Supporting Information

For

Metal-free annulation/aerobic oxidative dehydrogenation of cyclohexanones with *o*-acylanilines: Efficient Syntheses of acridines

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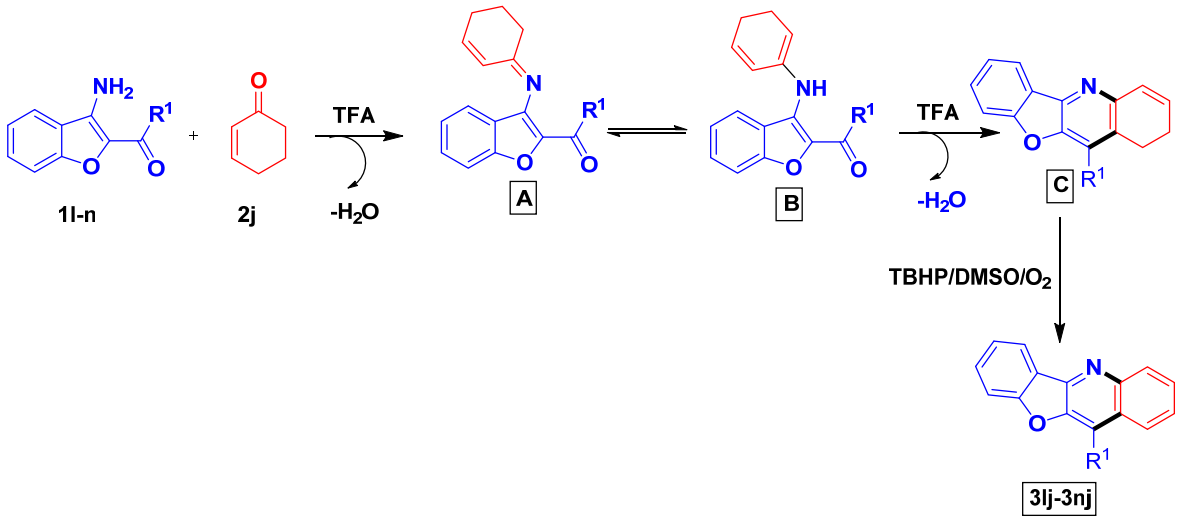
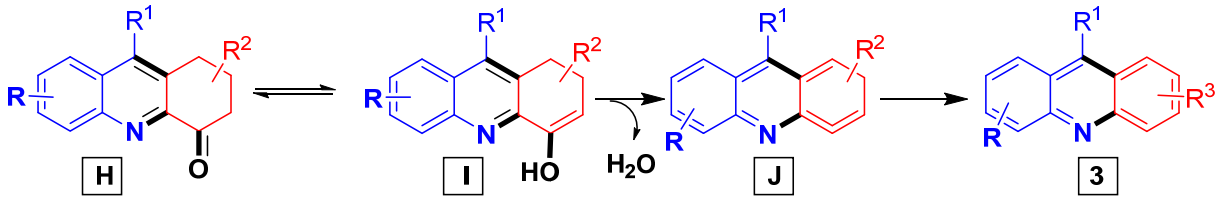
General Information

All commercial reagent and solvents were used as purchased without further purifications. ^{1}H, ^{13}C and DEPT NMR spectra were recorded on a 400 MHz Varian Unity Plus or Varian Mercury plus spectrometer or JEOL 400 MHz. The chemical shift (δ) values are reported in parts per million (ppm), and the coupling constants (J) are given in Hz. The spectra were recorded using $CDCl_3$ as a solvent. ^{1}H NMR chemical shifts are referenced to tetramethylsilane (TMS) (0 ppm). ^{13}C NMR was referenced to $CDCl_3$ (77.0 ppm). The abbreviations used are as follows: s, singlet; d, doublet; t, triplet; q, quartet; dd, doublet of doublet; ddd, doublet of doublet of doublet; dq, doublet of quartet; dt, doublet of triplet; m, multiplet. Mass spectra and high-resolution mass spectra (HRMS) was measured using the ESI (FT-MS solariX) at National Sun Yat-Sen University, Kaohsiung, Taiwan or LTQ Orbitrap XL (Thermo Fischer Scientific) at National Chung Hsing University. All IR (neat) νmax spectra were obtained as neat films with a Perkin-Elmer Model 2000 FT-IR $(neat)_{\nu max}$ SYSTEM and selected absorbance are reported in cm^{-1}. Melting points were determined on an EZ-Melt (Automated melting point apparatus). All products reported showed ^{1}H NMR spectra in agreement with the assigned structures. Reaction progress and product mixtures were routinely monitored by TLC using Merck TLC aluminum sheets (silica gel 60 F254). Column chromatography was carried out with 230–400 mesh silica gel 60 (Merck)/neutral alumina and a mixture of hexane/ethyl acetate or hexane as an eluent. The starting materials **1l-n** was prepared according to the reported literature methods.[1a]

Scheme S1. Mechanism for the reaction with Cyclohexenone

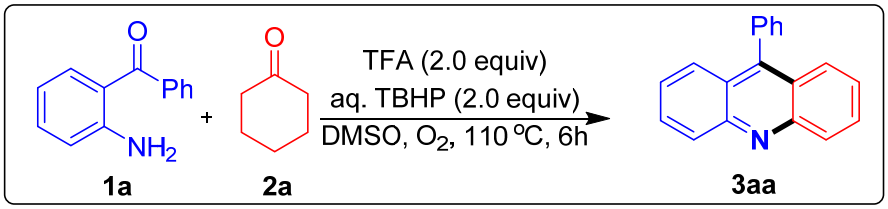


Scheme S2. Alternative mechanism for formation of 3 from intermediate I[1b,c]



General Procedure (A) for the Preparation of Acridines

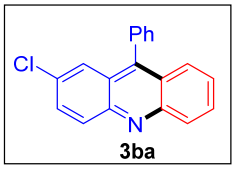
Preparation of 9-Phenylacridine (3aa)[2]



A 15 mL oven dried sealed tube was charged with 2-aminobenzophenone **1a** (197 mg, 1.0 mmol), DMSO (0.5 mL), cyclohexanone **2a** (156 µL, 1.5 mmol), trifluoroacetic acid (153 µL,

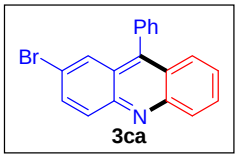
2.0 mmol) and 70% aq. TBHP solution (257 µL, 2.0 mmol). The resulting solution was sealed under O_2 and stirred at 110 ˚C for 6 h. After cooling to room temperature, the reaction mixture was diluted with 5 mL of water and neutralized with 10% aqueous $NaHCO_3$ solution. The aqueous layer was extracted with (3X10 mL) of ethyl acetate and the combined organic layer was given brine wash (2X10 mL). The final organic layer was evaporated in a rotary evaporator to remove the volatile components. The residue was purified by column chromatography on silica gel (3% ethyl acetate/ hexane to 6% ethyl acetate/hexane) to afford pure 9-phenylacridine **3aa** as a pale yellow solid (234 mg, 92%); m.p. 181-182 ˚C; IR (neat)$_{\nu max}$: 1362 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (d, J = 8.8 Hz, 2H), 7.76 (ddd, J = 8.0, 6.8, 1.6 Hz, 2H), 7.72 (d, J = 8.8 Hz, 2H), 7.66 – 7.56 (m, 3H), 7.49 – 7.41 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.66, 147.24, 135.85, 130.37, 129.96, 129.45, 128.40, 128.31, 126.81, 125.55, 125.09; HRMS (ESI) calcd for $C_{19}H_{14}N$ $[M + H]^+$: 256.1121; found: 256.1112.

2-Chloro-9-phenylacridine (3ba).[2] Following the general procedure (A) for 6 h on a 1.0 mmol scale, giving the compound as a beige color solid (205 mg, 71%); m.p. 149-151 ˚C; IR (neat)$_{\nu max}$ 1345, 738 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$,) δ 8.25 (m, 1H), 8.21 (dd, J = 9.2, 0.8 Hz, 1H), 7.78 (ddd, J = 8.0, 6.4, 1.2 Hz, 1H), 7.70 – 7.66 (m, 3H), 7.65 – 7.60 (m, 3H), 7.47 – 7.41 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.34, 148.81, 146.99, 146.40, 135.25, 131.47, 131.34, 131.17, 130.32, 130.24, 129.64, 128.63, 126.78, 126.23, 125.43, 125.38, 125.05; HRMS (ESI) calcd for $C_{19}H_{13}ClN$ $[M + H]^+$: 290.0731; found: 290.0724.

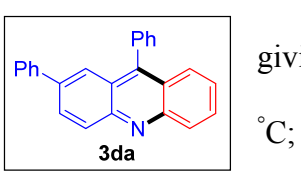


3ba

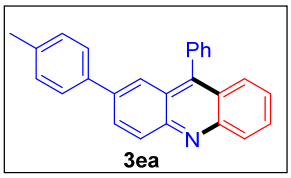
2-Bromo-9-phenylacridine (3ca). Following the general procedure (A) for 6 h on a 1.0 mmol scale, giving the compound as a pale yellow solid (266 mg, 80%); m.p. 137-138 °C; IR (neat)$_{\nu max}$, 1355, 552 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (d, J = 8.8 Hz, 1H), 8.12 (d, J = 9.2 Hz, 1H), 7.83 (d, J = 1.8 Hz, 1H), 7.80 – 7.75 (m, 2H), 7.66 (dd, J = 8.8, 0.6 Hz, 1H), 7.63 – 7.59 (m, 3H), 7.44 – 7.39 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.38, 148.90, 147.11, 146.35, 135.24, 133.51, 131.39, 130.34, 130.31, 129.68, 128.65, 128.55, 126.83, 126.27, 126.00, 125.37, 119.86; HRMS (ESI) calcd for $C_{19}H_{13}BrN$ $[M + H]^+$: 334.0226; found: 334.0220.



2,9-Diphenylacridine (3da). Following the general procedure (A) for 24 h on a 1.0 mmol scale, giving the compound as a pale yellow solid (287 mg, 87%); m.p. 158-160 °C; IR (neat)$_{\nu max}$ 1374 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 (d, J = 9.0 Hz, 1H), 8.29 (d, J = 8.7 Hz, 1H), 8.06 (dd, J = 9.0, 2.0 Hz, 1H), 7.88 (d, J = 2.0 Hz, 1H), 7.76 (ddd, J = 8.0, 6.4, 1.2 Hz, 1H), 7.70 (d, J = 8.5 Hz, 1H), 7.62 – 7.57 (m, 5H), 7.49 – 7.46 (m, 2H), 7.44-7.40 (m, 3H), 7.36 – 7.32 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.74, 148.20, 147.26, 140.42, 138.01, 135.84, 130.43, 130.11, 130.08, 129.87, 129.59, 128.84, 128.49, 128.38, 127.59, 127.33, 126.79, 125.68, 125.43, 125.18, 124.05; HRMS (ESI) calcd for $C_{25}H_{18}N$ $[M + H]^+$: 332.1433; found: 332.1433.

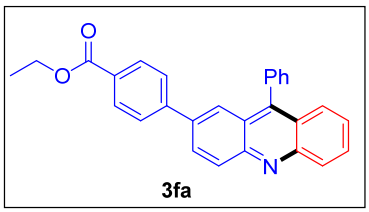


11-(*p*-Tolyl)benzofuro[3,2-*b*]quinolone (3ea). Following the general procedure (A) for 12 h on a 1.0 mmol scale, giving the compound as a brown solid (200 mg, 58%); m.p. 191-193 °C; IR (neat)$_{\nu max}$ 2854, 1375, 1184 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.34 (d, J = 9.1 Hz, 1H), 8.28 (d, J = 8.7 Hz, 1H), 8.06 (dd, J = 9.1, 2.1 Hz, 1H), 7.85 (d, J = 1.8 Hz, 1H), 7.77 (ddd, J = 8.2, 6.3, 1.1 Hz, 1H), 7.70 (d, J = 8.8 Hz, 1H), 7.64-7.58 (m, 3H), 7.51 – 7.47 (m, 4H), 7.43 (ddd, J = 8.0, 6.8, 1.2 Hz, 1H), 7.26 – 7.22 (m, 2H), 2.39 (s,



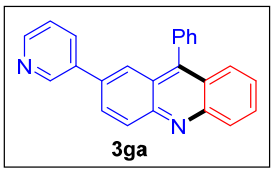
3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.58, 138.88, 130.78, 130.48, 129.47, 127.73, 125.94, 125.71, 123.52, 122.29, 112.32, 21.48; HRMS (ESI) calcd for $C_{26}H_{20}N$ $[M + H]^+$: 346.1590; found: 346.1585.

Ethyl 9-phenylacridine-2-carboxylate (3fa). Following the general procedure (A) for 10 h on a 1.0 mmol scale, giving the compound as a pale yellow solid (241 mg, 60%); m.p. 170-173 °C; IR (neat)$_{\nu max}$ 2928, 1715, 1362 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 (d, J = 9.2 Hz, 1H), 8.31 (d, J = 8.8 Hz, 1H), 8.11 (d, J = 8.4 Hz, 2H), 8.08 (dd, J = 8.8, 2.4 Hz, 1H), 7.93 (d, J = 2.0 Hz, 1H), 7.80 (ddd, J = 8.8, 6.4, 1.2 Hz, 1H), 7.72 (d, J = 8.0 Hz, 1H), 7.68 – 7.61 (m, 5H), 7.50 (dd, J = 8.0, 1.2 Hz, 2H), 7.46 (ddd, J = 7.6, 6.4, 1.2 Hz, 1H), 4.38 (q, J = 7.1 Hz, 2H), 1.38 (t, J = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.34, 149.02, 148.32, 147.70, 144.70, 136.89, 135.66, 130.44, 130.38, 130.22, 130.13, 129.65, 129.63, 129.54, 128.58, 127.20, 126.89, 125.90, 125.51, 125.10, 124.88, 61.02, 14.33; HRMS (ESI) calcd for $C_{28}H_{22}NO_2$ $[M + H]^+$: 404.1645; found:404.1652.



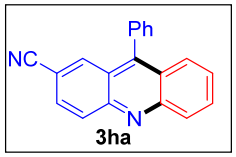
3fa

9-Phenyl-2-(pyridin-3-yl)acridine (3ga). Following the general procedure (A) for 8 h on a 1.0 mmol scale, giving the compound as a pale yellow solid (186 mg, 56%); m.p. 142-144 °C; IR (neat)$_{\nu max}$ 1565, 1367 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.86 (s, 1H), 8.59 (s, 1H), 8.40 (d, J = 9.0 Hz, 1H), 8.30 (d, J = 8.8 Hz, 1H), 8.02 (dd, J = 9.0, 2.1 Hz, 1H), 7.90 (td, J = 4.8, 2.0 Hz, 2H), 7.80 (ddd, J = 8.8, 6.5, 1.4 Hz, 1H), 7.72 (d, J = 8.4 Hz, 1H), 7.66 – 7.60 (m, 3H), 7.50 – 7.44 (m, 3H), 7.37 (dd, J = 7.9, 4.8 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.04, 148.98, 148.69, 148.47, 148.18, 148.15, 147.65, 135.55, 134.64, 134.48, 130.66, 130.36, 130.27, 129.62, 129.32, 128.62, 126.89, 125.96, 125.50, 125.10, 124.72, 123.60; HRMS (ESI) calcd for $C_{24}H_{17}N_2$ $[M + H]^+$: 333.1386; found: 333.1379.

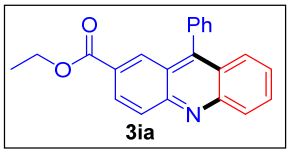


3ga

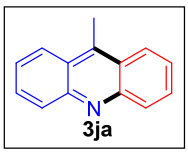
9-Phenylacridine-2-carbonitrile (3ha). Following the general procedure (A) for 8 h on a 1.0 mmol scale, giving the compound as a pale yellow solid (146 mg, 52%); m.p. 235-237 °C; IR (neat)$_{\nu max}$ 2220, 1338 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (dd, J = 9.0, 0.5 Hz, 1H), 8.28 (d, J = 8.4 Hz, 1H), 8.15 (d, J = 1.7 Hz, 1H), 7.86 (ddd, J = 8.8, 6.6, 1.4 Hz, 1H), 7.81 (dd, 8.8, 1.6 Hz, 1H), 7.75 (d, J = 8.8 Hz, 1H), 7.66 – 7.64 (m, 3H), 7.50 (ddd, J = 8.7, 6.5, 1.2 Hz, 1H), 7.44 – 7.41 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.36, 148.90, 148.40, 134.64, 134.36, 131.69, 131.24, 130.25, 129.76, 129.11, 129.00, 128.78, 127.16, 126.79, 125.58, 124.00, 118.90, 109.08; HRMS (ESI) calcd for $C_{20}H_{13}N_2$ [M + H]$^+$: 281.1073; found: 281.1068.



Ethyl 4-(9-phenylacridin-2-yl)benzoate (3ia).[3] Following the general procedure (A) for 8 h on a 1.0 mmol scale, giving the compound as a pale yellow solid (215 mg, 66%); m.p. 134-136 °C; IR (neat)$_{\nu max}$ 2913, 1715, 1362 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (dd, J = 1.6, 0.8, 1H), 8.31 (d, J = 8.7 Hz, 1H), 8.30 (d, J = 1.6 Hz, 1H), 8.28 (d, J = 8.0 Hz, 1H), 7.82 (ddd, J = 8.0, 6.8, 1.6 Hz, 1H), 7.74 (d, J = 8.7 Hz, 1H), 7.64 – 7.60 (m, 3H), 7.47 – 7.43 (m, 3H), 4.38 (q, J = 7.1 Hz, 2H), 1.37 (t, J = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.23, 149.98, 149.37, 149.64, 135.07, 131.09, 130.88, 130.46, 129.86, 129.65, 128.77, 128.56, 125.43, 124.09, 61.04, 29.68, 14.35; HRMS (ESI) calcd for $C_{22}H_{18}NO_2$ [M + H]$^+$: 328.1332; found: 328.1329.

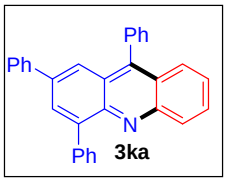


9-Methylacridine (3ja).[4] Following the general procedure (A) for 8 h on a 1.0 mmol scale, giving the compound as a pale yellow crystal (100 mg, 52%); m.p. 91-93 °C; IR (neat)$_{\nu max}$ 2850, 1368 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 – 8.19 (m, 4H),

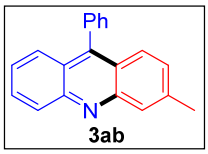


7.75 (ddd, J = 8.0, 6.8, 1.2 Hz, 2H), 7.54 (ddd, J = 8.4, 6.4, 1.2 Hz, 2H), 3.11 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.52, 130.32, 130.29, 129.83, 129.79, 125.63, 125.47, 124.59, 13.67; HRMS (ESI) calcd for $C_{14}H_{12}N$ $[M + H]^+$: 194.0964; found: 194.0961.

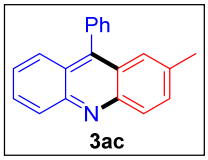
2,4,9-Triphenylacridine (3ka). Following the general procedure (A) for 24 h on a 1.0 mmol scale, giving the compound as a pale yellow solid (284 mg, 70%); m.p. 241-243 °C; IR (neat)$_{\nu max}$ 1368 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (dt, J = 8.8, 0.8 Hz, 1H), 8.08 (d, J = 2.1 Hz, 1H), 7.96 – 7.93 (m, 2H), 7.87 (d, J = 2.1 Hz, 1H), 7.70 – 7.67 (m, 1H), 7.65 – 7.57 (m, 7H), 7.57 – 7.53 (m, 2H), 7.50 – 7.45 (m, 3H), 7.42 – 7.34 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.45, 147.04, 146.19, 141.07, 140.58, 139.80, 137.63, 136.33, 131.15, 130.58, 130.54, 130.51, 129.32, 128.86, 128.52, 128.32, 127.88, 127.61, 127.41, 127.40, 126.56, 125.87, 125.66, 125.11, 123.73; HRMS (ESI) calcd for $C_{31}H_{22}N$ $[M + H]^+$: 408.1746; found: 408.1747.



3-Methyl-9-phenylacridine (3ab).[2] Following the general procedure (A) for 48 h on a 1.0 mmol scale, giving the compound as a pale yellow solid (244 mg, 91%); m.p. 119-120 °C; IR (neat)$_{\nu max}$ 2862, 1359 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (dt, J = 8.8, 0.8 Hz, 1H), 8.18 (d, J = 8.8 Hz, 1H), 7.71 (ddd, J = 8.0, 6.4, 1.6 Hz, 1H), 7.64 (d, J = 8.0 Hz, 1H), 7.62 – 7.58 (m, 4H), 7.44 – 7.37 (m, 4H), 2.44 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.19, 147.74, 145.93, 136.13, 135.39, 132.82, 130.39, 129.51, 129.40, 129.28, 128.39, 128.18, 127.67, 126.69, 125.75, 125.41, 124.70, 21.97; HRMS (ESI) calcd for $C_{20}H_{16}N$ $[M + H]^+$: 270.1277; found: 270.1265.

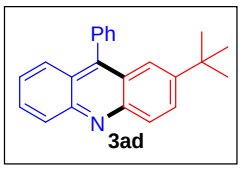


2-Methyl-9-phenylacridine (3ac).[4] Following the general procedure (A) for 48 h on a 1.0 mmol scale, giving the compound as a pale green solid (242 mg, 90%); m.p. 122-124 °C; IR (neat)$_{\nu max}$ 2844, 1368 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 (dt, J =

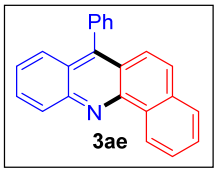


8.0, 0.8 Hz, 1H), 8.18 (d, J = 8.8 Hz, 1H), 7.73 (ddd, J = 8.0, 6.4, 1.6 Hz, 1H), 7.66, (ddd, J = 8.8, 1.4, 0.7 Hz, 1H), 7.63 – 7.59 (m, 4H), 7.45 – 7.38 (m, 4H), 2.46 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.20, 147.75, 145.98, 136.16, 135.42, 132.86, 130.42, 129.52, 129.44, 129.29, 128.42, 128.21, 126.72, 125.44, 125.28, 125.12, 124.72, 22.00; HRMS (ESI) calcd for $C_{20}H_{16}N$ $[M + H]^+$: 270.1277; found: 270.1269.

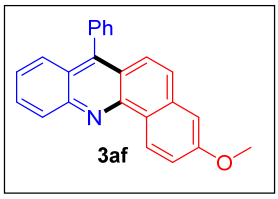
2-(*tert*-Butyl)-9-phenylacridine (3ad). Following the general procedure (A) for 48 h on a 1.0 mmol scale, giving the compound as an off-white solid (236 mg, 76%); m.p. 85-87 °C; IR (neat)$_{\nu max}$ 2916, 1338 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 (dt, J = 8.0, 0.8 Hz, 1H), 8.21 (dd, J = 9.2, 0.6 Hz, 1H), 7.88 (dd, J = 9.2, 2.2 Hz, 1H), 7.72 (ddd, J = 8.0, 6.4, 1.2 Hz, 1H), 7.68 (ddd, J = 8.8, 1.2, 0.8 Hz, 1H), 7.60 – 7.56 (m, 4H), 7.44 (dd, J = 8.0, 1.6 Hz, 1H), 7.39 (ddd, J = 8.0, 6.8, 1.6 Hz, 1H), 1.30 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.41, 148.02, 147.77, 146.68, 136.10, 130.41, 129.58, 129.51, 129.41, 129.10, 128.34, 128.22, 126.74, 125.31, 125.24, 124.77, 120.78, 35.06, 30.74; HRMS (ESI) calcd for $C_{23}H_{22}N$ $[M + H]^+$: 312.1747; found: 312.1736.



7-Phenylbenzo[*c*]acridine (3ae).[5] Following the general procedure (A) for 48 h on a 1.0 mmol scale, giving the compound as an off-white solid (136 mg, 45%); m.p. 123-124 °C; IR (neat)$_{\nu max}$ 1365 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.59 (dd, J = 8.0, 0.7 Hz, 1H), 8.42 (dd, J = 8.0, 0.8 Hz, 1H), 7.85 – 7.69 (m, 5H), 7.61 – 7.56 (m, 4H), 7.49 – 7.44 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.43, 147.29, 146.07, 136.26, 133.53, 131.63, 130.46, 129.92, 129.33, 129.04, 128.44, 128.19, 127.69, 127.34, 127.25, 126.56, 125.82, 125.77, 125.46, 124.07, 123.10; HRMS (ESI) calcd for $C_{23}H_{16}N$ $[M + H]^+$: 306.1277; found: 306.1272.

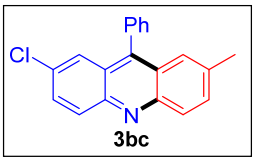


3-Methoxy-7-phenylbenzo[*c*]acridine (3af). Following the general procedure (A) for 48 h on a



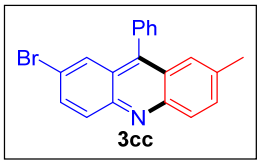
1.0 mmol scale, giving the compound as an off white solid (214 mg, 64%); m.p.193-194 °C; IR (neat)$_{\nu max}$ 1349 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.47 (d, J = 9.0 Hz, 1H), 8.36 (d, J = 8.4 Hz, 1H), 7.76 (ddd, J = 8.0, 6.4, 1.2 Hz, 1H), 7.66 (d, J = 8.0 Hz, 1H), 7.61 – 7.55 (m, 3H), 7.49 – 7.40 (m, 6H), 7.36 (dd, J = 9.0, 2.6 Hz, 1H), 3.95 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.39, 147.45, 147.42, 146.06, 136.31, 135.12, 130.44, 129.65, 129.29, 128.38, 128.13, 127.29, 127.09, 126.57, 125.58, 125.38, 125.29, 124.75, 122.33, 116.63, 108.76, 55.42; HRMS (ESI) calcd for $C_{24}H_{18}NO$ [M + H]$^{+}$: 336.1383; found: 336.1374.

2-Chloro-7-methyl-9-phenylacridine (3bc). Following the general procedure (A) for 48 h on a



1.0 mmol scale, giving the compound as a pale yellow solid (196 mg, 65%); m.p. 201-202 °C; IR (neat)$_{\nu max}$ 2922, 1344, 730 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (dd, J = 9.1, 0.5 Hz, 1H), 8.15 (d, J = 9.2 Hz, 1H) , 7.67 – 7.59 (m, 6H), 7.43 – 7.37 (m, 3H), 2.45 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.80, 146.42, 145.18, 136.23, 135.48, 133.16, 131.34, 131.28, 130.65, 130.33, 129.35, 128.63, 128.52, 125.60, 125.39, 124.95, 124.67, 22.03; HRMS (ESI) calcd for $C_{20}H_{15}ClN$ [M + H]$^{+}$: 304.0888; found: 304.0883.

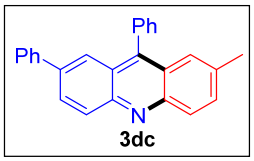
2-Bromo-7-methyl-9-phenylacridine (3cc). Following the general procedure (A) for 36 h on a



1.0 mmol scale, giving the compound as a pale yellow solid (252 mg, 73%); m.p. 202-204 °C; IR (neat)$_{\nu max}$ 2914, 1346, 565 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, J = 8.9 Hz, 1H), 8.11 (dd, J = 9.2, 0.5 Hz, 1H), 7.79 (d, J = 1.7 Hz, 1H), 7.76 (dd, J = 9.2, 2.2 Hz, 1H), 7.64 – 7.60 (m, 4H), 7.42 – 7.38 (m, 3H), 2.45 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.88, 146.53, 145.11, 136.25, 135.44, 133.23, 132.99, 131.34, 130.33,

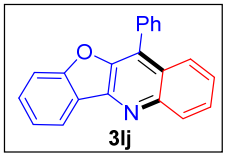
129.38, 128.63, 128.53, 128.44, 126.16, 125.37, 124.70, 119.71, 22.01; HRMS (ESI) calcd for $C_{20}H_{14}BrN$ $[M + H]^+$: 348.0382; found: 348.0386.

2-Methyl-7,9-diphenylacridine (3dc). Following the general procedure (A) for 24 h on a 1.0 mmol scale, giving the compound as a pale yellow solid (272 mg, 79%); m.p. 166-168 °C; IR (neat)$_{\nu max}$ 2922, 1322 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (dd, J = 8.8, 0.8 Hz, 1H), 8.18 (d, J = 9.2 Hz, 1H), 8.00 (dd, J = 8.8 2.1 Hz, 1H), 7.82 (d, J = 2.0 Hz, 1H), 7.63 – 7.56 (m, 6H), 7.45 (dd, J = 8.0, 2.0 Hz, 2H), 7.42 – 7.38 (m, 3H), 7.32 (ddd, J = 8.8, 6.4, 1.2 Hz, 1H), 2.43 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.72, 147.64, 146.07, 140.53, 137.89, 136.06, 135.55, 132.79, 130.44, 130.04, 129.60, 129.32, 128.82, 128.49, 128.27, 127.52, 127.32, 125.42, 125.33, 124.71, 124.00, 21.99; HRMS (ESI) calcd for $C_{26}H_{20}N$ $[M + H]^+$: 346.1590; found: 346.1588.



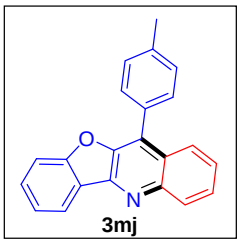
3dc

11-Phenylbenzofuro[3,2-*b*]quinolone (3lj). By replacing cyclohexanone with cyclohexenone and following the general procedure (A) for 6 h on a 1.0 mmol scale, giving the compound as a brown solid (153 mg, 52%); m.p. 184-186 °C; IR (neat)$_{\nu max}$ 1336, 1188 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (dd, J = 7.7, 0.6 Hz, 1H), 8.37 (d, J = 8.6 Hz, 1H), 8.01 (dd, J = 8.6, 1.0 Hz, 1H), 7.74 (ddd, J = 8.4, 6.8, 1.6 Hz, 1H), 7.70-7.64 (m, 4H), 7.60 (m, 3H), 7.54 (ddd, J = 8.4, 6.8, 1.2 Hz, 1H), 7.47 (ddd, J = 8.0, 6.8, 1.2 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.57, 146.98, 146.55, 145.19, 132.11, 130.83, 130.58, 129.50, 128.88, 128.72, 127.89, 127.77, 126.11, 126.05, 125.62, 123.57, 123.13, 122.28, 112.32; HRMS (ESI) calcd for $C_{21}H_{14}NO$ $[M + H]^+$: 296.1070; found: 296.1062.

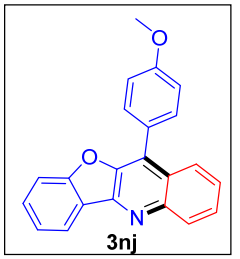


3lj

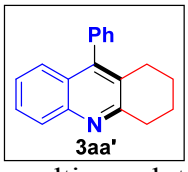
11-(*p*-Tolyl)benzofuro[3,2-*b*]quinolone (3mj). By replacing cyclohexanone with cyclohexenone and following the general procedure (A) for 8 h on a 1.0 mmol scale, giving the compound as a white solid (201 mg, 65%); m.p. 191-193 °C; IR (neat)$_{\nu max}$ 2855, 1338, 1130 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.42 (dq, J = 7.6, 0.8 Hz, 1H), 8.35 (dq, J = 8.0, 0.8 Hz, 1H), 8.03 (dq, J = 8.4, 1.2 Hz, 1H), 7.73 (ddd, J = 8.4, 6.4, 1.2 Hz, 1H), 7.62 – 7.52 (m, 5H), 7.48 – 7.44 (m, 3H), 2.52 (s, 3H) ; ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.58, 146.91, 146.58, 145.28, 144.75, 138.88, 130.78, 130.48, 129.47, 129.07, 127.73, 126.24, 125.94, 125.71, 125.01, 123.95, 123.52, 122.29, 112.32, 21.48; HRMS (ESI) calcd for $C_{22}H_{16}NO$ $[M + H]^+$: 310.1226; found 310.1220.



11-(4-Methoxyphenyl)benzofuro[3,2-*b*] quinolone (3nj). By replacing cyclohexanone with cyclohexenone and following the general procedure (A) for 8 h on a 1.0 mmol scale, giving the compound as a pale yellow solid (107 mg, 33%); m.p. 218-220 °C; IR(neat)$_{\nu max}$ 2850, 1341, 1256, 1183 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (dq, J = 8.4, 0.8 Hz, 1H), 8.35 (dq, J = 8.4, 0.8 Hz, 1H), 8.03 (dq, J = 8.4, 0.8 Hz, 1H), 7.75 (ddd, J = 8.3, 6.8, 1.4 Hz, 1H), 7.65 (d, J = 8.4 Hz, 2H), 7.63- 7.52 (m, 1H), 7.47 (ddd, J = 8.4, 7.2, 1.2 Hz, 1H), 7.18 (d = J = 8.8 Hz, 2H), 3.96 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.07, 159.52, 146.88, 146.65, 145.32, 131.92, 130.73, 129.51, 127.77, 127.70, 126.29, 125.91, 125.67, 124.12, 123.51, 123.22, 122.26, 114.26, 112.28, 55.40; HRMS (ESI) calcd for $C_{22}H_{16}NO_2$ $[M + H]^+$: 326.1175; found 326.1174.

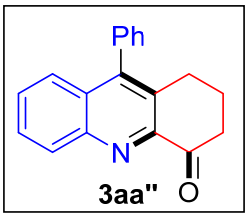


9-Phenyl-1,2,3,4-tetrahydroacridine (3aa').[6] A 15 mL oven dried sealed tube was charged with 2-aminobenzophenone **1a** (197 mg, 1.0 mmol), DMSO (0.5 mL), cyclohexanone **2a** (156 µL, 1.5 mmol) and trifluoroacetic acid (153 µL, 2.0 mmol). The resulting solution was stirred at 65 °C for 2 h. After cooling to room temperature, the reaction



mixture was diluted with 5 mL of water and neutralized with 10% aqueous $NaHCO_3$ solution. The aqueous layer was extracted with (3X10 mL) of ethyl acetate and the combined organic layer was given brine wash (2X10 mL). The final organic layer was evaporated in a rotary evaporator to remove the volatile components. The residue was purified by column chromatography on silica gel (8% ethyl acetate/hexane) to afford compound **3aa'** as an off white solid (245 mg, 95%); m.p. 144-145 °C ; IR (neat)$_{\nu max}$ 2871, 1350 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, J = 8.4 Hz, 1H), 7.57 (ddd, J = 8.4, 5.6, 2.4 Hz, 1H), 7.52 – 7.44 (m, 3H), 7.31 – 7.28 (m, 2H), 7.23 – 7.20 (m, 2H), 3.20 (t, J = 6.4 Hz, 2H), 2.60 (t, J = 6.4 Hz, 2H), 1.95 (quin, J = 6.8 Hz, 2H), 1.77 (quin, J = 6.4 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.00, 146.41, 146.25, 137.08, 129.04, 128.53, 128.30, 128.26, 127.65, 126.60, 125.71, 125.30, 34.20, 27.98, 22.97, 22.86; HRMS (ESI) calcd for $C_{19}H_{18}N$ $[M + H]^+$: 260.1434; found: 260.1430.

9-Phenyl-2,3-dihydroacridin-4(1*H*)-one (3aa"). A 15 mL oven dried sealed tube was charged



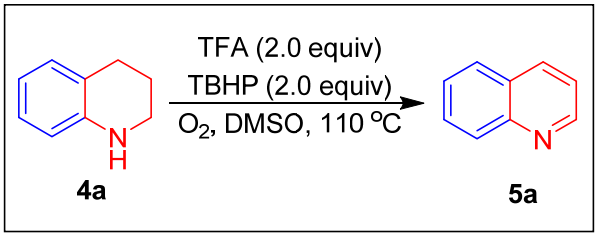
3aa"

with compound **3aa'** (259 mg, 1.0 mmol), DMSO (0.5 mL), trifluoroacetic acid (153 µL, 2.0 mmol) and 70% aq. TBHP solution (257 µL, 2.0 mmol). The resulting solution was sealed under O_2 and stirred at 110°C for 2 h. After cooling to room temperature, the reaction mixture was diluted with 5 mL of water and neutralized with 10% aqueous $NaHCO_3$ solution. The aqueous layer was extracted with (3X10 mL) of ethyl acetate and the combined organic layer was given brine wash (2X10 mL). The final organic layer was evaporated in a rotary evaporator to remove the volatile components. The residue was purified by column chromatography on silica gel (10% ethyl acetate/hexane) to afford compound **3aa''** as a pale yellow solid (68 mg, 25%); m.p. 157-159 °C; IR (neat)$_{\nu max}$ 2850, 1710, 1335 cm $^{-1}$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 (dq, J = 8.8, 0.8 Hz, 1H), 7.71 (ddd, J = 8.4, 6.8, 1.2 Hz, 1H), 7.58 – 7.44 (m, 5H), 7.28 (dd, J = 8.4, 2.0 Hz, 2H), 2.91 (t, J = 6.8 Hz,

2H), 2.86 (t, J = 6.0 Hz, 2H), 2.14 (quin, J = 6.4 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.58, 148.49, 148.20, 147.25, 136.23, 133.51, 131.59, 129.56, 129.23, 128.90, 128.81, 128.78, 128.43, 125.90, 40.29, 28.06, 22.67; HRMS (ESI) calcd for $C_{19}H_{15}NNaO$ $[M + Na]^+$: 296.1046 ; found: 296.1048.

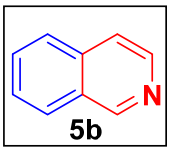
General procedure (B) for benzylic oxidation reaction

Preparation of Quinoline.[7]

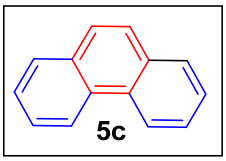


A 15 mL oven dried sealed tube was charged with 1,2,3,4-tetrahydroquinoline **4a** (134 mg, 1.0 mmol), DMSO (0.5 mL), trifluoroacetic acid (153 µL, 2.0 mmol) and 70% aq. TBHP solution (257 µL, 2.0 mmol). The resulting solution was sealed under O_2 and stirred at 110°C for 48 h. After cooling to room temperature, the reaction mixture was diluted with 5 mL of water and neutralized with 10% aqueous $NaHCO_3$ solution. The aqueous layer was extracted with (3X10 mL) of ethyl acetate and the combined organic layer was given brine wash (2X10 mL). The final organic layer was evaporated in a rotary evaporator to remove the volatile components. The residue was purified by column chromatography on silica gel (hexane to 10% ethyl acetate/hexane) to afford pure quinoline 5**a** as a dark yellow liquid (70 mg, 55%); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.91 (m, 1H), 8.14-8.10 (m, 2H), 7.79 (d, J = 8.4 Hz, 1H), 7.70 (td, J = 8.8, 1.6, 1H), 7.55-7.51 (m, 1H), 7.37 (dd, J = 8.4 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.46, 148.35, 136.12, 129.52, 128.36, 127.86, 126.61, 121.13.

Isoquinoline (5b).[8] Following the general procedure (B) for 24 h on a 1.0 mmol scale, giving the compound as dark yellow color liquid (81 mg, 63%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.21 (s, 1H), 8.48 (d, *J* = 6.0 Hz, 1H), 7.97 – 7.84 (m, 1H), 7.76 (dd, *J* = 9.2, 0.8 Hz, 1H), 7.71 – 7.41 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 152.57, 143.01, 135.84, 130.42, 128.74, 127.68, 127.31, 126.53, 120.54.



Phenanthrene (5c).[9] Following the general procedure (B) for 24 h on a 1.0 mmol scale, the reaction underwent 50% conversion by ^{1}H NMR. The compound could not be isolated due to the similar boiling point.[10] The spectral characterization complies with the reported literature data.



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checkCIF/PLATON report

Structure factors have been supplied for datablock(s) g1063

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: g1063

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                 alpha=90       beta=107.345(8)     gamma=90
Temperature:     150 K
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Hall group	-P 2yn	-P 2yn
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Sum formula	C19 H13 N	C19 H13 N
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Dx,g cm-3	1.296	1.296
Z	4	4
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F000’	536.19	
h,k,lmax	11,24,12	11,24,12
Nref	3595	3031
Tmin,Tmax	0.979,0.990	0.988,1.000
Tmin’	0.978	

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AbsCorr = MULTI-SCAN

Data completeness= 0.843          Theta(max)= 29.324

R(reflections)= 0.0639( 1885)     wR2(reflections)= 0.1479( 3031)

S = 1.012                 Npar= 181
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The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

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PLAT906_ALERT_3_C Large K value in the Analysis of Variance ......      2.762 Check
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Alert level G

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PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600        562 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density           4 Note
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   2 ALERT level C = Check. Ensure it is not caused by an omission or oversight
   4 ALERT level G = General information/check it is not something unexpected

   0 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   1 ALERT type 2 Indicator that the structure model may be wrong or deficient
   3 ALERT type 3 Indicator that the structure quality may be low
   2 ALERT type 4 Improvement, methodology, query or suggestion
   0 ALERT type 5 Informative message, check
```

Validation response form

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

```
# start Validation Reply Form
_vrf_PLAT906_g1063
;
PROBLEM: Large K value in the Analysis of Variance ......      9.846 Check
RESPONSE: ...
;
# end Validation Reply Form
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

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Publication of your CIF in other journals

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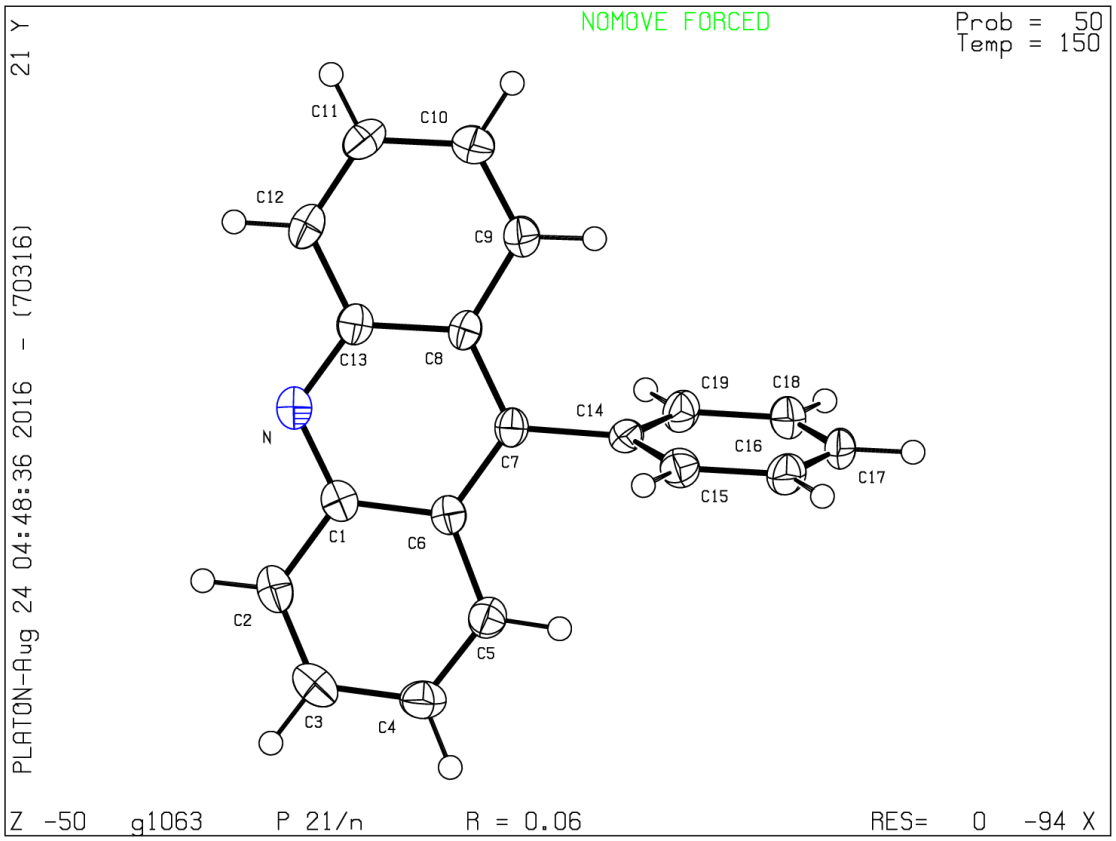
PLATON version of 11/08/2016; check.def file version of 04/08/2016

Datablock g1063 - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) g1025

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: g1025

```
Bond precision:  C-C = 0.0080 A          Wavelength=0.71073

Cell:         a=7.5233(11)       b=9.2661(15)     c=11.5013(15)
              alpha=109.068(14)  beta=96.475(12)  gamma=105.036(13)
Temperature:  297 K
```

	Calculated	Reported
Volume	714.5(2)	714.49(19)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C19 H12 Cl N	C19 H12 Cl N
Sum formula	C19 H12 Cl N	C19 H12 Cl N
Mr	289.75	289.75
Dx,g cm-3	1.347	1.347
Z	2	2
Mu (mm-1)	0.259	0.259
F000	300.0	300.0
F000’	300.39	
h,k,lmax	10,12,15	10,11,15
Nref	3876	3251
Tmin,Tmax	0.954,0.992	0.800,1.000
Tmin’	0.940	

```
Correction method= # Reported T Limits: Tmin=0.800 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.839          Theta(max)= 29.197

R(reflections)= 0.0807( 1184)     wR2(reflections)= 0.2825( 3251)

S = 0.984                 Npar= 190
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

```
PLAT026_ALERT_3_B Ratio Observed / Unique Reflections (too) Low ..         36 %
```

Alert level C

```
ABSTY02_ALERT_1_C  An _exptl_absorpt_correction_type has been given without
           a literature citation. This should be contained in the
           _exptl_absorpt_process_details field.
           Absorption correction given as multi-scan
RFACR01_ALERT_3_C  The value of the weighted R factor is > 0.25
           Weighted R factor given   0.282
PLAT084_ALERT_3_C High wR2 Value (i.e. > 0.25) ...................       0.28 Report
PLAT340_ALERT_3_C Low Bond Precision on  C-C Bonds ...............    0.00805 Ang.
PLAT906_ALERT_3_C Large K value in the Analysis of Variance ......     12.981 Check
PLAT906_ALERT_3_C Large K value in the Analysis of Variance ......      2.484 Check
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density          0 Note
```

Alert level G

```
PLAT072_ALERT_2_G SHELXL First  Parameter in WGHT  Unusually Large       0.11 Report
PLAT898_ALERT_4_G Second Reported H-M Symbol in CIF Ignored ......          ! Check
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min)           4 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600        572 Note
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
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   1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
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   7 ALERT type 3 Indicator that the structure quality may be low
   2 ALERT type 4 Improvement, methodology, query or suggestion
   0 ALERT type 5 Informative message, check
```

Validation response form

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

```
# start Validation Reply Form
_vrf_ABSTY02_g1025
;
PROBLEM: An _exptl_absorpt_correction_type has been given without
RESPONSE: ...
;
_vrf_RFACR01_g1025
;
PROBLEM: The value of the weighted R factor is > 0.25
RESPONSE: ...
;
_vrf_PLAT084_g1025
;
PROBLEM: High wR2 Value (i.e. > 0.25) ...................       0.28 Report
RESPONSE: ...
;
_vrf_PLAT340_g1025
;
PROBLEM: Low Bond Precision on  C-C Bonds ...............    0.00805 Ang.
```

```
RESPONSE: ...
;
_vrf_PLAT906_g1025
;
PROBLEM: Large K value in the Analysis of Variance ......     12.981 Check
RESPONSE: ...
;
_vrf_PLAT978_g1025
;
PROBLEM: Number C-C Bonds with Positive Residual Density           0 Note
RESPONSE: ...
;
# end Validation Reply Form
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

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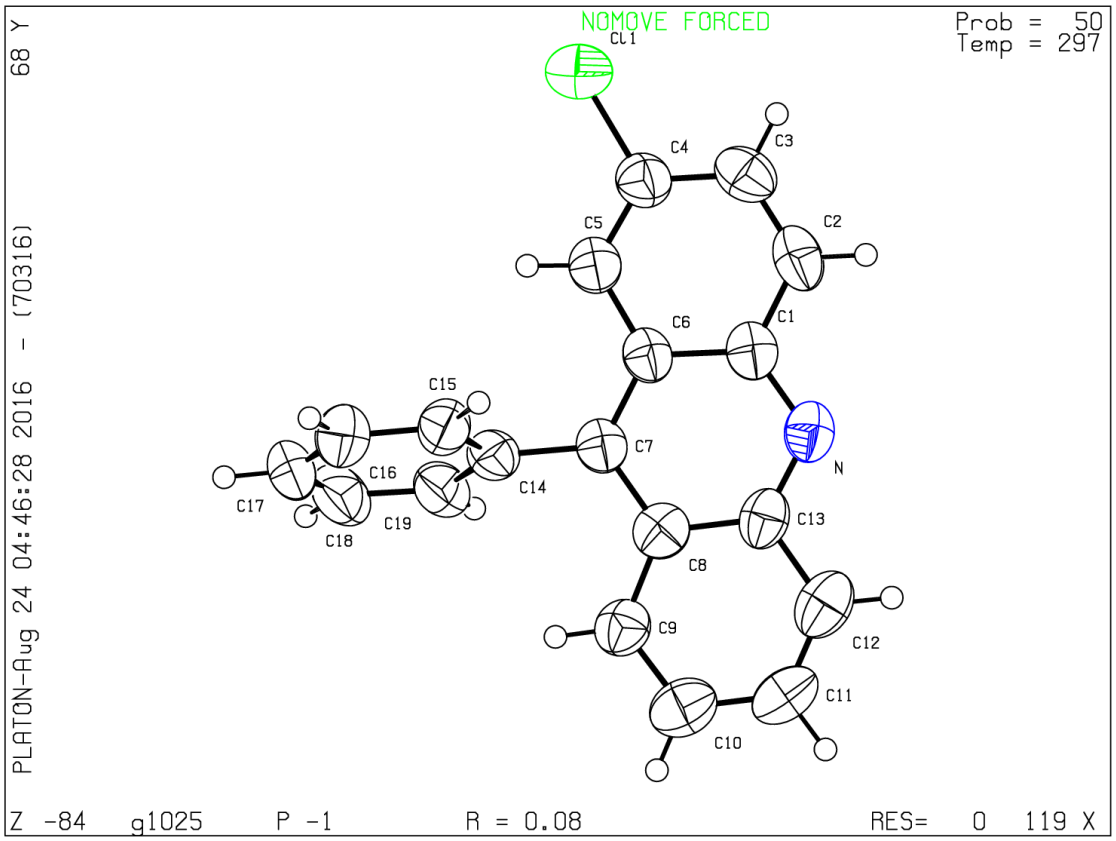
Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 11/08/2016; check.def file version of 04/08/2016

Datablock g1025 - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) int1

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: int1

```
Bond precision:  C-C = 0.0041 A          Wavelength=0.71073

Cell:          a=9.0735(9)       b=9.1093(10)     c=9.1688(9)
               alpha=71.085(3)   beta=75.202(3)   gamma=85.168(4)
Temperature:   150 K
```

	Calculated	Reported
Volume	693.13(12)	693.13(12)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C19 H17 N	C19 H17 N
Sum formula	C19 H17 N	C19 H17 N
Mr	259.34	259.33
Dx,g cm-3	1.243	1.243
Z	2	2
Mu (mm-1)	0.072	0.072
F000	276.0	276.0
F000’	276.09	
h,k,lmax	11,11,11	11,11,11
Nref	2850	2788
Tmin,Tmax	0.973,0.982	0.722,0.928
Tmin’	0.970	

```
Correction method= # Reported T Limits: Tmin=0.722 Tmax=0.928
AbsCorr = MULTI-SCAN

Data completeness= 0.978          Theta(max)= 26.387

R(reflections)= 0.0689( 1834)     wR2(reflections)= 0.2090( 2788)

S = 1.029                 Npar= 181
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

```
DIFMX01_ALERT_2_C  The maximum difference density is > 0.1*ZMAX*0.75
           _refine_diff_density_max given =      0.548
           Test value =      0.525
DIFMX02_ALERT_1_C  The maximum difference density is > 0.1*ZMAX*0.75
           The relevant atom site should be identified.
PLAT097_ALERT_2_C Large Reported Max.  (Positive) Residual Density       0.55 eA-3
PLAT340_ALERT_3_C Low Bond Precision on  C-C Bonds ..............     0.0041 Ang.
PLAT906_ALERT_3_C Large K value in the Analysis of Variance ......      2.024 Check
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min)          6 Note
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L=  0.600         41 Report
```

Alert level G

```
PLAT898_ALERT_4_G Second Reported H-M Symbol in CIF Ignored ......          ! Check
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600         15 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density           5 Note
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
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   7 ALERT level C = Check. Ensure it is not caused by an omission or oversight
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   1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
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   4 ALERT type 3 Indicator that the structure quality may be low
   2 ALERT type 4 Improvement, methodology, query or suggestion
   0 ALERT type 5 Informative message, check
```

Validation response form

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

```
# start Validation Reply Form
_vrf_DIFMX01_int1
;
PROBLEM: The maximum difference density is > 0.1*ZMAX*0.75
RESPONSE: ...
;
_vrf_DIFMX02_int1
;
PROBLEM: The maximum difference density is > 0.1*ZMAX*0.75
RESPONSE: ...
;
_vrf_PLAT097_int1
;
PROBLEM: Large Reported Max.  (Positive) Residual Density       0.55 eA-3
RESPONSE: ...
;
_vrf_PLAT340_int1
;
PROBLEM: Low Bond Precision on  C-C Bonds ..............     0.0041 Ang.
RESPONSE: ...
;
_vrf_PLAT906_int1
;
PROBLEM: Large K value in the Analysis of Variance ......      2.024 Check
RESPONSE: ...
;
```

```
_vrf_PLAT910_int1
;
PROBLEM: Missing # of FCF Reflection(s) Below Theta(Min)           6 Note
RESPONSE: ...
;
_vrf_PLAT911_int1
;
PROBLEM: Missing # FCF Refl Between THmin & STh/L=  0.600         41 Report
RESPONSE: ...
;
# end Validation Reply Form
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

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Publication of your CIF in other journals

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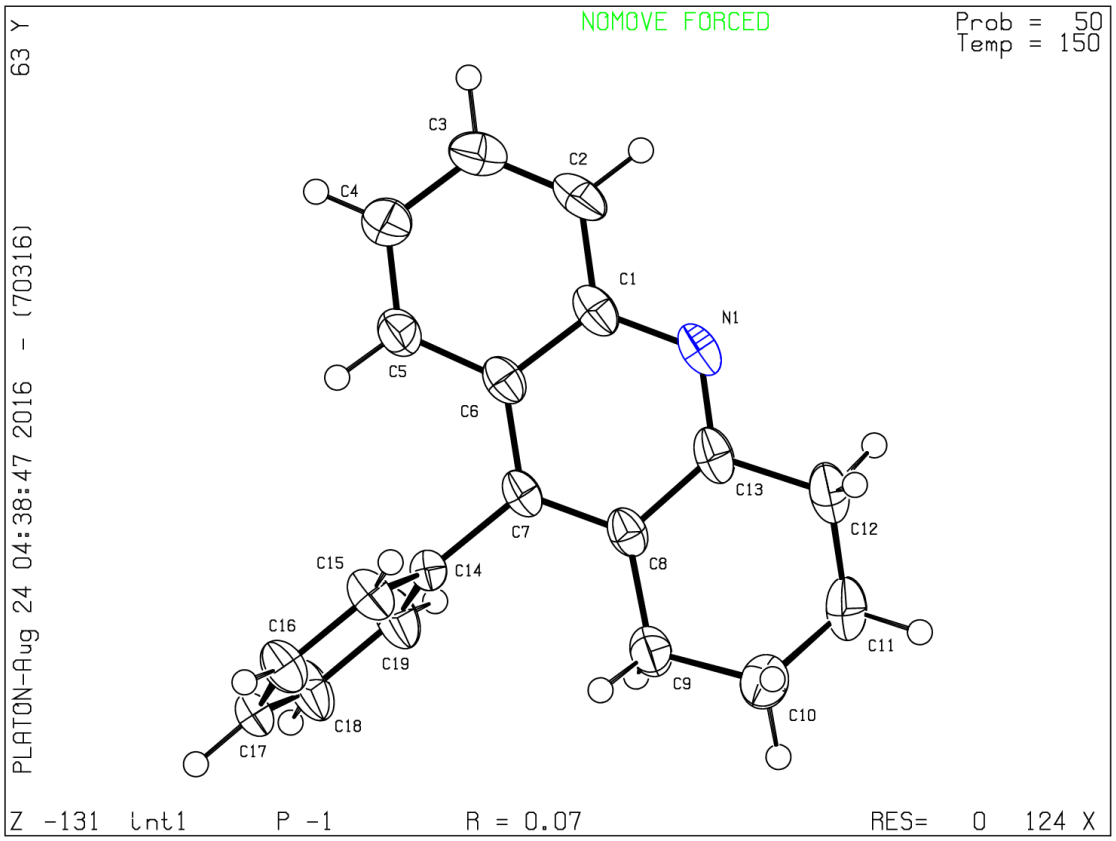
PLATON version of 11/08/2016; check.def file version of 04/08/2016

Datablock int1 - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) int2

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: int2

```
Bond precision:  C-C = 0.0021 A          Wavelength=0.71073

Cell:          a=8.2767(7)        b=8.6984(6)      c=11.4987(9)
               alpha=110.318(2)   beta=90.656(3)   gamma=115.299(3)
Temperature:   150 K
```

	Calculated	Reported
Volume	689.29(10)	689.29(9)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C19 H15 N O	C19 H15 N O
Sum formula	C19 H15 N O	C19 H15 N O
Mr	273.32	273.32
Dx,g cm-3	1.317	1.317
Z	2	2
Mu (mm-1)	0.081	0.081
F000	288.0	288.0
F000’	288.11	
h,k,lmax	10,10,14	10,10,14
Nref	2829	2813
Tmin,Tmax	0.966,0.968	0.832,0.928
Tmin’	0.966	

```
Correction method= # Reported T Limits: Tmin=0.832 Tmax=0.928
AbsCorr = MULTI-SCAN

Data completeness= 0.994          Theta(max)= 26.392

R(reflections)= 0.0533( 2375)     wR2(reflections)= 0.1625( 2813)

S = 1.021                 Npar= 190
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

```
PLAT094_ALERT_2_C Ratio of Maximum / Minimum Residual Density ....       2.28 Report
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min)          6 Note
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L=  0.600         5 Report
```

Alert level G

```
PLAT072_ALERT_2_G SHELXL First  Parameter in WGHT  Unusually Large       0.10 Report
PLAT898_ALERT_4_G Second Reported H-M Symbol in CIF Ignored ......          ! Check
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600          6 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density           7 Note
```

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
3 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
4 **ALERT level G** = General information/check it is not something unexpected

0 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
3 ALERT type 2 Indicator that the structure model may be wrong or deficient
2 ALERT type 3 Indicator that the structure quality may be low
2 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

Validation response form

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

```
# start Validation Reply Form
_vrf_PLAT094_int2
;
PROBLEM: Ratio of Maximum / Minimum Residual Density ....       2.28 Report
RESPONSE: ...
;
_vrf_PLAT910_int2
;
PROBLEM: Missing # of FCF Reflection(s) Below Theta(Min)          6 Note
RESPONSE: ...
;
_vrf_PLAT911_int2
;
PROBLEM: Missing # FCF Refl Between THmin & STh/L=  0.600         5 Report
RESPONSE: ...
;
# end Validation Reply Form
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

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Publication of your CIF in other journals

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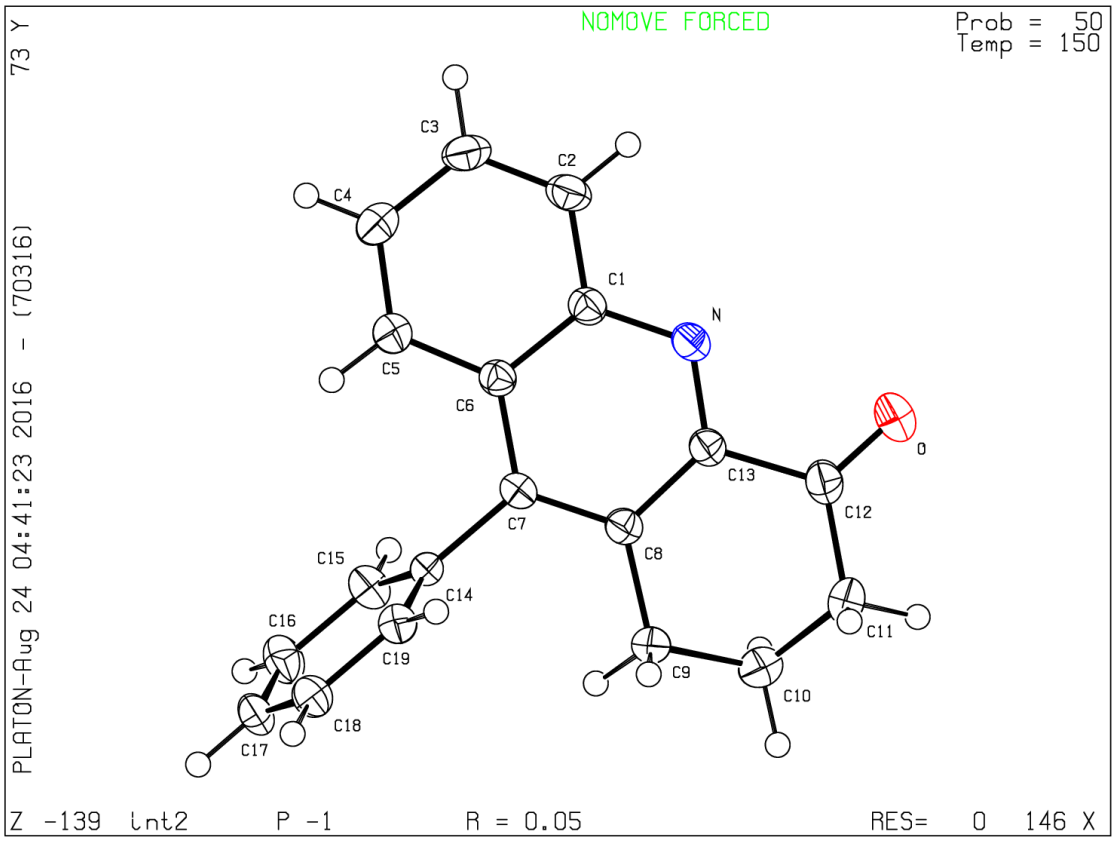
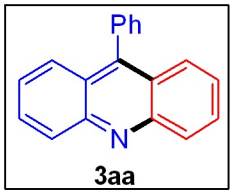
PLATON version of 11/08/2016; check.def file version of 04/08/2016

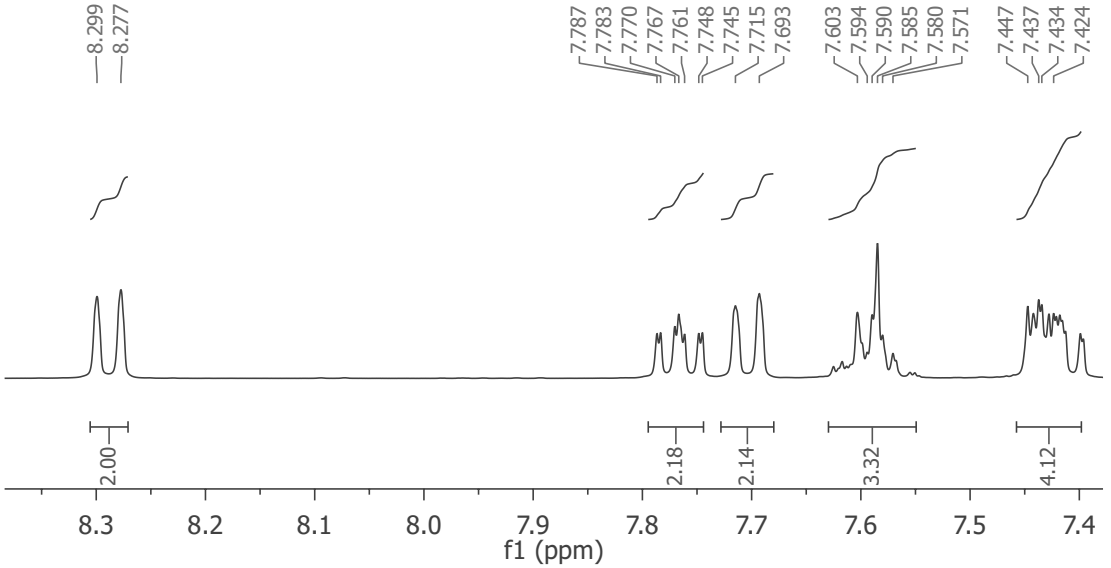
Datablock int2 - ellipsoid plot

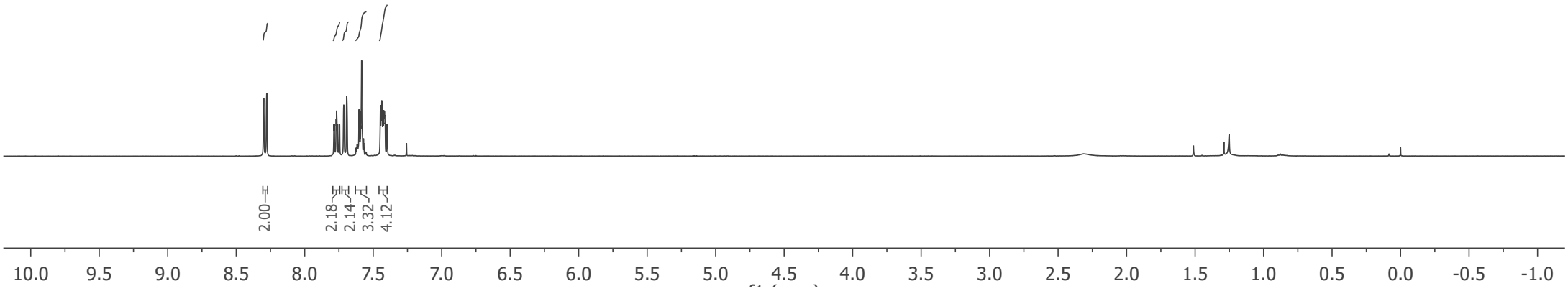


Solvent CDCl3

Spectrometer Frequency 400.40

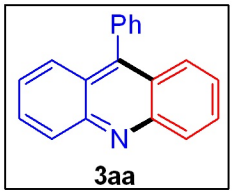




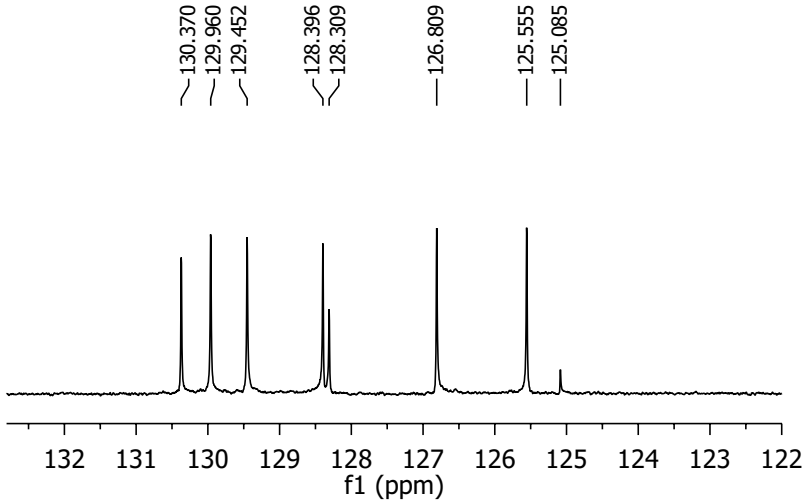


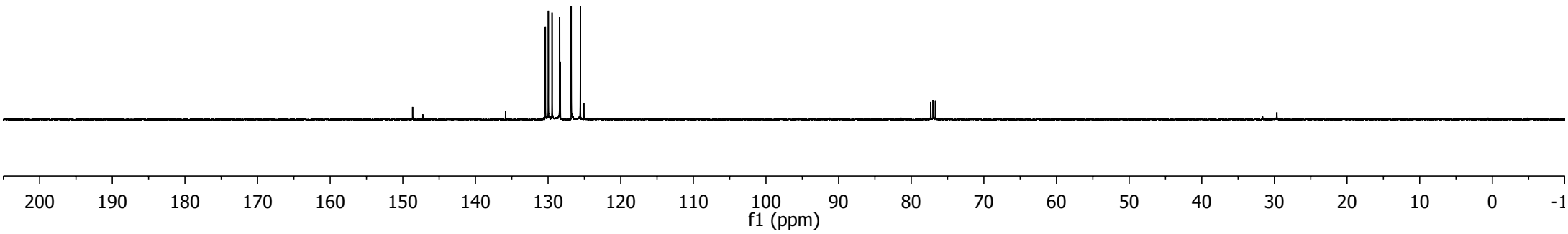
Solvent CDCl3
Spectrometer Frequency 100.69

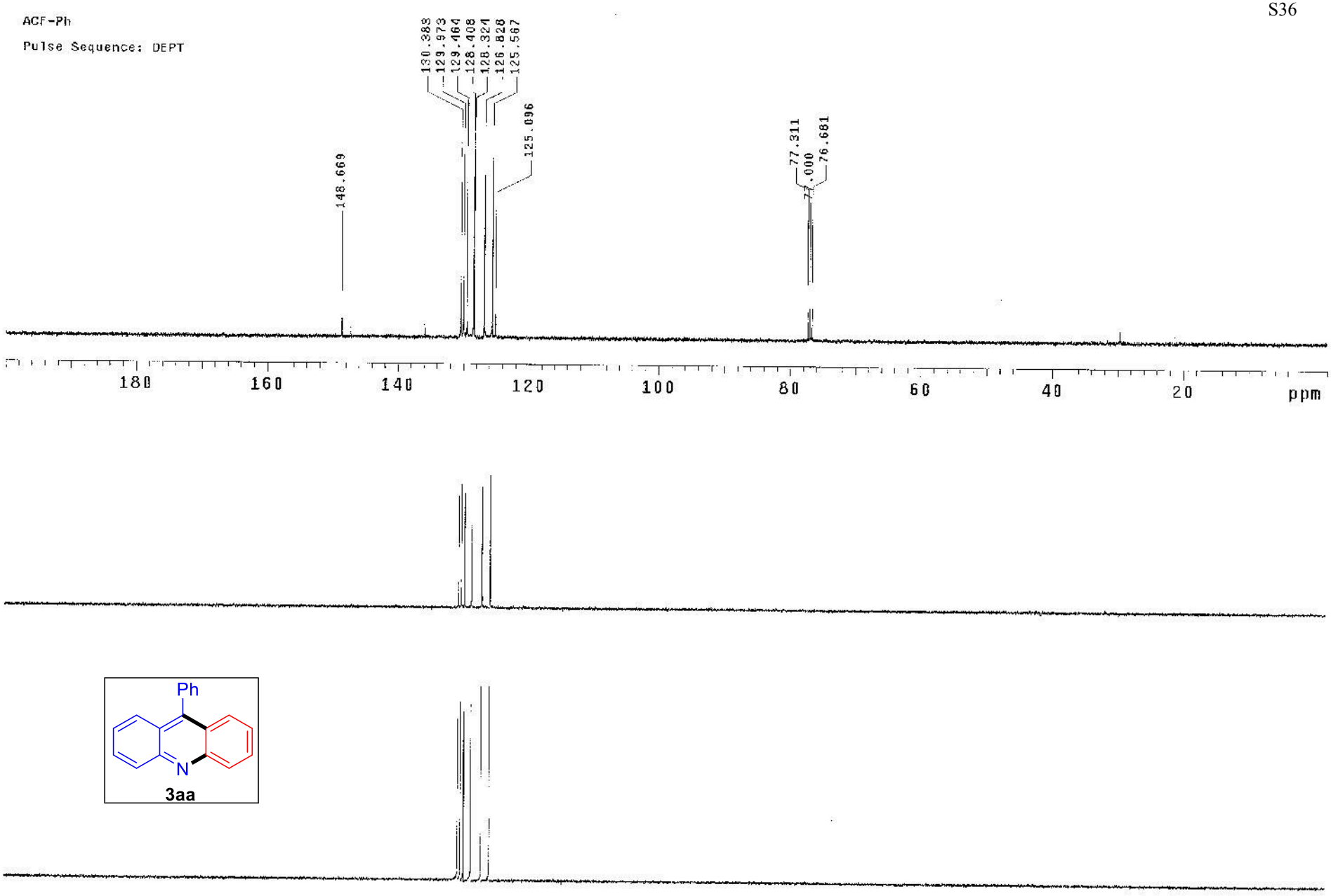
148.655
147.247
135.859
130.370
129.960
129.452
128.396
128.309
126.809
125.555
125.085

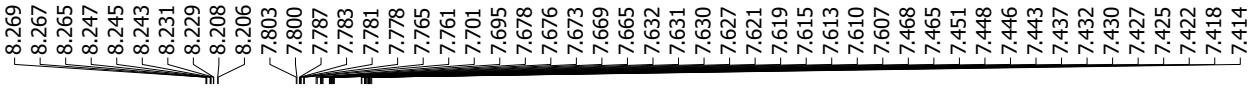


3aa



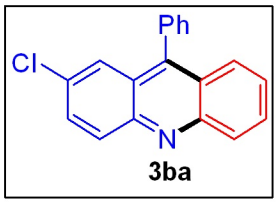


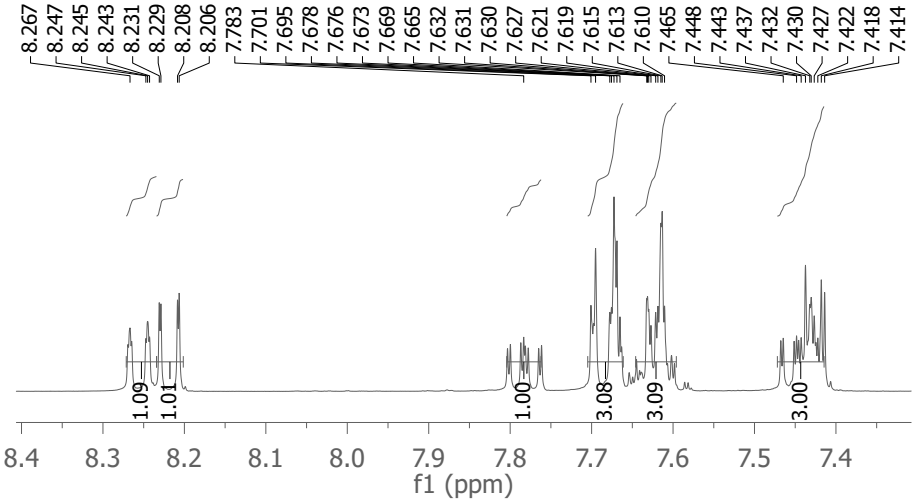

ACF-Ph
Pulse Sequence: DEPT
130.388
129.973
129.464
128.408
128.321
126.828
125.567
125.096
148.669
77.311
77.000
76.681
180
160
140
120
100
80
60
40
20
ppm
Ph
N
3aa

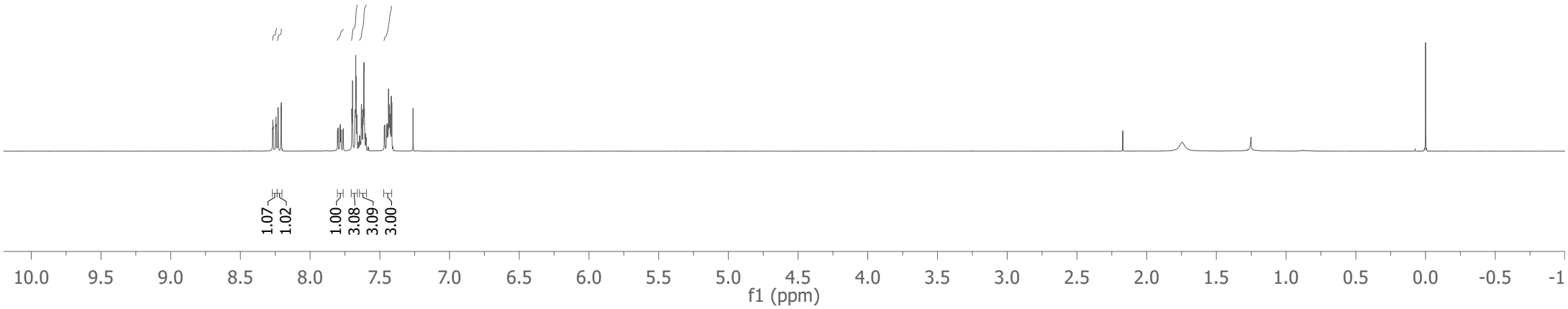


Solvent CDCl3

Spectrometer Frequency 400.40

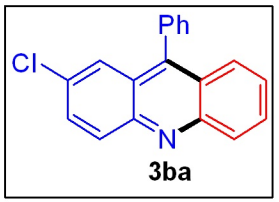






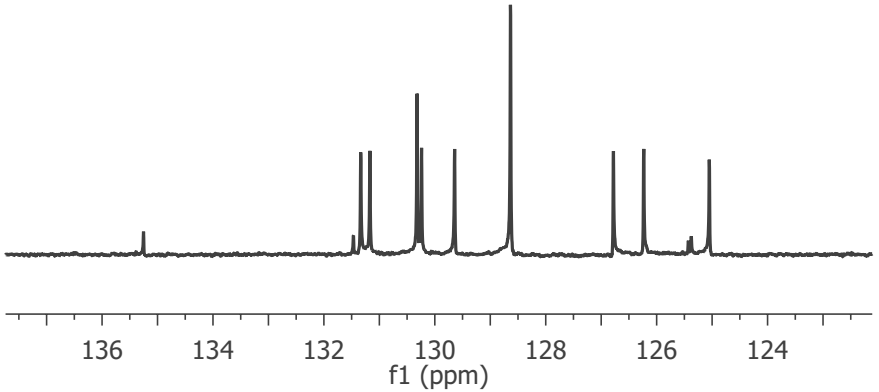
148.807
146.992
146.395
135.252
131.467
131.336
131.170
130.321
130.239
129.643
128.635
126.829
126.780
126.233
125.428
125.375
125.050

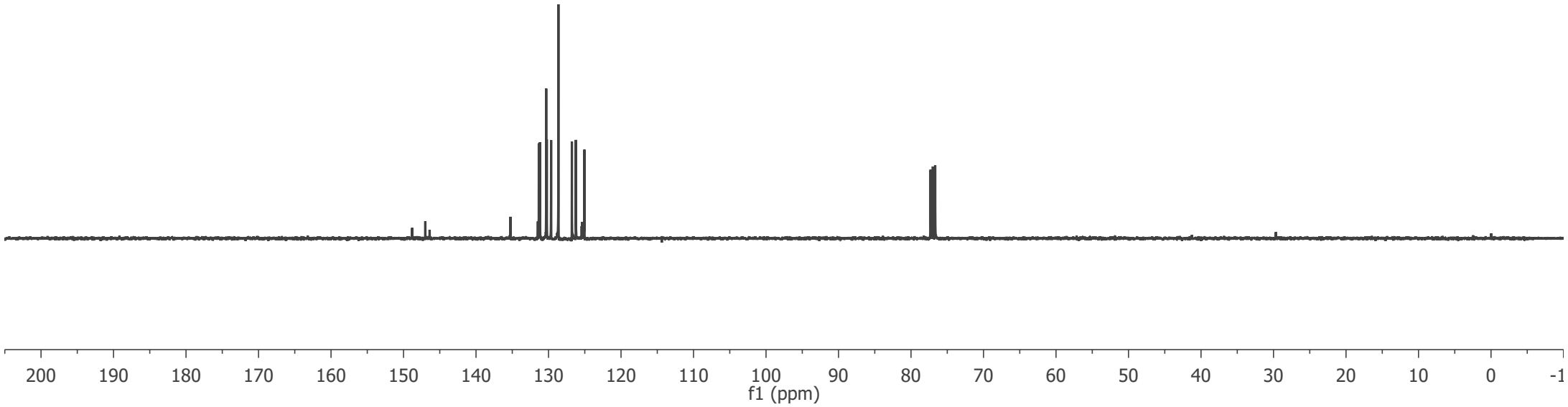
Solvent CDCl3
Spectrometer Frequency 100.69

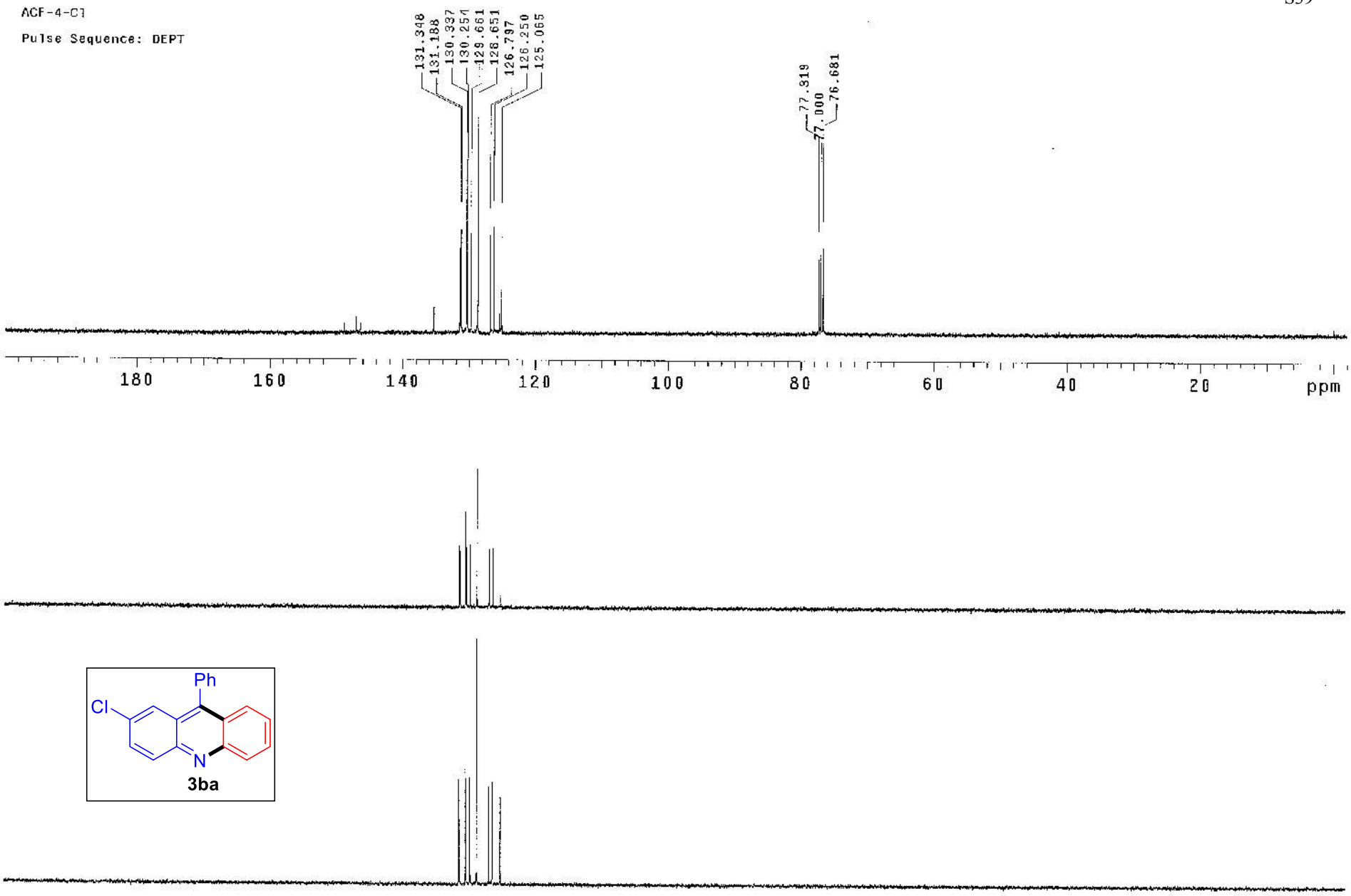


3ba

135.252
131.467
131.336
131.170
130.321
130.239
129.643
128.635
126.829
126.780
126.233
125.428
125.375
125.050



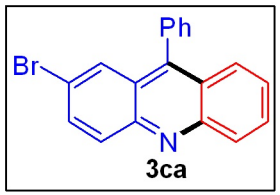



ACF-4-Cl
Pulse Sequence: DEPT
131.348
131.188
130.337
130.254
129.661
128.651
126.797
126.250
125.065
77.319
77.000
76.681
180
160
140
120
100
80
60
40
20
ppm
Ph
Cl
N
3ba

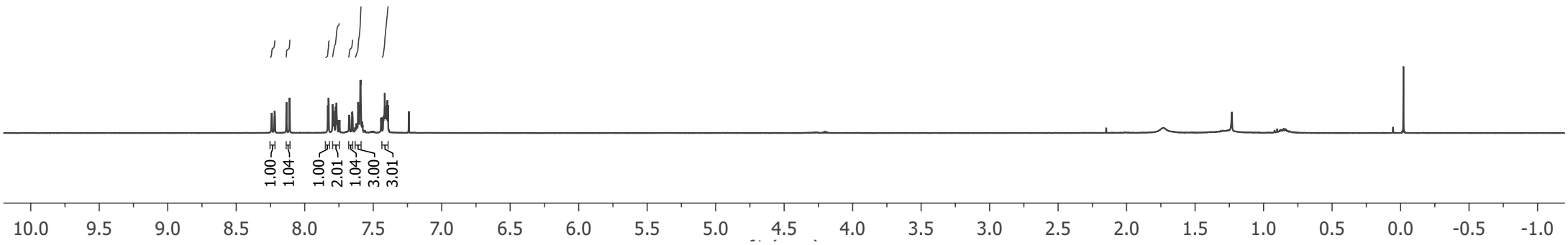
Solvent CDCl3

Spectrometer Frequency 400.28

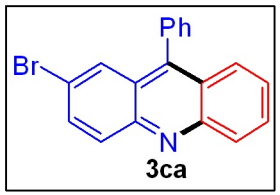
8.241 8.219 8.134 8.133 8.111 8.110 7.675 7.673 7.653 7.651 7.632 7.628 7.623 7.619 7.611 7.610 7.608 7.605 7.600 7.594 7.592 7.585 7.581 7.577 7.565 7.561 7.443 7.440 7.426 7.423 7.421 7.416 7.410 7.405 7.401 7.396 7.392



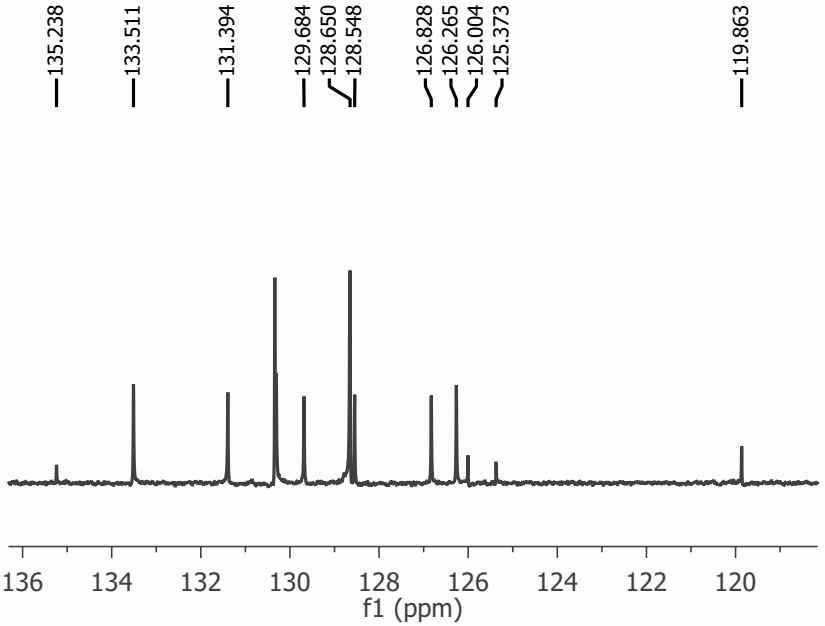
3ca

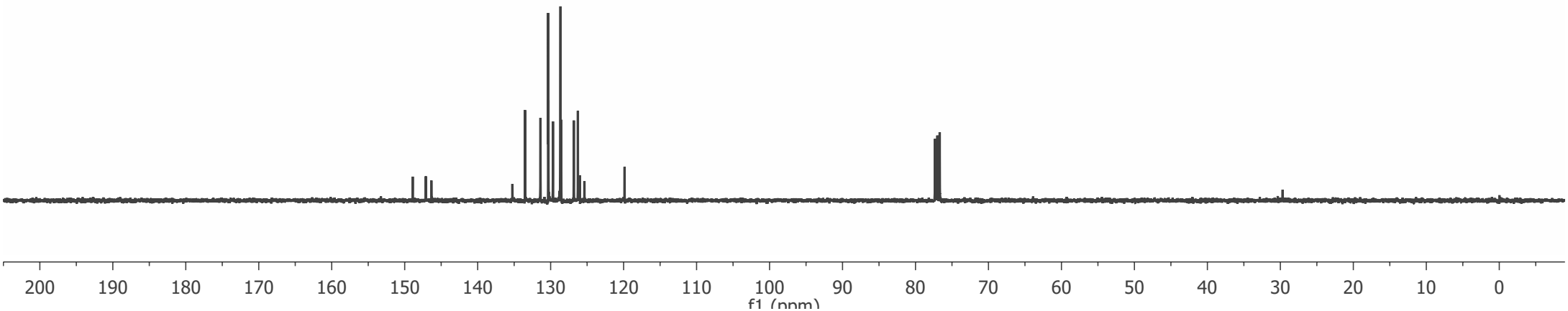


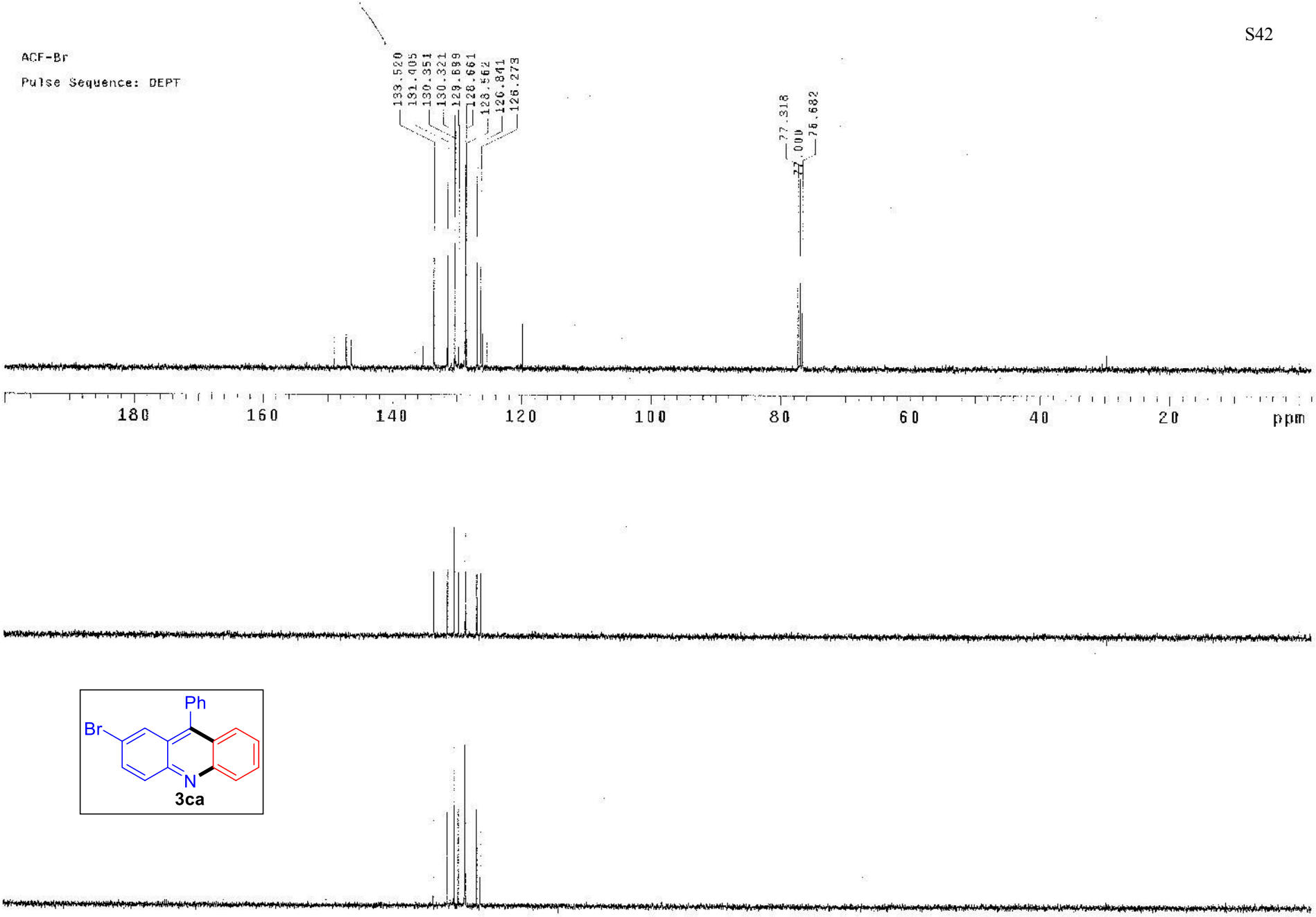
Solvent CDCl3
Spectrometer Frequency 100.66

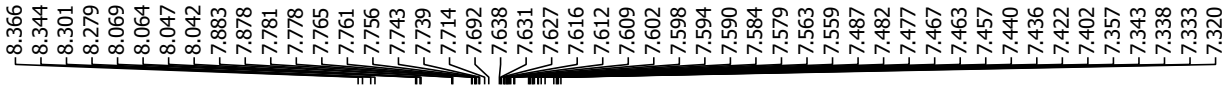


148.902
147.105
146.345
133.511
131.394
129.684
128.650
128.548
126.828
126.265
126.004
125.373
119.863



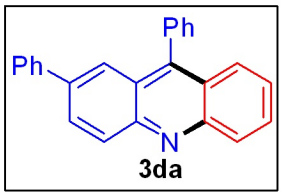


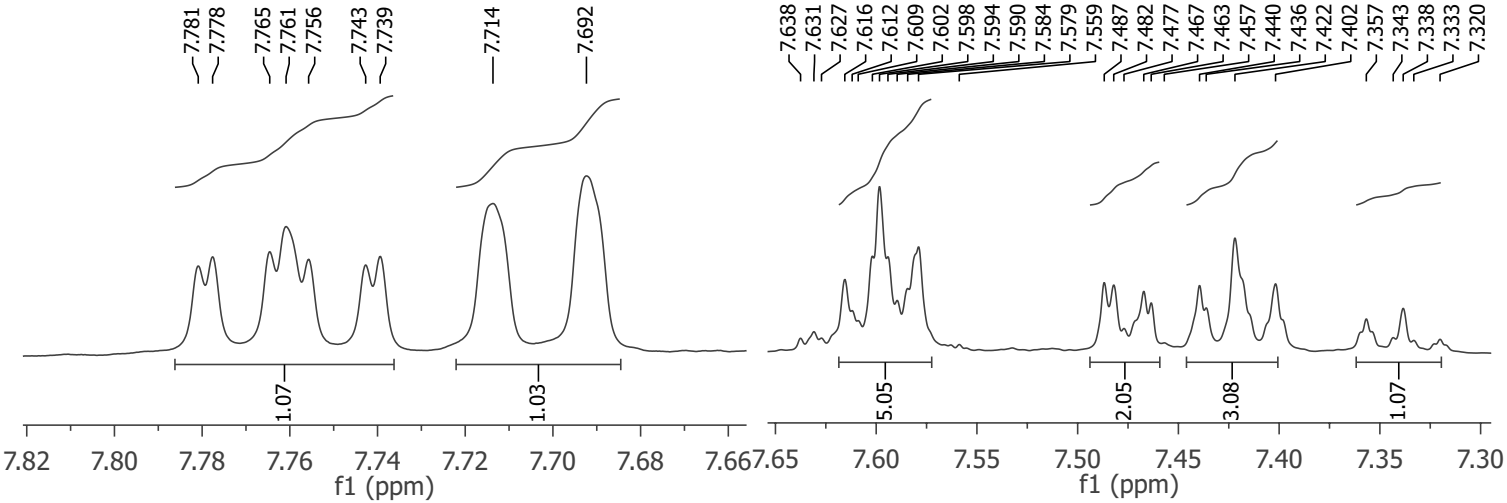

ACF-Br
Pulse Sequence: DEPT
133.520
131.405
130.351
130.321
129.689
128.661
128.562
126.841
126.273
77.318
77.000
76.682
180
160
140
120
100
80
60
40
20
ppm
Ph
Br
N
3ca

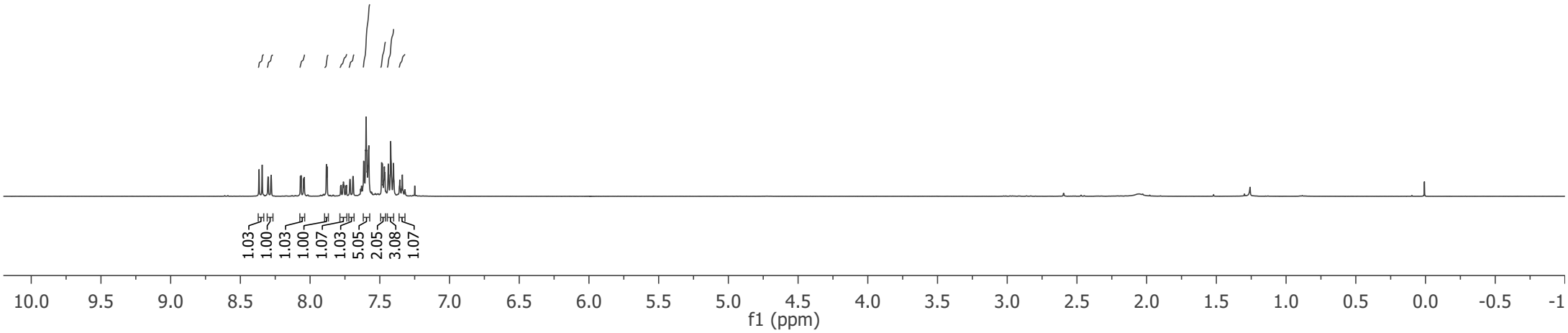


Solvent CDCl3

Spectrometer Frequency 400.40

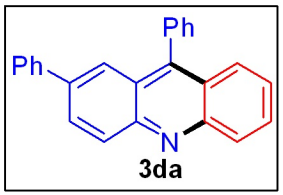




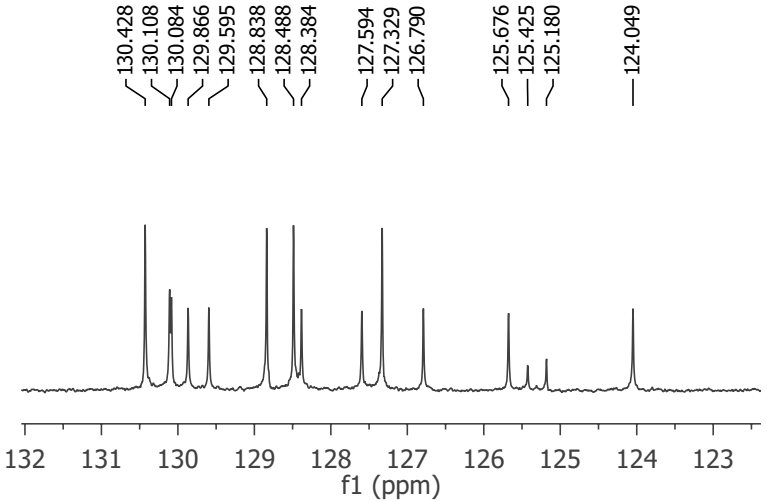


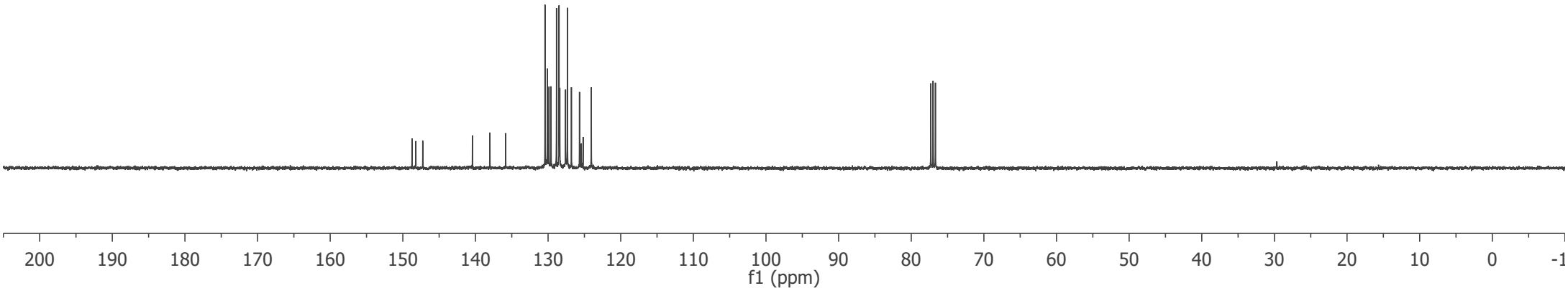
148.738
148.202
147.256
140.417
138.009
135.839
130.428
130.108
130.084
129.866
129.595
128.838
128.488
128.384
127.594
127.329
126.790
125.676
125.425
125.180
124.049

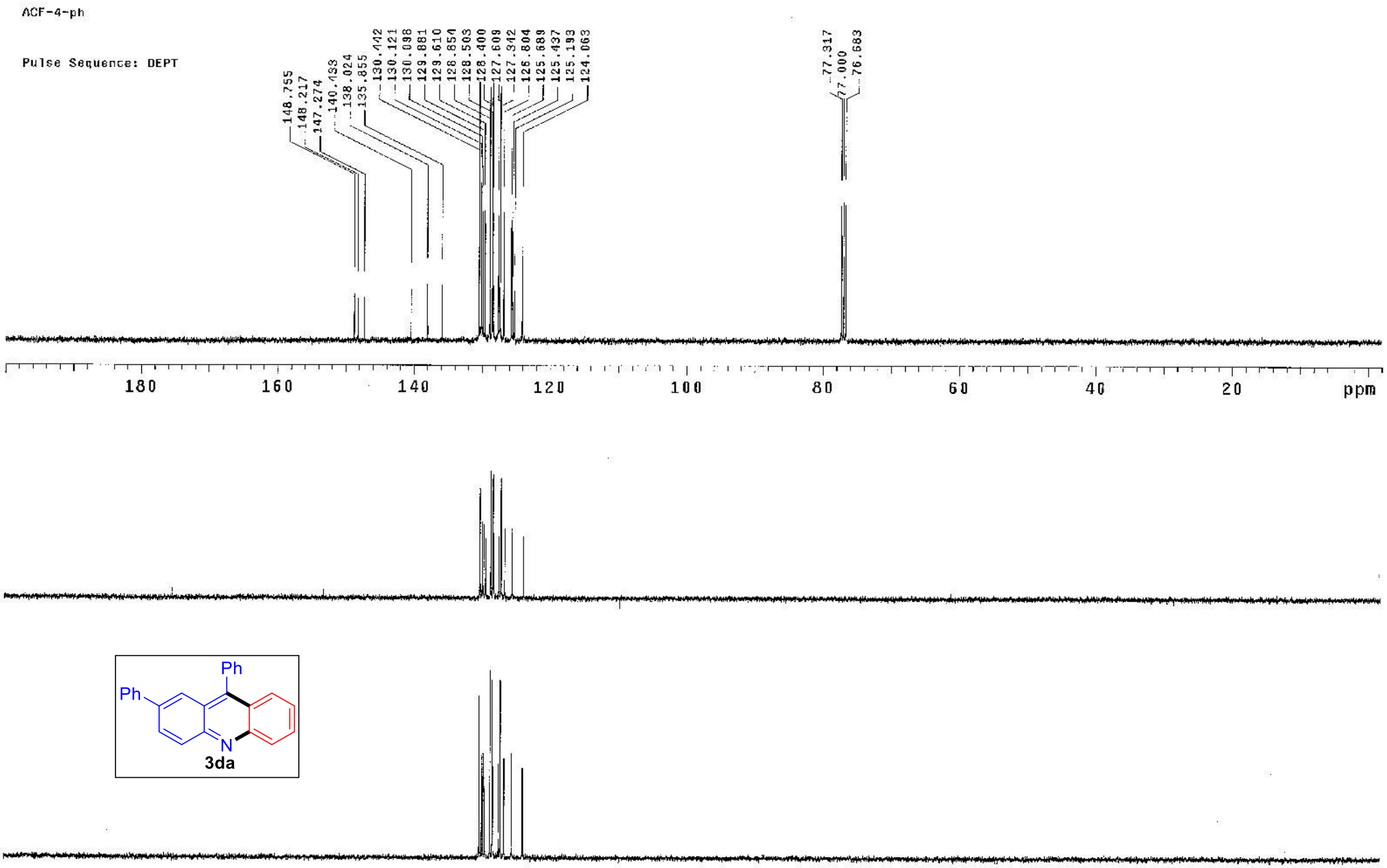
Solvent CDCl3
Spectrometer Frequency 100.69



3da

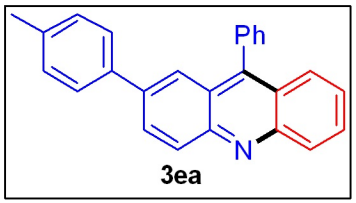


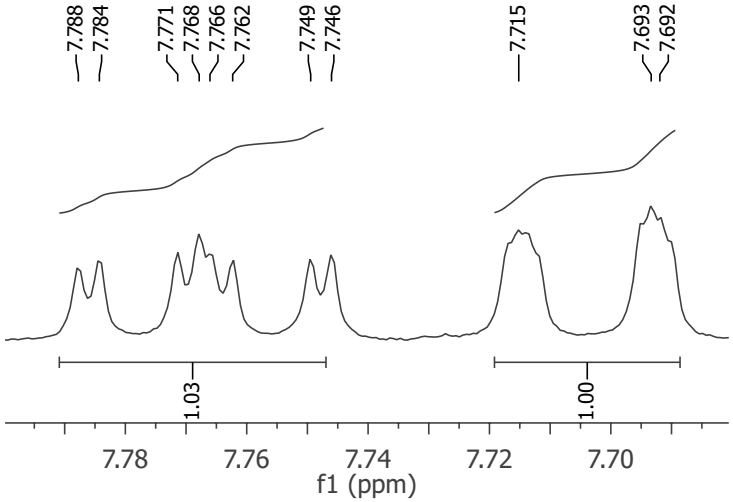


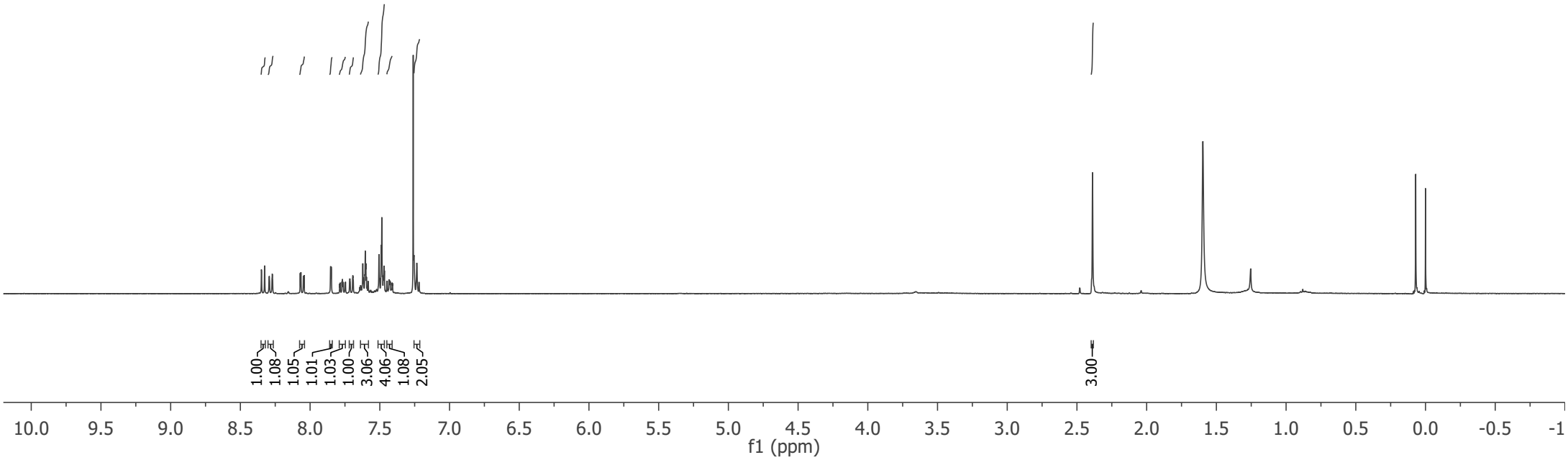

ACF-4-ph
Pulse Sequence: DEPT
148.755
148.217
147.274
140.133
138.024
135.855
130.442
130.121
130.098
129.881
129.610
128.854
128.503
128.400
127.609
127.342
126.804
125.689
125.437
125.193
124.063
77.317
77.000
76.683
180
160
140
120
100
80
60
40
20
ppm
Ph
Ph
N
3da

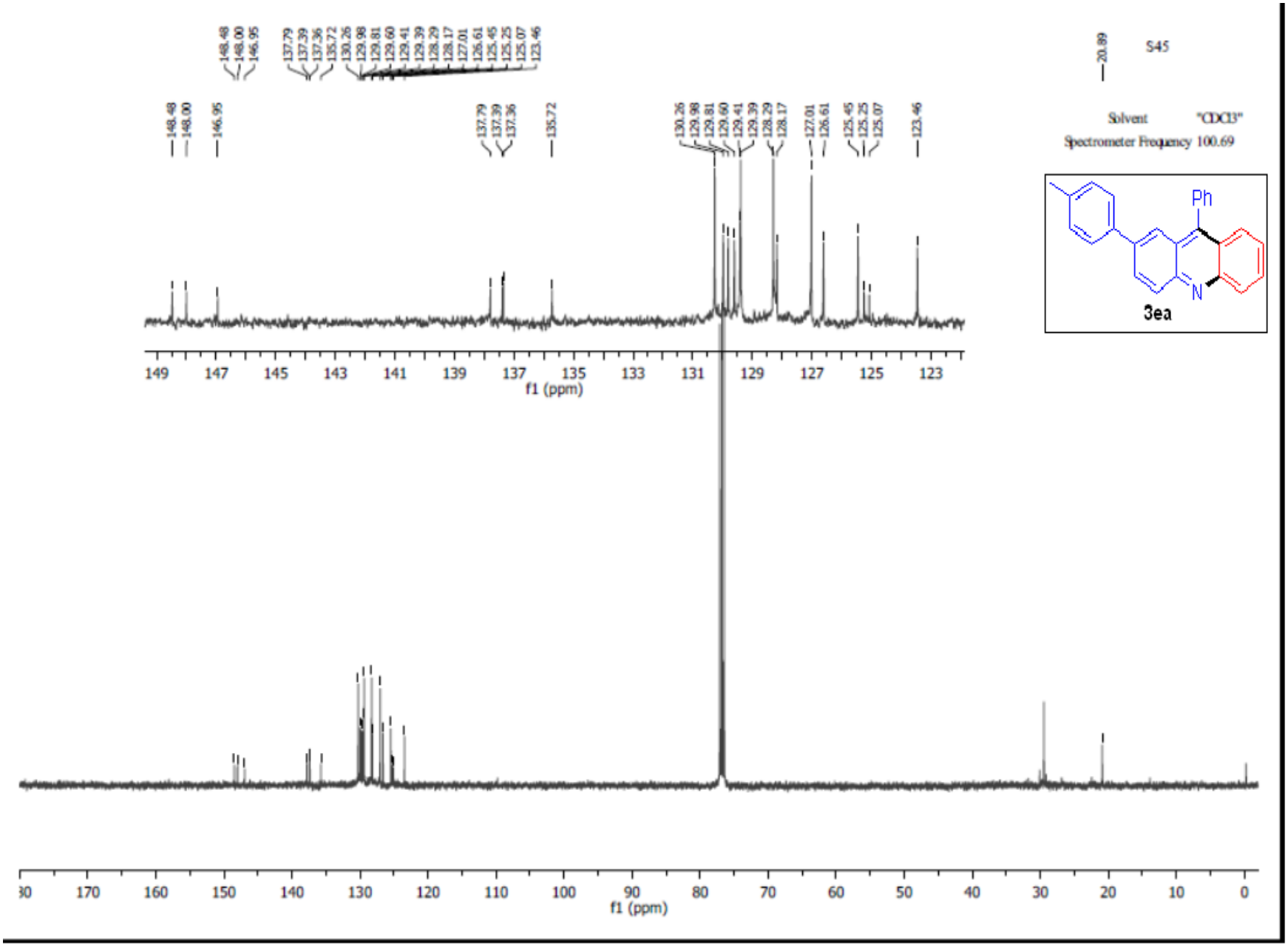
Solvent CDCl3

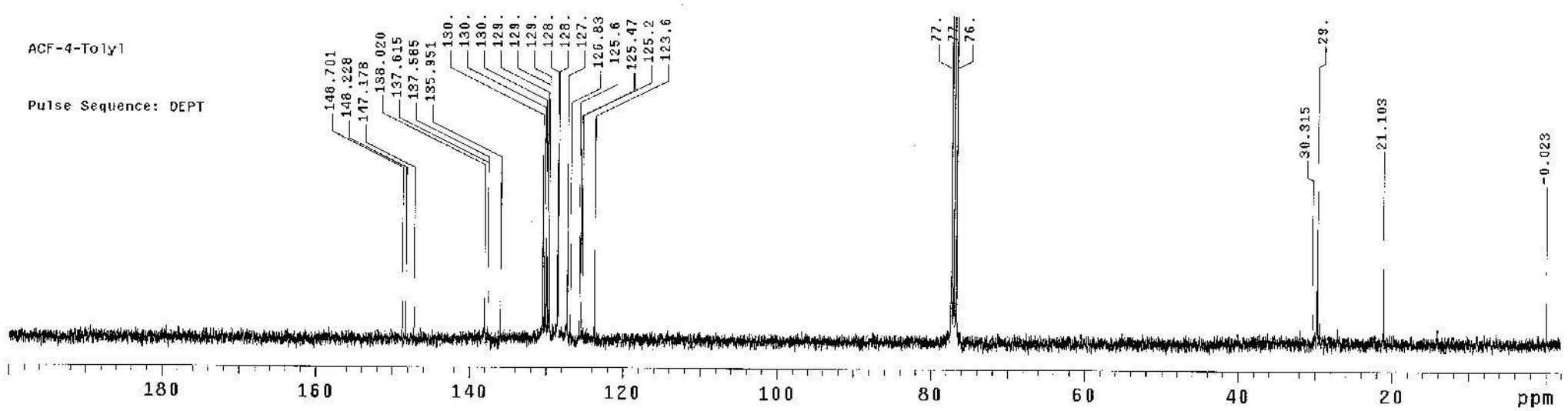
Spectrometer Frequency 400.4

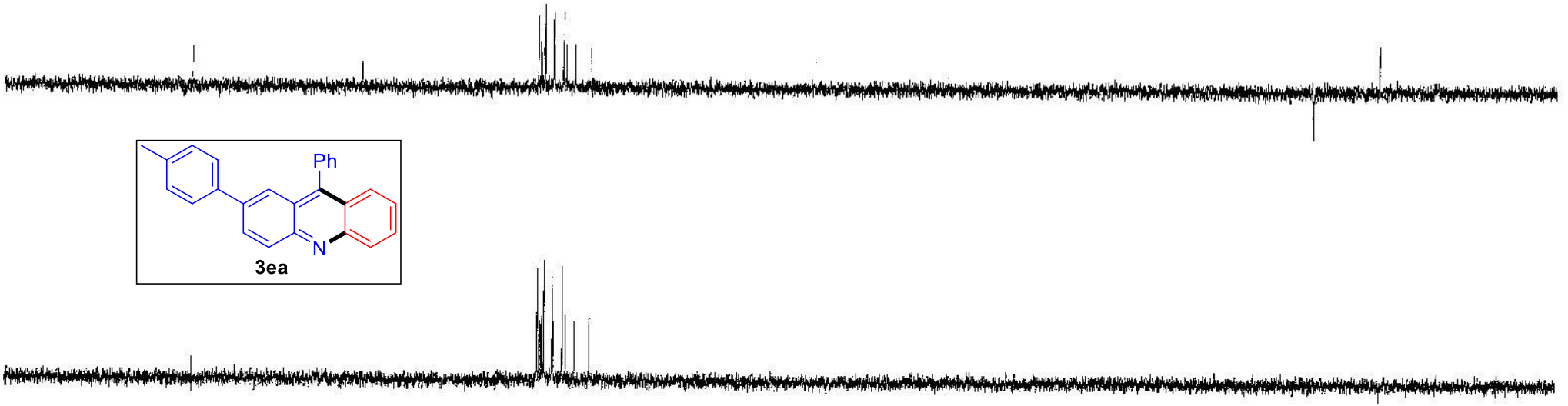






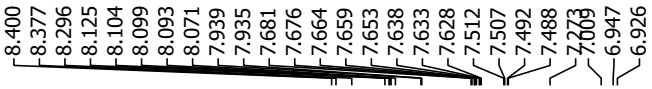

148.48
148.00
146.95
137.79
137.39
137.36
135.72
130.26
129.98
129.81
129.60
129.41
129.39
128.29
128.17
127.01
126.61
125.45
125.25
125.07
123.46
20.89
S45
Solvent "CDCl3"
Spectrometer Frequency 100.69
Ph
N
3ea
f1 (ppm)
f1 (ppm)

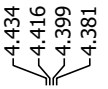

ACF-4-Tolyl
Pulse Sequence: DEPT
148.701
148.228
147.178
138.020
137.615
137.585
135.951
130.
130.
130.
129.
129.
129.
128.
128.
127.
126.83
125.6
125.47
125.2
123.6
77.
76.
30.315
29.
21.103
-0.023
180
160
140
120
100
80
60
40
20
ppm



Ph
N

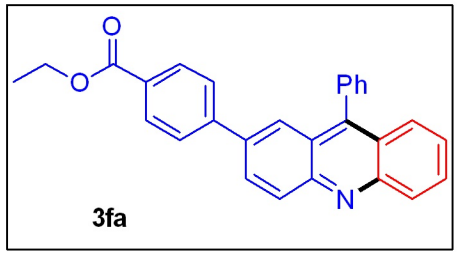
3ea

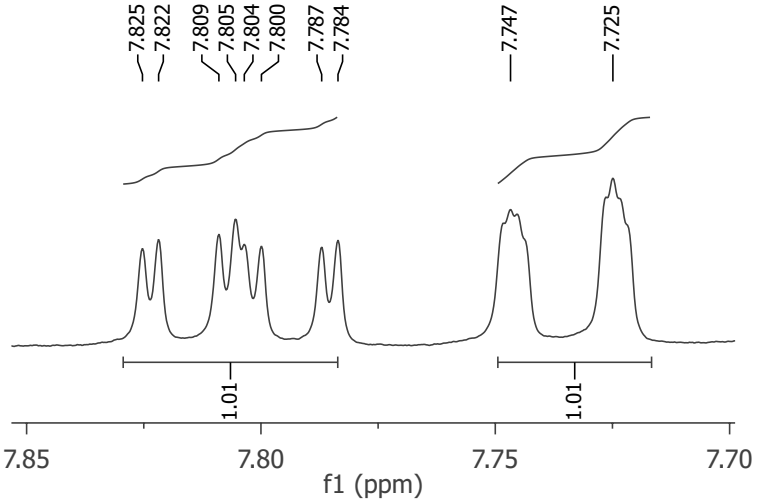


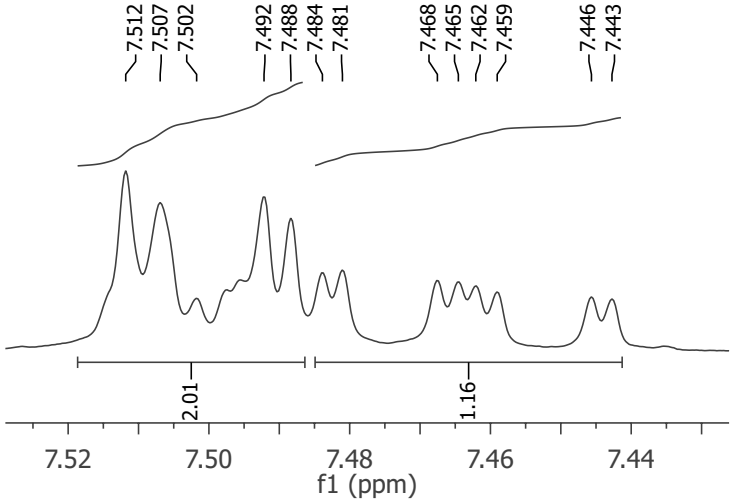


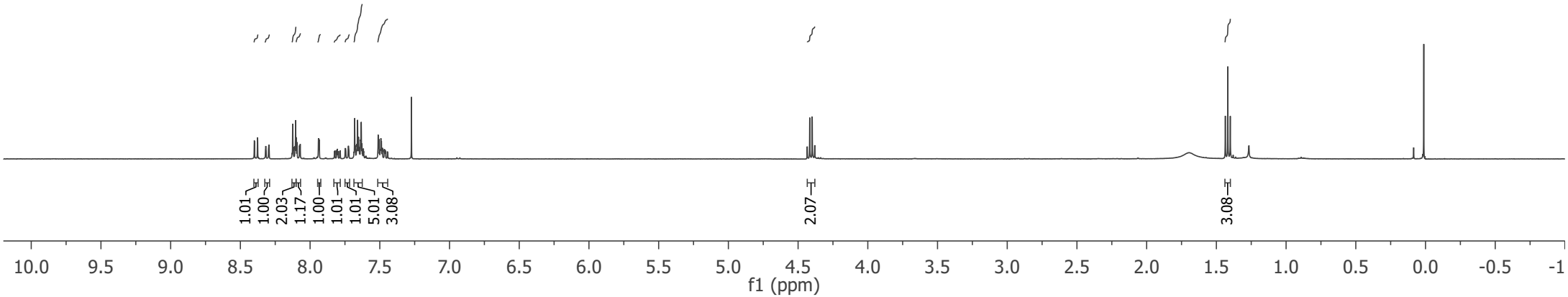
Solvent CDCl3

Spectrometer Frequency 400.40



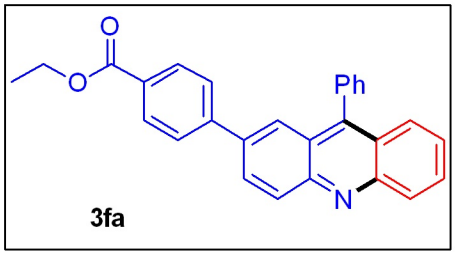


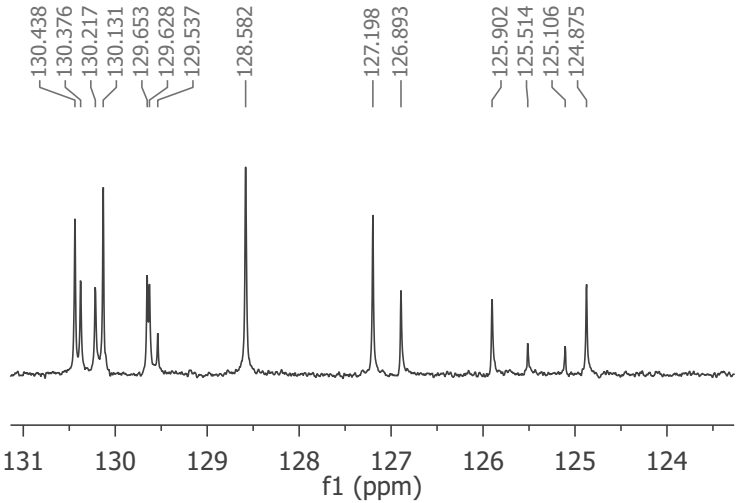


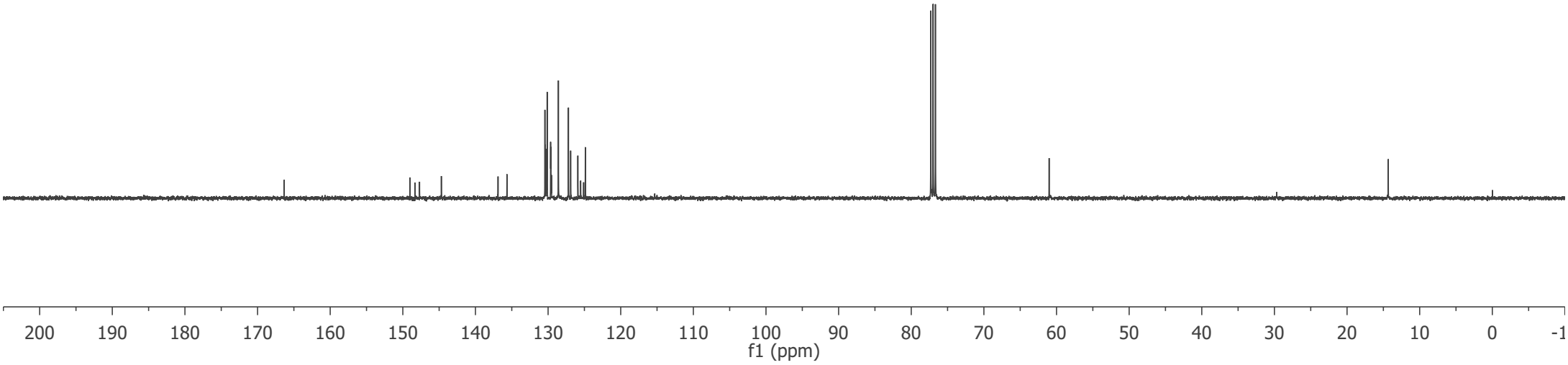


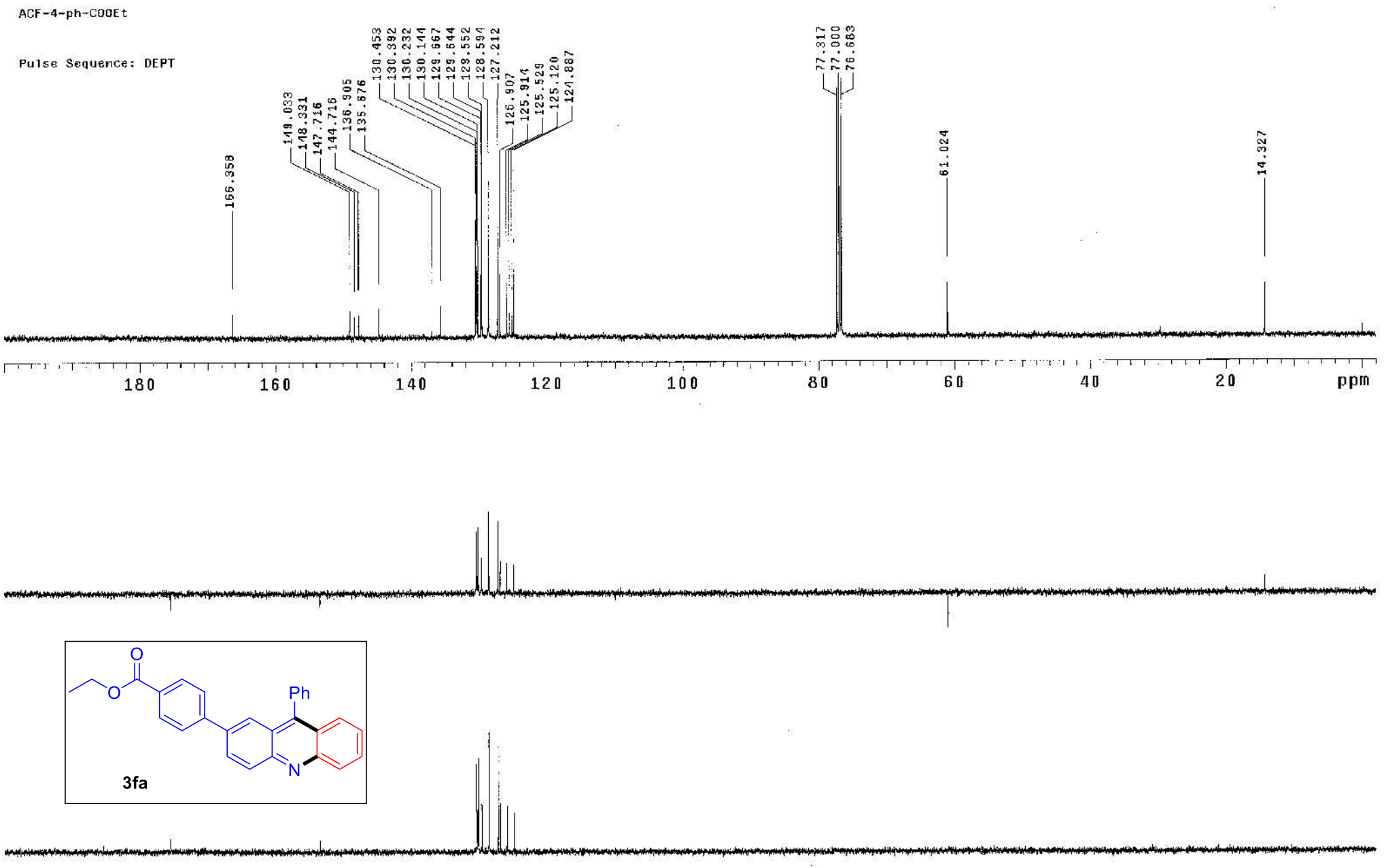
166.340
149.019
148.316
147.701
144.699
136.890
135.661
130.438
130.376
130.217
130.131
129.653
129.628
129.537
128.582
127.198
126.893
125.902
125.514
125.106
124.875
61.018
14.325

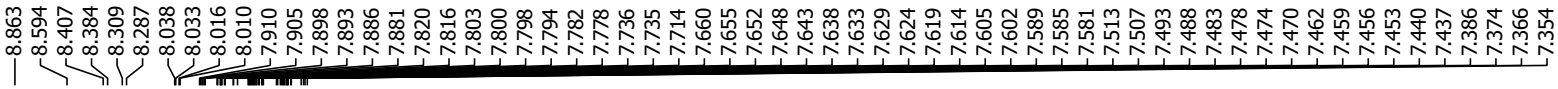
Solvent CDCl3
Spectrometer Frequency 100.69





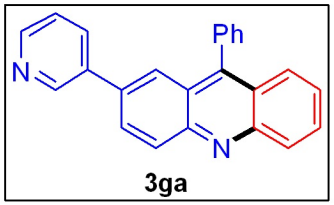



ACF-4-ph-COOEt
Pulse Sequence: DEPT
166.358
149.033
148.331
147.716
144.716
136.905
135.676
130.453
130.392
130.232
130.144
129.667
129.644
129.552
128.594
127.212
126.907
125.914
125.529
125.120
124.887
77.317
77.000
76.683
61.024
14.327
180
160
140
120
100
80
60
40
20
ppm
O
O
Ph
N
3fa

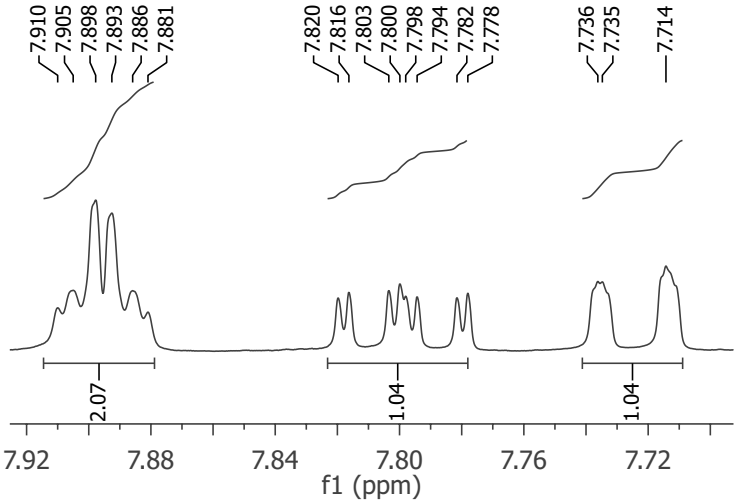


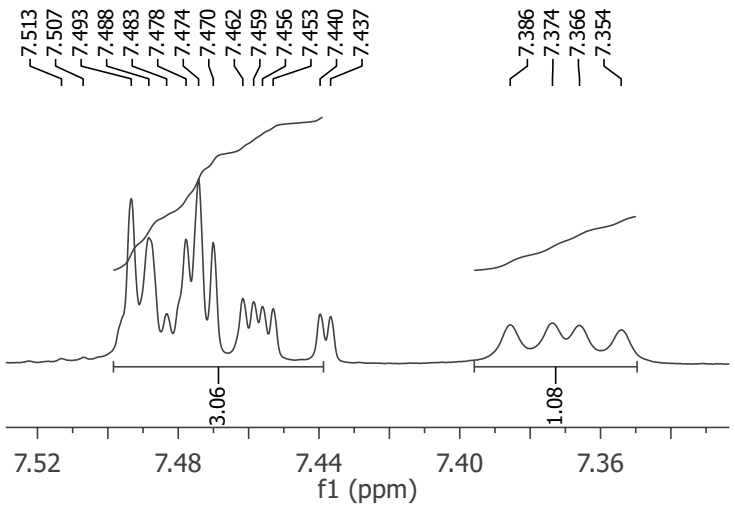
Solvent CDCl3

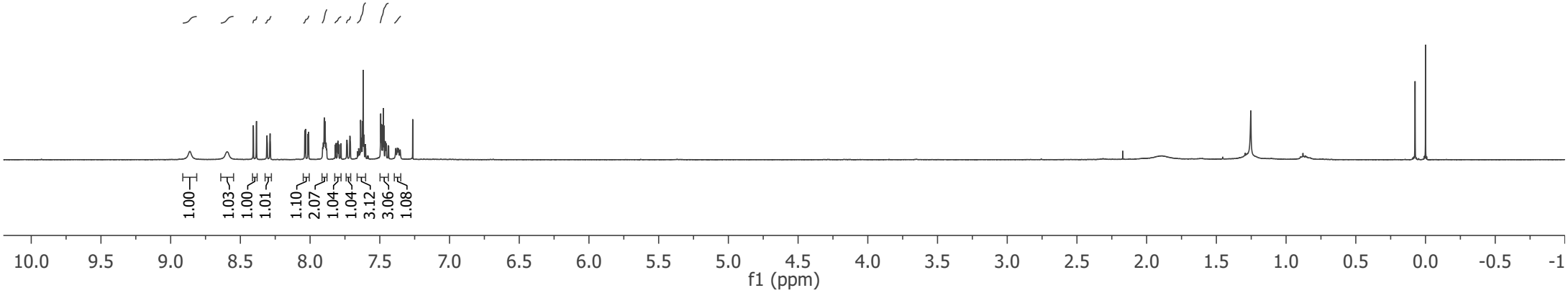
Spectrometer Frequency 400.40



3ga

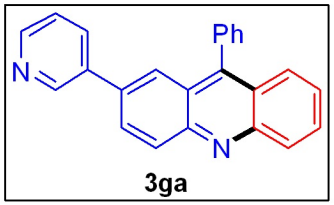




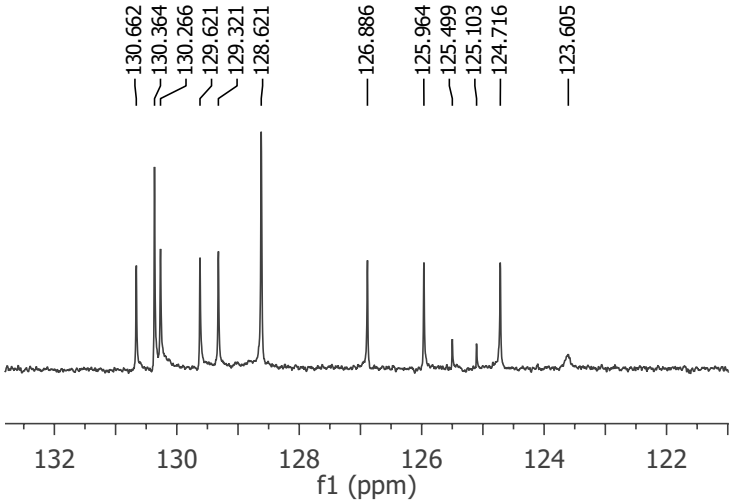


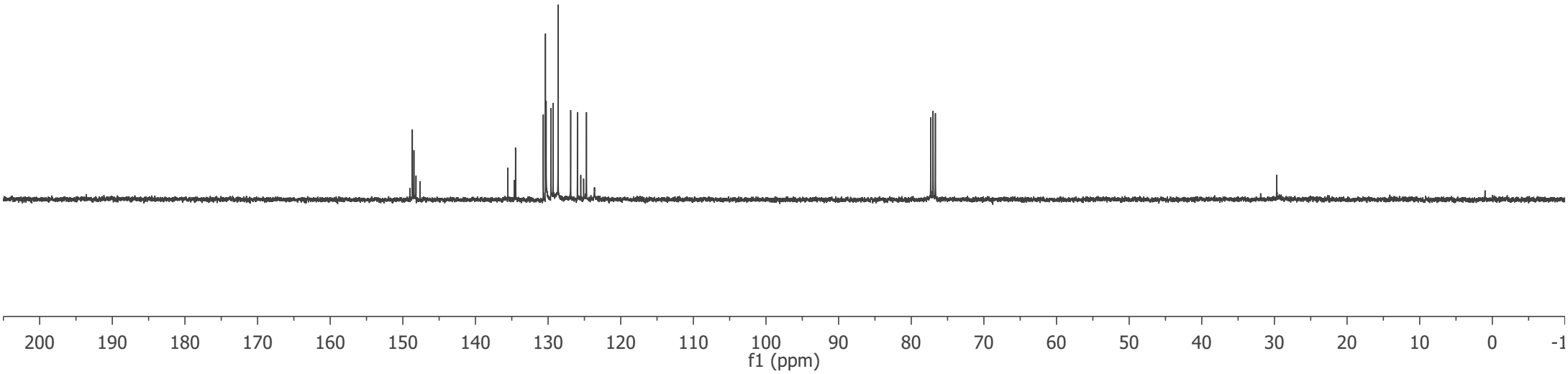
Solvent CDCl3
Spectrometer Frequency 100.69

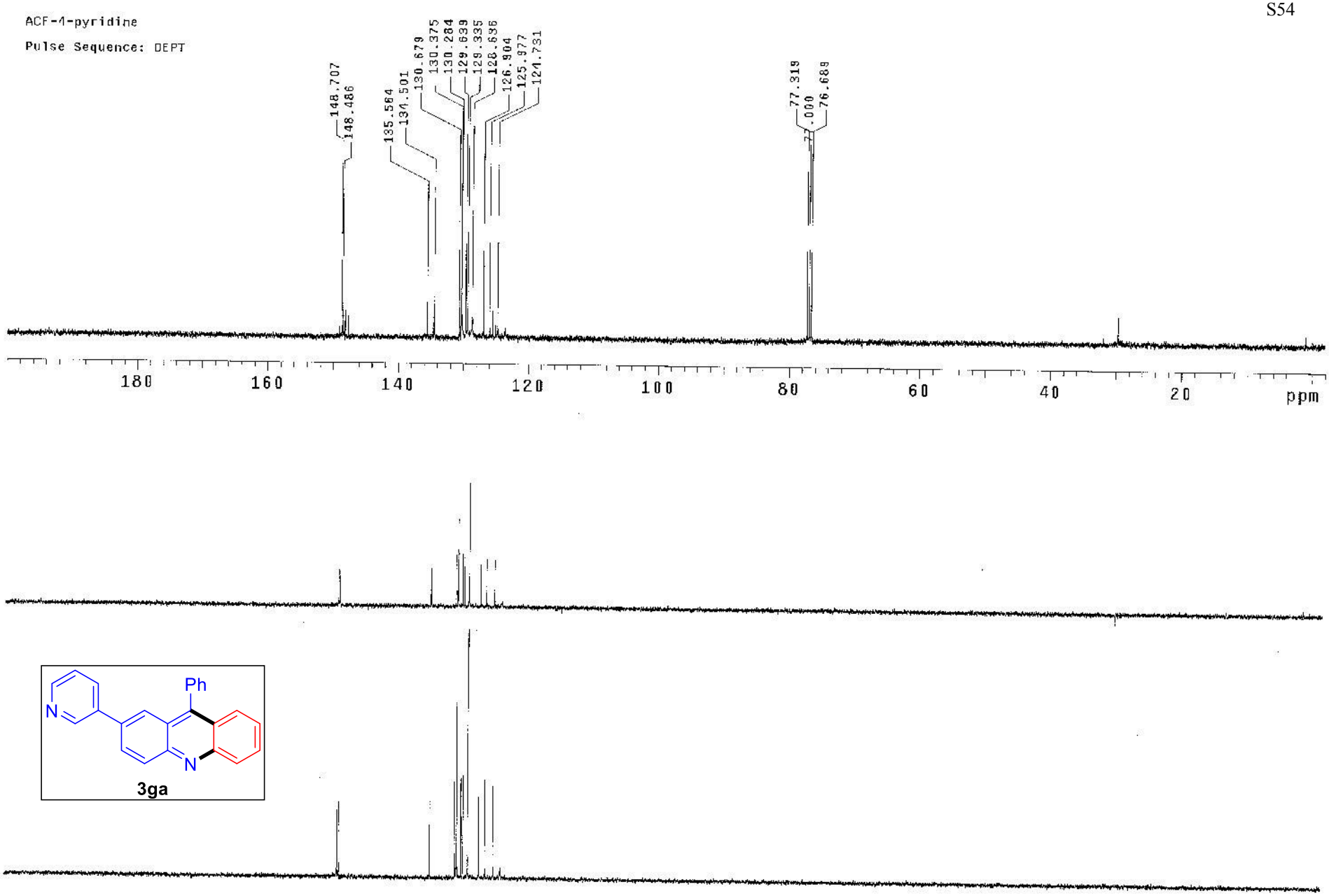
149.035
148.979
148.690
148.468
148.182
148.147
147.649
135.550
134.643
134.484
130.662
130.364
130.266
129.621
129.321
128.621
126.886
125.964
125.499
125.103
124.716
123.605

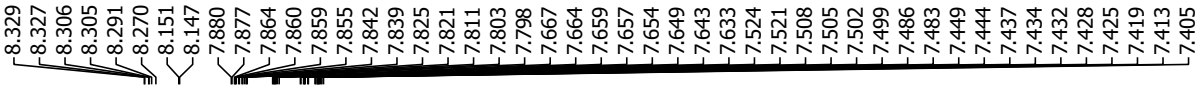


3ga



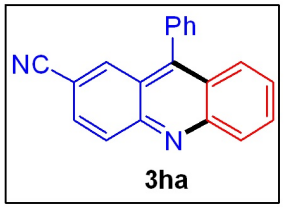


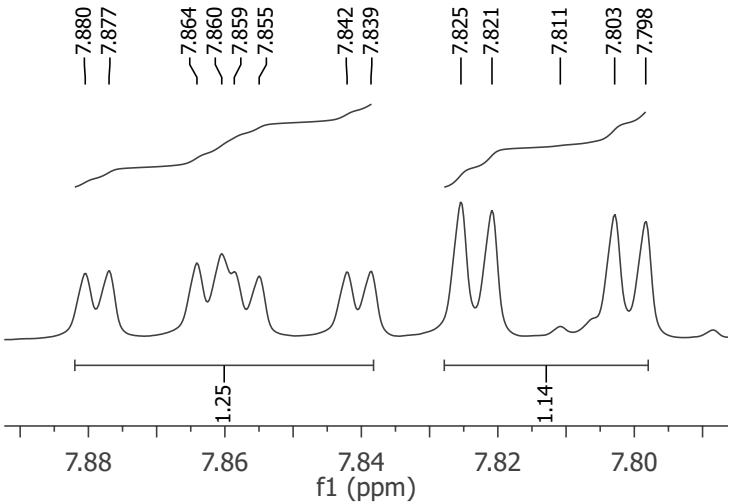

ACF-4-pyridine
Pulse Sequence: DEPT
148.707
148.486
135.564
134.501
130.679
130.375
130.284
129.639
129.335
128.836
126.904
125.977
124.731
77.319
77.000
76.689
180
160
140
120
100
80
60
40
20
ppm
Ph
N
N
3ga

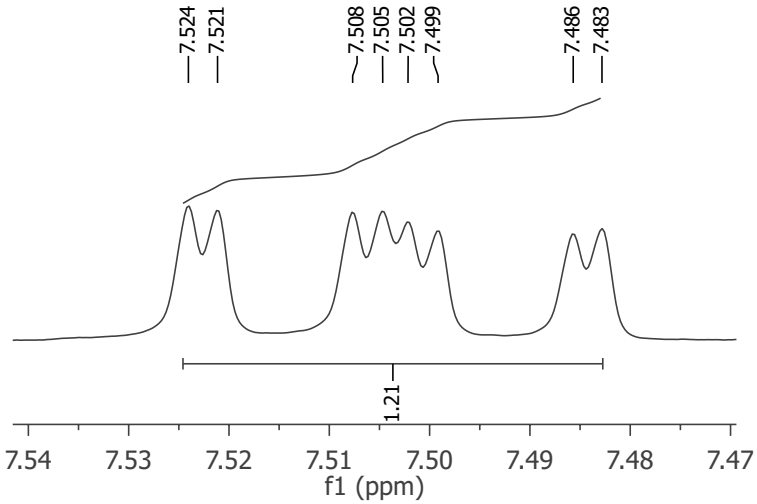


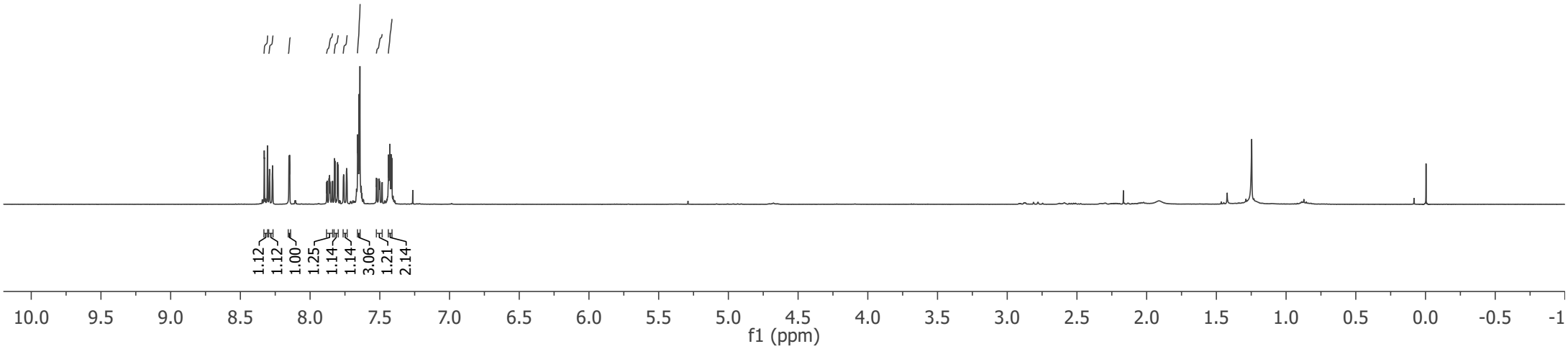
Solvent CDCl3

Spectrometer Frequency 400.28



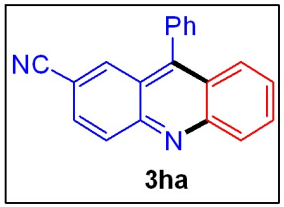




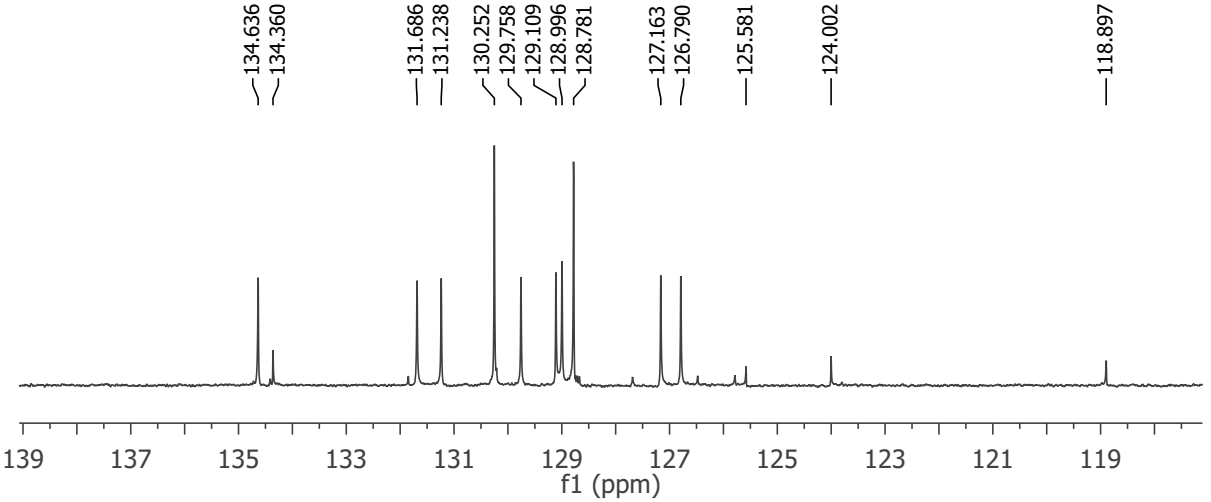


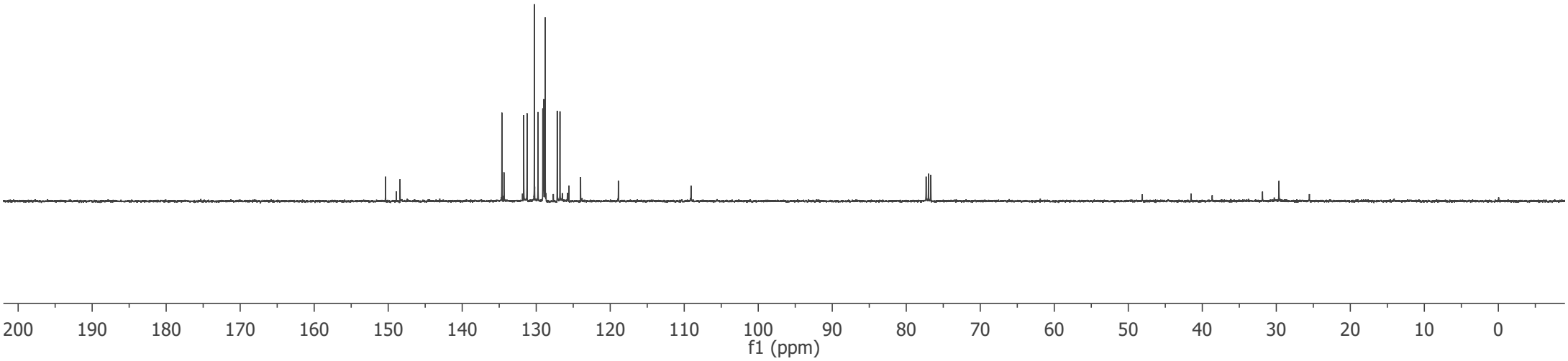
150.355
148.902
148.400
134.636
131.686
131.238
130.252
129.758
129.109
128.996
128.781
127.163
126.790
118.897
109.084

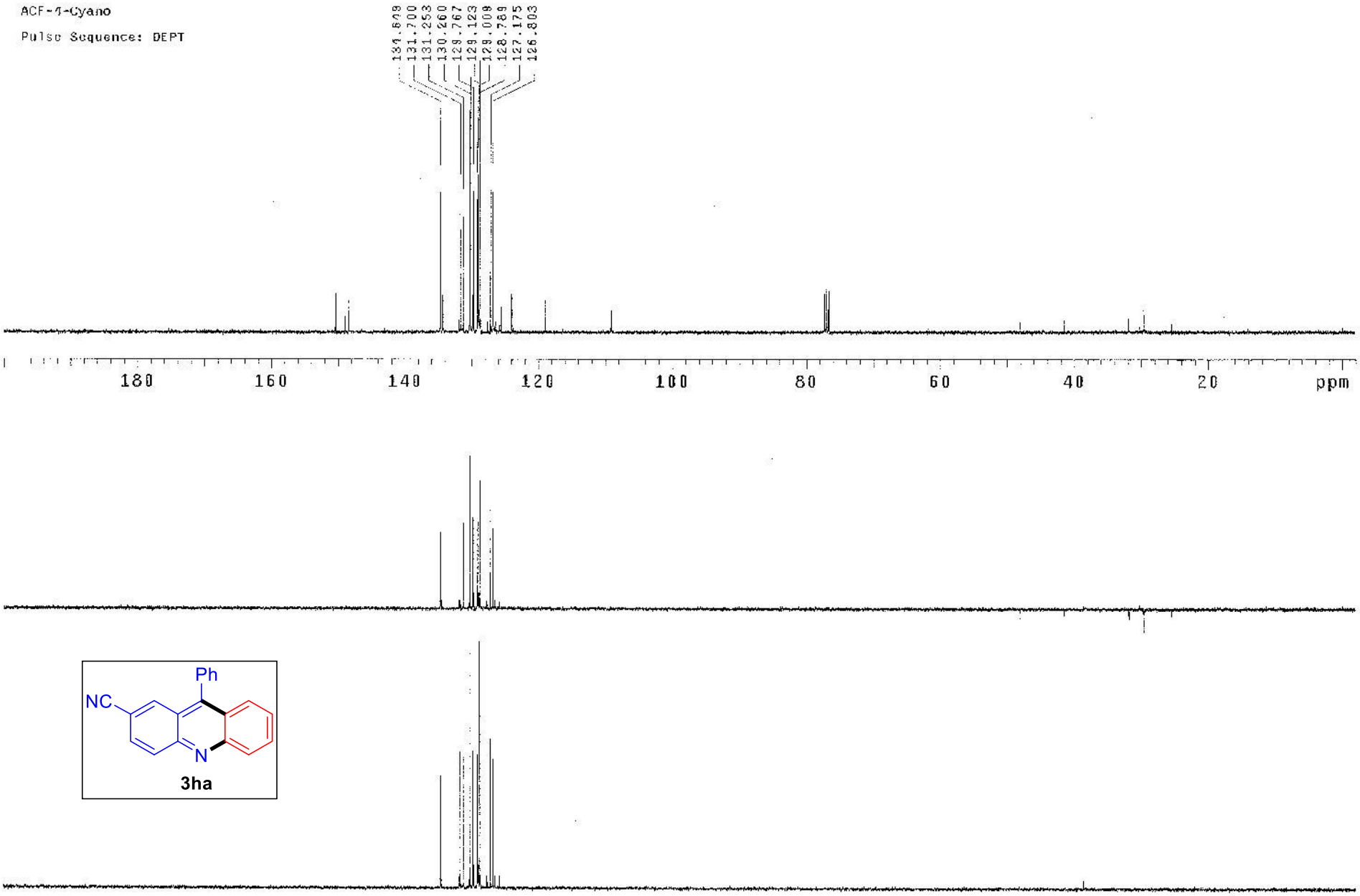
Solvent CDCl3
Spectrometer Frequency 100.66



3ha

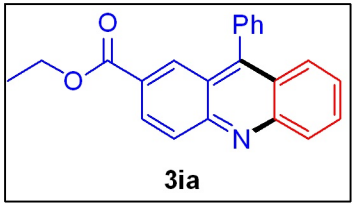




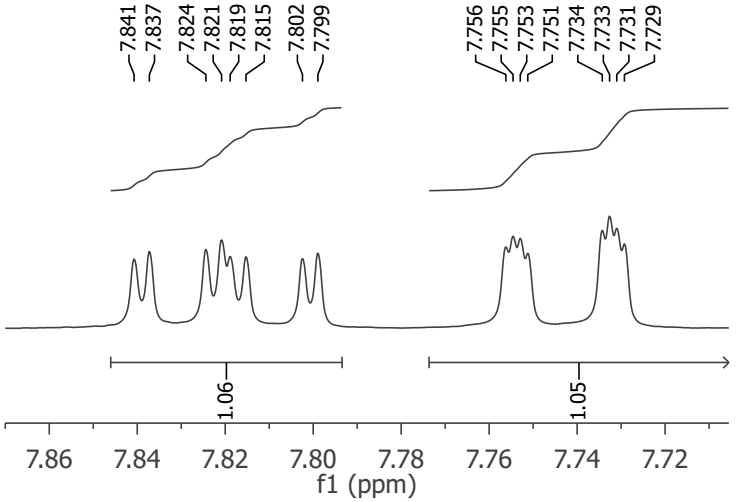

ACF-4-Cyano
Pulse Sequence: DEPT
134.649
131.700
131.253
130.260
129.767
129.123
129.009
128.789
127.175
126.803
180
160
140
120
100
80
60
40
20
ppm
Ph
NC
N
3ha

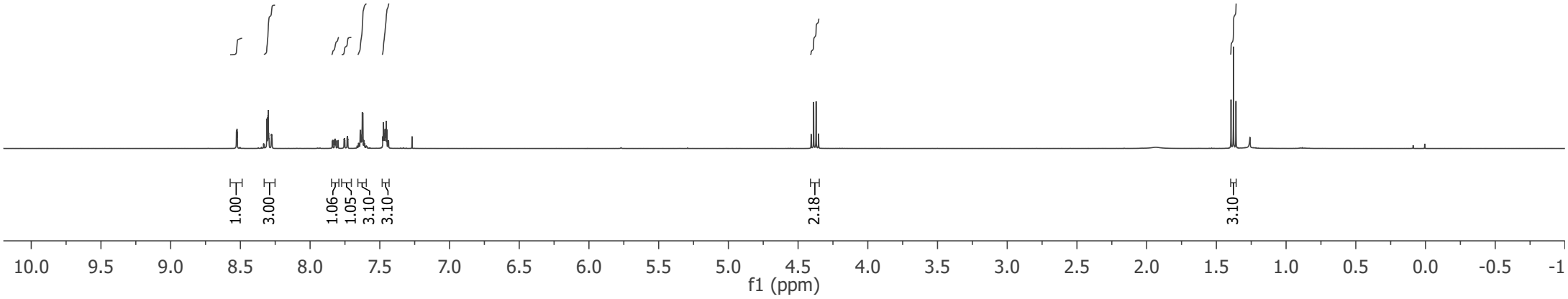
8.528 8.526 8.524 8.522 8.333 8.329 8.310 8.306 8.302 8.300 8.297 8.295 8.277 8.275 8.273
7.837 7.824 7.821 7.799 7.756 7.755 7.753 7.734 7.733 7.731 7.729 7.640 7.638 7.635 7.632 7.625 7.622 7.614 7.478 7.473 7.467 7.462 7.459 7.454 7.450 7.406 4.389 4.371 4.353
1.395 1.377 1.359

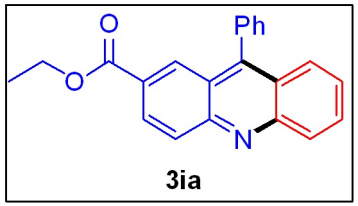
Solvent CDCl3
Spectrometer Frequency 400.40



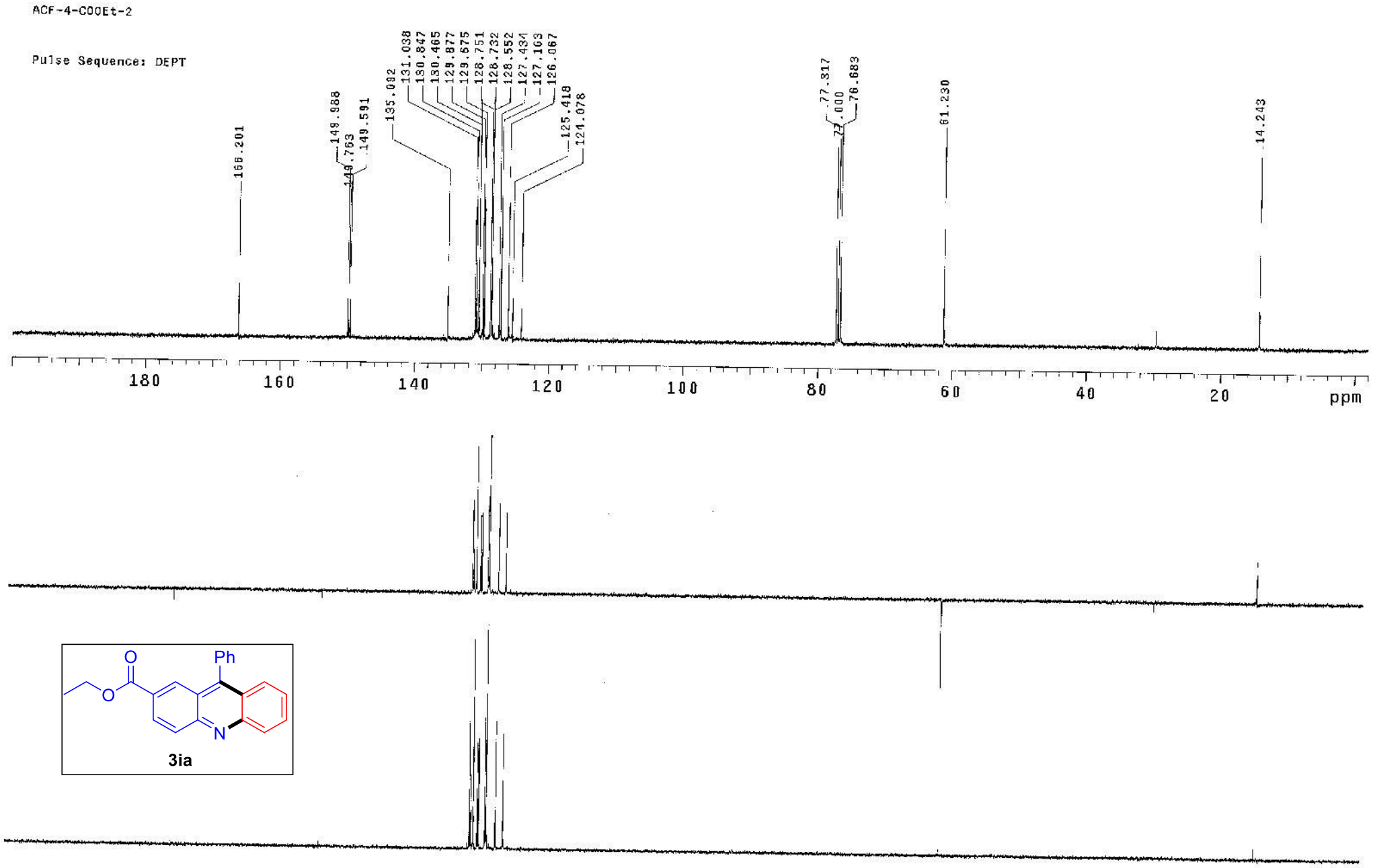
3ia







3ia

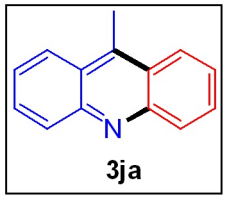

ACF-4-COOEt-2
Pulse Sequence: DEPT
166.201
149.988
149.763
149.591
135.092
131.038
130.847
130.465
129.877
129.675
128.751
128.732
128.552
127.431
127.163
126.067
125.418
124.078
77.317
77.000
76.683
61.230
14.243
180
160
140
120
100
80
60
40
20
ppm
O
O
Ph
N
3ia

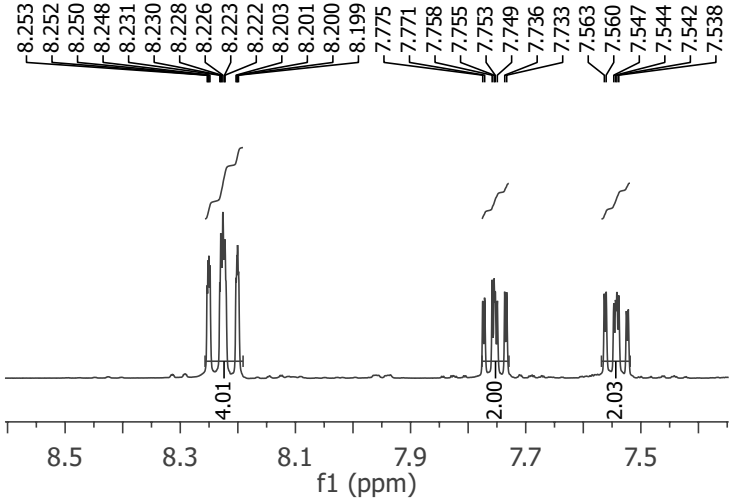
8.253 8.252 8.250 8.248 8.231 8.230 8.228 8.226 8.223 8.222 8.203 8.201 8.200 8.199 7.775 7.771 7.758 7.755 7.753 7.749 7.736 7.733 7.563 7.560 7.547 7.544 7.542 7.538 7.525 7.522

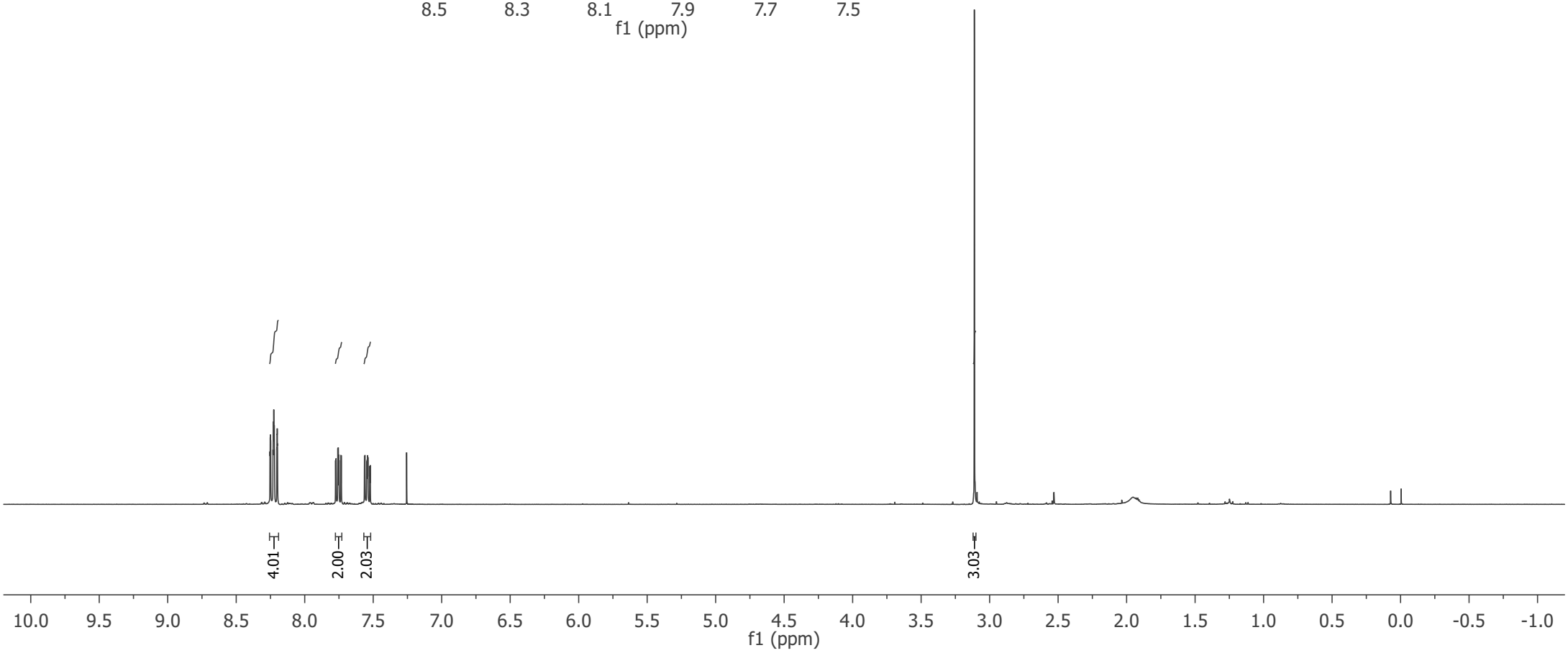
3.111

Solvent CDCl3

Spectrometer Frequency 399.78



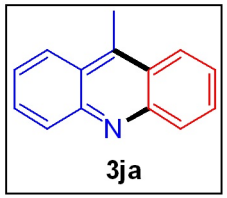




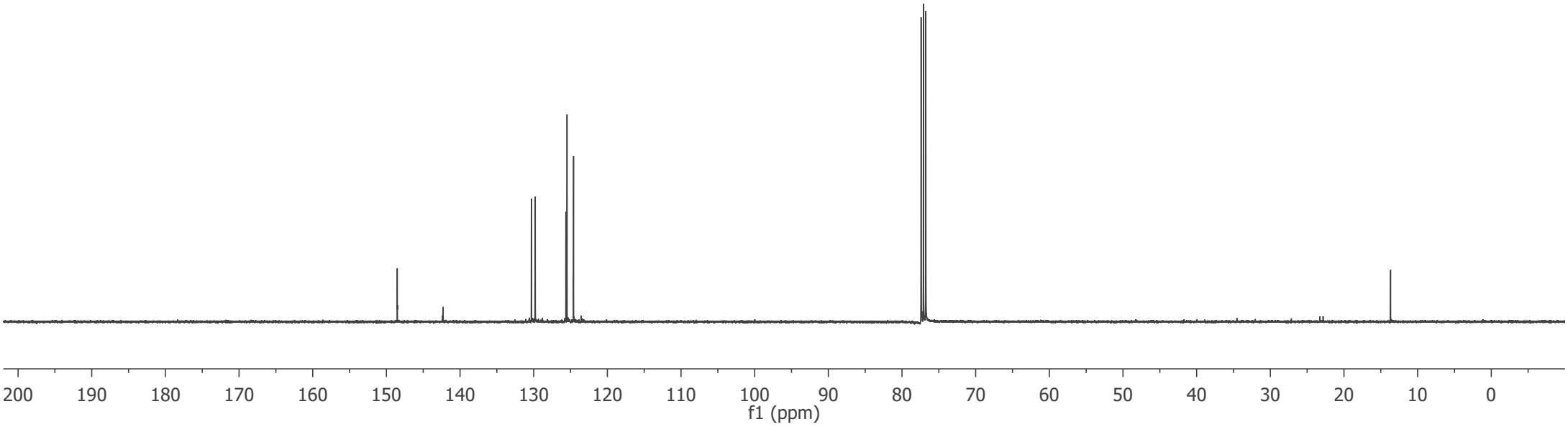
148.523
130.317
130.288
129.830
129.786
125.635
125.473
124.591
13.671

Solvent CDCl3

Spectrometer Frequency 100.53

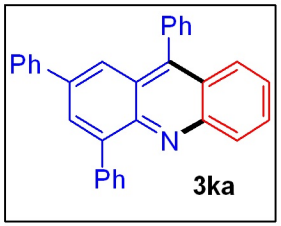


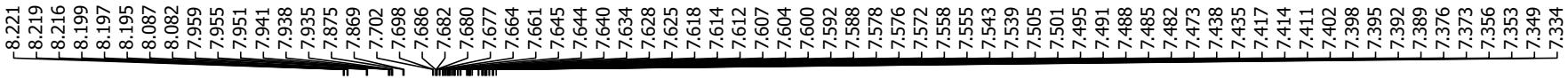
3ja

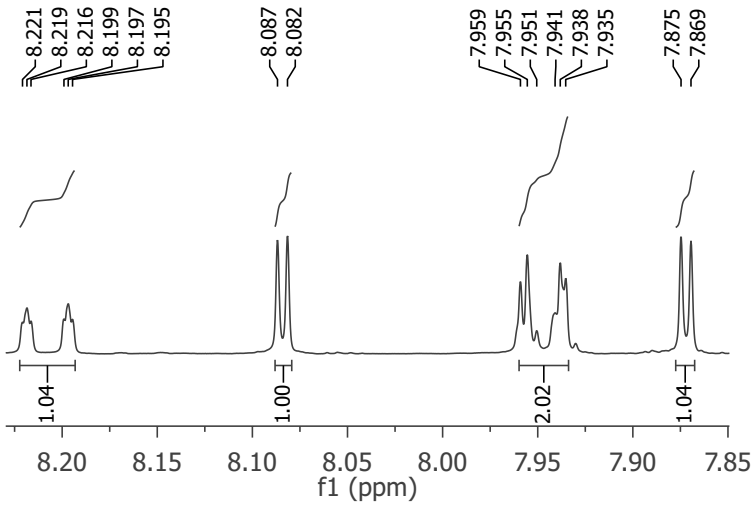


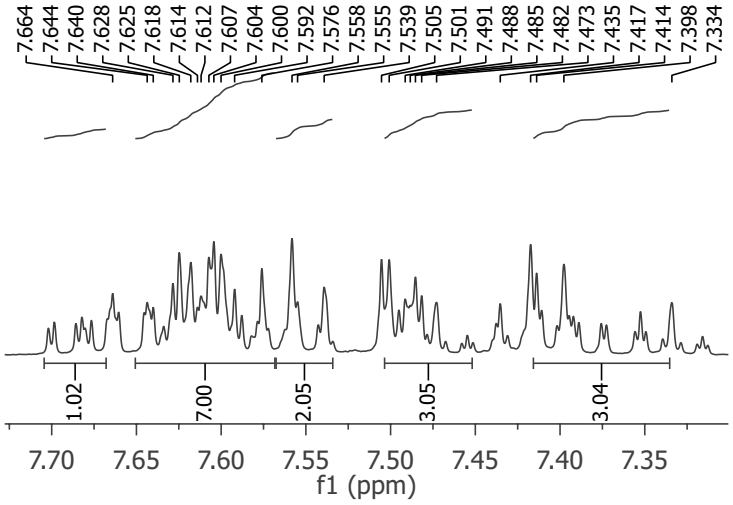
Solvent CDCl3

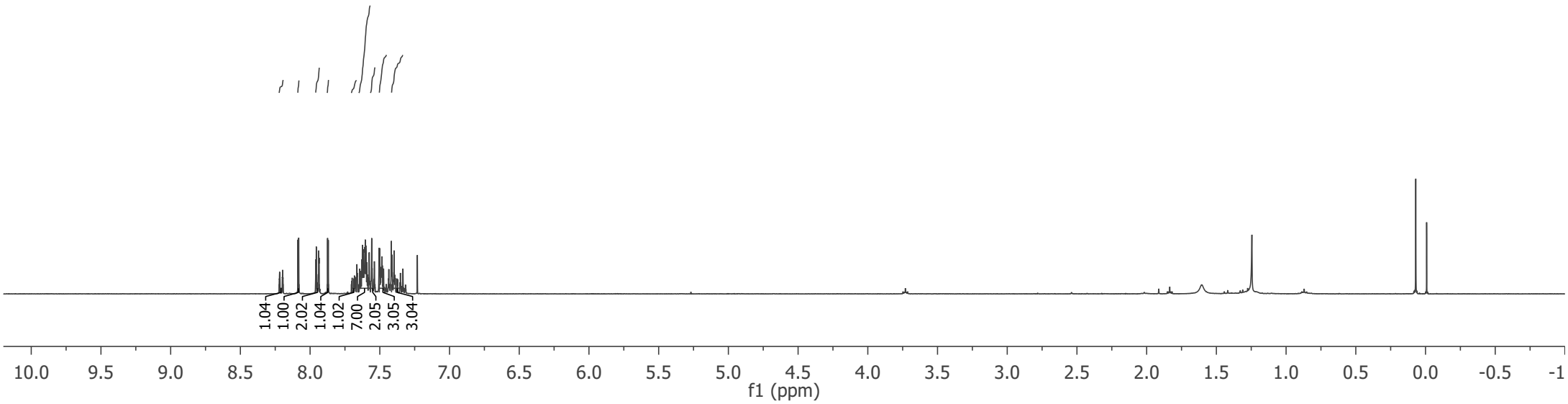
Spectrometer Frequency 400.40

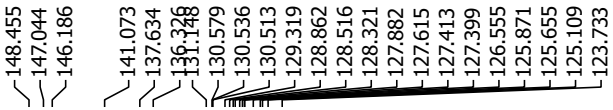






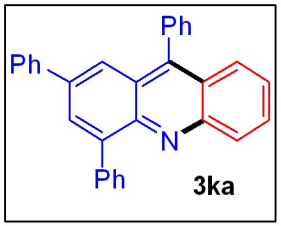


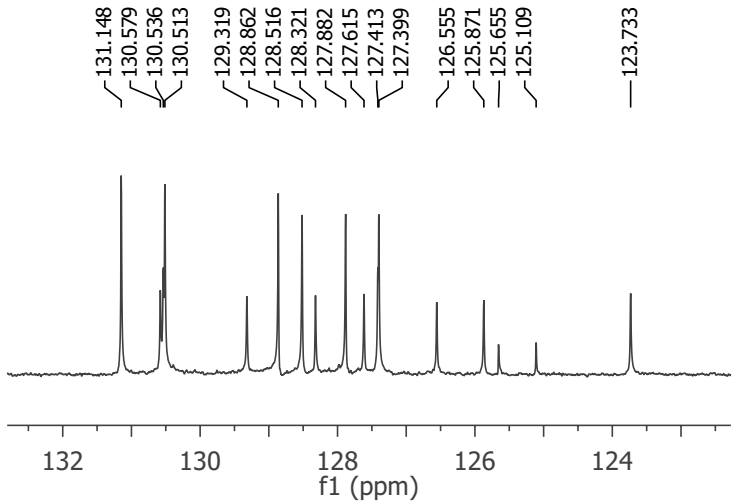


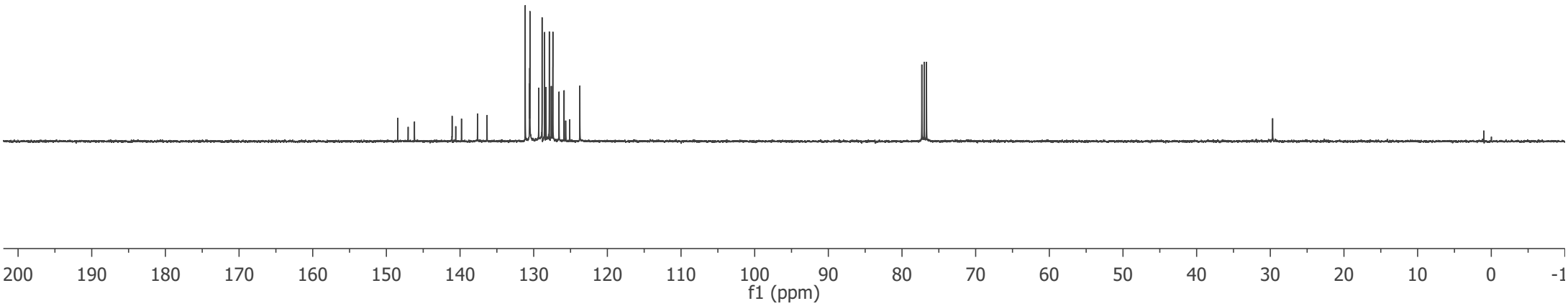


Solvent CDCl3

Spectrometer Frequency 100.69

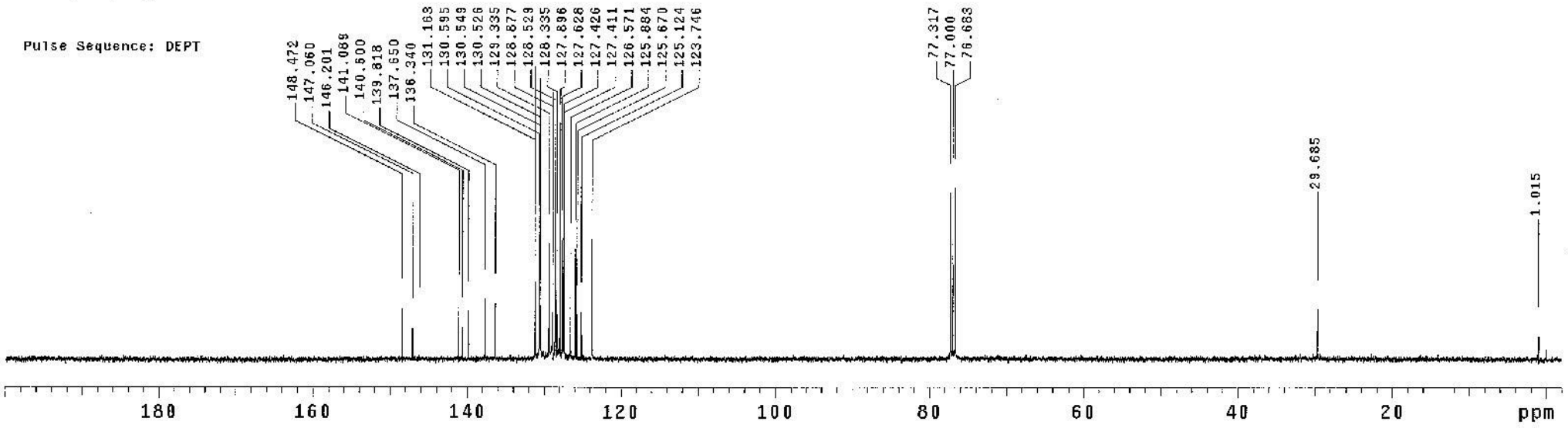


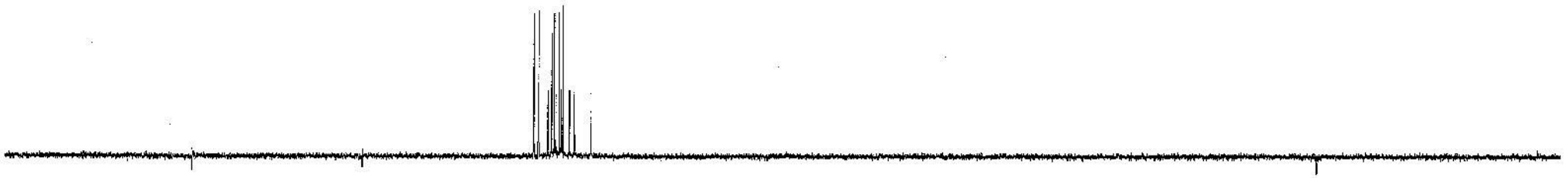


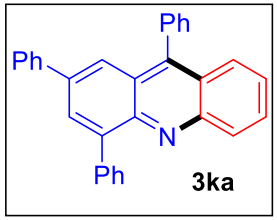


ACF-2,4-Diphenyl

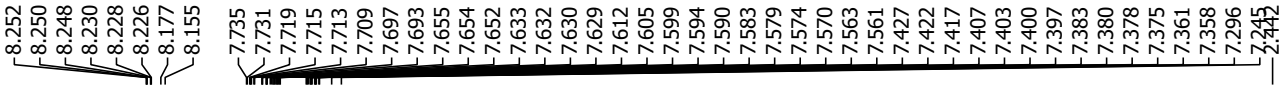
Pulse Sequence: DEPT





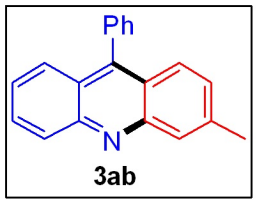


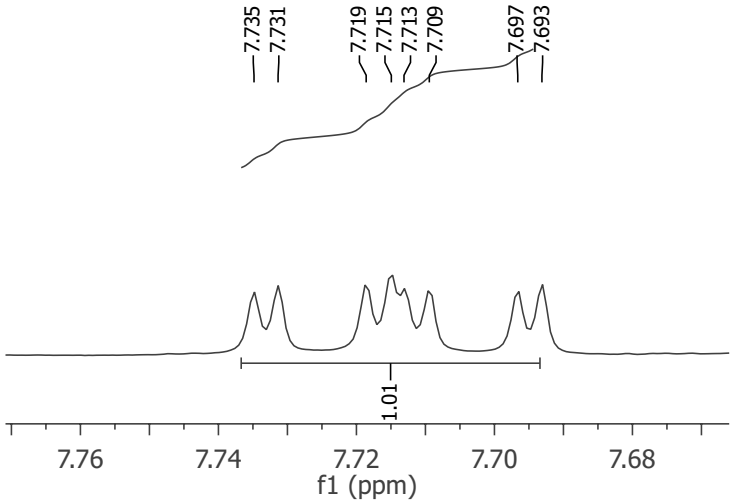
3ka

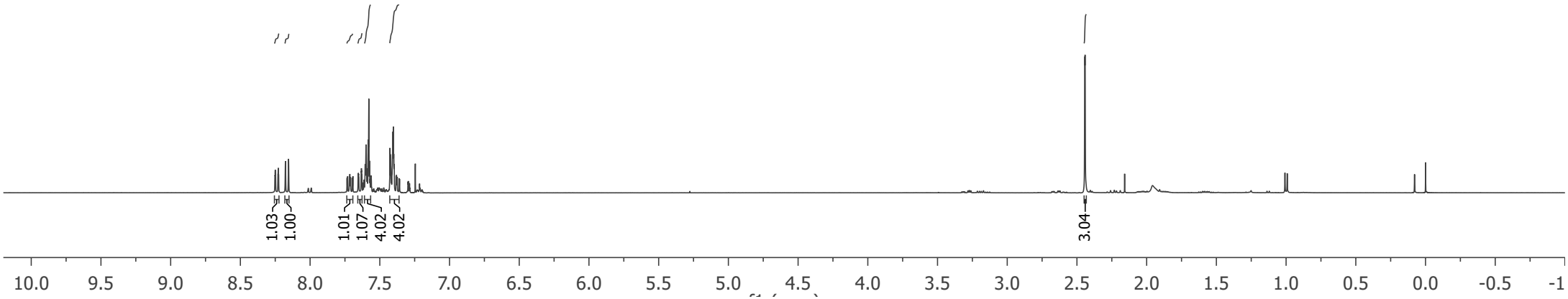


Solvent CDCl3

Spectrometer Frequency 400.40



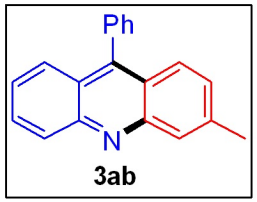




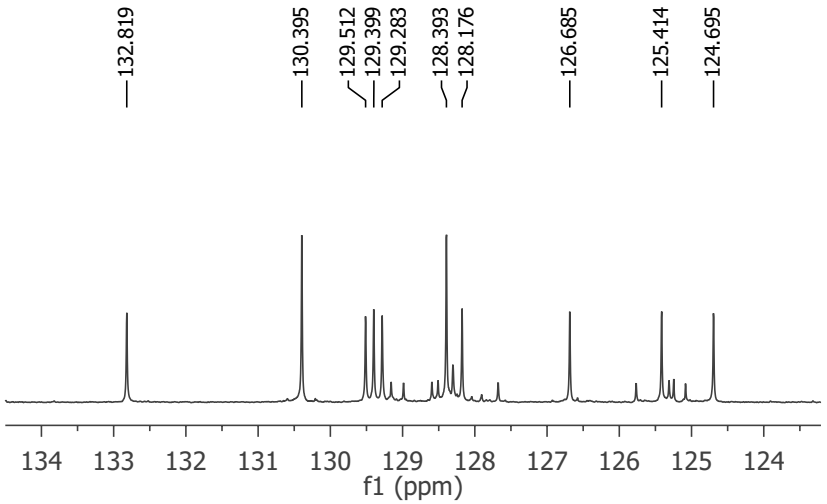
Solvent CDCl3
Spectrometer Frequency 100.66

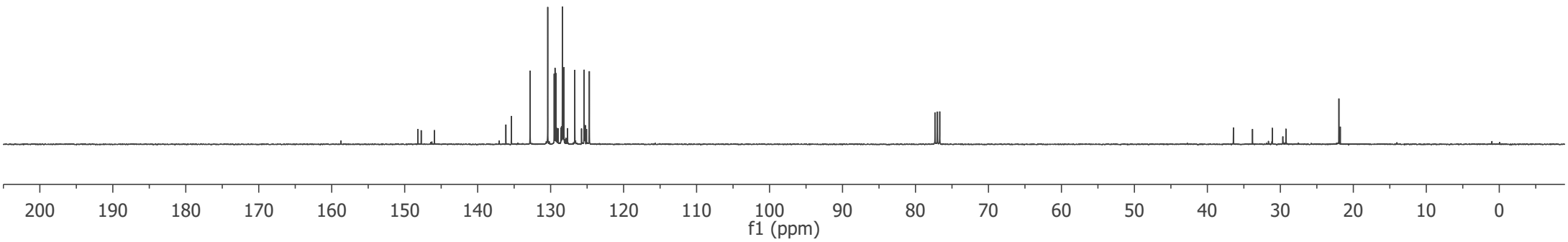
148.189
147.737
145.926
136.130
135.387
132.819
130.395
129.512
129.399
129.283
128.393
128.176
126.685
125.414
124.695

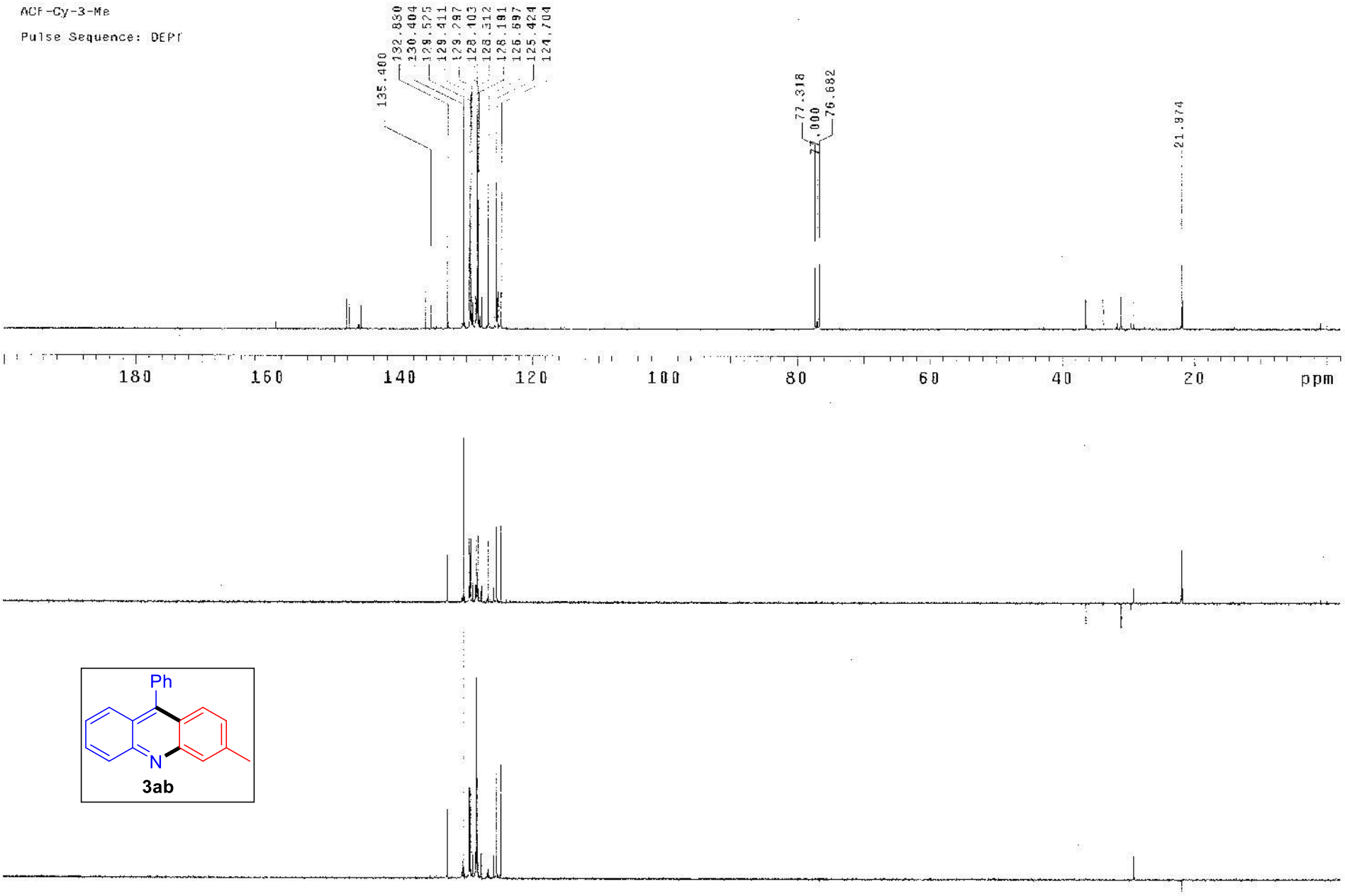
21.974



3ab

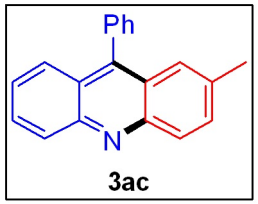


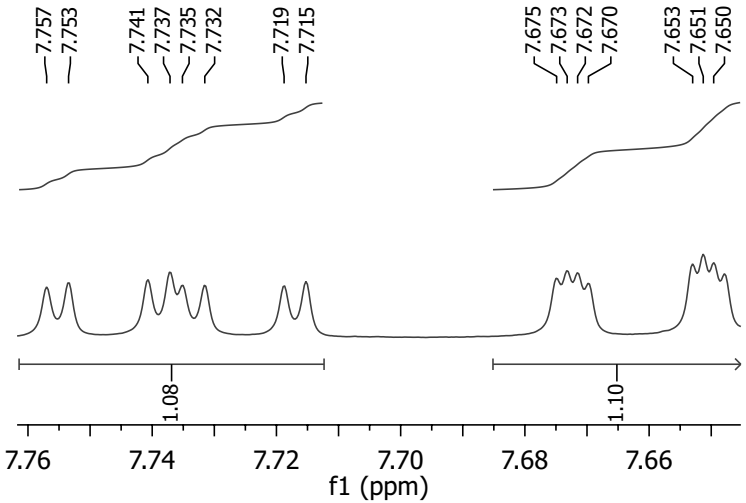


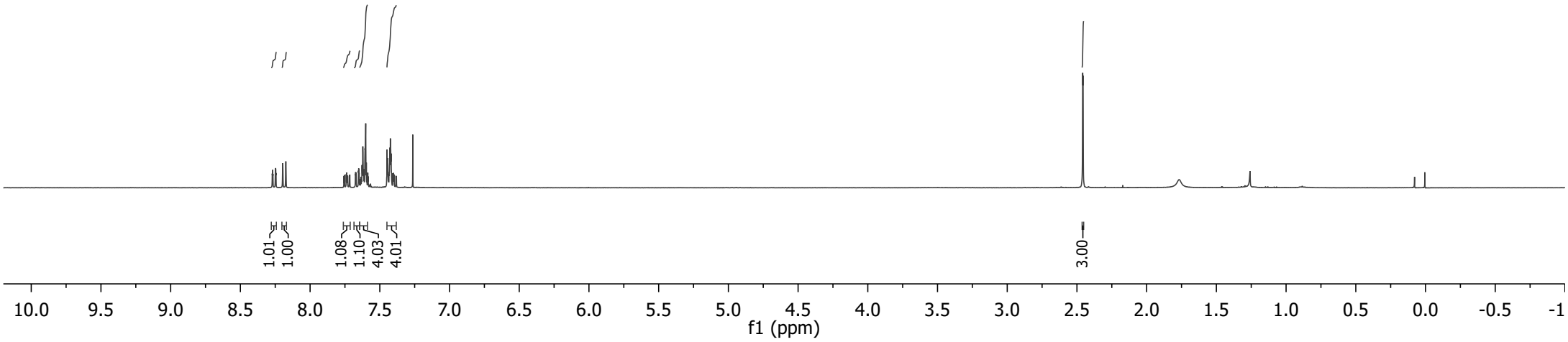

ACF-Cy-3-Me
Pulse Sequence: DEPT
135.400
132.850
130.404
129.525
129.411
129.297
128.103
128.312
128.191
126.697
125.424
124.704
77.318
77.000
76.682
21.974
180
160
140
120
100
80
60
40
20
ppm
Ph
N
3ab

Solvent CDCL3

Spectrometer Frequency 400.40



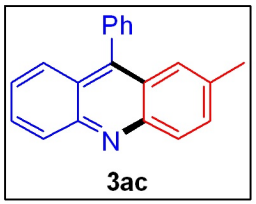




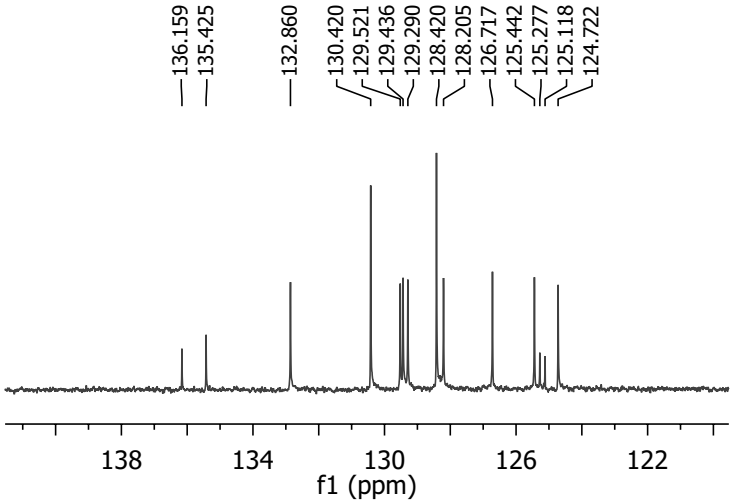
Solvent cdcl3
Spectrometer Frequency 100.69

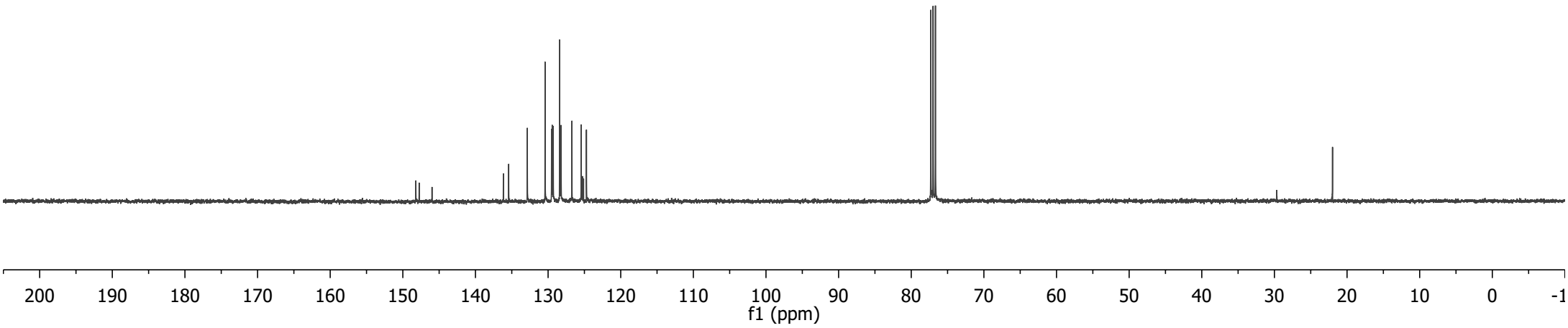
148.205
147.754
145.982
136.159
135.425
132.860
130.420
129.521
129.436
129.290
128.420
128.205
126.717
125.442
125.277
125.118
124.722

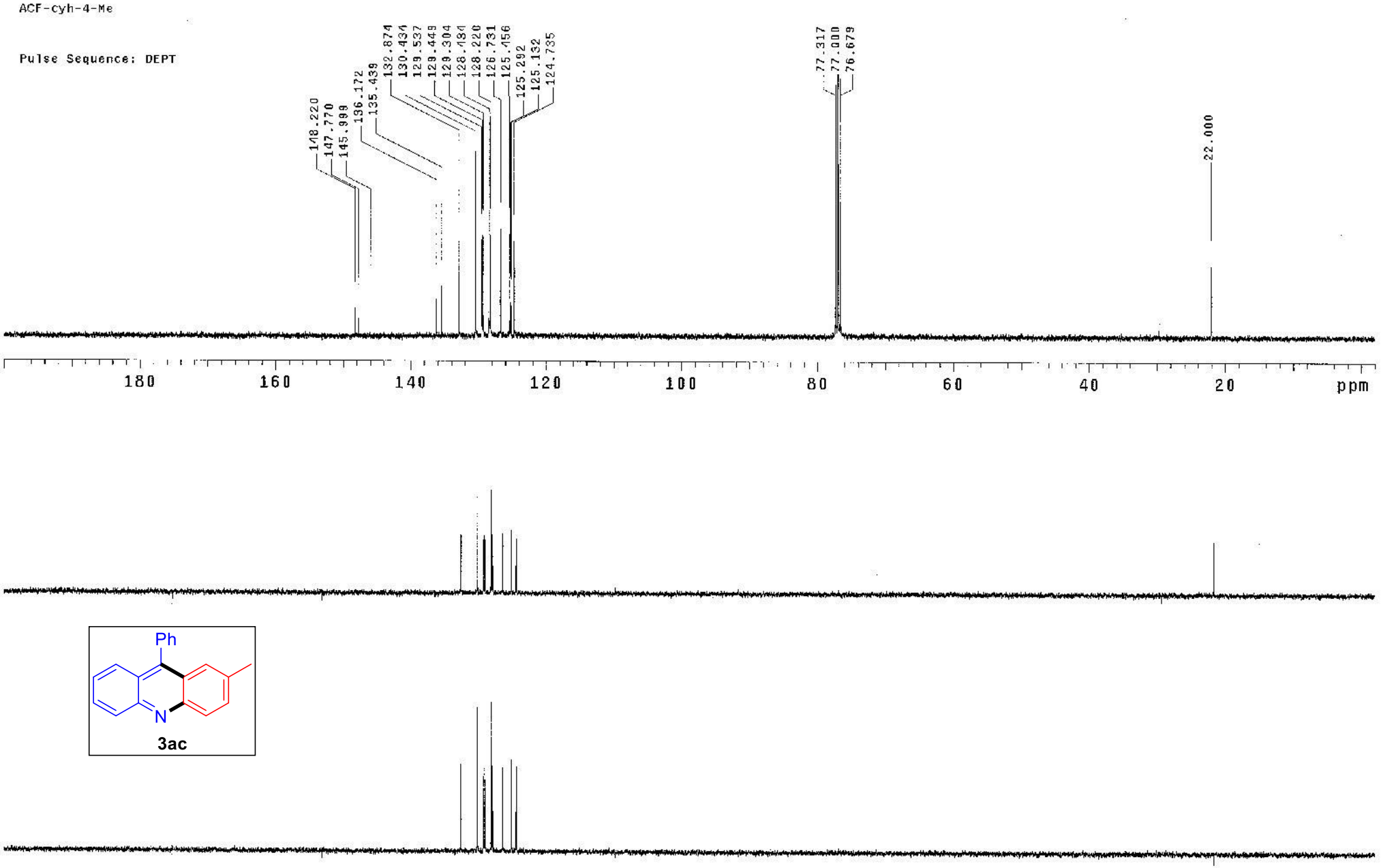
21.999

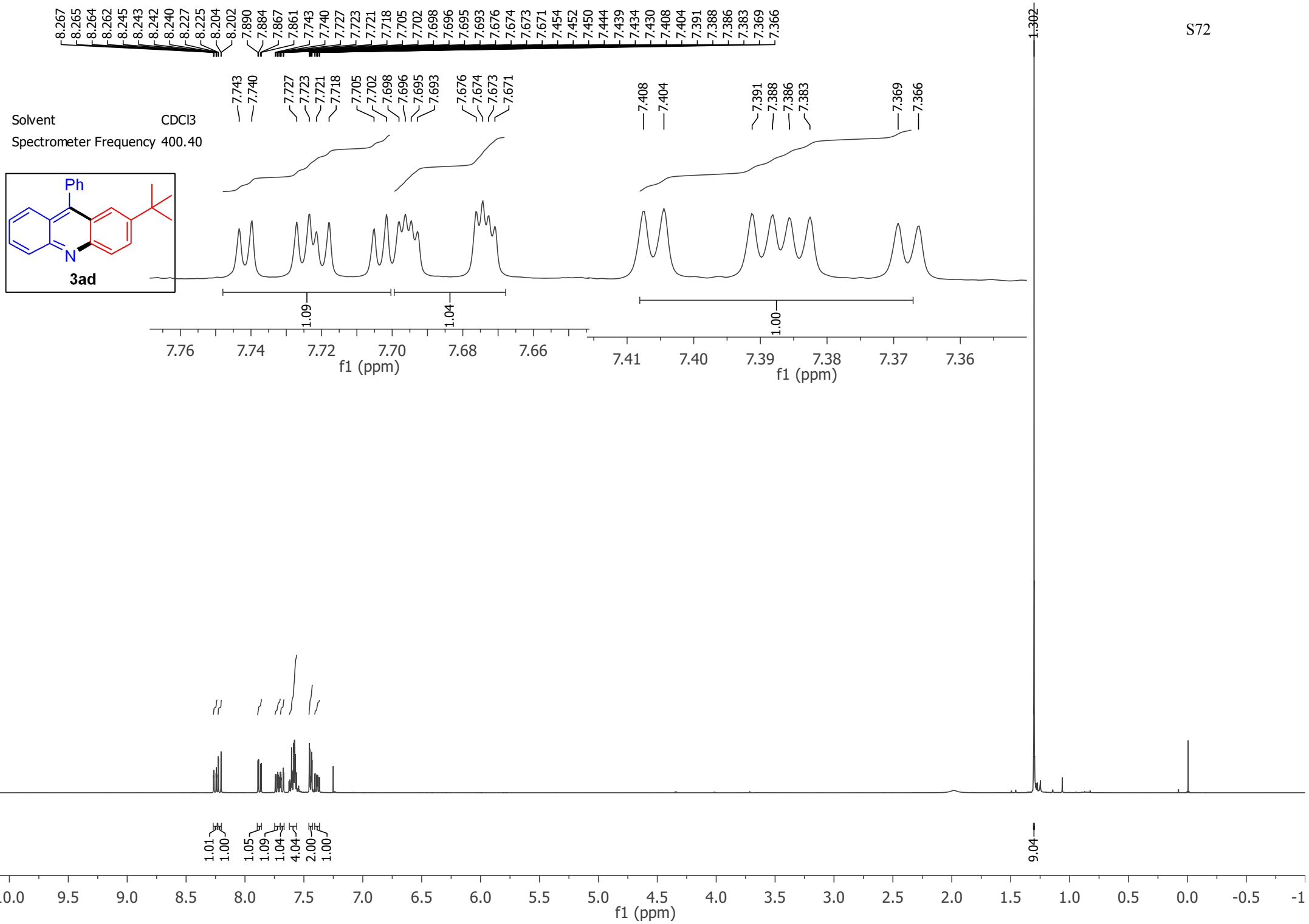


3ac



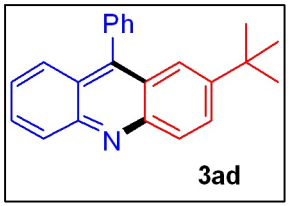


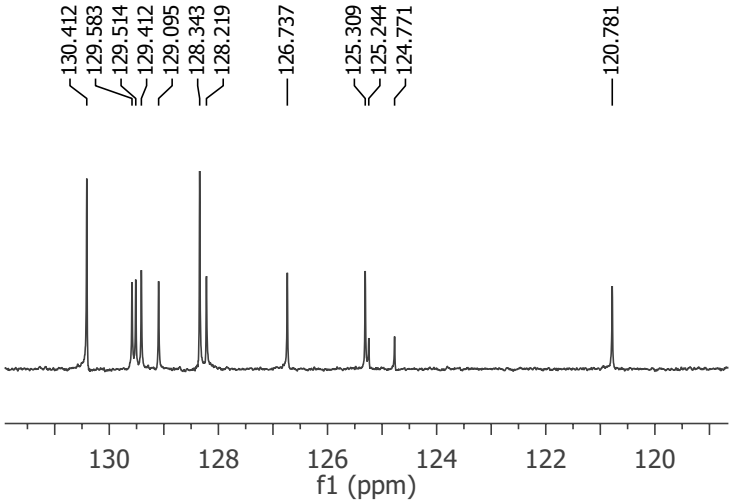

ACF-cyh-4-Me
Pulse Sequence: DEPT
148.220
147.770
145.999
136.172
135.439
132.874
130.434
129.537
129.449
129.304
128.180
128.220
126.731
125.456
125.292
125.132
124.735
77.317
77.000
76.679
22.000
180
160
140
120
100
80
60
40
20
ppm
Ph
N
3ac

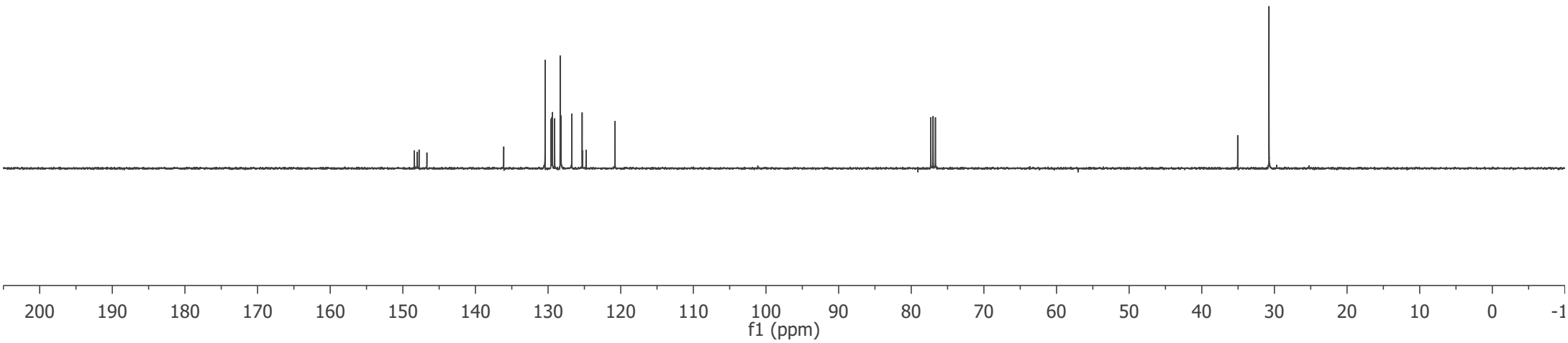

Solvent CDCl3
Spectrometer Frequency 400.40
Ph
N
3ad
f1 (ppm)
f1 (ppm)
f1 (ppm)

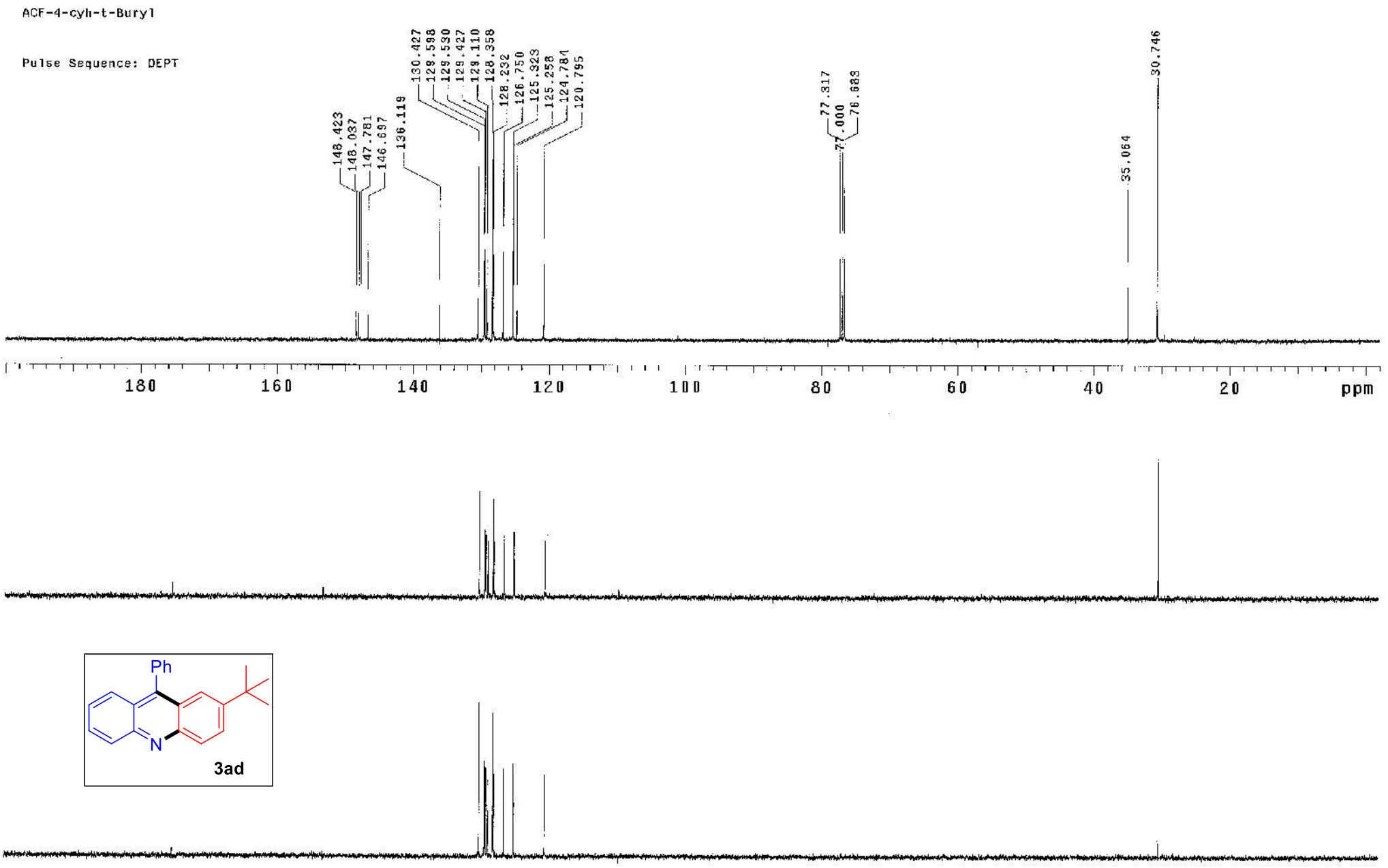
148.405
148.022
147.765
146.682
136.104
130.412
129.583
129.514
129.412
129.095
128.343
128.219
126.737
125.309
125.244
124.771
120.781
35.061
30.745

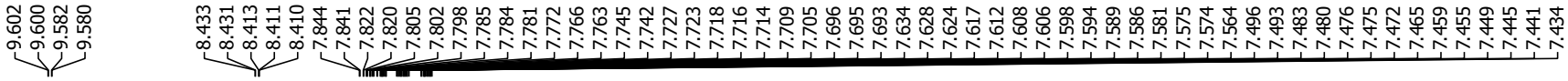
Solvent CDCl3
Spectrometer Frequency 100.69





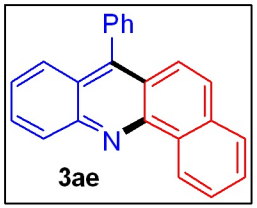


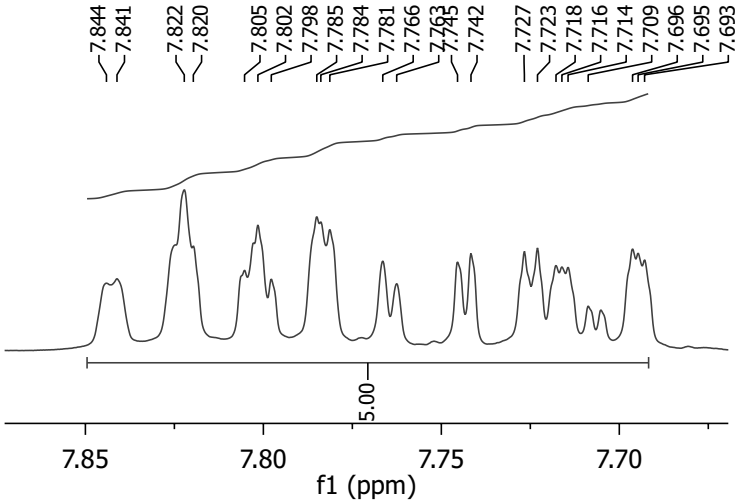

ACF-4-cyh-t-Buryl
Pulse Sequence: DEPT
148.423
148.037
147.781
146.697
136.119
130.427
129.588
129.530
129.427
129.110
128.358
128.232
126.750
125.323
125.258
124.784
120.795
77.317
77.000
76.683
35.064
30.746
180
160
140
120
100
80
60
40
20
ppm
Ph
N
3ad

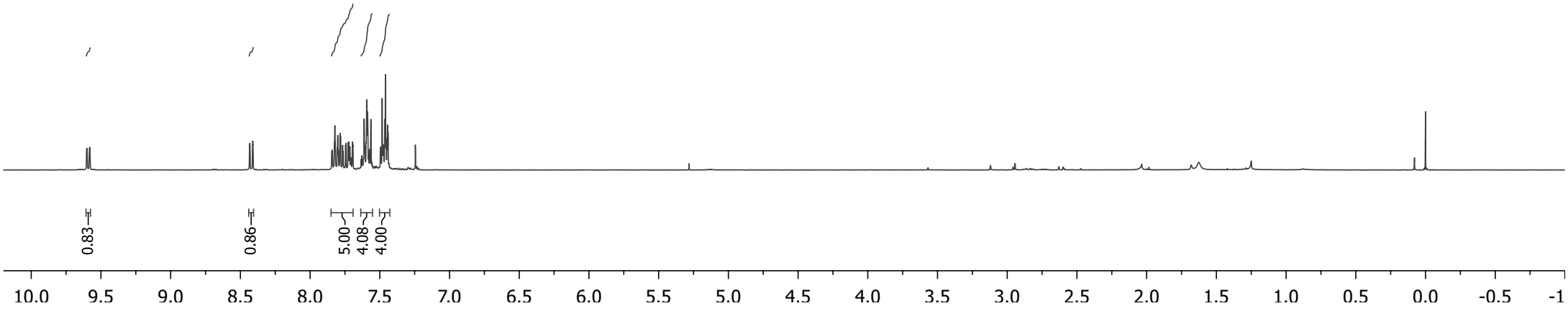


Solvent CDCl3

Spectrometer Frequency 400.40

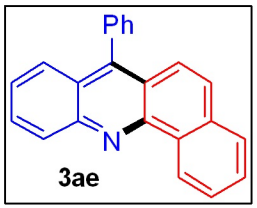


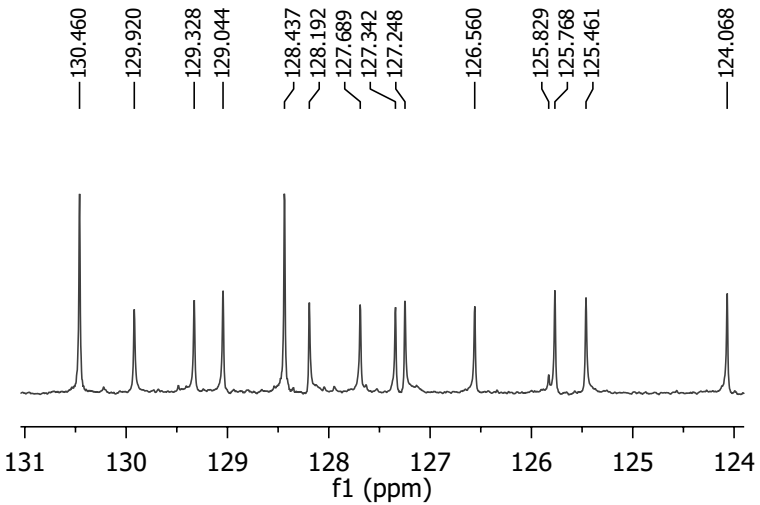


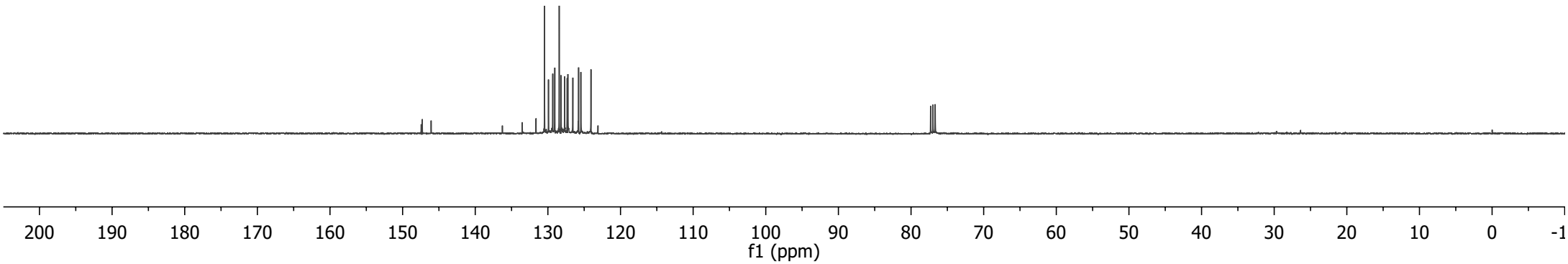


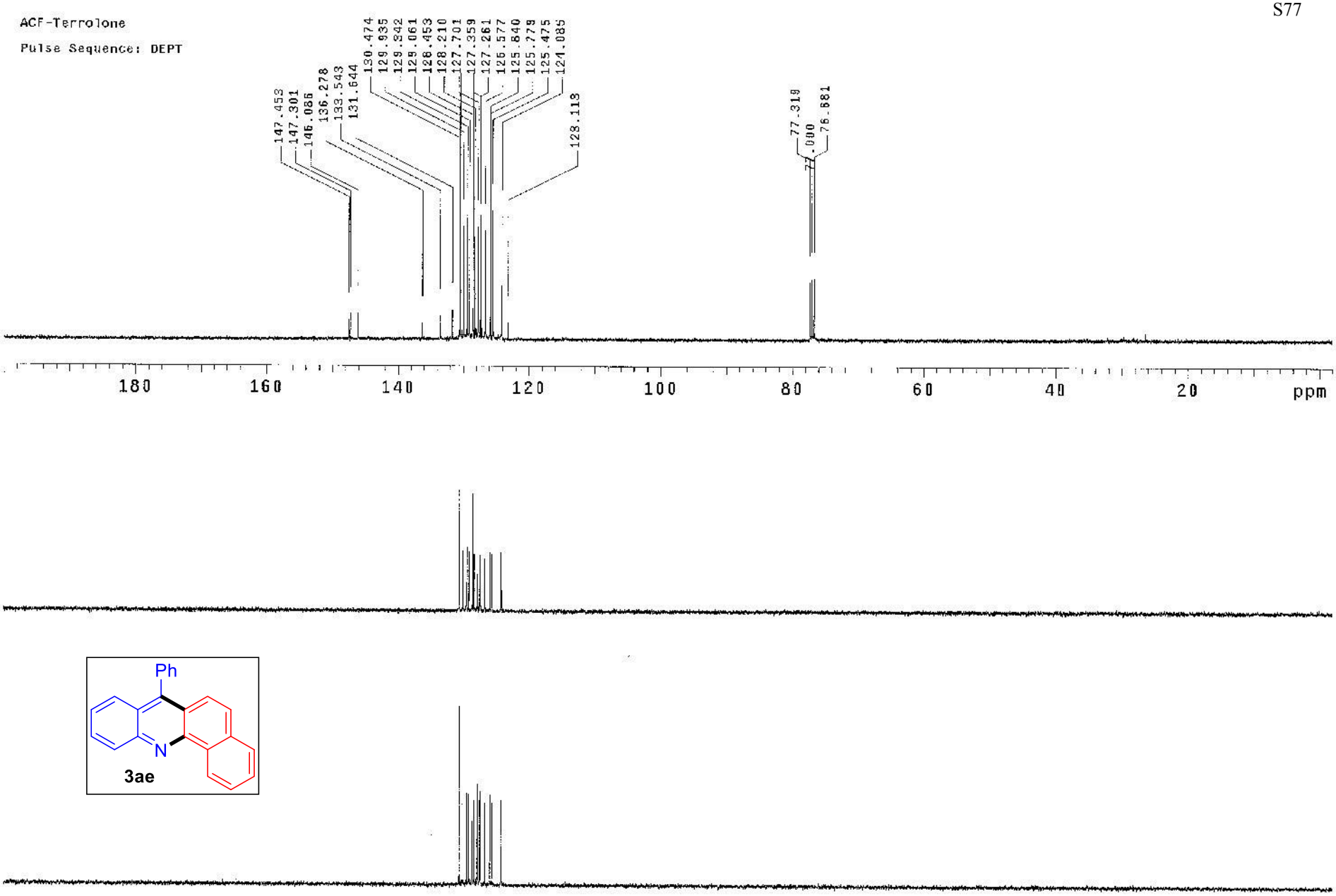
Solvent CDCl3
Spectrometer Frequency 100.69

147.435
147.288
146.071
136.263
133.527
131.626
130.460
129.920
129.328
129.044
128.437
128.192
127.689
127.342
127.248
126.560
125.829
125.768
125.461
124.068
123.100



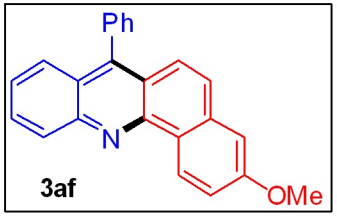


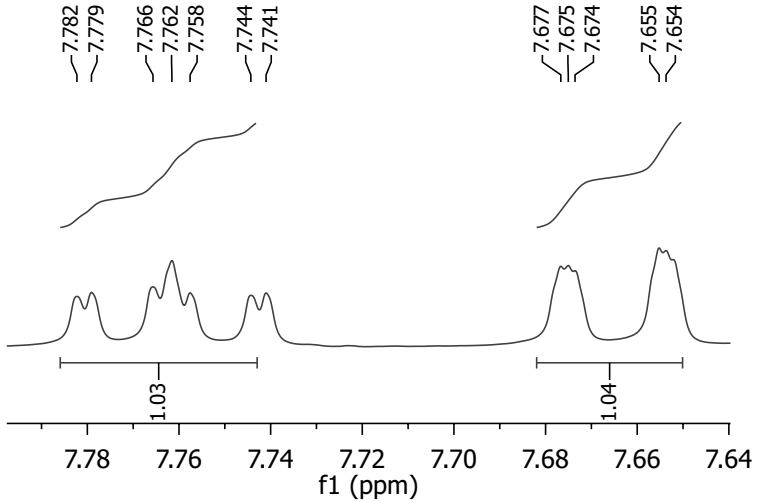


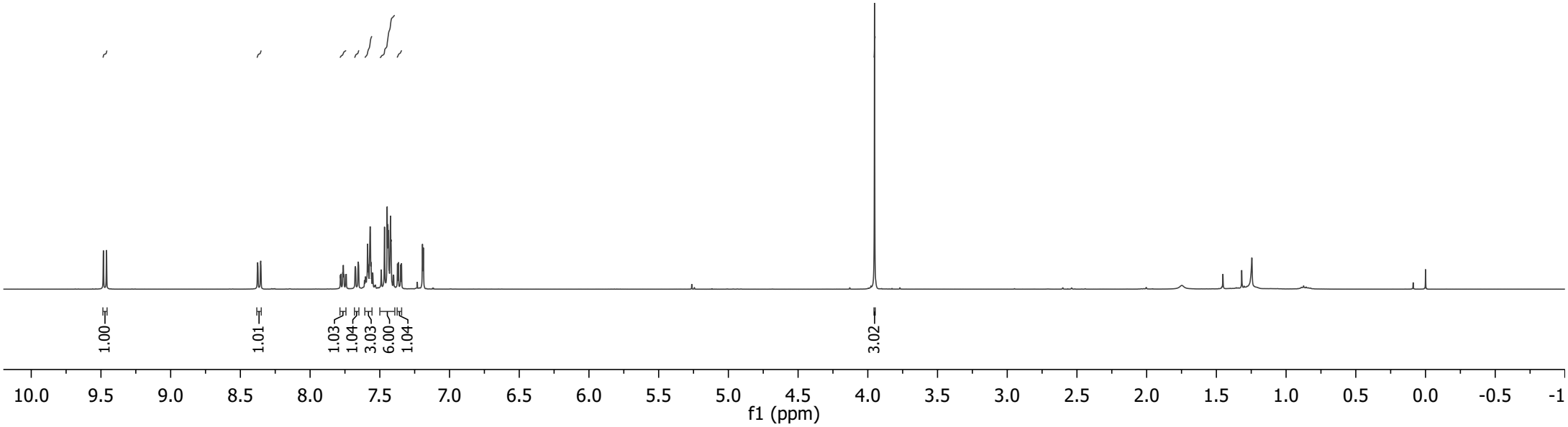

ACF-Terrolone
Pulse Sequence: DEPT
147.453
147.301
146.086
136.278
133.543
131.644
130.474
129.935
129.342
129.061
128.453
128.210
127.701
127.359
127.261
126.577
125.840
125.779
125.475
124.085
128.113
77.319
77.000
76.681
180
160
140
120
100
80
60
40
20
ppm
Ph
N
3ae

9.483
9.460
8.376
8.355
8.354
7.782
7.779
7.766
7.762
7.758
7.744
7.741
7.677
7.675
7.674
7.655
7.654
7.588
7.584
7.581
7.569
7.563
7.551
7.490
7.466
7.447
7.442
7.438
7.433
7.423
7.420
7.401
7.373
7.367
7.351
7.344
3.952

Solvent CDCl3
Spectrometer Frequency 400.40



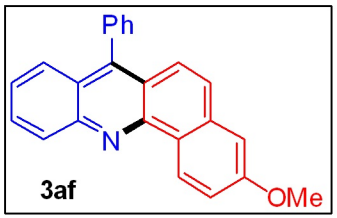


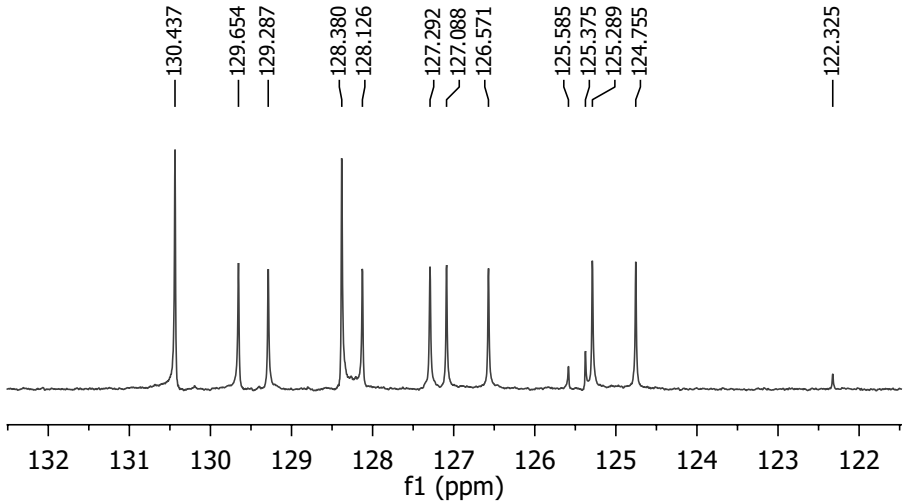


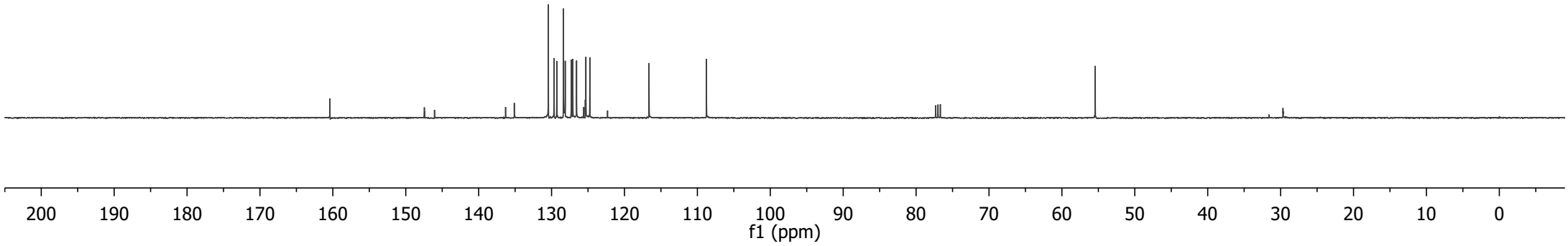
Solvent CDCl3

Spectrometer Frequency 100.69

160.393
147.449
147.418
146.062
130.437
129.654
129.287
128.380
128.126
126.571
125.375
125.289
124.755
116.629
108.760
55.424





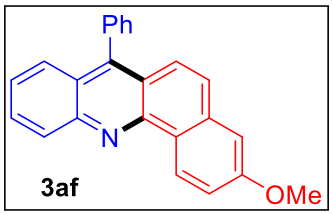


ACF-6-OMe-Tet

Pulse Sequence: DEPT

130.151
129.669
129.301
128.393
128.112
127.306
127.101
126.585
125.301
124.769
116.640
108.770
55.433

180 160 140 120 100 80 60 40 20 ppm



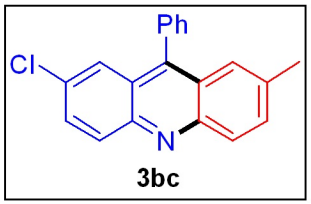
3af

8.199 8.197 8.176 8.175 8.162 8.151 8.139 7.656 7.651 7.645 7.641 7.632 7.631 7.628 7.622 7.617 7.613 7.610 7.607 7.605 7.600 7.584 7.421 7.416 7.411 7.408 7.405 7.401 7.397 7.391

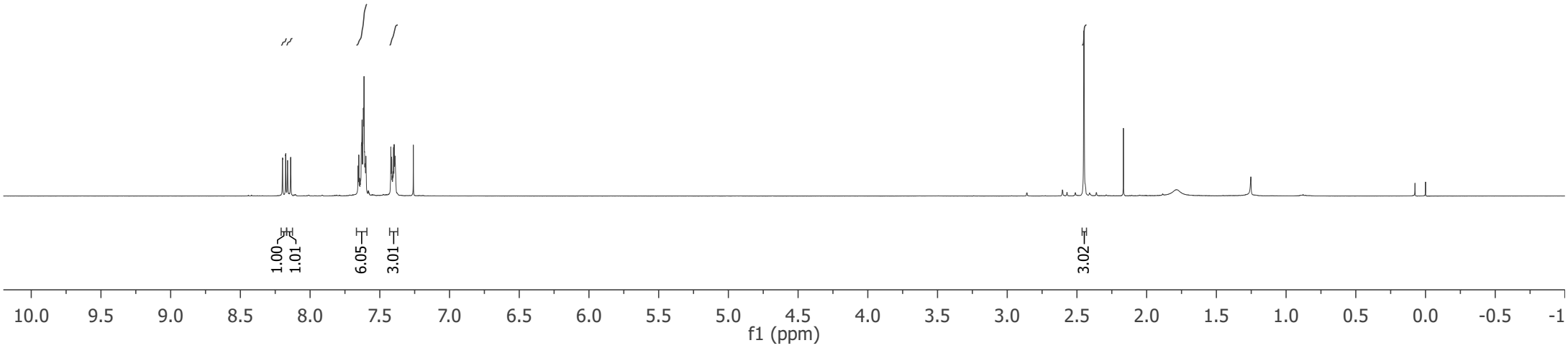
2.451 2.449

Solvent CDCL3

Spectrometer Frequency 400.40

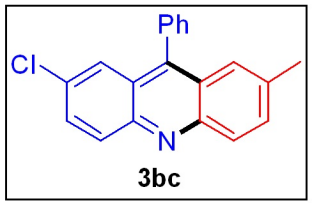


3bc

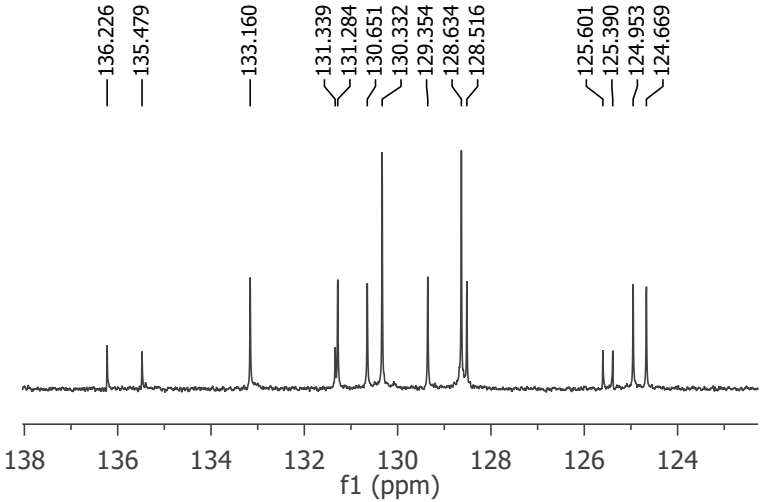


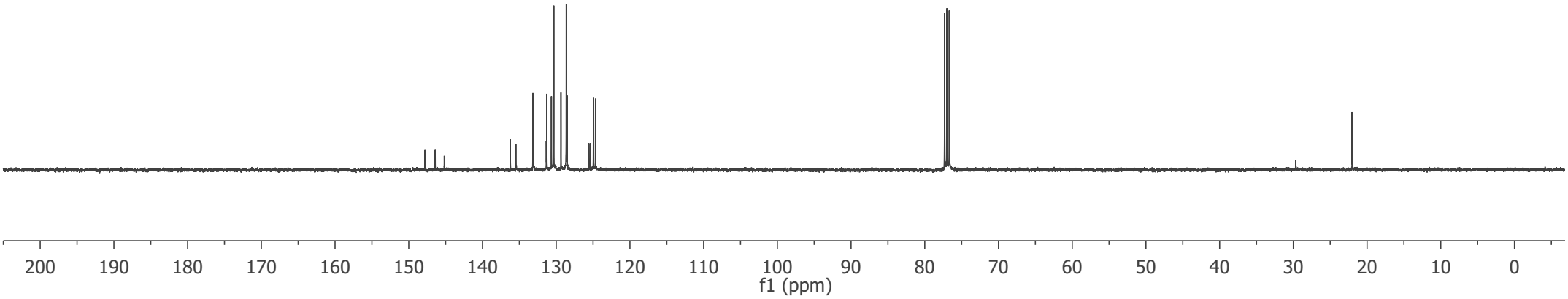
147.802
146.420
145.175
136.226
135.479
133.160
131.339
131.284
130.651
130.332
129.354
128.634
128.516
125.601
125.390
124.953
124.669
77.308
76.991
76.674
29.675
22.025

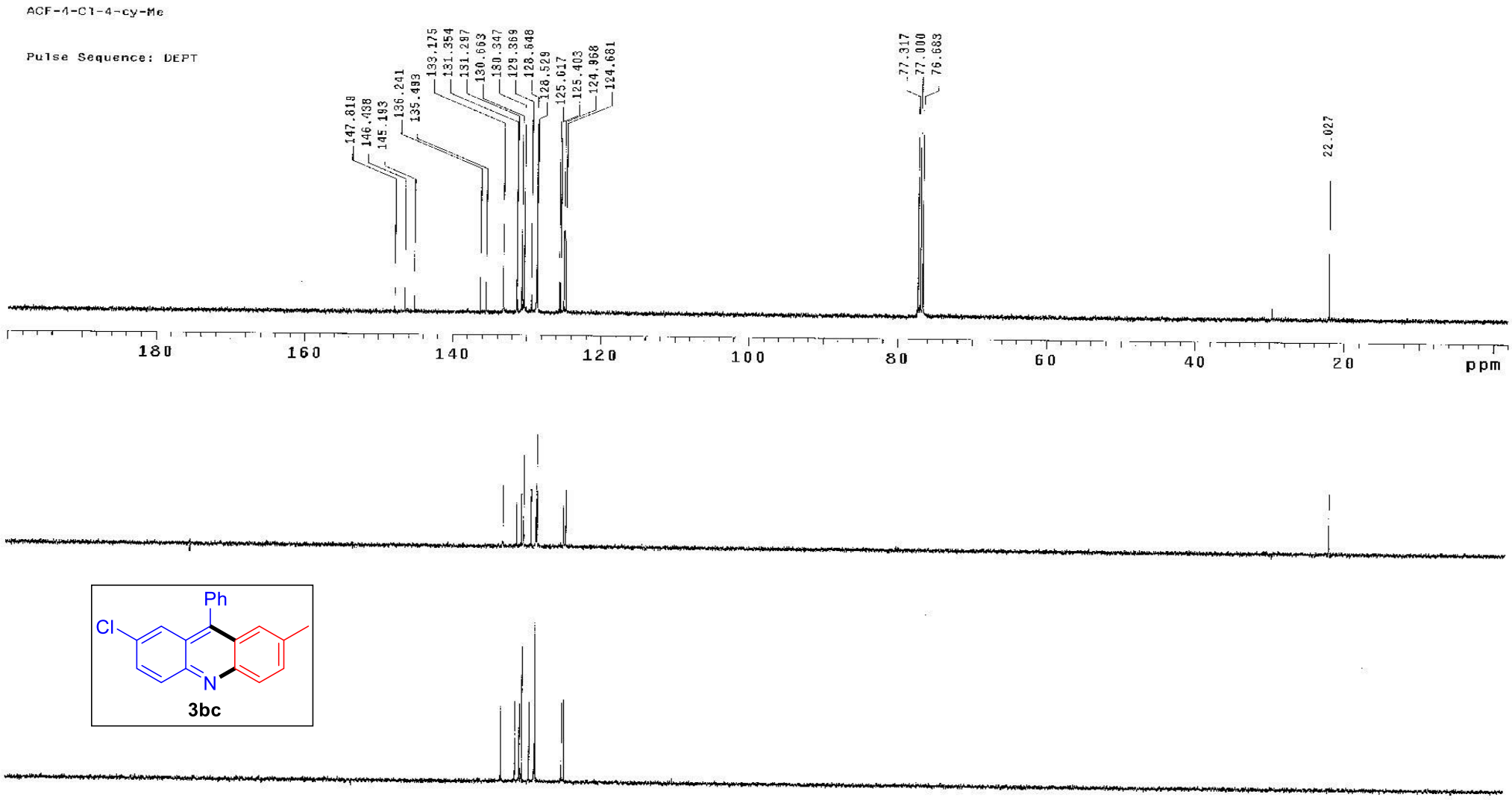
Solvent CDCL3
Spectrometer Frequency 100.69



3bc





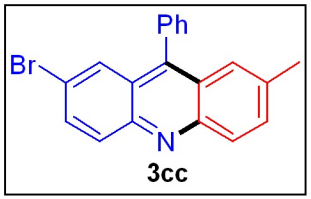

ACF-4-Cl-4-cy-Me
Pulse Sequence: DEPT
147.818
146.438
145.193
136.241
135.493
133.175
131.354
131.287
130.663
130.347
129.369
128.648
128.529
125.617
125.403
124.968
124.681
77.317
77.000
76.683
22.027
180
160
140
120
100
80
60
40
20
ppm
Ph
Cl
N
3bc

8.154 8.131 8.120 8.119 8.097 8.096 7.795 7.791 7.773 7.767 7.750 7.744 7.650 7.645 7.641 7.637 7.628 7.626 7.623 7.616 7.614 7.609 7.606 7.603 7.597 7.594 7.416 7.411 7.409 7.406 7.403 7.401 7.397 7.392 7.388 7.386

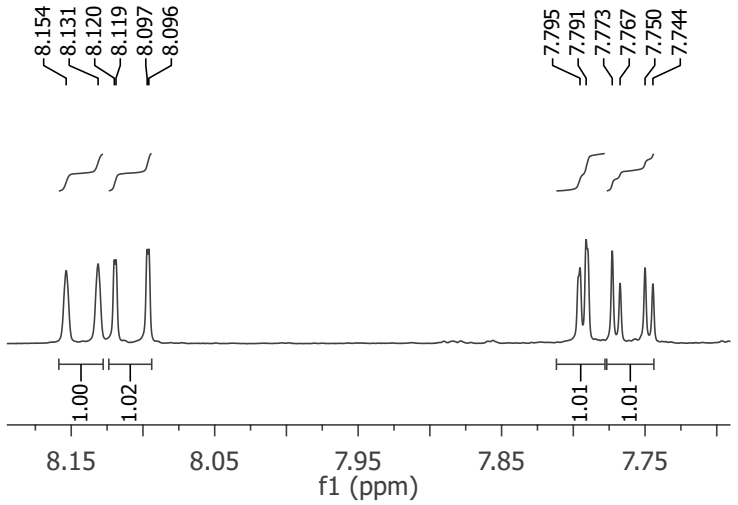
2.448

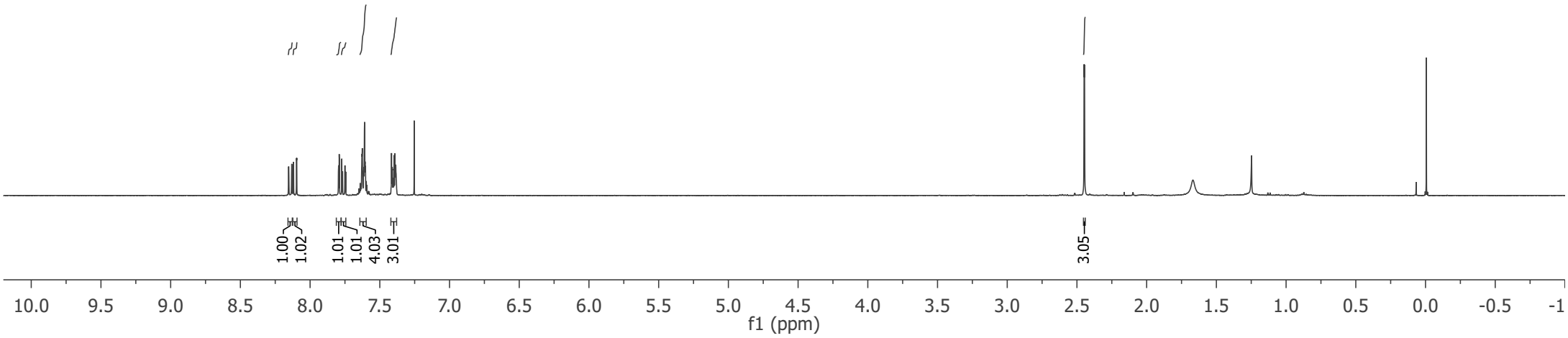
Solvent CDCl3

Spectrometer Frequency 400.40



3cc

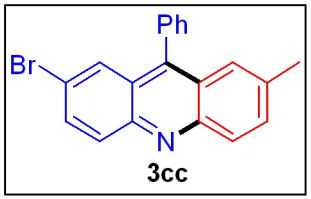


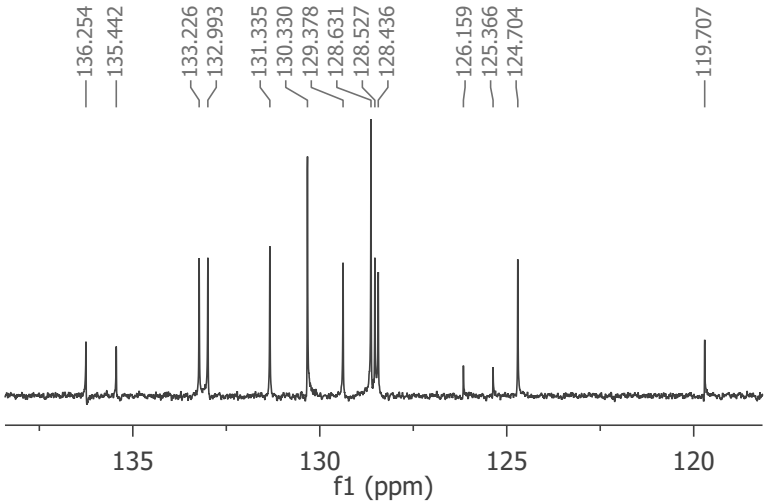


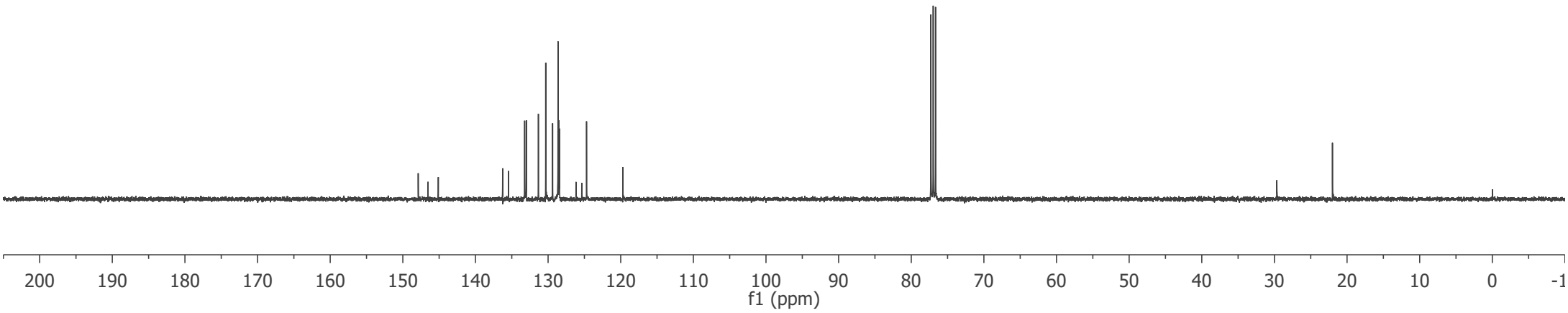
147.879
146.530
145.114
136.254
135.442
133.226
132.993
131.335
130.330
129.378
128.631
128.527
128.436
126.159
125.366
124.704
119.707

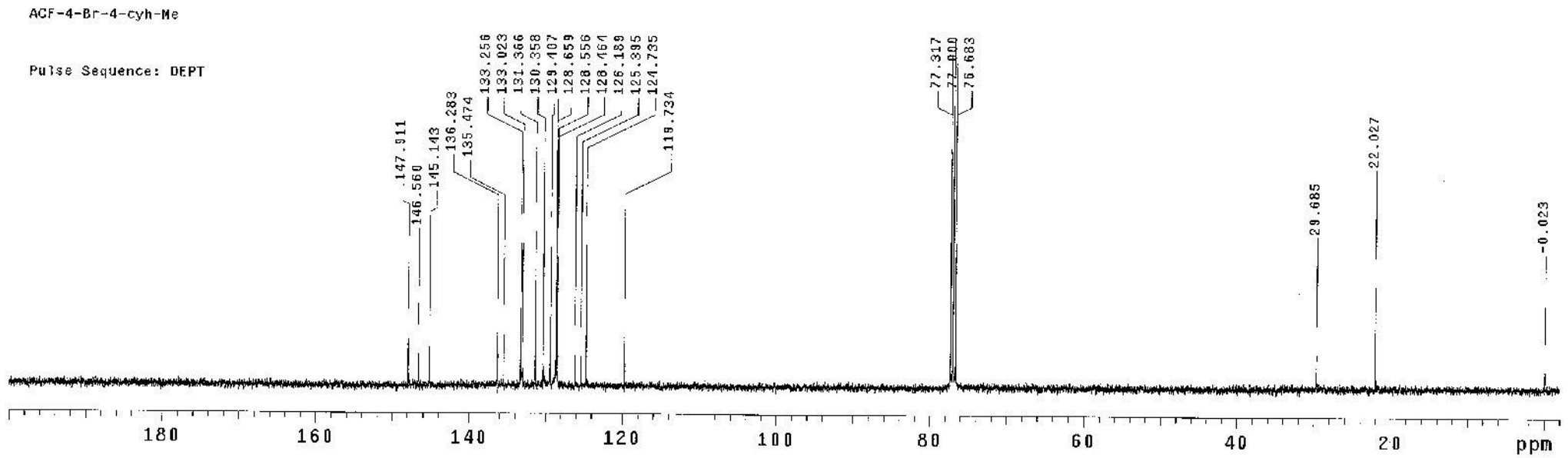
22.011

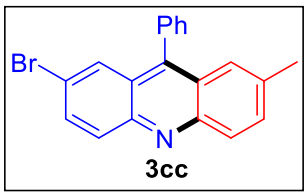
Solvent CDCl3
Spectrometer Frequency 100.69

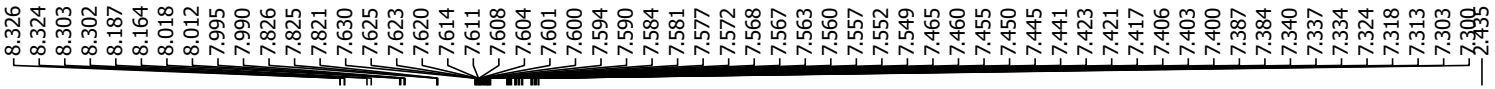






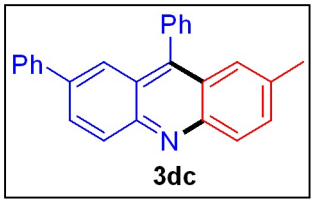

ACF-4-Br-4-cyh-Me
Pulse Sequence: DEPT
147.911
146.560
145.143
136.283
135.474
133.256
133.023
131.366
130.358
129.107
128.659
128.556
128.461
126.189
125.395
124.735
119.734
77.317
77.000
76.683
29.685
22.027
-0.023
180
160
140
120
100
80
60
40
20
ppm


Ph
Br
N
3cc

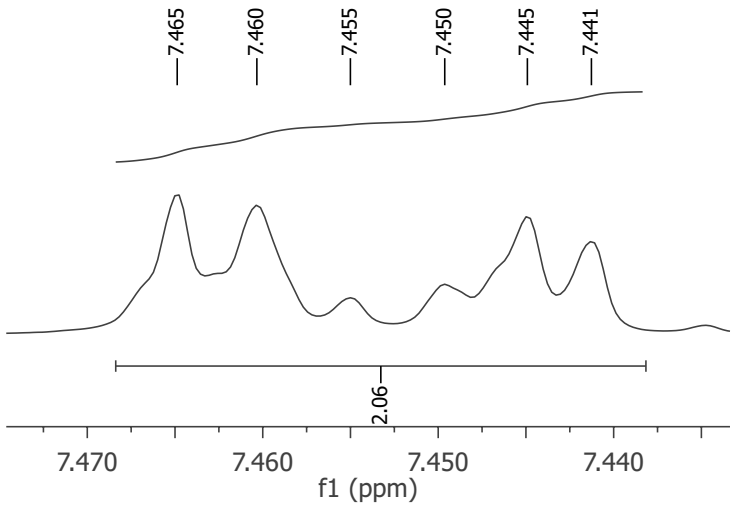


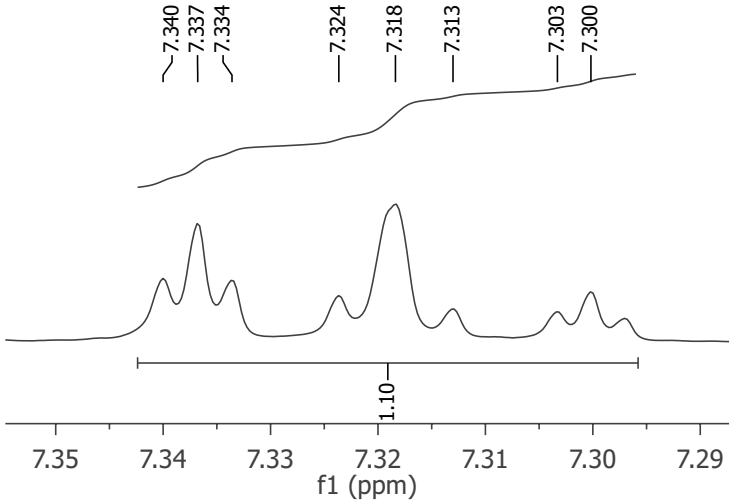
Solvent CDCl3

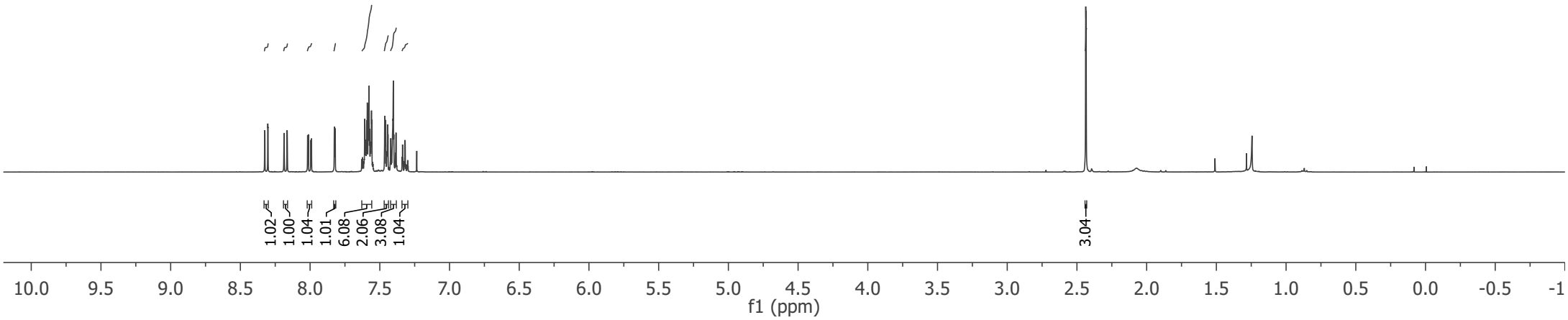
Spectrometer Frequency 400.40



3dc



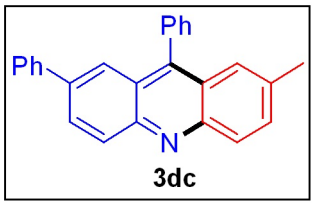




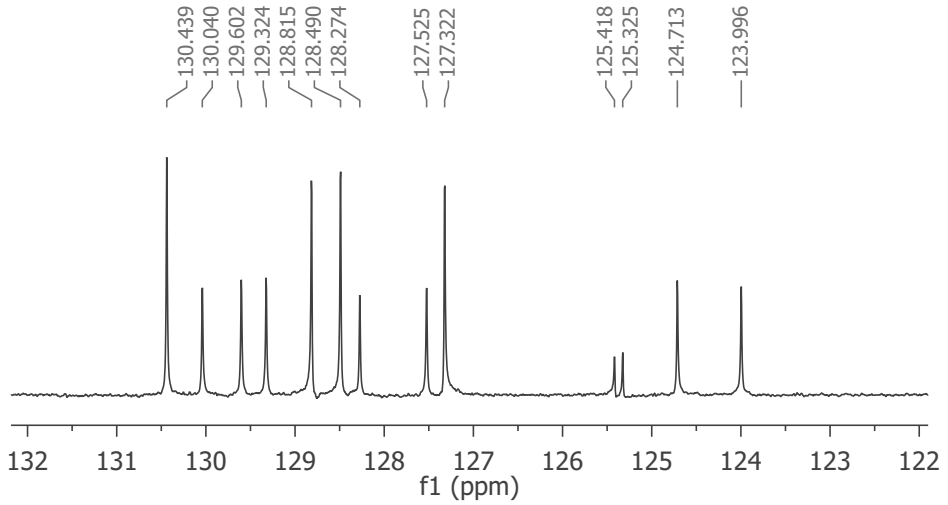
147.724
147.638
146.071
140.531
137.893
136.055
135.553
132.788
130.439
130.040
129.602
129.324
128.815
128.490
128.274
127.525
127.322
125.418
125.325
124.713
123.996

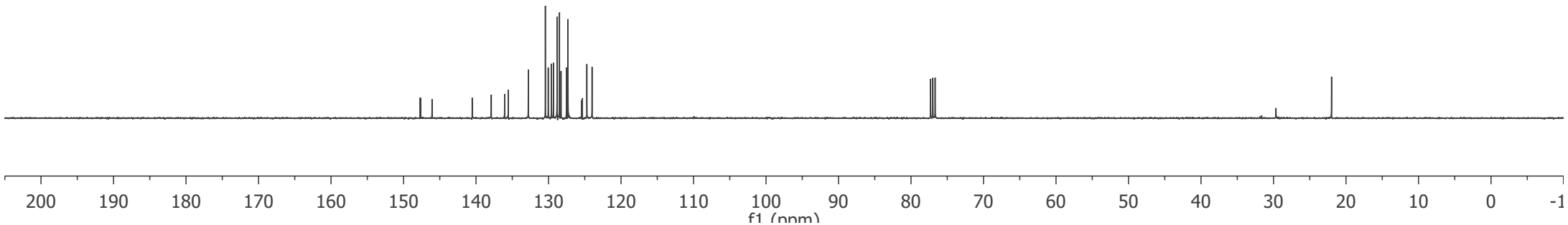
21.993

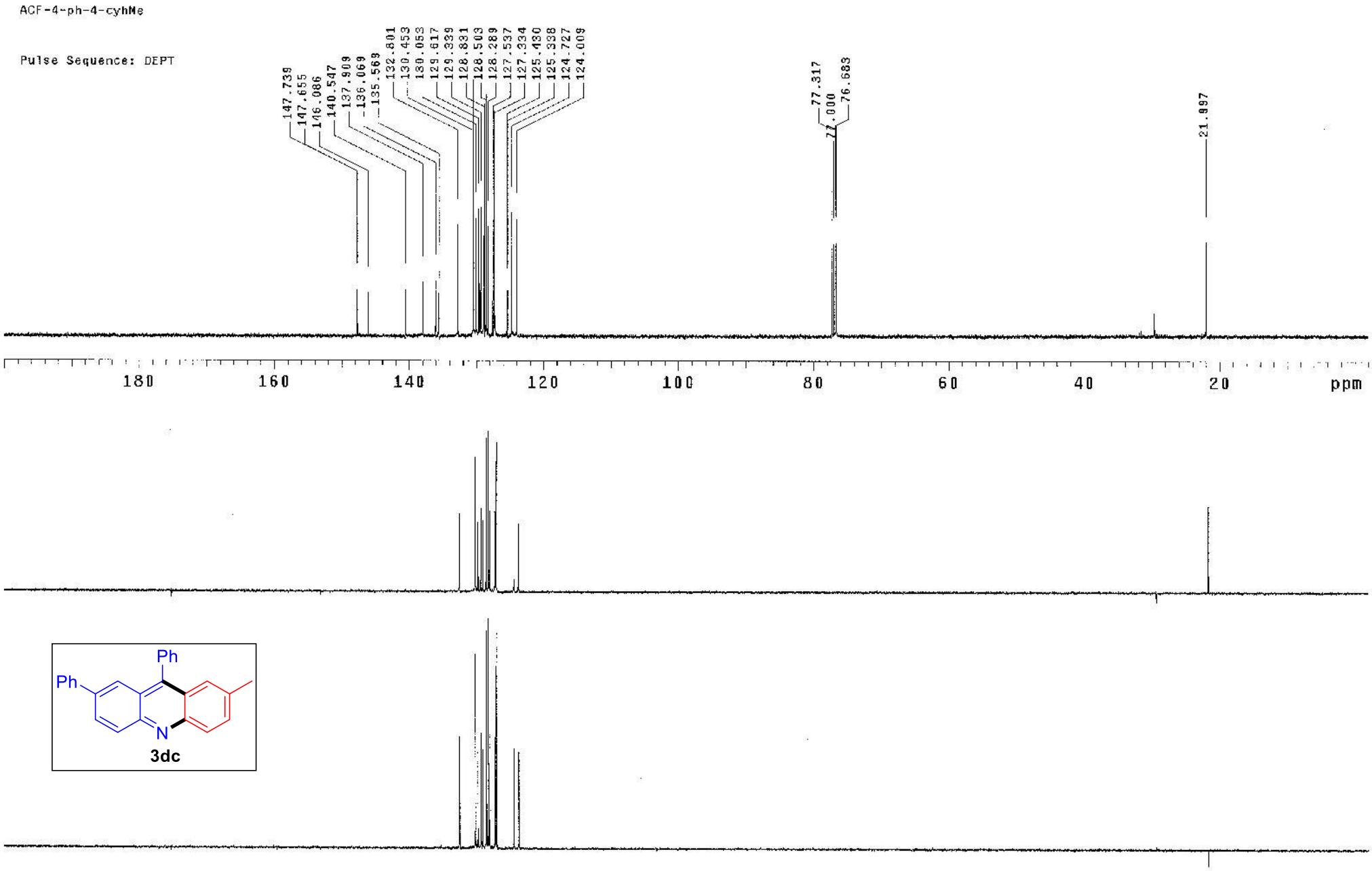
Solvent CDCl3
Spectrometer Frequency 100.69

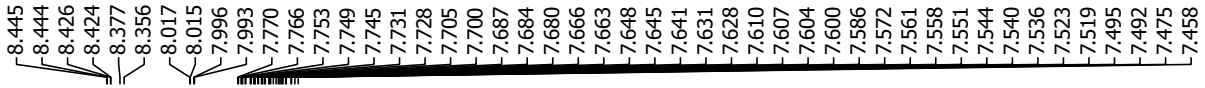


3dc



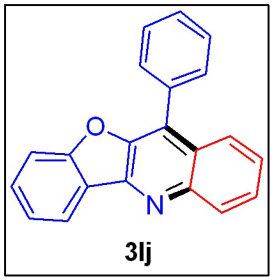


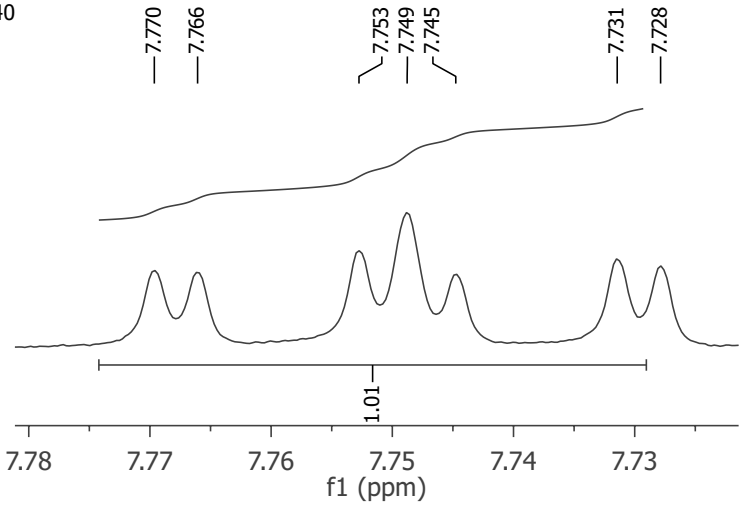

ACF-4-ph-4-cyhMe
Pulse Sequence: DEPT
147.739
147.655
146.086
140.547
137.909
136.069
135.569
132.801
130.453
130.053
129.617
129.339
128.831
128.503
128.289
127.537
127.334
125.430
125.338
124.727
124.009
77.317
77.000
76.683
21.997
180
160
140
120
100
80
60
40
20
ppm
Ph
Ph
N
3dc

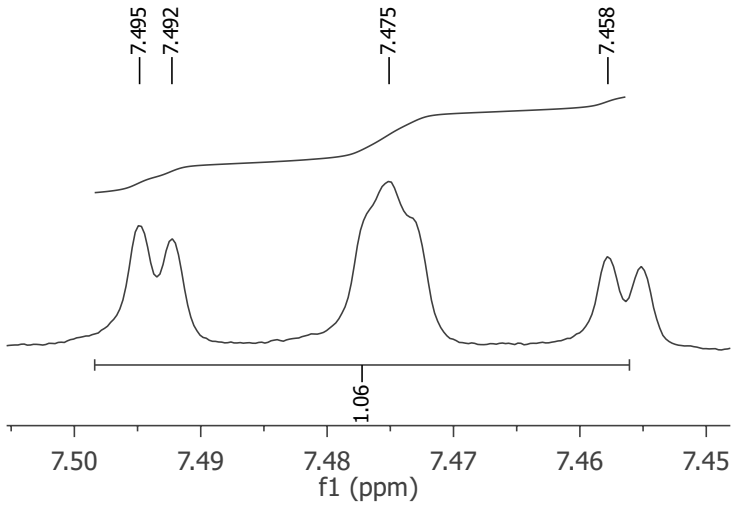


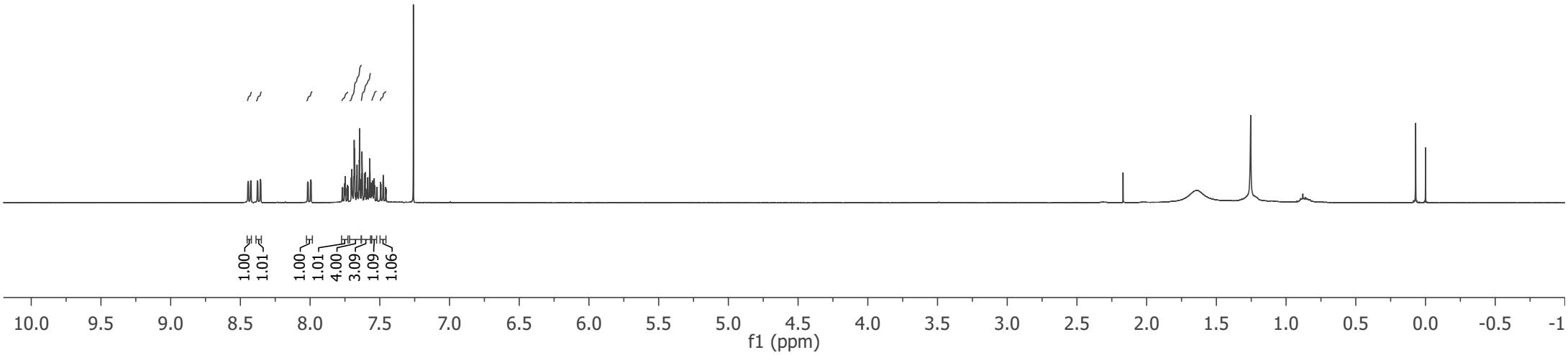
Solvent CDCl3

Spectrometer Frequency 400.40





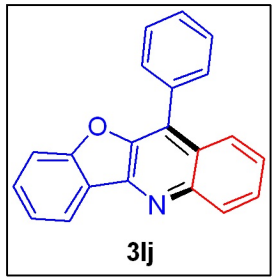




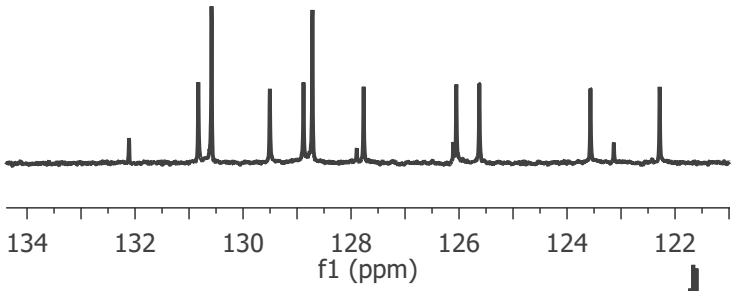
159.574
146.984
146.546
145.186
132.111
130.828
130.583
129.504
128.880
128.715
127.767
126.051
125.624
123.566
122.285
112.315

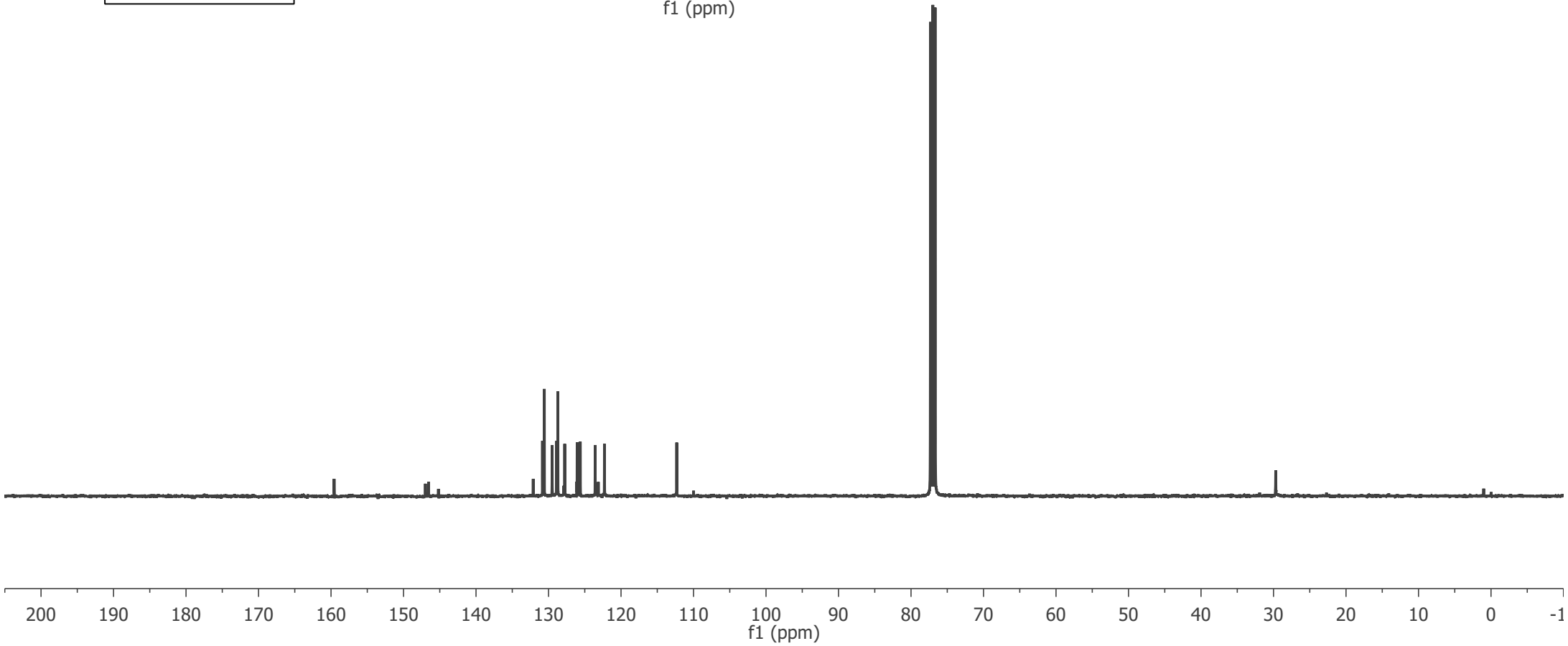
132.111
130.828
130.583
129.504
128.880
128.715
127.894
127.767
126.106
126.051
125.624
123.566
123.134
122.285

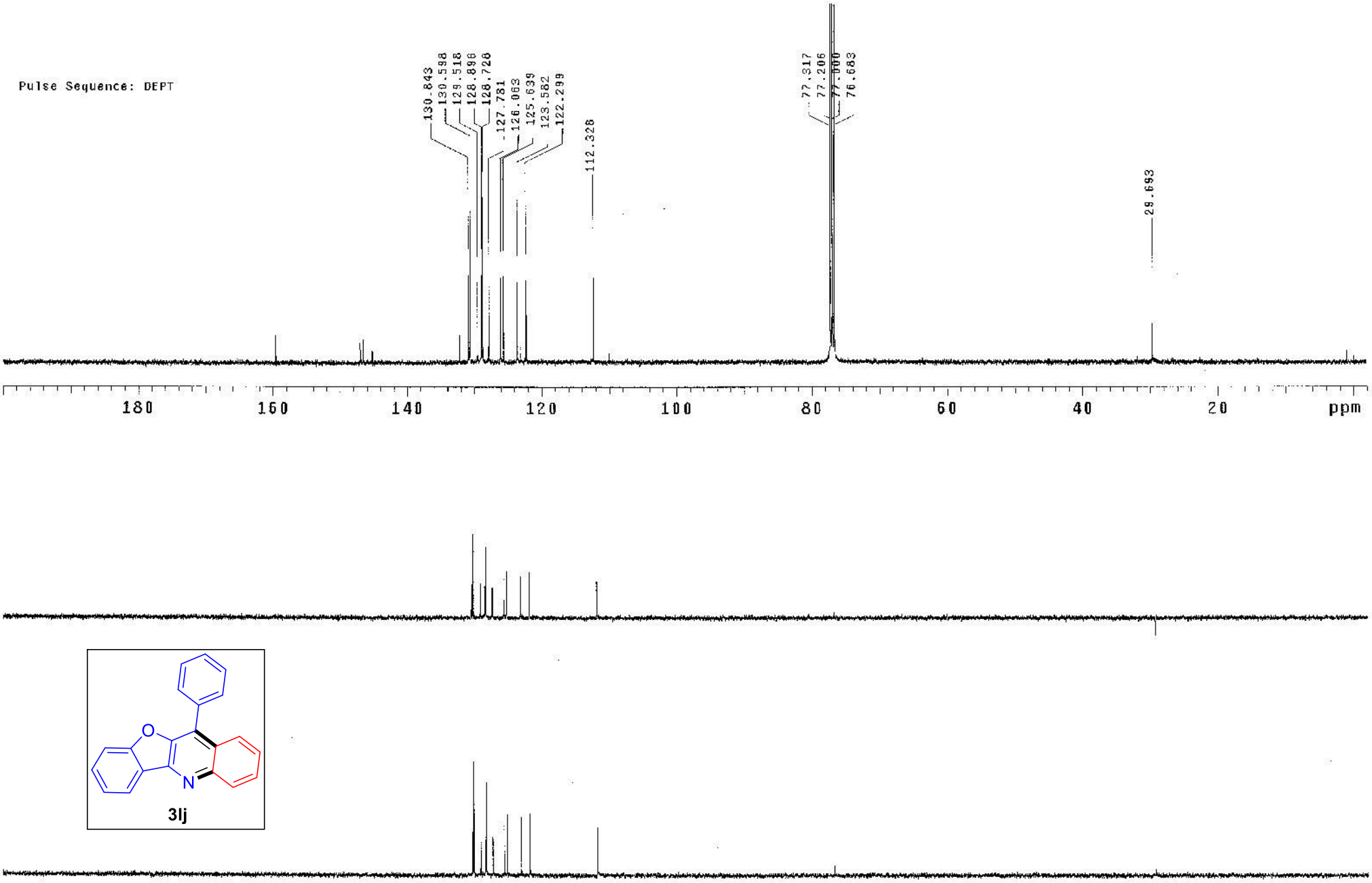
Solvent CDCl3
Spectrometer Frequency 100.69



3lj



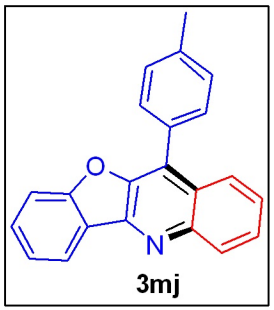


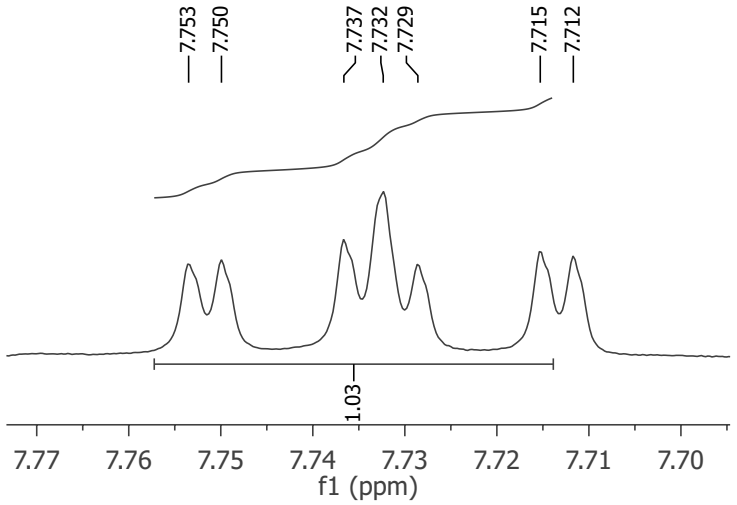

Pulse Sequence: DEPT
130.843
130.598
129.518
128.896
128.720
127.781
126.063
125.639
123.582
122.299
112.328
77.317
77.206
77.000
76.683
29.693
180
160
140
120
100
80
60
40
20
ppm
O
N
3lj

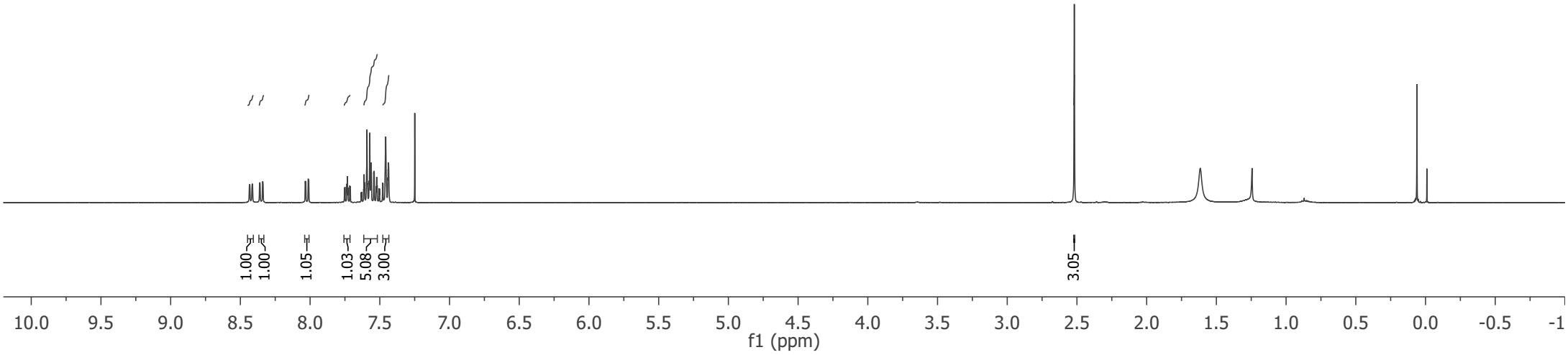
8.433 8.414 8.360 8.340 8.034 8.032 8.031 8.013 8.011 8.010 7.753 7.750 7.737 7.732 7.729 7.715 7.712 7.634 7.630 7.616 7.613 7.609 7.592 7.577 7.572 7.567 7.562 7.543 7.541 7.527 7.522 7.519 7.505 7.502 7.480 7.478 7.458 7.457 7.443 7.439 7.437 2.519

Solvent CDCl3

Spectrometer Frequency 400.28

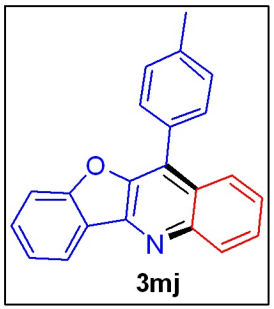




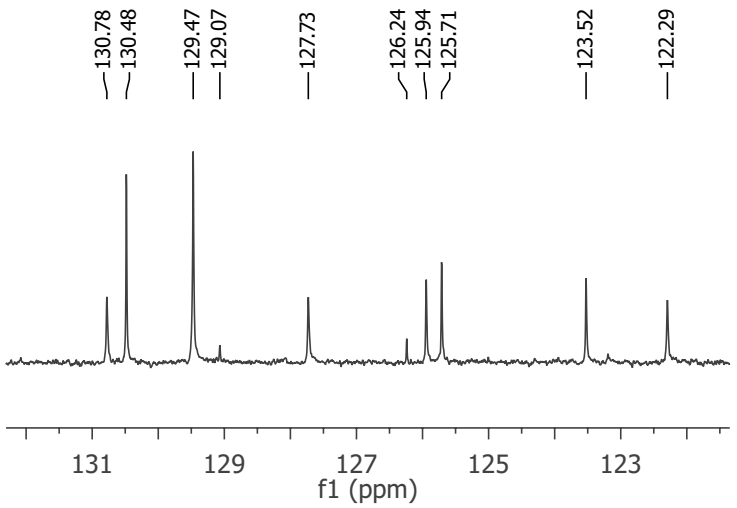


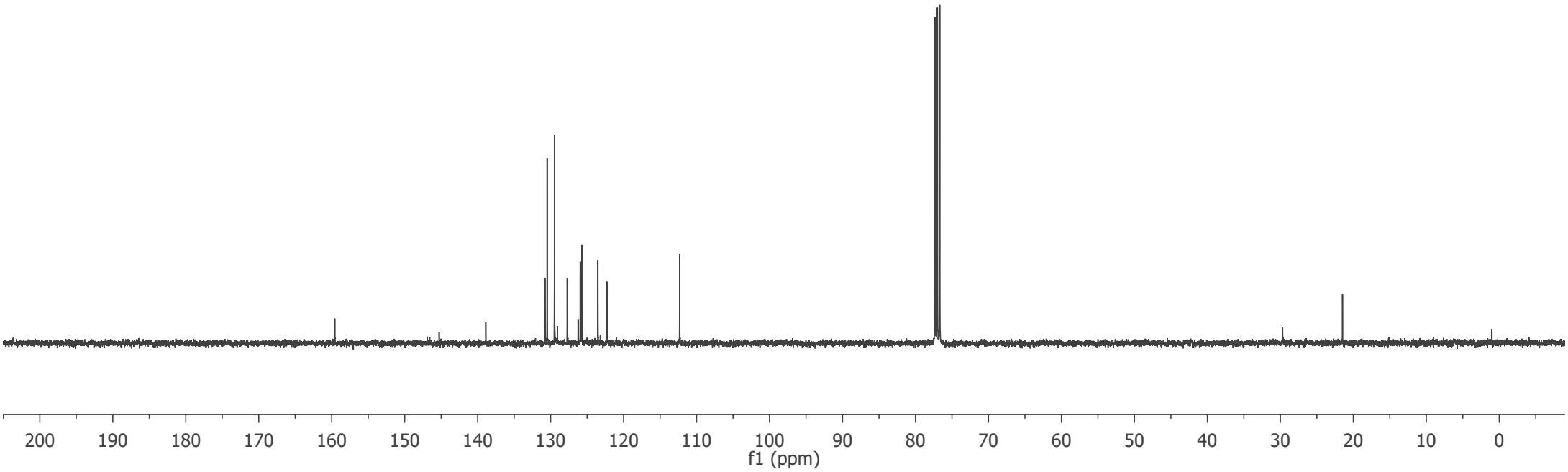
159.58
145.28
144.75
138.88
130.78
130.48
129.47
127.73
125.94
125.71
123.52
122.29
112.32
21.48

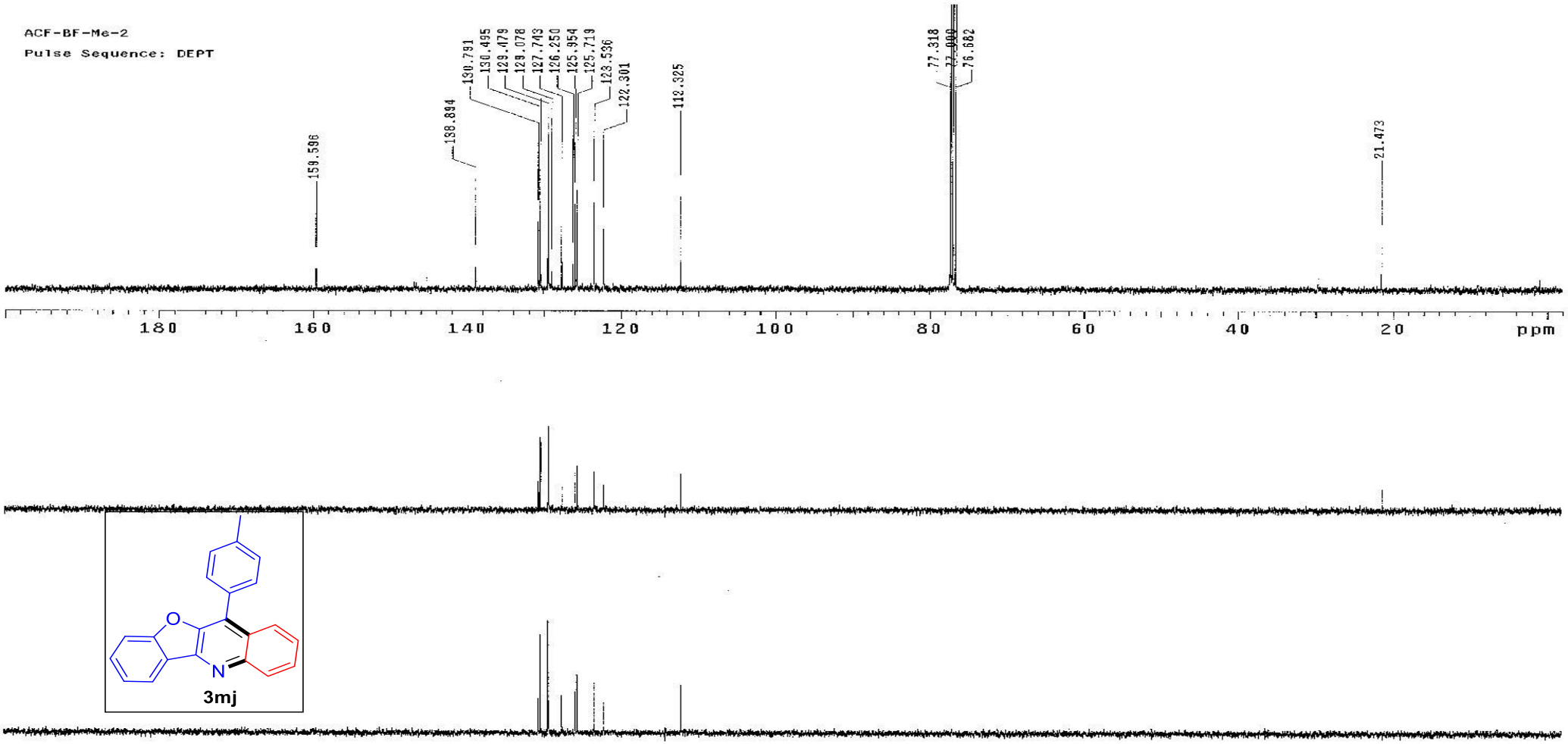
Solvent CDCl3
Spectrometer Frequency 100.66



3mj



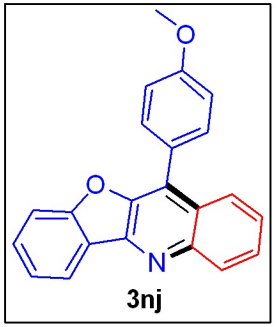


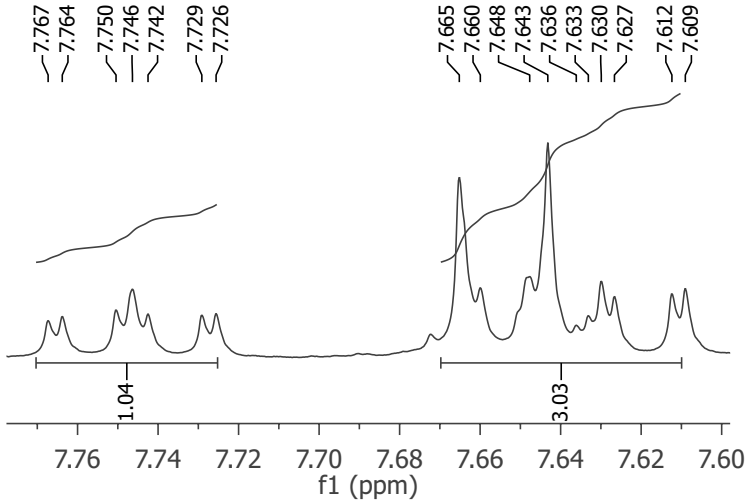

ACF-BF-Me-2
Pulse Sequence: DEPT
159.596
138.884
130.791
130.495
129.479
129.078
127.743
126.250
125.954
125.719
123.536
122.301
112.325
77.318
77.000
76.682
21.473
180
160
140
120
100
80
60
40
20
ppm
O
N
3mj

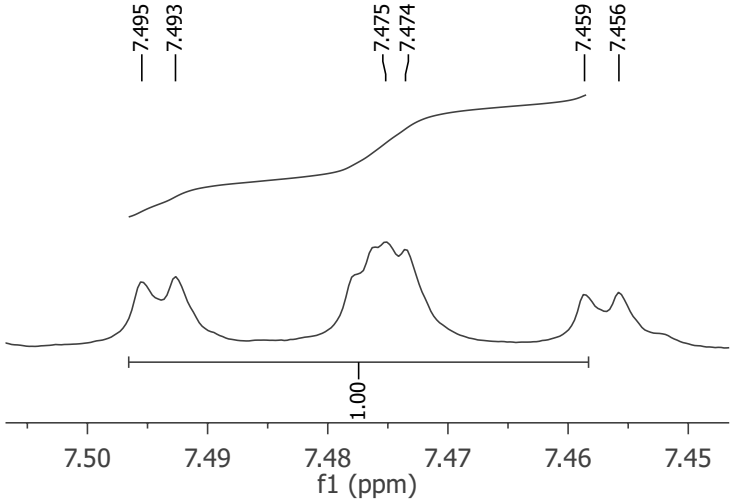
8.445 8.443 8.442 8.426 8.424 8.422 8.371 8.369 8.368 8.349 8.348 8.346 8.054 8.052 7.750 7.746 7.742 7.729 7.726 7.665 7.660 7.648 7.643 7.630 7.627 7.612 7.609 7.584 7.582 7.565 7.562 7.548 7.544 7.541 7.495 7.493 7.475 7.474 7.266 7.195 7.189 7.178 7.173 3.960

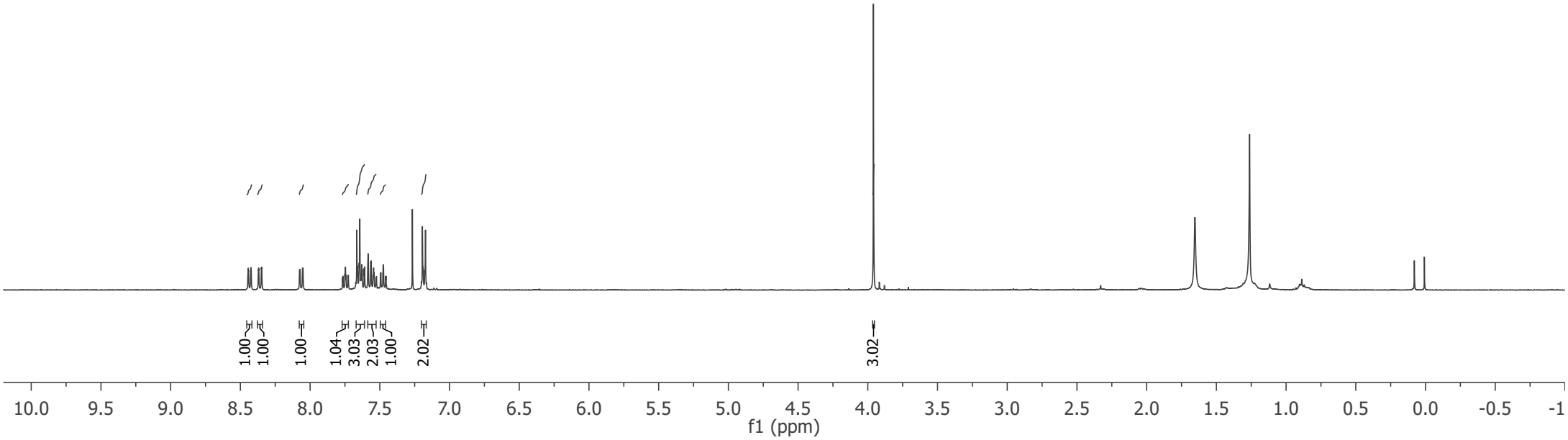
Solvent CDCl3

Spectrometer Frequency 400.40



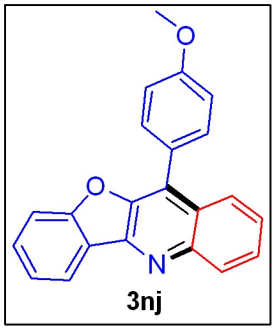


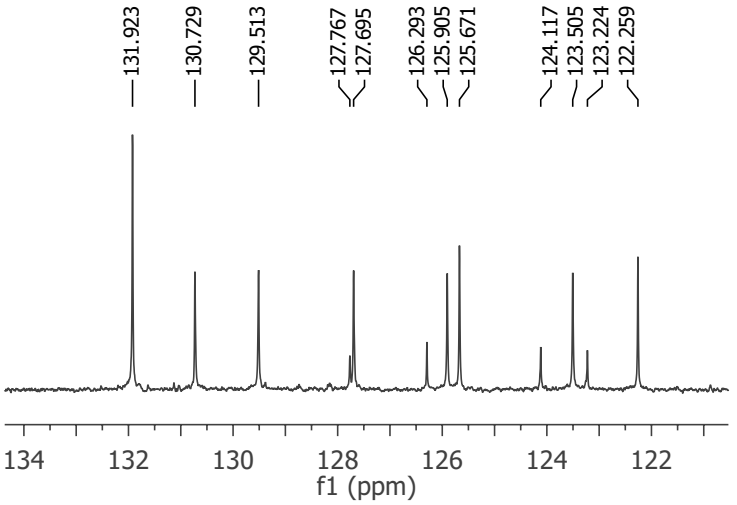


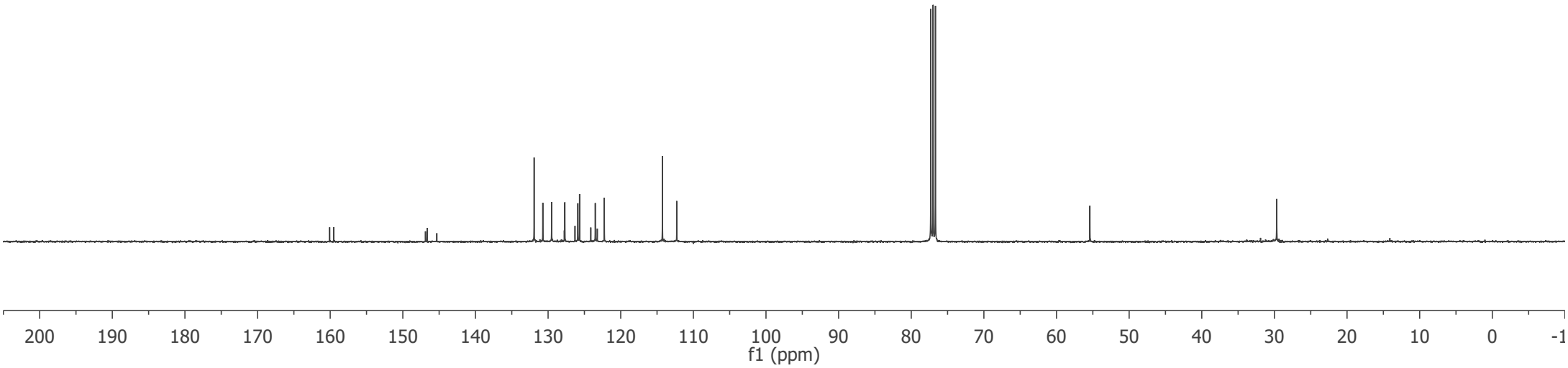


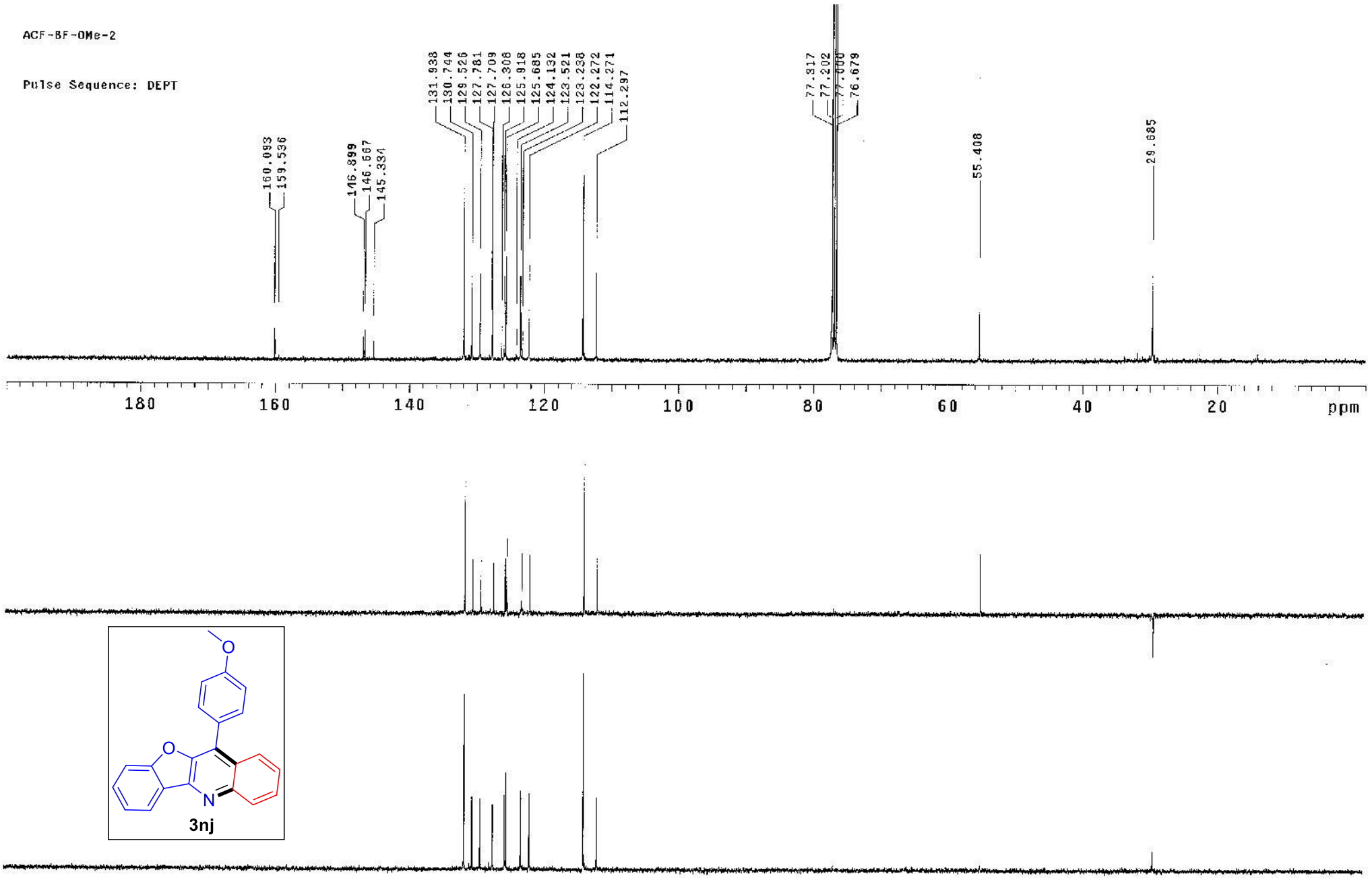
160.074
159.517
146.883
146.650
145.318
131.923
130.729
129.513
127.695
126.293
125.905
125.671
124.117
123.505
122.259
114.257
112.285
55.404

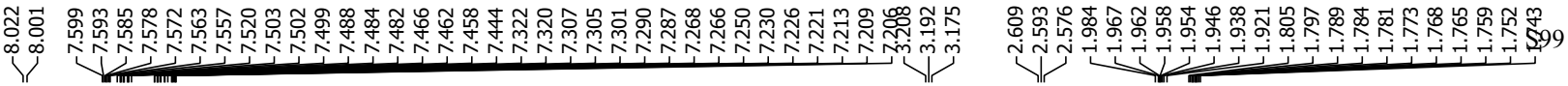
Solvent CDCl3
Spectrometer 100.69





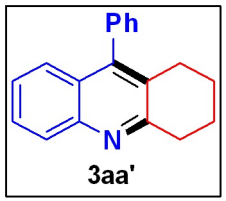


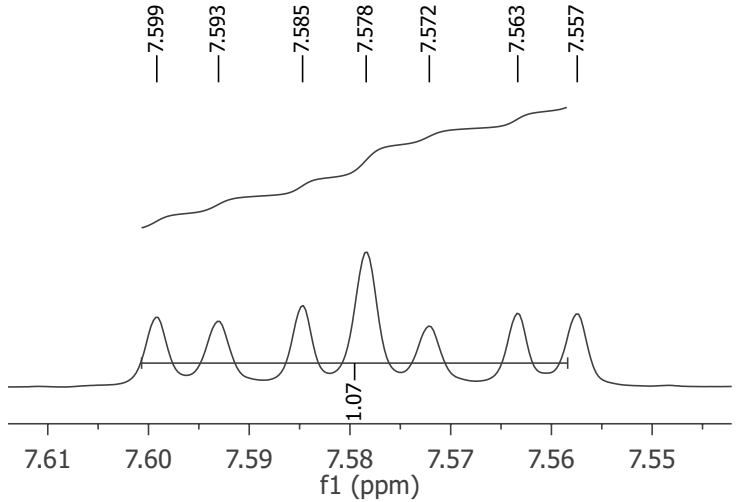

ACF-BF-OMe-2
Pulse Sequence: DEPT
160.093
159.536
146.899
146.667
145.334
131.938
130.744
129.526
127.781
127.709
126.308
125.818
125.685
124.132
123.521
123.238
122.272
114.271
112.297
77.317
77.202
77.000
76.679
55.408
29.685
180
160
140
120
100
80
60
40
20
ppm
O
O
N
3nj

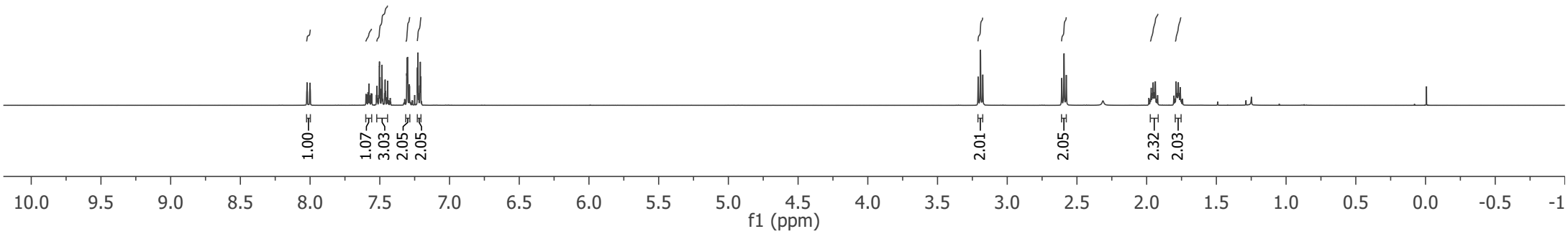


Solvent CDCl3

Spectrometer Frequency 400.40

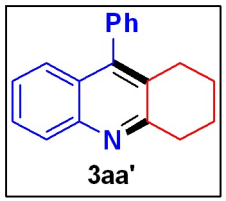


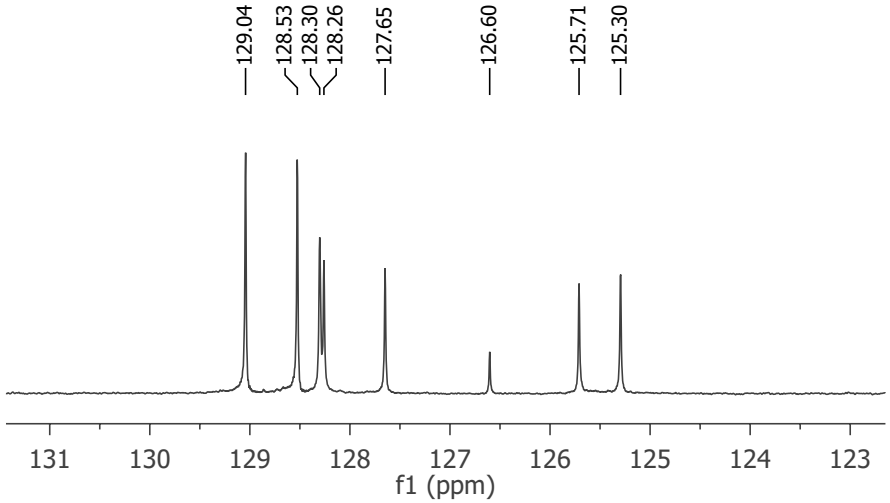


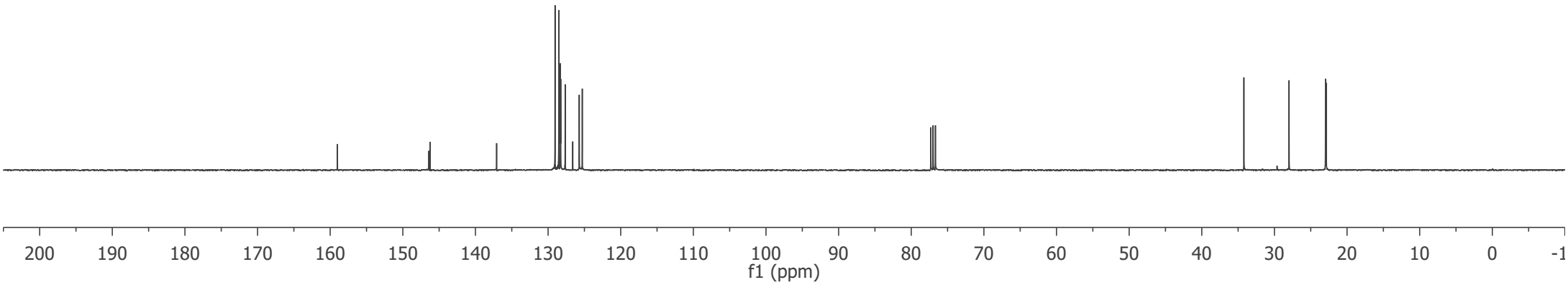


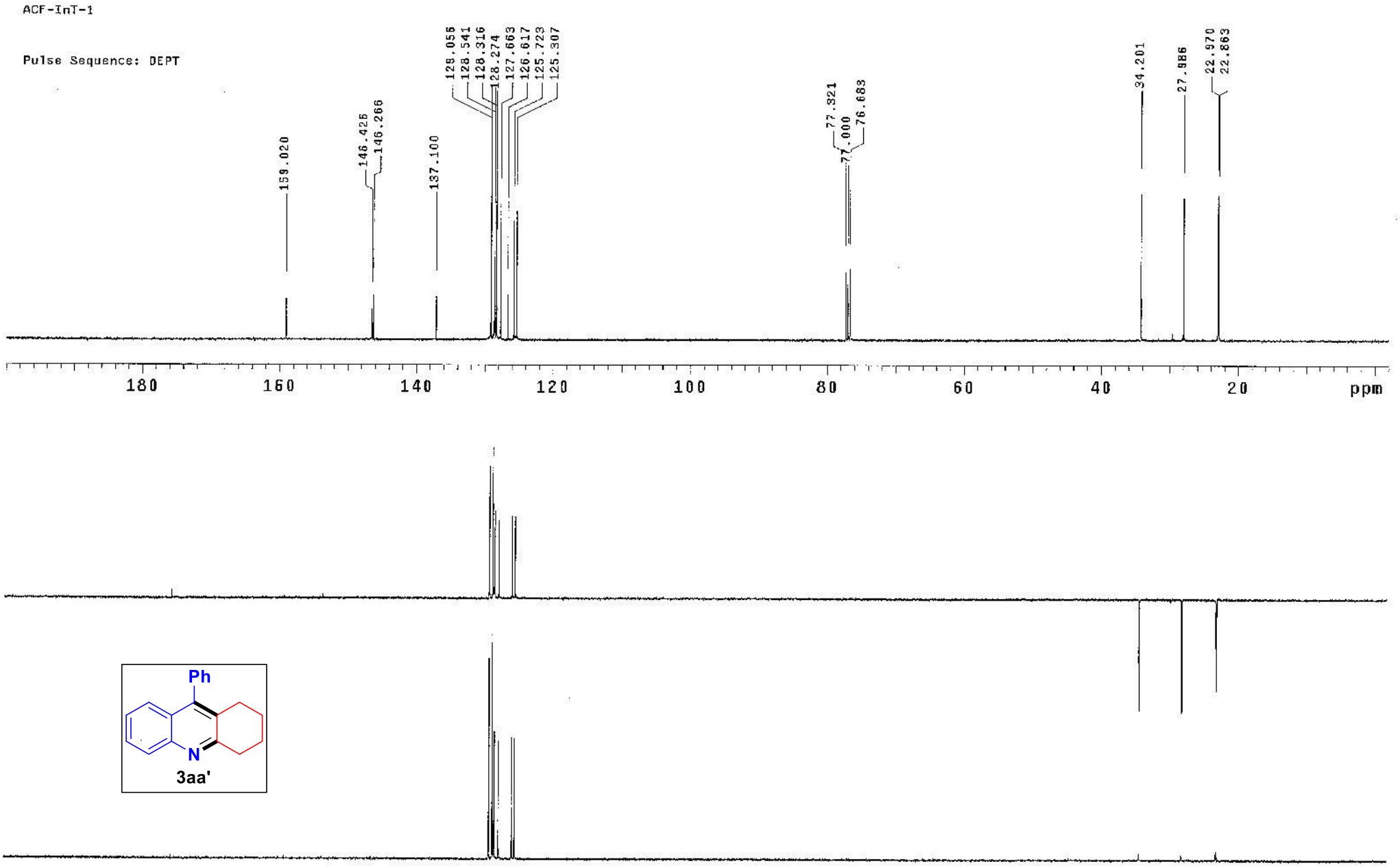
159.00
146.41
146.25
137.08
129.04
128.53
128.30
128.26
127.65
126.60
125.71
125.30
34.20
27.98
22.97
22.86

Solvent CDCl3
Spectrometer Frequency 100.69







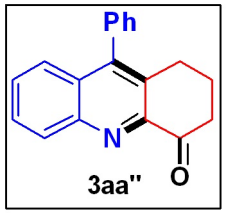

ACF-InT-1
Pulse Sequence: DEPT
159.020
146.425
146.266
137.100
128.055
128.541
128.316
128.274
127.663
126.617
125.723
125.307
77.321
77.000
76.683
34.201
27.986
22.970
22.863
180
160
140
120
100
80
60
40
20
ppm
Ph
N
3aa'

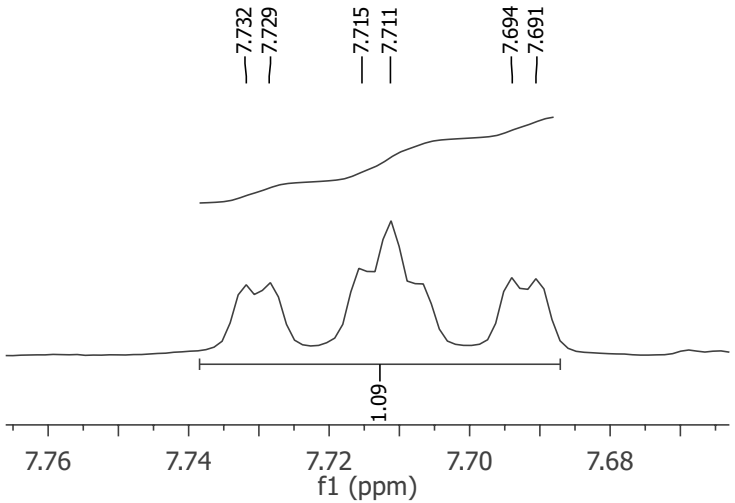
8.391 8.370 7.732 7.729 7.715 7.711 7.694 7.691 7.585 7.581 7.564 7.545 7.536 7.532 7.521 7.515 7.491 7.472 7.442 7.423 7.287 7.283 7.267

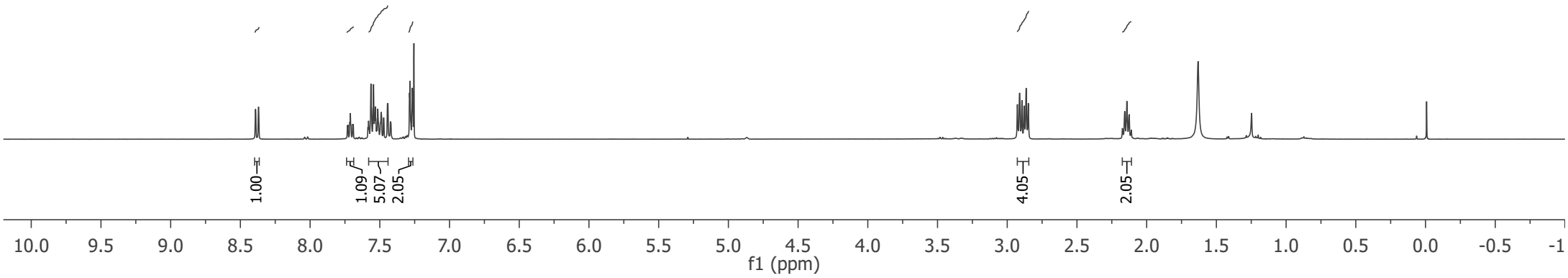
2.927 2.911 2.894 2.879 2.864 2.849 2.174 2.157 2.142 2.126 2.110

Solvent CDCl3

Spectrometer Frequency 399.78



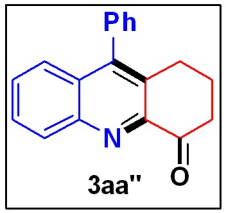




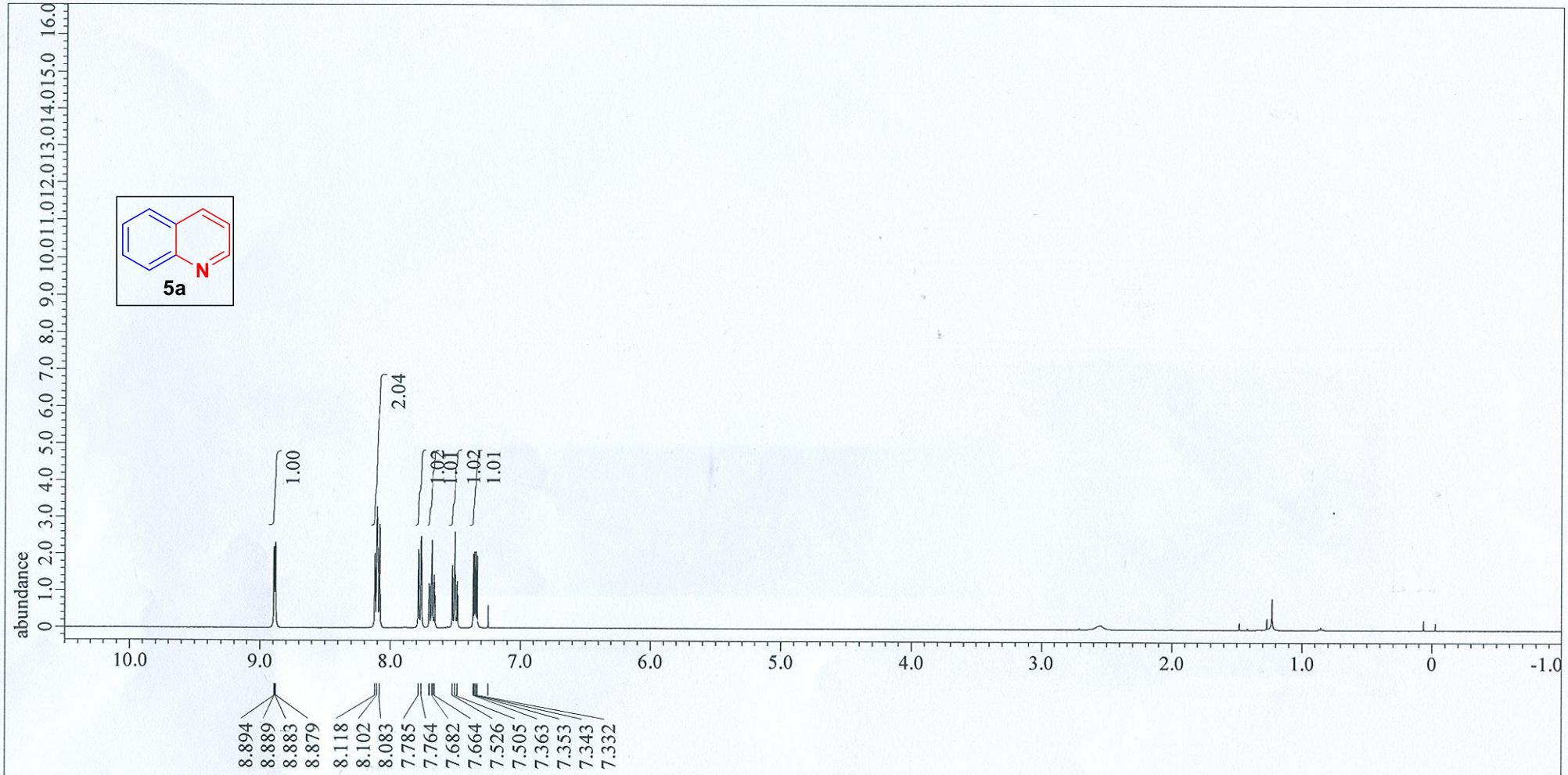
197.58
148.49
148.20
147.25
136.23
133.51
131.59
129.56
129.23
128.90
128.81
128.78
128.43
125.90
40.29
28.06
22.67

Solvent CDCl3

Spectrometer Frequency 100.53

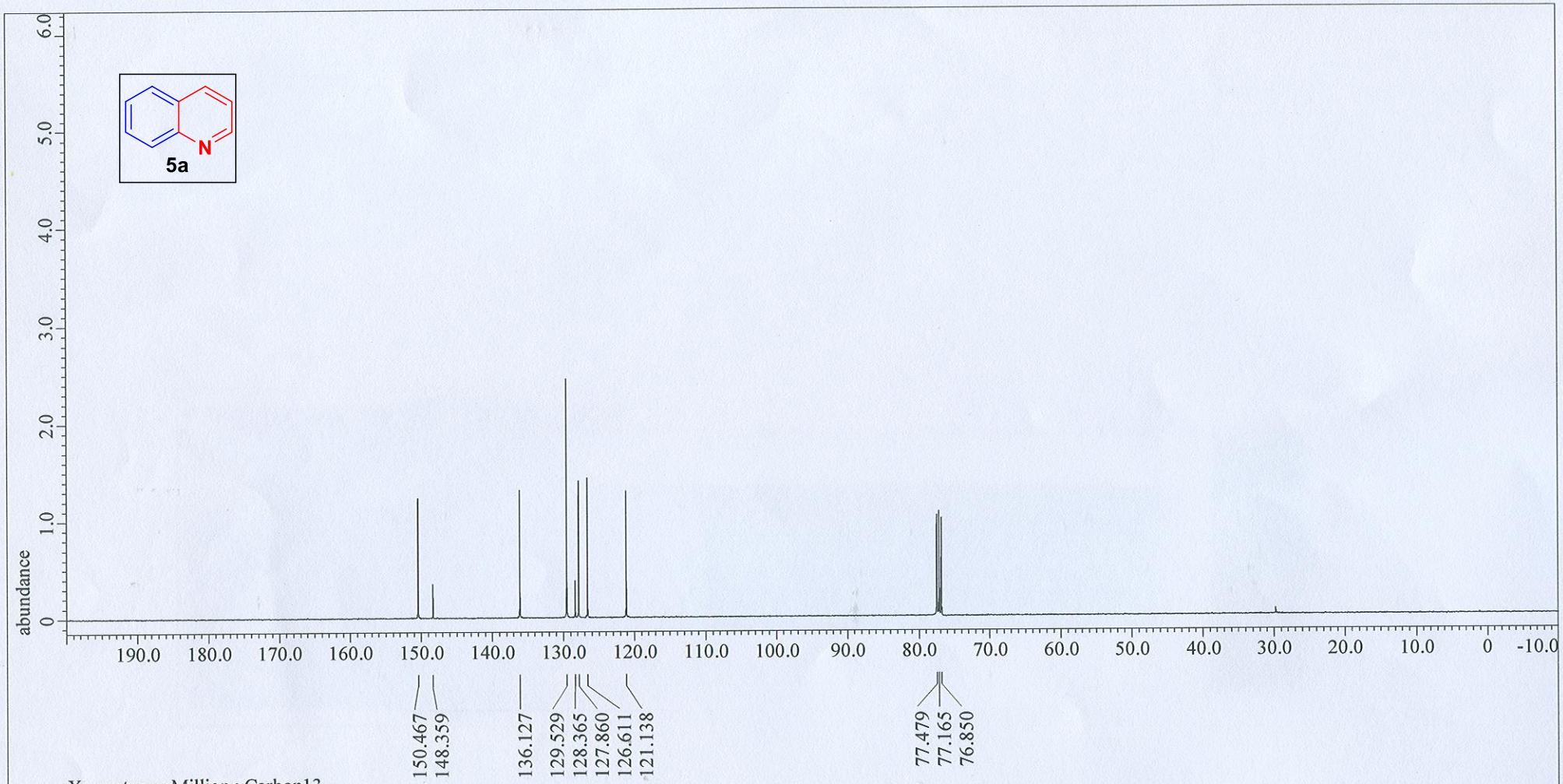


3aa''



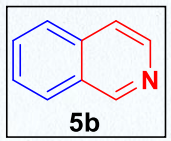
Filename	= ACF-QUINO_Proton-1-4.jdf	Spectrometer	= DELTA2_NMR	Tri_Freq	= 399.78219838[MHz]
Author	= delta			Tri_Offset	= 5[ppm]
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])	Clipped	= FALSE
Sample_Id	= ACF-QUINO	X_Acq_Duration	= 2.18365952[s]	Scans	= 8
Solvent	= CHLOROFORM-D	X_Domain	= 1H	Total_Scans	= 8
Creation_Time	= 6-AUG-2016 13:25:54	X_Freq	= 399.78219838[MHz]		
Revision_Time	= 23-AUG-2016 23:11:41	X_Offset	= 5[ppm]	Relaxation_Delay	= 5[s]
Current_Time	= 23-AUG-2016 23:11:55	X_Points	= 16384	Recvr_Gain	= 32
		X_Prescans	= 1	Temp_Get	= 25.4[dC]
Comment	= single_pulse	X_Resolution	= 0.45794685[Hz]	X_90_Width	= 12.795[us]
Data_Format	= 1D COMPLEX	X_Sweep	= 7.5030012[kHz]	X_Acq_Time	= 2.18365952[s]
Dim_Size	= 13107	X_Sweep_Clipped	= 6.00240096[kHz]	X_Angle	= 45[deg]
Dim_Title	= Proton	Irr_Domain	= Proton	X_Atn	= 2.4[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	X_Pulse	= 6.3975[us]
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Mode	= Off
Site	= JNM-ECS400	Tri_Domain	= Proton	Tri_Mode	= Off

JEOL RESONANCE



Filename	= ACF-QUINO_Carbon-1-3.jdf	Spectrometer	= DELTA2_NMR	Scans	= 1024		
Author	= delta			Total_Scans	= 1024		
Experiment	= carbon.jxp	Field_Strength	= 9.389766[T] (400[MHz])				
Sample_Id	= ACF-QUINO	X_Acq_Duration	= 1.04333312[s]	Relaxation_Delay	= 2[s]		
Solvent	= CHLOROFORM-D	X_Domain	= 13C	Recvr_Gain	= 60		
Creation_Time	= 6-AUG-2016 13:28:56	X_Freq	= 100.52530333[MHz]	Temp_Get	= 25.6[dC]		
Revision_Time	= 23-AUG-2016 23:13:13	X_Offset	= 100[ppm]	X_90_Width	= 10.33[us]		
Current_Time	= 23-AUG-2016 23:13:30	X_Points	= 32768	X_Acq_Time	= 1.04333312[s]		
		X_Prescans	= 4	X_Angle	= 30[deg]		
Comment	= single pulse decoupled gat	X_Resolution	= 0.95846665[Hz]	X_Atn	= 5.3[dB]		
Data_Format	= 1D COMPLEX	X_Sweep	= 31.40703518[kHz]	X_Pulse	= 3.44333333[us]		
Dim_Size	= 26214	X_Sweep_Clipped	= 25.12562814[kHz]	Irr_Atn_Dec	= 21.473[dB]		
Dim_Title	= Carbon13	Irr_Domain	= Proton	Irr_Atn_Noe	= 21.473[dB]		
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	Irr_Noise	= WALTZ		
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Pwidth	= 0.115[ms]		
Site	= JNM-ECS400	Clipped	= FALSE	Decoupling	= TRUE		

JEOL RESONANCE

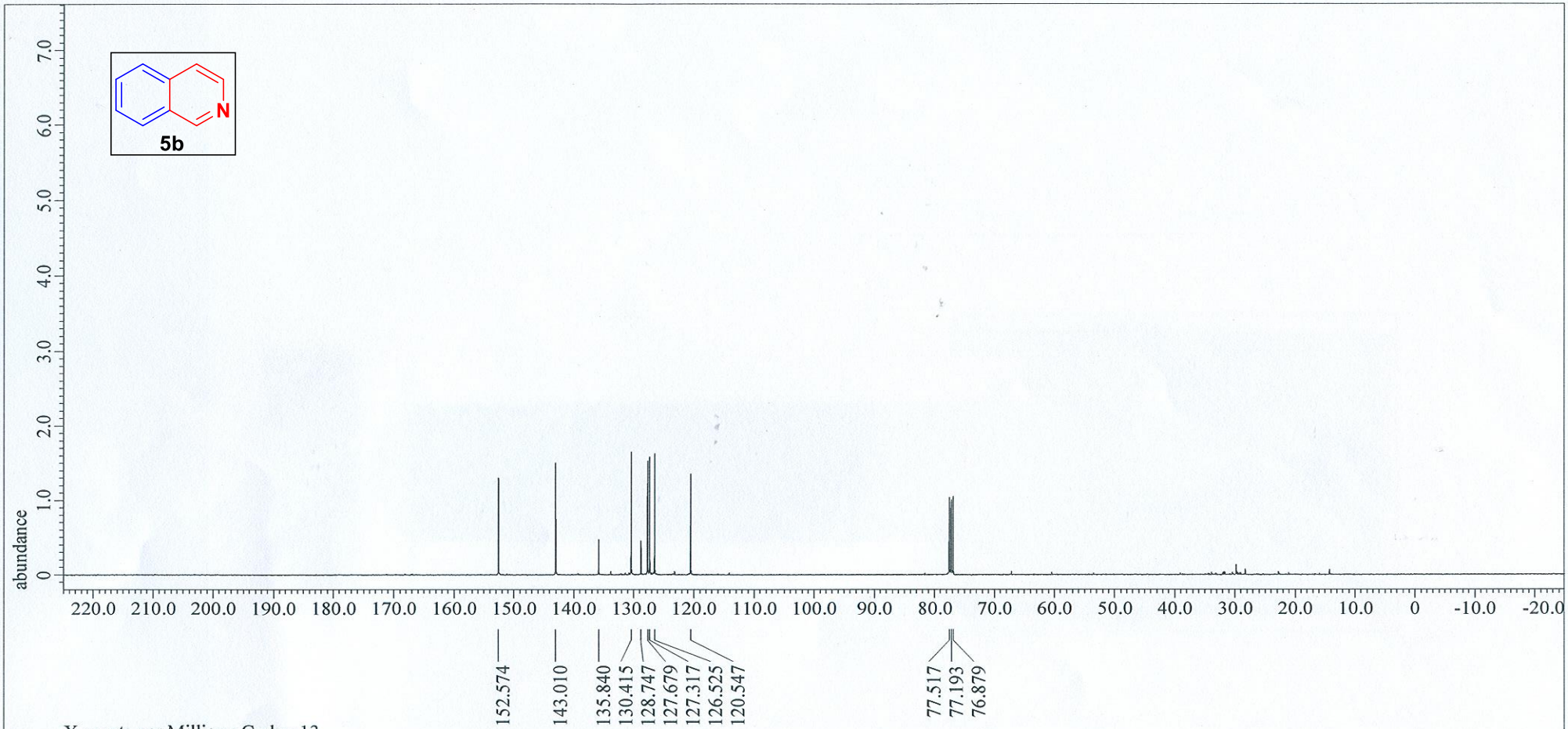


5b

X : parts per Million : Proton

Filename	= acf-oxi-isoqu-3_Proton-1-4	Spectrometer	= DELTA2_NMR	Tri_Freq	= 399.78219838[MHz]
Author	= delta			Tri_Offset	= 5[ppm]
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])	Clipped	= FALSE
Sample_Id	= acf-oxi-isoqu-3	X_Acq_Duration	= 2.18365952[s]	Scans	= 8
Solvent	= CHLOROFORM-D	X_Domain	= 1H	Total_Scans	= 8
Creation_Time	= 17-AUG-2016 23:08:48	X_Freq	= 399.78219838[MHz]		
Revision_Time	= 23-AUG-2016 23:19:20	X_Offset	= 5[ppm]	Relaxation_Delay	= 5[s]
Current_Time	= 23-AUG-2016 23:19:51	X_Points	= 16384	Recvr_Gain	= 30
		X_Prescans	= 1	Temp_Get	= 24.9[dC]
Comment	= single_pulse	X_Resolution	= 0.45794685[Hz]	X_90_Width	= 12.795[us]
Data_Format	= 1D COMPLEX	X_Sweep	= 7.5030012[kHz]	X_Acq_Time	= 2.18365952[s]
Dim_Size	= 13107	X_Sweep_Clipped	= 6.00240096[kHz]	X_Angle	= 45[deg]
Dim_Title	= Proton	Irr_Domain	= Proton	X_Atn	= 2.4[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	X_Pulse	= 6.3975[us]
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Mode	= Off
Site	= JNM-ECS400	Tri_Domain	= Proton	Tri_Mode	= Off

JEOL RESONANCE



Filename	= acf-oxi-isoqu-3_Carbon-1-3	Spectrometer	= DELTA2_NMR	Scans	= 1024
Author	= delta			Total_Scans	= 1024
Experiment	= carbon.jxp	Field_Strength	= 9.389766[T] (400[MHz])		
Sample_Id	= acf-oxi-isoqu-3	X_Acq_Duration	= 1.04333312[s]	Relaxation_Delay	= 2[s]
Solvent	= CHLOROFORM-D	X_Domain	= 13C	Recvr_Gain	= 60
Creation_Time	= 17-AUG-2016 23:15:13	X_Freq	= 100.52530333[MHz]	Temp_Get	= 26.4[dC]
Revision_Time	= 23-AUG-2016 23:14:36	X_Offset	= 100[ppm]	X_90_Width	= 10.33[us]
Current_Time	= 23-AUG-2016 23:15:08	X_Points	= 32768	X_Acq_Time	= 1.04333312[s]
		X_Prescans	= 4	X_Angle	= 30[deg]
Comment	= single pulse decoupled gat	X_Resolution	= 0.95846665[Hz]	X_Atn	= 5.3[dB]
Data_Format	= 1D COMPLEX	X_Sweep	= 31.40703518[kHz]	X_Pulse	= 3.44333333[us]
Dim_Size	= 26214	X_Sweep_Clipped	= 25.12562814[kHz]	Irr_Atn_Dec	= 21.473[dB]
Dim_Title	= Carbon13	Irr_Domain	= Proton	Irr_Atn_Noe	= 21.473[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	Irr_Noise	= WALTZ
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Pwidth	= 0.115[ms]
Site	= JNM-ECS400	Clipped	= FALSE	Decoupling	= TRUE

JEOL RESONANCE