

Supporting Information
for
A Spiroborate-Based Anionic Bis-N-Heterocyclic Carbene

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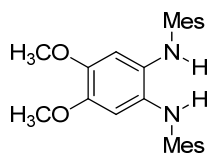
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General Information:

Air and moisture sensitive reactions were carried out under an atmosphere of N₂ using standard Schlenk techniques or inside a glovebox. Toluene and dichloromethane were dried via passing through the molecular sieves packed drying column under nitrogen. THF and THF-*d*₈ were dried using Na/K alloy, and distilled under inert atmosphere. ¹H, ¹¹B, and ¹³C NMR spectra were recorded using Bruker Avance III 400 MHz NMR, Bruker AVIII 500MHz FT-NMR and Bruker AVIII 800MHz NMR. The ¹H, and ¹³C NMR spectra reported in ppm (δ) are relative to the chemical shift of solvent residual signals for CDCl₃ at 7.26 and 77.00 ppm, [*d*₆]DMSO at 2.50 and 39.52 ppm, [*d*₈]THF at 3.58, 1.73 ppm, 25.4, 67.6 ppm, and [*d*₄]MeOH at 4.78, 3.31 ppm and 49.2 ppm. The elemental analysis was recorded on Thermo Flash 2000. The HR-ESI-MS was recorded on Bruker micrOTOF-QII. The infrared spectra were recorded using BOMEM MB-series.

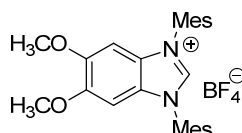
X-ray Crystallography: Diffraction data were collected at T = 150(2) K with a Bruker AXS APEX CCD diffractometer equipped with a rotation anode using graphite-monochromated Mo-Kα radiation (λ = 0.71073 Å). Diffraction data were collected over the full sphere and were corrected for absorption. Cell parameters were retrieved and refined using DENZO-SMN software on all reflections. Data reduction was performed with DENZO-SMN software. Structural analysis was conducted using the SHELXS program on a personal computer. All the structures were solved and refined using the SHELXL-97 program by full-matrix least-squares on F² values. Hydrogen atoms were placed geometrically and refined using a riding model. Crystallographic data have been deposited at the Cambridge Crystallographic Data Center with deposition number CCDC-950338, CCDC-950339, and CCDC-950340, for [1][Br], [2][Br], and K[4], respectively. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. The relatively high wR2 value for [2][Br] and K[4] was due to the presence of volatile solvent molecules in the crystal lattice that resulted in crystal cracking during the mounting process. Although the R values for both structures were not perfect, we were able to precisely determine the unit cell parameters by using a large amount of reflections (27631 reflections for [2][Br], and 25545 reflections for K[4]).

Synthesis of 4,5-dimethoxy-*N,N'*-dimesityl-1,2-benzenediamine



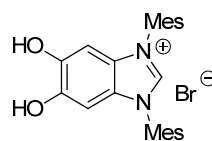
A catalyst for Buchwald-Hartwig coupling reaction was prepared by charging a 100 mL Schlenk flask with 1,3-bis(2,6-diisopropylphenyl)imidazol-2-ylidene (1.44 g, 3.4 mmol), Pd(OAc)₂ (0.39 g, 1.7 mmol), sodium *tert*-butoxide (0.49 g, 5.1 mmol) and toluene (30 mL), and then refluxed for 30 minutes. The resulting mixture was transferred into a 300 mL flask containing 4,5-dibromoveratrole (5.00 g, 17 mmol), sodium *tert*-butoxide (4.87 g, 51 mmol) in toluene (150 mL). Then 2,4,6-trimethylaniline (4.7 mL, 34 mmol) was added subsequently. The resulting mixture was heated at 110 °C for 16 h. After cooling to ambient temperature, the reaction mixture was quenched with saturated aqueous NH₄Cl (150 mL), and added 100 mL diethyl ether. The organic layer was then washed with 2 x 50 mL deionized water. The combined organic layer was dried over MgSO₄, filtered and concentrated under vacuum to yield brown oil. The crude product was recrystallized from methanol at -40 °C, and the product was collected as an off-white solid (6.24 g, yield 91 %) after filtration. ¹H NMR (CDCl₃, 400 MHz): δ 7.01 (s, 4H, Ar-H), δ 6.09 (s, 2H, Ar-H), δ 5.07 (s, 2H, NH), δ 3.68 (s, 6H, CH₃), δ 2.37 (s, 6H, CH₃), δ 2.27 (s, 12H, CH₃). ¹³C NMR (CDCl₃, 100 MHz): δ 143.1, 137.3, 133.4, 132.7, 129.4, 128.9, 101.9, 56.7, 20.7, 18.1 ppm.

Synthesis of 5,6-dimethoxy-1,3-dimesitylbenzimidazolium tetrafluoroborate



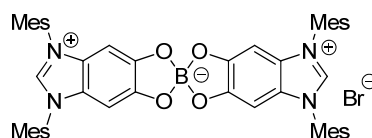
As-synthesized 4,5-dimethoxy-*N,N'*-dimesityl-1,2-benzenediamine (6.24 g, 15.4 mmol) was dissolved in the excess amount of triethyl orthoformate (40 mL). Then adding excess amount of HBF₄·etherate (5 mL) and stirred for overnight at room temperature. After reaction, the mixture was poured into diethyl ether (150 mL) to yield precipitates. The solids were collected by filtration and washed with diethyl ether, and was further purified by recrystallization from methanol and diethyl ether to give **3a** as an off-white solid (5.2 g, yield 67 %). ¹H NMR ([*d*₆]DMSO, 400 MHz): δ 9.90 (s, 1H, CH), δ 7.28 (s, 4H, Ar-H), δ 6.90 (s, 2H, Ar-H), δ 3.82 (s, 6H, CH₃), δ 2.41 (s, 6H, CH₃) δ 2.06 (s, 12H, CH₃). ¹³C NMR ([*d*₆]DMSO, 100 MHz): δ 151.2, 140.9, 140.8, 135.2, 129.7, 128.2, 124.9, 94.6, 56.6, 20.7, 16.8 ppm. HR-ESI-MS: *m/z* calc. for [C₂₇H₃₁N₂O₂]⁺: 415.2380; Found: 415.2387.

Synthesis of 5,6-dihydroxy-1,3-dimesitylbenzimidazolium bromide [1][Br]



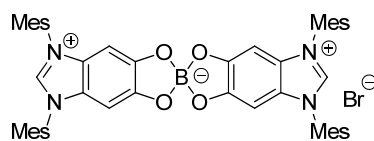
To a solution of 5,6-dimethoxy-1,3-dimesitylbenzimidazolium tetrafluoroborate (3.0 g, 6 mmol) in dried dichloromethane (30 mL), 1 M BBr₃ in dichloromethane (12 mL, 13 mmol) was added dropwise at -78 °C. The colour of the mixture changed from dark green to brown. After addition of BBr₃, the mixture was slowly warmed to room temperature, and stirred for additional 1 hour. After reaction, all volatile species were evaporated under vacuum. The solid residual was re-dissolved in methanol. After 30 minutes, deionized water was added till precipitates formed. The crude product was collected as an off-white solid after filtration, and was further purified by recrystallization from methanol and diethyl ether (2.7 g, yield 97 %). Single crystals were obtained from a methanol/ether solution of [1][Br] at room temperature. ¹H NMR ([d₆]DMSO, 400 MHz): δ 10.20 (s, 2H, OH), δ 9.89 (s, 1H, CH), δ 7.27 (s, 4H, Ar-H), δ 6.66 (s, 2H, Ar-H), δ 2.40 (s, 6H, CH₃) δ 2.06 (s, 12H, CH₃). ¹³C NMR ([d₆]DMSO, 100 MHz): δ 149.0, 141.4, 140.6, 135.5, 130.2, 128.8, 124.8, 97.5, 21.2, 17.2 ppm. Anal. Calc. for C₂₅H₂₇BrN₂O₂·H₂O: C, 61.86; H, 6.02; N, 5.77; Found: C, 62.85; H, 5.79; N, 5.72.

Synthesis of [2][Br] from benzenediboronic acid



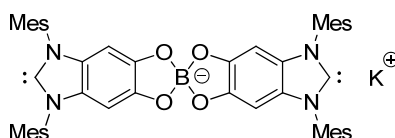
Benzenediboronic acid (90 mg, 0.54 mmol), [1][Br] (500 mg, 1.07 mmol) and 20 mL ethanol were added into a 50 mL round-bottom flask that was equipped with a Dean-Stark apparatus. The mixture was refluxed overnight. All volatiles were removed under vacuum. The crude product was purified by recrystallization from methanol and diethyl ether, and then washed with THF for several times to yield [2][Br] as off-white solids (401 mg, yield 87 %). Single crystals suitable for X-ray diffraction analysis were obtained from an ethyl acetate/ether/acetone solution of [2][Br] at room temperature. ¹H NMR ([d₆]DMSO, 400 MHz): δ 9.74 (s, 2H, CH), δ 7.25 (s, 8H, Ar-H), δ 6.48 (s, 4H, Ar-H), δ 2.39 (s, 12H, CH₃), δ 2.05 (s, 24H, CH₃). ¹³C NMR ([d₆]DMSO, 100 MHz): δ 153.3, 140.7, 138.1, 135.1, 129.6, 128.6, 124.5, 90.8, 20.7, 16.7. ¹¹B NMR ([d₆]DMSO, 128 MHz): δ 15 ppm. Anal. Calc. for C₅₀H₅₀BBrN₄O₄: C, 69.69; H, 5.85; N, 6.50; Found: C, 68.93; H, 5.83; N, 6.39.

Synthesis of [2][Br] from phenylboronic acid



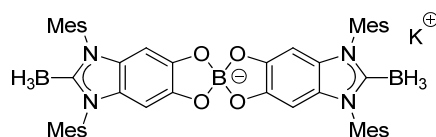
Phenylboronic acid (27 mg, 0.22 mmol), [1][Br] (200 mg, 0.43 mmol) and 10 mL ethanol were added into a 25 mL round-bottom flask that was equipped with a Dean-Stark apparatus. The mixture was refluxed overnight. All volatiles were removed under vacuum. The crude product was purified by recrystallization from methanol and diethyl ether, and then washed with THF for several times to yield [2][Br] as off-white solids (159 mg, yield 86 %).

Synthesis of anionic bis-NHC K[3]



To a 25 mL Schlenk flask containing [2][Br] (100 mg, 0.12 mmol) in 10 mL dried THF was added 0.26 mL of 1 M THF solution of KHMDS (0.26 mL, 0.24 mmol). The mixture was allowed to stir at room temperature for 16 hours. The resulting solution was filtered through 0.22 μ m syringe filter. The collected filtrate was dried under vacuum to give product as a white solid (86 mg, yield 90 %). ¹H NMR ([*d*₈]THF, 500 MHz): δ 7.00 (s, 8H, Ar-H), δ 6.04 (s, 4H, Ar-H), δ 2.34 (s, 12H, CH₃), δ 2.02 (s, 24H, CH₃). ¹³C NMR ([*d*₈]THF, 125 MHz): δ 224.5 (carbene), 149.9, 137.6, 137.5, 136.6, 129.3, 128.3, 90.9, 21.0, 17.9 ppm. ¹¹B NMR ([*d*₈]THF, 128 MHz): δ 15 ppm.

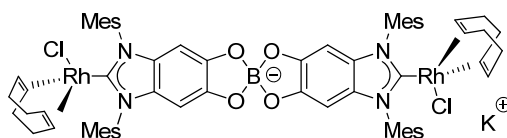
Synthesis of di-borane adduct K[4]



To a 50 mL Schlenk flask charging with 100 mg of [2][Br] (0.12 mmol) and 10 mL of dried THF was added 28 mg of KO^tBu (0.24 mmol). After stirring at room temperature for 30 minutes, BH₃(SMe₂) (2 M solution in THF, 0.21 mL, 0.48 mmol) was added slowly at ambient temperature. The resulting mixture was allowed to stir for overnight. The precipitates slowly formed during reaction, and was collected after reaction by filtration to yield K[4] as a white solid (56 mg, yield 57 %). Single crystal was obtained from a methanol/ether solution of K[4] at room temperature. ¹H NMR ([*d*₄]MeOH, 500 MHz): δ 7.09 (s, 8H, Ar-H), δ 6.17 (s, 4H, Ar-H), δ 2.38 (s, 12H, CH₃), δ 1.97 (s, 24H, CH₃), δ 0.60 (s, 6H, BH₃). ¹³C NMR ([*d*₄]MeOH, 125 MHz): δ

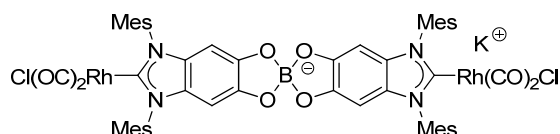
176.7 (q, broad, H₃B-C), 151.5, 140.4, 136.8, 133.5, 130.2, 127.1, 92.3, 21.2, 17.6 ppm. ¹¹B NMR ([d₄]MeOH, 128 MHz): δ 14, -36 ppm. HR-ESI-MS: m/z calc. for C₅₀H₅₄B₃N₄O₄ [[4]⁻]: 807.4452; Found: 807.4443.

Synthesis of di-Rh(COD)Cl complex K[5]



Cyclooctadiene rhodium chloride dimer (60 mg, 0.13 mmol) was dissolved in 5 mL of toluene, and was slowly added in to the *in situ* generated anionic bis-NHC from [2][Br] (100 mg, 0.12 mmol) and KO^tBu (28 mg, 0.24 mmol) in THF (10 mL). The resulting mixture was refluxed overnight to result in precipitations. The product was collected by filtration, and washed with small portion of THF and diethyl ether (126 mg, yield 79 %). ¹H NMR ([d₆]DMSO, 800 MHz): δ 7.14 (s, 4H, Ar-H), δ 7.12 (s, 4H, Ar-H), δ 5.94 (s, 4H, Ar-H), δ 4.29 (m, 4H, COD-CH), δ 3.37 (m, 4H, COD-CH), δ 2.38 (s, 12H, CH₃), δ 2.19 (s, 12H, CH₃), δ 1.93 (s, 12H, CH₃), δ 1.74 (m, 8H, COD-CH₂), δ 1.51 (m, 8H, COD-CH₂). ¹³C NMR ([d₆]DMSO, 125 MHz): δ 186.0 (¹J_{Rh-C}=52 Hz), 149.4, 138.0, 137.4, 135.0, 133.5, 129.3, 128.3, 127.4, 94.7, 89.7, 66.9, 32.3, 27.9, 20.8, 19.1, 17.5. ¹¹B NMR ([d₆]DMSO, 128 MHz): δ 14 ppm. HR-ESI-MS: m/z calc. for C₆₆H₇₂BCl₂N₄O₄Rh₂ [[5]⁻]: 1271.3150; Found: 1271.3134.

Synthesis of di-Rh(CO)₂Cl complex K[6]



The CO gas was bubbled through a 10 mL of THF solution of K[5] (27 mg, 0.02 mmol) at ambient temperature for 30 minutes. After reaction, diethyl ether was added into the solution to result in precipitation of complex K[6] as yellowish solid, which was collected by filtration (19 mg, yield 76 %). ¹H NMR ([d₆]DMSO, 400 MHz): δ 7.12, 7.114, 6.00, 5.97, 5.96, 2.37, 2.05, 2.04 ppm. ¹³C NMR ([d₆]DMSO, 200 MHz): δ 184.2, 138.8, 178.1, 177.8, 149.9, 149.5, 138.3, 135.8, 135.0, 133.2, 129.6, 129.0, 128.9, 128.6, 128.2, 127.1, 125.3, 124.2, 90.0, 21.0, 20.8, 17.9, 16.7, 15.4 ppm. HR-ESI-MS: m/z calc. for C₅₄H₄₈BCl₂N₄O₈Rh₂ [[6]⁻]: 1167.1067; Found: 1167.1076. calc. for C₅₃H₄₈BCl₂N₄O₇Rh₂ [[6]⁻-CO]: 1139.1117; Found: 1139.1142. FT-IR: 2073, 1992 cm⁻¹($\bar{\nu}_{\text{CO}}$)

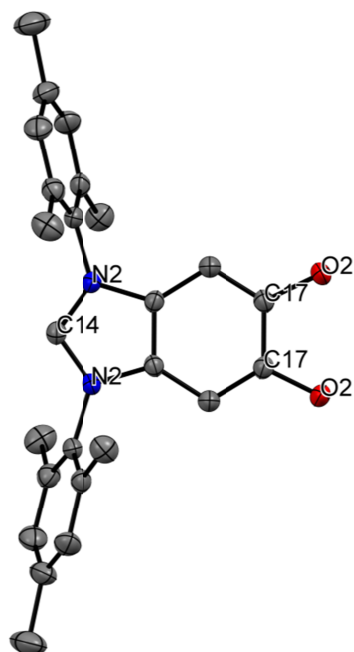


Figure S1 Molecular structure of $[1]^+$. Hydrogen atoms and counter anion are omitted for clarity. Selected bond distance (\AA) and bond angle (deg): C14-N2 1.338(3), C17-O2 1.351(3), N2-C14-N2 108.9(3).

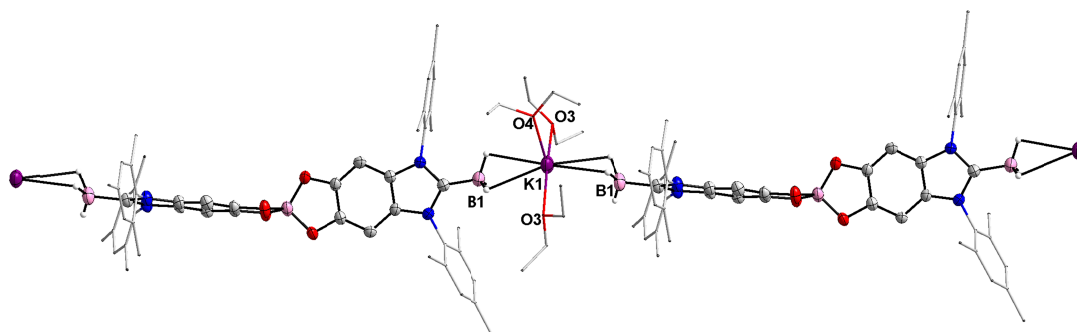


Figure S2 Polymeric structure of $K[4]$ in solid state formed via potassium-hydrogen interactions. Each potassium atom is coordinated by three disordered diethyl ether molecules.

Study on the condensation process

The experiments are designed to identify how many boron atoms were provided by a BDBA (benzeneboronic acid) molecule. As shown in the table below, the experiments were carried out with different molar ratio of [1][Br] and BDBA. After reaction, all volatile species were removed under vacuum. The crude reaction mixtures were subjected to ^1H NMR measurement without purification. The isolation yield was obtained after recrystallization.

Molar ratio [1][Br] : BDBA	Isolation yield (based on [1][Br])
1:1	94%
2:1	80%
4:1	87%

Table S1 Isolation yield of [2][Br] with different molar ratio of reactants.

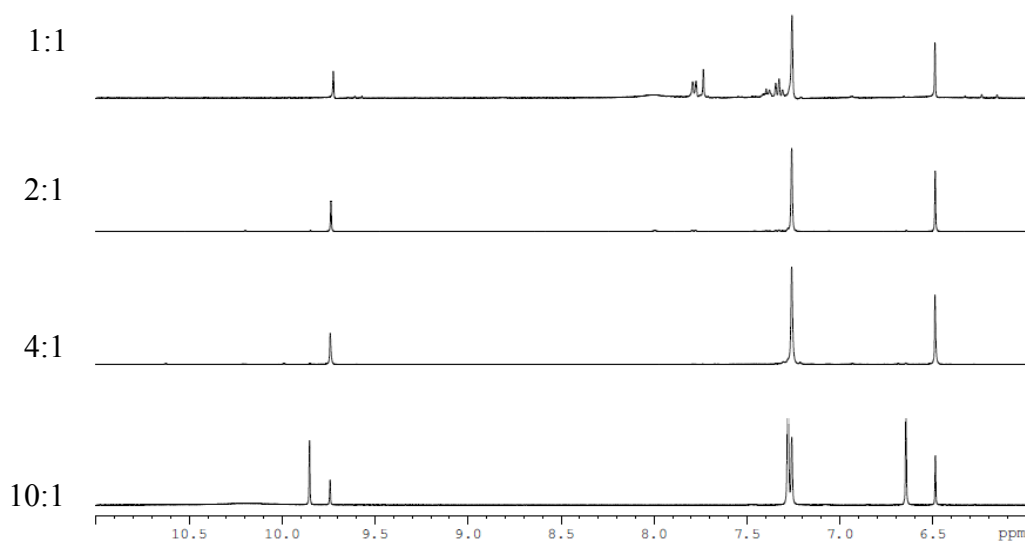


Figure S3 Crude ^1H NMR of the reaction mixtures.

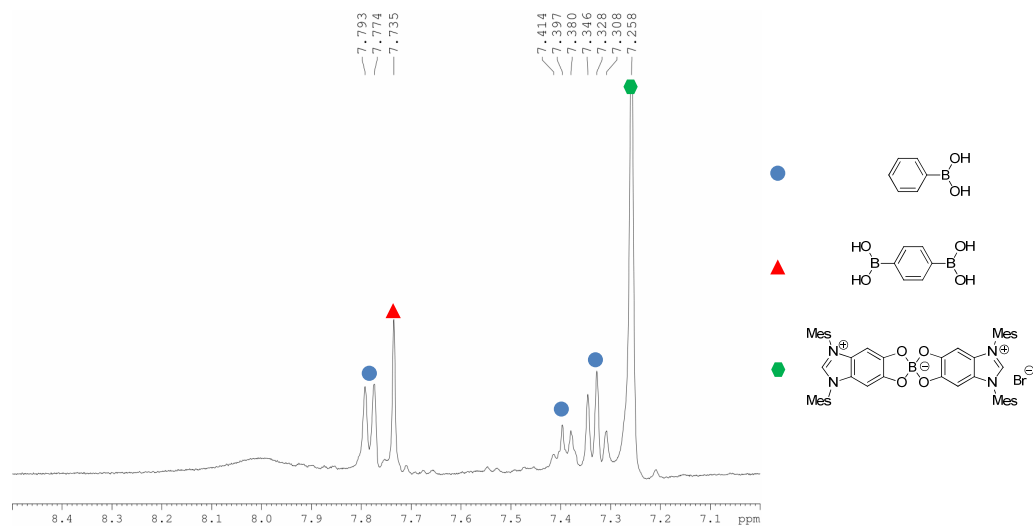


Figure S4 Crude ¹H NMR of the reaction mixture having 1:1 molar ratio of [1][Br] and BDBA.

Table S2 Crystal data and structure refinement for [1][Br].

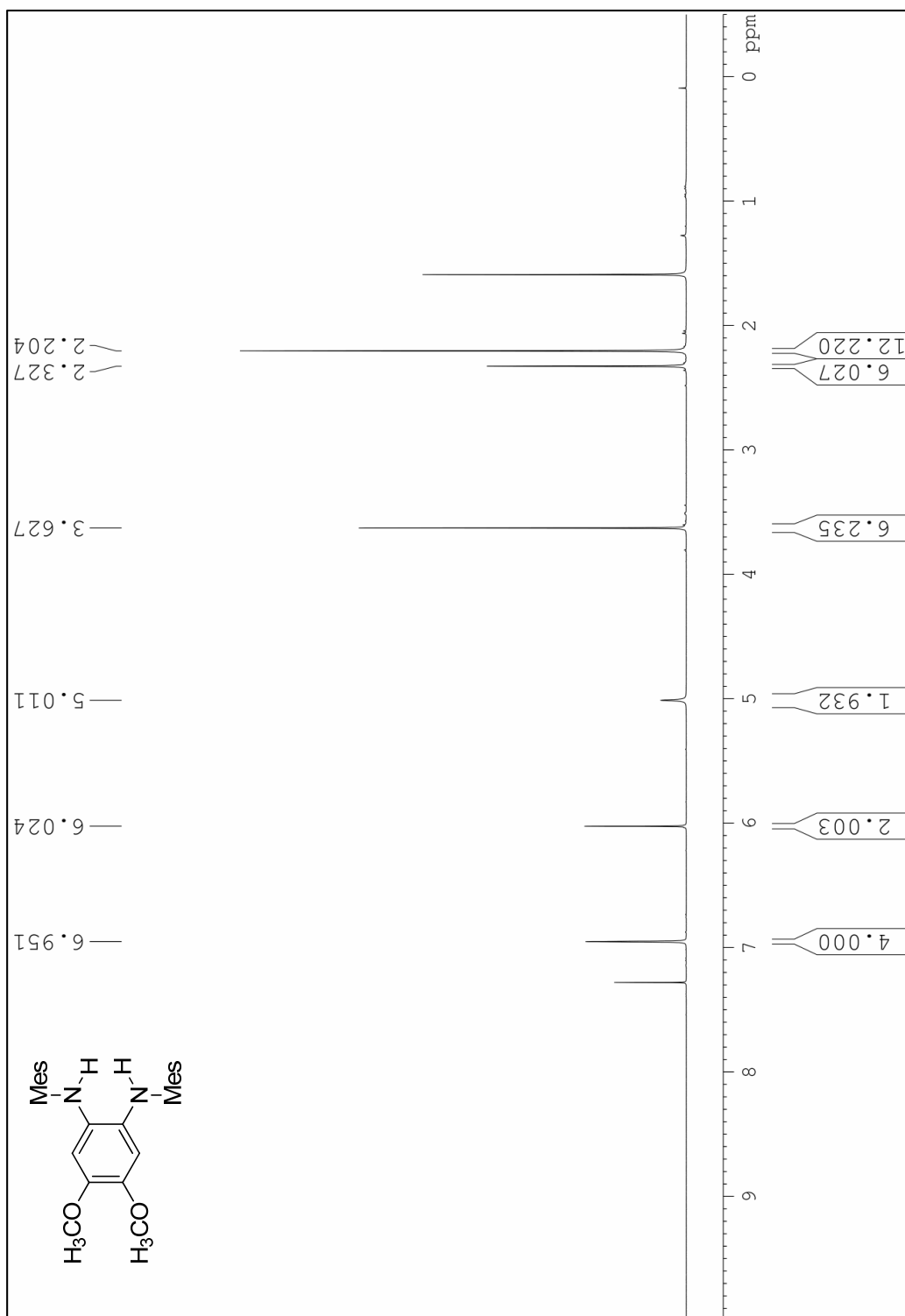
Identification code	ic15773
Empirical formula	C ₂₅ H ₂₉ Br N ₂ O ₃
Formula weight	485.41
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	Pccn
Unit cell dimensions	a = 18.7580(18) Å α = 90°. b = 15.0409(14) Å β = 90°. c = 17.1991(16) Å γ = 90°.
Volume	4852.5(8) Å ³
Z	8
Density (calculated)	1.329 g/cm ³
Absorption coefficient	1.722 mm ⁻¹
F(000)	2016
Crystal size	0.47 x 0.45 x 0.08 mm ³
Theta range for data collection	1.74 to 27.50°.
Index ranges	-24 ≤ h ≤ 23, -19 ≤ k ≤ 19, -22 ≤ l ≤ 22
Reflections collected	35835
Independent reflections	5580 [R(int) = 0.0721]
Completeness to theta = 27.50°	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.8746 and 0.4984
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	5580 / 0 / 287
Goodness-of-fit on F ²	1.028
Final R indices [I > 2σ(I)]	R1 = 0.0450, wR2 = 0.1093
R indices (all data)	R1 = 0.0698, wR2 = 0.1237
Largest diff. peak and hole	0.535 and -0.495 e.Å ⁻³

Table S3 Crystal data and structure refinement for [2][Br].

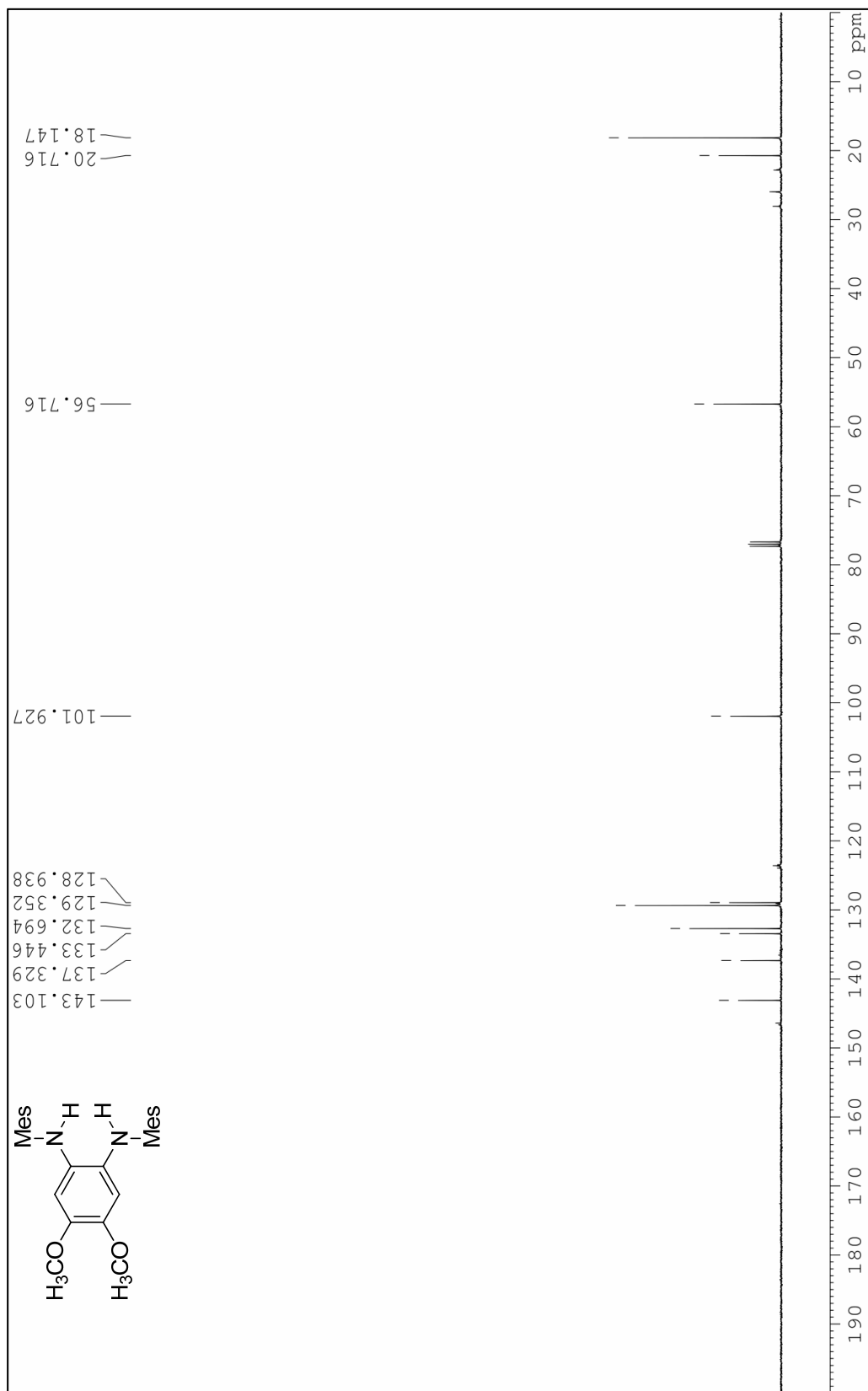
Identification code	ic15764
Empirical formula	C ₅₄ H _{58.78} B Br N ₄ O _{5.61}
Formula weight	944.35
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P2(1)/n
Unit cell dimensions	a = 8.8956(2) Å α = 90°. b = 17.9979(4) Å β = 90.3726(9)°. c = 30.9329(7) Å γ = 90°.
Volume	4952.32(19) Å ³
Z	4
Density (calculated)	1.267 g/cm ³
Absorption coefficient	0.882 mm ⁻¹
F(000)	1983
Crystal size	0.45 x 0.20 x 0.16 mm ³
Theta range for data collection	1.31 to 25.00°.
Index ranges	-10 ≤ h ≤ 9, -21 ≤ k ≤ 21, -36 ≤ l ≤ 36
Reflections collected	27631
Independent reflections	8707 [R(int) = 0.0518]
Completeness to theta = 25.00°	99.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.873 and 0.739
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	8707 / 7 / 586
Goodness-of-fit on F ²	1.063
Final R indices [I > 2σ(I)]	R1 = 0.0645, wR2 = 0.1788
R indices (all data)	R1 = 0.0944, wR2 = 0.1956
Largest diff. peak and hole	0.800 and -1.079 e.Å ⁻³

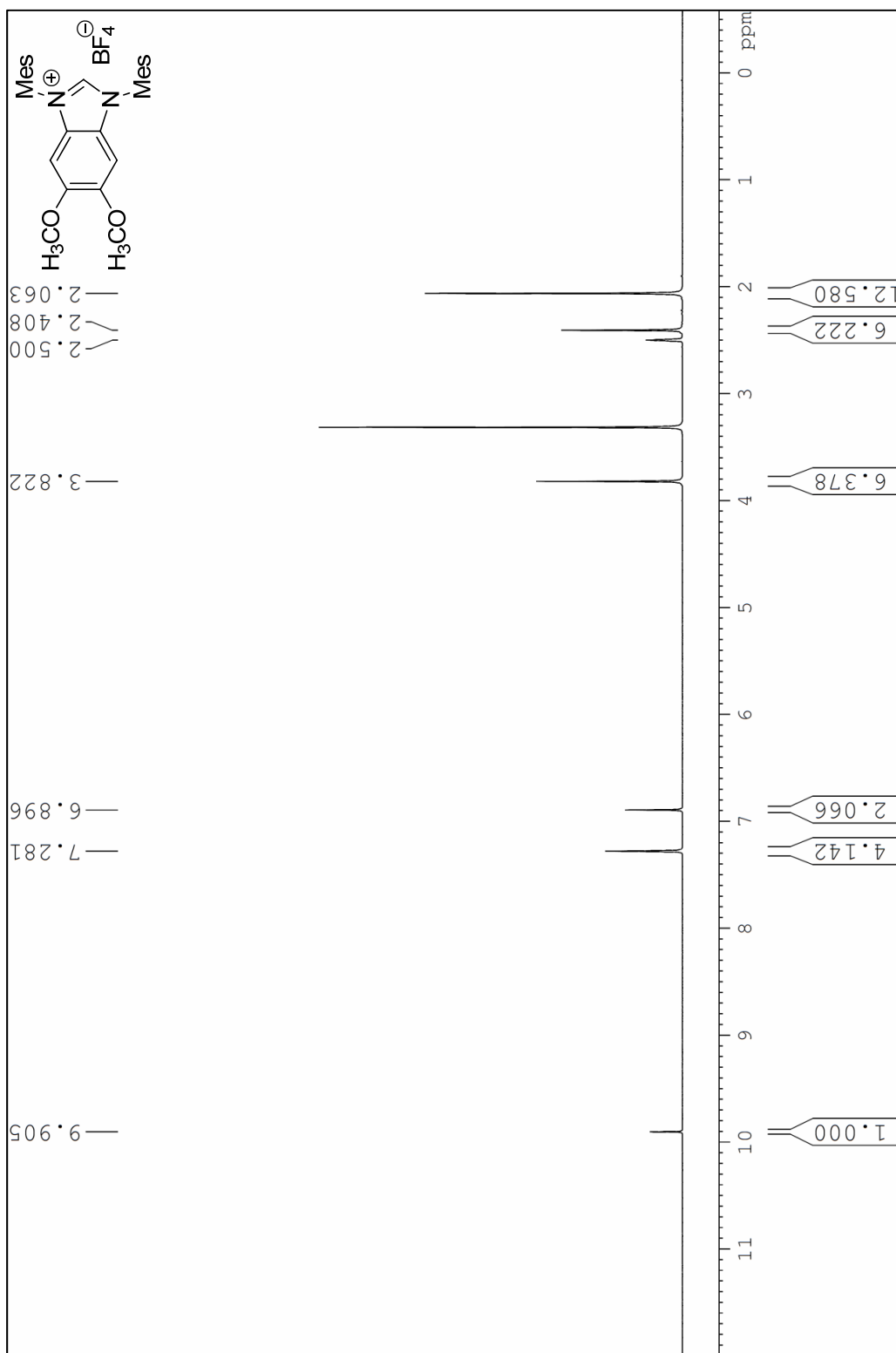
Table S4 Crystal data and structure refinement for K[4].

Identification code	ic16249
Empirical formula	C64.64 H80.55 B3 K N4 O8.64
Formula weight	1111.25
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	C2/c
Unit cell dimensions	a = 22.2188(4) Å $\alpha = 90^\circ$. b = 19.7828(4) Å $\beta = 117.3330(9)^\circ$. c = 16.8614(3) Å $\gamma = 90^\circ$.
Volume	6584.0(2) Å ³
Z	4
Density (calculated)	1.121 Mg/m ³
Absorption coefficient	0.134 mm ⁻¹
F(000)	2374
Crystal size	0.40 x 0.22 x 0.20 mm ³
Theta range for data collection	1.46 to 27.50°.
Index ranges	-28 ≤ h ≤ 28, -25 ≤ k ≤ 25, -21 ≤ l ≤ 21
Reflections collected	25445
Independent reflections	7542 [R(int) = 0.0423]
Completeness to theta = 27.50°	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.976 and 0.884
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	7542 / 40 / 397
Goodness-of-fit on F ²	1.045
Final R indices [I > 2σ(I)]	R1 = 0.0811, wR2 = 0.2483
R indices (all data)	R1 = 0.1245, wR2 = 0.2823
Largest diff. peak and hole	0.987 and -0.500 e.Å ⁻³

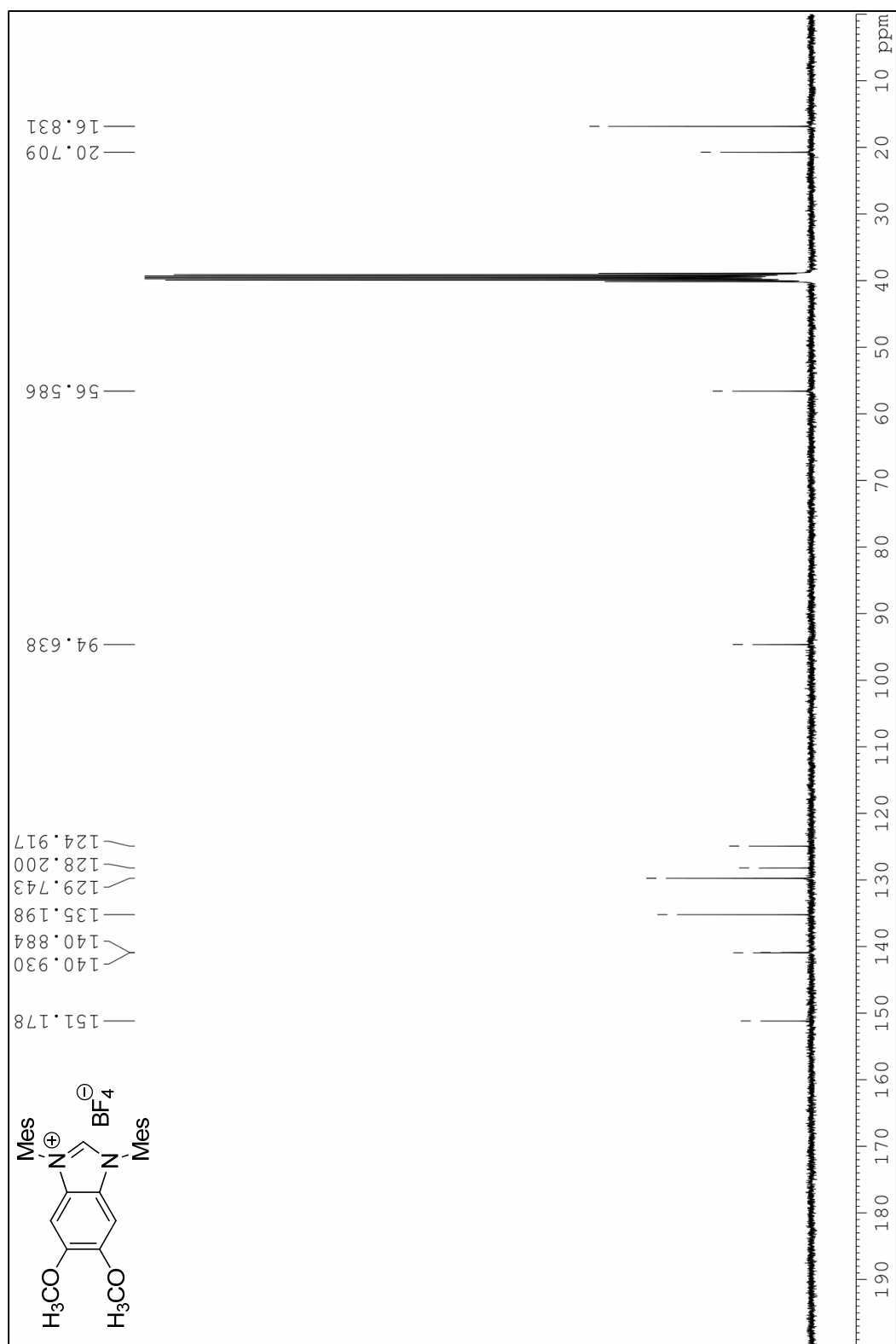


¹H NMR of 4,5-dimethoxy-N,N'-dimesityl-1,2-benzenediamine in CDCl₃

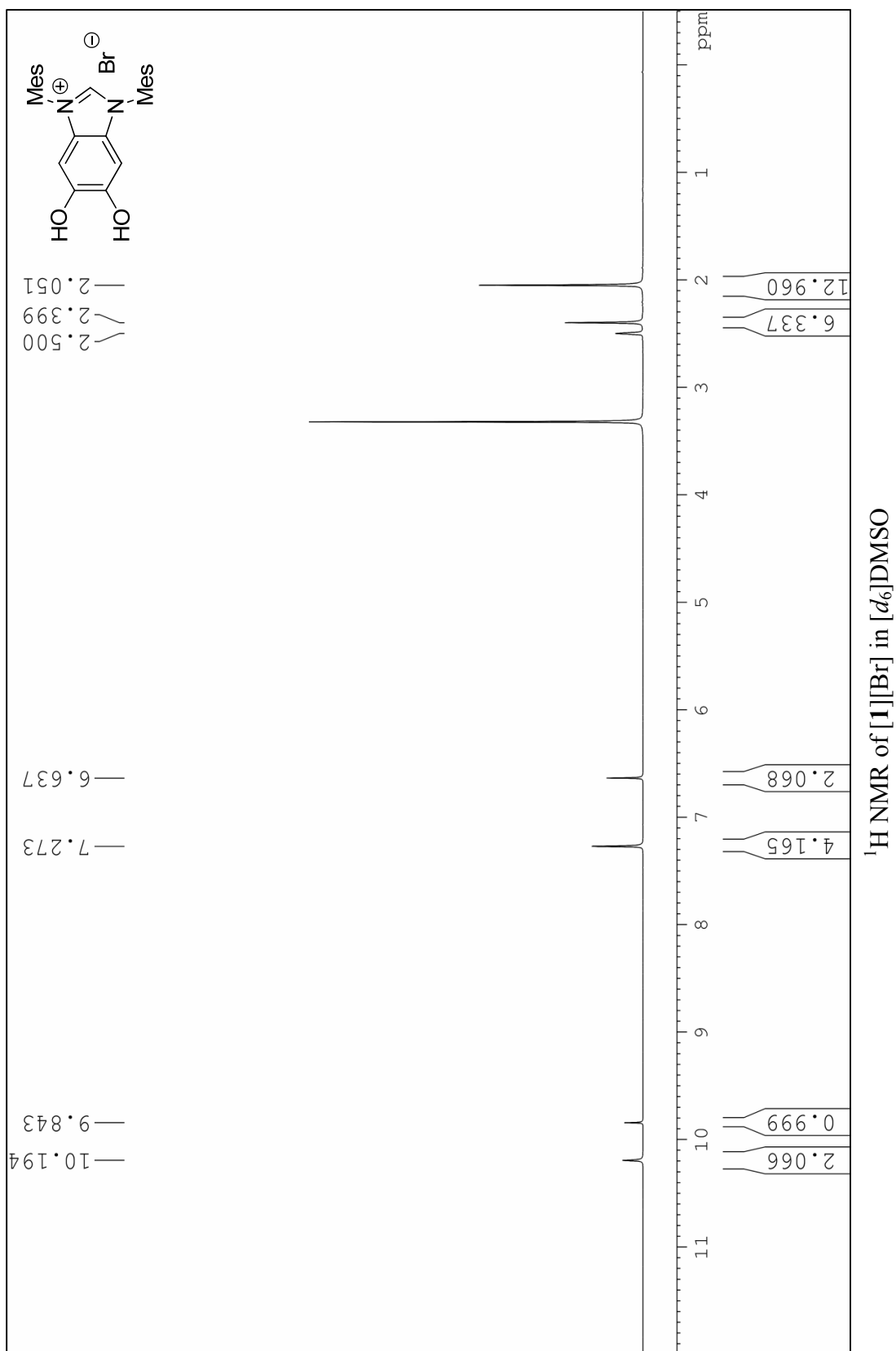


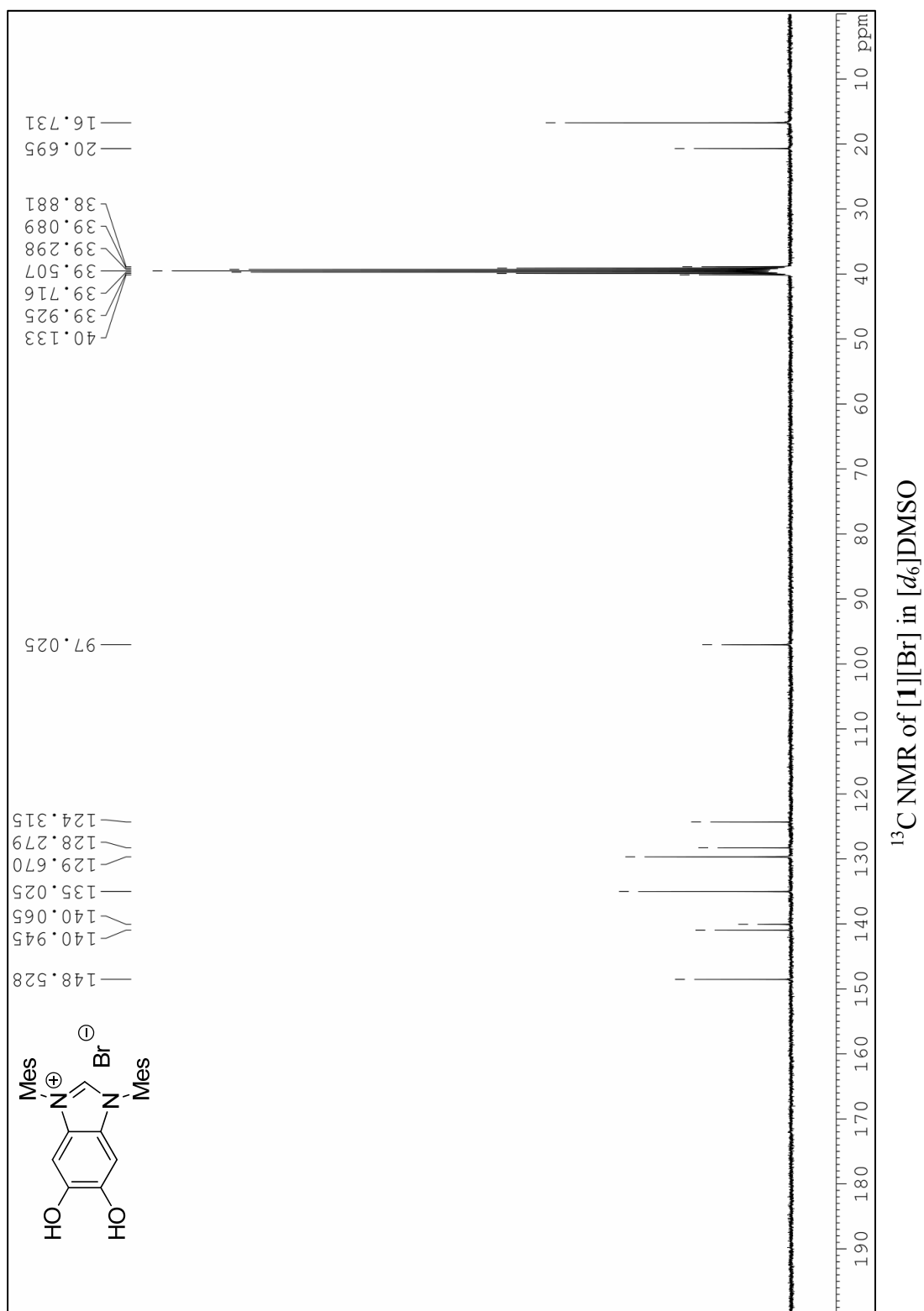


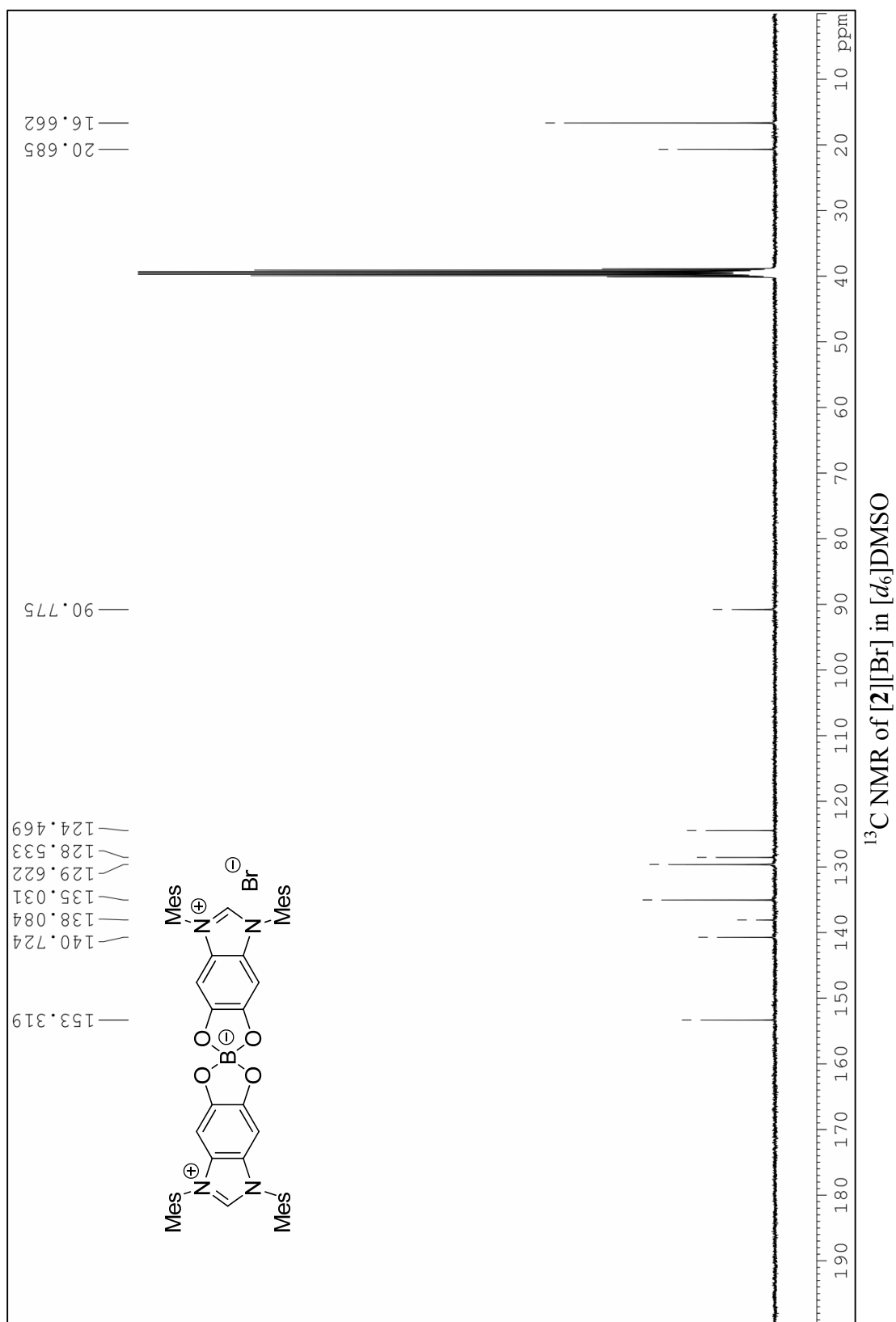
¹H NMR of 5,6-dimethoxy-1,3-dimesitylbenzimidazolium tetrafluoroborate in [d₆]DMSO

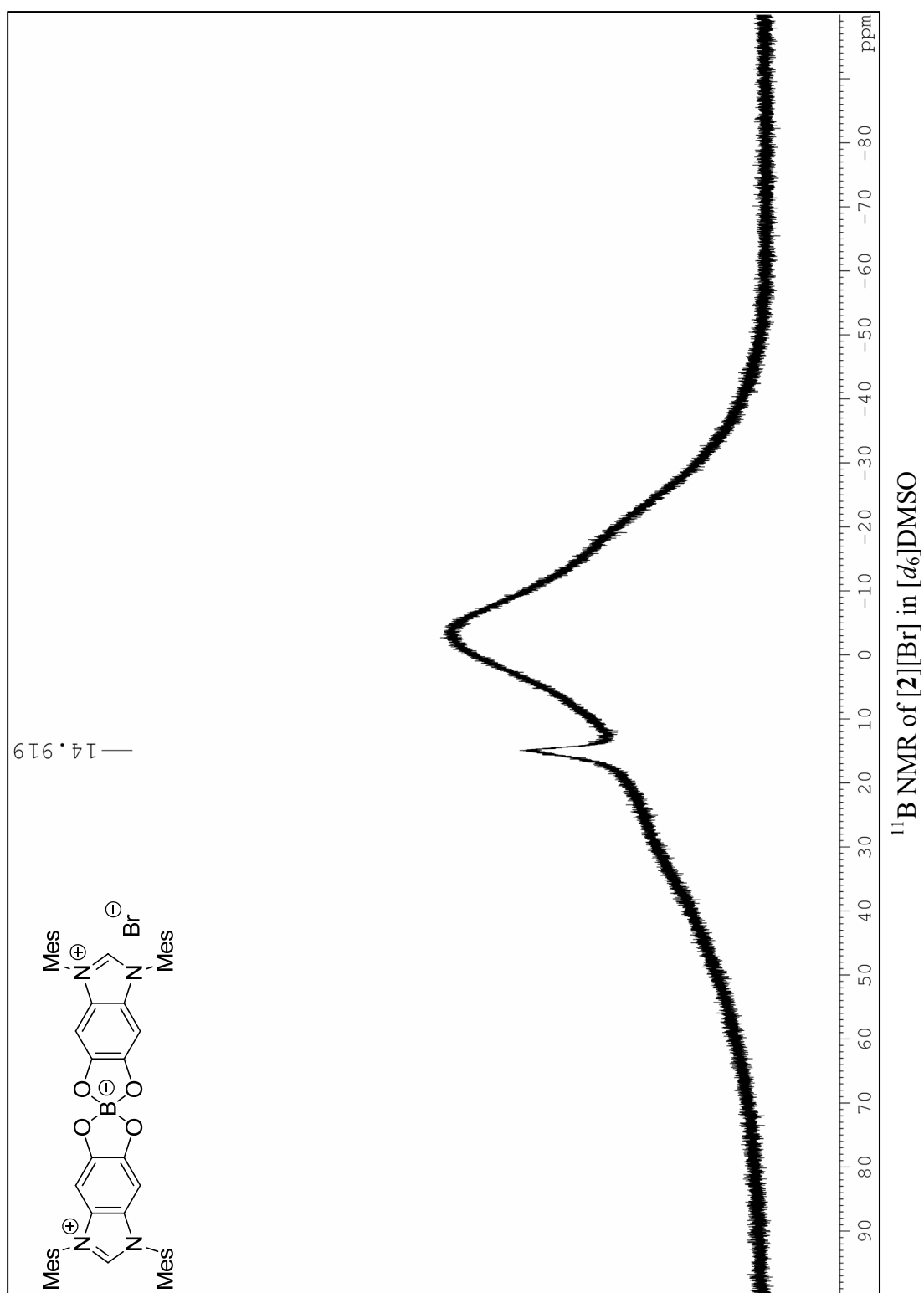


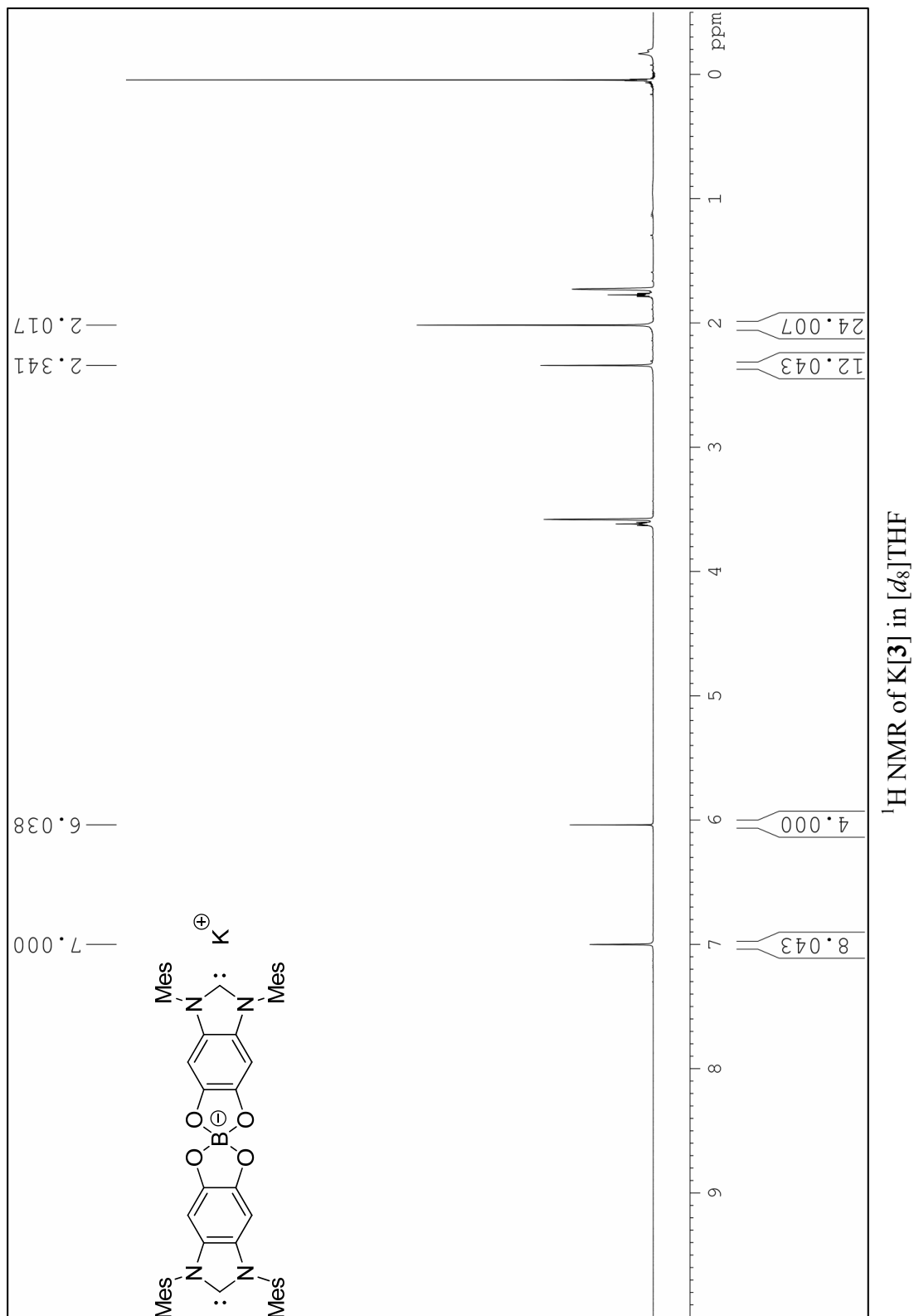
^{13}C NMR of 5,6-dimethoxy-1,3-dimesitylbenzimidazolium tetrafluoroborate in $[\text{d}_6]\text{DMSO}$

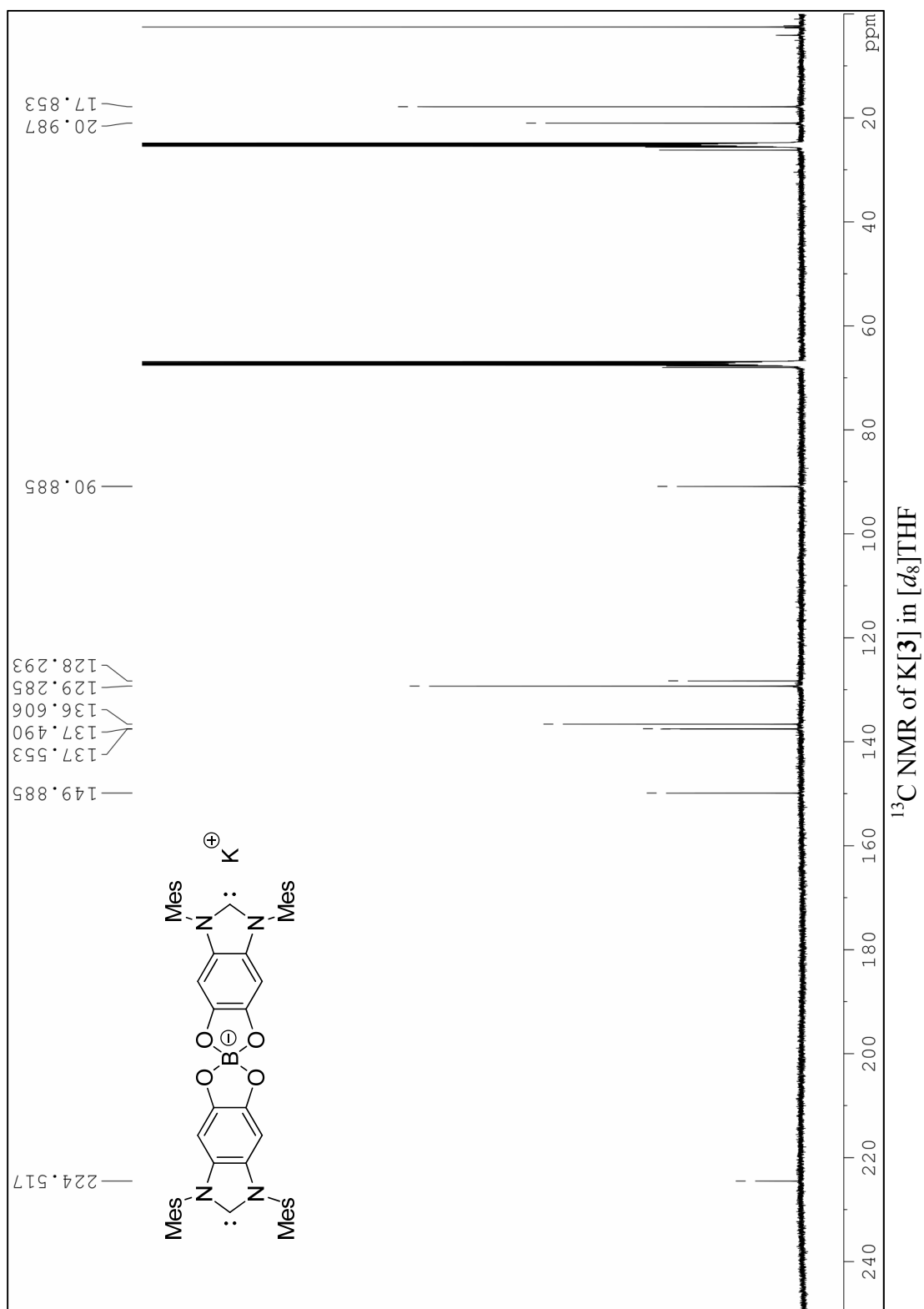


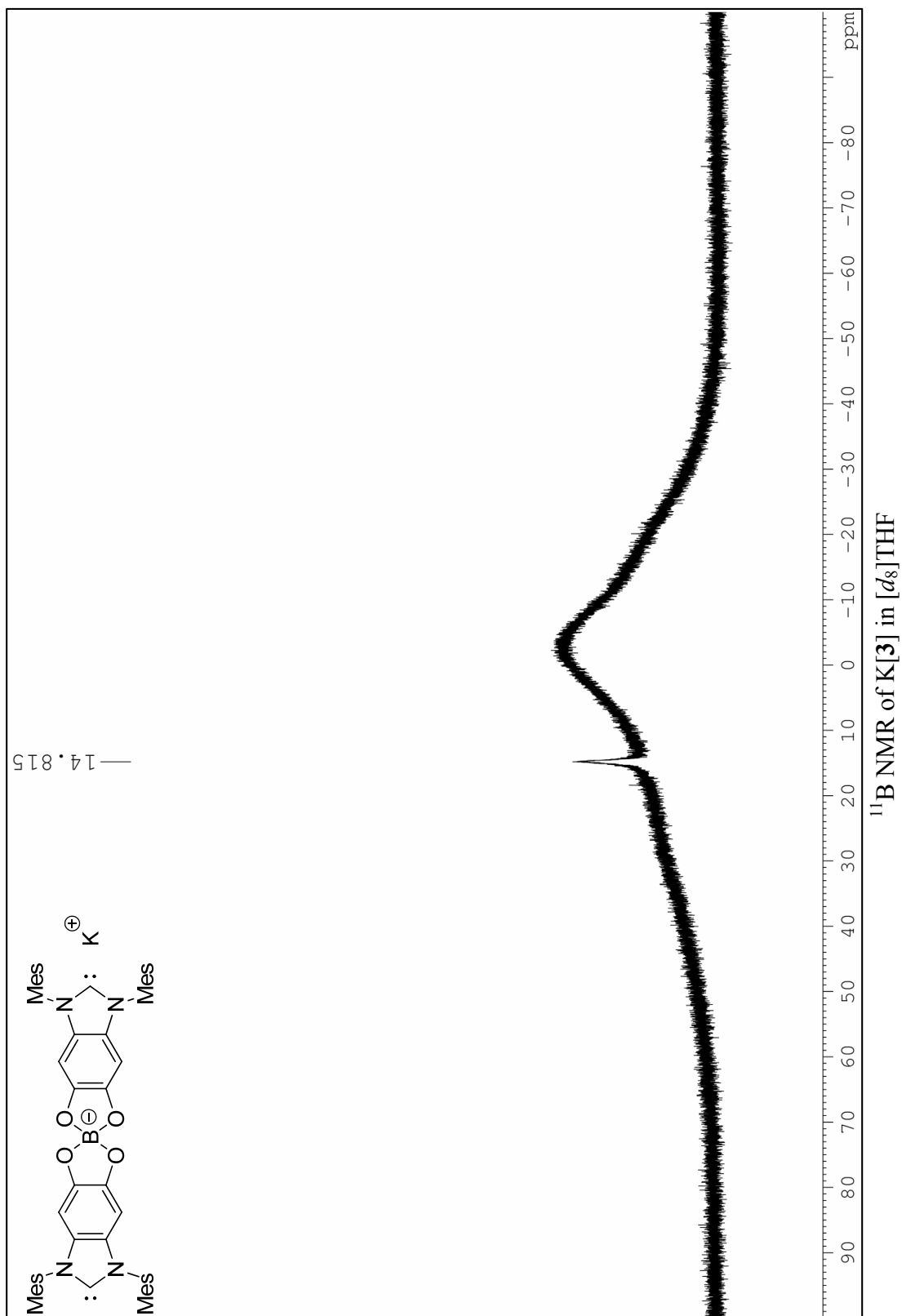


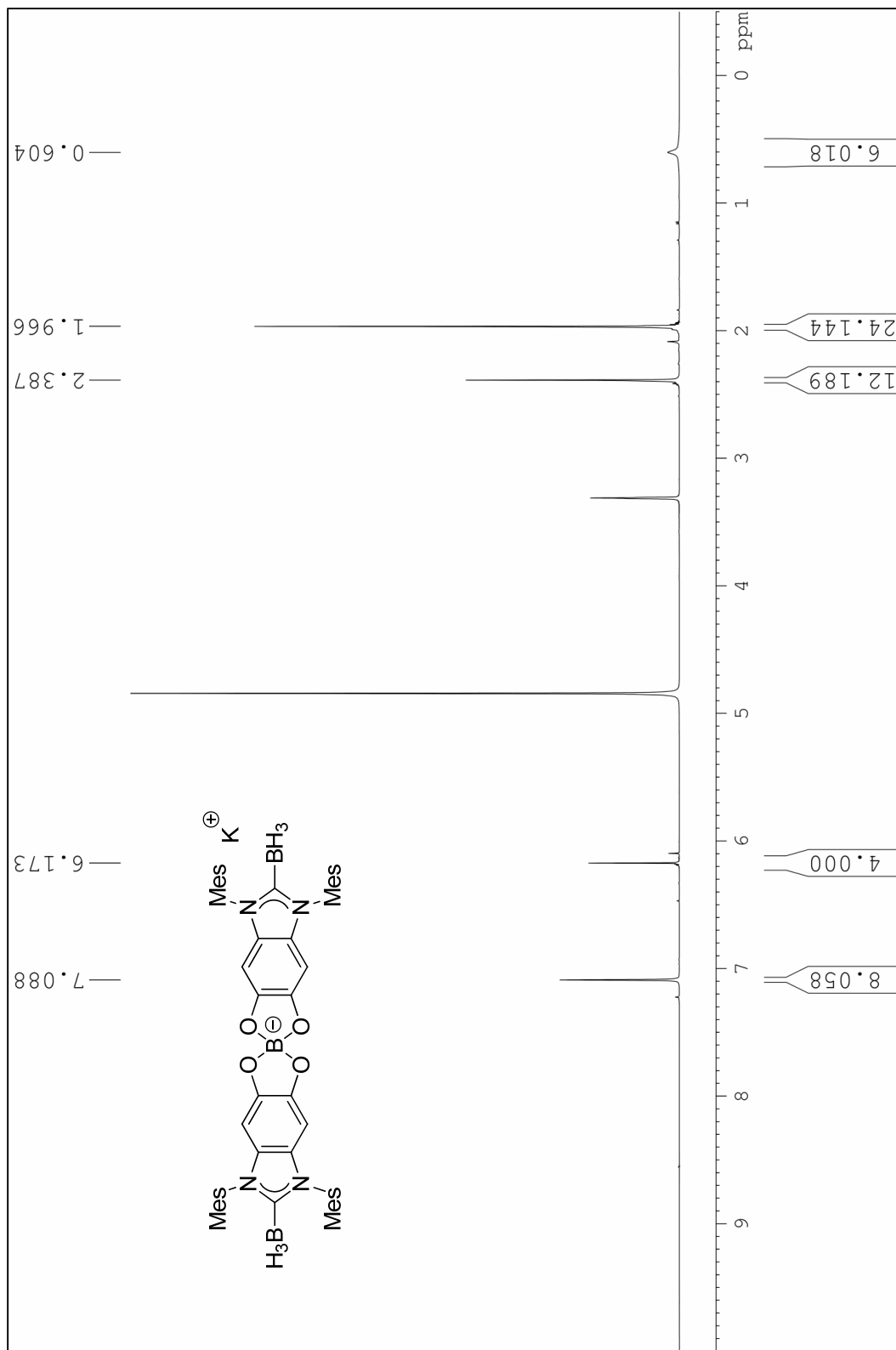


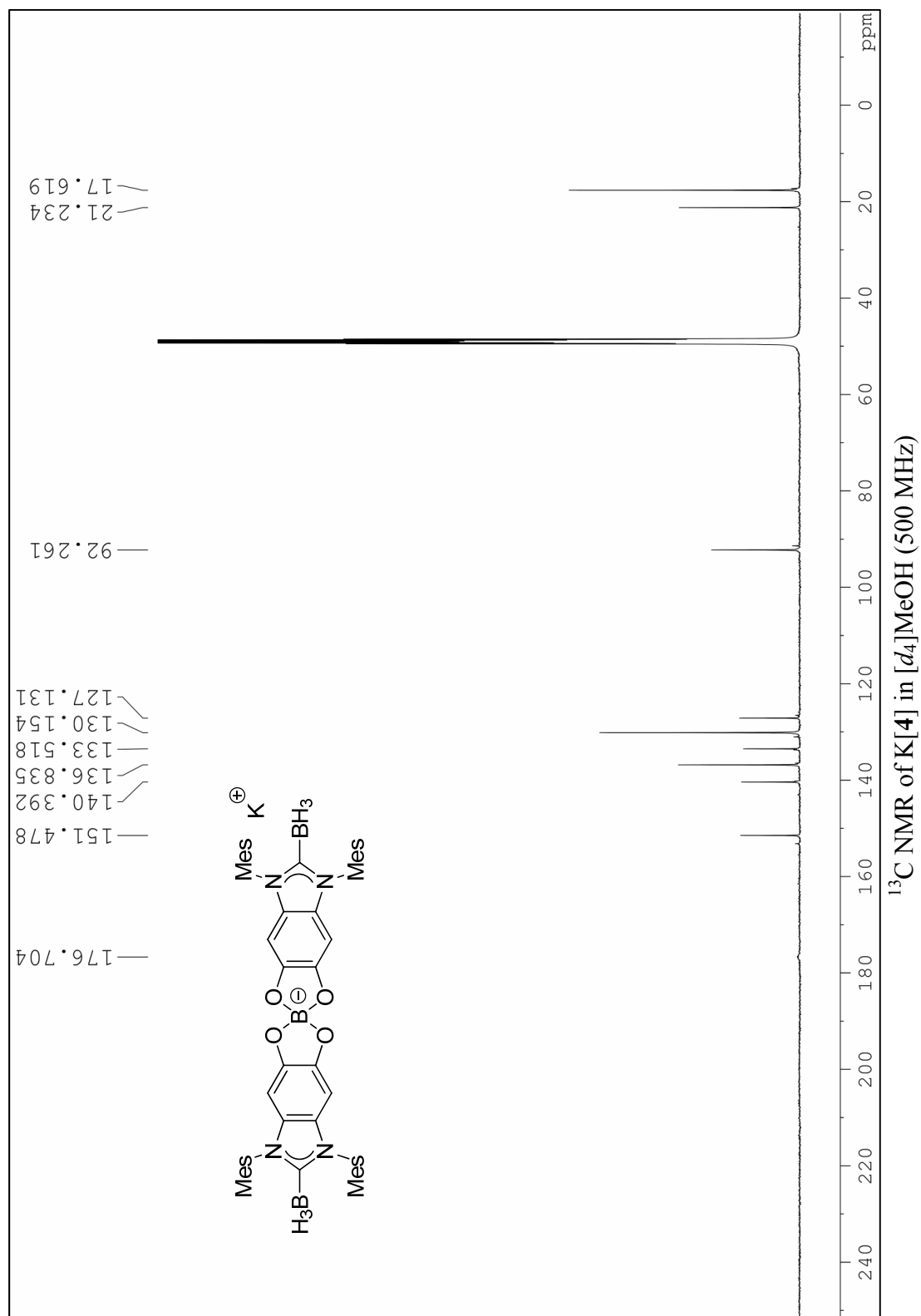


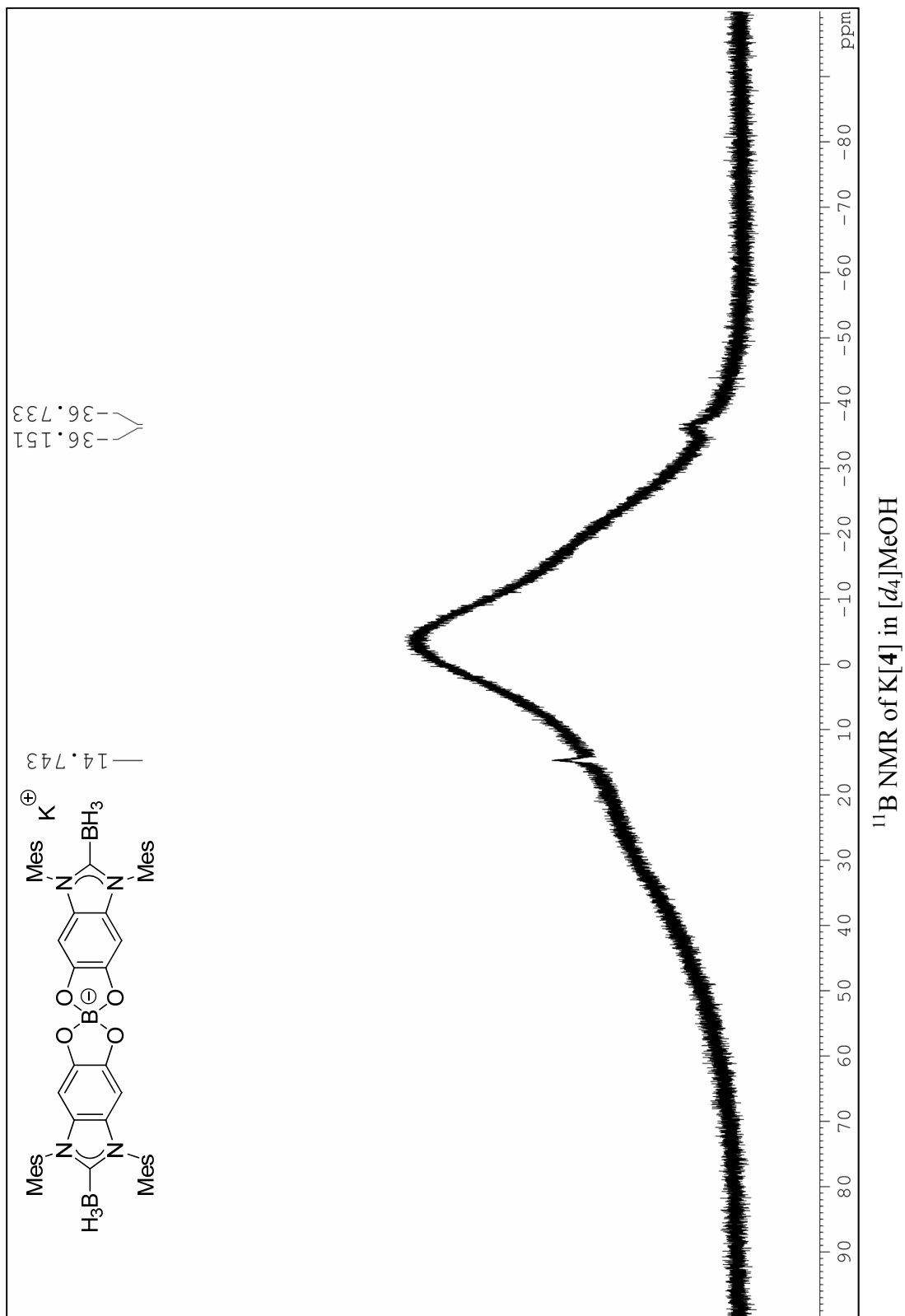


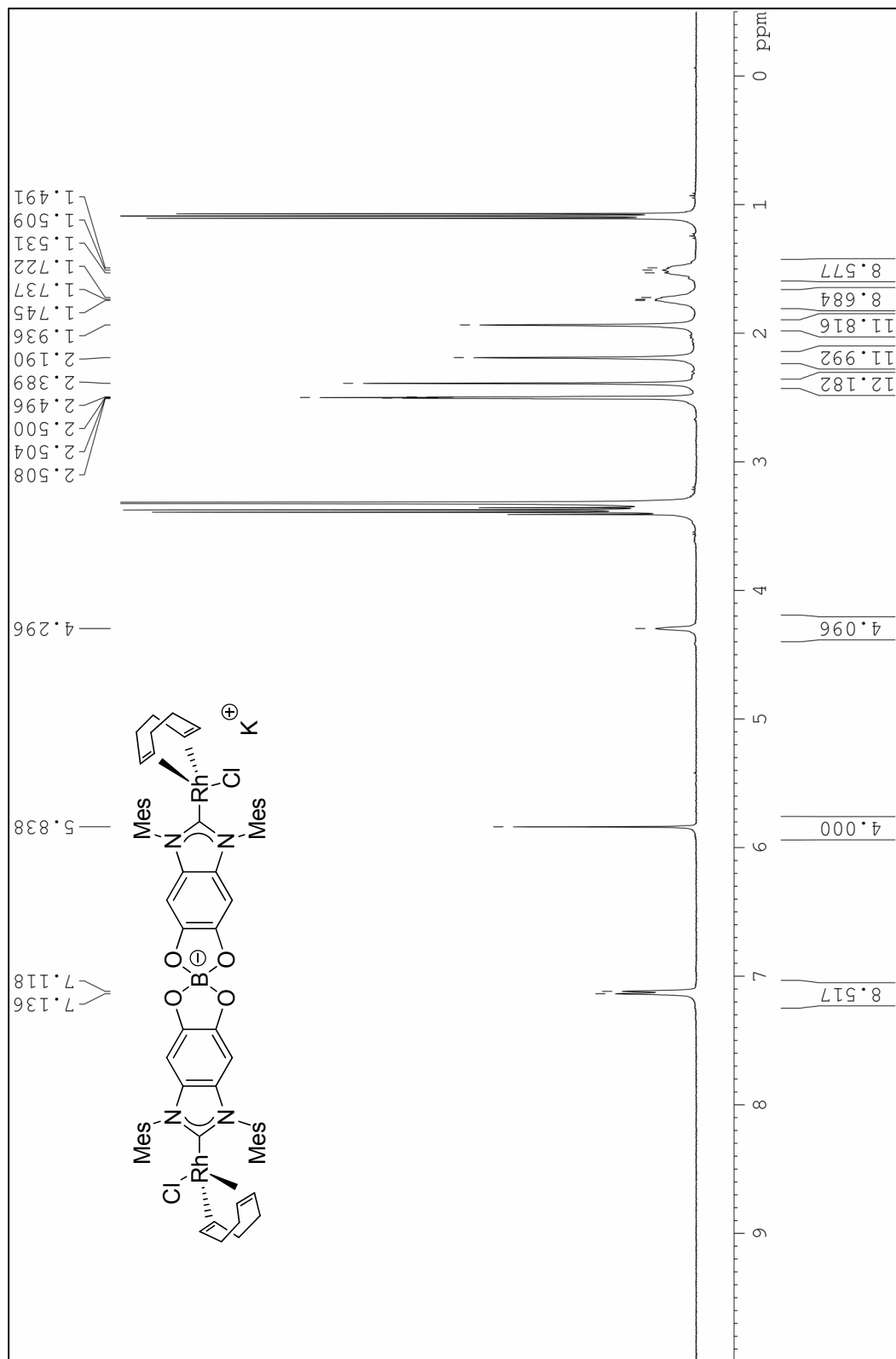


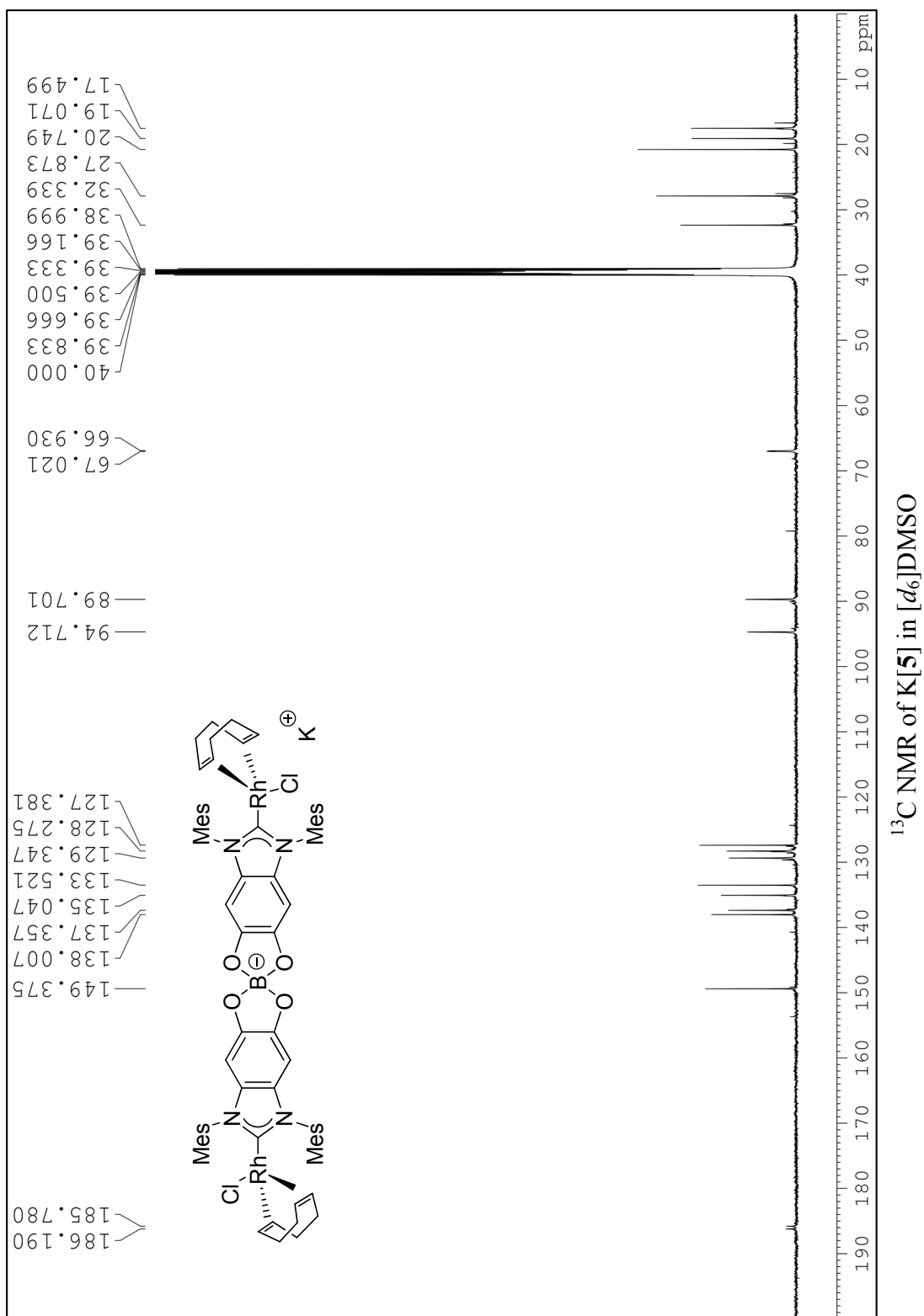


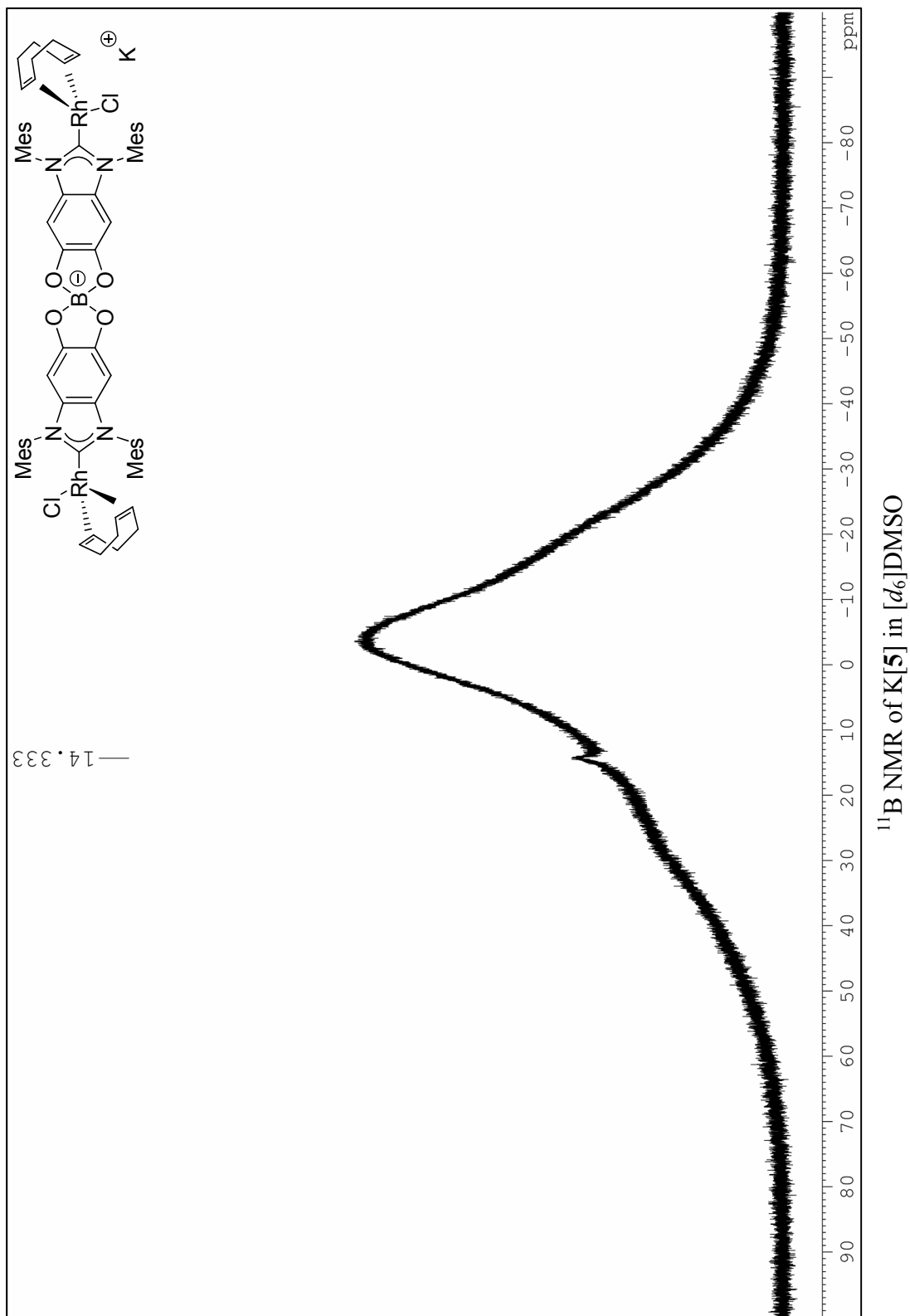


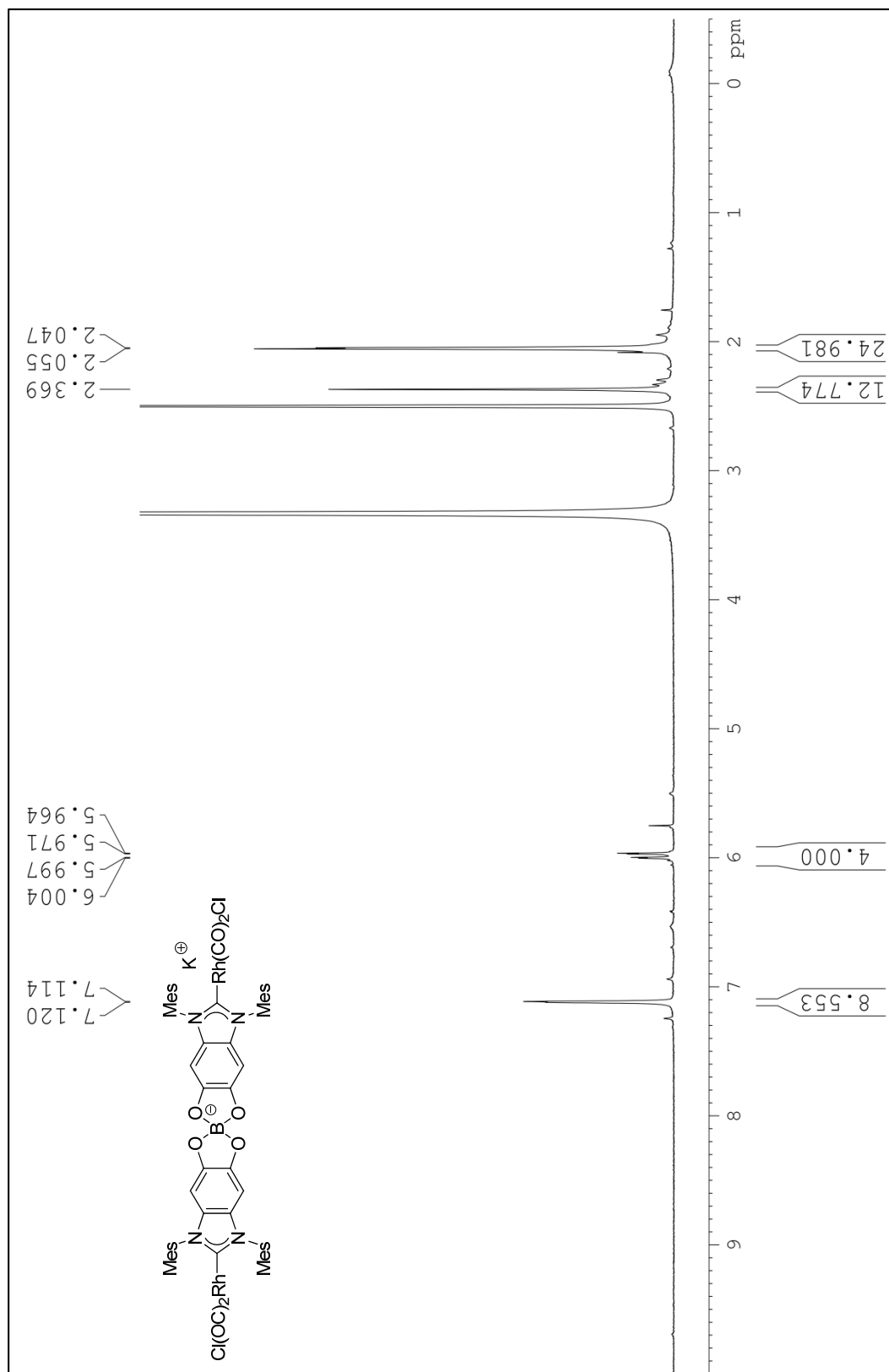


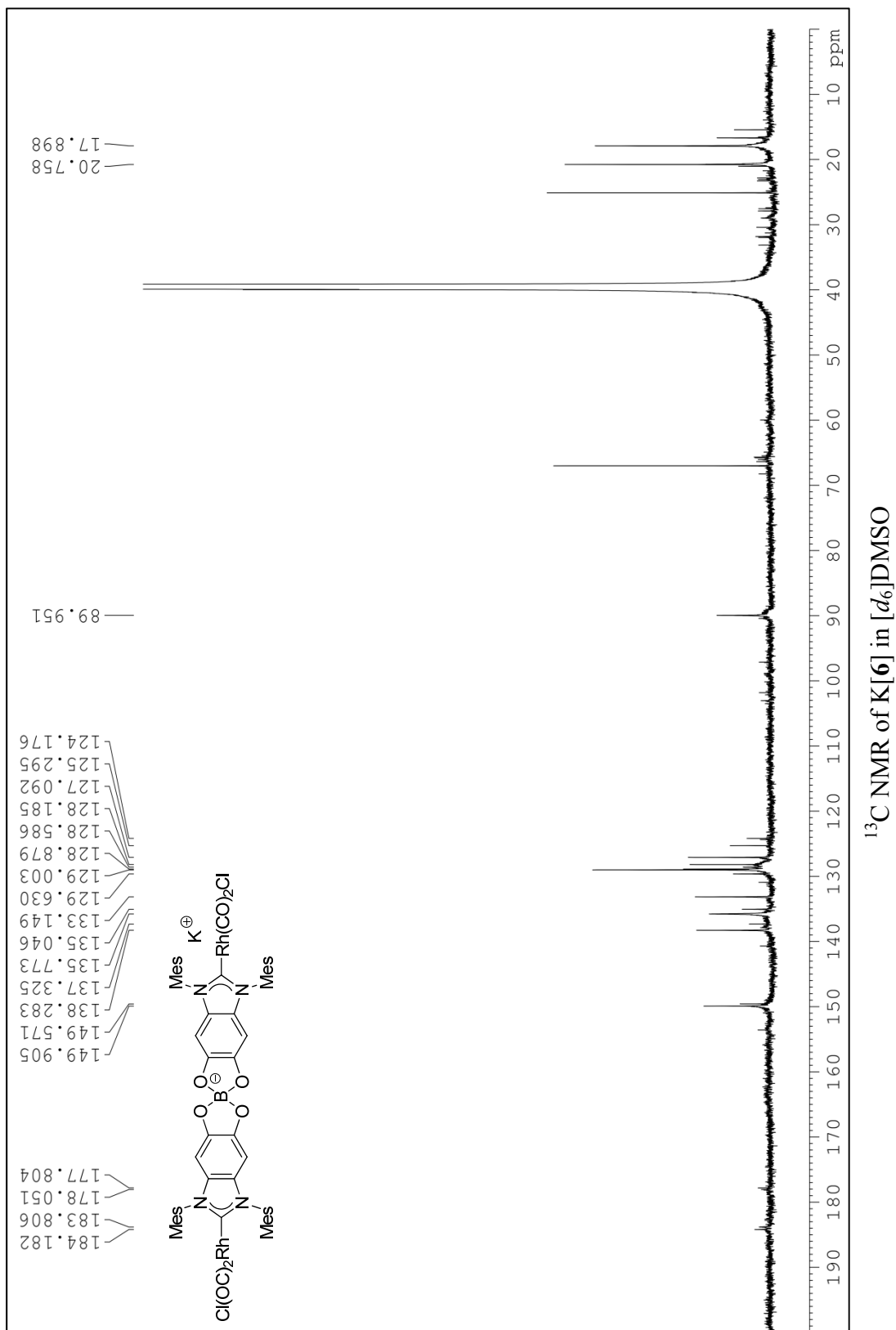












HR-ESI-MS for 5,6-dimethoxy-1,3-dimesitylbenzimidazolium tetrafluoroborate

Mass Spectrum SmartFormula Report

Analysis Info

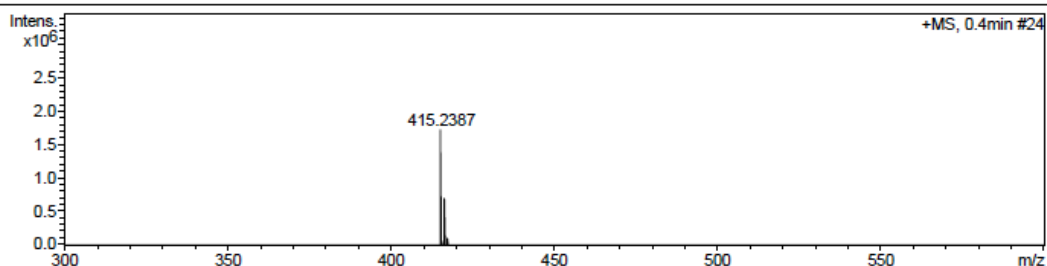
Analysis Name D:\Data\FishMS20130823_Mes-imidzolium_pos_wide000001.d
Method tune_wide.m
Sample Name TuningMix 1:100
Comment

Acquisition Date 8/23/2013 5:24:14 PM
Operator BDAL@DE
Instrument / Ser# micrOTOF-Q 10183

Acquisition Parameter

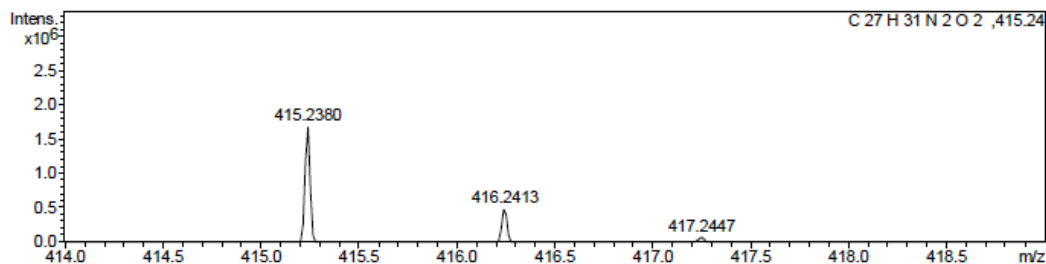
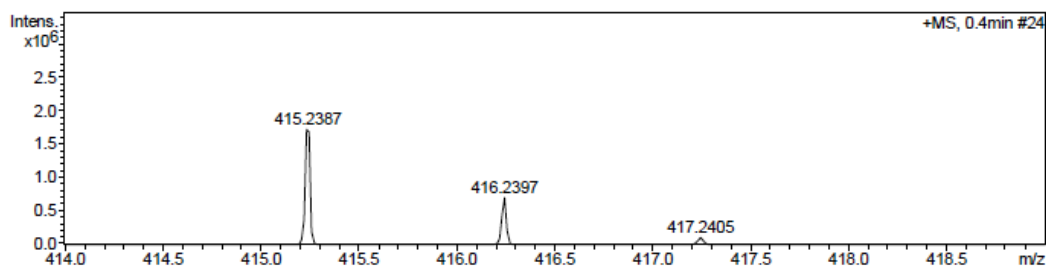
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Source

+MS, 0.4min #24



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e ⁻ Conf	N-Rule
415.2387	1	C 27 H 31 N 2 O 2	100.00	415.2380	-0.7	-1.6	52.9	13.5	even	ok

+MS, 0.4min #24



+MS, 0.4min #24

HR-ESI-MS for K[4]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\FishMS20130610_BH3NHC_neg_wide000002.d
Method tune_wide_neg_20130103.m
Sample Name TuningMix 1:100
Comment

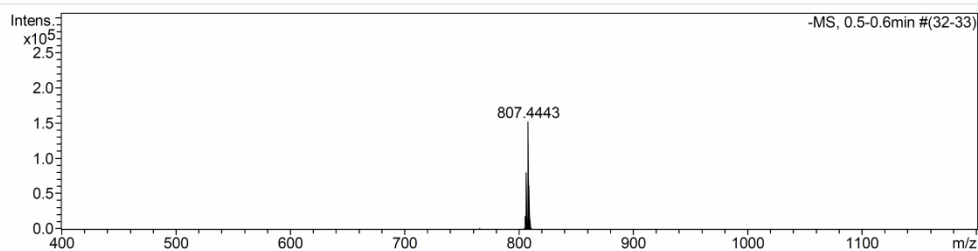
Acquisition Date 6/10/2013 6:01:40 PM

Operator BDAL@DE
Instrument / Ser# micrOTOF-Q 10183

Acquisition Parameter

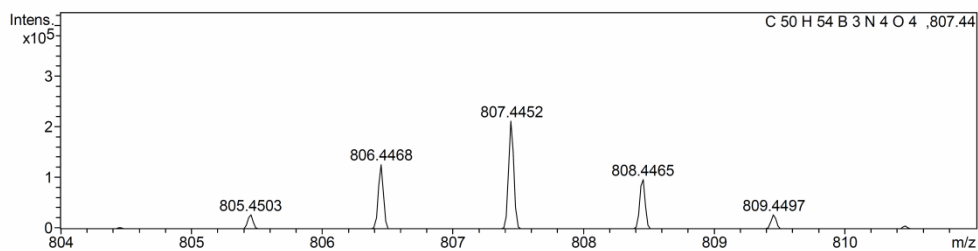
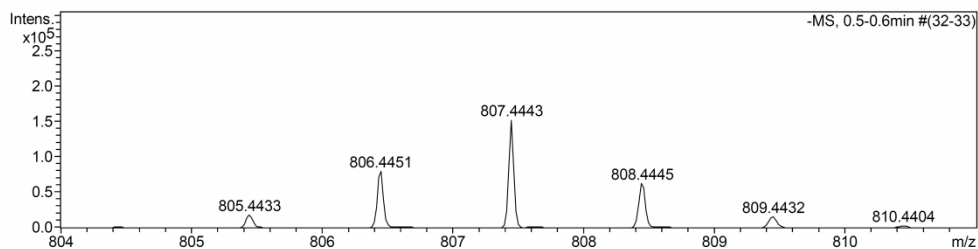
Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	3300 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Source

-MS, 0.5-0.6min #(32-33)



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e ⁻ Conf	N-Rule
807.4443	1	C ₅₀ H ₅₄ B ₃ N ₄ O ₄	100.00	807.4452	0.9	1.2	35.6	27.5	even	ok

-MS, 0.5-0.6min #(32-33)



HR-ESI-MS for K[5]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\Fish\MS20130613_RhCODNHC_neg_wide000001.d
Method tune_wide_neg_20130103.m
Sample Name TuningMix 1:100
Comment

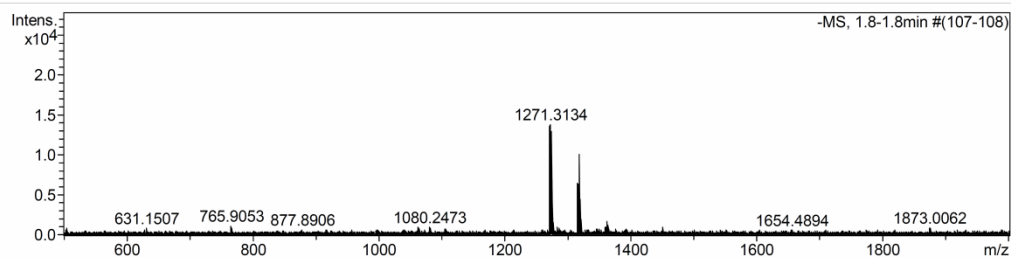
Acquisition Date 6/13/2013 12:10:56 PM

Operator BDAL@DE
Instrument / Ser# micrOTOF-Q 10183

Acquisition Parameter

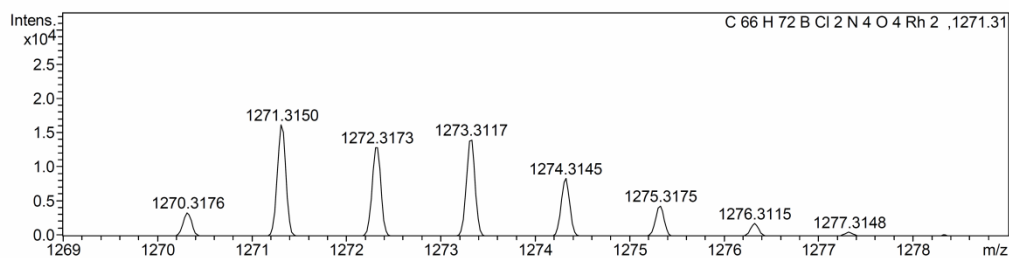
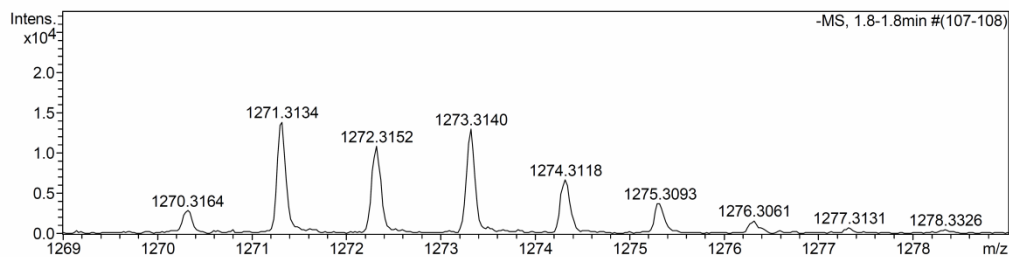
Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	3300 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Source

-MS, 1.8-1.8min #(107-108)



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e ⁻ Conf	N-Rule
1271.3134	1	C 66 H 72 B Cl 2 N 4 O 4 Rh 2	100.00	1271.3150	0.5	0.4	26.8	33.5	even	ok

-MS, 1.8-1.8min #(107-108)



HR-ESI-MS for K[6]

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\Fish\MS20130703_RhCO_neg_wide000002.d
Method tune_wide_neg_20130103.m
Sample Name TuningMix 1:100
Comment

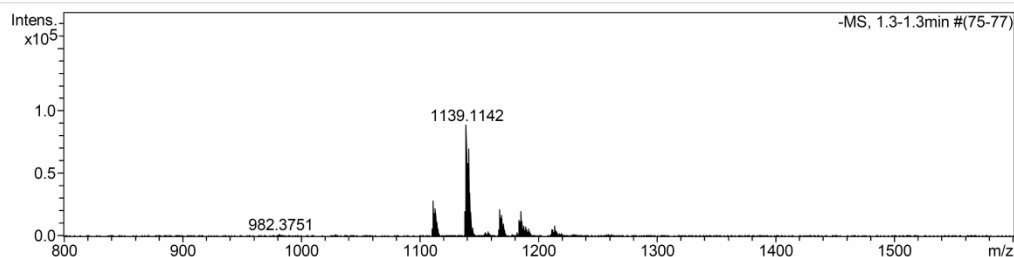
Acquisition Date 7/3/2013 5:47:51 PM

Operator BDAL@DE
Instrument / Ser# micrOTOF-Q 10183

Acquisition Parameter

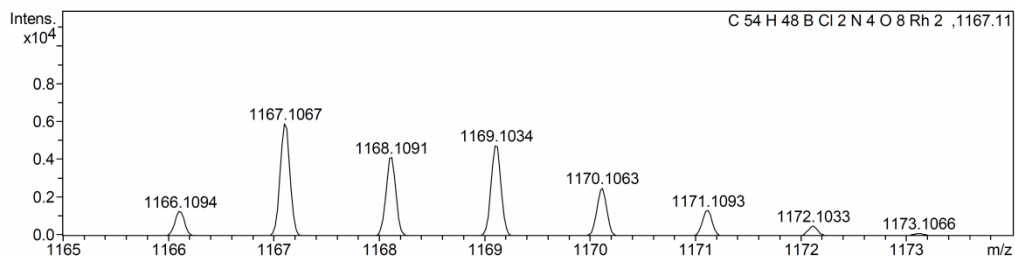
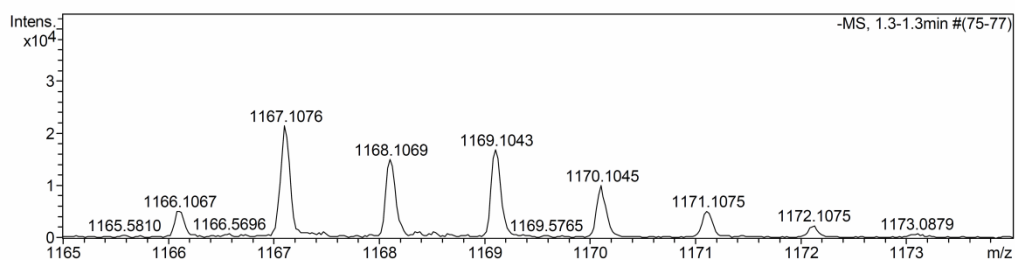
Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	3300 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Source

-MS, 1.3-1.3min #(75-77)



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e ⁻ Conf	N-Rule
1166.1067	1	C ₅₄ H ₄₈ B ₂ Cl ₂ N ₄ O ₈ Rh ₂	100.00	1167.1067	2.7	2.3	26.5	33.5	even	ok

-MS, 1.3-1.3min #(75-77)



HR-ESI-MS for K[6]

[6]⁻ - CO

Mass Spectrum SmartFormula Report

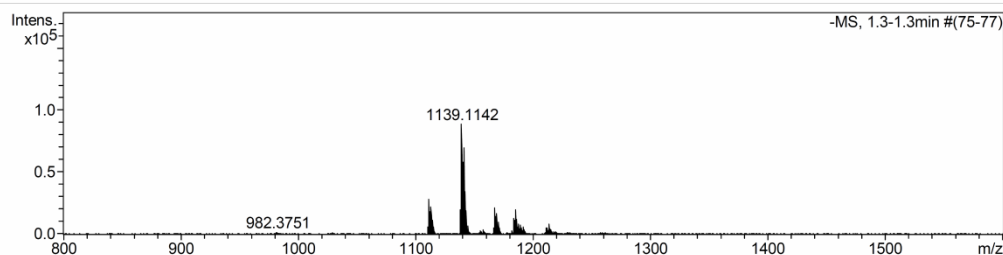
Analysis Info

Analysis Name D:\Data\Fish\Data\2013\130703\MS20130703_RhCO_neg_wide000002.d
Method tune_wide_neg_20130103.m
Sample Name TuningMix 1:100
Comment
Acquisition Date 7/3/2013 5:47:51 PM
Operator BDAL@DE
Instrument / Ser# micrOTOF-Q 10183

Acquisition Parameter

Source Type ESI Ion Polarity Negative Set Nebulizer 0.4 Bar
Focus Active Set Capillary 3300 V Set Dry Heater 180 °C
Scan Begin 50 m/z Set End Plate Offset -500 V Set Dry Gas 4.0 l/min
Scan End 3000 m/z Set Collision Cell RF 600.0 Vpp Set Divert Valve Source

-MS, 1.3-1.3min #(75-77)



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e ⁻ Conf	N-Rule
1139.1142	1	C 53 H 48 B Cl 2 N 4 O 7 Rh 2	100.00	1139.1117	-2.4	-2.1	18.0	32.5	even	ok
1166.1067	1	C 54 H 48 B Cl 2 N 4 O 8 Rh 2	100.00	1167.1067	2.7	2.3	26.5	33.5	even	ok

-MS, 1.3-1.3min #(75-77)

