ppm)

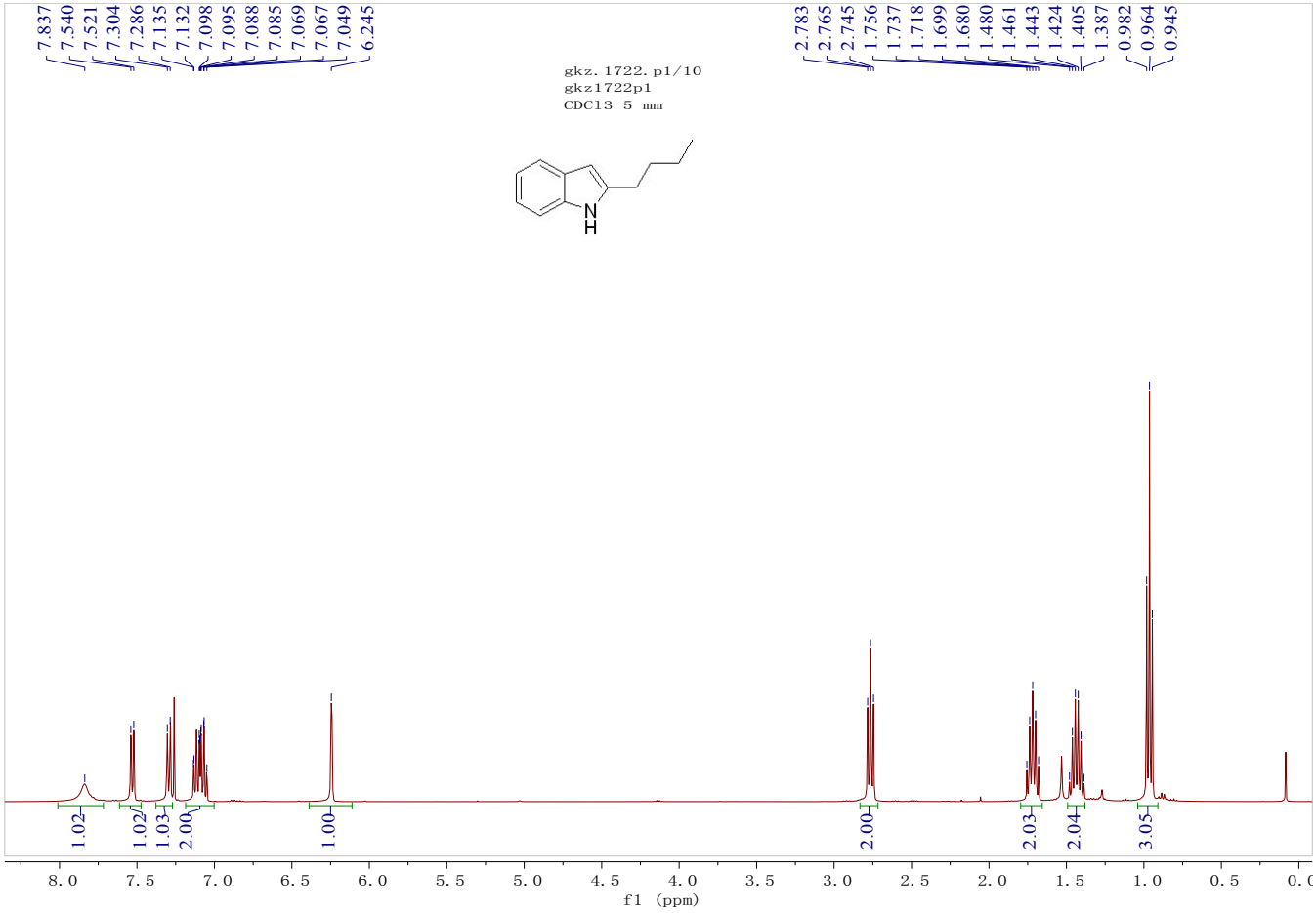

135.478
130.240
130.045
124.383
123.363
112.751
110.737
100.639
33.025
2019-01-14.21.fid
gkz1459
JMOD CDCl3 E:\\ chit 5
Br
N
f1 (ppm)

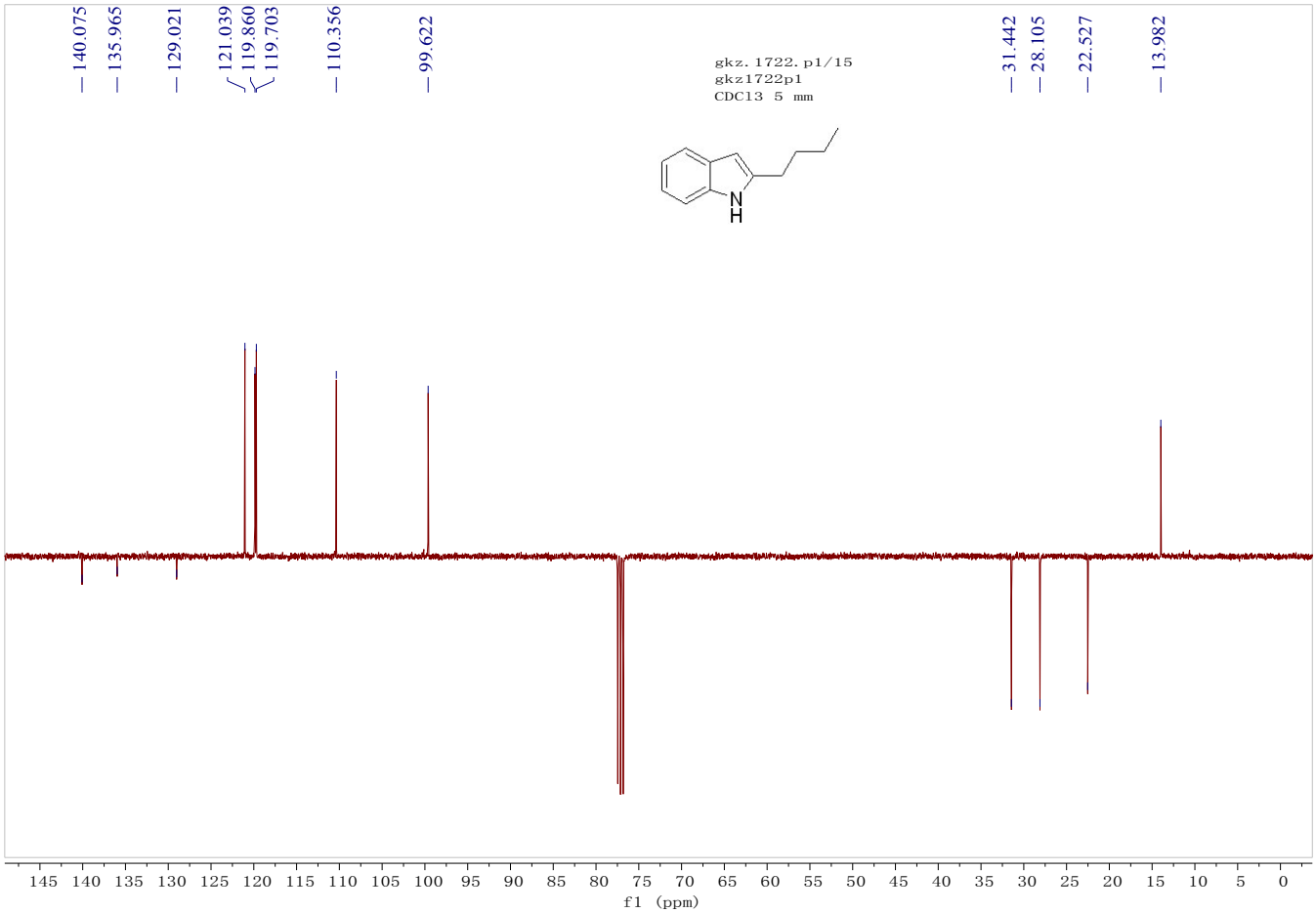

7.544
7.519
7.298
7.271
7.260
7.156
7.132
7.107
7.078
7.053
7.029
6.240
4.133
4.108
4.083
3.501
3.477
3.453
3.431
3.410
2.761
2.736
2.709
1.895
1.872
1.846
1.822
1.796
1.778
1.752
1.727
1.700
1.692
1.670
1.644
1.622
1.599
1.549
1.524
1.499
1.474
1.450
1.426
1.226
1.202
1.179
1.018
0.994
0.969
gkz1572p2n3/1
gkz1572p2n3
N
O
1.00
1.01
2.03
1.01
2.02
4.08
2.06
8.33
3.01
3.03
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

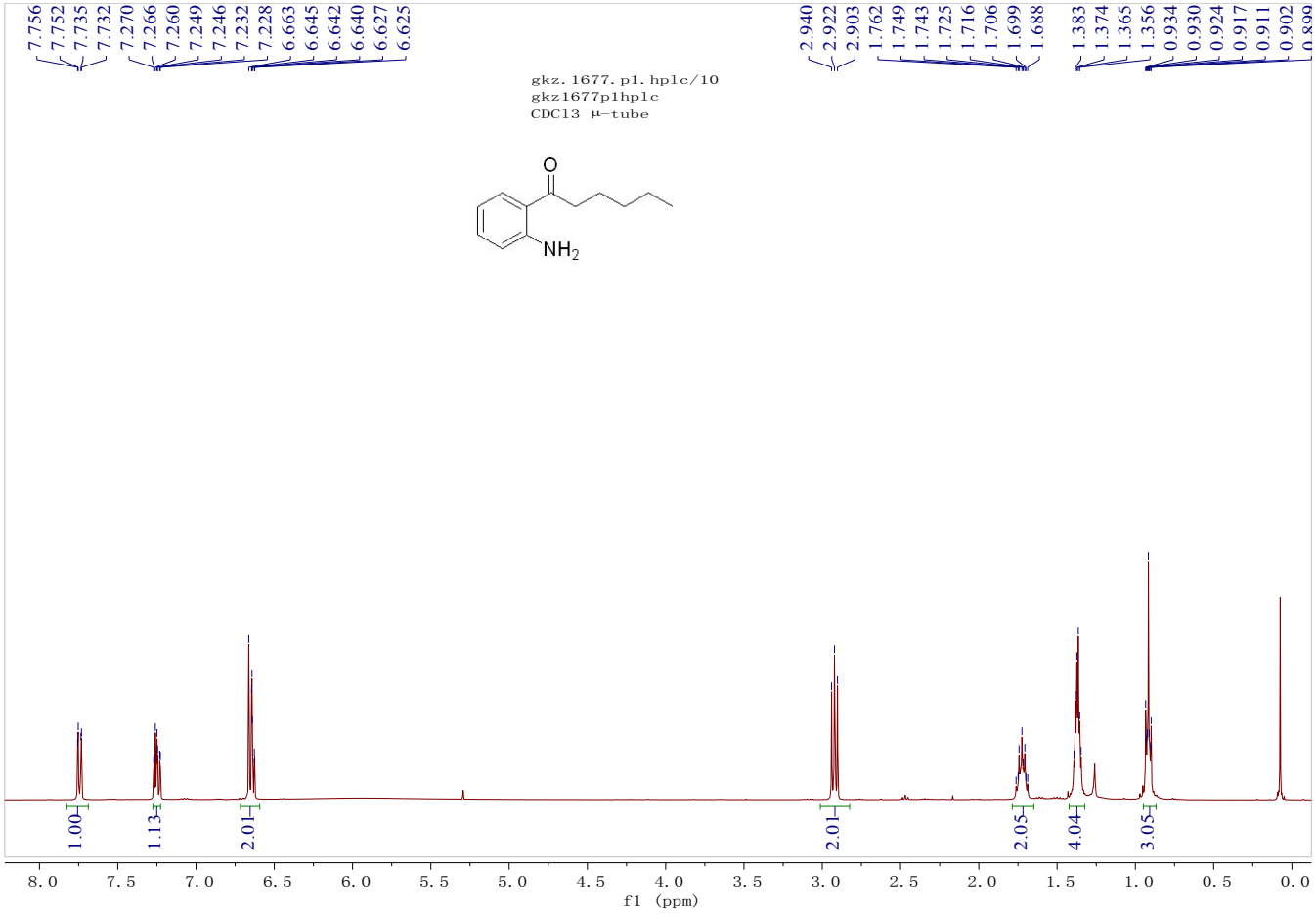

141.142
136.673
128.263
120.441
119.856
119.191
109.173
98.826
70.254
66.359
43.086
30.856
27.437
27.317
26.555
22.741
15.322
14.077
2019-03-01.61.fid
gkz1572p2n2
JMOD CDCl3 E:\\ chit 15
N
O
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
f1 (ppm)

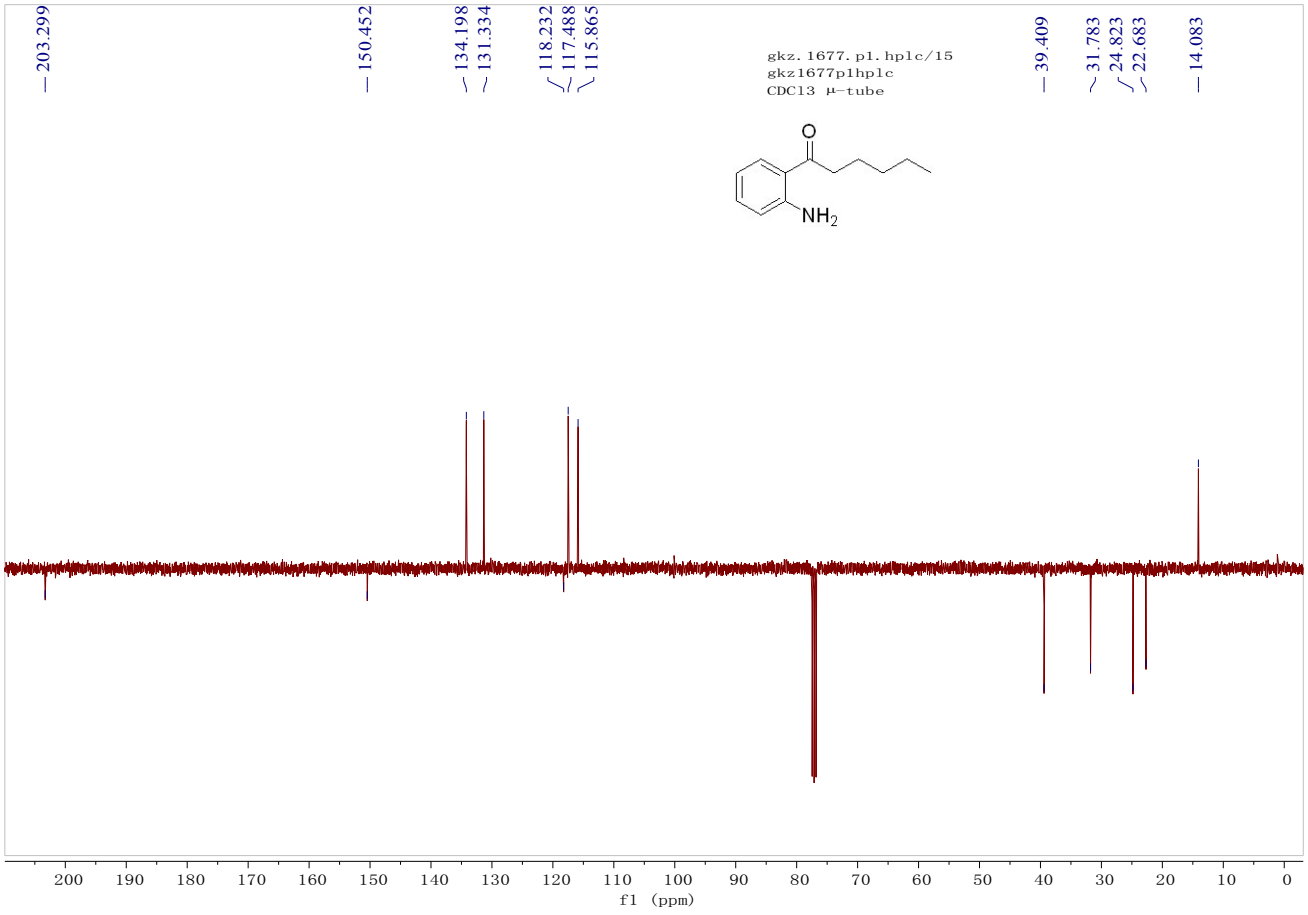

7.555
7.529
7.415
7.389
7.366
7.340
7.327
7.314
7.305
7.286
7.162
7.137
7.111
7.064
7.040
7.014
6.430
4.122
4.097
4.072
3.138
3.122
3.101
1.726
1.702
1.676
1.652
1.626
1.371
1.349
1.323
1.300
1.278
2019-05-24.181.fid
gkz1725p2
PROTON CDCl3 E:\\ chit 16
N
O
1.00
6.05
1.02
1.01
1.00
2.02
5.01
2.07
2.05
f1 (ppm)


141.422
137.476
133.377
129.557
128.593
128.371
128.009
121.626
120.680
119.873
110.172
102.294
72.110
58.591
43.821
26.902
26.844
2019-05-24.182.fid
gkz1725p2
JMOD CDCl3 E:\\ chit 16
N
O
f1 (ppm)


7.837
7.540
7.521
7.304
7.286
7.135
7.132
7.098
7.095
7.088
7.085
7.069
7.067
7.049
6.245
2.783
2.765
2.745
1.756
1.737
1.718
1.699
1.680
1.480
1.461
1.443
1.424
1.405
1.387
0.982
0.964
0.945
gkz. 1722. p1/10
gkz1722p1
CDC13 5 mm
N
H
1.02
1.02
1.03
2.00
1.00
2.00
2.03
2.04
3.05
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


140.075
135.965
129.021
121.039
119.860
119.703
110.356
99.622
31.442
28.105
22.527
13.982
gkz. 1722. p1/15
gkz1722p1
CDC13 5 mm
N
H
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
f1 (ppm)


7.756
7.752
7.735
7.732
7.270
7.266
7.260
7.249
7.246
7.232
7.228
6.663
6.645
6.642
6.640
6.627
6.625
2.940
2.922
2.903
1.762
1.749
1.743
1.725
1.716
1.706
1.699
1.688
1.383
1.374
1.365
1.356
0.934
0.930
0.924
0.917
0.911
0.902
0.899
gkz. 1677. p1. hplc/10
gkz1677p1hplc
CDCl3 μ-tube
O
NH2
1.00
1.13
2.01
2.01
2.05
4.04
3.05
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


203.299
150.452
134.198
131.334
118.232
117.488
115.865
39.409
31.783
24.823
22.683
14.083
gkz. 1677. p1. hplc/15
gkz1677p1hplc
CDCl3 μ-tube
O
NH2
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

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