Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

Rh-catalyzed tandem C-C/C-N bond formation of quinoxalines with alkynes leading to heterocyclic ammonium salts

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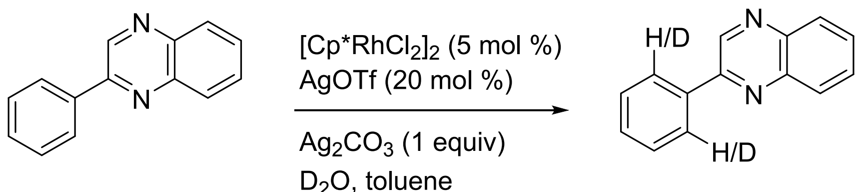
General Information. $[Cp^*RhCl_2]_2$, $AgBF_4$ (98%) and $Cu(OAc)_2{\cdot}H_2O$ (≥98%) were purchased from Aldrich and used as received. 2-Arylqunoxalines **1a-l** and alkynes **2a-l** were prepared according to literature.[1,2] Merck silica gel G/GF 254 plates were used for analytical TLC and Rankem silica gel (60-120 mesh) was used for column chromatography. NMR (^1H and ^{13}C) spectra were recorded in Bruker 300, Bruker Avance III 400 and 600 spectrometers using $CDCl_3$/DMSO-d_6 as a solvent and TMS as an internal standard. Chemical shifts (δ) and spin-spin coupling constant (J) are reported in ppm and in Hz, respectively and other data are reported as follows: s = singlet, d = doublet, dd= doublet of doublet, t = triplet, m = multiplet, q = quartet. PerkinElmer IR instrument was used for recording FT-IR spectra. Using Q-Tof ESI-MS instrument (model HAB 273), mass spectra were recorded. Single crystal X-ray data were collected using Bruker SMART APEX-II CCD diffractometer, which is equipped with 1.75 kW sealed-tube Mo-Kα irradiation (λ = 0.71073 Å) at 296(2) K. The crystal structure was solved by direct method using *SHELLX-97* (Göttingen, Germany) and refined with full-matrix least squares on F^2 using *SHELXL*-97.

Procedure for the Synthesis of Rhodacycle 1I′. To a 10 mL round bottom flask, 2-phenylquinoxaline **1a** (0.25 mmol, 52 mg), $[Cp^*RhCl_2]_2$ (0.10 mmol, 62 mg) and $AgBF_4$ (0.25 mmol, 48.6 mg) were added. The flask was purged with nitrogen three times followed by the addition of 3 mL of $(CH_2Cl)_2$. The mixture was stirred at room temperature for 10 h and was then passed through a short pad of celite using MeOH. The filtrate was concentrated in vacuo and the residue washed with EtOAc (1 x 5 mL). The resultant yellow solid was dried and isolated in 55% yield. The formation of the rhodacycle **1I′** was confirmed by ^1H NMR and HRMS analysis.

Procedure for the Reaction of 1I′ with 1,2-diphenylethyne 2a. To a 10 mL round bottom flask, 1,2-diphenylethyne **2a** (0.05 mmol, 9 mg), **1I′** (0.05 mmol, 26 mg), $AgBF_4$ (0.05 mmol, 9.7 mg) and $Cu(OAc)_2{\cdot}H_2O$ (0.05 mmol, 10 mg) were added in 1.5 mL of $(CH_2Cl)_2$ and stirred at room temperature for 5 h. The reaction mixture was then diluted with CH_2Cl_2 (5 mL) and passed through a short pad of celite using CH_2Cl_2 (3 x 5 mL). The combined filtrate was concentrated in vacuo and the residue was purified on silica gel column chromatography employing CH_2Cl_2/MeOH (96:4) as an eluent to afford **3aa** in 91% yield.

Procedure for the Synthesis of 1a-*d*$_2$.[3] In an oven-dried 10 mL round bottom flask, 2-phenylqunoxaline **1a** (0.25 mmol, 52 mg), $[Cp^*RhCl_2]_2$ (5 mol %, 7.6 mg), AgOTf (0.05 mmol, 12.8 mg), Ag_2CO_3 (0.25 mmol, 68.8 mg), toluene (1.5 mL) and D_2O (0.5 mL) were stirred at 130

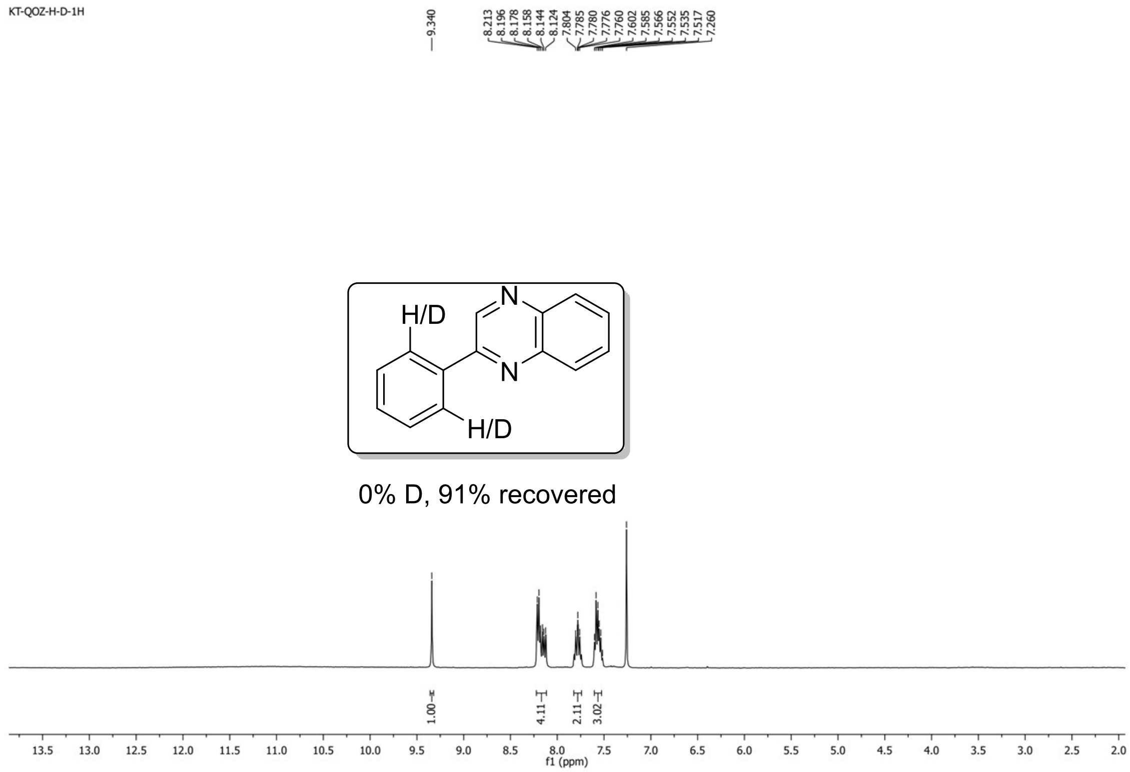
°C for 14 h under air. The solvent was then removed on a rotary evaporator and the residue was dissolved with EtOAc and passed through a short pad of celite. The filtrate was concentrated and the residue was purified on silica gel column chromatography using EtOAc/hexane (1:10) as an eluent to afford **1a-*d*$_2$** as a pale yellow solid. The deuterium incorporation was determined using 400 MHz ^{1}H NMR as 96.5%.



1a **1a-*d*$_2$**, 96.5% deutorium incorporation

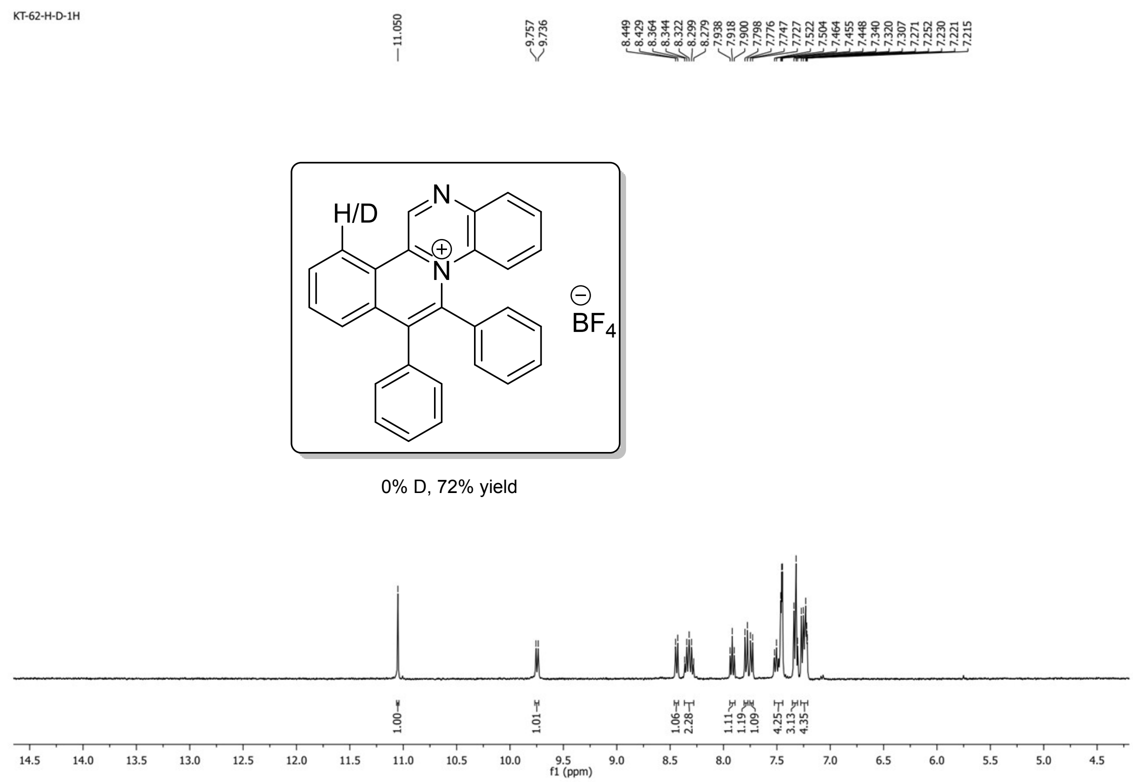
Mechanistic investigation

H/D Exchange Experiment with D_2O. 2-Phenylquinoxaline **1a** (0.25 mmol, 52 mg), [Cp*RhCl$_2$]$_2$ (2.5 mol %, 3.8 mg), $AgBF_4$ (0.25 mmol, 48.6 mg), $Cu(OAc)_2 \cdot H_2O$ (0.25 mmol, 49.9 mg) and D_2O (1 mmol) were stirred at room temperature for 10 h in $(CH_2Cl)_2$ (3 mL) under air. The reaction mixture was then diluted with CH_2Cl_2 (5 mL), and passed through a short pad of celite using CH_2Cl_2 (3 x 5 mL). Drying (Na_2SO_4) and evaporation of the solvent on vacuo produced a residue, which was purified on silica gel column chromatography using EtOAc/hexane (1:10) as eluent. No deutration incorporation was observed from 400 MHz ^{1}H NMR.

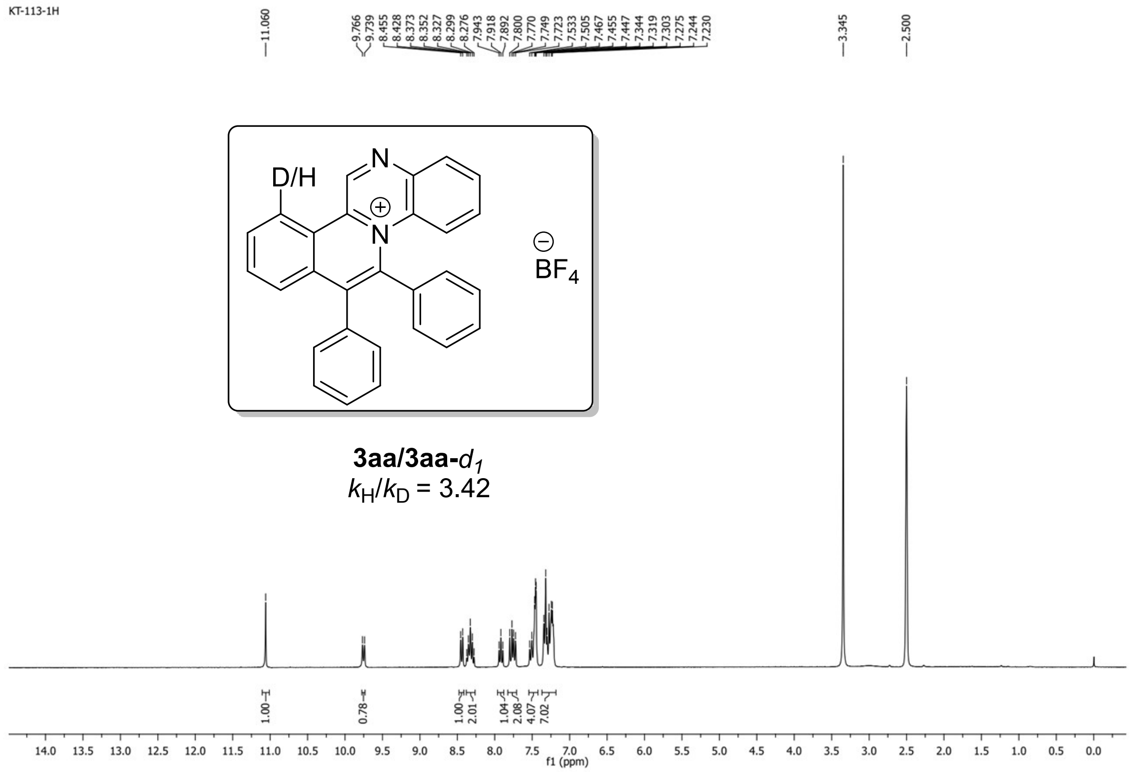


H/D Exchange Experiment with D_2O in Presence of Alkyne

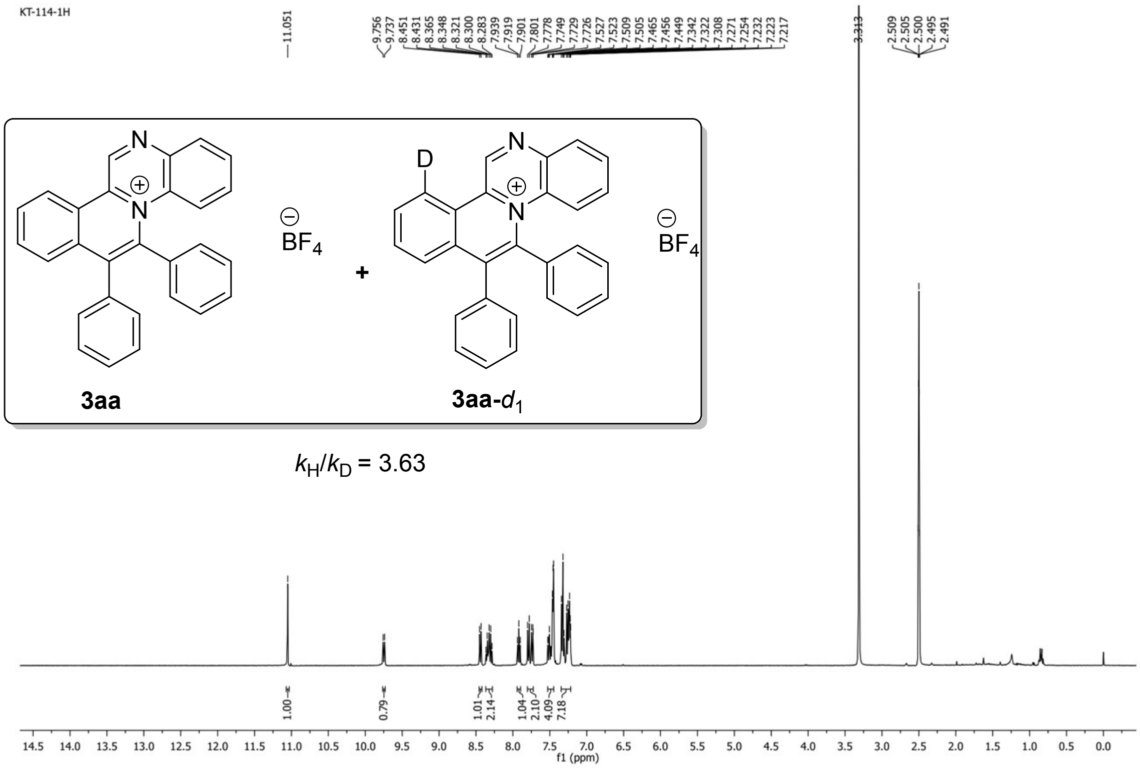
H/D Exchange Experiment with D_2O in Presence of Alkyne. 2-Phenylquinoxaline **1a** (0.25 mmol, 52 mg), 1,2-diphenylethyne **2a** (0.25 mmol, 44.5 mg), $[Cp^*RhCl_2]_2$ (2.5 mol %, 3.8 mg), $AgBF_4$ (0.25 mmol, 48.6 mg), $Cu(OAc)_2{\cdot}H_2O$ (0.25 mmol, 49.9 mg) and D_2O (1 mmol) were stirred at room temperature for 10 h in $(CH_2Cl)_2$ (3 mL) under air. The reaction mixture was diluted with CH_2Cl_2 (5 mL), and passed through a short pad of celite using CH_2Cl_2 (3 x 5 mL). Drying (Na_2SO_4) and evaporation of the solvent on vacuo produced a residue, which was purified on silica gel column chromatography using CH_2Cl_2/MeOH (96:4) as an eluent. No deutration incorporation was observed from 400 MHz ^{1}H NMR.



Competition Isotope Experiment. Compound **1a** (0.25 mmol, 52 mg) and **1a**-d_2 (0.25 mmol, 52 mg) were subjected to the standard reaction conditions for 4 h. The reaction mixture was diluted with CH_2Cl_2 (5 mL) and passed through a short pad of celite using CH_2Cl_2 (3 x 5 mL). The filtrate was concentrated on vacuo and the residue was purified on silica gel column chromatography using CH_2Cl_2/MeOH (96:4) as an eluent. The k_H/k_D was determined using 300 MHz ^{1}H NMR as 3.42.



Parallel Kinetic Isotope Experiment. Compound **1a** (0.25 mmol, 52 mg) and **1a**-d_2 (0.25 mmol, 52 mg) were separately subjected to the standard reaction conditions for 4 h. The reaction mixtures were diluted with CH_2Cl_2 (5 mL). Both the reaction mixtures were combined together and the resultant solution was passed through a short pad of celite using CH_2Cl_2 (3 x 5 mL). The filtrate was concentrated on vacuo and the residue was purified on silica gel column chromatography using CH_2Cl_2/MeOH (96:4) as an eluent. The k_H/k_D was determined using 400 MHz ^{1}H NMR as 3.63.


KT-114-1H
3aa
3aa-d_1
k_H/k_D = 3.63
f1 (ppm)

Crystal Structure and Data of 3aa

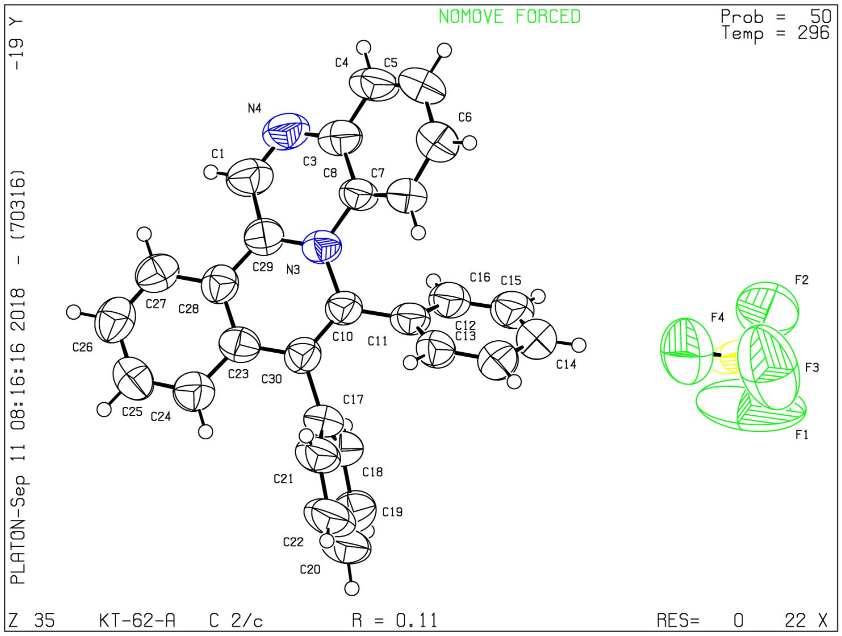
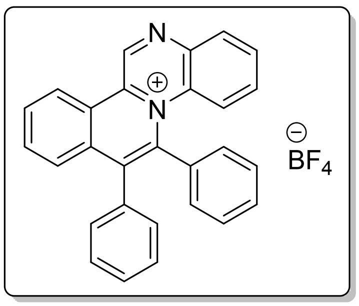


Figure S1. ORTEP diagram of 6,7-Diphenylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate **3aa** with 50% thermal ellipsoids (CCDC 1867227).

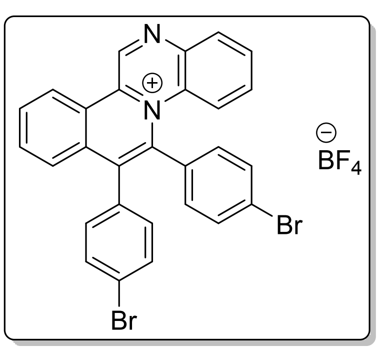
Identification code	**3aa**
Empirical formula	$C_{28}H_{19}N_2BF_4$
Formula weight	470.26
Crystal habit, colour	block/yellow
Crystal size, mm^3	0.32 x 0.25 x 0.16
Temperature, T/K	296(2)
Wavelength, λ/Å	0.71073
Crystal system	monoclinic
Space group	'C 2/c'
Unit cell dimensions	a = 31.900(4) Å b = 9.7621(10) Å c = 15.5417(16) Å $\alpha = \gamma = 90.00°$ $\beta = 107.394(14)$
Volume, V/Å^3	4618.5(9)
Z	8
Calculated density, Mg·m^{-3}	1.353
Absorption coefficient, μ/mm^{-1}	0.101
F(000)	1936
θ range for data collection	1.338 to 25.048 °

Limiting indices	$-37 \leq h \leq 37$, $-11 \leq k \leq 11$, $-18 \leq l \leq 18$
Reflection collected / unique	4093/ 1585 [R(int) = 0.0742]
Completeness to θ	1.000 (θ = 25.048 °)
Refinement method	SHELXL-97 (Sheldrick, 1997)
Data / restraints / parameters	4093 / 0 / 316
Goodness–of–fit on F^2	1.349
Final R indices [I>2sigma(I)]	R1 = 0.1092, wR2 = 0.2628
R indices (all data)	R1 = 0.2352, wR2 = 0.3187

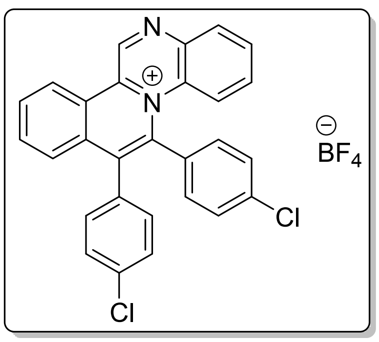
Characterization data



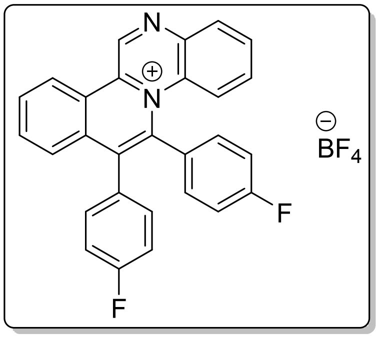
6,7-Diphenylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3aa. Yellow solid; yield 98% (115 mg); ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.04 (s, 1H), 9.75 (d, J = 8.4 Hz, 1H), 8.44 (d, J = 8.0 Hz, 1H), 8.34-8.29 (m, 2H), 7.91 (t, J = 8.0 Hz, 1H), 7.79-7.72 (m, 2H), 7.52-7.44 (m, 4H), 7.33-7.21 (m, 7H); ^{13}C NMR (150 MHz, DMSO-d_6/$CDCl_3$, 1:10) δ 144.95, 144.93, 142.0, 141.6, 140.2, 136.1, 136.0, 135.7, 134.3, 132.6, 132.1, 130.8, 130.4, 129.7, 129.44, 129.42, 128.5, 128.4, 128.0, 127.4, 127.1, 125.8, 124.4, 123.4; FT-IR (KBr) 2924, 2853, 1612, 1572, 1520, 1495, 1464, 1345, 1261, 1061, 766, 698 cm^{-1}; HRMS (ESI) *m/z* $[M\text{-}BF_4]^+$ calcd for $C_{28}H_{19}N_2^+$: 383.1543, found: 383.1548.



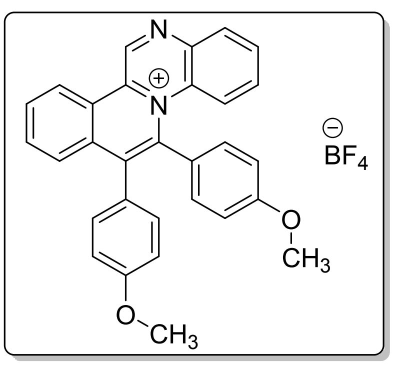
6,7-Bis(4-bromophenyl)isoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3ab. Yellow solid; yield 95% (149 mg); ^{1}H NMR (400 MHz, DMSO-d$_6$) δ 11.05 (s, 1H), 9.75 (d, J = 8.0 Hz, 1H), 8.47-8.44 (m, 1H), 8.38-8.28 (m, 2H), 7.97-7.93 (m, 1H), 7.79 (d, J = 8.4 Hz, 1H), 7.75-7.70 (m, 3H), 7.64-7.59 (m, 1H), 7.56-7.53 (m, 2H), 7.31-7.28 (m, 2H), 7.21-7.18 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d$_6$) δ 147.1, 141.7, 139.7, 139.3, 137.8, 137.1, 135.4, 134.3, 132.8, 132.5, 132.4, 132.1, 131.8, 131.7, 131.5, 130.1, 127.38, 127.31, 127.0, 124.8, 124.2, 123.4, 122.4; FT-IR (KBr) 2924, 2853, 1590, 1490, 1437, 1396, 1346, 1250, 1081, 1058, 1013, 820, 775, 735 cm^{-1}; HRMS (ESI) *m/z* [M-BF$_4$]$^+$ calcd for $C_{28}H_{17}Br_2N_2^+$: 540.9733, found: 540.9753.



6,7-Bis(4-chlorophenyl)isoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3ac. Yellow solid; yield 92% (124 mg); ^{1}H NMR (600 MHz, DMSO-d$_6$) δ 11.06 (s, 1H), 9.75 (d, J = 8.4 Hz, 1H), 8.46 (d, J = 7.8 Hz, 1H), 8.36 (t, J = 8.4 Hz, 1H), 8.31 (t, J = 7.8 Hz, 1H), 7.96-7.94 (m, 1H), 7.79 (d, J = 8.4 Hz, 1H), 7.75 (d, J = 8.4 Hz, 1H), 7.61 (t, J = 7.8 Hz, 1H), 7.59-7.57 (m, 2H), 7.41-7.36 (m, 4H), 7.27 (d, J = 8.4 Hz, 2H); ^{13}C NMR (150 MHz, DMSO-d$_6$) δ 147.1, 141.7, 139.7, 139.4, 137.8, 137.1, 135.4, 134.5, 134.0, 133.7, 132.46, 132.42, 131.8, 131.4, 130.1, 128.9, 128.8, 127.37, 127.31, 127.0, 124.8, 124.2; FT-IR (KBr) 2924, 2853, 1598, 1519, 1493, 1439, 1347, 1212, 1083, 1061, 968, 823, 769, 755, 669 cm^{-1}; HRMS (ESI) *m/z* [M-BF$_4$]$^+$ calcd for $C_{28}H_{17}Cl_2N_2^+$: 451.0763, found: 451.0769.

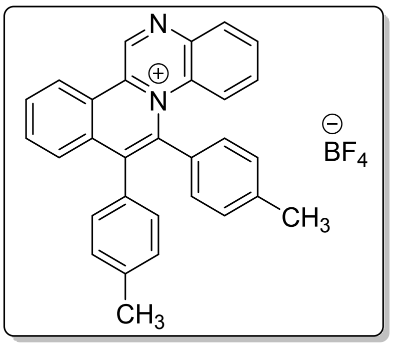


6,7-Bis(4-fluorophenyl)isoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3ad. Yellow solid; yield 91% (115 mg); ^{1}H NMR (600 MHz, DMSO-d$_6$) δ 11.04 (s, 1H), 9.74 (d, *J* = 8.4 Hz, 1H), 8.45 (d, *J* = 8.4 Hz, 1H), 8.36-8.29 (m, 2H), 7.93 (t, *J* = 7.8 Hz, 1H), 7.76 (t, *J* = 7.8 Hz, 2H), 7.58 (t, *J* = 8.4 Hz, 1H), 7.39 (s, 2H), 7.33 (t, *J* = 8.4 Hz, 2H), 7.27 (s, 1H), 7.16 (t, *J* = 8.4 Hz, 2H), 6.56 (s, 1H); ^{13}C NMR (150 MHz, DMSO-d$_6$) δ 162.9 (J_{C-F} = 247.6 Hz), 162.7 (J_{C-F} = 245.1 Hz), 147.2, 147.0, 141.6, 140.0, 139.8, 137.6, 136.9, 135.6, 133.0, 132.28, 132.20, 131.7, 131.3, 130.0, 129.9, 127.4, 127.2 (J_{C-F} = 6.7 Hz), 127.0 (J_{C-F} = 5.4 Hz), 124.7, 124.1, 115.9 (J_{C-F} = 22.2 Hz), 115.8 (J_{C-F} = 24.1 Hz); FT-IR (KBr) 2924, 2854, 1604, 1509, 1437, 1375, 1336, 1230, 1162, 1059, 969, 832, 776, 698 cm^{-1}; HRMS (ESI) *m/z* [M-BF$_4$]$^+$ calcd for $C_{28}H_{17}F_2N_2^+$: 419.1354, found: 419.1356.

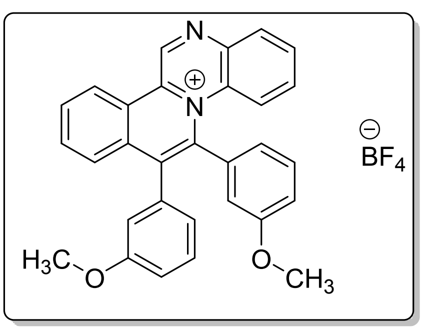


6,7-Bis(4-methoxyphenyl)isoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3ae. Yellow solid; yield 79% (105 mg); ^{1}H NMR (600 MHz, DMSO-d$_6$) δ 10.97 (s, 1H), 9.68 (d, *J* = 8.4 Hz, 1H), 8.41 (d, *J* = 8.4 Hz, 1H), 8.30-8.25 (m, 2H), 7.90 (t, *J* = 7.8 Hz, 1H), 7.80 (d, *J* = 9.0 Hz, 1H), 7.75 (d, *J* = 7.8 Hz, 1H), 7.52 (t, *J* = 8.4 Hz, 1H), 7.24 (d, *J* = 9.0 Hz, 2H), 7.15 (d, *J* = 8.4 Hz, 2H), 7.04 (d, *J* = 9.0 Hz, 2H), 6.84 (d, *J* = 9.0 Hz, 2H), 3.80 (s, 3H), 3.71 (s, 3H); ^{13}C NMR (150

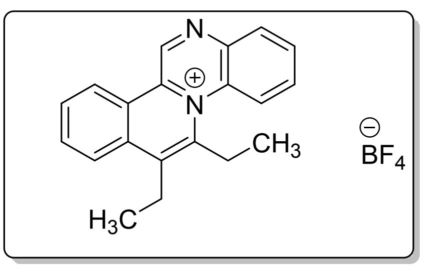
MHz, DMSO-d_6) δ 159.7, 159.2, 147.1, 141.6, 141.1, 140.9, 137.5, 136.7, 136.4, 132.1, 132.0, 131.4, 131.2, 130.1, 129.7, 127.99, 127.92, 127.1, 127.0, 125.8, 124.8, 124.0, 114.18, 114.13, 55.38, 55.31; FT-IR (KBr) 2960, 2925, 2839, 1611, 1573, 1512, 1439, 1335, 1246, 1179, 1061, 1029, 964, 826, 797 cm^{-1}; HRMS (ESI) *m/z* [M-BF_4]$^+$ calcd for $C_{30}H_{23}O_2N_2^+$: 443.1754, found: 443.1764.



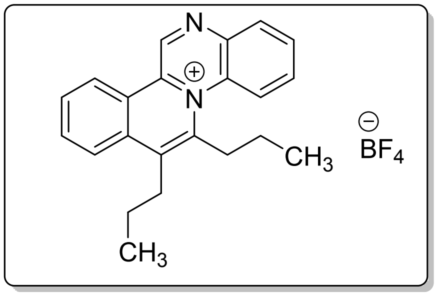
6,7-Di-*p*-tolylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3af. Yellow solid; yield 83% (103 mg); ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.01 (s, 1H), 9.72 (d, *J* = 8.4 Hz, 1H), 8.42 (d, *J* = 8.0 Hz, 1H), 8.33-8.25 (m, 2H), 7.90 (t, *J* = 7.6 Hz, 1H), 7.78 (d, *J* = 8.8 Hz, 1H), 7.71 (d, *J* = 8.0 Hz, 1H), 7.53-7.48 (m, 1H), 7.29 (d, *J* = 7.6 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 7.10 (q, *J* = 6.8 Hz, 4H), 2.37 (s, 3H), 2.25 (s, 3H); ^{13}C NMR (150 MHz, DMSO-d_6/$CDCl_3$,1:10) δ 144.8, 142.2, 141.6, 140.5, 139.7, 138.3, 136.3, 135.9, 135.6, 131.9, 131.5, 130.6, 130.3, 129.6, 129.2, 129.0, 128.7, 127.4, 127.3, 125.7, 124.3, 123.4, 20.85, 20.80; FT-IR (KBr) 2927, 2838, 1611, 1573, 1512, 1465, 1439, 1335, 1288, 1185, 1060, 1028, 964, 826, 772, 755 cm^{-1}; HRMS (ESI) *m/z* [M-BF_4]$^+$ calcd for $C_{30}H_{23}N_2^+$: 411.1856, found: 411.1860.



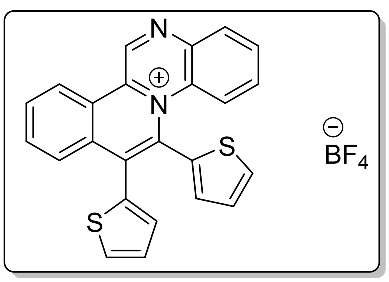
6,7-Bis(3-methoxyphenyl)isoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3ag. (Rotational isomers 1:1) Yellow solid; yield 82% (109 mg); ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.04 (s, 2H), 9.74 (d, *J* = 8.4 Hz, 2H), 8.45 (d, *J* = 8.4 Hz, 2H), 8.35-8.29 (m, 5H), 7.94 (d, *J* = 7.8 Hz, 1H), 7.92-7.89 (m, 2H), 7.81 (d, *J* = 7.8 Hz, 2H), 7.56 (t, *J* = 8.4 Hz, 2H), 7.42 (t, *J* = 7.8 Hz, 1H), 7.35 (t, *J* = 7.8 Hz, 1H), 7.19 (t, *J* = 7.8 Hz, 1H), 7.15 (t, *J* = 7.8 Hz, 1H), 7.08 (s, 1H), 7.04 (d, *J* = 8.4 Hz, 2H), 6.97 (d, *J* = 6.6 Hz, 2H), 6.92-6.88 (m, 4H), 6.84 (d, *J* = 7.2 Hz, 1H), 6.70 (s, 1H), 6.68 (d, *J* = 7.2 Hz, 1H), 3.73 (s, 3H), 3.66 (s, 3H), 3.54 (s, 3H), 3.52 (s, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 159.09, 159.06, 159.03, 158.9, 147.1, 141.6, 140.48, 140.45, 140.1, 137.6, 136.9, 136.42, 136.40, 135.6, 135.07, 135.05, 132.3, 131.3, 130.1, 130.0, 129.9, 129.89, 129.82, 127.7, 127.1, 124.8, 123.9, 122.6, 122.5, 122.4, 122.0, 115.98, 115.92, 115.87, 115.84, 115.7, 115.6, 114.2, 55.3, 55.2; FT-IR (KBr) 2924, 2853, 1601, 1589, 1519, 1458, 1425, 1322, 1264, 1157, 1059, 940, 769, 695 cm^{-1}; HRMS (ESI) *m/z* $[M-BF_4]^+$ calcd for $C_{30}H_{23}O_2N_2^+$: 443.1754, found: 443.1752.



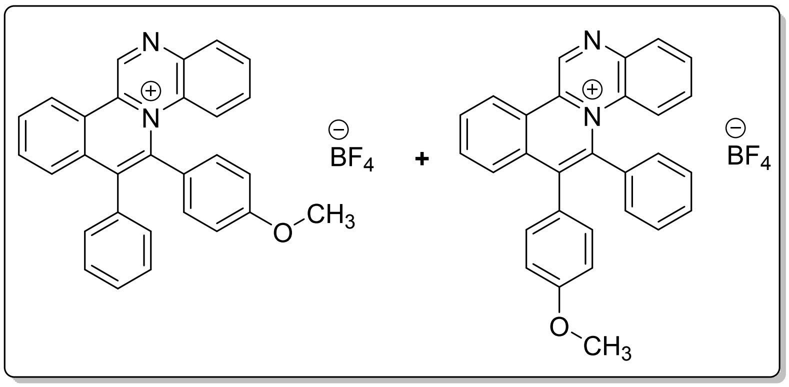
6,7-Diethylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3ah. Yellow solid; yield 74% (69 mg); ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.79 (s, 1H), 9.53 (d, *J* = 8.8 Hz, 1H), 8.61 (t, *J* = 8.0 Hz, 2H), 8.48 (d, *J* = 8.0 Hz, 1H), 8.36 (t, *J* = 8.0 Hz, 1H), 8.22 (t, *J* = 7.6 Hz, 1H), 8.12 (t, *J* = 8.0 Hz, 1H), 8.05 (t, *J* = 8.0 Hz, 1H), 3.68 (q, *J* = 7.2 Hz, 2H), 3.50 (q, *J* = 7.2 Hz, 2H), 1.41 (t, *J* = 7.2 Hz, 3H), 1.12 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 146.9, 143.8, 142.0, 141.1, 136.9, 136.6, 134.7, 131.7, 131.3, 130.6, 129.7, 127.5, 127.1, 125.0, 124.3, 124.0, 26.9, 22.5, 14.9, 13.8; FT-IR (KBr) 2924, 2853, 1610, 1589, 1517, 1435, 1384, 1335, 1212, 1129, 1092, 1050, 898, 781, 761, 699 cm^{-1}; HRMS (ESI) *m/z* $[M-BF_4]^+$ calcd for $C_{20}H_{19}N_2^+$: 287.1543, found: 287.1546.



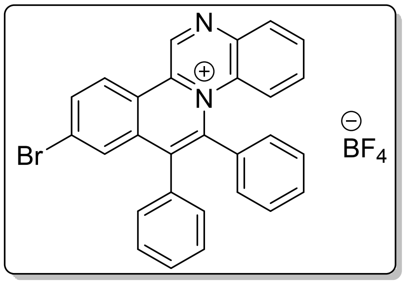
6,7-Dipropylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3ai. Yellow solid; yield 72% (72 mg); ^{1}H NMR (400 MHz, DMSO-d$_6$) δ 10.81 (s, 1H), 9.54 (d, *J* = 8.4 Hz, 1H), 8.62 (q, *J* = 6.0 Hz, 2H), 8.48 (q, *J* = 6.8 Hz, 1H), 8.36 (t, *J* = 7.6 Hz, 1H), 8.22 (t, *J* = 8.0 Hz, 1H), 8.13 (t, *J* = 7.6 Hz, 1H), 8.06 (t, *J* = 8.8 Hz, 1H), 3.67 (t, *J* = 8.0 Hz, 2H), 3.43 (t, *J* = 8.0 Hz, 2H), 1.83 (q, *J* = 8.0 Hz, 2H), 1.37 (q, *J* = 7.6 Hz, 2H), 1.19 (t, *J* = 7.2 Hz, 3H), 0.70 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d$_6$) δ 147.0, 142.9, 141.26, 141.21, 136.9, 136.8, 135.2, 132.0, 131.6, 130.9, 129.9, 127.7, 127.2, 125.4, 124.3, 124.2, 35.6, 31.3, 24.2, 22.7, 14.3, 13.8; FT-IR (KBr) 2924, 1610, 1588, 1516, 1459, 1435, 1384, 1335, 1262, 1084, 1050, 898, 781, 760 cm^{-1}; HRMS (ESI) *m/z* [M-BF$_4$]$^+$ calcd for $C_{22}H_{23}N_2^+$: 315.1856, found: 315.1857.



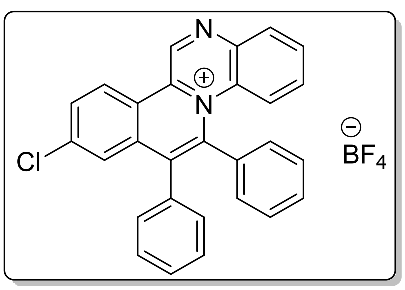
6,7-Di(thiophen-2-yl)isoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3aj. Yellow solid; yield 67% (81 mg); ^{1}H NMR (400 MHz, DMSO- d$_6$) δ 10.97 (s, 1H), 9.69-9.66 (m, 1H), 8.44 (q, *J* = 7.2 Hz, 1H), 8.35-8.32 (m, 2H), 7.96-7.93 (m, 3H), 7.87 (d, *J* = 6.0 Hz, 1H), 7.81 (d, *J* = 6.0 Hz, 1H), 7.63-7.59 (m, 1H), 7.47-7.46 (m, 1H), 7.32-7.29 (m, 1H), 7.20-7.19 (m, 1H), 7.02-7.00 (m, 1H); ^{13}C NMR (100 MHz, DMSO-d$_6$) δ 147.0, 141.5, 138.2, 137.2, 136.5, 136.2, 135.6, 135.5, 133.4, 132.68, 132.60, 132.2, 132.1, 131.4, 130.16, 130.13, 129.9, 128.0, 127.5, 127.3, 127.2, 127.0, 124.8, 122.6; FT-IR (KBr) 2927, 2845, 1578, 1509, 1481, 1443, 1321, 1201, 1067, 964, 812, 783, 721, 651 cm^{-1}; HRMS (ESI) *m/z* $[M\text{-}BF_4]^+$ calcd for $C_{24}H_{15}S_2N_2^+$: 395.0671, found: 395.0683.



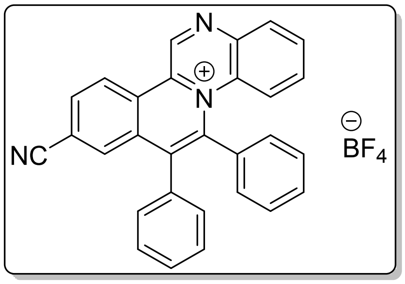
6-(4-Methoxyphenyl)-7-phenylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate and 7-(4-Methoxyphenyl)-6-phenylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate.(3:2) 3al. Yellow solid; yield 84% (105 mg); ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.03 (s, 0.9H), 11.01 (s, 0.6H), 9.72 (t, *J* = 7.8 Hz, 1.6H), 8.43 (t, *J* = 7.8 Hz, 1.6H), 8.34-8.27 (m, 3.4 H), 7.91 (t, *J* = 7.8 Hz, 1.7H), 7.83 (d, *J* = 9.0 Hz, 0.7H), 7.79 (d, *J* = 9.0 Hz, 2H), 7.72 (d, *J* = 9.0 Hz, 0.7H), 7.54 (t, *J* = 9.0 Hz, 0.8H), 7.51-7.48 (m, 3.2H), 7.34 (t, *J* = 7.2 Hz, 3.3H), 7.28 (t, *J* = 7.8 Hz, 2.2H), 7.25 (d, *J* = 9.0 Hz, 2.9H), 7.16 (d, *J* = 8.4 Hz, 2.1H), 7.03 (d, *J* = 8.4 Hz, 2.1H), 6.82 (d, *J* = 9.0 Hz, 1.4H), 3.79 (s, 3H), 3.70 (s, 1.9H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 159.7, 159.2, 147.2, 141.7, 140.96, 140.91, 140.88, 140.86, 137.69, 137.60, 136.8, 136.3, 135.8, 135.5, 133.9, 132.2, 132.15, 132.10, 131.4, 131.3, 131.2, 130.6, 130.15, 130.13, 130.0, 129.8, 129.7, 129.6, 128.7, 128.6, 127.9, 127.7, 127.2, 127.19, 127.17, 127.0, 125.6, 124.9, 124.8, 124.2, 124.0, 114.1, 114.0, 55.36, 55.31; FT-IR (KBr) 2955, 2924, 2853, 1609, 1586, 1512, 1439, 1397, 1337, 1292, 1249, 1177, 1059, 1028, 771, 698 cm^{-1}; HRMS (ESI) *m/z* $[M\text{-}BF_4]^+$ calcd for $C_{29}H_{21}ON_2^+$: 413.1648, found: 413.1654.



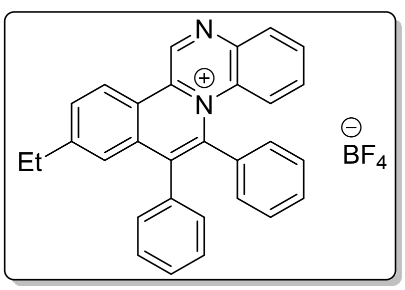
9-Bromo-6,7-diphenylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3ba. Yellow solid; yield 97% (133 mg); ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.02 (s, 1H), 9.67 (d, J = 9.0 Hz, 1H), 8.55 (d, J = 10.2 Hz, 1H), 8.45 (d, J = 8.4 Hz, 1H), 7.93 (t, J = 7.8 Hz, 1H), 7.77-7.75 (m, 2H), 7.52-7.46 (m, 4H), 7.32 (t, J = 9.0 Hz, 3H), 7.26-7.23 (m, 4H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 147.1, 141.8, 141.7, 139.4, 137.7, 136.6, 135.2, 135.1, 133.1, 131.8, 131.5, 131.1, 130.4, 130.1, 130.0, 129.8, 129.3, 129.2, 129.0, 128.8, 128.7, 127.6, 124.0, 123.8; FT-IR (KBr) 2923, 2853, 1596, 1519, 1462, 1445, 1396, 1328, 1261, 1083, 1030, 785, 759 cm^{-1}; HRMS (ESI) *m/z* $[M-BF_4]^+$ calcd for $C_{28}H_{18}BrN_2^+$: 461.0648, found: 461.0639.



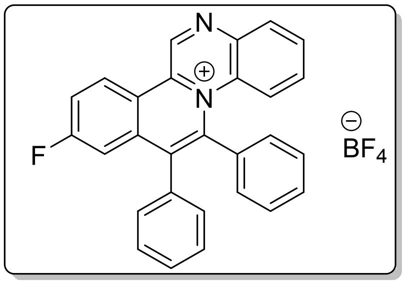
9-Chloro-6,7-diphenylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3ca. Yellow solid; yield 91% (114 mg); ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.01 (s, 1H), 9.76 (d, J = 9.2 Hz, 1H), 8.45-8.40 (m, 2H), 7.92 (t, J = 8.0 Hz, 1H), 7.78 (d, J = 9.2 Hz, 1H), 7.59 (s, 1H), 7.47-7.46 (m, 3H), 7.32-7.23 (m, 8H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 147.1, 142.2, 141.86, 141.80, 139.6, 137.6, 136.8, 135.1, 133.2, 132.6, 131.5, 131.2, 130.5, 130.2, 130.07, 130.06, 129.8, 129.7, 129.0, 128.7, 127.6, 125.7, 124.1, 123.6; FT-IR (KBr) 2924, 2854, 1607, 1519, 1462, 1377, 1083, 824, 759, 700 cm^{-1}; HRMS (ESI) *m/z* $[M-BF_4]^+$ calcd for $C_{28}H_{18}ClN_2^+$: 417.1153, found: 417.1160.



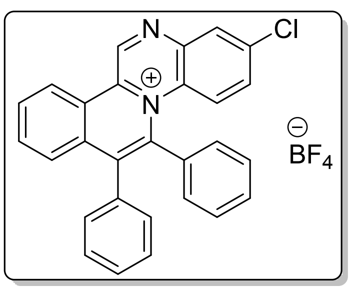
9-Cyano-6,7-diphenylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3da. Yellow solid; yield 75% (93 mg); ^{1}H NMR (600 MHz, DMSO-d$_6$) δ 11.11 (s, 1H), 9.89 (d, J = 8.4 Hz, 1H), 8.70 (d, J = 10.2 Hz, 1H), 8.49 (d, J = 7.8 Hz, 1H), 8.10 (s, 1H), 7.97 (t, J = 7.8 Hz, 1H), 7.83 (d, J = 9.0 Hz, 1H), 7.55 (t, J = 8.4 Hz, 1H), 7.48-7.47 (m, 3H), 7.34-7.31 (m, 3H), 7.26-7.23 (m, 4H); ^{13}C NMR (150 MHz, DMSO-d$_6$) δ 147.2, 142.4, 142.0, 140.4, 137.4, 135.1, 134.8, 132.9, 132.7, 132.09, 132.06, 130.4, 130.3, 130.2, 130.1, 129.9, 129.1, 128.7, 128.6, 128.5, 127.6, 126.7, 124.2, 118.1, 117.3; FT-IR (KBr) 2923, 2853, 1618, 1588, 1517, 1462, 1439, 1372, 1330, 1131, 1084, 1028, 970, 831, 762, 699 cm^{-1}; HRMS (ESI) *m/z* [M-BF$_4$]$^+$ calcd for $C_{29}H_{18}N_3^+$: 408.1495, found: 408.1500.



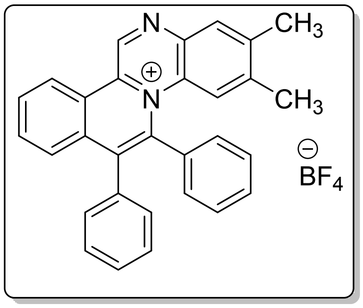
9-Ethyl-6,7-diphenylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3ea. Yellow solid; yield 88% (110 mg); ^{1}H NMR (600 MHz, DMSO-d$_6$) δ 10.98 (s, 1H), 9.66 (d, J = 9.0 Hz, 1H), 8.41 (d, J = 9.0 Hz, 1H), 8.25 (d, J = 9.0 Hz, 1H), 7.88 (t, J = 8.4 Hz, 1H), 7.75 (d, J = 9.0 Hz, 1H), 7.48-7.44 (m, 5H), 7.31 (t, J = 8.4 Hz, 3H), 7.25-7.21 (m, 4H), 2.89 (q, J = 7.2 Hz, 2H), 1.19 (t, J = 7.8 Hz, 3H); ^{13}C NMR (150 MHz, DMSO-d$_6$) δ 154.3, 147.3, 141.5, 140.7, 140.4, 137.4, 136.3, 135.4, 133.8, 133.2, 131.1, 130.6, 130.1, 130.0, 129.7, 129.6, 128.79, 128.72, 128.6, 127.7, 127.3, 124.7, 124.1, 123.4, 29.0, 14.9; FT-IR (KBr) 2963, 2924, 2854, 1614, 1571, 1523, 1462, 1436, 1331, 1213, 1144, 1056, 928, 832, 769, 700 cm^{-1}; HRMS (ESI) *m/z* [M-BF$_4$]$^+$ calcd for $C_{30}H_{23}N_2^+$: 411.1856, found: 411.1861.



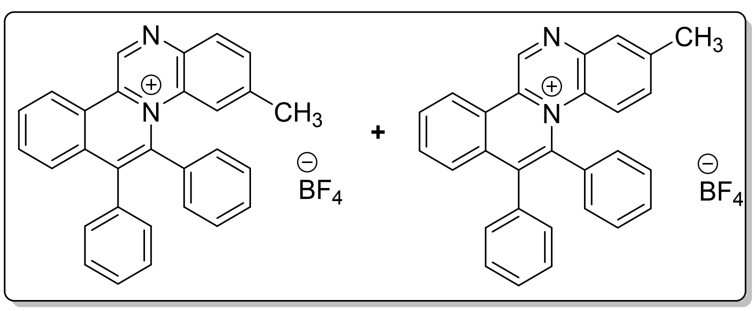
9-Fluoro-6,7-diphenylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3fa. Yellow solid; yield 93% (113 mg); ^{1}H NMR (400 MHz, DMSO-d$_6$) δ 11.01 (s, 1H), 9.87 (d, J = 9.6 Hz, 1H), 8.44 (d, J = 8.0 Hz, 1H), 8.34-8.29 (m, 1H), 7.91 (t, J = 8.0 Hz, 1H), 7.77 (d, J = 8.8 Hz, 1H), 7.52-7.45 (m, 4H), 7.34-7.31 (m, 4H), 7.27-7.21 (m, 4H); ^{13}C NMR (150 MHz, DMSO-d$_6$) δ 167.0 ($J_{C\text{-}F}$ = 258.9 Hz), 147.2, 141.5, 141.4, 140.0 ($J_{C\text{-}F}$ = 4.9 Hz), 138.5, 138.4, 137.5, 135.1, 133.3, 131.8, 131.7, 131.3, 130.5, 130.1, 129.9, 129.8, 129.0, 128.7, 127.5, 124.1, 122.3, 122.2, 111.6 ($J_{C\text{-}F}$ = 23.2 Hz); FT-IR (KBr) 2924, 2853, 1619, 1524, 1463, 1428, 1404, 1376, 1330, 1209, 1179, 1083, 1035, 765, 701 cm^{-1}; HRMS (ESI) *m/z* [M-BF$_4$]$^+$ calcd for $C_{28}H_{18}FN_2^+$: 401.1449, found: 401.1433



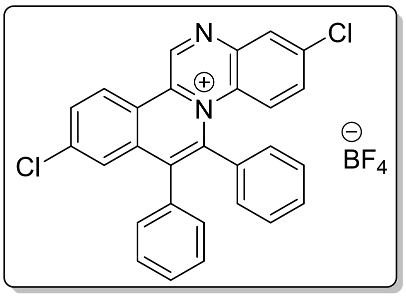
2-Chloro-6,7-diphenylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3ga. Yellow solid; yield 92% (116 mg); ^{1}H NMR (600 MHz, DMSO-d$_6$) δ 11.06 (s, 1H), 9.76 (d, J = 8.4 Hz, 1H), 8.47 (d, J = 8.4 Hz, 1H), 8.37-8.31 (m, 2H), 8.01 (q, J = 6.6 Hz, 1H), 7.75-7.73 (m, 2H), 7.47-7.45 (m, 3H), 7.39-7.37 (m, 3H), 7.31 (t, J = 8.4 Hz, 2H), 7.24-7.23 (m, 2H); ^{13}C NMR (150 MHz, DMSO-d$_6$) δ 147.7, 140.9, 140.6, 140.4, 137.9, 137.3, 136.0, 134.8, 134.2, 133.6, 132.4, 131.7, 131.6, 130.6, 130.03, 130.00, 128.9, 128.8, 128.6, 128.1, 127.4, 127.1, 124.8, 124.0; FT-IR (KBr) 2924, 2853, 1602, 1584, 1508, 1444, 1397, 1335, 1164, 1081, 1032, 918, 751, 698 cm^{-1}; HRMS (ESI) *m/z* [M-BF$_4$]$^+$ calcd for $C_{28}H_{18}ClN_2^+$: 417.1153, found: 417.1155.



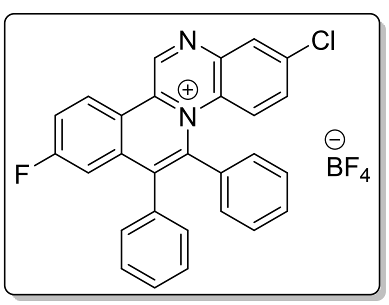
2,3-Dimethyl-6,7-diphenylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3ha. Yellow solid; yield 85% (106 mg); ^{1}H NMR (400 MHz, DMSO-d$_6$) δ 10.98 (s, 1H), 9.73 (d, J = 9.2 Hz, 1H), 8.33-8.23 (m, 3H), 7.71 (d, J = 8.0 Hz, 1H), 7.48-7.44 (m, 4H), 7.37-7.33 (m, 3H), 7.30-7.26 (m, 2H), 7.24-7.22 (m, 2H), 2.44 (s, 3H), 2.02 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d$_6$) δ 145.7, 141.6, 140.7, 140.6, 140.4, 140.0, 136.6, 136.3, 135.3, 135.1, 133.8, 132.0, 130.5, 129.9, 129.4, 129.3, 128.6, 128.5, 128.4, 126.9, 126.8, 125.6, 124.9, 123.8, 20.0, 19.0; FT-IR (KBr) 2924, 2853, 1609, 1583, 1507, 1443, 1398, 1375, 1337, 1229, 1149, 1082, 1030, 869, 766, 696 cm^{-1}; HRMS (ESI) *m/z* [M-BF$_4$]$^+$ calcd for $C_{30}H_{23}N_2^+$: 411.1856, found: 411.1866.



3-Methyl-6,7-diphenylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate and 2-Methyl-6,7-diphenylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate (2.5:1) 3ia. Yellow solid; yield 82% (99 mg); ^{1}H NMR (400 MHz, DMSO-d$_6$) 11.02 (s, 0.4H), 10.99 (s, 1H), 9.72 (t, J = 8.0 Hz, 1.6H), 8.34-8.25 (m, 5.1 H), 7.79 (d, J = 8.0 Hz, 1.2H), 7.71 (t, J = 8.0 Hz, 1.6H), 7.66 (d, J = 9.2 Hz, 0.5H), 7.51 (s, 1.1H), 7.46-7.44 (m, 4.9H), 7.35-7.22 (m, 12.3H), 2.12 (s, 3.3H); ^{13}C NMR (100 MHz, DMSO-d$_6$) δ 146.9, 145.8, 142.0, 141.8, 140.8, 140.7, 140.5, 140.2, 137.3, 136.9, 136.6, 136.5, 135.4, 135.3, 135.2, 133.7, 132.6, 132.0, 131.2, 130.5, 130.4, 129.97, 129.90, 129.7, 129.4, 129.2, 128.64, 128.61, 128.5, 128.4, 127.3, 127.0, 126.9, 125.6, 124.88, 124.82, 123.9, 123.5, 21.4, 20.4; FT-IR (KBr) 2924, 2854, 1608, 1519, 1444, 1375, 1339, 1209, 1161, 1083, 1061, 839, 777, 696 cm^{-1}; HRMS (ESI) *m/z* $[M\text{-}BF_4]^+$ calcd for $C_{29}H_{21}N_2^+$: 397.1699, found: 397.1699.

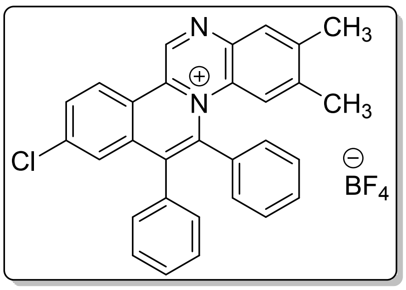


2,9-Dichloro-6,7-diphenylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3ja. Yellow solid; yield 85% (114 mg); ^{1}H NMR (400 MHz, DMSO-d$_6$) δ 11.04 (s, 1H), 9.79 (d, *J* = 9.2 Hz, 1H), 8.48-8.45 (m, 2H), 8.03 (dd, *J* = 9.2, 6.4 Hz, 1H), 7.73-7.72 (m, 1H), 7.62-7.61 (m, 1H), 7.48-7.47 (m, 3H), 7.39-7.36 (m, 3H), 7.31 (t, *J* = 7.6 Hz, 2H), 7.26-7.24 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d$_6$) δ 147.5, 142.5, 141.7, 140.4, 139.6, 137.7, 136.8, 134.5, 134.3, 132.8, 132.7, 131.7, 131.6, 130.4, 130.0, 129.9, 129.7, 129.0, 128.8, 128.6, 127.9, 125.6, 123.9, 123.4; FT-IR (KBr) 2924, 2853, 1597, 1510, 1434, 1393, 1331, 1205, 1187, 1096, 1056, 1033, 973, 851, 729, 698 cm^{-1}; HRMS (ESI) *m/z* $[M\text{-}BF_4]^+$ calcd for $C_{28}H_{17}Cl_2N_2^+$: 451.0763, found: 451.0763.

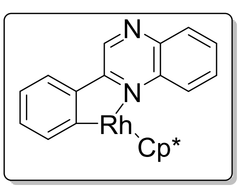


2-Chloro-9-fluoro-6,7-diphenylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3ka. Yellow solid; yield 88% (115 mg); ^{1}H NMR (400 MHz, DMSO-d$_6$) δ 11.03 (s, 1H), 9.89 (q, *J* = 4.4 Hz, 1H), 8.47 (d, *J* = 8.8 Hz, 1H), 8.37-8.32 (m, 1H), 8.01 (dd, *J* = 8.8, 6.8 Hz, 1H), 7.71 (s, 1H), 7.48-7.46 (m, 3H), 7.39-7.31 (m, 6H), 7.25-7.22 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d$_6$) δ 167.6 ($J_{C\text{-}F}$ = 260.6 Hz), 147.6, 141.4, 140.2, 138.7, 137.5, 134.4, 134.2, 133.0, 131.9, 131.8, 131.6

($J_{C\text{-}F}$ = 4.5 Hz), 130.5, 130.0, 129.8, 128.9, 128.8, 128.6, 127.9, 123.9, 122.5, 122.3, 122.0, 111.6 ($J_{C\text{-}F}$ = 23.1 Hz); FT-IR (KBr) 2924, 2853, 1617, 1519, 1463, 1377, 1329, 1288, 1199, 1062, 1031, 909, 831, 765, 697 cm^{-1}; HRMS (ESI) *m/z* [M-BF$_4$]$^+$ calcd for $C_{28}H_{17}ClFN_2^+$: 435.1059, found: 435.1071.



9-Chloro-2,3-dimethyl-6,7-diphenylisoquinolino[2,1-a]quinoxalin-5-ium tetrafluoroborate 3la. Yellow solid; yield 90% (120 mg); ^{1}H NMR (400 MHz, DMSO-d$_6$) δ 10.95 (s, 1H), 9.75 (d, *J* = 9.2 Hz, 1H), 8.41 (dd, *J* = 8.8, 6.8 Hz, 1H), 8.24 (s, 1H), 7.57-7.56 (m, 1H), 7.47-7.46 (m, 4H), 7.36-7.28 (m, 5H), 7.26-7.23 (m, 2H) 2.44 (s, 3H), 2.02 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d$_6$) δ 145.6, 141.9, 141.4, 141.2, 140.9, 140.8, 139.2, 136.5, 136.1, 135.1, 133.1, 132.3, 130.4, 129.9, 129.6, 129.33, 129.31, 128.8, 128.6, 125.59, 125.55, 123.69, 123.64, 20.1, 19.0; FT-IR (KBr) 2923, 1599, 1508, 1451, 1396, 1346, 1333, 1224, 1154, 1096, 1083, 1032, 968, 826, 751, 698 cm^{-1}; HRMS (ESI) *m/z* [M-BF$_4$]$^+$ calcd for $C_{30}H_{22}ClN_2^+$: 445.1466, found: 445.1466.



Rhodacycle 1l′. Yellow solid; yield 55 % (29 mg); ^{1}H NMR (400 MHz, DMSO-d$_6$) δ 9.59 (s, 1H), 8.35 (dd, *J* = 8.0, 6.4 Hz, 2H), 8.17-8.12 (m, 2H), 7.92-7.83 (m, 2H), 7.62-7.58 (m, 2H), 1.71 (s, 3H), 1.61 (s, 1H), 1.54 (s, 11H); HRMS (ESI) *m/z* [M-BF$_4$]$^+$ calcd for $C_{24}H_{24}RhN_2^+$: 443.0995, found: 443.0995.

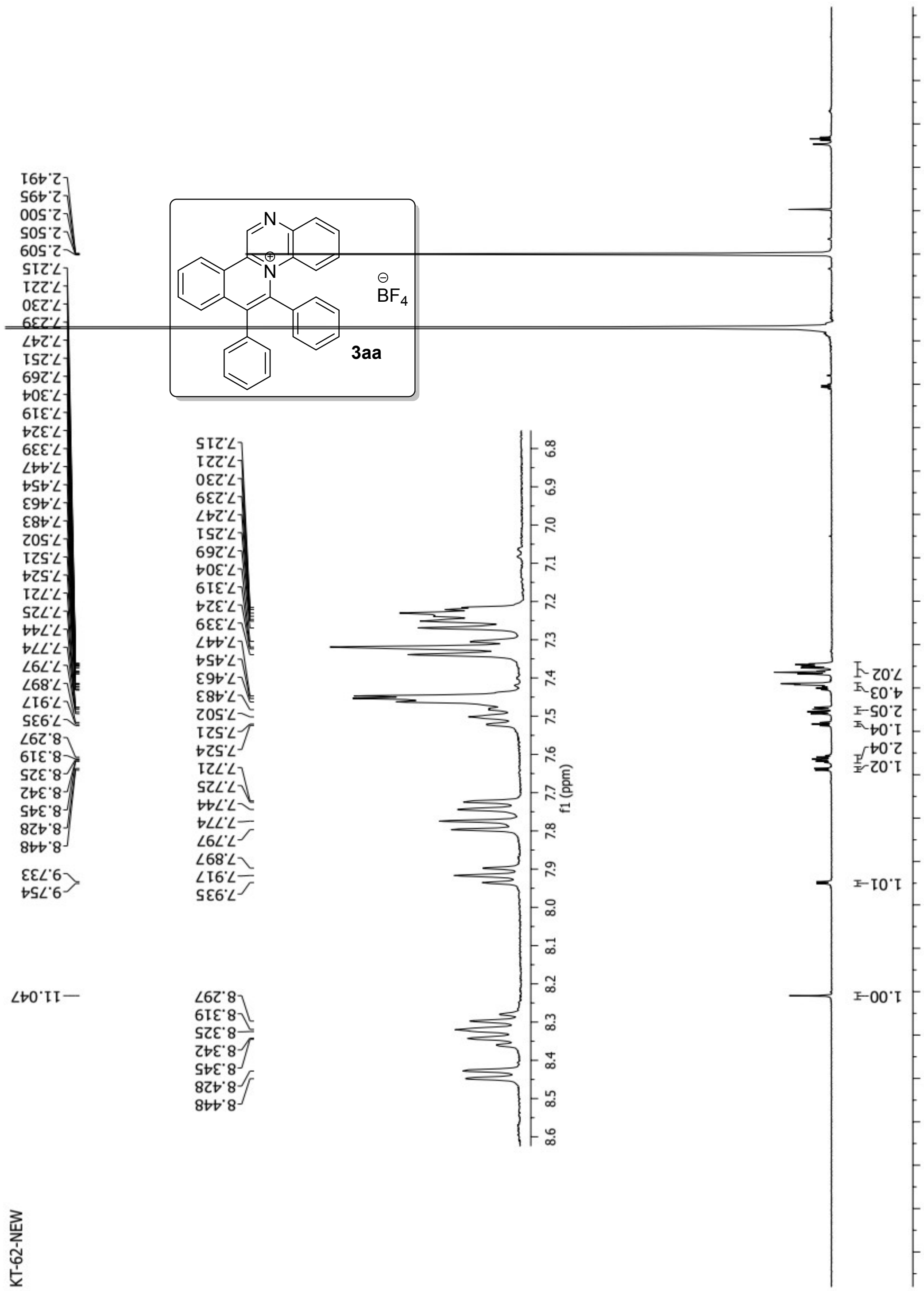
References

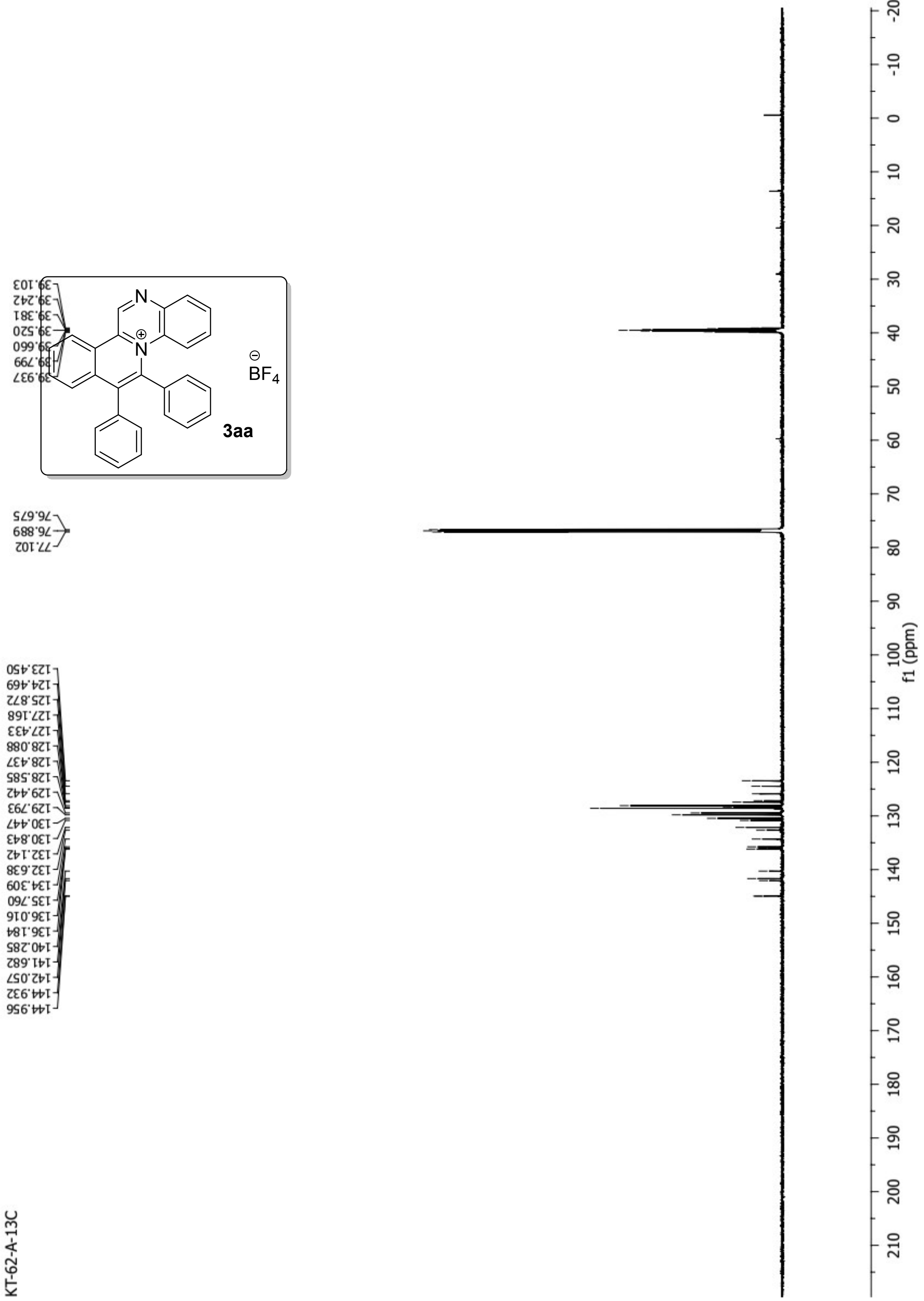
1. D. Lu, Q. Xiang, L. Zhou and Q. Zeng*, Asian J. Chem.*, 2015, **27**, 2639.

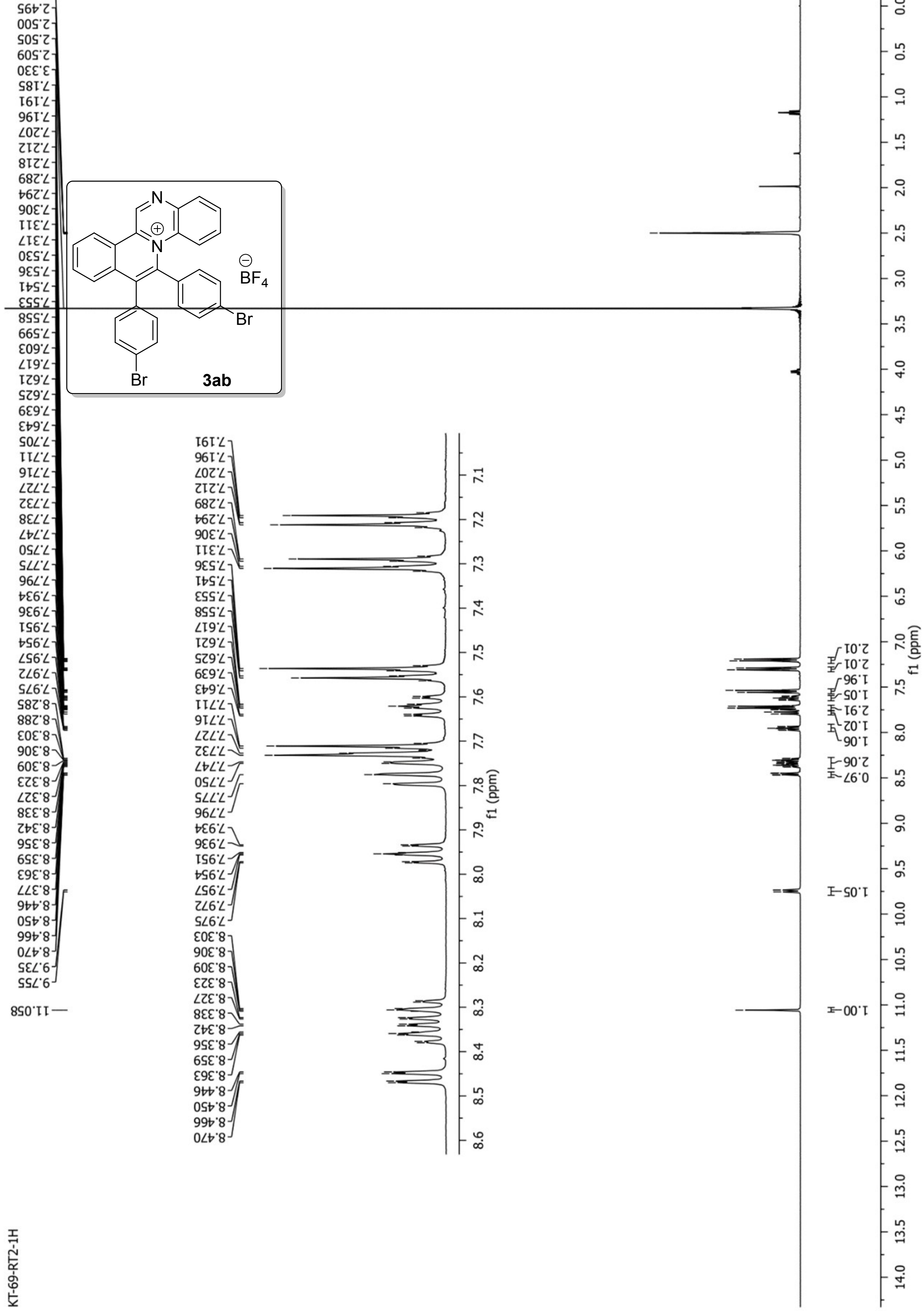
2. M. J. Mio, L. C. Kopel, J. B. Braun, T. L. Gadzikwa, K. L. Hull, R. G. Brisbois, C. J. Markworth and P. A. Grieco, *Org. Lett.*, 2002, **4**, 3199

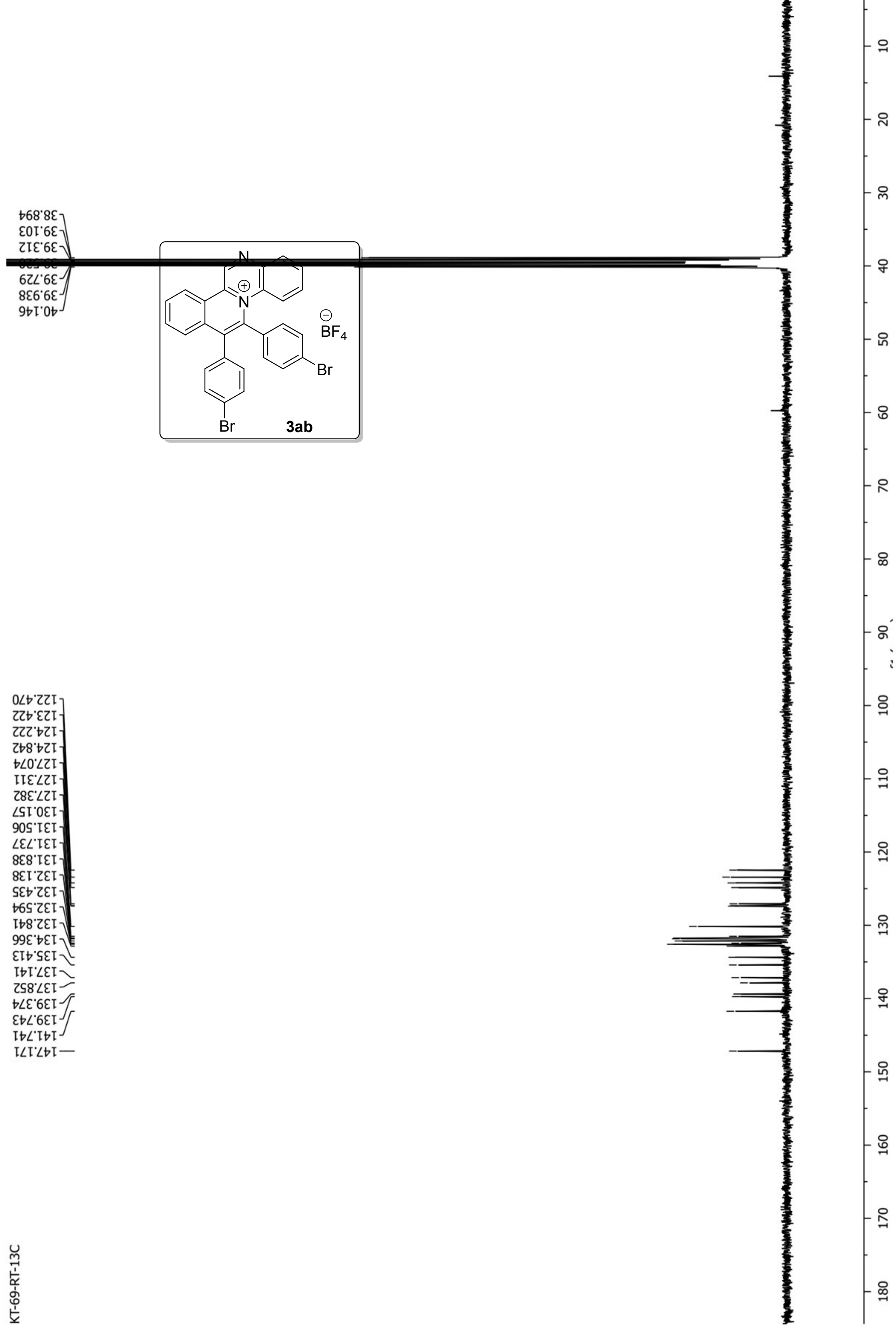
3. W. Xie, B. Li and B. Wang, *J. Org. Chem.,* 2016, **81**, 396.

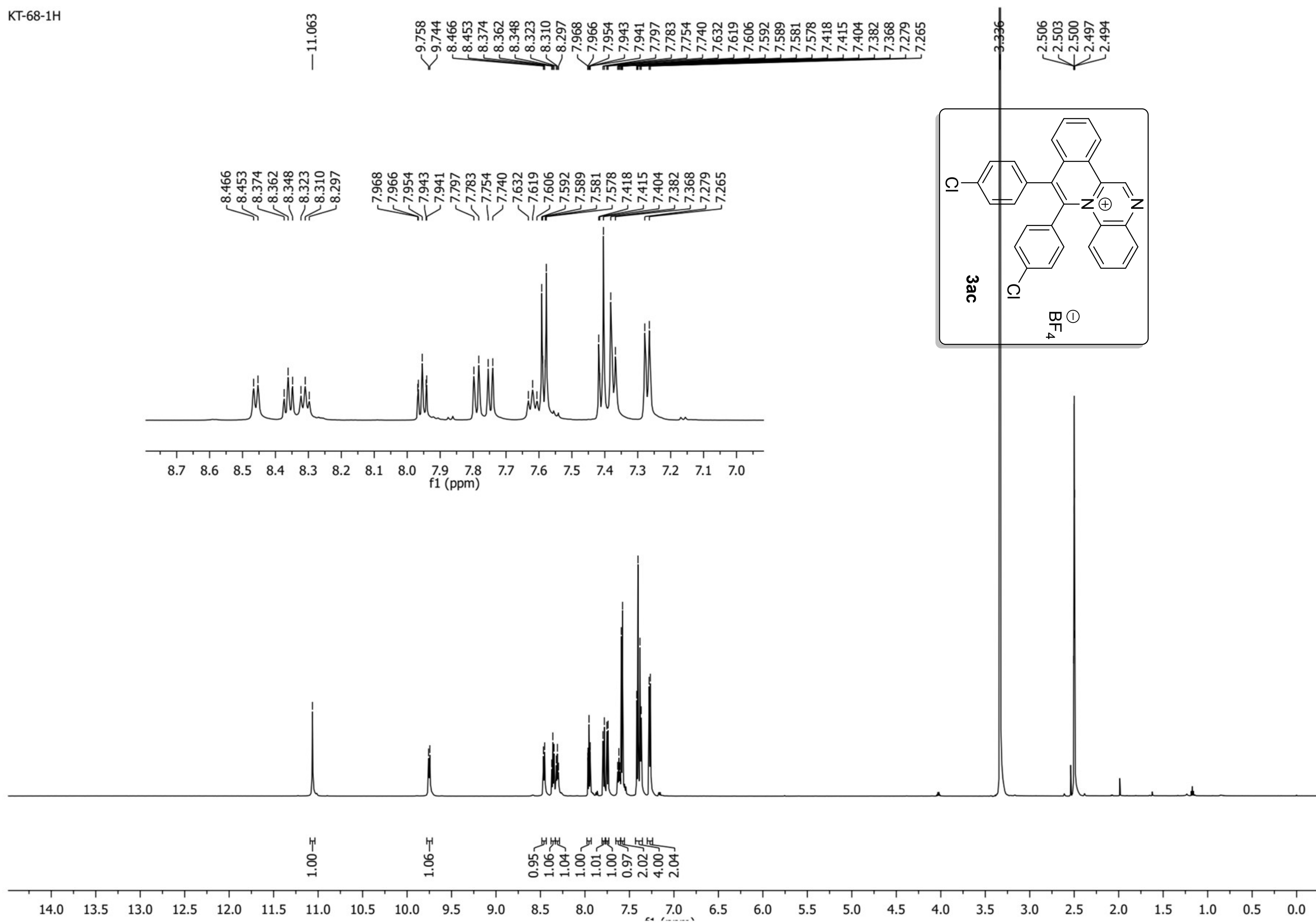
^{1}H and ^{13}C NMR Spectra




KT-62-A-13C
3aa
$BF_4^{\ominus}$
39.103
39.242
39.381
39.520
39.660
39.799
39.937
76.675
76.889
77.102
123.450
124.469
125.872
127.168
127.433
128.088
128.437
128.585
129.442
129.793
130.447
130.843
132.142
132.638
134.309
135.760
136.016
136.184
140.285
141.682
142.057
144.932
144.956
f1 (ppm)


KT-69-RT2-1H
3ab
BF4
Br
Br
f1 (ppm)
f1 (ppm)


KT-69-RT-13C
3ab
BF4
Br
Br
N
38.894
39.103
39.312
39.729
39.938
40.146
122.470
123.422
124.222
124.842
127.074
127.311
127.382
130.157
131.506
131.737
131.838
132.138
132.435
132.594
132.841
134.366
135.413
137.141
137.852
139.374
139.743
141.741
147.171
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180


KT-68-1H
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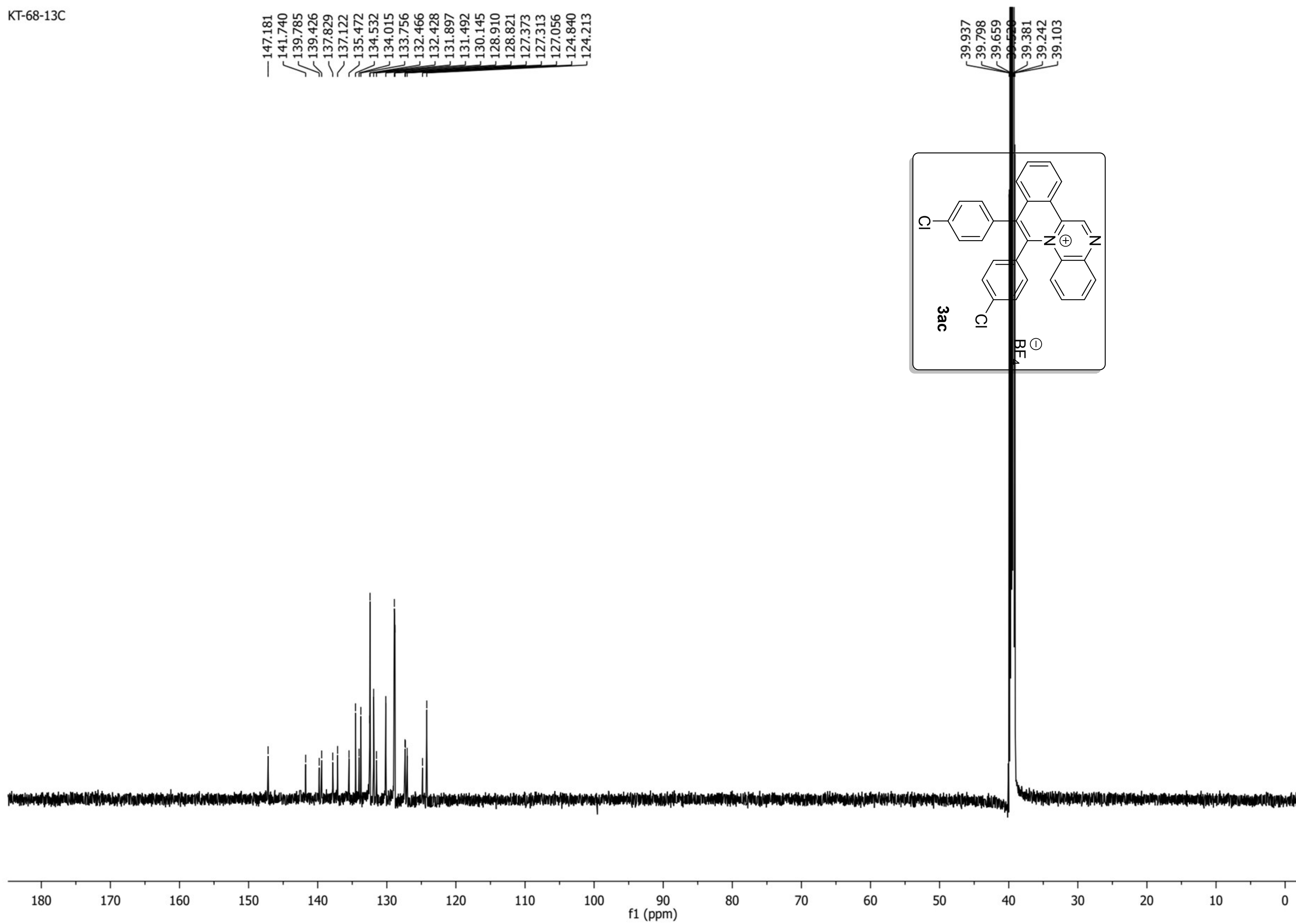
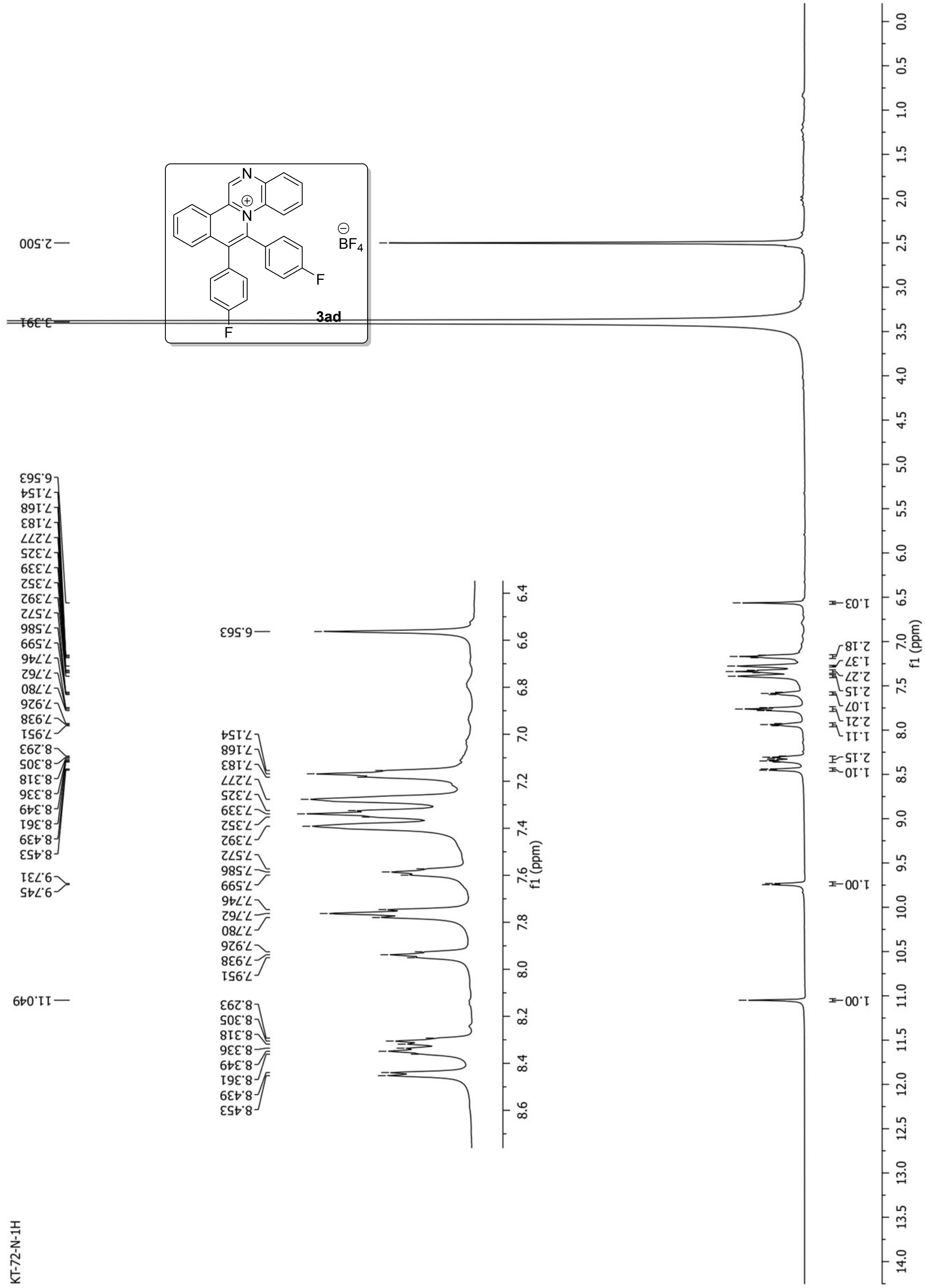
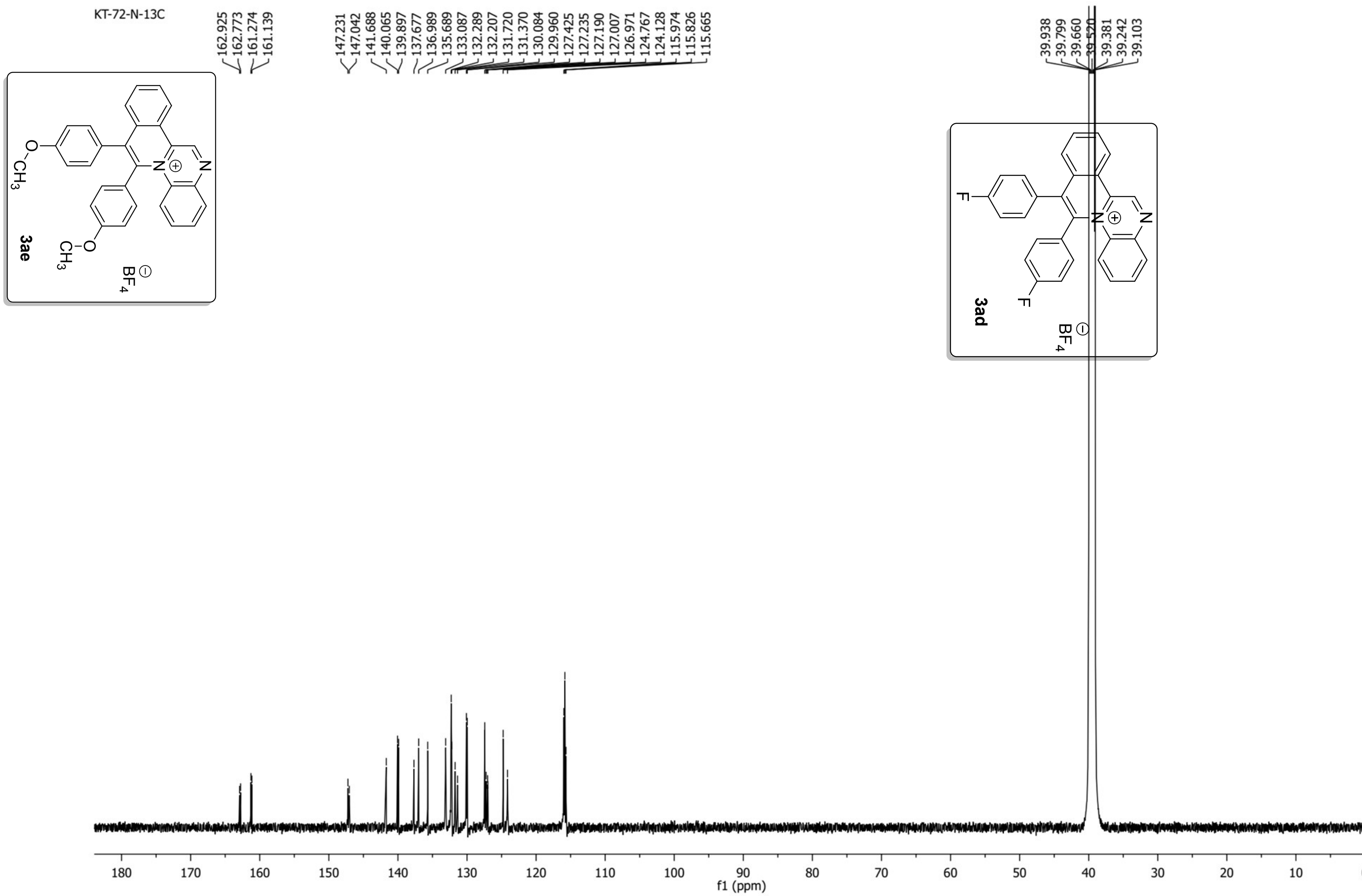
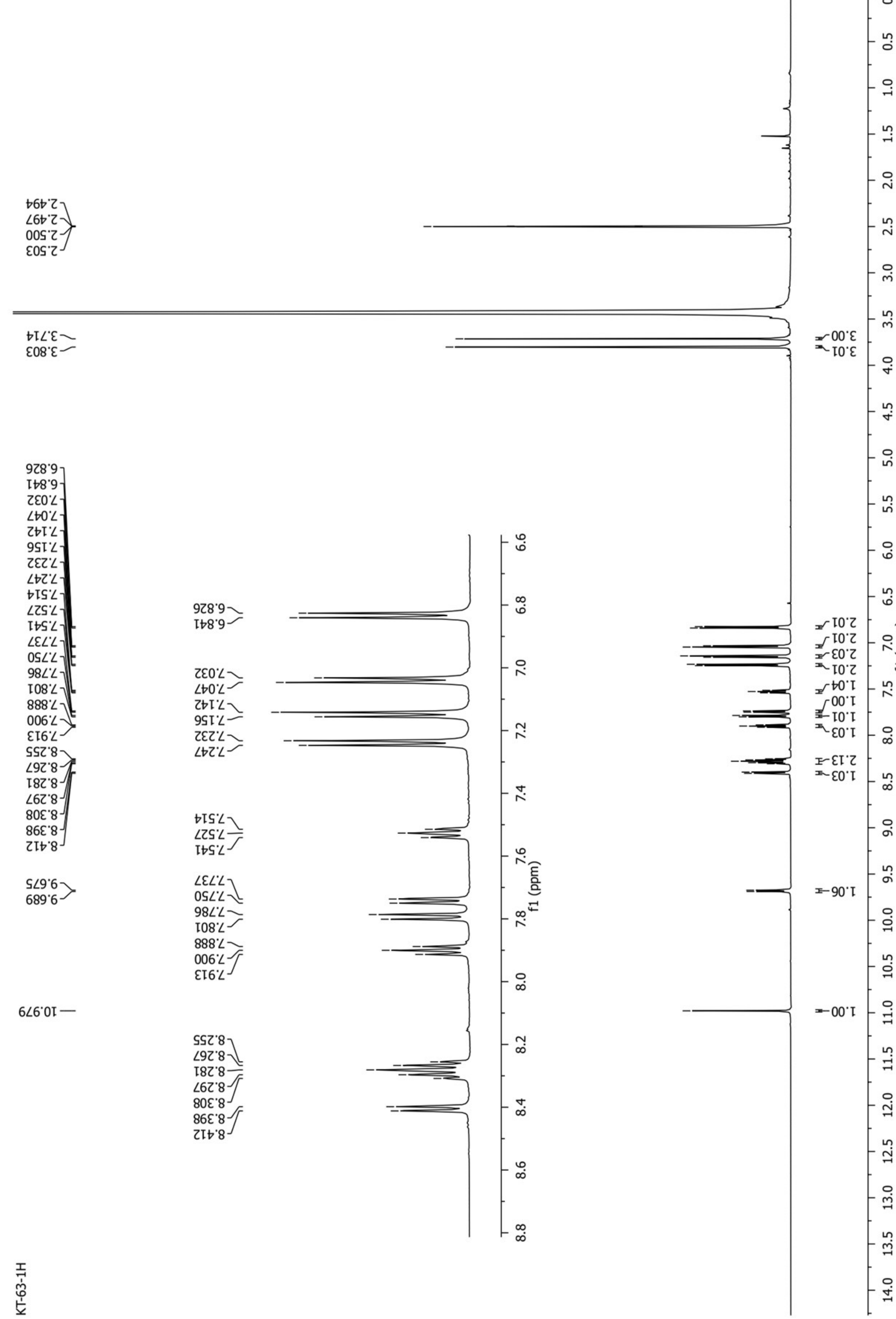
9.758
9.744
8.466
8.453
8.374
8.362
8.348
8.323
8.310
8.297
7.968
7.966
7.954
7.943
7.941
7.797
7.783
7.754
7.740
7.632
7.619
7.606
7.592
7.589
7.581
7.578
7.418
7.415
7.404
7.382
7.368
7.279
7.265
3.336
2.506
2.503
2.500
2.497
2.494
N
N
Cl
Cl
BF_4
3ac
f1 (ppm)
1.00
1.06
0.95
1.06
1.04
1.00
1.01
1.00
0.97
2.02
4.00
2.04

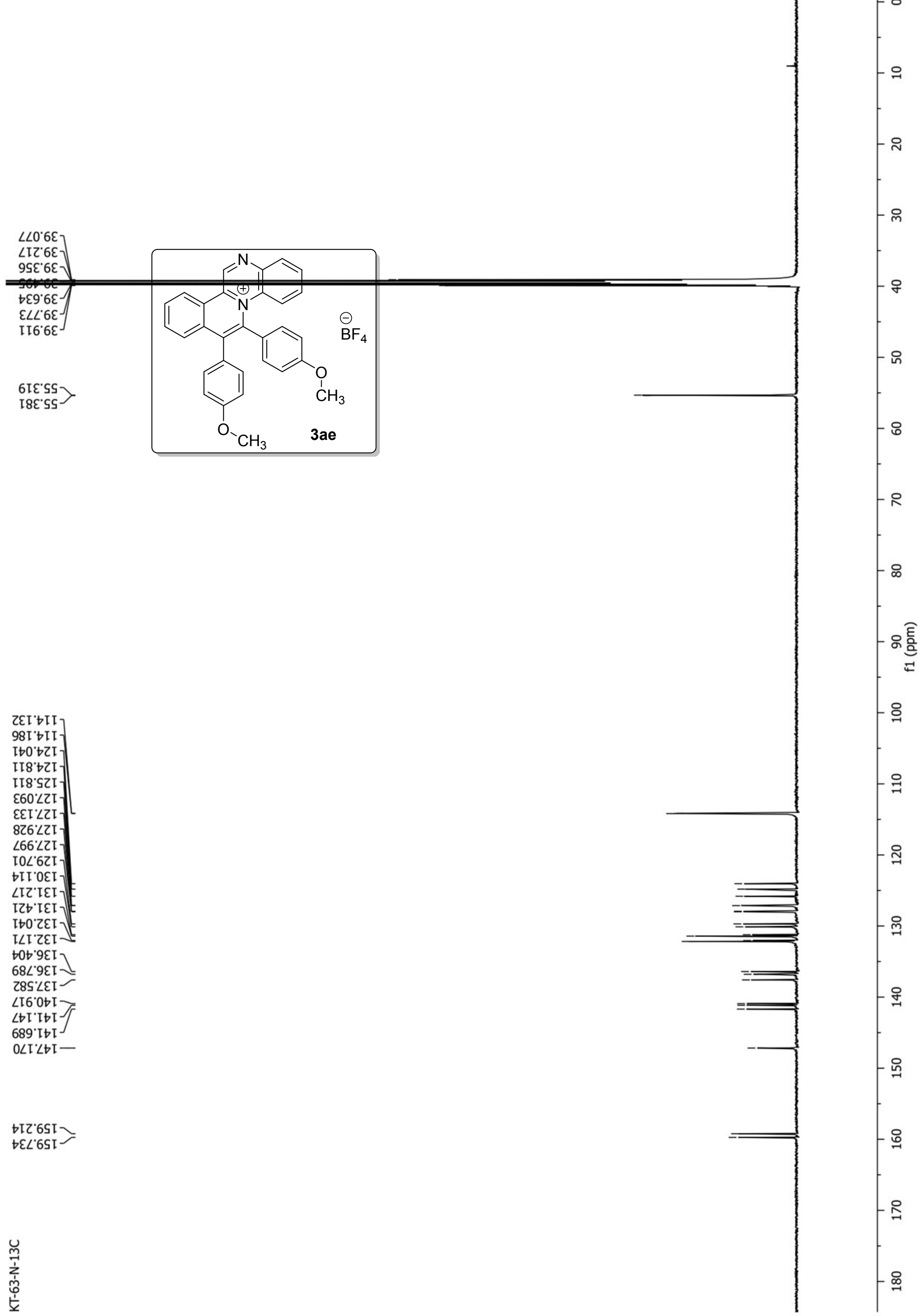

KT-68-13C
147.181
141.740
139.785
139.426
137.829
137.122
135.472
134.532
134.015
133.756
132.466
132.428
131.897
131.492
130.145
128.910
128.821
127.373
127.313
127.056
124.840
124.213
39.937
39.798
39.659
39.381
39.242
39.103
3ac
f1 (ppm)

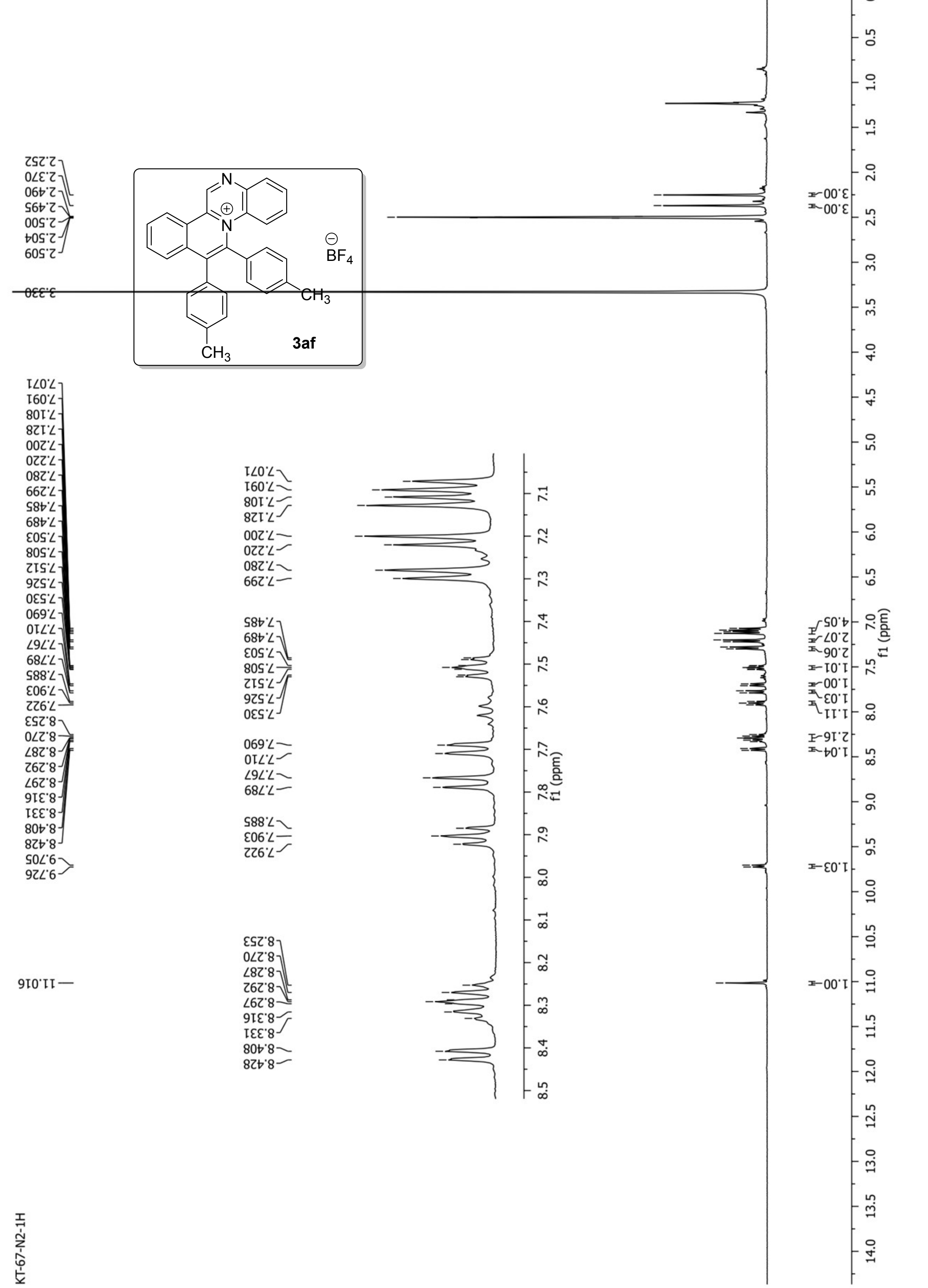


KT-72-N-1H
3ad
N
F
F
BF4
2.500
3.391
11.049
6.563
f1 (ppm)

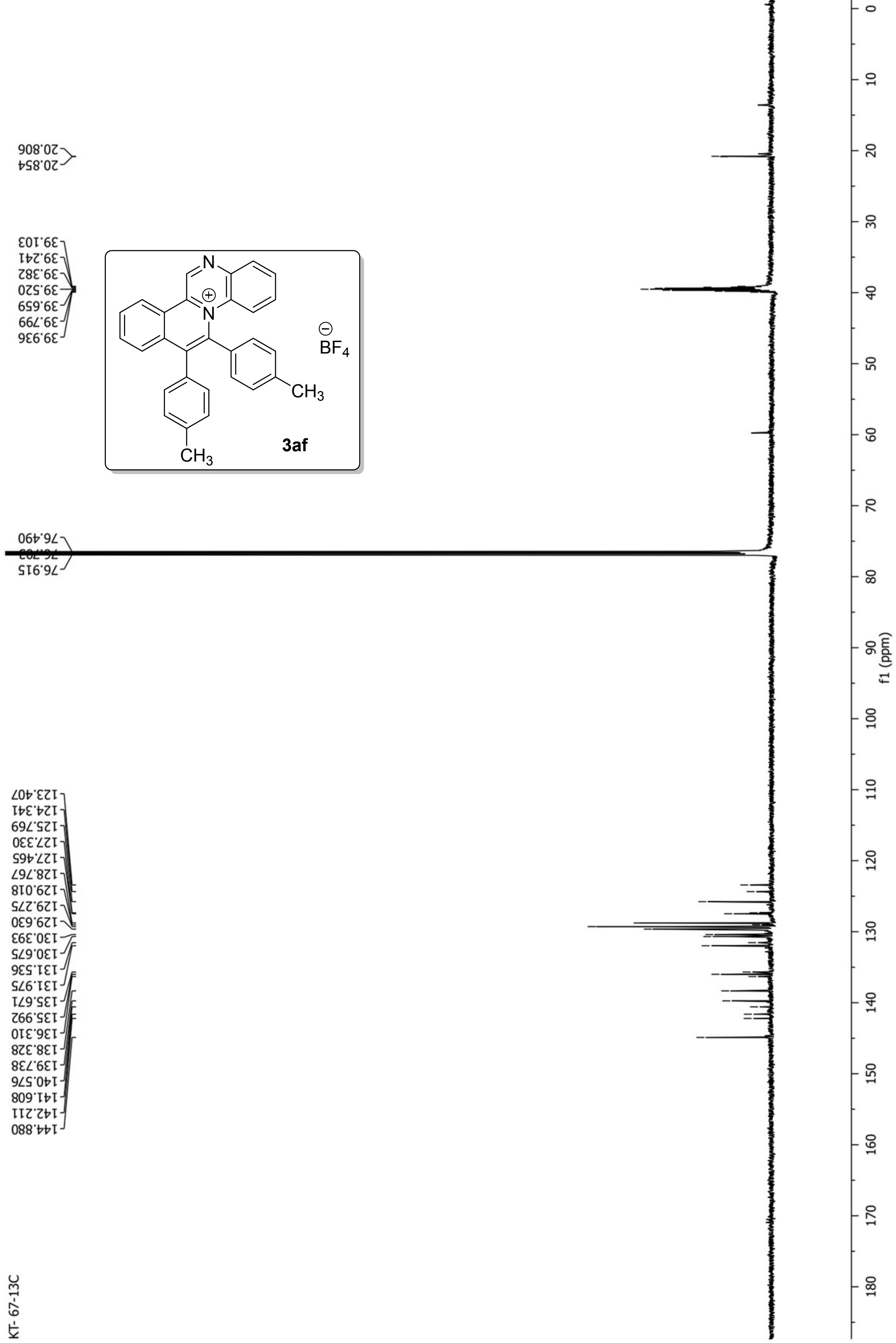

KT-72-N-13C
162.925
162.773
161.274
161.139
147.231
147.042
141.688
140.065
139.897
137.677
136.989
135.689
133.087
132.289
132.207
131.720
131.370
130.084
129.960
127.425
127.235
127.190
127.007
126.971
124.767
124.128
115.974
115.826
115.665
39.938
39.799
39.660
39.520
39.381
39.242
39.103
3ae
3ad
f1 (ppm)
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

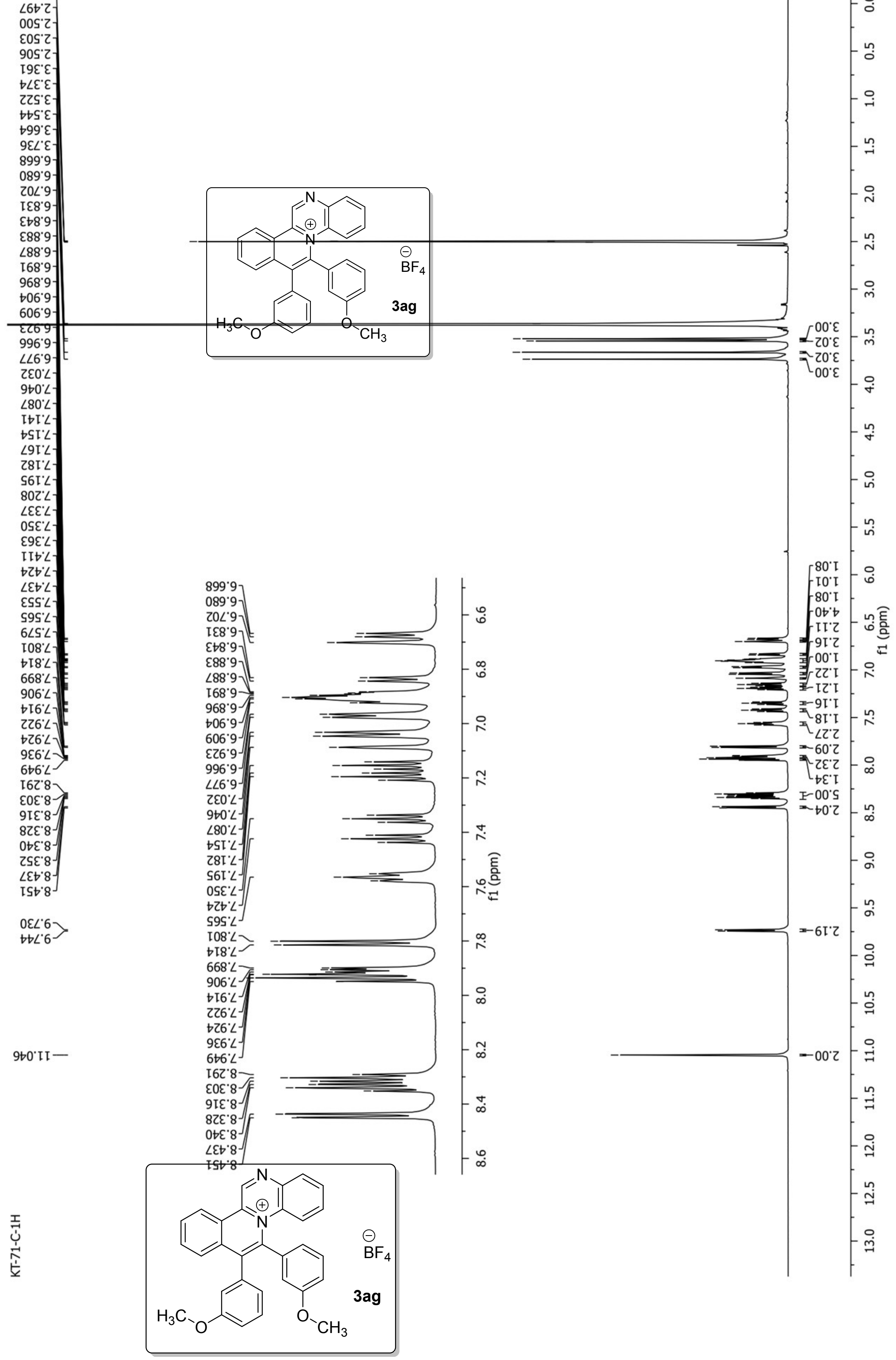

KT-63-1H
2.494
2.497
2.500
2.503
3.714
3.803
6.826
6.841
7.032
7.047
7.142
7.156
7.232
7.247
7.514
7.527
7.541
7.737
7.750
7.786
7.801
7.888
7.900
7.913
8.255
8.267
8.281
8.297
8.308
8.398
8.412
9.675
9.689
10.979
3.00
3.01
2.01
2.01
2.03
2.01
1.04
1.00
1.01
1.03
2.13
1.03
1.06
1.00
f1 (ppm)

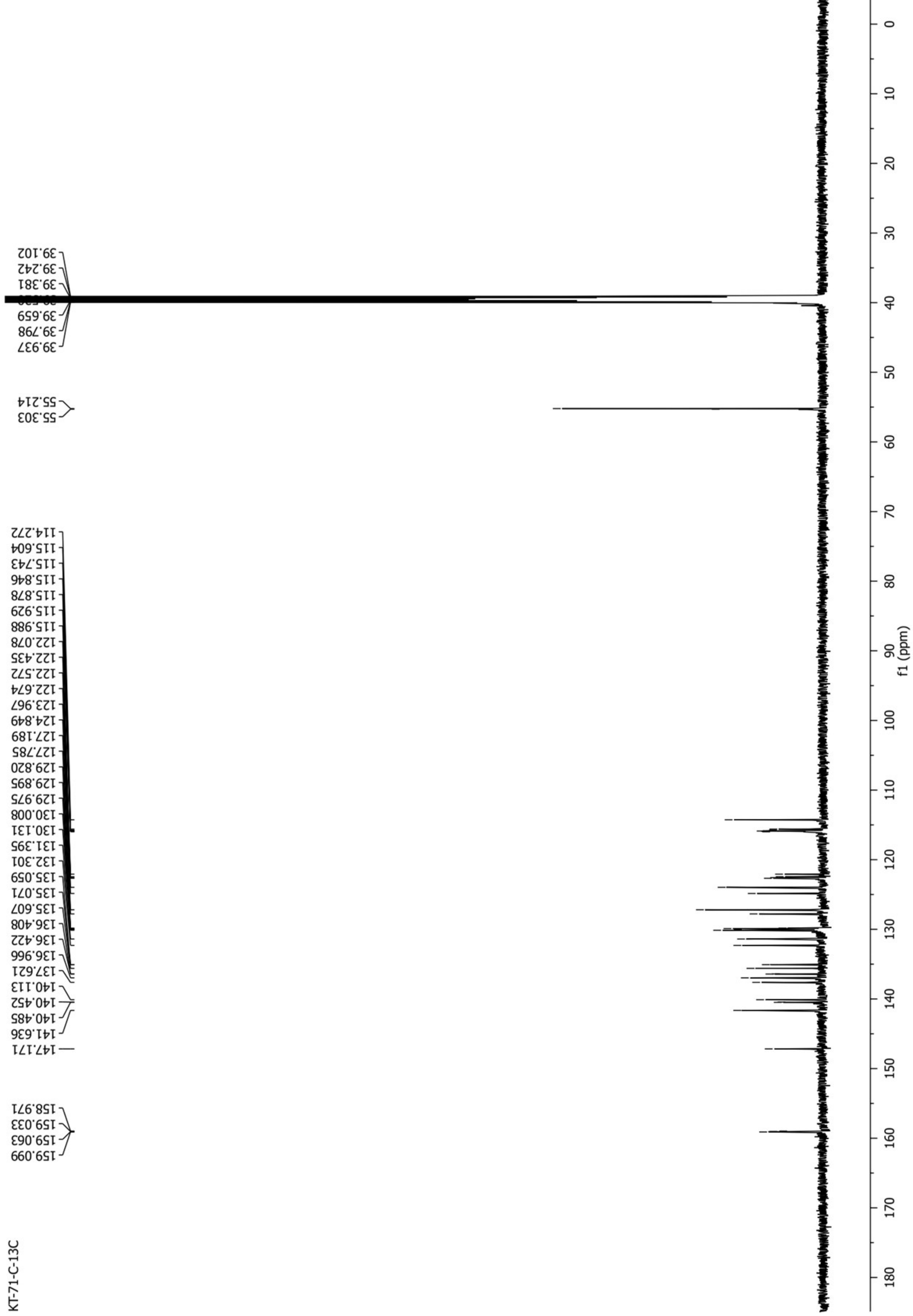

39.077
39.217
39.356
39.495
39.634
39.773
39.911
55.319
55.381
114.132
114.186
124.041
124.811
125.811
127.093
127.133
127.928
127.997
129.701
130.114
131.217
131.421
132.041
132.171
136.404
136.789
137.582
140.917
141.147
141.689
147.170
159.214
159.734
KT-63-N-13C
f1 (ppm)
BF4
3ae

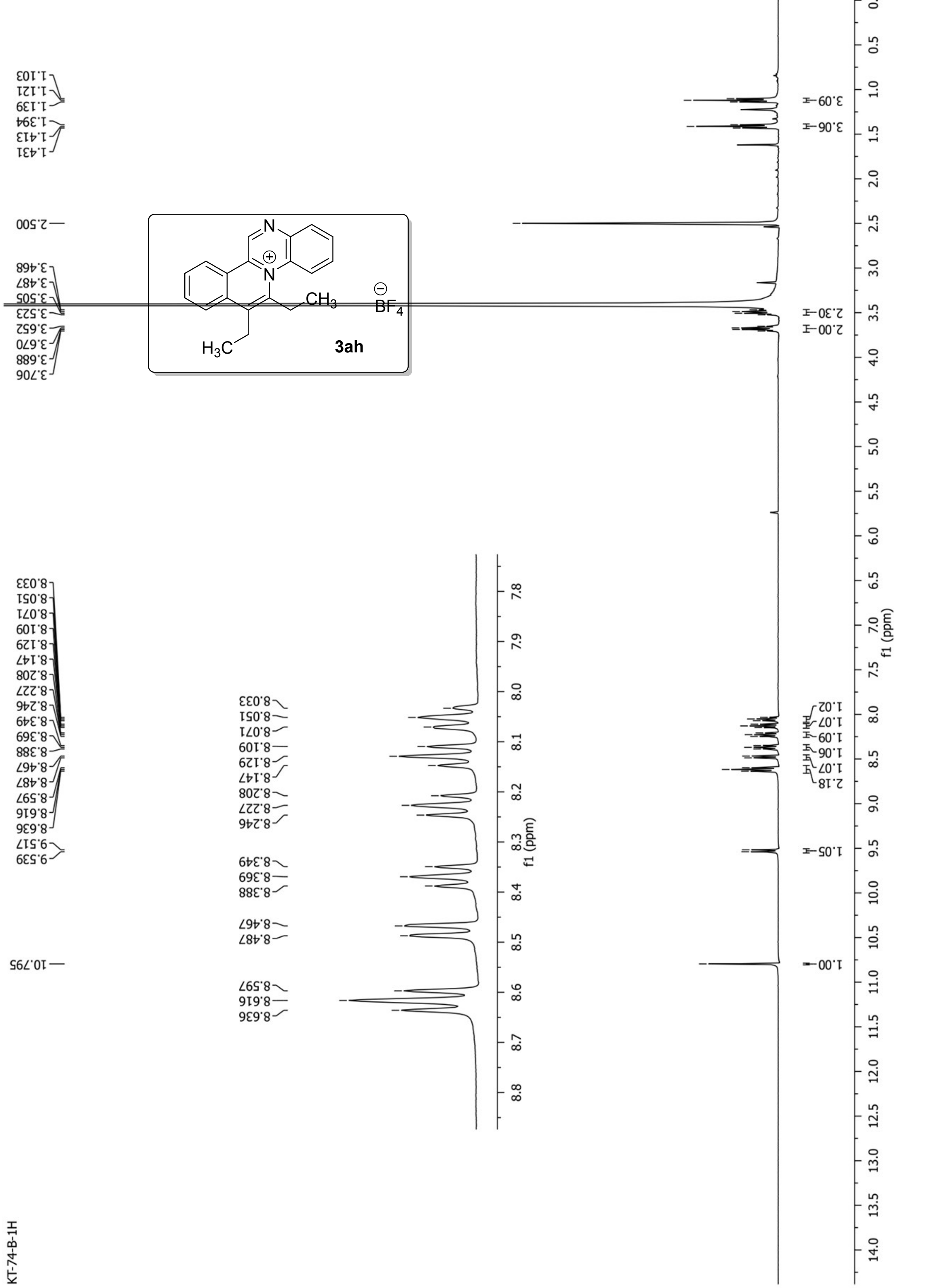


KT-67-N2-1H
3af
BF4
CH3
f1 (ppm)


KT- 67-13C
20.806
20.854
39.103
39.241
39.382
39.520
39.659
39.799
39.936
76.490
76.703
76.915
123.407
124.341
125.769
127.330
127.465
128.767
129.018
129.275
129.630
130.393
130.675
131.536
131.975
135.671
135.992
136.310
138.328
139.738
140.576
141.608
142.211
144.880
N
N
BF4
CH3
CH3
3af
f1 (ppm)


KT-71-C-1H
3ag
BF4
f1 (ppm)


KT-71-C-13C
39.102
39.242
39.381
39.659
39.798
39.937
55.214
55.303
114.272
115.604
115.743
115.846
115.878
115.929
115.988
122.078
122.435
122.572
122.674
123.967
124.849
127.189
127.785
129.820
129.895
129.975
130.008
130.131
131.395
132.301
135.059
135.071
135.607
136.408
136.422
136.966
137.621
140.113
140.452
140.485
141.636
147.171
158.971
159.033
159.063
159.099
f1 (ppm)


KT-74-B-1H
3ah
CH3
H3C
BF4
10.795
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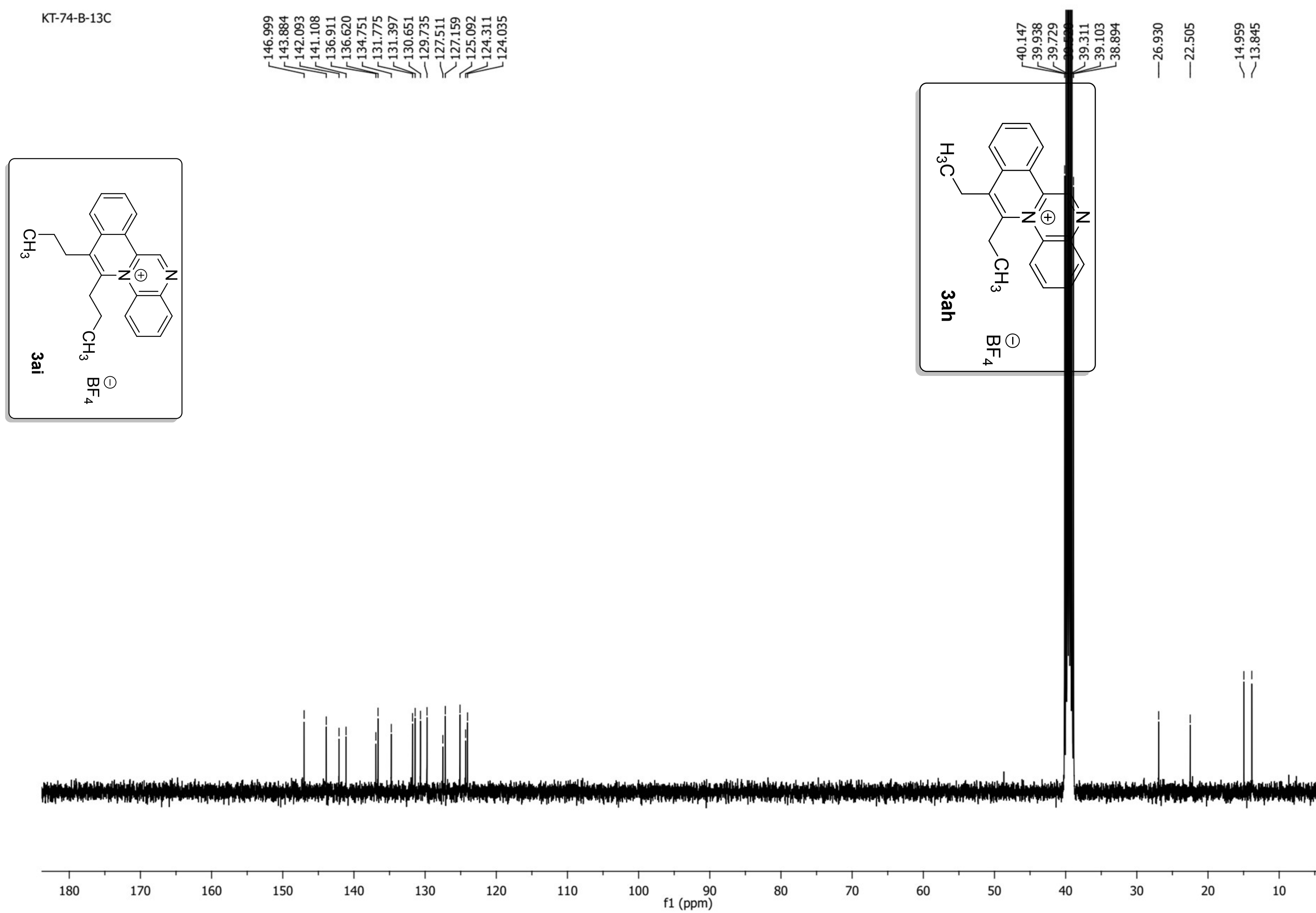
9.517
8.636
8.616
8.597
8.487
8.467
8.388
8.369
8.349
8.246
8.227
8.208
8.147
8.129
8.109
8.071
8.051
8.033
3.706
3.688
3.670
3.652
3.523
3.505
3.487
3.468
2.500
1.431
1.413
1.394
1.139
1.121
1.103
1.00
1.05
2.18
1.07
1.06
1.09
1.07
1.02
2.00
2.30
3.06
3.09
f1 (ppm)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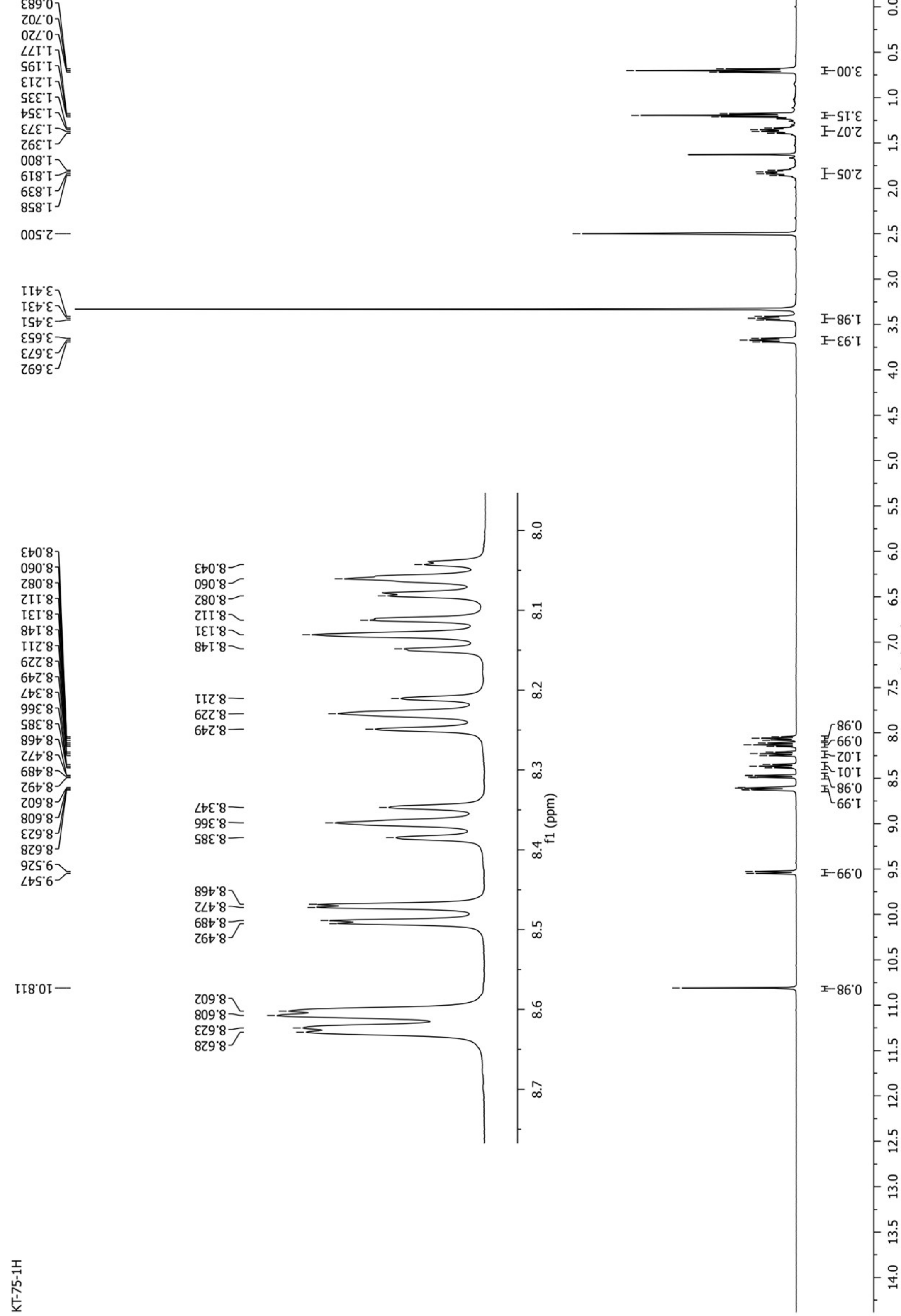
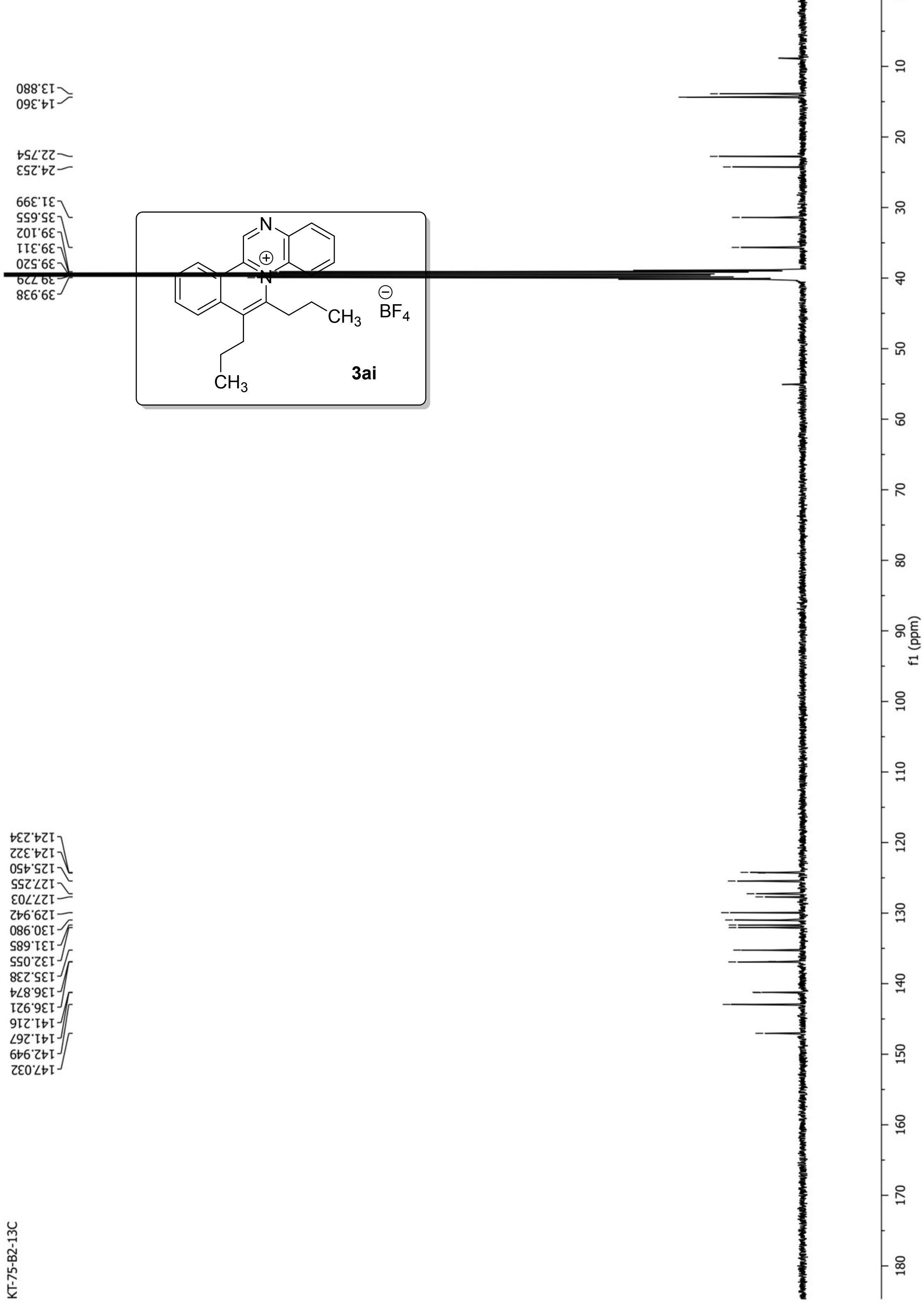
142.093
141.108
136.911
136.620
134.751
131.775
131.397
130.651
129.735
127.511
127.159
125.092
124.311
124.035
40.147
39.938
39.729
39.311
39.103
38.894
26.930
22.505
14.959
13.845
3ai
3ah
f1 (ppm)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


KT-75-1H
0.683
0.702
0.720
1.177
1.195
1.213
1.335
1.354
1.373
1.392
1.800
1.819
1.839
1.858
2.500
3.411
3.431
3.451
3.653
3.673
3.692
8.043
8.060
8.082
8.112
8.131
8.148
8.211
8.229
8.249
8.347
8.366
8.385
8.468
8.472
8.489
8.492
8.602
8.608
8.623
8.628
9.526
9.547
10.811
3.00
3.15
2.07
2.05
1.98
1.93
0.98
0.99
1.02
1.01
0.98
1.99
0.99
0.98
f1 (ppm)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147.032
142.949
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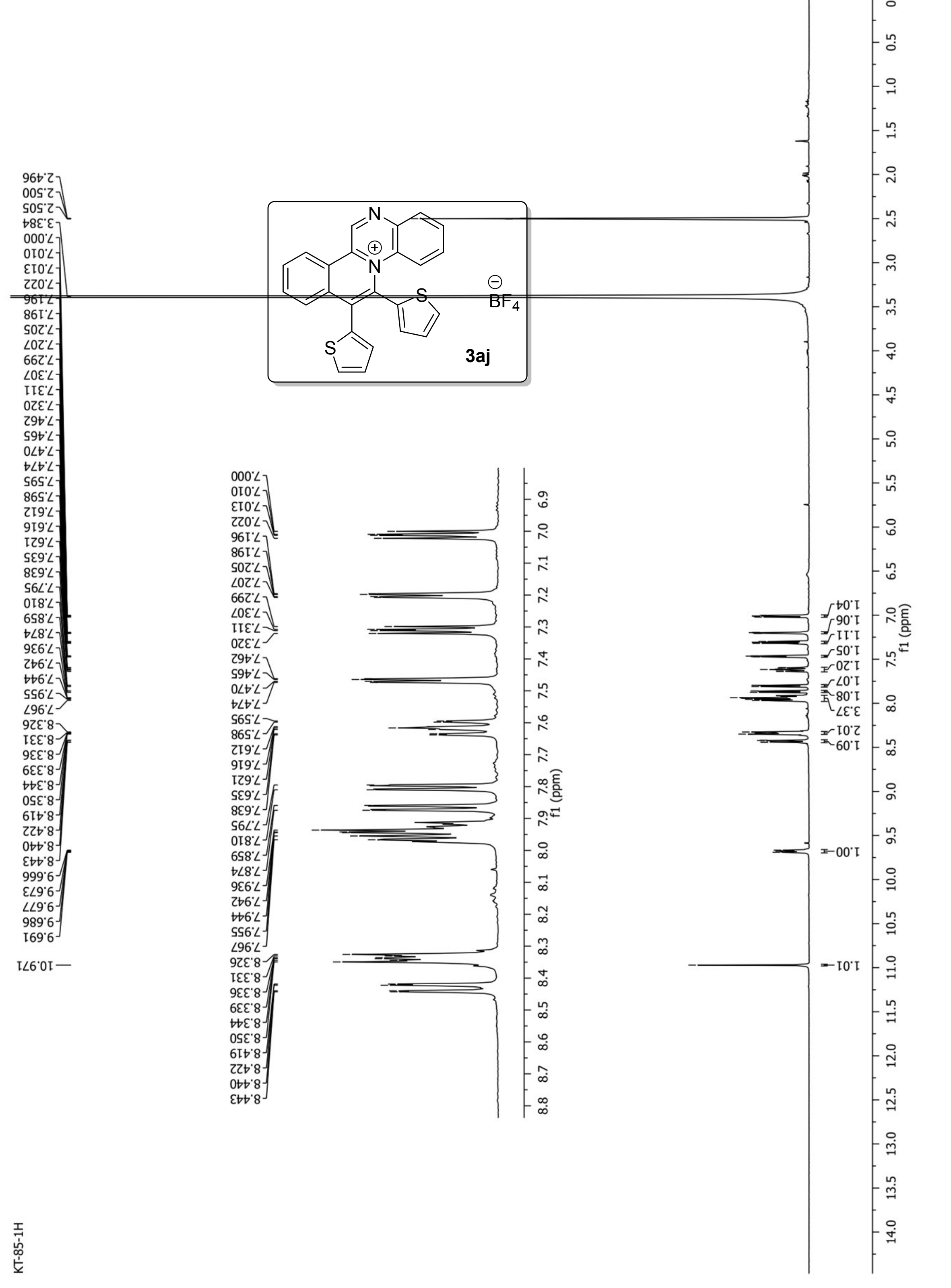
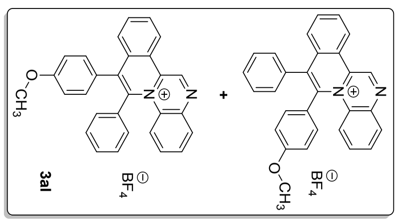
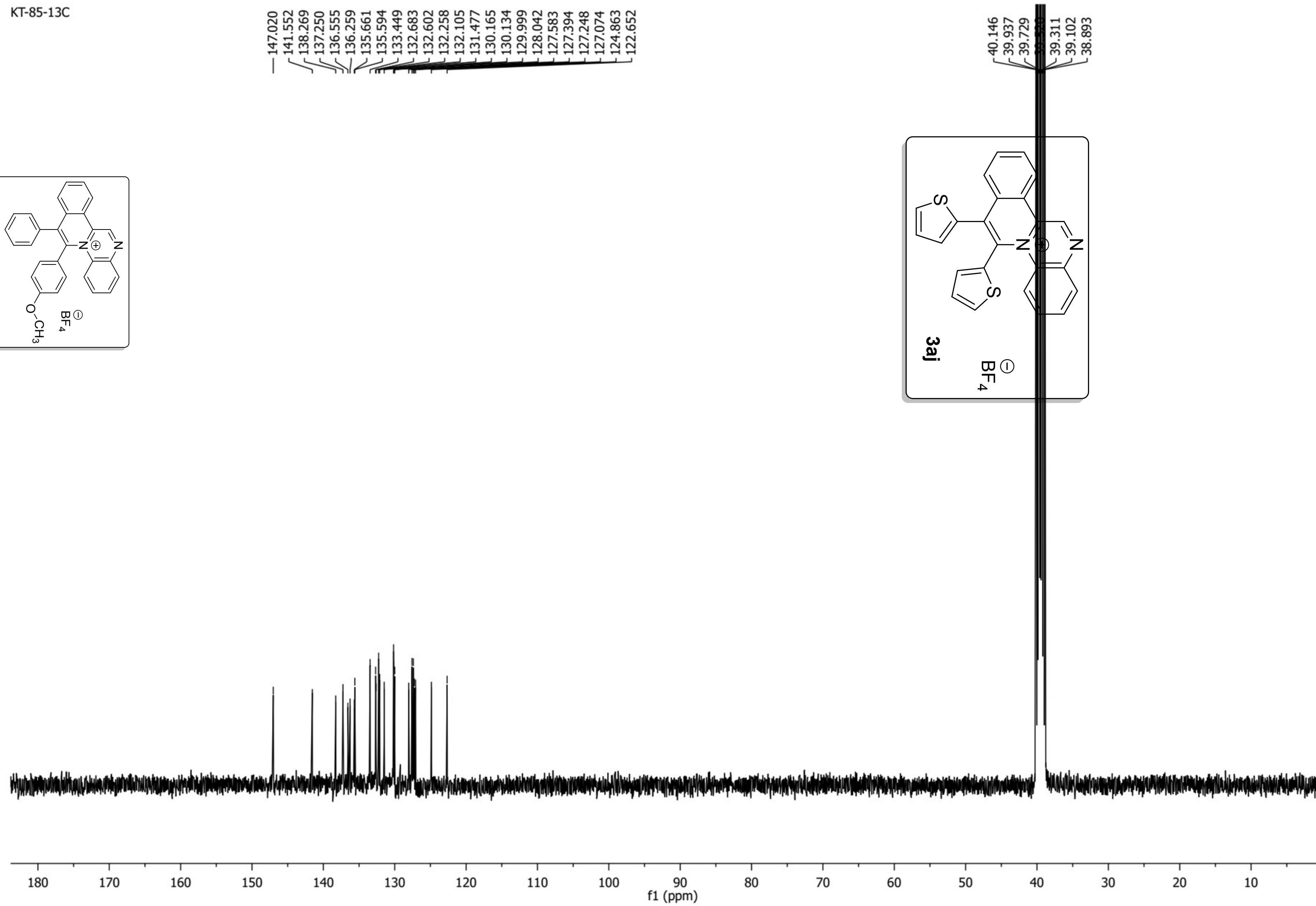
141.216
136.921
136.874
135.238
132.055
131.685
130.980
129.942
127.703
127.255
125.450
124.322
124.234
39.938
39.729
39.520
39.311
39.102
35.655
31.399
24.253
22.754
14.360
13.880
3ai
f1 (ppm)


KT-85-1H
3aj
f1 (ppm)

KT-85-13C



3al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135.594
133.449
132.683
132.602
132.258
132.105
131.477
130.165
130.134
129.999
128.042
127.583
127.394
127.248
127.074
124.863
122.652
40.146
39.937
39.729
39.311
39.102
38.893
3aj
f1 (ppm)
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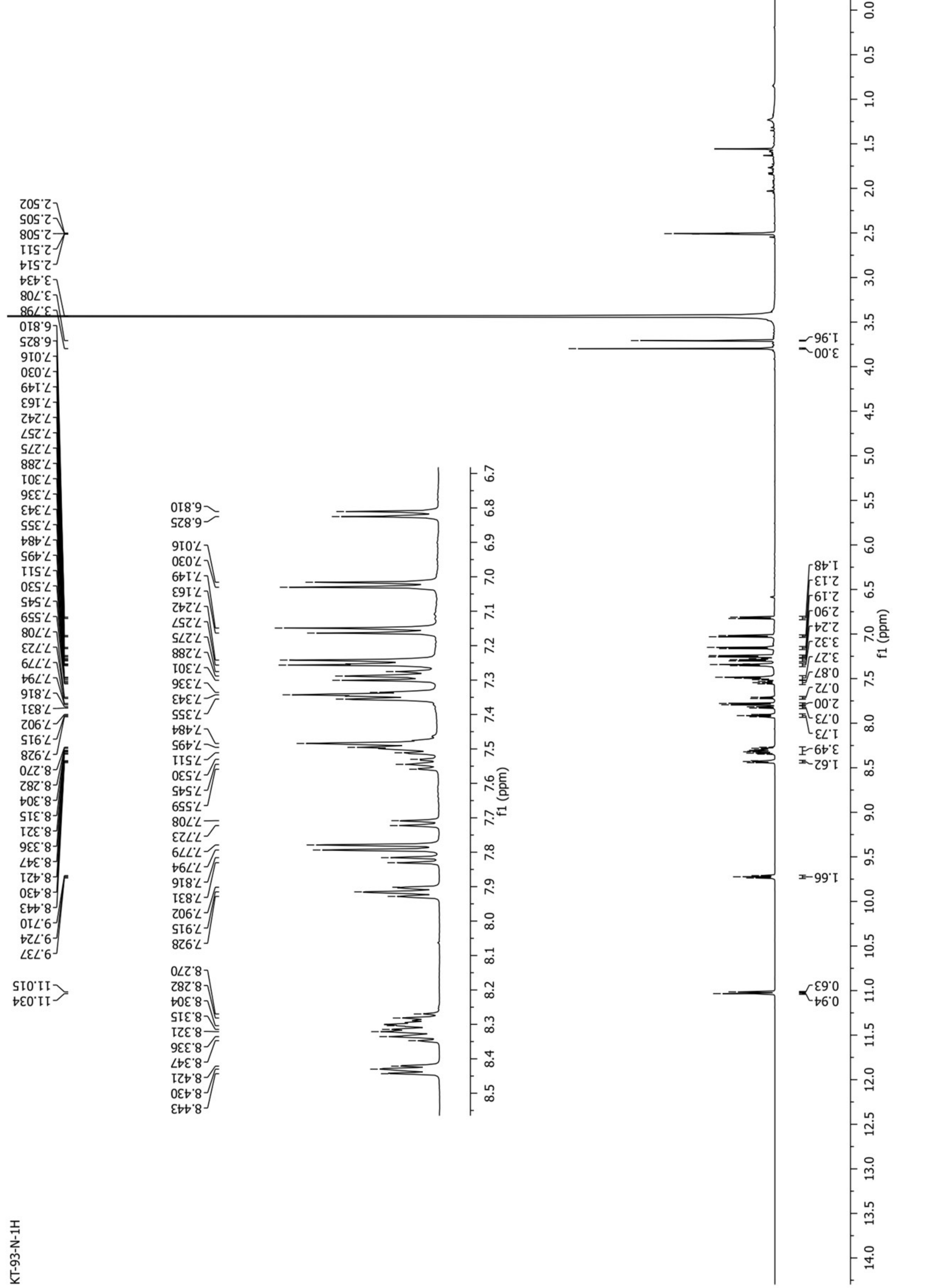
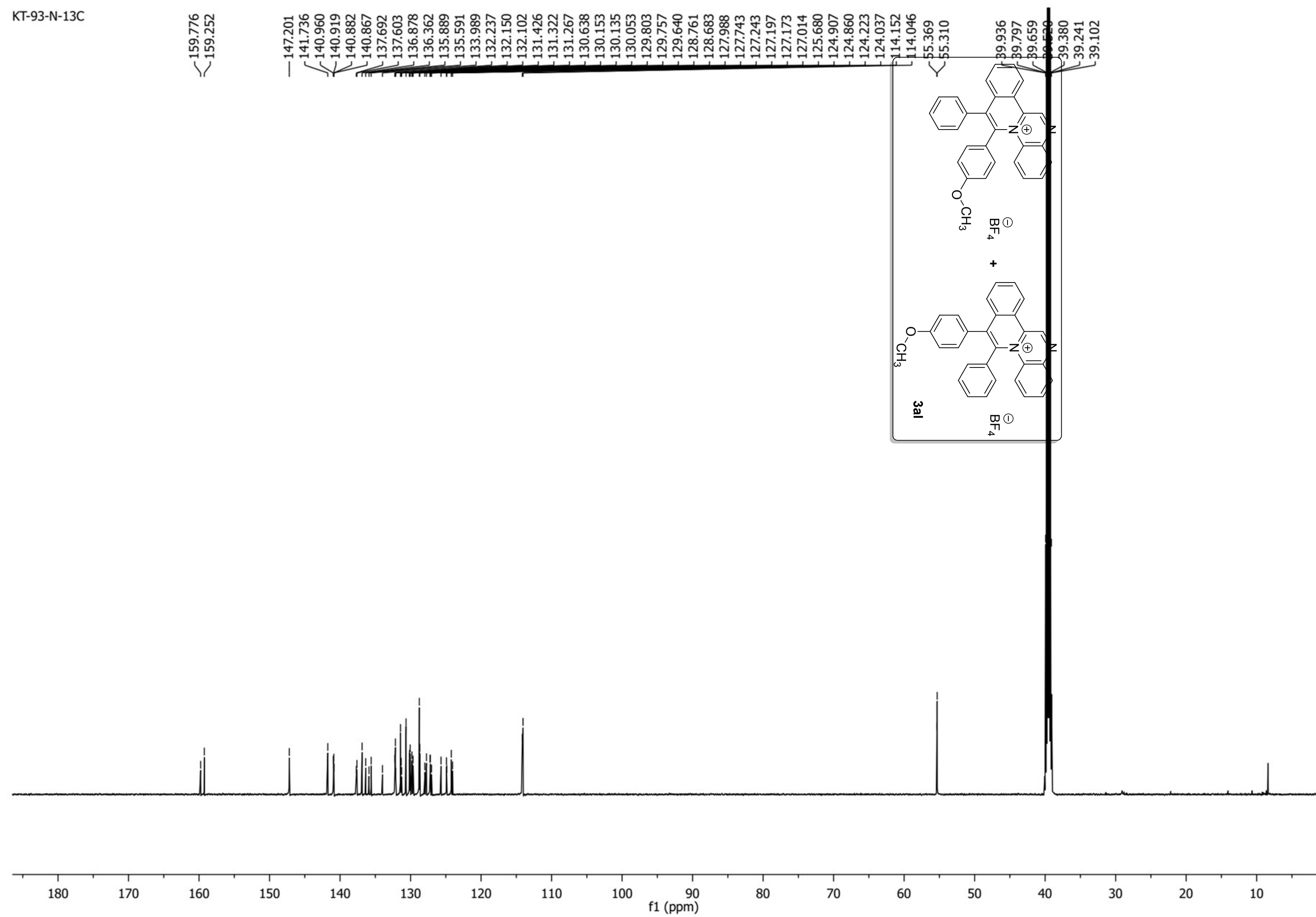
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


KT-93-N-1H
f1 (ppm)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140.919
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140.867
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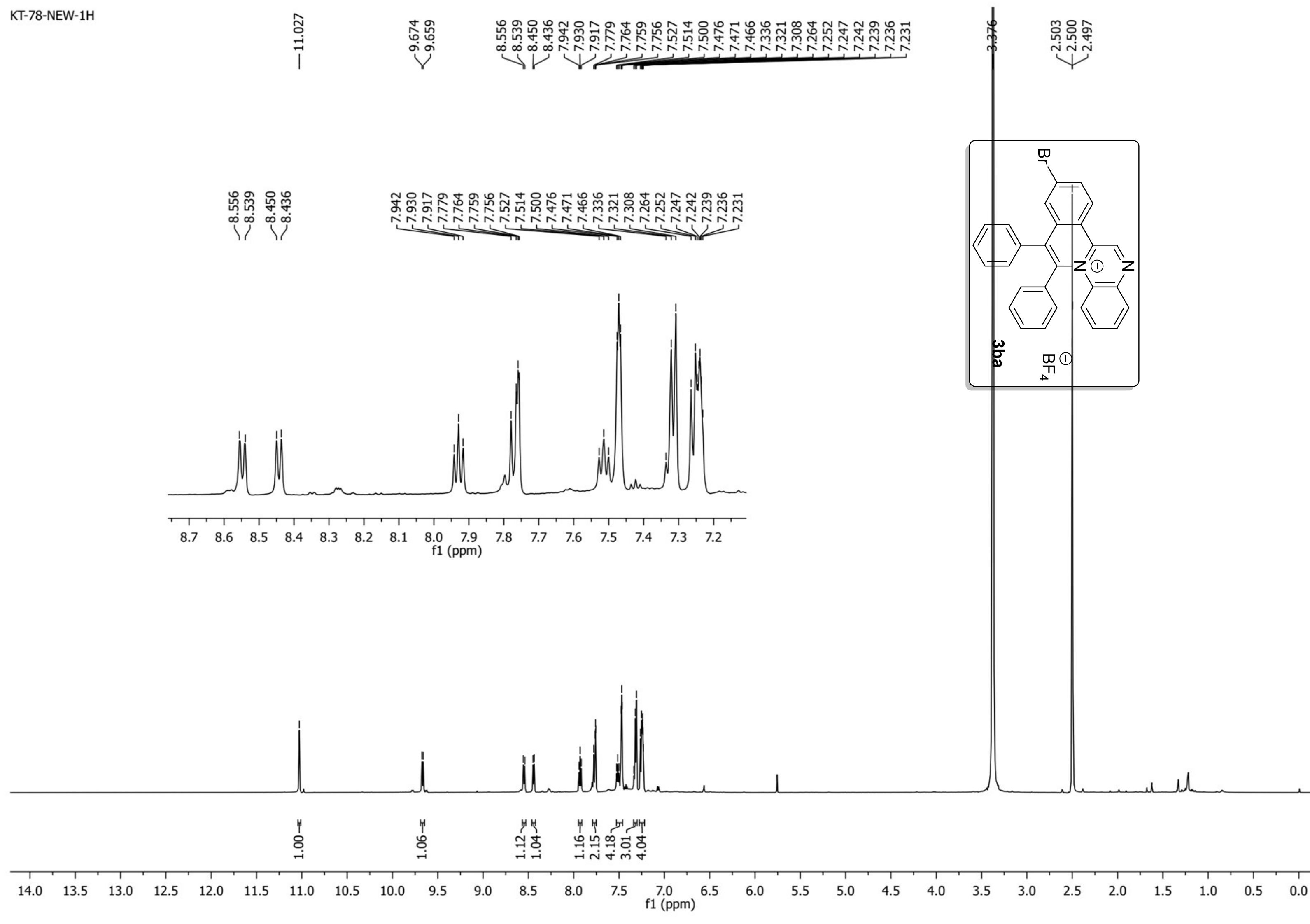
137.603
136.878
136.362
135.889
135.591
133.989
132.237
132.150
132.102
131.426
131.322
131.267
130.638
130.153
130.135
130.053
129.803
129.757
129.640
128.761
128.683
127.988
127.743
127.243
127.197
127.173
127.014
125.680
124.907
124.860
124.223
124.037
114.152
114.046
55.369
55.310
39.936
39.797
39.659
39.380
39.241
39.102
3al
f1 (ppm)


KT-78-NEW-1H
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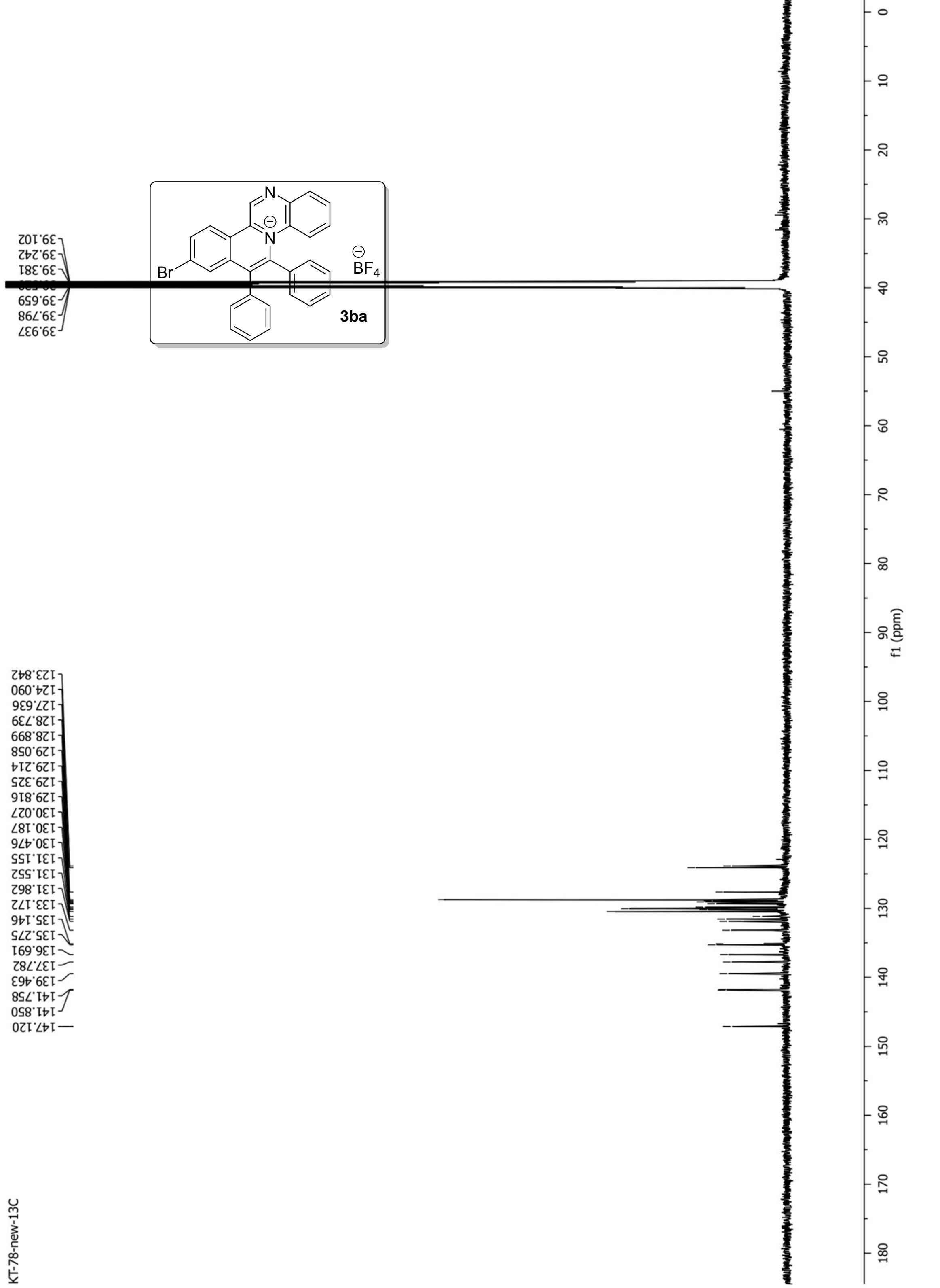
9.674
9.659
8.556
8.539
8.450
8.436
7.942
7.930
7.917
7.779
7.764
7.759
7.756
7.527
7.514
7.500
7.476
7.471
7.466
7.336
7.321
7.308
7.264
7.252
7.247
7.242
7.239
7.236
7.231
3.376
2.503
2.500
2.497
Br
N
N
BF4
3ba
1.00
1.06
1.12
1.04
1.16
2.15
4.18
3.01
4.04
f1 (ppm)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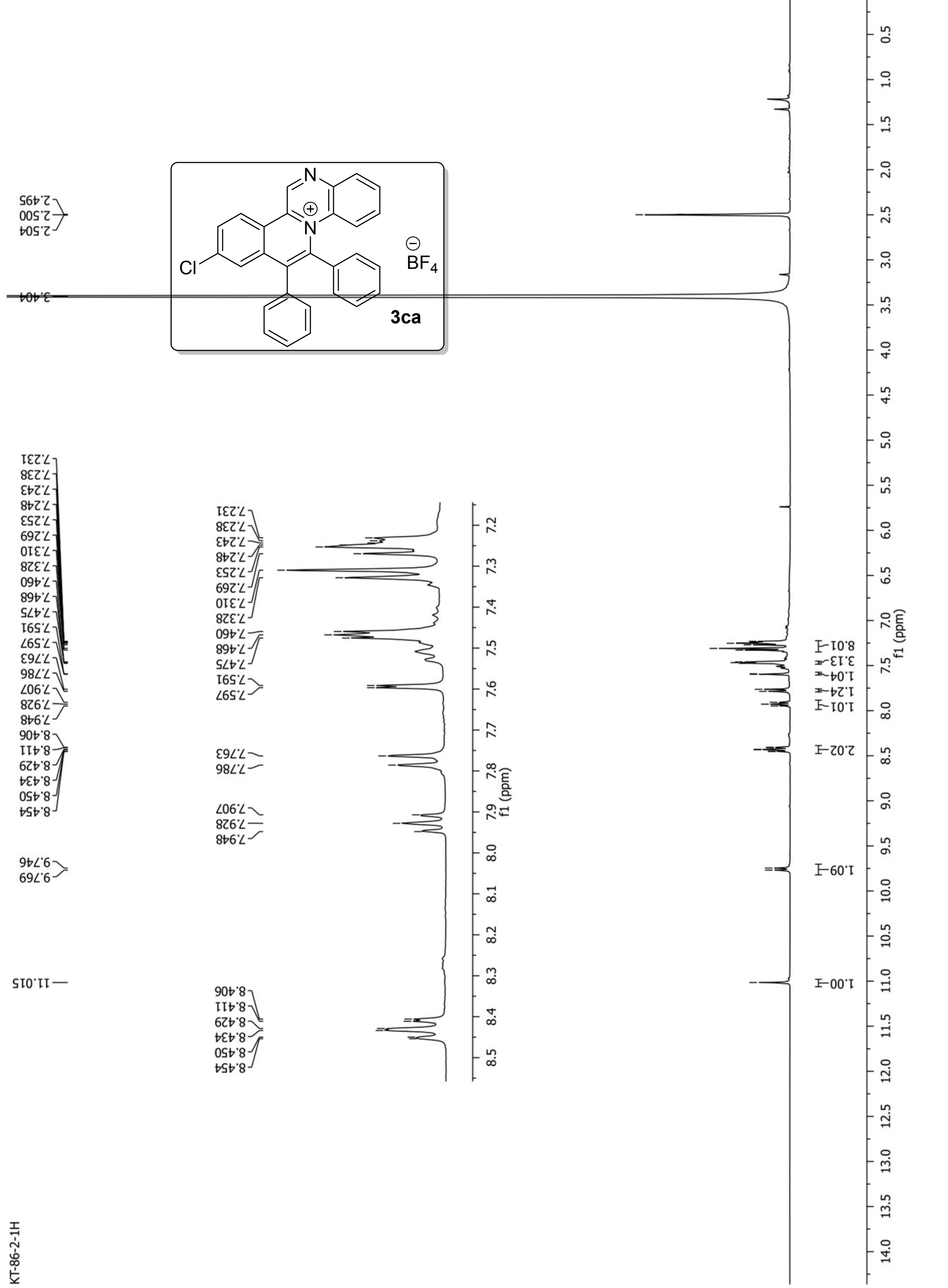
141.758
139.463
137.782
136.691
135.275
135.146
133.172
131.862
131.552
131.155
130.476
130.187
130.027
129.816
129.325
129.214
129.058
128.899
128.739
127.636
124.090
123.842
39.937
39.798
39.659
39.381
39.242
39.102
Br
N
N
BF4
3ba
180
170
160
150
140
130
120
110
100
90
f1 (ppm)
80
70
60
50
40
30
20
10
0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Cl
BF4
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2.500
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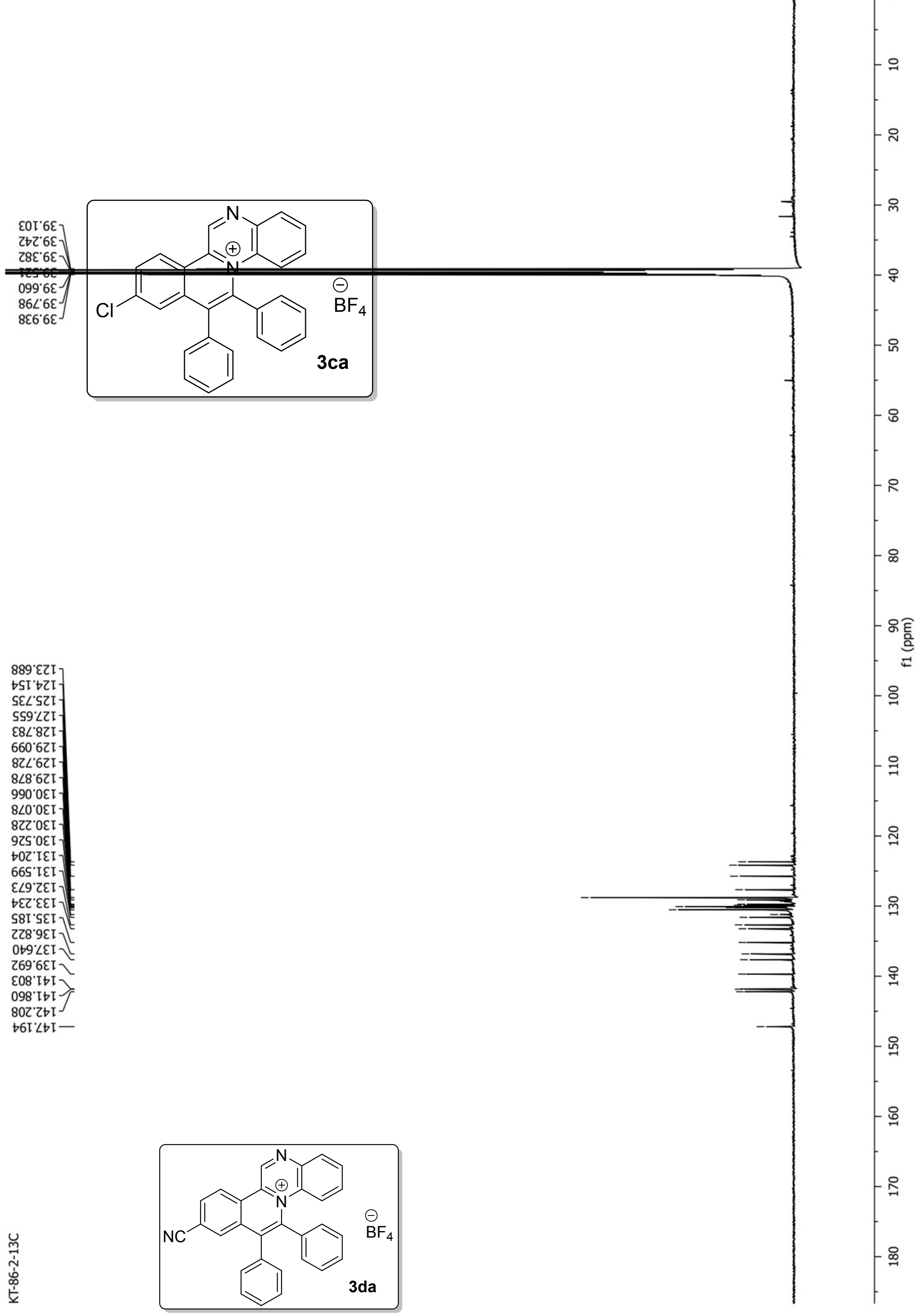
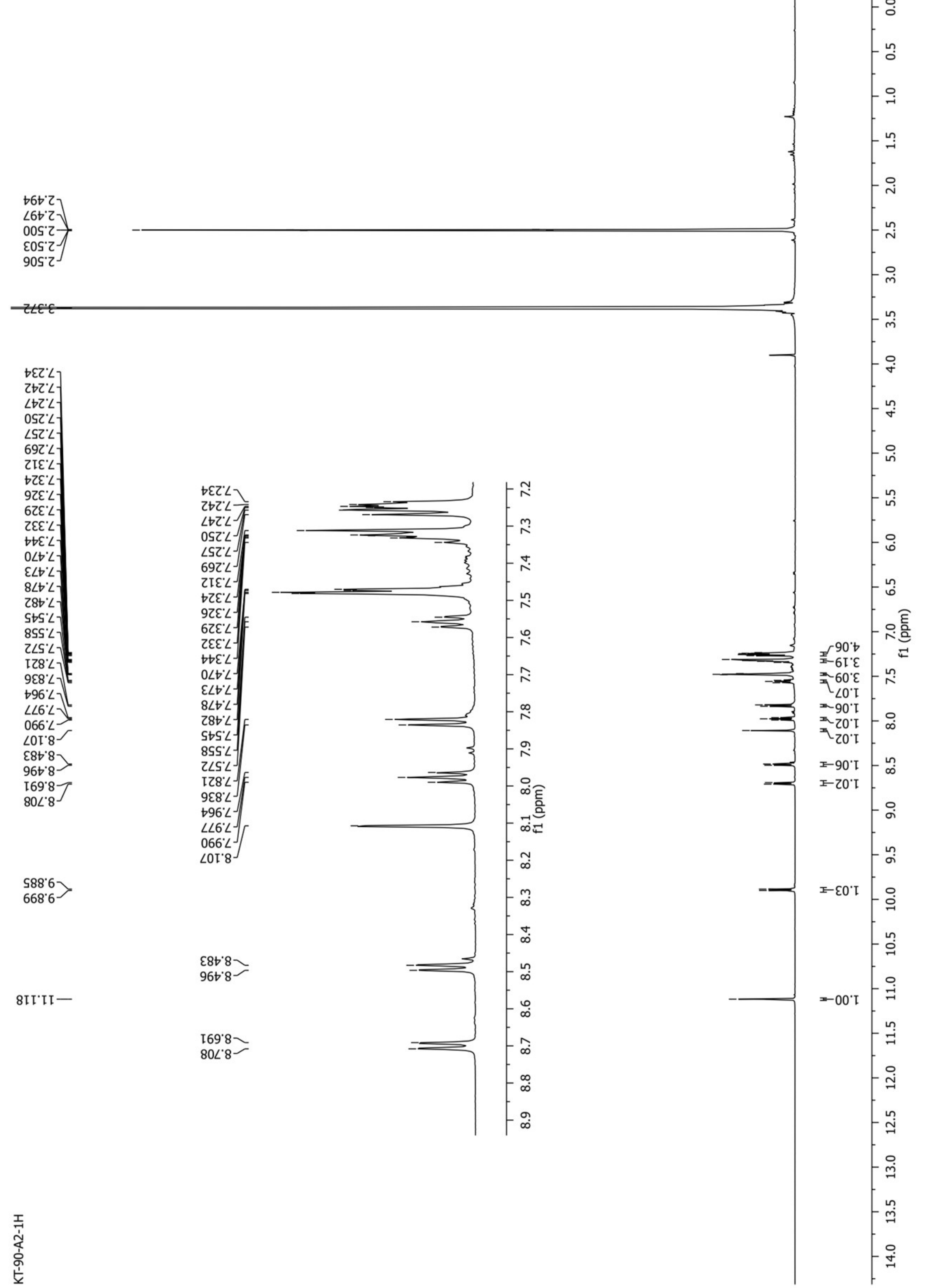
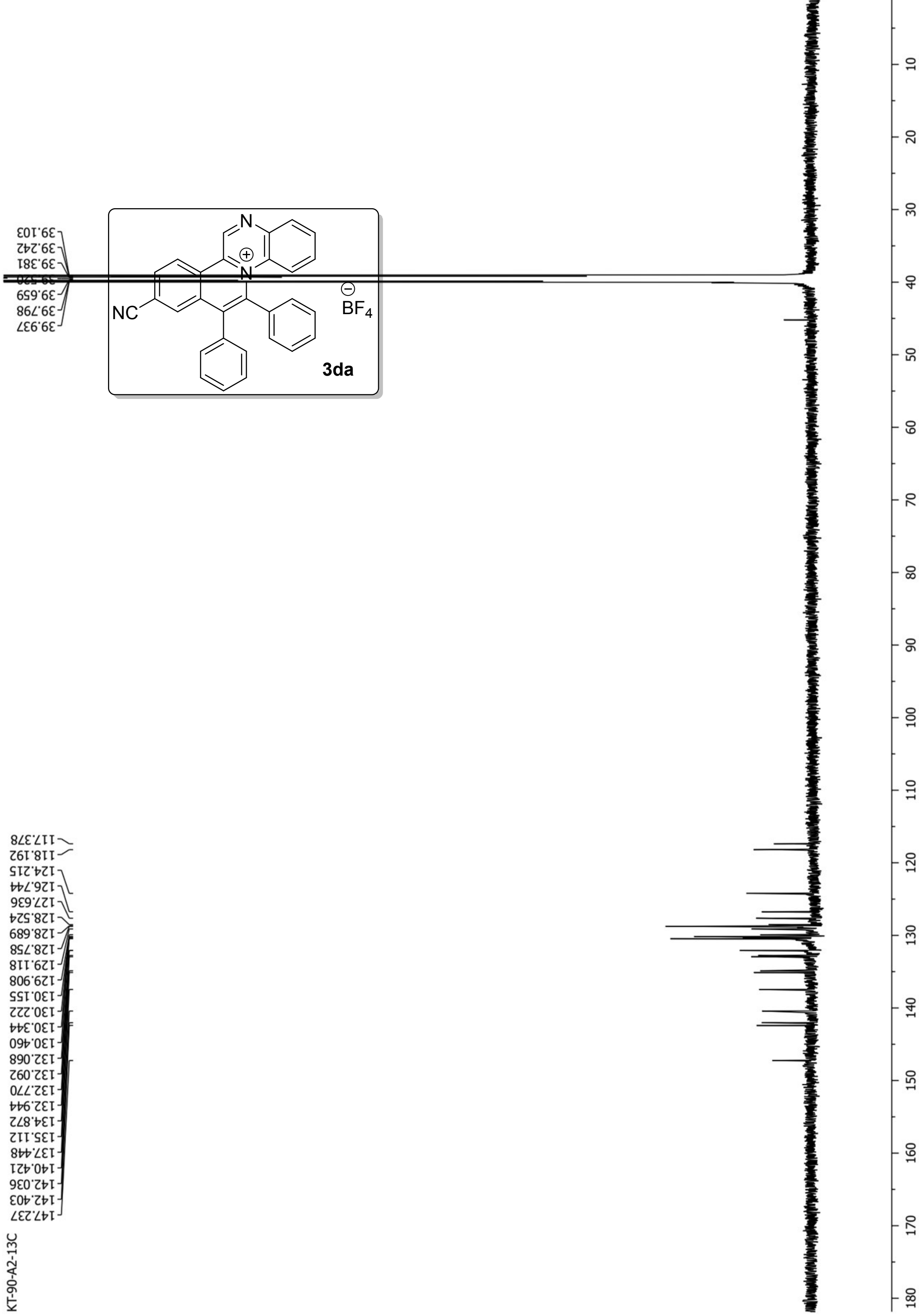
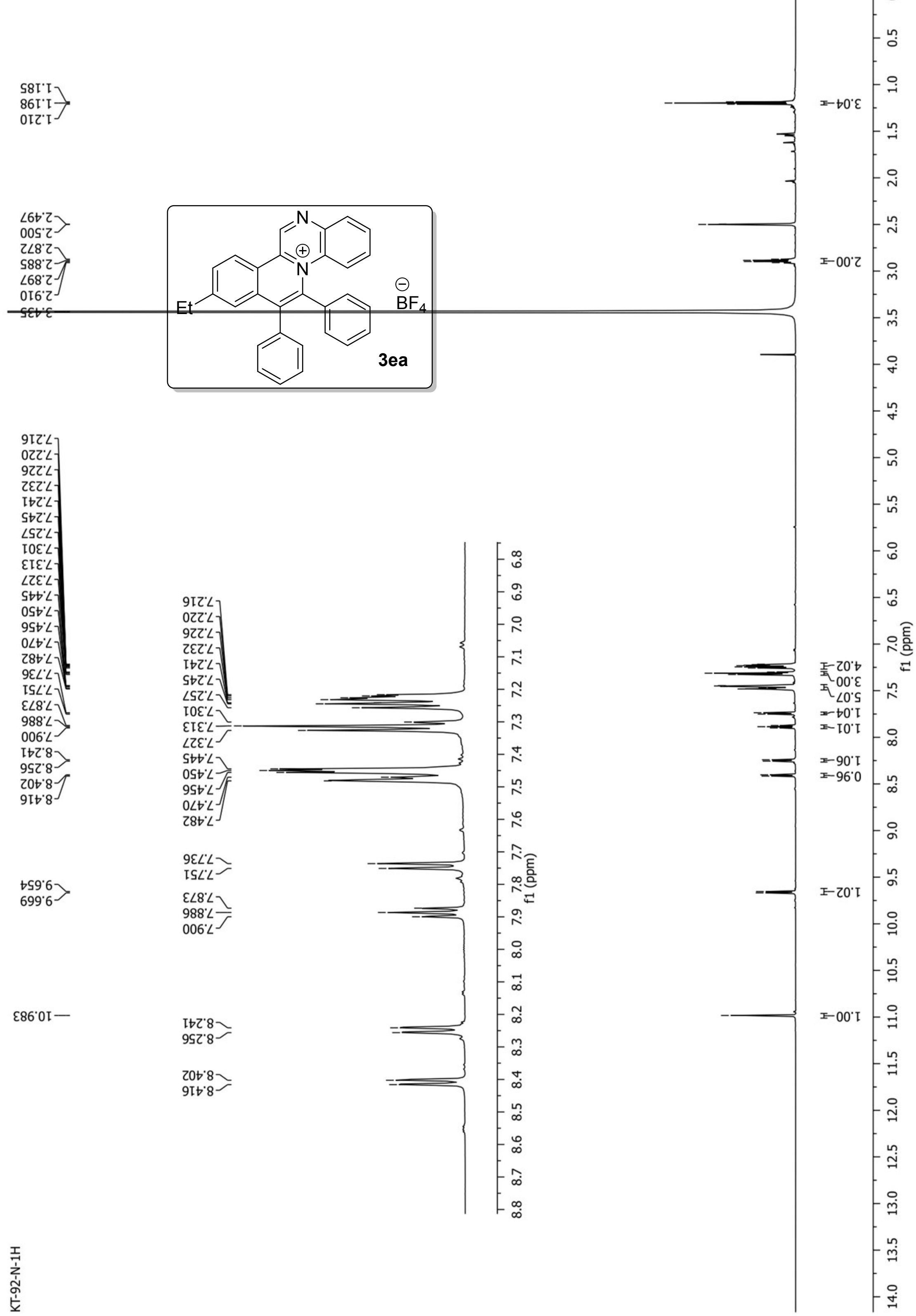
7.253
7.269
7.310
7.328
7.460
7.468
7.475
7.591
7.597
7.763
7.786
7.907
7.928
7.948
8.406
8.411
8.429
8.434
8.450
8.454
9.746
9.769
11.015
8.01
3.13
1.04
1.24
1.01
2.02
1.09
1.00
f1 (ppm)

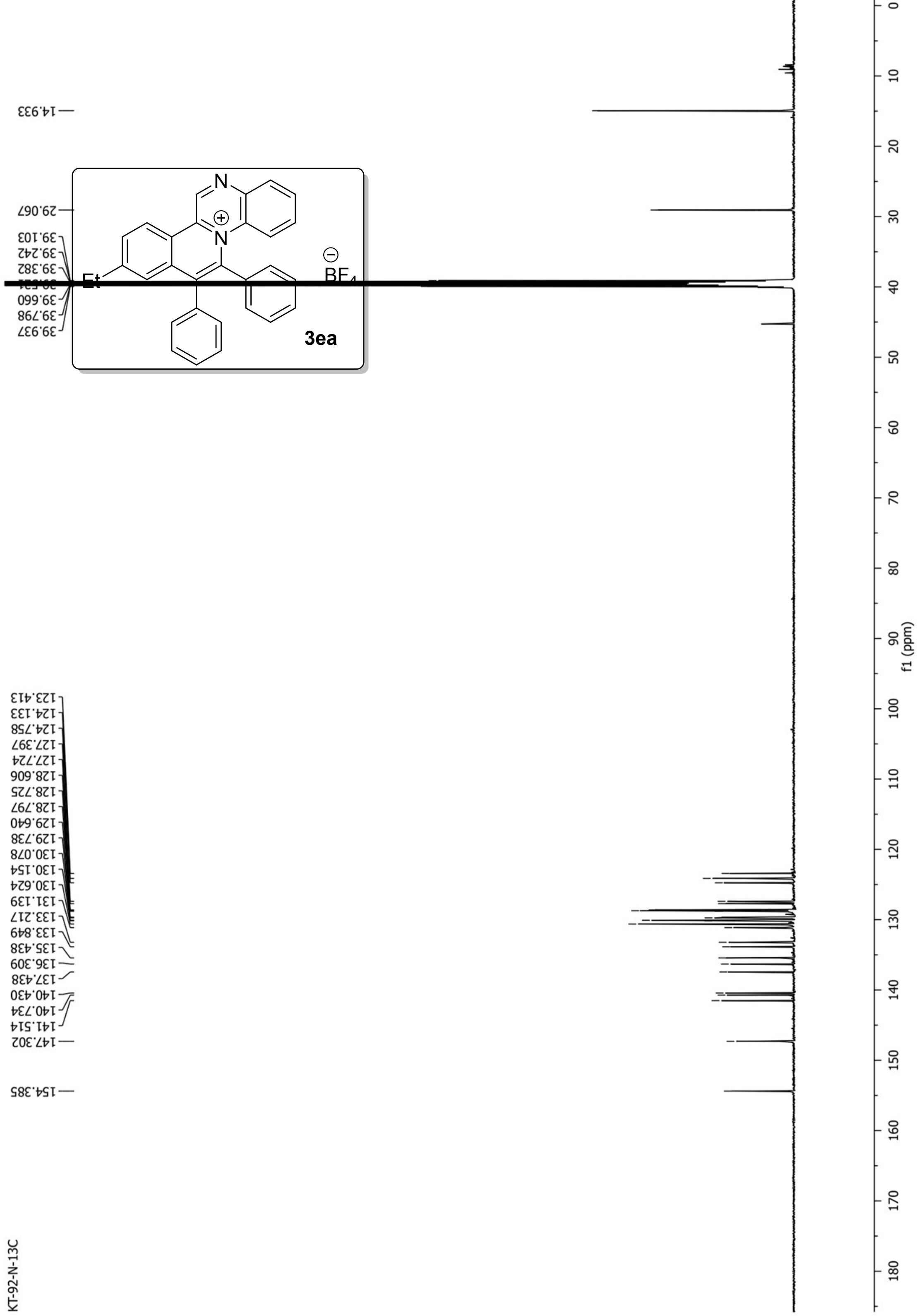

KT-86-2-13C
147.194
142.208
141.860
141.803
139.692
137.640
136.822
135.185
133.234
132.673
131.599
131.204
130.526
130.228
130.078
130.066
129.878
129.728
129.099
128.783
127.655
125.735
124.154
123.688
39.938
39.798
39.660
39.521
39.382
39.242
39.103
3ca
3da
f1 (ppm)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


KT-90-A2-1H
f1 (ppm)

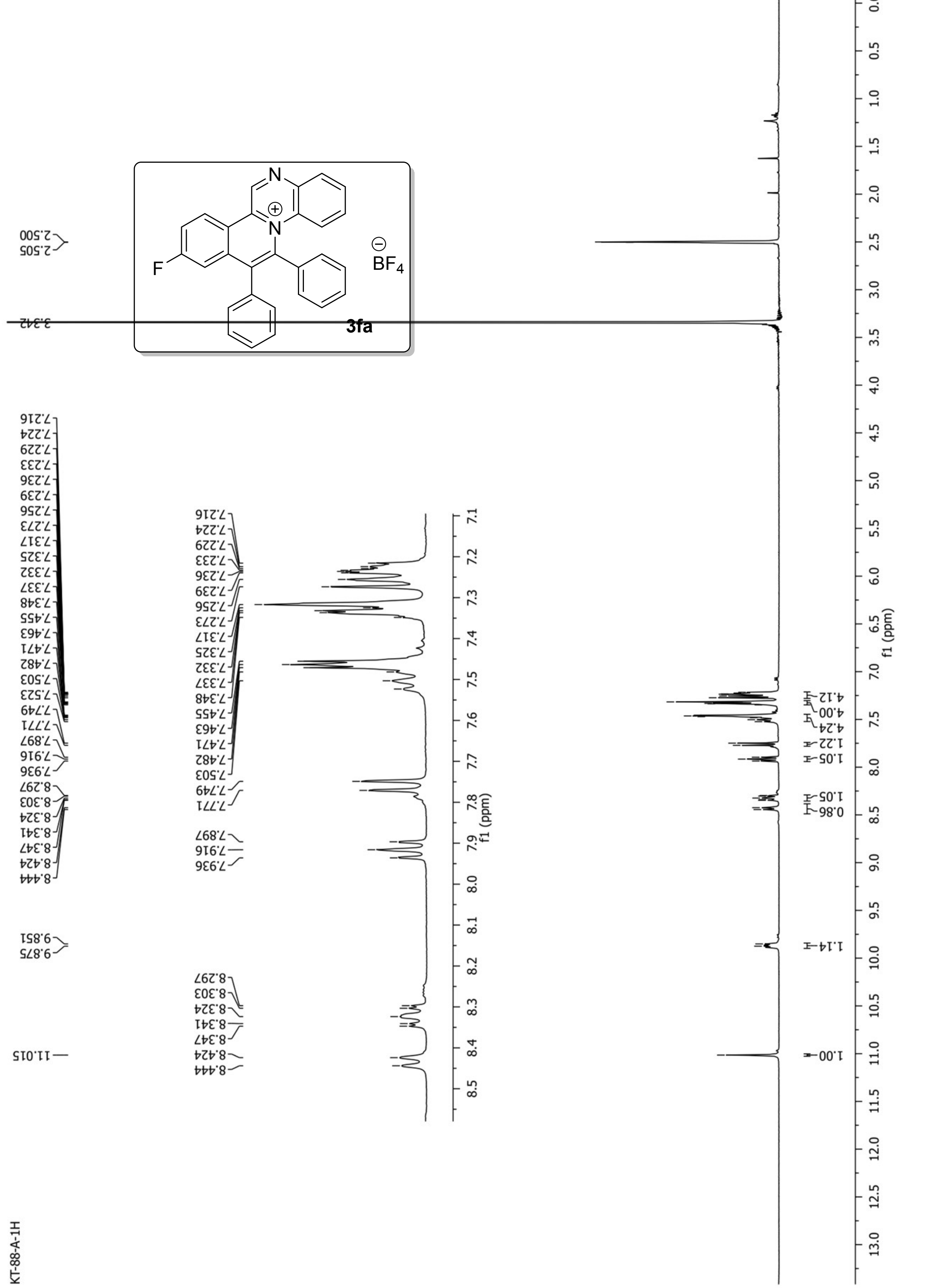


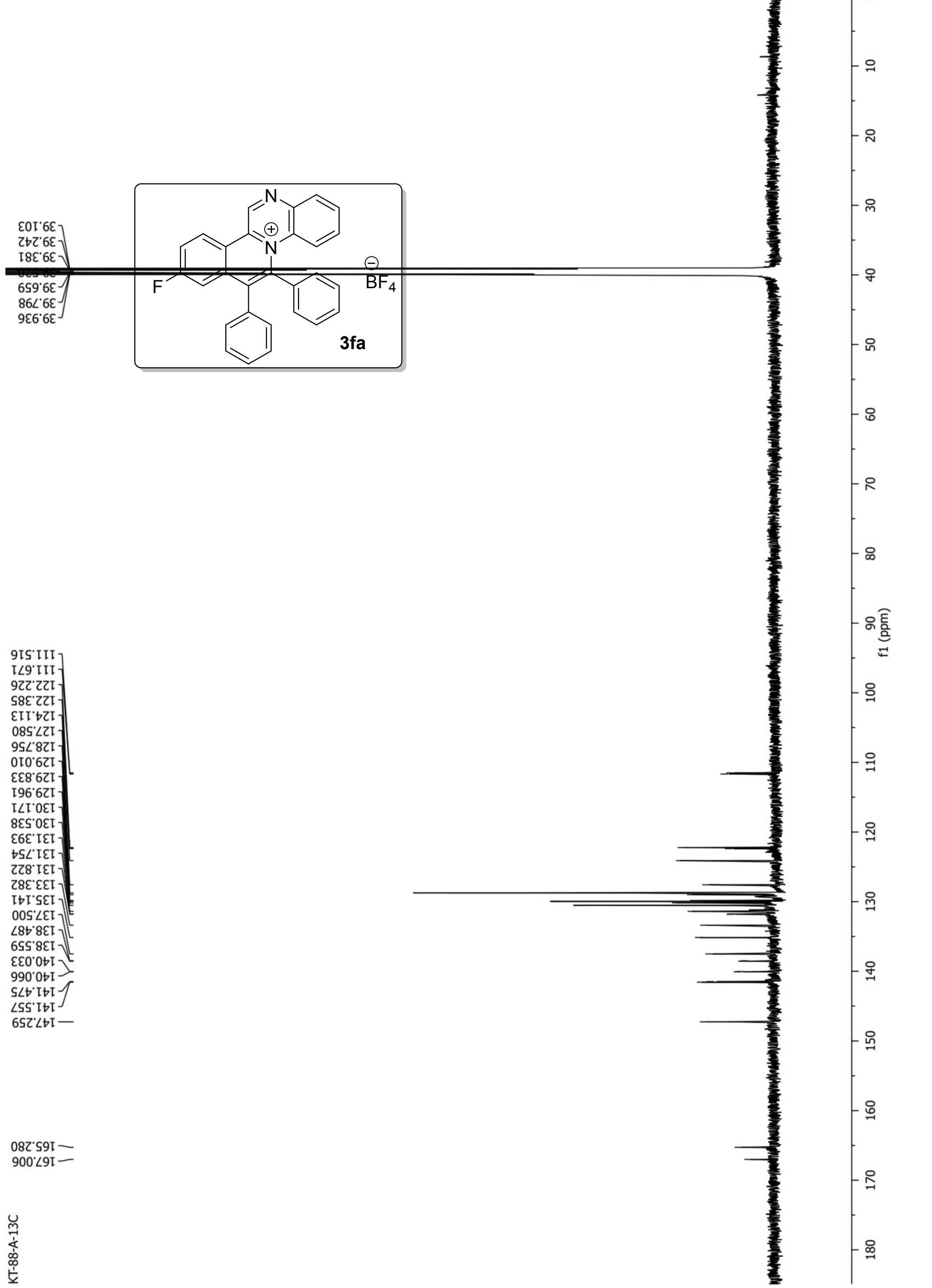
KT-90-A2-13C
39.103
39.242
39.381
39.659
39.798
39.937
117.378
118.192
124.215
126.744
127.636
128.524
128.689
128.758
129.118
129.908
130.155
130.222
130.344
130.460
132.068
132.092
132.770
132.944
134.872
135.112
137.448
140.421
142.036
142.403
147.237
NC
N
N
BF4
3da
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180

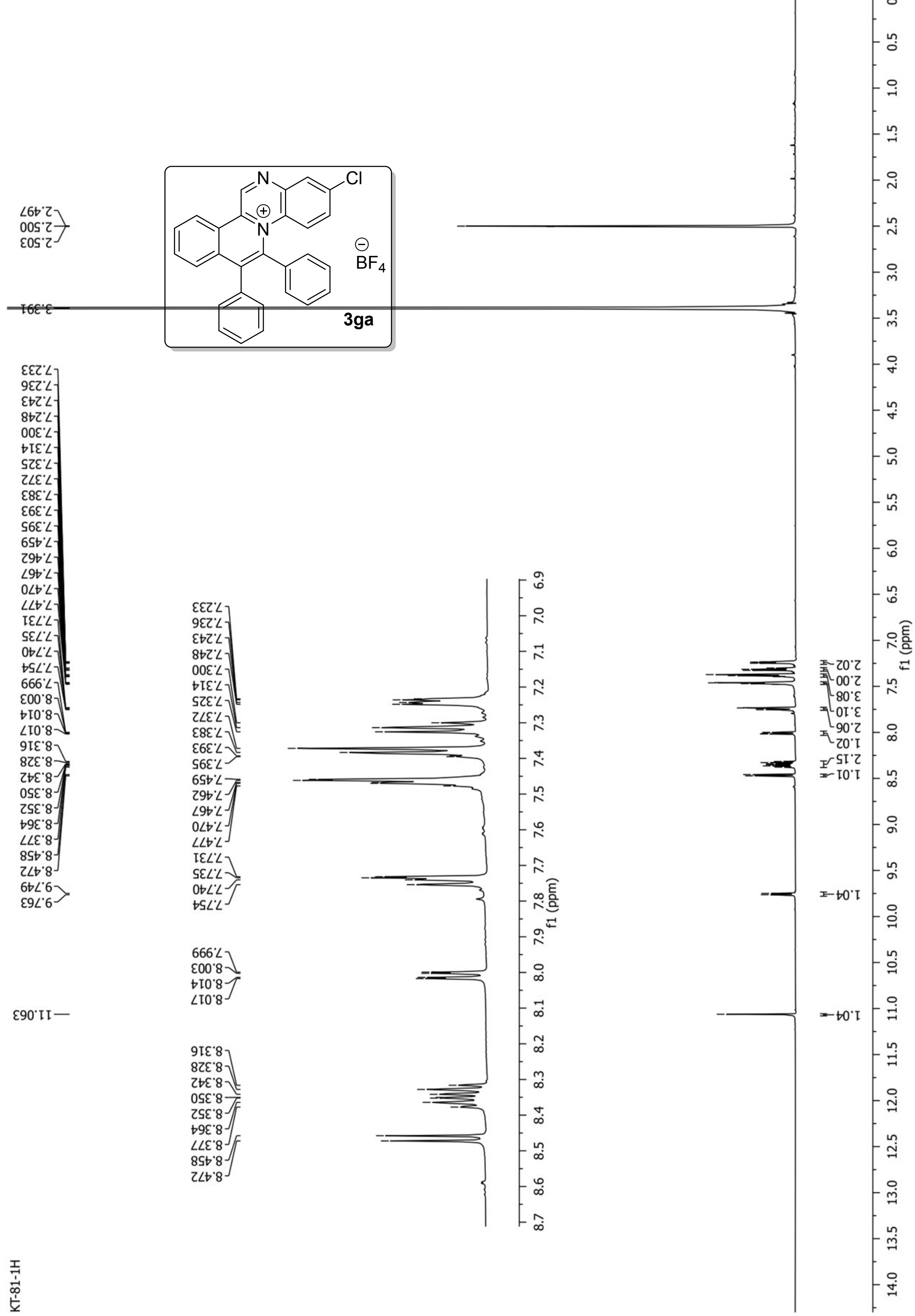

KT-92-N-1H
3ea
Et
BF_4
1.185
1.198
1.210
2.497
2.500
2.872
2.885
2.897
2.910
3.435
7.216
7.220
7.226
7.232
7.241
7.245
7.257
7.301
7.313
7.327
7.445
7.450
7.456
7.470
7.482
7.736
7.751
7.873
7.886
7.900
8.241
8.256
8.402
8.416
9.654
9.669
10.983
3.04
2.00
4.02
3.00
5.07
1.04
1.01
1.06
0.96
1.02
1.00
f1 (ppm)

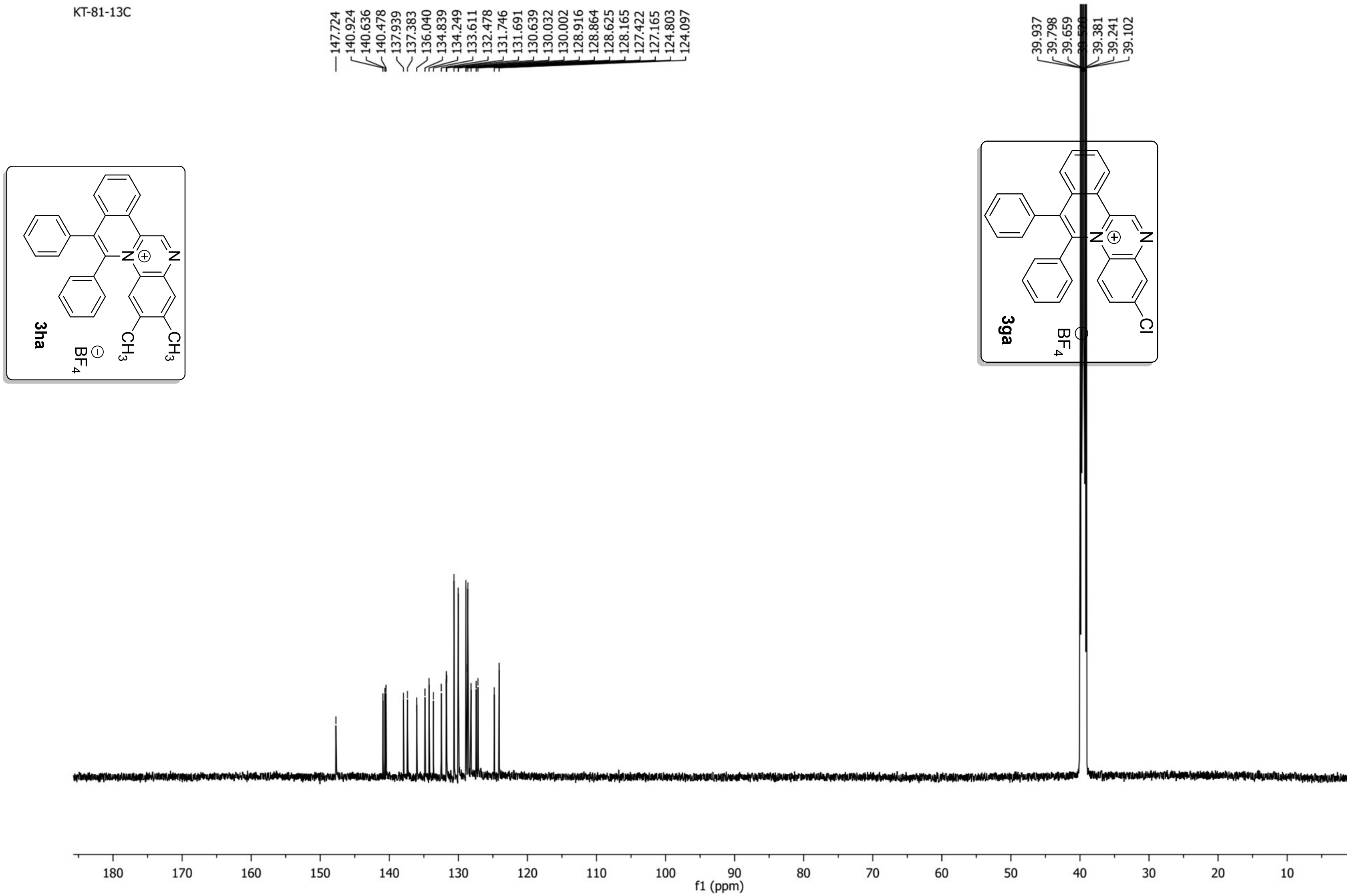


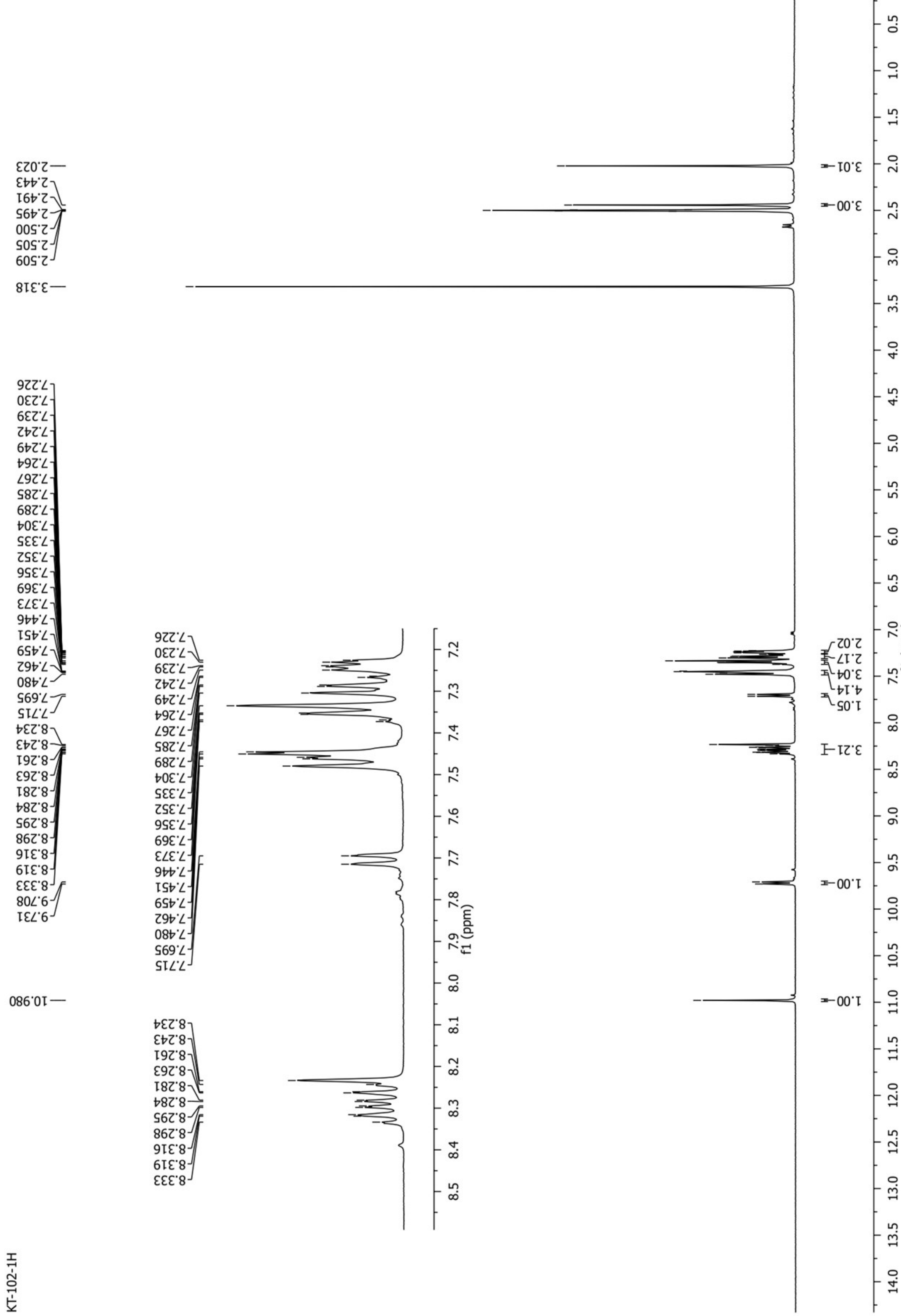
KT-92-N-13C
154.385
147.302
141.514
140.734
140.430
137.438
136.309
135.438
133.849
133.217
131.139
130.624
130.154
130.078
129.738
129.640
128.797
128.725
128.606
127.724
127.397
124.758
124.133
123.413
39.937
39.798
39.660
39.382
39.242
39.103
29.067
14.933
N
N
Et
BF4
3ea
f1 (ppm)

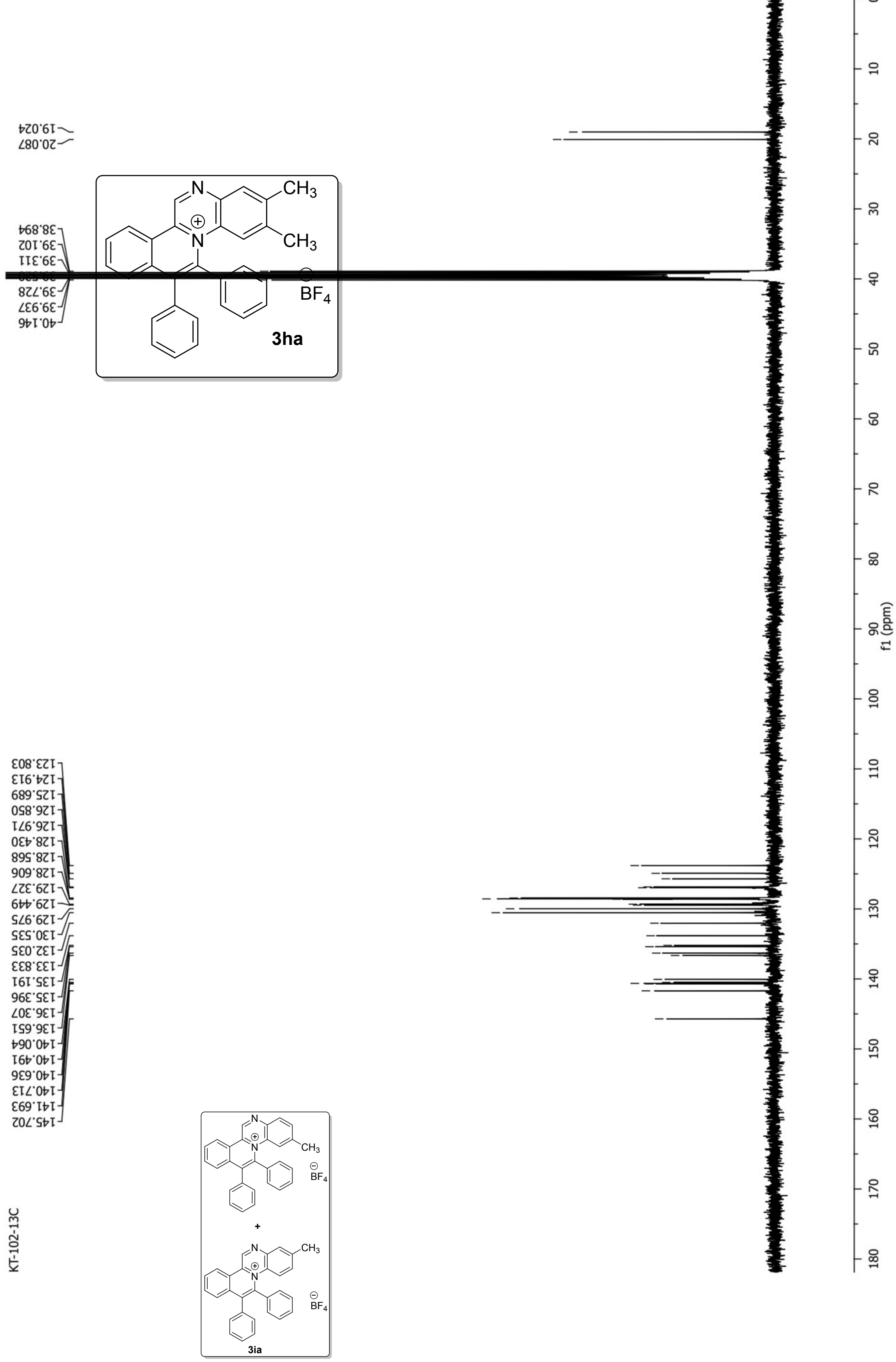

3fa
KT-88-A-1H
f1 (ppm)


KT-88-A-13C
3fa
F
N
BF4
39.103
39.242
39.381
39.659
39.798
39.936
111.516
111.671
122.226
122.385
124.113
127.580
128.756
129.010
129.833
129.961
130.171
130.538
131.393
131.754
131.822
133.382
135.141
137.500
138.487
138.559
140.033
140.066
141.475
141.557
147.259
165.280
167.006
f1 (ppm)

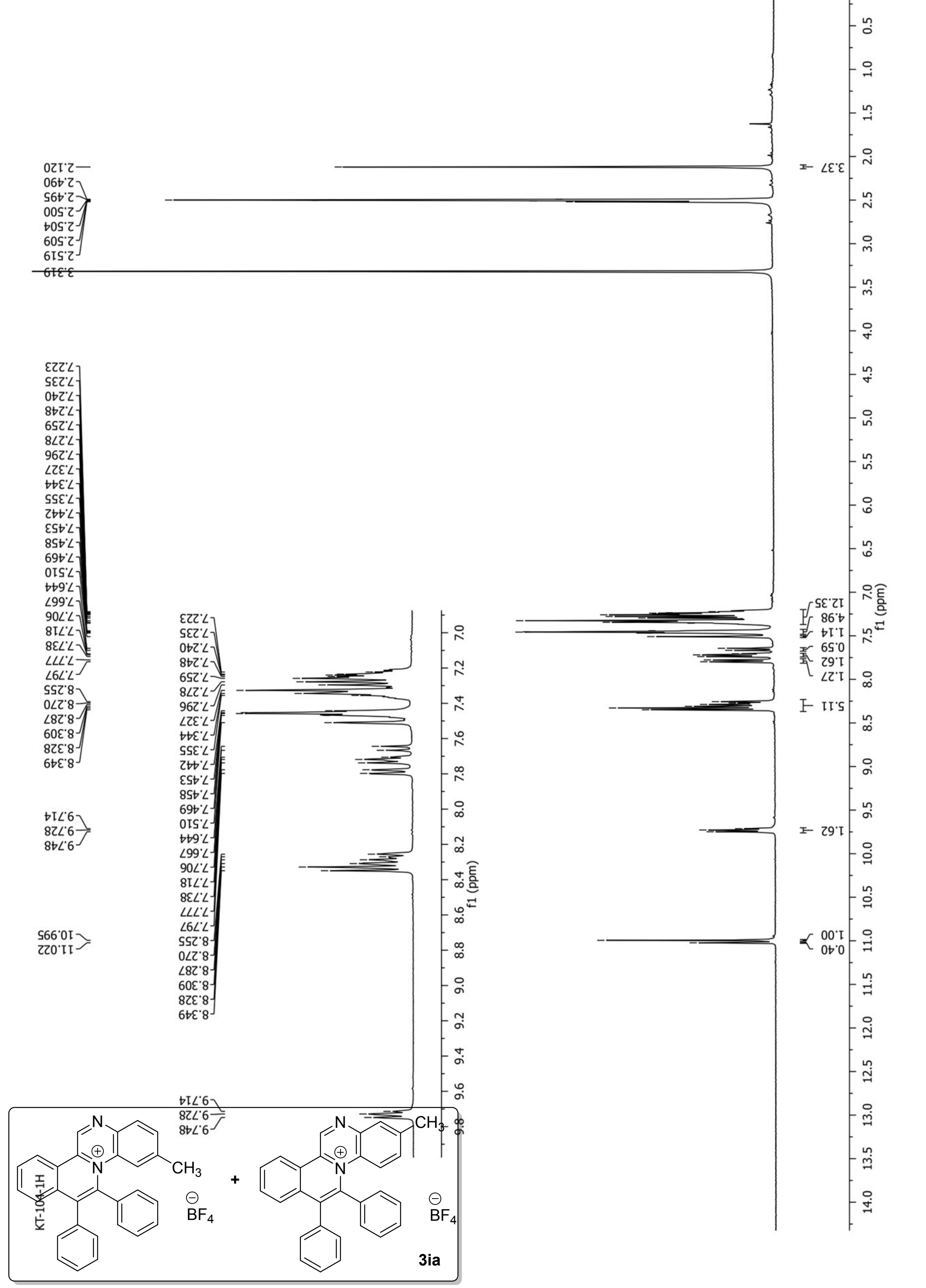

KT-81-1H
3ga
f1 (ppm)

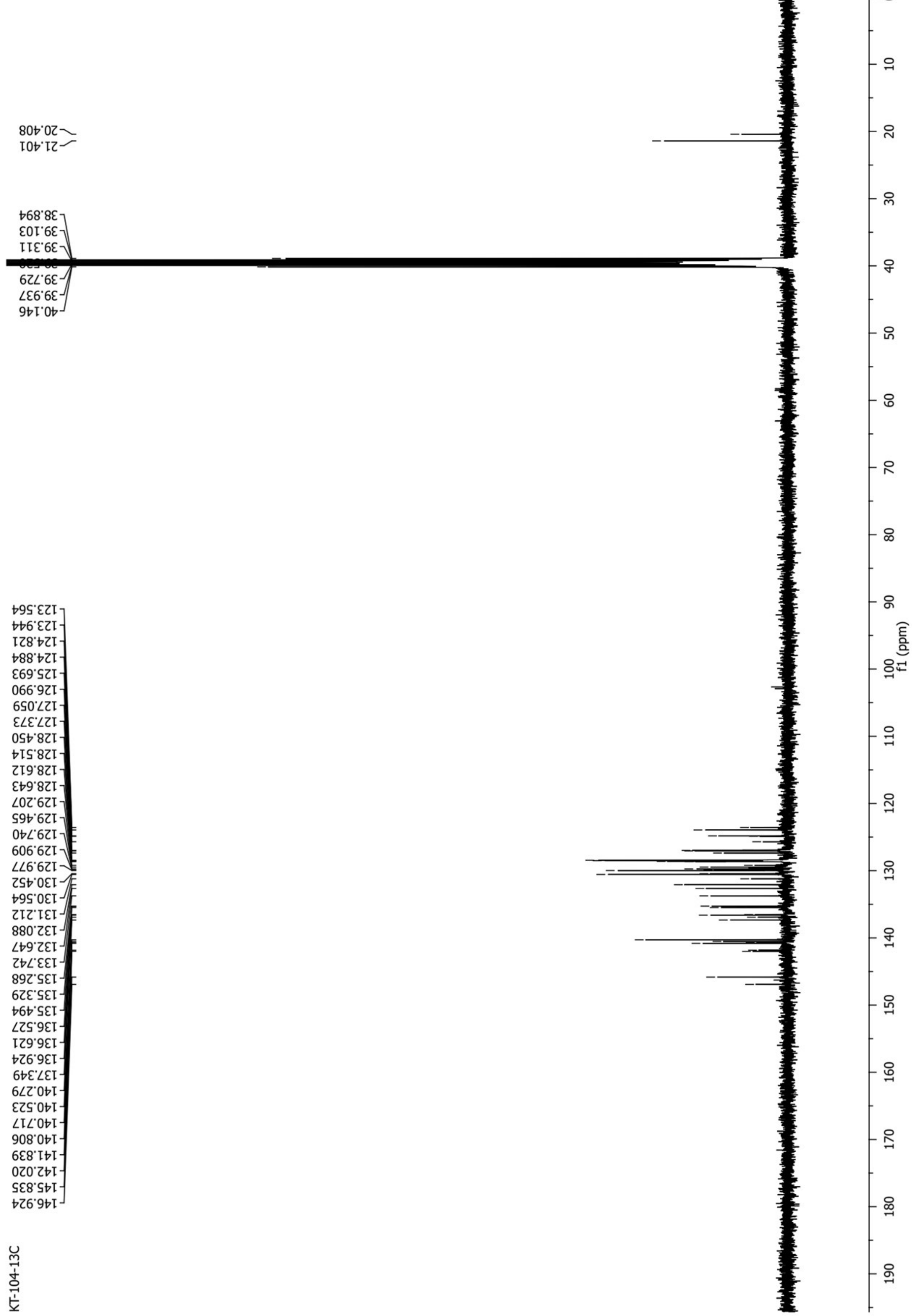

KT-81-13C
147.724
140.924
140.636
140.478
137.939
137.383
136.040
134.839
134.249
133.611
132.478
131.746
131.691
130.639
130.032
130.002
128.916
128.864
128.625
128.165
127.422
127.165
124.803
124.097
39.937
39.798
39.659
39.381
39.241
39.102
N
N
CH3
CH3
BF4
3ha
N
N
Cl
BF4
3ga
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

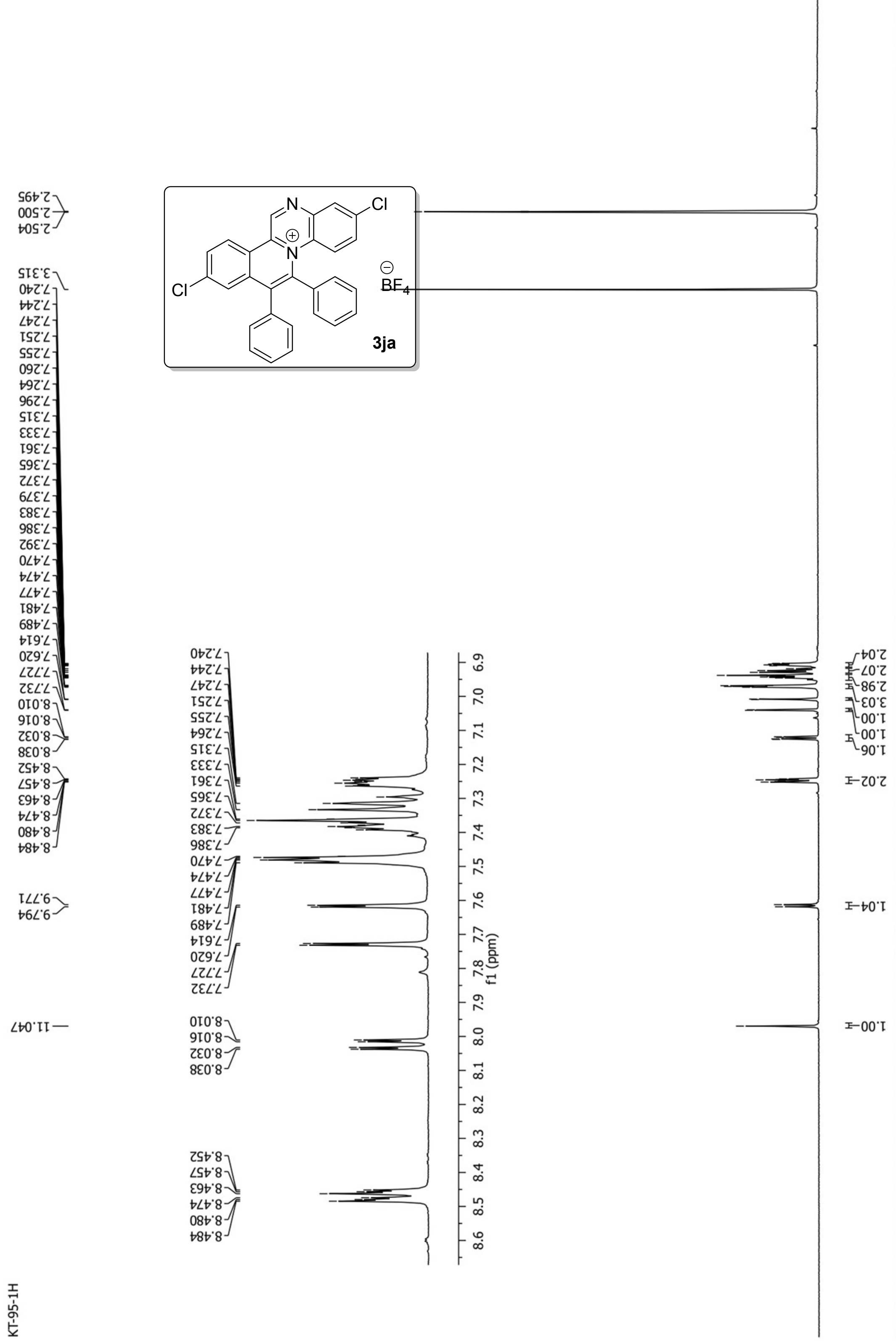

KT-102-1H
f1 (ppm)



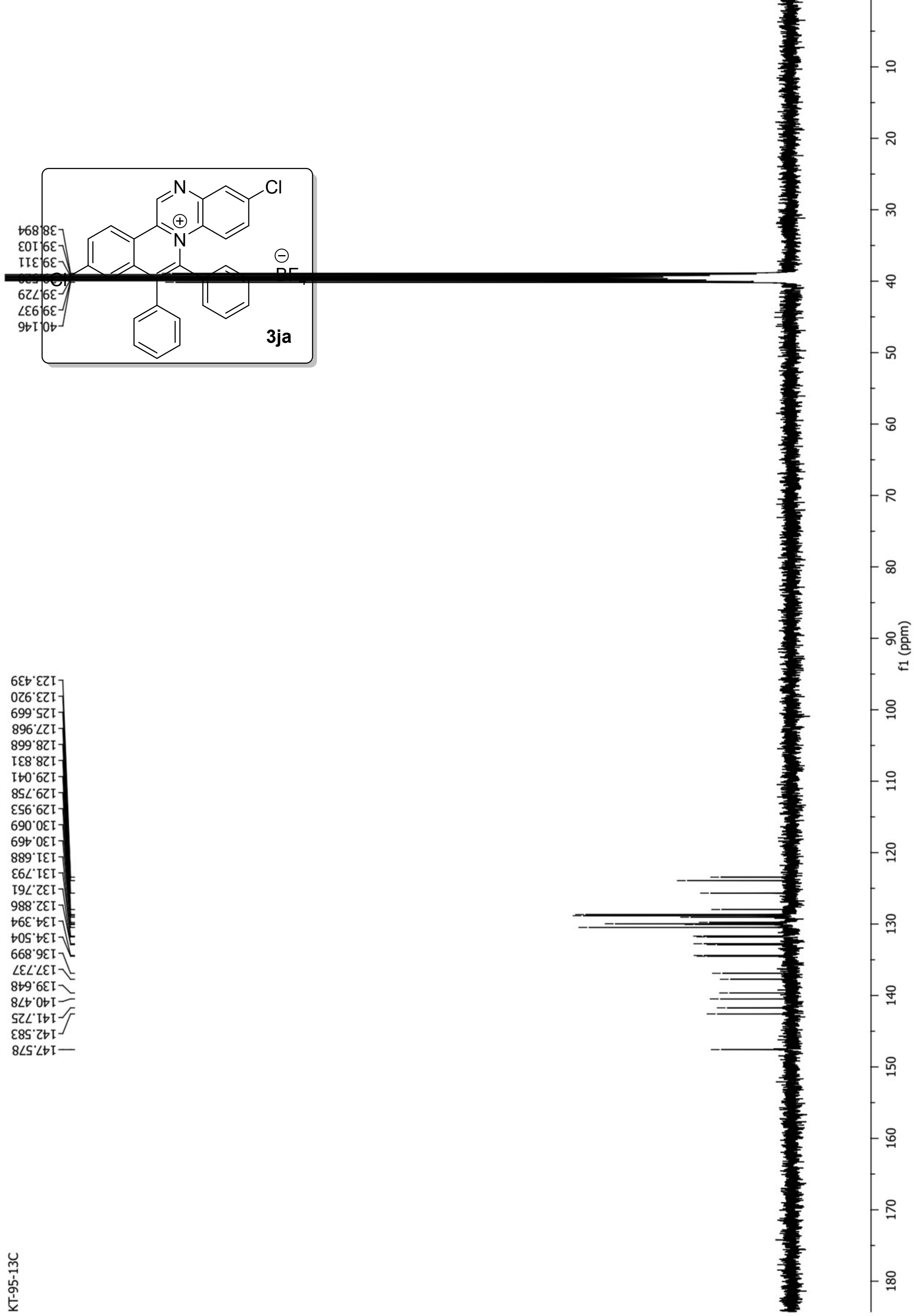
19.024
20.087
38.894
39.102
39.311
39.728
39.937
40.146
123.803
124.913
125.689
126.850
126.971
128.430
128.568
128.606
129.327
129.449
129.975
130.535
132.035
133.833
135.191
135.396
136.307
136.651
140.064
140.491
140.636
140.713
141.693
145.702
KT-102-13C
3ha
3ia
f1 (ppm)

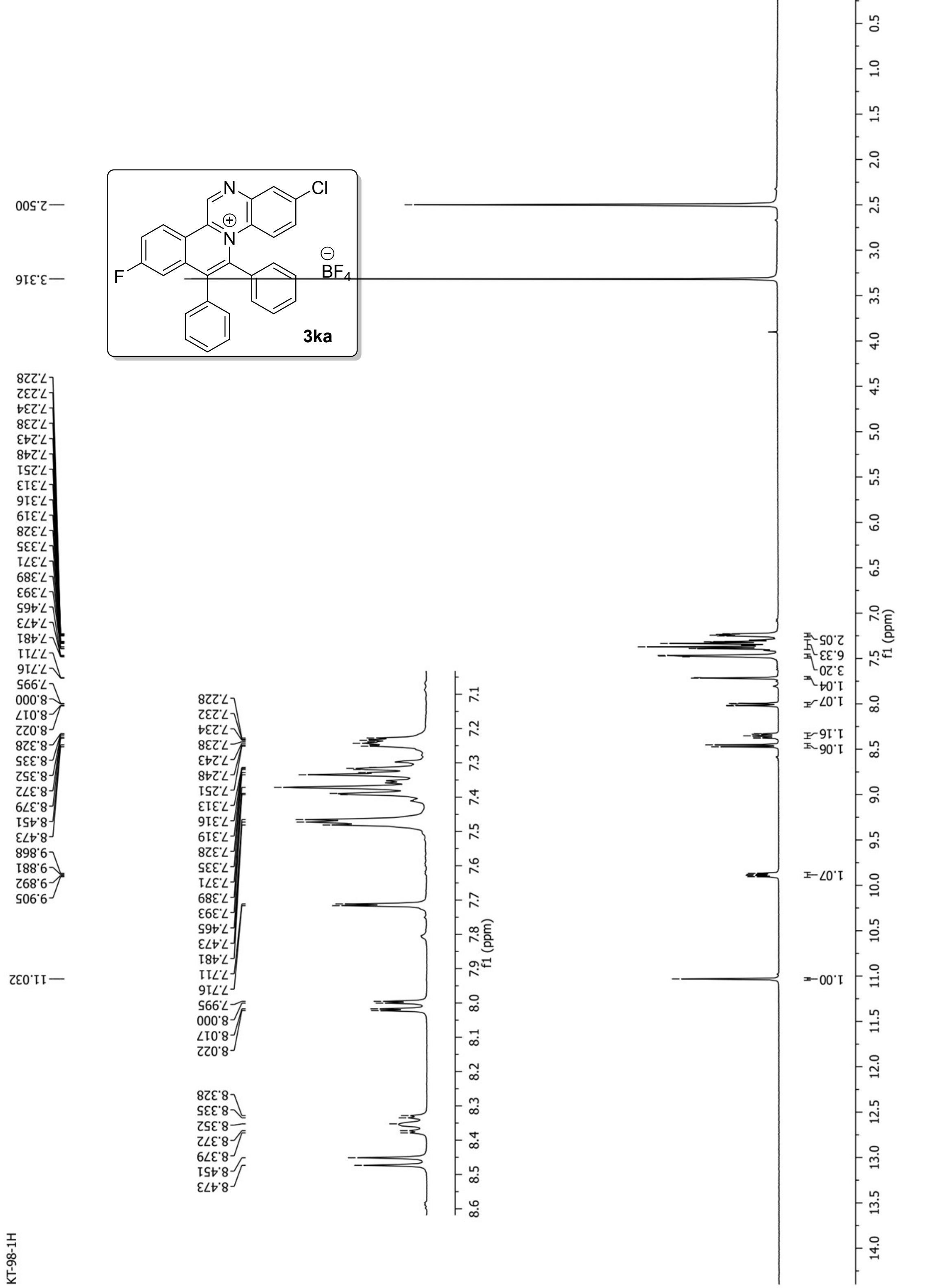

KT-104-1H
3ia
f1 (ppm)

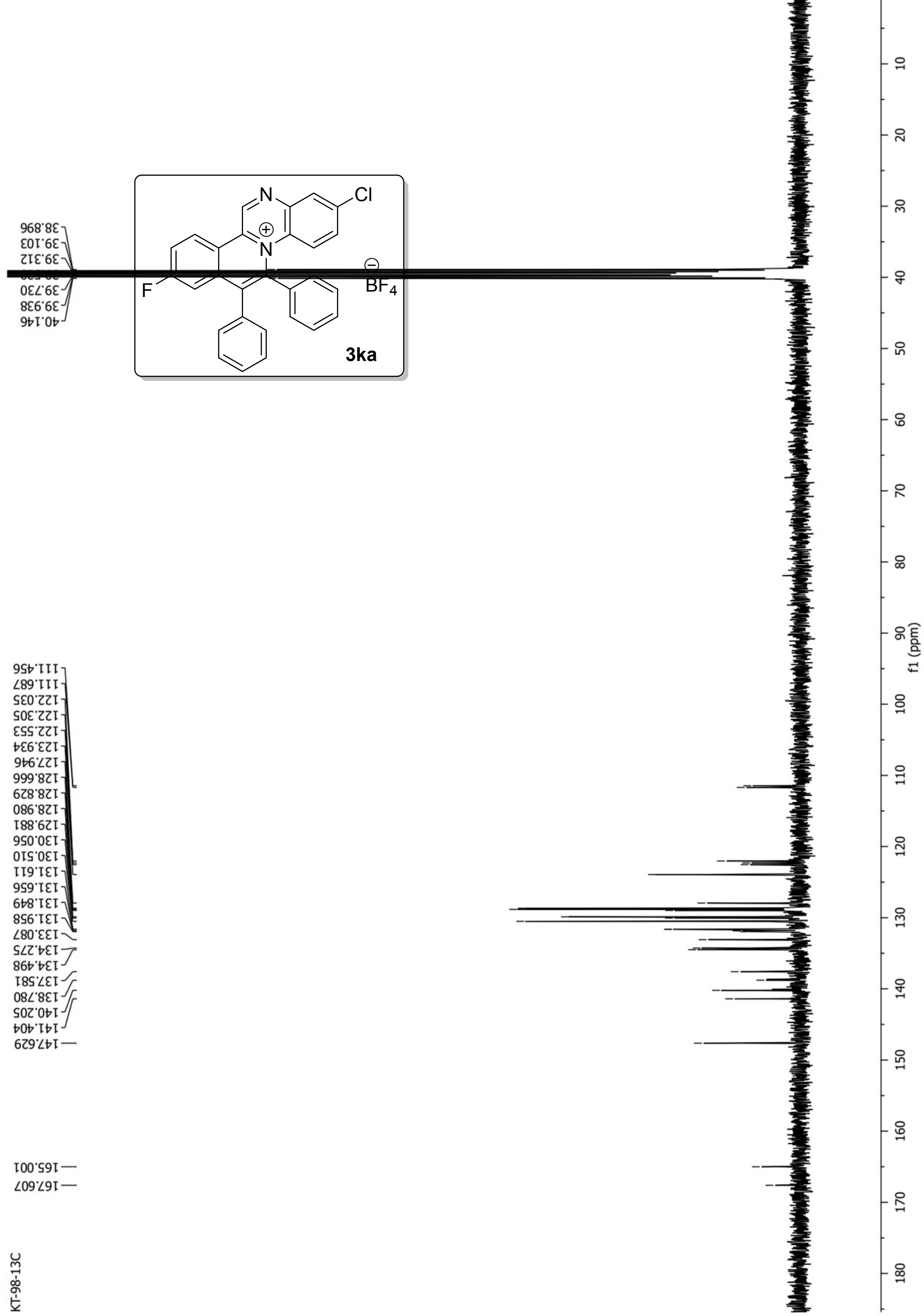

KT-104-13C
20.408
21.401
38.894
39.103
39.311
39.729
39.937
40.146
123.564
123.944
124.821
124.884
125.693
126.990
127.059
127.373
128.450
128.514
128.612
128.643
129.207
129.465
129.740
129.909
129.977
130.452
130.564
131.212
132.088
132.647
133.742
135.268
135.329
135.494
136.527
136.621
136.924
137.349
140.279
140.523
140.717
140.806
141.839
142.020
145.835
146.924
f1 (ppm)

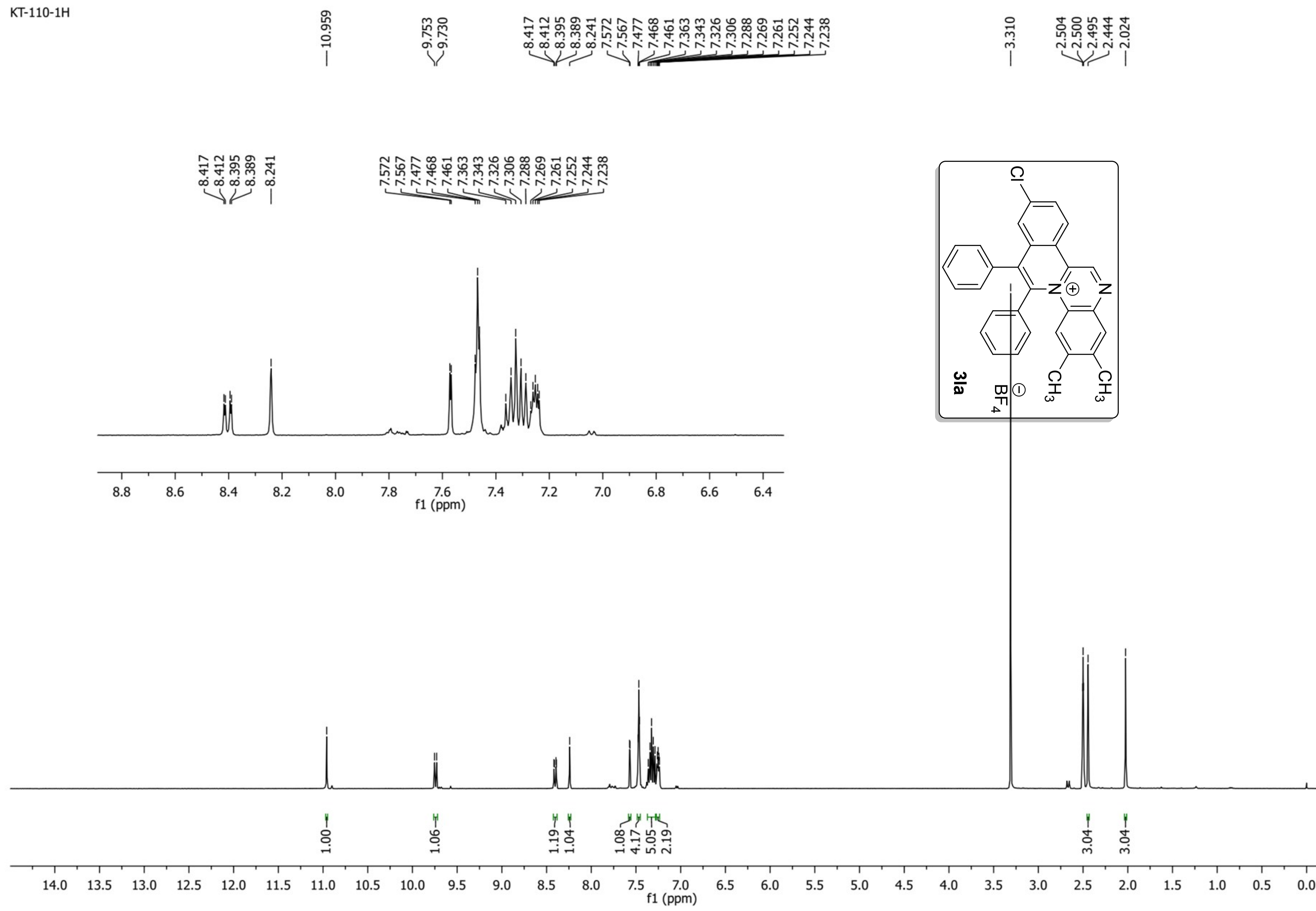


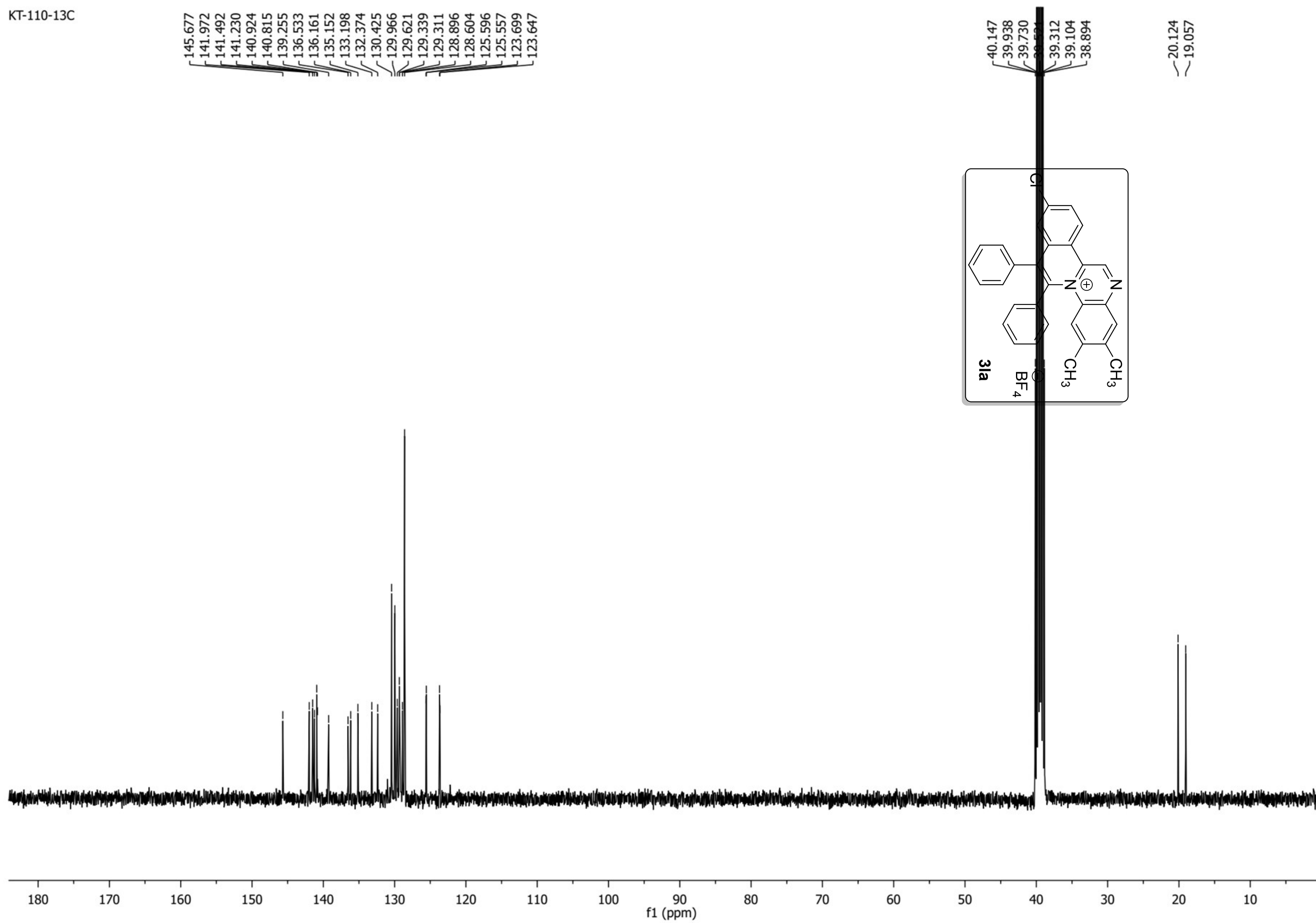
KT-95-1H
11.047
9.794
9.771
8.484
8.480
8.474
8.463
8.457
8.452
8.038
8.032
8.016
8.010
7.732
7.727
7.620
7.614
7.489
7.481
7.477
7.474
7.470
7.392
7.386
7.383
7.379
7.372
7.365
7.361
7.333
7.315
7.296
7.264
7.260
7.255
7.251
7.247
7.244
7.240
3.315
2.504
2.500
2.495
BF4
3ja
Cl
N
f1 (ppm)
1.00
1.04
2.02
1.06
1.00
1.00
3.03
2.98
2.07
2.04

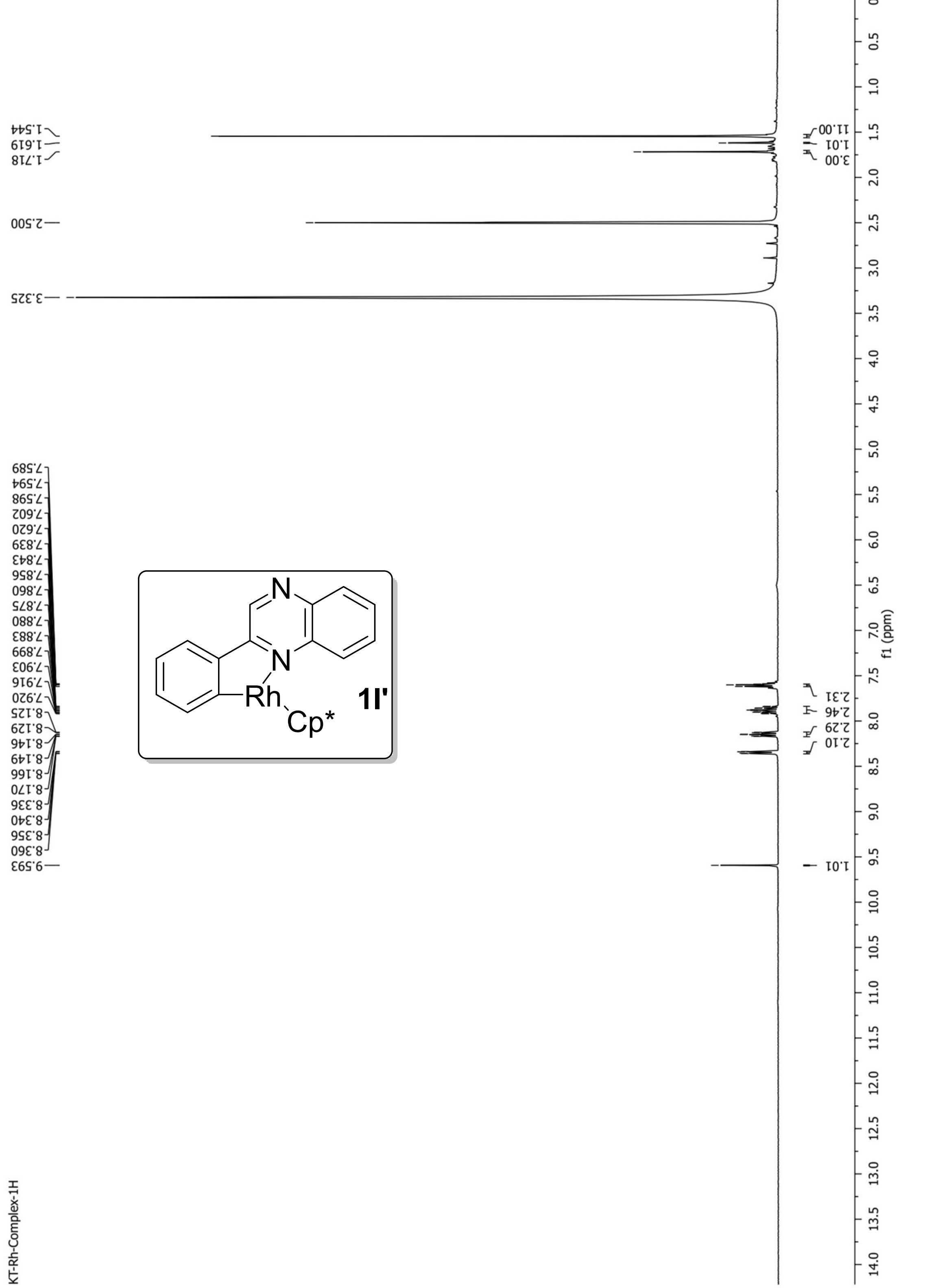

KT-95-13C
147.578
142.583
141.725
140.478
139.648
137.737
136.899
134.504
134.394
132.886
132.761
131.793
131.688
130.469
130.069
129.953
129.758
129.041
128.831
128.668
127.968
125.669
123.920
123.439
40.146
39.937
39.729
39.311
39.103
38.894
Cl
N
N
Cl
3ja
f1 (ppm)


KT-98-1H
3ka
BF4
F
Cl
N
2.500
3.316
7.228 7.232 7.234 7.238 7.243 7.248 7.251 7.313 7.316 7.319 7.328 7.335 7.371 7.389 7.393 7.465 7.473 7.481 7.711 7.716 7.995 8.000 8.017 8.022 8.328 8.335 8.352 8.372 8.379 8.451 8.473 9.868 9.881 9.892 9.905
11.032
2.05 6.33 3.20 1.04 1.07 1.16 1.06 1.07 1.00
f1 (ppm)

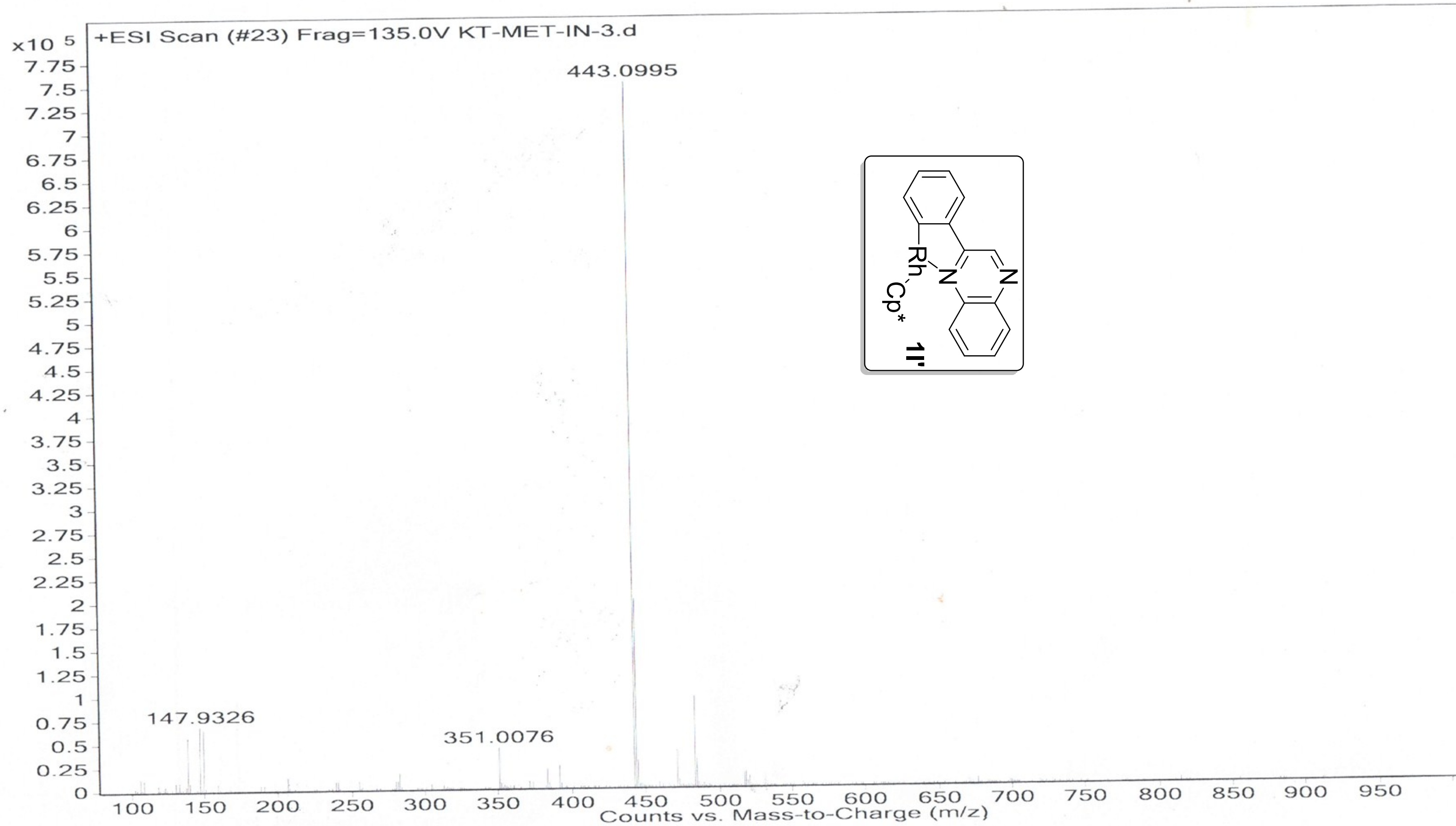

KT-98-13C
3ka
N
Cl
N
F
BF4
38.896
39.103
39.312
39.730
39.938
40.146
111.456
111.687
122.035
122.305
122.553
123.934
127.946
128.666
128.829
128.980
129.881
130.056
130.510
131.611
131.656
131.849
131.958
133.087
134.275
134.498
137.581
138.780
140.205
141.404
147.629
165.001
167.607
f1 (ppm)

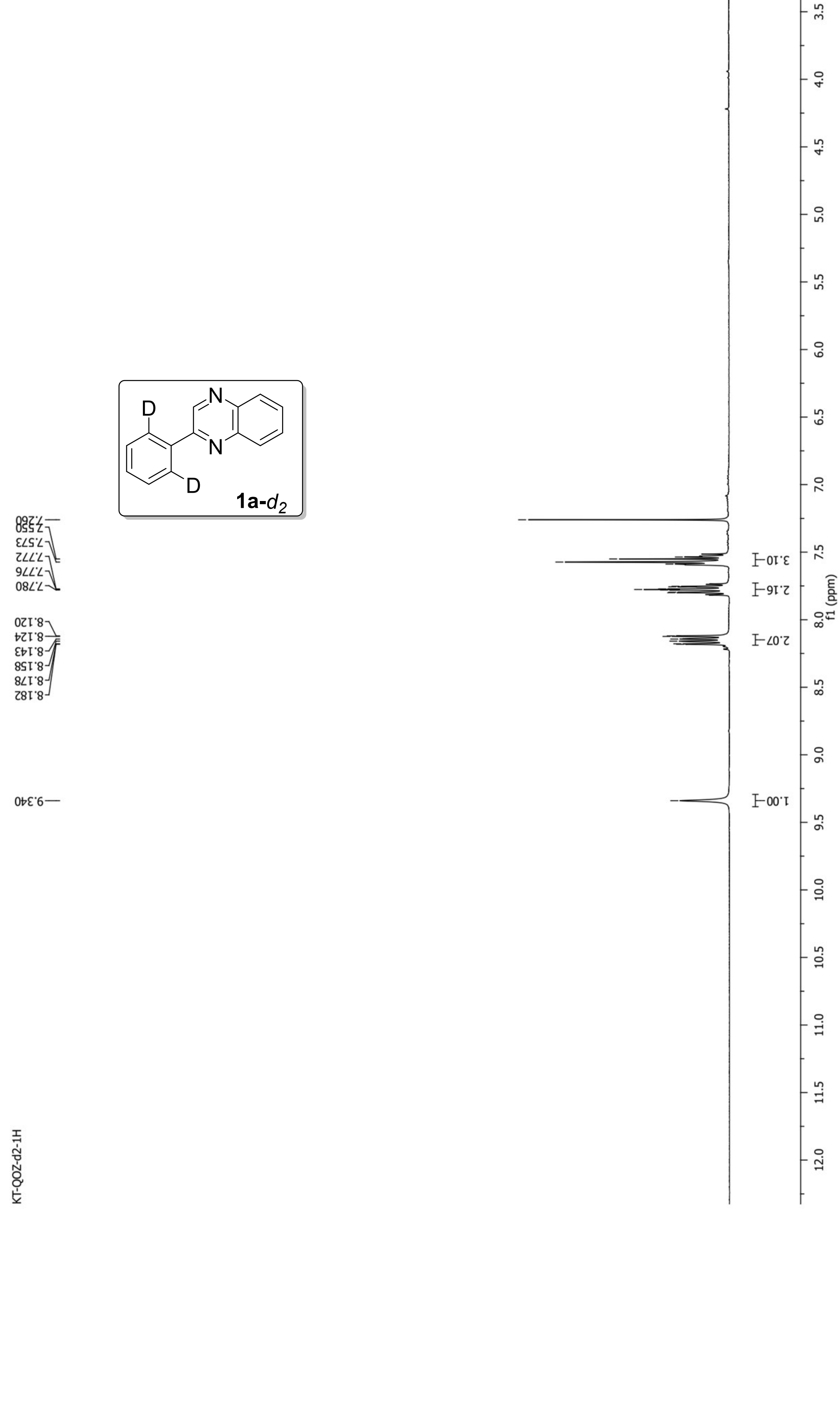

KT-110-1H
10.959
9.753
9.730
8.417
8.412
8.395
8.389
8.241
7.572
7.567
7.477
7.468
7.461
7.363
7.343
7.326
7.306
7.288
7.269
7.261
7.252
7.244
7.238
3.310
2.504
2.500
2.495
2.444
2.024
Cl
N
N
CH3
CH3
BF4
3la
1.00
1.06
1.19
1.04
1.08
4.17
5.05
2.19
3.04
3.04
f1 (ppm)


KT-110-13C
145.677
141.972
141.492
141.230
140.924
140.815
139.255
136.533
136.161
135.152
133.198
132.374
130.425
129.966
129.621
129.339
129.311
128.896
128.604
125.596
125.557
123.699
123.647
40.147
39.938
39.730
39.312
39.104
38.894
20.124
19.057
CH3
CH3
N
N
Cl
BF4
3la
f1 (ppm)


KT-Rh-Complex-1H
1.544
1.619
1.718
2.500
3.325
7.589
7.594
7.598
7.602
7.620
7.839
7.843
7.856
7.860
7.875
7.880
7.883
7.899
7.903
7.916
7.920
8.125
8.129
8.146
8.149
8.166
8.170
8.336
8.340
8.356
8.360
9.593
11.00
1.01
3.00
2.31
2.46
2.29
2.10
1.01
N
N
Rh
Cp*
1l'
f1 (ppm)

Sample Name	KT-MET-IN-3	**Position**	Vial 1	**Instrument Name**	QTOF	**User Name**	
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	KT-MET-IN-3.d	**ACQ Method**		**Comment**		**Acquired Time**	8/8/2018 12:52:3




KT-QOZ-d2-1H
7.260
7.550
7.573
7.772
7.776
7.780
8.120
8.124
8.143
8.158
8.178
8.182
9.340
3.10
2.16
2.07
1.00
f1 (ppm)
1a-d2