–Supporting Information–

Revisiting Polyfluoroarenes as Radical Acceptors: Radical C−F Bond Borylation of Polyfluoroarenes with N-Heterocyclic Carbene Boranes and Synthesis of Borane-Containing Liquid Crystals

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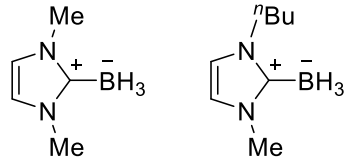
1. General remarks

All reactions were performed in oven-dried glassware. A pressure vessel [ACE GLASS Inc. (15 mL)] was used in radical reactions. All reagents purchased commercially were used without further purification unless otherwise noted. Dehydrated solvents were purchased from Kanto Chemical Co., Inc and Sigma-Aldrich. Thin-layer chromatography (TLC) analysis was performed by illumination with a UV lamp (254 nm) or staining with phosphomolybdic acid (PMA) and heating. Column chromatography was carried out on silica gel 60N (Kanto Chemical Co., Inc., spherical, neutral, 40−50 μm). DSC analysis including measurement of melting points was performed on TA Instruments DSC 250. ^{1}H, ^{13}C, ^{11}B and ^{19}F NMR spectra (in $CDCl_3$) were recorded on Bruker Avance 400, JEOL JNM-ECA 500 and JEOL JNM-ECA600 spectrometers at 20 °C. Chemical shifts (δ) are quoted relative to tetramethylsilane (^{1}H NMR, δ 0 ppm), the signals of the solvent (^{13}C NMR, $CDCl_3$: δ 77.0 ppm), boron trifluoride diethyl ether complex (^{11}B NMR, external standard: δ 0 ppm) and hexafluorobenzene (^{19}F NMR, external standard: δ −164.90 ppm). The resonances for carbons bonded to boron in the ^{13}C NMR spectra were weak and not often observed. Coupling constants (J) are given in Hz. Multiplicities are indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet) or br (broadened). IR spectra were recorded with a JASCO Fourier Transform IR-460 spectrometer. Mass spectra were recorded on JEOL JMS-T100TD (direct analysis in real time, DART). Polarizing optical microscopy (POM) images were recorded on Olympus BX53F polarizing microscope.

2. Synthetic experiments

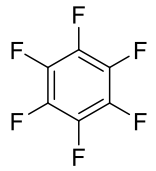
Starting materials and reagents:



2 **13**

1,3-Dimethylimidazol-2-ylidene borane (**2**)[1] and 1-butyl-3-methylimidazol-2-ylidene borane (**13**)[2] was prepared according to the established procedure.

Di-*tert*-butyl peroxide (*t*-BuOO*t*-Bu, DTBP) was purchased from nakalai tesque Inc.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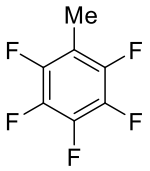
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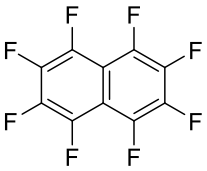
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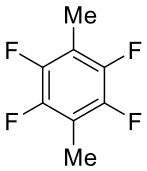
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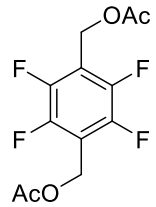
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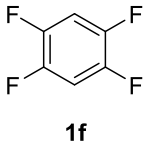
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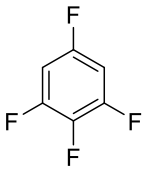
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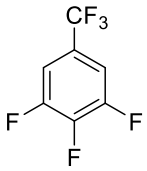
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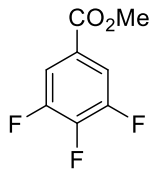
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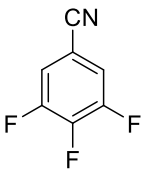
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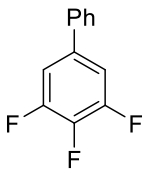
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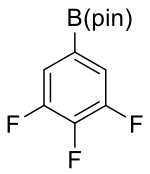
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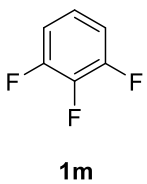
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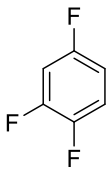
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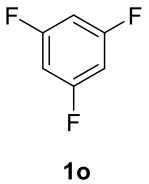
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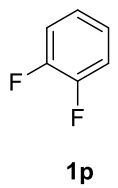
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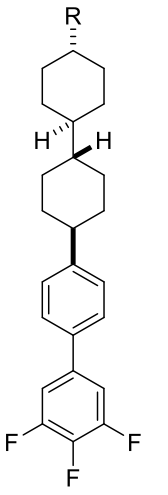
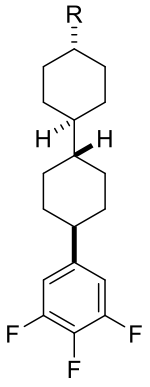


12a (R = $^{n}C_5H_{11}$)
12b (R = $^{n}C_3H_7$)



16a (R = $^{n}C_5H_{11}$)
16b (R = $^{n}C_3H_7$)

1a–d, 1f–j, 1l, 1m–p, 12a,b and **16a,b** were commercially available from some suppliers.

1k[3] was prepared according to methods previously reported.

1e was prepared as shown below.

(2,3,5,6-Tetrafluoro-1,4-phenylene)bis(methylene) diacetate (1e).

To a solution of commercially available 2,3,5,6-Tetrafluoro-1,4-benzenedimethanol (1.00 g, 4.75

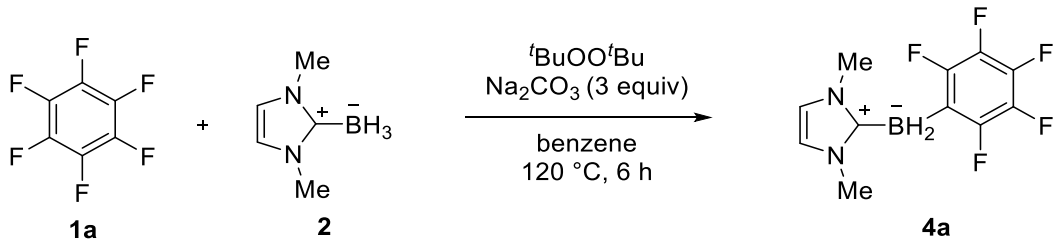
mmol) in CH_2Cl_2 (24 mL), pyridine (1.88 g, 23.8 mmol) and acetic anhydride (2.43 g, 23.8 mmol) were slowly added at room temperature. After the mixture was stirred for 21 h at the same temperature, 1 M HCl aq. solution was added to the reaction solution, and the mixture was extracted with CH_2Cl_2 three times. The combined organic phase was successively washed with a saturated $NaHCO_3$ aq. solution and brine, and dried over Na_2SO_4. After the solvent was evapolated, the residue was purified by silica gel chromatography (eluent: *n*-hexane/EtOAc, 90:10) to give **1e** (743 mg, 2.53 mmol, 53% yield) as a white solid (mp: 78.6–79.8 °C).

^{1}H NMR (600 MHz, $CDCl_3$): δ 5.22 (s, 4H), 2.10 (s, 6H).
^{13}C NMR (150 MHz, $CDCl_3$): δ 170.2, 144.2–146.0 (m including a large d, $J_{C–F}$ = 253 Hz), 115.1–115.3 (m), 53.6, 20.6.
^{19}F NMR (564 MHz, $CDCl_3$): δ –145.75.
IR ($CHCl_3$, cm^{-1}): ν_{max} 1747, 1494, 1223.
HRMS (DART+): calcd for $C_{12}H_{14}F_4NO_4$ [M+NH_4] 312.0859, found 312.0864.

Optimization of the reaction of hexafluorobenzene (1a) with NHC-borane 2:

The amounts of NHC-borane **2** and DTBP and concentration of the substrates significantly affected to yield of product **4a** (Table S1, entries 1–4). In the reaction of **1a**, $PhCF_3$ was not a good solvent (entry 5). We found that the reaction occurred at 120 °C in the absence of the initiator (entry 6), suggesting that a non-radical pathway might compete (e.g., ionic hydroboration induced by hydride transfer to hexafluorobenzene). But the reaction was very sluggish, so the radical mechanism would be a main pathway.

Table S1. Reaction of hexafluorobenzene (**1a**) and diMe-Imd-BH_3 (**2**)[a]



entry	**2** (equiv)	^{t}BuOO^{t}Bu (equiv)	conc. of **1a** (M)	yield[b] (%)
1	1.5	0.5	0.5	35
2	1.5	1.0	0.5	40
3	1.5	1.0	0.2	58
4	2.0	1.0	0.2	66[c]
5[d]	2.0	1.0	0.2	47
6[e]	2.0	0	0.5	11

[a]Conditions: **1a** (0.4 mmol), **2** (0.6–0.8 mmol), DTBP (0–0.4 mmol), Na_2CO_3 (1.2 mmol) in benzene (0.8–2.0 mL) at 120 ˚C in a sealed tube. [b]Yield estimated by ^{1}H NMR analysis of the crude product (internal standard: dimethyl sulfone). [c]Yield isolated by silica gel chromatography. [d]Benzotrifluoride ($PhCF_3$) was used as a solvent. [e]The reaction was conducted for 48 h in the absence of Na_2CO_3.

General procedure 1 (GP1):

A mixture of polyfluoroarene (0.40 mmol), NHC-borane **2** (88.0 mg, 0.80 mmol), di-*tert*-butyl peroxide (58.5 mg, 74 μL, 0.40 mmol) and Na_2CO_3 (127 mg, 1.20 mmol) in benzene or benzotrifluoride (2 mL) was heated for given time (see below for details) at 120 °C (oil bath) in a sealed pressure tube. After the solvent was removed under reduced pressure, the crude product was purified by silica gel chromatography (eluent: *n*-hexane/EtOAc, see below for details) to give the corresponding *B*-aryl NHC-borane product.

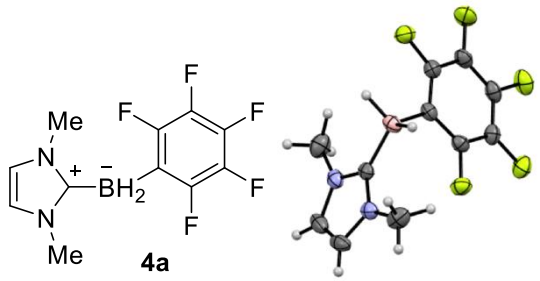
General procedure 2 (GP2):

A mixture of polyfluoroarene (0.40 mmol), NHC-borane **2** (132.0 mg, 1.20 mmol) and di-*tert*-butyl peroxide (87.7 mg, 0.11 mL, 0.60 mmol) in benzotrifluoride (0.8 mL) was heated for given time (see below for details) at 120 °C (oil bath) in a sealed pressure tube. After the solvent was removed under reduced pressure, the crude product was purified by silica gel chromatography (eluent: *n*-hexane/EtOAc, see below for details) to give the corresponding *B*-aryl NHC-borane product.

Cautions:

DTBP is a high-energy compound and sensitive to heating and shock. In addition, the present reaction causes high pressure due to heating of the reaction mixture in a sealed tube and because the present reaction generates HF, which is then quenched by NHC-boranes or Na_2CO_3 to generate H_2 and CO_2 gas. Therefore, appropriate precautions against explosion (e.g., use of a safety shield and tougher pressure tubes) should be paid, especially in large scale reactions.

Experimental details and analytical data for synthesized compounds:



(1,3-Dimethyl-1*H*-imidazol-3-ium-2-yl)(perfluorophenyl)dihydroborate (4a).

Hexafluorobenzene (**1a**) (74.4 mg, 46 μL, 0.40 mmol) was used according to GP1.

Reaction time: 6 h.

Eluent in chromatography: *n*-hexane/EtOAc, 70:30.

72.3 mg, 0.262 mmol, 66% yield.

White solid, mp: 85.8–87.8 °C.

The crystal for X-ray crystallography was grown from a *n*-hexane/ethyl acetate solution.

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.86 (s, 2H), 3.76 (s, 6H), 2.19 (2H, br-q, $J_{H–B}$ = 89 Hz).

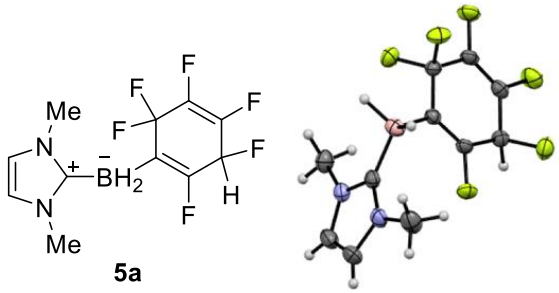
^{13}C NMR (100 MHz, $CDCl_3$): δ 167.1–168.7 (m), 146.5–149.0 (m a including large d, $J_{C–F}$ = 230 Hz), 136.7–139.5 (m including a large d, $J_{C–F}$ = 244 Hz), 135.3–138.2 (m including a large d, $J_{C–F}$ = 248 Hz), 120.6, 35.9, 29.7.

^{11}B NMR (128 MHz, $CDCl_3$): δ –32.69 (t, $J_{B–H}$ = 89 Hz).

^{19}F NMR (376 MHz, $CDCl_3$): δ –136.28 (d, J = 20.8 Hz, 2F), –165.04 (t, J = 20.1 Hz, 1F), –168.36 - –168.21 (m, 2F).

IR ($CHCl_3$, cm^{-1}): ν_{max} 2367, 1508, 1464.

HRMS (DART+): calcd for $C_{11}H_{14}BF_5N_3$ [M+NH_4] 294.1201, found 294.1197.



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(1,3-Dimethyl-1*H*-imidazol-3-ium-2-yl)(2,3,4,5,6,6-hexafluorocyclohexa-1,4-dien-1-yl)dihydroborate (5a).

A mixture of hexafluorobenzene (**1a**) (74.4 mg, 46 µL, 0.40 mmol), NHC-borane **2** (66.0 mg, 0.60 mmol) and di-*tert*-butyl peroxide (29.2 mg, 37 µL, 0.20 mmol) in benzene (0.8 mL) was heated for 4 h at 120 °C (oil bath) in a sealed pressure tube. After the solvent was removed under reduced pressure, the crude product was purified by silica gel chromatography (eluent: *n*-hexane/EtOAc, 70:30) to give an inseparable mixture (71.3 mg, ca. 65%) of **4a** and **5a** (30/70) as a white solid. Recrystallization (two times) from a *n*-hexane/ethyl acetate solution gave pure **5a** as colorless crystals (mp: 116.2–119.2 °C).

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.86 (s, 2H), 5.42 (br-d, $J_{H–F}$ = 52 Hz, 1H), 3.76 (s, 6H), 1.93 (br-q, $J_{H–B}$ = 89 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 152.1 (dt. $J_{C–F}$ = 248, 14.4 Hz), 138.5–142.0 (m including a large d, $J_{C–F}$ = 274 Hz), 137.7–140.9 (m including a large d, $J_{C–F}$ = 264 Hz), 120.6, 113.1–118.1 (m including a large t, $J_{C–F}$ = 229 Hz), 79.6 (ddd. $J_{C–F}$ = 175, 37.6, 24.6 Hz), 36.0.

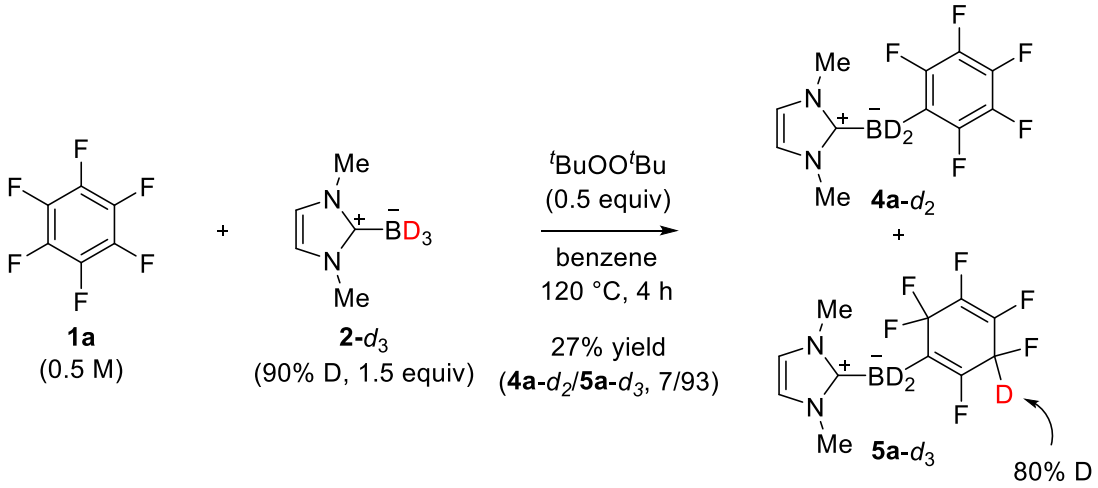
^{11}B NMR (128 MHz, $CDCl_3$): δ –33.32 (t, $J_{B–H}$ = 89 Hz).

^{19}F NMR (376 MHz, $CDCl_3$): δ –98.31 (d, J = 302 Hz, 1F), –101.15 (d, J = 302 Hz, 1F), –115.77 (d, J = 30.5 Hz, 1F), –150.15 (tt, J = 17.1, 5.5 Hz, 1F), –160.65 - –160.53 (m, 1F), –189.08 - –188.69 (m, 1F).

IR ($CHCl_3$, cm^{-1}): ν_{max} 2374, 1770, 1672, 1485, 1315, 1227.

HRMS (DART+): calcd for $C_{11}H_{15}BF_6N_3$ [M+NH_4] 314.1263, found 314.1257.

Reaction of **1a** with NHC-BD_3 (**2**-d_3):



According to GP1 (reaction time: 4 h) in the absence of Na_2CO_3, **1a** (37.2 mg, 0.20 mmol), **2**-d_3 (33.9 mg, 0.30 mmol, 90% D) and DTBP (14.6 mg, 18.5 µL, 0.10 mmol) were used in benzene (0.4 mL), and an inseparable mixture (16.0 mg, ca. 27%) of **4a**-d_2 and **5a**-d_3 (ca. 7/93 from the ^{19}F NMR spectrum) was obtained as a white solid. The D/H ratio was estimated at 80/20 from the ^{1}H NMR spectrum.

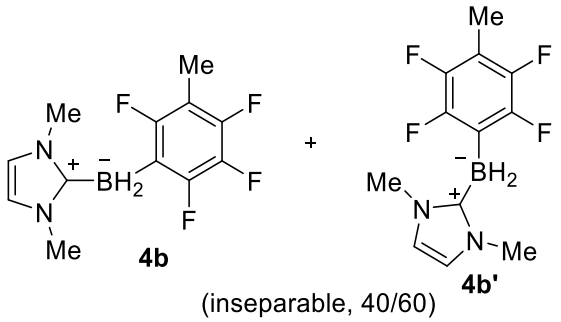
5a-d_3:

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.86 (s, 2H), 5.42 (br-d, J_{H-F} = 52 Hz, 0.2H), 3.76 (s, 6H).

^{11}B NMR (128 MHz, $CDCl_3$): δ –33.56 (br).

^{19}F NMR (376 MHz, $CDCl_3$): δ –98.33 (d, J = 302 Hz, 1F), –101.15 (d, J = 302 Hz, 1F), –115.79 (d, J = 30.5 Hz, 1F), –150.34 - –150.11 (m, 1F), –160.63 - –160.47 (m, 1F), –189.11 - –188.72 (m, ca. 0.2F, for C(H)F), –189.68 - –189.39 (m, ca. 0.8F, for C(D)F).

HRMS (DART+): calcd for $C_{11}H_{12}D_3BF_6N_3$ [M+NH_4] 317.1452, found 317.1458.

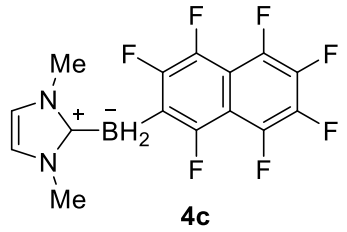


(1,3-Dimethyl-1*H*-imidazol-3-ium-2-yl)(2,3,4,6-tetrafluoro-5-methylphenyl)dihydroborate (4b) and (1,3-dimethyl-1*H*-imidazol-3-ium-2-yl)(2,3,5,6-tetrafluoro-4-methylphenyl)dihydroborate (4b')

Pentafluorotoluene (**1b**) (72.8 mg, 0.40 mmol) was used according to GP2 [but $PhCF_3$ (2.0 mL) and Na_2CO_3 (127 mg, 1.20 mmol) were used].

Reaction time: 24 h.
Eluent in chromatography: *n*-hexane/EtOAc, 70:30.
52.6 mg, 0.193 mmol, 48% yield (**4b**/**4b'**, 40/60). [Very small amounts of dearomatized products are detected in NMR spectra]
White solid, mp: 68.6–73.2 °C (dec.).

^{1}H NMR (600 MHz, $CDCl_3$): δ 6.84 (s, 2H for **4b** and 2H for **4b'**), 3.763 (s, 6H, for **4b'**), 3.760 (s, 6H, for **4b**), 1.96–2.44 (m, 2H for **4b** and 2H for **4b'**), 2.18 (t, $J_{H–F}$ = 1.2 Hz, 3H, for **4b'**), 2.09 (dd, $J_{H–F}$ = 3.6, 1.8 Hz, 3H, for **4b**).
^{13}C NMR (100 MHz, $CDCl_3$): δ 156.5–159.1 (m including a large d, $J_{C–F}$ = 224 Hz, for **4b**), 149.5–152.3 (m including a large d, $J_{C–F}$ = 233 Hz, for **4b**), 147.8 (dt, $J_{C–F}$ = 231, 14.5 Hz, for **4b'**), 145.6–148.3 (m including a large d, $J_{C–F}$ = 233 Hz, for **4b**), 143.2–145.9 (m including a large d, $J_{C–F}$ = 246 Hz, for **4b'**), 135.2–138.0 (m including a large d, $J_{C–F}$ = 248 Hz, for **4b**), 120.5 (for **4b'**), 120.4 (for **4b**), 111.0 (t, $J_{C–F}$ = 19.5 Hz, for **4b'**), 108.0–108.5 (m, for **4b**), 35.9 (for **4b** and **4b'**), 7.3 (for **4b'**), 7.2 (for **4b**).
^{11}B NMR (128 MHz, $CDCl_3$): δ –32.53 (t, $J_{B–H}$ = 89 Hz).
^{19}F NMR (376 MHz, $CDCl_3$): δ –115.12 (s, 1F, for **4b**), –134.56 (d, J = 20.8 Hz, 1F, for **4b**), –138.37 (s, 2F, for **4b'**), –147.77 (d, J = 20.8 Hz, 1F, for **4b**), –150.37 (q, J = 12.9 Hz, 2F, for **4b'**), –172.53 (td, J = 21.1, 12.8 Hz, 1F, for **4b**).
IR ($CHCl_3$, cm^{-1}): ν_{max} 2356, 1484, 1448.
HRMS (DART+): calcd for $C_{12}H_{12}BF_4N_2$ [M–H] 271.1030, found 271.1025.



4c

(1,3-Dimethyl-1*H*-imidazol-3-ium-2-yl)(perfluoronaphthalen-2-yl)dihydroborate (4c).
Octafluoronaphthalene (**1c**) (108.8 mg, 0.40 mmol) was used according to GP1.
Reaction time: 24 h.
Eluent in chromatography: *n*-hexane/EtOAc, 70:30.
57.8 mg, 0.160 mmol, 40% yield. [A very small amounts of a regioisomer (tentative) was detected in NMR spectra along with dearomatized products (**4c**/regioisomer is >95/5)]
White solid, mp: 156.8–163.0 °C (dec.).

^{1}H NMR (600 MHz, $CDCl_3$): δ 6.89 (s, 2H), 3.80 (s, 6H), 2.33 (br-q, J = 88 Hz, 2H).
^{13}C NMR (100 MHz, $CDCl_3$): δ 149.9–154.7 (m, 2C), 136.2–142.4 (m, 6C), 120.7, 107.7–109.9 (m

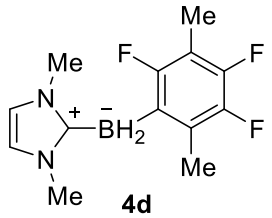
including a large d, $J_{C–F}$ = 193 Hz), 36.0.

^{11}B NMR (128 MHz, $CDCl_3$): δ –32.30 (t, $J_{B–H}$ = 89 Hz) [regioisomer (tentative): δ –29.38 (t, $J_{B–H}$ = 88 Hz)].

^{19}F NMR (376 MHz, $CDCl_3$): δ –113.88 (d, J = 72.1 Hz, 1F), –128.84 (s, 1F), –149.34 (dt, J = 70.7, 16.0 Hz, 1F), –151.65 (dt, J = 56.4, 16.0 Hz, 1F), –156.53 (dt, J = 55.3, 19.2 Hz, 1F), –161.85 (t, J = 18.0 Hz, 1F), –162.94 - –162.76 (m, 1F)

IR ($CHCl_3$, cm^{-1}): ν_{max} 2364, 1667, 1635, 1519, 1490, 1394, 1273.

HRMS (DART+): calcd for $C_{15}H_9BF_7N_2$ [M–H] 361.0747, found 361.0737.



4d

(1,3-Dimethyl-1*H*-imidazol-3-ium-2-yl)(2,4,5-trifluoro-3,6-dimethylphenyl)dihydroborate (4d). 2,3,5,6-Tetrafluoro-*p*-xylene (**1d**) (71.3 mg, 0.40 mmol) was used according to GP2.

Reaction time: 24 h.

Eluent in chromatography: *n*-hexane/EtOAc, 70:30.

54.2 mg, 0.202 mmol, 51% yield (in $PhCF_3$)

57.0 mg, 0.213 mmol, 53% yield (in benzene).

White solid, mp: 131.1–133.8 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.82 (s, 2H), 3.67 (s, 6H), 2.33 (m, $J_{H–F}$ = 2.8 Hz, 3H), 2.17 (br-q, $J_{H–B}$ = 87 Hz, 2H), 2.04 (s, 3H).

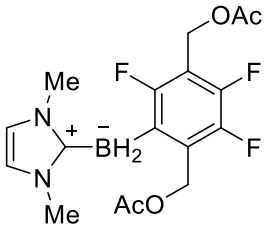
^{13}C NMR (100 MHz, $CDCl_3$): δ 157.2–159.7 (m including a large d, $J_{C–F}$ = 229 Hz), 145.1–147.7 (m including a large d, $J_{C–F}$ = 243 Hz), 127.2 (dd, $J_{C–F}$ = 13.7, 10.9 Hz), 120.2, 109.4 (dd, $J_{C–F}$ = 29.6, 15.9 Hz), 35.9, 13.6, 7.4.

^{11}B NMR (128 MHz, $CDCl_3$): δ –30.58 (t, $J_{B–H}$ = 87 Hz).

^{19}F NMR (376 MHz, $CDCl_3$): δ –118.47 (s, 1F), –150.00 (d, J = 20.8 Hz, 1F), –151.68 (t, J = 18.7 Hz, 1F).

IR ($CHCl_3$, cm^{-1}): ν_{max} 2326, 1473, 1417, 1242.

HRMS (DART+): calcd for $C_{13}H_{15}BF_3N_2$ [M–H] 267.1280, found 267.1279.



4e

(2,5-Bis(acetoxymethyl)-3,4,6-trifluorophenyl)(1,3-dimethyl-1*H*-imidazol-3-ium-2-yl)dihydroborate (4e).

(2,3,5,6-Tetrafluoro-1,4-phenylene)bis(methylene) diacetate (**1e**) (117.7 mg, 0.40 mmol) was used according to GP2.

Reaction time: 24 h.

Eluent in chromatography: *n*-hexane/EtOAc, 70:30.

77.3 mg, 0.201 mmol, 50% yield.

White solid, mp: 136.9–138.9 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.84 (s, 2H), 5.406 (d, $J_{H–F}$ = 2.4 Hz, 2H), 5.08 (s, 2H), 3.67 (s, 6H), 2.11 (br-q, $J_{H–B}$ = 88 Hz, 2H), 2.039 (s, 3H), 2.033 (s, 3H).

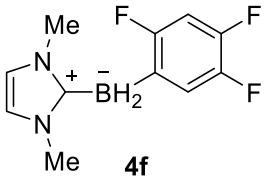
^{13}C NMR (100 MHz, $CDCl_3$): δ 170.8, 170.6, 158.6 (d, $J_{C–F}$ = 233 Hz), 145.1–148.0 (m including a large d, $J_{C–F}$ = 246 Hz), 128.6 (dd, $J_{C–F}$ = 13.7, 8.7 Hz), 120.4, 111.6 (dd, $J_{C–F}$ = 27.4, 14.4 Hz), 59.6, 54.8, 36.0, 20.9, 20.8.

^{11}B NMR (128 MHz, $CDCl_3$): δ –31.72 (t, $J_{B–H}$ = 88 Hz).

^{19}F NMR (376 MHz, $CDCl_3$): δ –116.46 (s, 1F), –147.97 (d, J = 20.8 Hz, 1F), –150.24 (t, J = 18.7 Hz, 1F).

IR ($CHCl_3$, cm^{-1}): ν_{max} 2342, 1736, 1474, 1232.

HRMS (DART+): calcd for $C_{17}H_{18}BF_3N_2O_4$ [M–H] 383.1390, found 383.1388.



4f

(1,3-Dimethyl-1*H*-imidazol-3-ium-2-yl)(2,4,5-trifluorophenyl)dihydroborate (4f).

1,2,4,5-Tetrafluorobenzene (**1f**) (60.0 mg, 0.40 mmol) was used according to GP2.

Reaction time: 24 h.

Eluent in chromatography: *n*-hexane/EtOAc, 70:30.

42.4 mg, 0.177 mmol, 44% yield [NMR yield (internal standard: dimethyl sulfone): 60%].

White solid, mp: 68.4–72.0 °C.

^{1}H NMR (600 MHz, $CDCl_3$):δ 7.11 (br, 1H), 6.84 (s, 2H), 6.61–6.65 (m, 1H), 3.74 (s, 6H), 2.21 (br-q, $J_{H–B}$ = 87 Hz, 2H).

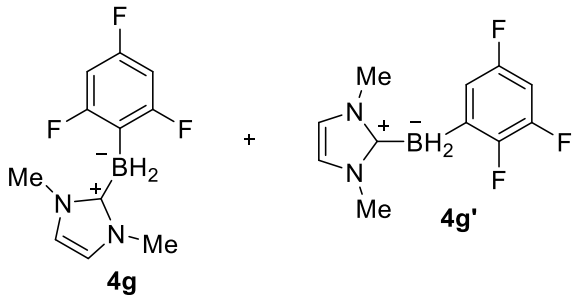
^{13}C NMR (100 MHz, $CDCl_3$): δ 160.3 (dd, $J_{C–F}$ = 233, 7.2 Hz), 147.4 (dt, $J_{C–F}$ = 242, 14.4 Hz), 145.1–147.7 (m including a large d, $J_{C–F}$ = 238 Hz), 123.7 (t, $J_{C–F}$ = 15.1 Hz), 120.4. 103.3 (dd, $J_{C–F}$ = 33.0, 18.8 Hz), 36.0.

^{11}B NMR (192 MHz, $CDCl_3$): δ –28.23 (t, $J_{B–H}$ = 89 Hz).

^{19}F NMR (376 MHz, $CDCl_3$): δ –112.72 (s, 1F), –144.77 (dt, $J_{F–H}$ = 21.1, 9.8 Hz 1F), –150.47 - –150.32 (m, 1F).

IR ($CHCl_3$, cm^{-1}): ν_{max} 2332, 1495, 1391.

HRMS (DART+): calcd for $C_{11}H_{11}BF_3N_2$ [M–H] 239.0967, found 239.0956.



(inseparable, 85/15)

(1,3-Dimethyl-1*H*-imidazol-3-ium-2-yl)(2,3,5-trifluorophenyl)dihydroborate (4g) and (1,3-dimethyl-1*H*-imidazol-3-ium-2-yl)(2,4,6-trifluorophenyl)dihydroborate (4g').

1,2,3,5-Tetraflurobenzene (**1g**) (60.0 mg, 0.40 mmol) was used according to GP2.

Reaction time: 12 h.

Eluent in chromatography: *n*-hexane/EtOAc, 70:30.

65.8 mg, 0.274 mmol, 69% yield (**4g**/**4g'**, 85/15) [in $PhCF_3$].

37.6 mg, 0.157 mmol, 39% yield (**4g**/**4g'**, 85/15) [in benzene].

White solid, mp: 55.6–63.7 °C (dec.).

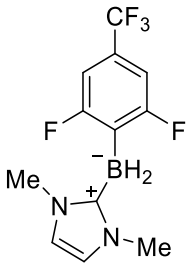
^{1}H NMR (400 MHz, $CDCl_3$): δ 6.86 (s, 2H, for **4g'**), 6.83 (s, 2H, for **4g**), 6.79–6.87 (m, 1H, for **4g'**), 6.53–6.60 (m, 1H, for **4g'**), 6.41–6.49 (m, 2H, for **4g**), 3.76 (s, 6H, for **4g'**), 3.75 (s, 6H, for **4g**), 2.26 (2H, br-q, $J_{H–B}$ = 87 Hz, for **4g'**), 2.17 (2H, br-q, $J_{H–B}$ = 87 Hz, for **4g**).

^{13}C NMR (150 MHz, $CDCl_3$): δ 165.9 (ddd, $J_{C–F}$ = 238, 20.0, 14.4 Hz, for **4g**), 160.8 (dt, $J_{C–F}$ = 241, 15.9, Hz for **4g**), 120.5 (for **4g'**), 120.3 (for **4g**), 98.5 (dd, $J_{C–F}$ = 36.1, 23.9 Hz, for **4g**), 36.0 (for **4g'**), 35.9 (for **4g**), aromatic signals of **4g'** were not detected due to the small amount.

^{11}B NMR (192 MHz, $CDCl_3$): δ –29.00 (t, $J_{B–H}$ = 87 Hz, for **4g'**), –33.68 (t, $J_{B–H}$ = 87 Hz, for **4g**).

^{19}F NMR (376 MHz, $CDCl_3$): δ –103.71 (s, 2F, for **4g**), –119.69 - –119.61 (m, 1F, for **4g**), –122.51 (dt, $J_{F–H}$ = 16.5, 8.3 Hz, 1F, for **4g'**), –140.58 (dd, J = 24.8, 10.9 Hz, 1F, for **4g'**), –142.42 (s, 1F, for **4g'**).

IR ($CHCl_3$, cm^{-1}): ν_{max} 2348, 1622, 1587, 1484, 1410.
HRMS (DART+): calcd for $C_{11}H_{11}BF_3N_2$ [M–H] 239.0967, found 239.0956.



4h

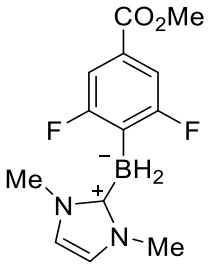
(2,6-Difluoro-4-(trifluoromethyl)phenyl)(1,3-dimethyl-1*H*-imidazol-3-ium-2-yl)dihydroborate (4h).

1,2,3-Trifluoro-5-(trifluoromethyl)benzene (**1h**) (82.5 mg, 0.40 mmol) was used according to GP2.
Reaction time: 12 h.
Eluent in chromatography: *n*-hexane/EtOAc, 70:30.
87.1 mg, 0.300 mmol, 75% yield.
White solid, mp: 59.0–62.6 °C.

2 mmol scale reaction:

A mixture of **1h** (412.5 mg, 2.0 mmol), NHC-borane **2** (659.8 mg, 6.0 mmol) and di-*tert*-butyl peroxide (438.7 mg, 0.56 mL, 3.0 mmol) in benzotrifluoride (2.0 mL) was heated for 12 h at 120 °C (oil bath) in a sealed pressure tube. After the solvent was removed under reduced pressure, the crude product was purified by silica gel chromatography (eluent: *n*-hexane/EtOAc, 70:30) to give **4h** (360.1 mg, 1.24 mmol, 62%) as a white solid.

^{1}H NMR (600 MHz, $CDCl_3$): δ 6.91–6.95 (m, 2H), 6.85 (s, 2H), 3.76 (s, 6H), 2.22 (br-q, $J_{H–B}$ = 89 Hz, 2H).
^{13}C NMR (100 MHz, $CDCl_3$): δ 166.1 (dd, $J_{C–F}$ = 239, 16.6 Hz), 128.0–129.3 (m), 123.6 (q, $J_{C–F}$ = 270 Hz), 120.5, 107.0–107.5 (m), 36.0.
^{11}B NMR (192 MHz, $CDCl_3$): δ –32.59 (t, $J_{B–H}$ = 89 Hz).
^{19}F NMR (376 MHz, $CDCl_3$): δ –65.53 (s, 3F), –103.63 (s, 2F).
IR ($CHCl_3$, cm^{-1}): ν_{max} 2355, 1564, 1484, 1410, 1339, 1230, 1168, 1126, 1082.
HRMS (DART+): calcd for $C_{12}H_{11}BF_5N_2$ [M–H] 289.0935, found 289.0942.



4i

(2,6-Difluoro-4-(methoxycarbonyl)phenyl)(1,3-dimethyl-1*H*-imidazol-3-ium-2-yl)dihydroborate (4i).

Methyl-3,4,5-trifluorobenzoate (**1i**) (75.3 mg, 0.40 mmol) was used according to GP2.

Reaction time: 12 h.

Eluent in chromatography: *n*-hexane/EtOAc, 65:35.

50.0 mg, 0.189 mmol, 47% yield.

White solid, mp: 134.6–138.6 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30–7.36 (m, 2H), 6.84 (s, 2H), 3.87 (s, 3H), 3.74 (s, 6H), 2.24 (br-q, $J_{H–B}$ = 88 Hz, 2H).

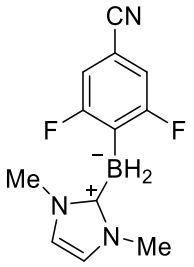
^{13}C NMR (100 MHz, $CDCl_3$): δ 166.2 (t, $J_{C–F}$ = 3.6 Hz), 166.0 (dd, $J_{C–F}$ = 238, 16.6 Hz), 128.5 (t, $J_{C–F}$ = 10.1 Hz), 120.4, 110.9–111.2 (m), 50.1, 36.0.

^{11}B NMR (128 MHz, $CDCl_3$): δ –32.41 (t, $J_{B–H}$ = 88 Hz).

^{19}F NMR (376 MHz, $CDCl_3$): δ –105.44 (s, 2F).

IR ($CHCl_3$, cm^{-1}): ν_{max} 2348, 1720, 1556, 1484, 1403, 1307, 1229.

HRMS (DART+): calcd for $C_{13}H_{19}BF_2N_3O_2$ [M+NH_4] 298.1538, found 298.1537.



4j

(4-Cyano-2,6-difluorophenyl)(1,3-dimethyl-1*H*-imidazol-3-ium-2-yl)dihydroborate (4j).

4-Cyano-1,2,3-trifluorobenzene (**1j**) (62.8 mg, 0.40 mmol) was used according to GP2.

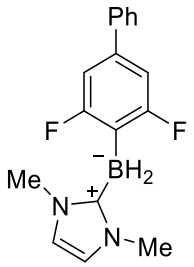
Reaction time: 12 h.

Eluent in chromatography: *n*-hexane/EtOAc, 65:35.

66.5 mg, 0.269 mmol, 67% yield.

White solid, mp: 113.0–116.0 °C.

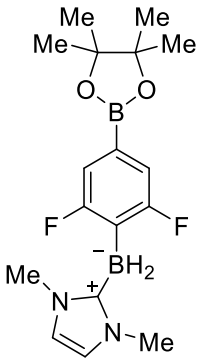
^{1}H NMR (400 MHz, $CDCl_3$): δ 6.92–6.99 (m, 2H), 6.87 (s, 2H), 3.74 (s, 6H), 2.21 (br-q, $J_{H–B}$ = 87 Hz, 2H).
^{13}C NMR (100 MHz, $CDCl_3$): δ 166.0 (dd, $J_{C–F}$ = 240 Hz), 120.6, 118.1 (d, $J_{C–F}$ = 4.2 Hz), 113.6–113.9 (m), 108.9 (t, $J_{C–F}$ = 13.0 Hz), 36.0.
^{11}B NMR (128 MHz, $CDCl_3$): δ –32.52 (t, $J_{B–H}$ = 89 Hz).
^{19}F NMR (376 MHz, $CDCl_3$): δ –99.83 (s, 2F).
IR ($CHCl_3$, cm^{-1}): ν_{max} 2349, 2229, 1544, 1485, 1407, 1299.
HRMS (DART+): calcd for $C_{12}H_{16}BF_2N_4$ [M+NH_4] 265.1436, found 265.1444.



4k

(3,5-Difluoro-[1,1'-biphenyl]-4-yl)(1,3-dimethyl-1*H*-imidazol-3-ium-2-yl)dihydroborate (4k).
3,4,5-Trifluoro-1-1'-biphenyl (**1k**) (83.3 mg, 0.40 mmol) was used according to GP2.
Reaction time: 12 h.
Eluent in chromatography: *n*-hexane/EtOAc, 70:30.
84.7 mg, 0.284 mmol, 71% yield [A very small amount of a regioisomer (tentative) was detected in NMR spectra (**4k**/regioisomer is >95/5)].
White solid, mp: 55.0–64.9 °C (dec.).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51–7.54 (m, 2H), 7.37–7.41 (m, 2H), 7.27–7.32 (m, 1H), 6.92–6.97 (m, 2H), 6.80 (s, 2H), 3.76 (s, 6H), 2.26 (br-q, $J_{H–B}$ = 88 Hz, 2H).
^{13}C NMR (100 MHz, $CDCl_3$): δ 166.7 (dd, $J_{C–F}$ = 237.6, 17.4 Hz), 139.9, 139.5 (t, $J_{C–F}$ = 10.5 Hz), 128.7, 127.3, 126.6, 120.3, 108.3–108.6 (m), 35.9.
^{11}B NMR (128 MHz, $CDCl_3$): δ –32.45 (t, $J_{B–H}$ = 88 Hz) [regioisomer (tentative): –27.91 (t, $J_{B–H}$ = 88 Hz)].
^{19}F NMR (376 MHz, $CDCl_3$): δ –106.12 (s, 2F).
IR ($CHCl_3$, cm^{-1}): ν_{max} 2346, 1540, 1484, 1444, 1398, 1232.
HRMS (DART+): calcd for $C_{17}H_{16}BF_2N_2O_3$ [M–H] 297.1375, found 297.1370.



4l

(2,6-Difluoro-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)(1,3-dimethyl-1*H*-imidazol-3-ium-2-yl)dihydroborate (4l).

3,4,5-Trifluoro-1-phenylboronic acid pinacol ester (**1l**) (103.2 mg, 0.40 mmol) was used according to GP2.

Reaction time: 21 h.

Eluent in chromatography: *n*-hexane/EtOAc, 70:30.

65.8 mg, 0.189 mmol, 47% yield.

White solid, mp: 170.0–174.3 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.06–7.12 (m, 2H), 6.80 (s, 2H), 3.73 (s, 6H), 2.23 (br-q, $J_{H–B}$ = 87 Hz, 2H), 1.31 (s, 12H).

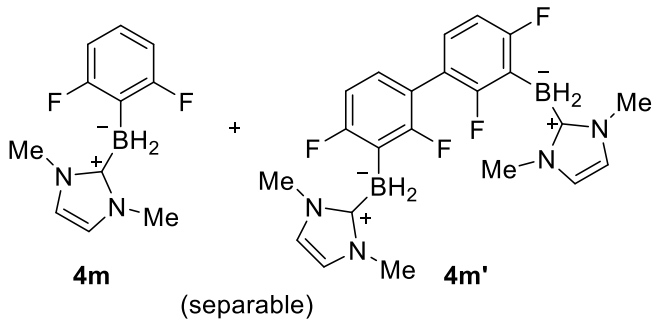
^{13}C NMR (100 MHz, $CDCl_3$): δ 166.2 (dd, $J_{C–F}$ = 238, 15.1 Hz), 126.4–128.4 (m),120.3, 115.4 (dd, $J_{C–F}$ = 21.1, 8.7 Hz), 83.7, 35.9, 24.8.

^{11}B NMR (128 MHz, $CDCl_3$): δ 30.30 (br-s), –32.45 (t, $J_{B–H}$ = 87 Hz).

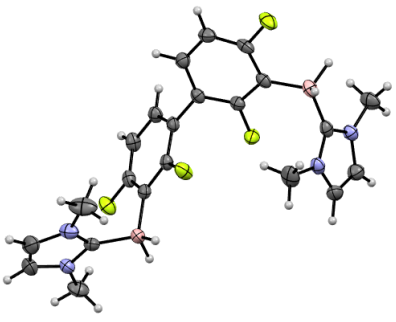
^{19}F NMR (376 MHz, $CDCl_3$): δ –107.13 (s, 2F).

IR ($CHCl_3$, cm^{-1}): ν_{max} 2349, 1481, 1392, 1356.

HRMS (DART+): calcd for $C_{17}H_{23}BF_2N_2O_2$ [M–H] 347.1914, found 347.1923.



4m (separable) **4m'**



CCDC 1982158

(2,6-Difluorophenyl)(1,3-dimethyl-1*H*-imidazol-3-ium-2-yl)dihydroborate (4m) and (2,2',4,4'-tetrafluoro-[1,1'-biphenyl]-3,3'-diyl)bis((1,3-dimethyl-1*H*-imidazol-3-ium-2-yl)dihydroborate) (4m').

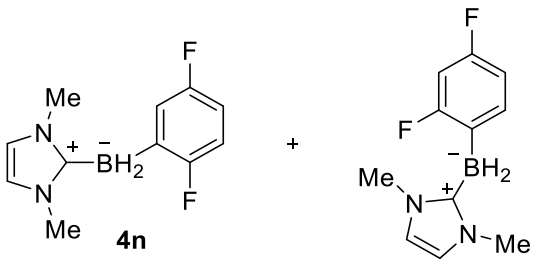
1,2,3-Trifluorobenzene (**1m**) (52.8 mg, 0.40 mmol) was used according to GP2.
Reaction time: 12 h.
Eluent in chromatography: *n*-hexane/EtOAc, 70:30 then 50:50.

The first elution gave **4m**.
45.4 mg, 0.205 mmol, 51% yield.
White solid, mp: 67.3–89.3 °C (dec.).

^{1}H NMR (600 MHz, $CDCl_3$): δ 6.96–7.01 (m, 1H), 6.82 (s, 2H), 6.65–6.70 (m, 2H), 3.76 (s, 6H), 2.23 (br-q, $J_{H–B}$ = 89 Hz, 2H).
^{13}C NMR (100 MHz, $CDCl_3$): δ 166.5 (dd, $J_{C–F}$ = 238, 16.6 Hz), 125.9 (t, $J_{C–F}$ = 10.9 Hz), 120.3, 109.7–110.0 (m), 35.9.
^{11}B NMR (192 MHz, $CDCl_3$): δ –32.53 (t, $J_{B–H}$ = 89 Hz).
^{19}F NMR (376 MHz, $CDCl_3$): δ –106.22 (s, 2F).
IR ($CHCl_3$, cm^{-1}): ν_{max} 2343, 1540, 1484, 1444, 1398, 1232.
HRMS (DART+): calcd for $C_{11}H_{12}BF_2N_2$ [M–H] 221.1062, found 221.1067.

The second elution gave **4m'**.
30.6 mg, 0.0691 mmol (0.138 mmol for a monomer), 35% yield.
Off-white solid, mp: 211.6–216.9 °C.
The crystal for X-ray crystallography was grown from a toluene/$CHCl_3$ solution.

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.95–7.01 (m, 1H), 6.79 (s, 2H), 6.71 (t, J = 8.0 Hz, 1H), 3.75 (s, 6H), 2.25 (br-q, $J_{H–B}$ = 87 Hz, 2H).
^{13}C NMR (100 MHz, $CDCl_3$): δ 165.8 (dd, $J_{C–F}$ = 236, 15.2 Hz), 162.9 (dd, $J_{C–F}$ = 243, 18,8 Hz), 128.0 (d, $J_{C–F}$ = 10.1 Hz), 120.25, 119.1–119.5 (m), 109.5 (d, $J_{C–F}$ = 27.4 Hz), 36.0.
^{11}B NMR (128 MHz, $CDCl_3$): δ –32.33 (t, $J_{B–H}$ = 87 Hz).
^{19}F NMR (376 MHz, $CDCl_3$): δ –109.86 (s, 2F), –110.23 (s, 2F).
IR ($CHCl_3$, cm^{-1}): ν_{max} 2349, 1597, 1576, 1483, 1448, 1407, 1234.
HRMS (DART+): calcd for $C_{22}H_{23}B_2F_4N_4$ [M–H] 441.2045, found 441.2036.



(inseparable, 75/25)

(2,5-Difluorophenyl)(1,3-dimethyl-1*H*-imidazol-3-ium-2-yl)dihydroborate (4n) and (2,4-difluorophenyl)(1,3-dimethyl-1*H*-imidazol-3-ium-2-yl)dihydroborate (4n').

1,2,5-Trifluorobenzene (**1n**) (52.8 mg, 0.40 mmol) was used according to GP2.

Reaction time: 24 h.

Eluent in chromatography: *n*-hexane/EtOAc, 70:30.

28.4 mg, 0.129 mmol, 32% yield (**4n**/**4n'**, 75/25).

Colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 (br, 1H, for **4n'**), 7.01 (br, 1H, for **4n**), 6.83 (s, 2H, for **4n**), 6.81 (s, 2H, for **4n'**), 6.62–6.73 (m, 2H for **4n** and 1H for **4n'**), 6.54 (td, J = 9.4, 2.3 Hz, 1H for **4n'**), 3.735 (s, 6H, for **4n**), 3.729 (s, 6H, for **4n'**), 2.26 (br-q, $J_{H–B}$ = 87 Hz, 2H for **4n** and 2H for **4n'**).

^{13}C NMR (100 MHz, $CDCl_3$): δ 165.8 (dd, $J_{C–F}$ = 238, 10.9 Hz, for **4n'**), 162.0 (d, $J_{C–F}$ = 230 Hz, for **4n**), 161.2 (dd, $J_{C–F}$ = 241, 13.0 Hz, for **4n'**), 158.7 (d, $J_{C–F}$ = 241 Hz, for **4n**), 137.9 (dd, $J_{C–F}$ = 14.2, 8.4 Hz, for **4n'**), 122.8 (dd, $J_{C–F}$ = 19.6, 13.1 Hz, for **4n**),120.3 (for **4n**), 120.3 (for **4n'**), 114.1 (dd, $J_{C–F}$ = 30.5, 8.0 Hz, for **4n**), 111.6 (dd, $J_{C–F}$ = 24.3, 9.1 Hz, for **4n**), 109.9 (d, $J_{C–F}$ = 18.2 Hz, for **4n'**), 101.8 (dd, $J_{C–F}$ = 31.2, 24.0 Hz, for **4n'**), 36.0 (for **4n** and **4n'**).

^{11}B NMR (128 MHz, $CDCl_3$): δ –27.98 (t, $J_{B–H}$ = 87 Hz, for **4n**), –28.13 (t, $J_{B–H}$ = 86 Hz, for **4n'**).

^{19}F NMR (376 MHz, $CDCl_3$): δ –106.39 (s, 1F, for **4n'**), –117.13 (s, 1F, for **4n**), –121.08 - –120.99 (m, 1F, for **4n'**), –126.20 - – 126.09 (m, 1F, for **4n**).

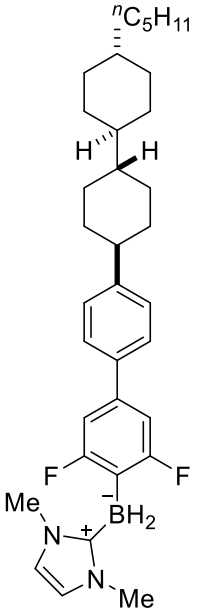
IR ($CHCl_3$, cm^{-1}): ν_{max} 2322, 1466, 1232, 1169.

HRMS (DART+): calcd for $C_{11}H_{17}BF_2N_3$ [M+NH_4] 240.1484, found 240.1476.

Unsuccessful reactions of 1o and 1p:

After 1,3,5-trifluorobenzene (**1o**) (52.8 mg, 0.40 mmol) and difluorobenzene (**1p**) (45.6 mg, 0.40 mmol) were reacted for 24 h according to GP1, respectively, ^{1}H and ^{11}B NMR analyses of the crude product were performed, and a very small amount of the corresponding borylated product was detected in both cases.

Synthesis of liquid crystalline compounds:



14a

(1,3-Dimethyl-1*H*-imidazol-3-ium-2-yl)(3,5-difluoro-4'-((*trans*, *trans*)-4'-pentyl-[1,1'-bi(cyclohexan)]-4-yl)-[1,1'-biphenyl]-4-yl)dihydroborate (14a).

12a (177.0 mg, 0.40 mmol) was used according to GP2.

Reaction time: 24 h.

Eluent in chromatography: *n*-hexane/EtOAc, 70:30.

63.0 mg, 0.118 mmol, 30% yield, white solid. [A very small amount of a regioisomer (tentative) was detected in NMR spectra (**14a**/regioisomer, >95/5)]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45 (app. d, J = 8.0 Hz, 2H), 7.23 (app. d, J = 8.4 Hz, 2H), 6.89–6.96 (m, 2H), 6.82 (s, 2H), 3.78 (s, 6H), 0.82–2.58 (m, 33H).

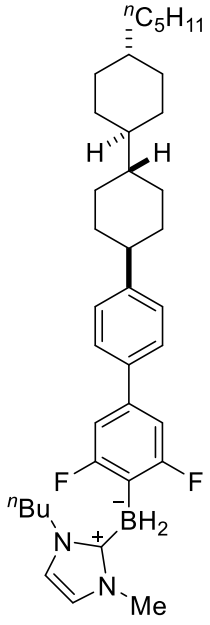
^{13}C NMR (100 MHz, $CDCl_3$): δ 166.8 (dd, $J_{C–F}$ = 236, 18.0 Hz), 147.2, 139.6 (t, $J_{C–F}$ = 10.1 Hz), 137.4, 127.2, 126.5, 120.3, 108.1–108.4 (m), 44.3, 43.5, 42.9, 37.9, 37.5, 36.0, 34.9, 33.7, 32.2, 30.4, 30.1, 26.7, 22.7, 14.1.

^{11}B NMR (128 MHz, $CDCl_3$): δ –32.47 (t, $J_{B–H}$ = 87 Hz). [regioisomer (tentative): –27.94 (t, $J_{B–H}$ = 85 Hz)]

^{19}F NMR (376 MHz, $CDCl_3$): δ –109.55 (s, 2F).

IR ($CHCl_3$, cm^{-1}): ν_{max} 2929, 2347, 1461, 1394, 1282, 1171.

HRMS (DART+): calcd for $C_{34}H_{46}BF_2N_2$ [M–H] 531.3722, found 531.3729.



15a

(1-Butyl-3-methyl-1*H*-imidazol-3-ium-2-yl)(3,5-difluoro-4'-((*trans, trans*)-4'-pentyl-[1,1'-bi(cyclohexan)]-4-yl)-[1,1'-biphenyl]-4-yl)dihydroborate (15a).

12a (177.0 mg, 0.40 mmol) and NHC-borane **13** (182.6 mg, 1.20 mmol) were used according to GP2. Reaction time: 24 h.

Eluent in chromatography: *n*-hexane/EtOAc, 70:30.

147.9 mg, 0.257 mmol, 64% yield, white solid. [A very small amount of a regioisomer (tentative) was detected in NMR spectra (**15a**/regioisomer, >95/5)]

DSC and POM experiments were performed using colorless crystals by recrystallization from a *n*-hexane/ethyl acetate solution.

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 (app. d, J = 8.3 Hz, 2H), 7.23 (app. d, J = 8.3 Hz, 2H), 6.89–6.95 (m, 2H), 6.85 (d, J = 2.0 Hz, 1H), 6.83 (d, J = 2.0 Hz, 1H), 4.15 (t, J = 7.8 Hz, 2H), 3.77 (s, 3H), 0.82–2.58 (m, 40H).

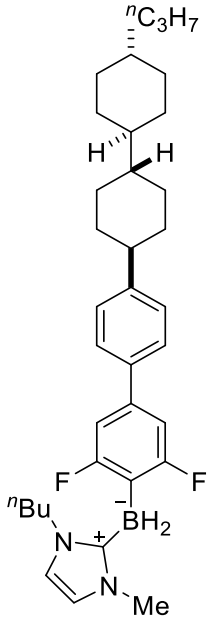
^{13}C NMR (100 MHz, $CDCl_3$): δ 166.7 (dd, $J_{C–F}$ = 236, 18.1 Hz), 147.2, 139.5 (t, $J_{C–F}$ = 10.2 Hz), 137.4, 127.2, 126.5, 120.4, 108.1–118.9 (m), 48.5, 44.3, 43.4, 42.9, 37.9, 37.5, 35.9, 34.6, 33.7, 32.4, 32.2, 30.4, 30.1, 26.7, 22.7, 19.7, 14.1, 13.6.

^{11}B NMR (128 MHz, $CDCl_3$): δ –32.36 (t, $J_{B–H}$ = 86 Hz). [regioisomer (tentative): –27.92 (t, $J_{B–H}$ = 85 Hz)]

^{19}F NMR (376 MHz, $CDCl_3$): δ –106.43 (s, 2F)

IR ($CHCl_3$, cm^{-1}): ν_{max} 2925, 2348, 1617, 1540, 1460, 1394.

HRMS (DART+): calcd for $C_{37}H_{52}BF_2N_2$ [M–H] 573.4192, found 573.4198.



15b

(1-Butyl-3-methyl-1*H*-imidazol-3-ium-2-yl)(3,5-difluoro-4'-((*trans, trans*)-4'-propyl-[1,1'-bi(cyclohexan)]-4-yl)-[1,1'-biphenyl]-4-yl)dihydroborate (15b).

12b (165.8 mg, 0.40 mmol) and NHC-borane **13** (182.6 mg, 1.20 mmol) were used according to GP2. Reaction time: 24 h.

Eluent in chromatography: *n*-hexane/EtOAc, 70:30.

120.0 mg, 0.220 mmol, 55% yield, white solid. [A very small amount of a regioisomer (tentative) was detected in NMR spectra (**15b**/regioisomer, >95/5)]

DSC and POM experiments were performed using colorless crystals by recrystallization from a *n*-hexane/ethyl acetate solution.

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 (d, J = 8.3 Hz, 2H), 7.23 (d, J = 8.3 Hz, 2H), 6.89–6.95 (m, 2H), 6.85 (d, J = 2.0 Hz, 1H), 6.83 (d, J = 2.0 Hz, 1H), 4.15 (t, J = 7.6 Hz, 2H), 3.77 (s, 3H), 0.82–2.50 (m, 36H).

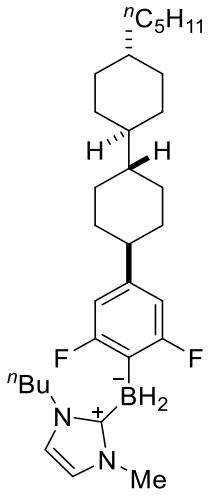
^{13}C NMR (100 MHz, $CDCl_3$): δ 168.0 (dd, $J_{C–F}$ = 236, 17.3 Hz), 147.2, 139.5 (t, $J_{C–F}$ = 10.1 Hz), 137.4, 127.2, 126.5, 120.4, 118.9, 108.1–108.4 (m), 48.5, 44.3, 43.4, 42.9, 39.8, 37.6, 35.9, 34.6, 33.6, 32.4, 30.4, 30.1, 20.0, 19.7, 14.4, 13.6.

^{11}B NMR (128 MHz, $CDCl_3$): δ –32.39 (t, $J_{B–H}$ = 87 Hz). [regioisomer (tentative): –27.93 (t, $J_{B–H}$ = 87 Hz)]

^{19}F NMR (376 MHz, $CDCl_3$): δ –106.44 (s, 2F).

IR ($CHCl_3$, cm^{-1}): ν_{max} 2924, 2348, 1617, 1541, 1477, 1394.

HRMS (DART+): calcd for $C_{35}H_{48}BF_2N_2$ [M–H] 545.3879, found 545.3865.



17a

(1-Butyl-3-methyl-1*H*-imidazol-3-ium-2-yl)(2,6-difluoro-4-((*trans*, *trans*)-4'-pentyl-[1,1'-bi(cyclohexan)]-4-yl)phenyl)dihydroborate (17a).

16a (146.6 mg, 0.40 mmol) and NHC-borane **13** (182.6 mg, 1.20 mmol) were used according to GP2. Reaction time: 24 h.

Eluent in chromatography: *n*-hexane/EtOAc, 70:30.

97.1 mg, 0.195 mmol, 49% yield, white solid. [A very small amount of a regioisomer (tentative) was detected in NMR spectra (**17a**/regioisomer, 95/5)]

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.82 (d, J = 2.0 Hz, 1H), 6.80 (d, J = 2.0 Hz, 1H), 6.49–6.55 (m, 2H), 4.12 (t, J = 7.8 Hz, 2H), 3.74 (s, 3H), 0.80–2.52 (m, 40H).

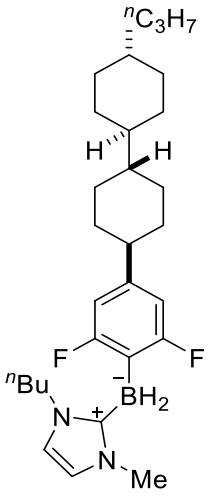
^{13}C NMR (100 MHz, $CDCl_3$): δ 166.3 (dd, $J_{C–F}$ = 236, 17.4 Hz), 146.8 (t, $J_{C–F}$ = 9.4 Hz), 120.3, 108.0–118.7 (m), 48.4, 43.9, 43.4, 42.9, 37.9, 37.5, 35.9, 34.4, 33.6, 32.4, 32.2, 30.3, 30.1, 26.7, 22.7, 19.7, 14.1, 13.6.

^{11}B NMR (128 MHz, $CDCl_3$): δ –32.45 (t, $J_{B–H}$ = 85 Hz). [regioisomer (tentative): –27.93 (t, $J_{B–H}$ = 86 Hz)]

^{19}F NMR (376 MHz, $CDCl_3$): δ –107.14 (s, 2F).

IR ($CHCl_3$, cm^{-1}): ν_{max} 2924, 2347, 1620, 1551, 1449, 1412.

HRMS (DART+): calcd for $C_{31}H_{48}BF_2N_2$ [M–H] 497.3879, found 497.3874.



17b

(1-Butyl-3-methyl-1*H*-imidazol-3-ium-2-yl)(2,6-difluoro-4-((*trans*, *trans*)-4'-propyl-[1,1'-bi(cyclohexan)]-4-yl)phenyl)dihydroborate (17b).

16b (135.4 mg, 0.40 mmol) and NHC-borane **13** (182.6 mg, 1.20 mmol) were used according to GP2. Reaction time: 24 h.

Eluent in chromatography: *n*-hexane/EtOAc, 70:30.

93.3 mg, 0.198 mmol, 50% yield, white solid. [A very small amount of a regioisomer (tentative) was detected in NMR spectra (**17b**/regioisomer, 93/7)]

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.82 (d, J = 2.0 Hz, 1H), 6.80 (d, J = 2.0 Hz, 1H), 6.49–6.55 (m, 2H), 4.11 (t, J = 7.8 Hz, 2H), 3.74 (s, 3H), 0.81–2.52 (m, 36H).

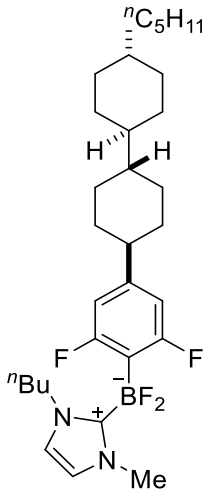
^{13}C NMR (100 MHz, $CDCl_3$): δ 166.3 (dd, $J_{C–F}$ = 236, 17.4 Hz), 146.8 (t, $J_{C–F}$ = 9.4 Hz), 120.3, 118.7, 108.0–108.4 (m), 48.4, 43.9, 43.4, 42.9, 39.8, 37.6, 35.9, 34.4, 33.6, 32.4, 30.3, 30.1, 20.0, 19.7, 14.4, 13.6.

^{11}B NMR (128 MHz, $CDCl_3$): δ –32.45 (t, $J_{B–H}$ = 86 Hz). [regioisomer (tentative): –27.92 (t, $J_{B–H}$ = 86 Hz)]

^{19}F NMR (376 MHz, $CDCl_3$): δ –107.13 (s, 2F).

IR ($CHCl_3$, cm^{-1}): ν_{max} 2924, 2347, 1620, 1550, 1449, 1412.

HRMS (DART+): calcd for $C_{29}H_{44}BF_2N_2$ [M–H] 469.3566, found 469.3571.



18a

(1-Butyl-3-methyl-1*H*-imidazol-3-ium-2-yl)(2,6-difluoro-4-((*trans*, *trans*)-4'-pentyl-[1,1'-bi(cyclohexan)]-4-yl)phenyl)difluoroborate (18a).

Selectfluor® (85.0 mg, 0.24 mmol) was added to a solution of **17a** (39.9 mg, 0.08 mmol) in acetonitrile (1.6 mL) at 0 °C, and the mixture was stirred for 0.5 h at 0 °C and for 3.5 h at room temperature. The solvent was evaporated, and the residue was purified by silica gel chromatography (eluent: *n*-hexane/EtOAc, 80:20) to give **18a** (13.3 mg, 0.0249 mmol, 31% yield) as a white solid. DSC and POM experiments were performed using colorless crystals by recrystallization from a *n*-hexane/ethyl acetate solution.

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.88 (d, J = 1.8 Hz, 1H), 6.85 (d, J = 1.8 Hz, 1H), 6.53-6.59 (m, 2H), 4.21 (t, J = 7.8 Hz, 2H), 3.88 (s, 3H), 2.35 (tt, J = 12.0, 3.2 Hz, 1H), 1.61–1.88 (m, 10H), 0.81–1.35 (m, 27H).

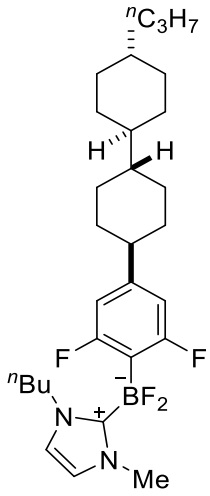
^{13}C NMR (100 MHz, $CDCl_3$): δ 166.0 (dd, $J_{C–F}$ = 240, 15.9 Hz), 150.1 (t, $J_{C–F}$ = 8.7 Hz), 121.8, 119.9, 109.0–109.3 (m), 48.9, 44.1, 43.4, 42.8, 37.9, 37.5, 35.4, 34.2, 33.6, 32.9, 32.2, 30.2, 30.1, 26.7, 22.7, 19.7, 14.1, 13.6.

^{11}B NMR (128 MHz, $CDCl_3$): δ 3.23 (br).

^{19}F NMR (376 MHz, $CDCl_3$): δ –110.73 - –110.66 (m, 2F), –156.13 (br, 2F).

IR ($CHCl_3$, cm^{-1}): ν_{max} 2925, 1626, 1554, 1449, 1415.

HRMS (DART+): calcd for $C_{31}H_{47}BF_3N_2$ [M–F] 515.3784, found 515.3774.



18b

(1-Butyl-3-methyl-1*H*-imidazol-3-ium-2-yl)(2,6-difluoro-4-((*trans*, *trans*)-4'-propyl-[1,1'-bi(cyclohexan)]-4-yl)phenyl)difluoroborate (18b).

Selectfluor® (53.1 mg, 0.15 mmol) was added to a solution of **17b** (23.5 mg, 0.05 mmol) in acetonitrile (1.0 mL) at 0 °C, and the mixture was stirred for 0.5 h at 0 °C and for 24 h at room temperature. The solvent was evaporated, and the residue was purified by silica gel chromatography (eluent: *n*-hexane/EtOAc, 80:20) to give **18b** (13.7 mg, 0.0270 mmol, 54% yield) as a white solid. DSC and POM experiments were performed using colorless crystals by recrystallization from a *n*-hexane/ethyl acetate solution.

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.88 (d, J = 2.0 Hz, 1H), 6.85 (d, J = 2.0 Hz, 1H), 6.53–6.59 (m, 2H), 4.21 (t, J = 7.8 Hz, 2H), 3.88 (s, 3H), 2.35 (tt, J = 12.1, 3.2 Hz, 1H), 1.58–1.88 (m, 12H), 0.84–1.41 (m, 33H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 166.0 (dd, $J_{C–F}$ = 238, 18.8 Hz), 150.1 (t, $J_{C–F}$ = 10.1 Hz), 121.8, 119.9, 109.0–109.3 (m), 48.9, 44.1, 43.4, 42.9, 39.8, 37.6, 36.4, 34.2, 33.6, 32.9, 30.2, 30.1, 20.0, 19.7, 14.4, 13.4.

^{11}B NMR (128 MHz, $CDCl_3$): δ 3.25 (br).

^{19}F NMR (376 MHz, $CDCl_3$): δ –110.73 - –110.66 (m, 2F), –156.13 (br, 2F).

IR ($CHCl_3$, cm^{-1}): ν_{max} 2925, 1625, 1552, 1449, 1414.

HRMS (DART+): calcd for $C_{29}H_{43}BN_2O_2$ [M–F] 487.3471, found 487.3491.

3. Thermal analysis of liquid crystalline compounds

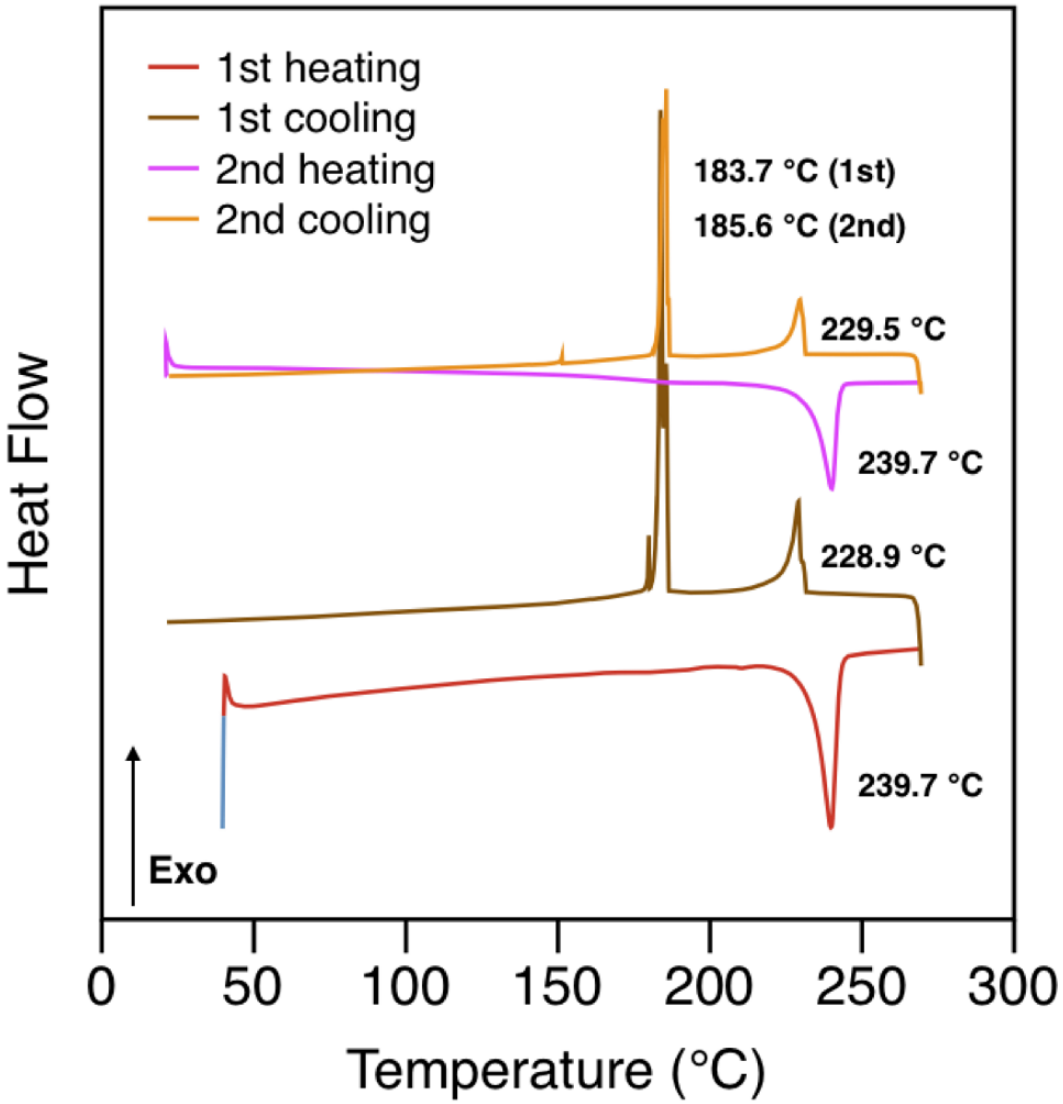


Figure S1. DSC trace of **14a** showing a monotropic mesophase upon cooling (10 °C/min).

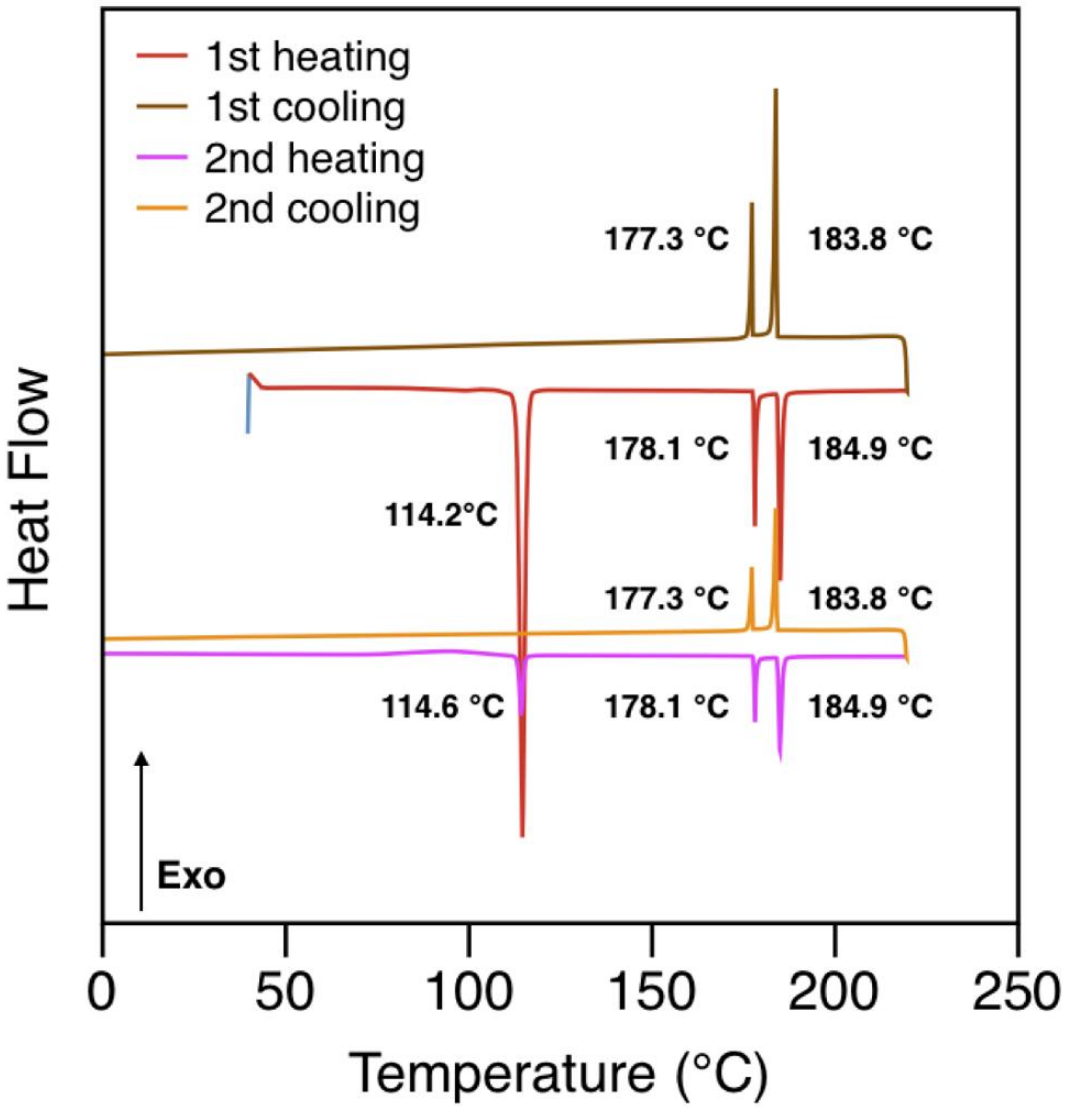


Figure S2. DSC trace of **15a** showing enantiotropic mesophases upon heating/cooling (5 °C/min).

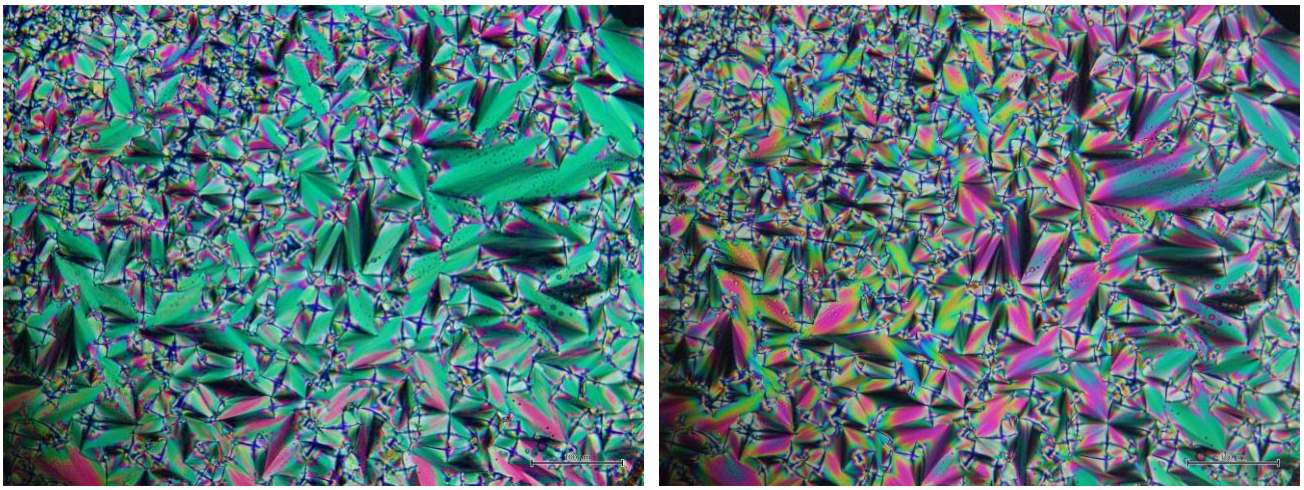


Figure S3. POM images of **15a** at 170 °C (left) and at 180 °C (right).

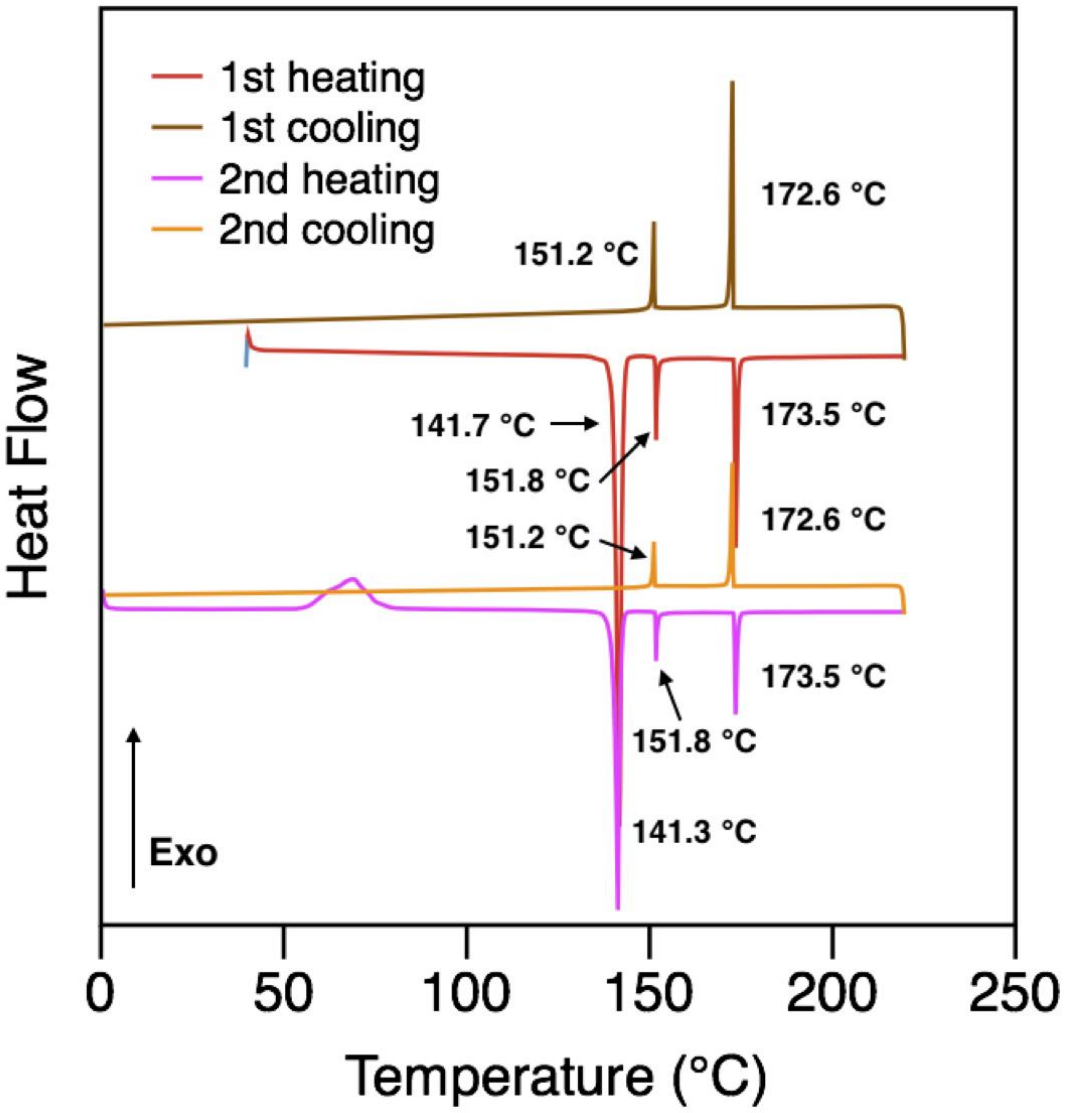


Figure S4. DSC trace of **15b** showing enantiotropic mesophases upon heating/cooling (5 °C/min).

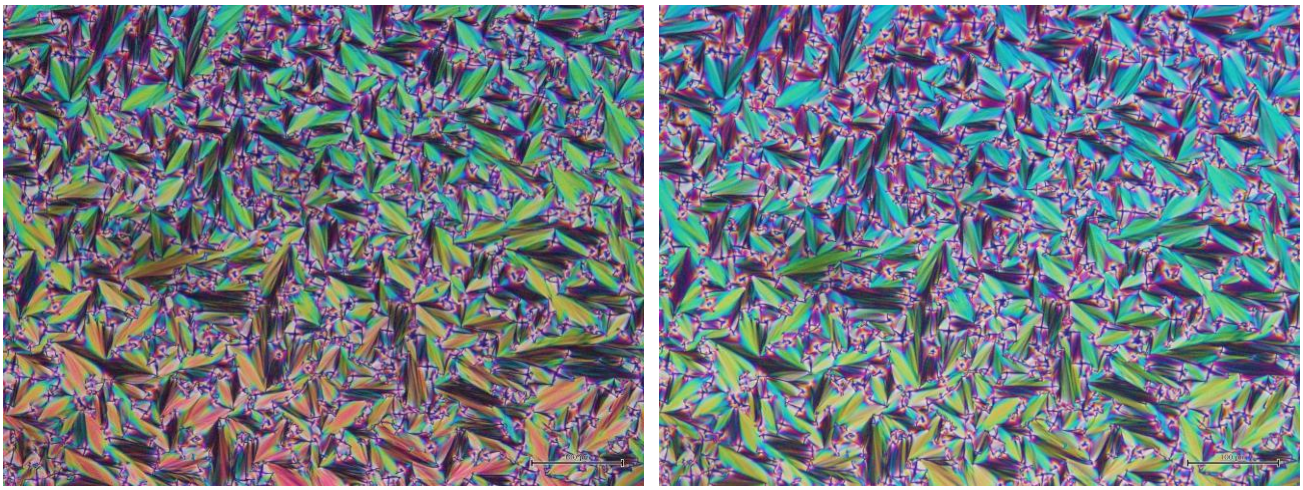


Figure S5. POM images of **15b** at 145 °C (left) and at 170 °C (right).

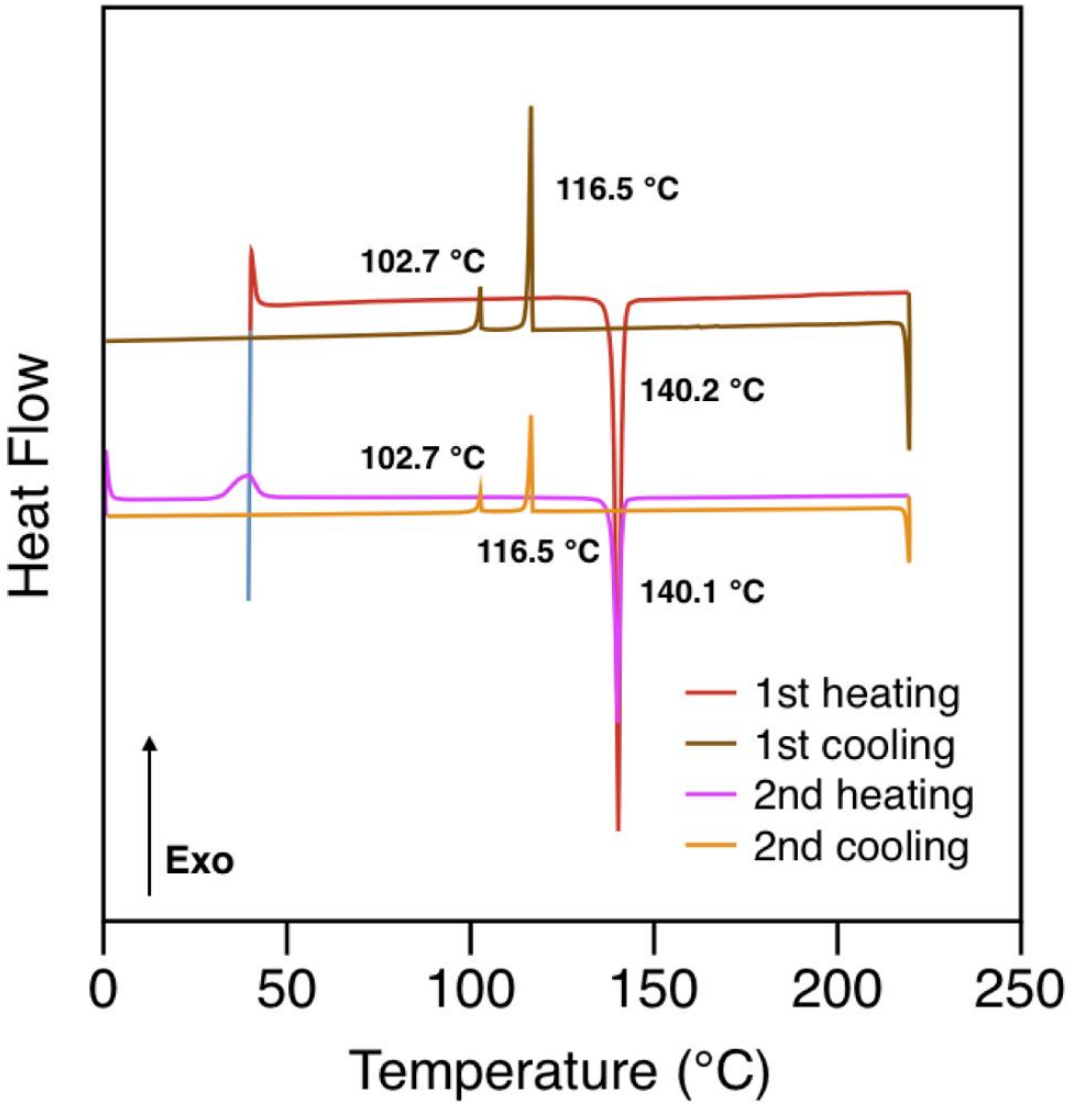


Figure S6. DSC trace of **17a** showing a monotropic mesophase upon cooling (10 °C/min).

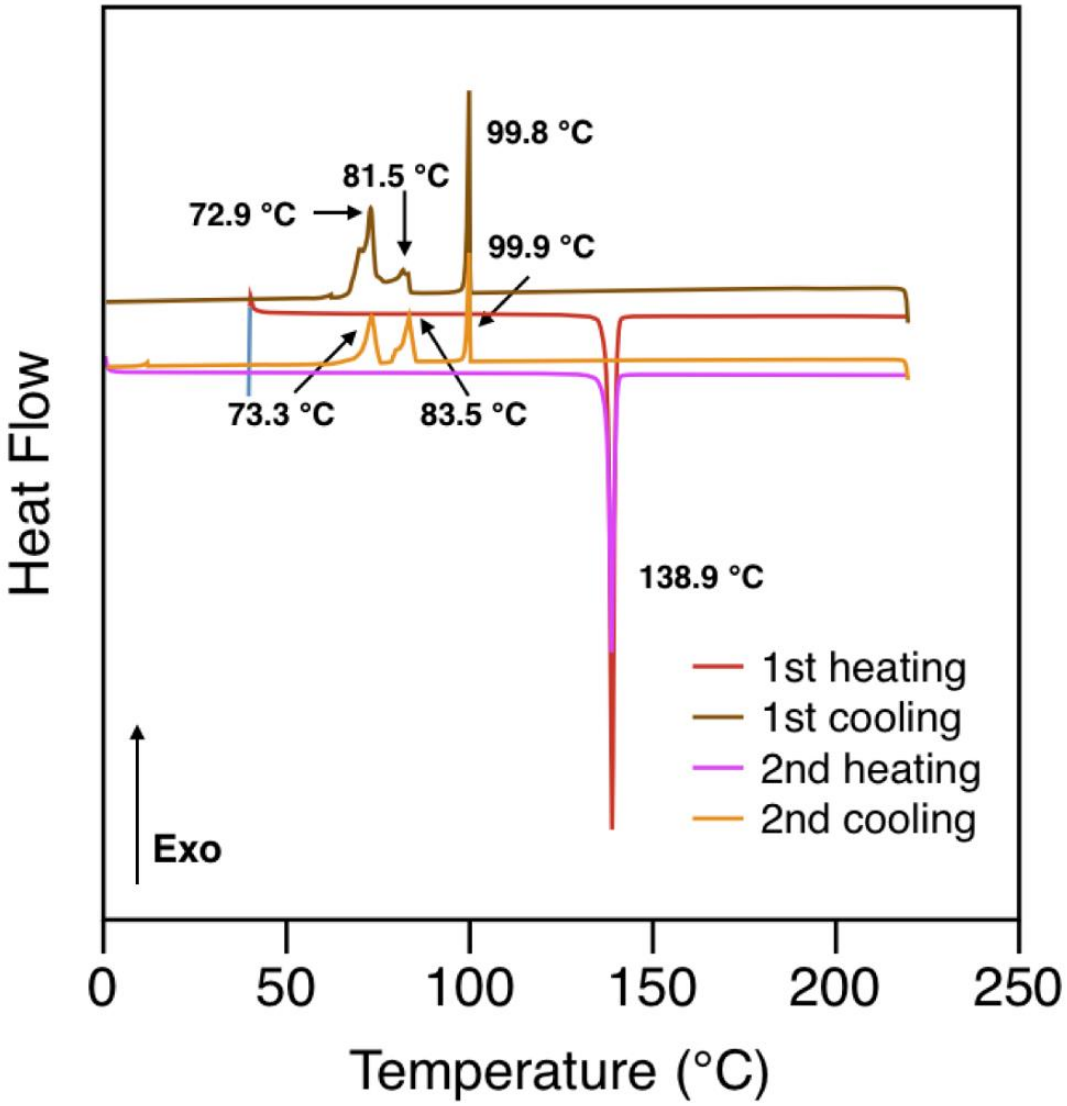


Figure S7. DSC trace of **17b** showing a monotropic mesophase upon cooling (10 °C/min).

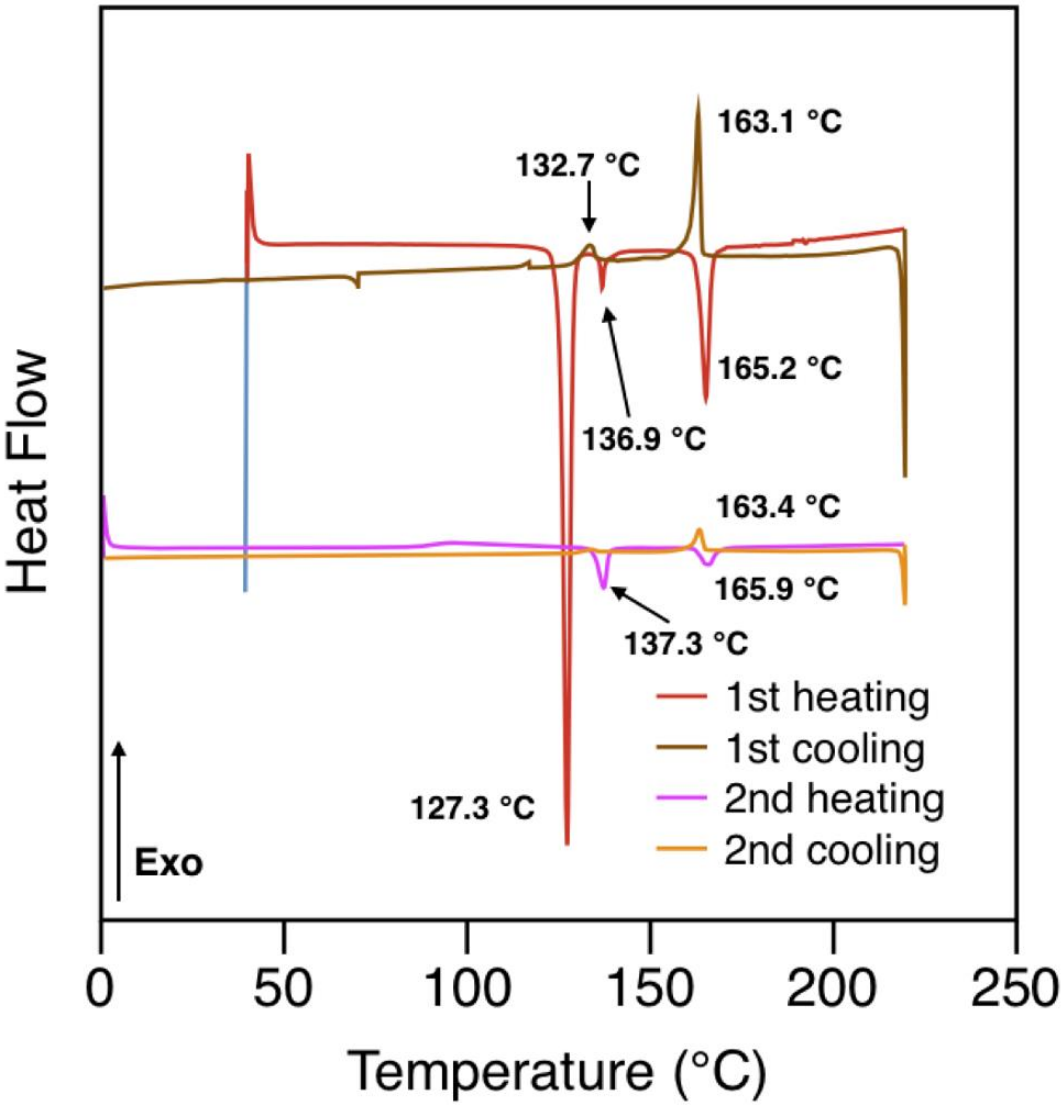


Figure S8. DSC trace of **18a** showing an enantiotropic mesophase upon heating/cooling (5 °C/min).

Figure S9. POM images of **18a** at 160 °C.

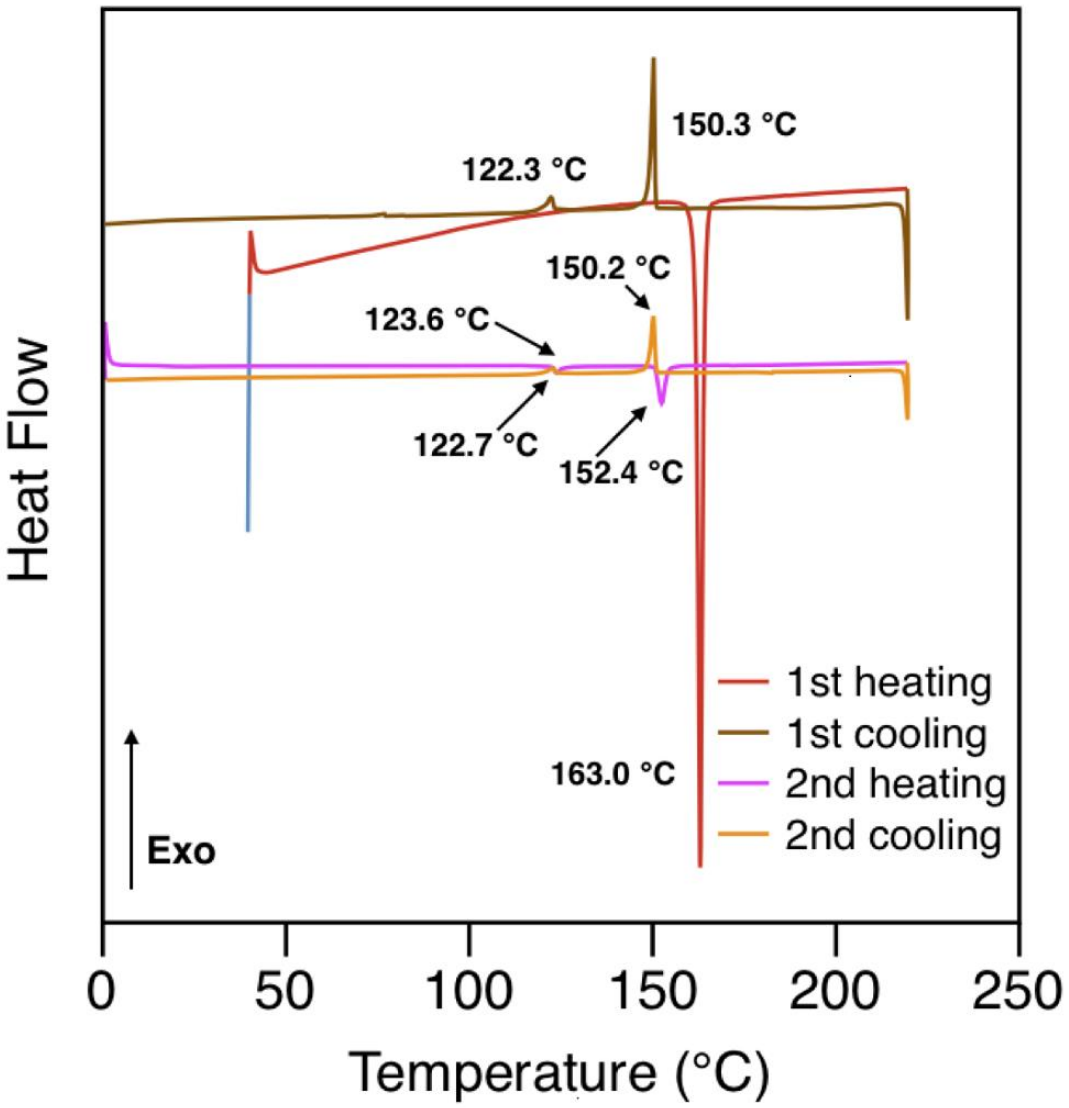


Figure S10. DSC trace of **18b** showing an enantiotropic mesophase upon heating/cooling (5 °C/min).

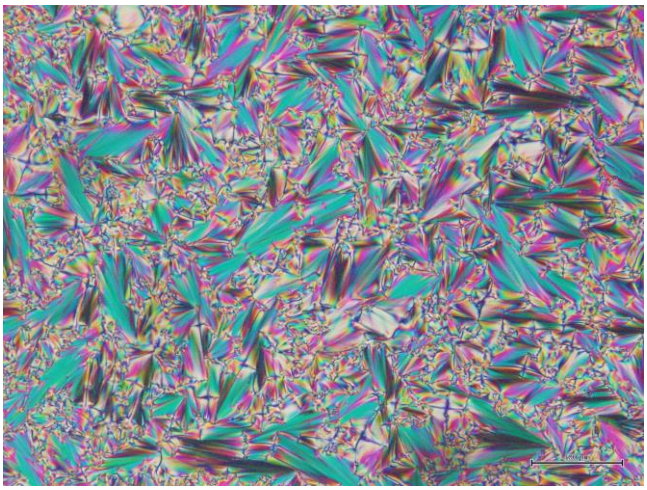
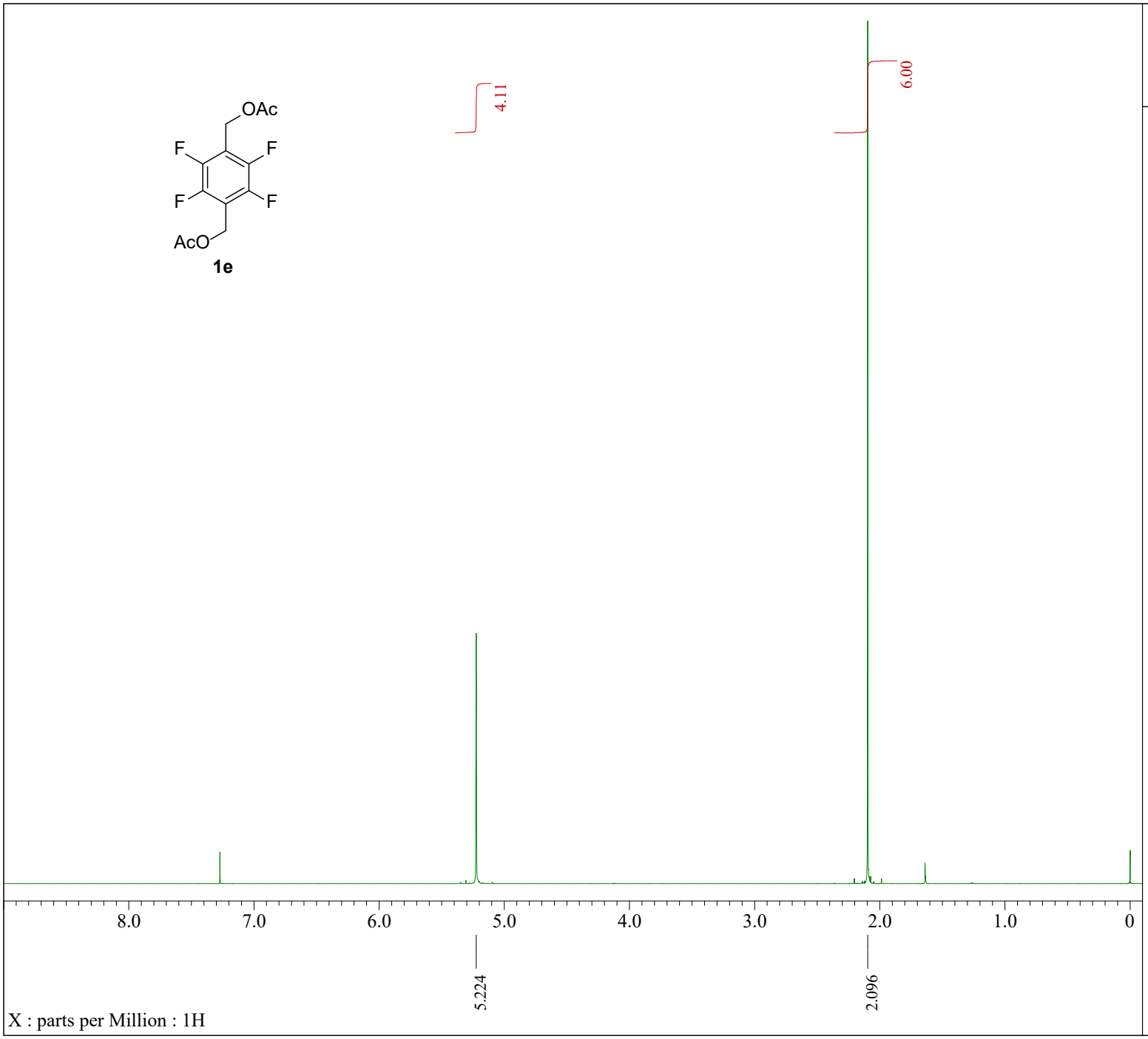


Figure S11. POM images of **18a** at 150 °C.

4. References

1. Gardner, S.; Kawamoto, T. Curran, D. P. *J. Org. Chem.* **2015**, *80*, 9794−9797.
2. Bolt, D. A.; Curran, D. P. *J. Org. Chem.* **2017**, *82*, 13746–13750.
3. Tang, Y.; Zeng, Y.; Hu, Q.; Huang, F.; Jin, L.; Mo, W.; Sun, N.; Hu, B.; Shen, Z.; Hu, X.; Sun, W.-H. *Adv. Synth. Catal.* **2016**, *358*, 2642–2651.

5. Copies of NMR spectra



JEOL Solutions for Innovation

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Author            = delta
Experiment        = single_pulse.ex2
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Creation_Time     = 18-JAN-2019 11:19:56
Revision_Time     =  6-FEB-2020 09:54:58
Current_Time      =  6-FEB-2020 09:55:03

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Dim_Title         = 1H
Dim_Units         = [ppm]
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Spectrometer      = JNM-ECA600

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X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.34366642[Hz]
X_Sweep           = 11.26126126[kHz]
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Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 2[s]
Recvr_Gain        = 40
Temp_Get          = 18.5[dC]
X_90_Width        = 13.05[us]
X_Acq_Time        = 2.9097984[s]
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X_Atn             = 2[dB]
X_Pulse           = 6.525[us]
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Dante_Presat      = FALSE
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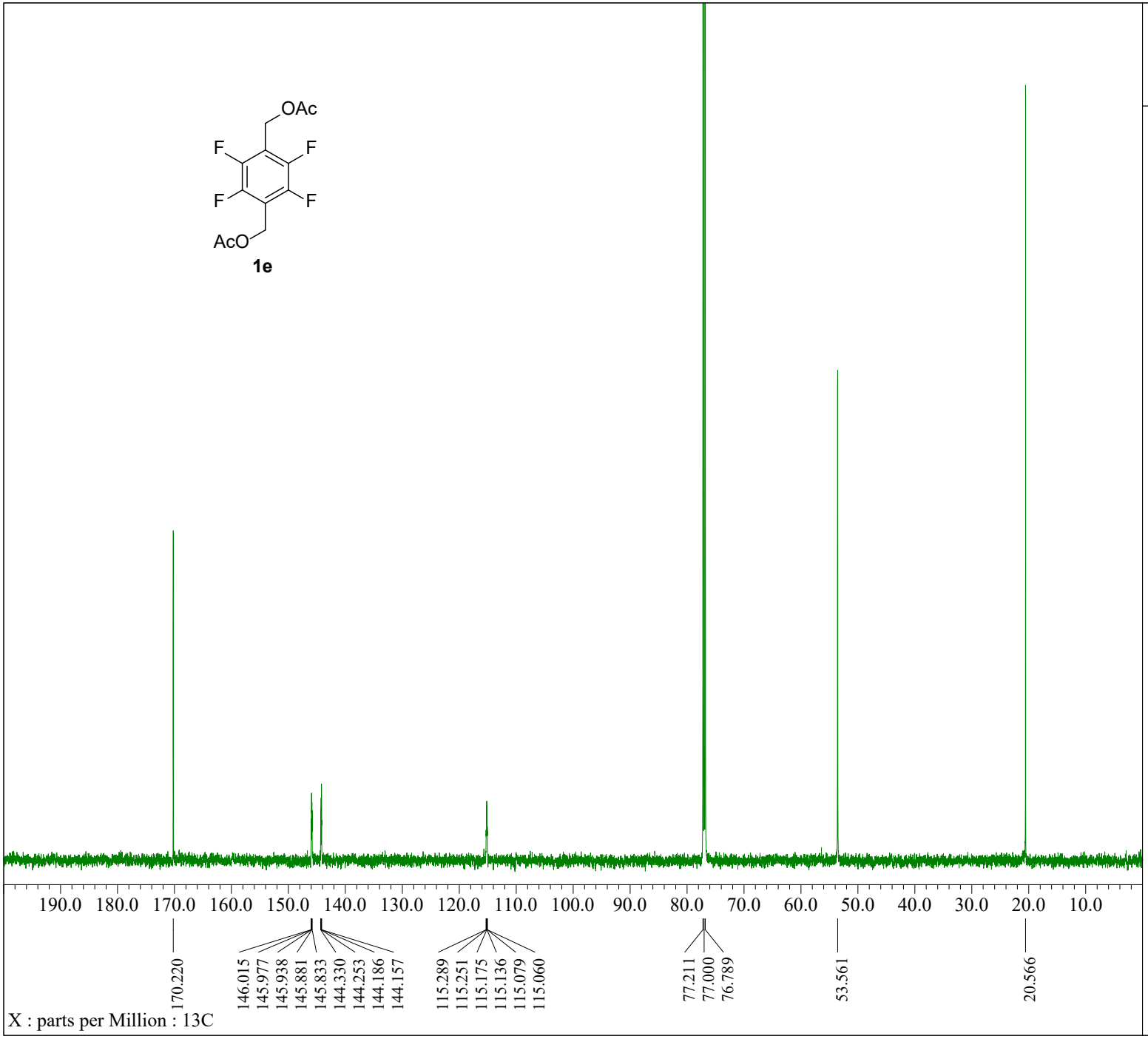

JEOL Solutions for Innovation

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Author            = delta
Experiment        = single_pulse_dec
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Solvent           = CHLOROFORM-D
Creation_Time     = 18-JAN-2019 11:32:59
Revision_Time     = 27-JAN-2020 15:08:48
Current_Time      =  6-FEB-2020 09:56:08

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Dim_Size          = 26214
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Spectrometer      = JNM-ECA600

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X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
Irr_Domain        = 1H
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 400
Total_Scans       = 400

Relaxation_Delay  = 1.2[s]
Recvr_Gain        = 56
Temp_Get          = 19.5[dC]
X_90_Width        = 10.675[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 8[dB]
X_Pulse           = 3.55833333[us]
Irr_Atn_Dec       = 17.058[dB]
Irr_Atn_Noe       = 17.058[dB]
Irr_Noise         = WALTZ
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Repetition_Time   = 1.89206016[s]
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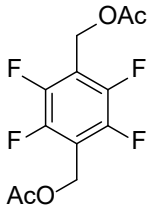

1e


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Experiment       = zg
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Revision_Time    = 27-JAN-2020 15:24:22
Current_Time     =  6-FEB-2020 09:56:56

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Spectrometer     = BRUKER_DMX_NMR

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X_Domain         = 19F
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X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
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Filter_Factor    = 220
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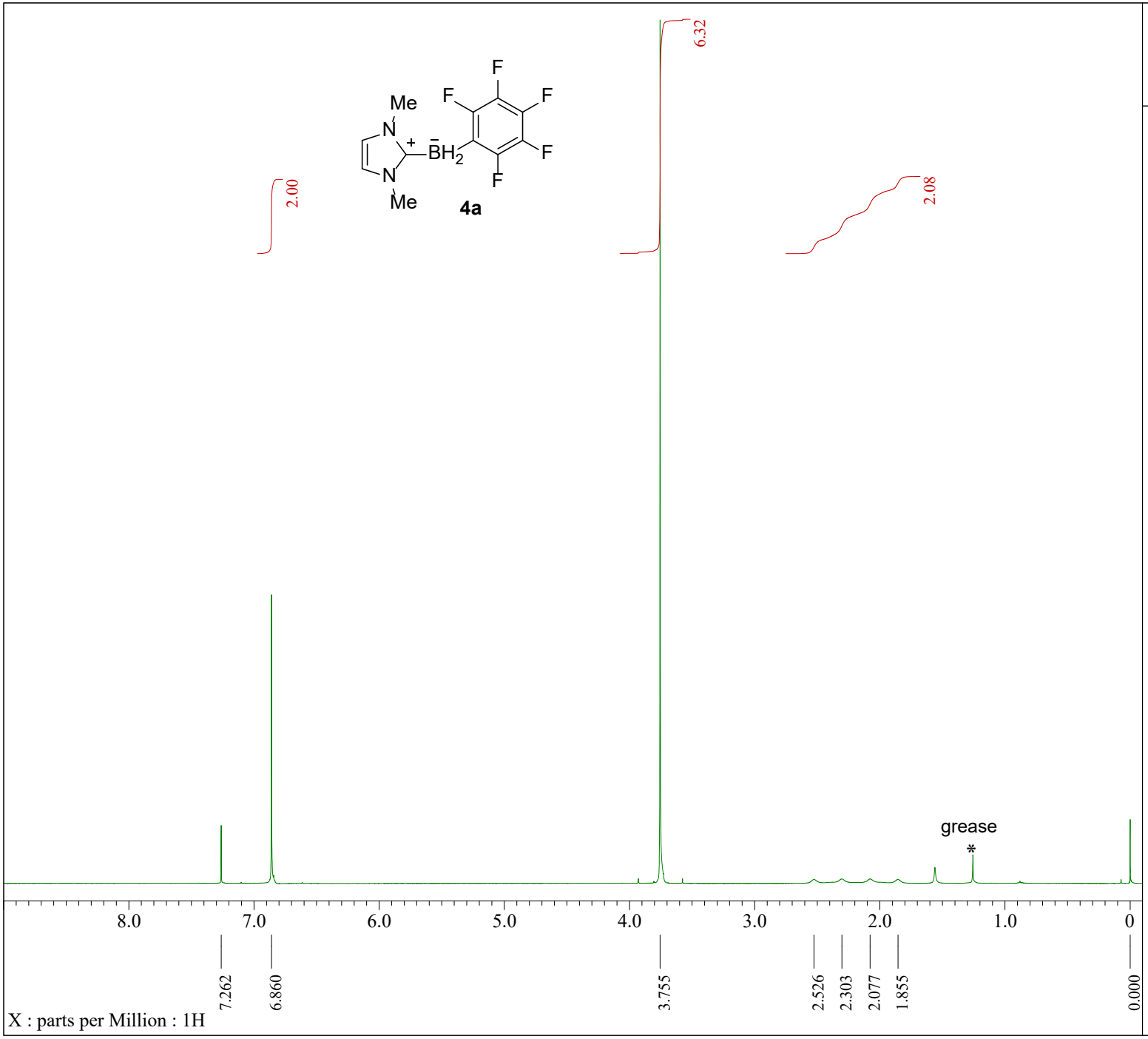

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Experiment       = zg30
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Solvent          = CHLOROFORM-D
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Revision_Time    =  5-FEB-2020 18:20:59
Current_Time     =  5-FEB-2020 18:21:47

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Dim_Size         = 32768
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Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

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X_Domain         = 1H
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X_Freq_Flip      = TRUE
X_Offset         = 2.470802[kHz]
X_Points         = 32768
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X_Sweep          = 8.19672131[kHz]
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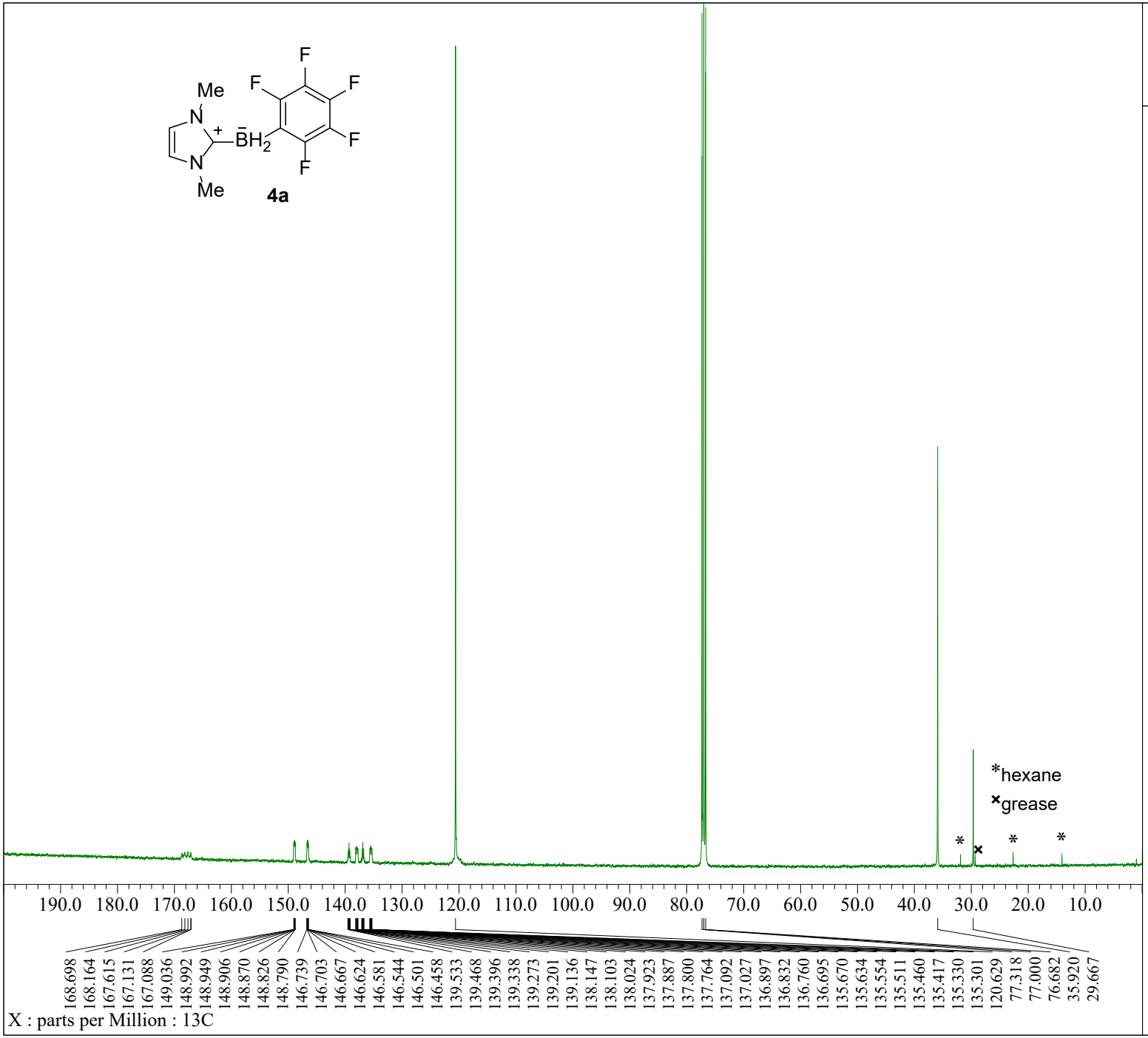

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Experiment       = zgpg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 09:54:51
Revision_Time    = 27-JAN-2020 12:05:42
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Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
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X_Freq_Flip      = TRUE
X_Offset         = 10.061277[kHz]
X_Points         = 32768
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Filter_Factor    = 840
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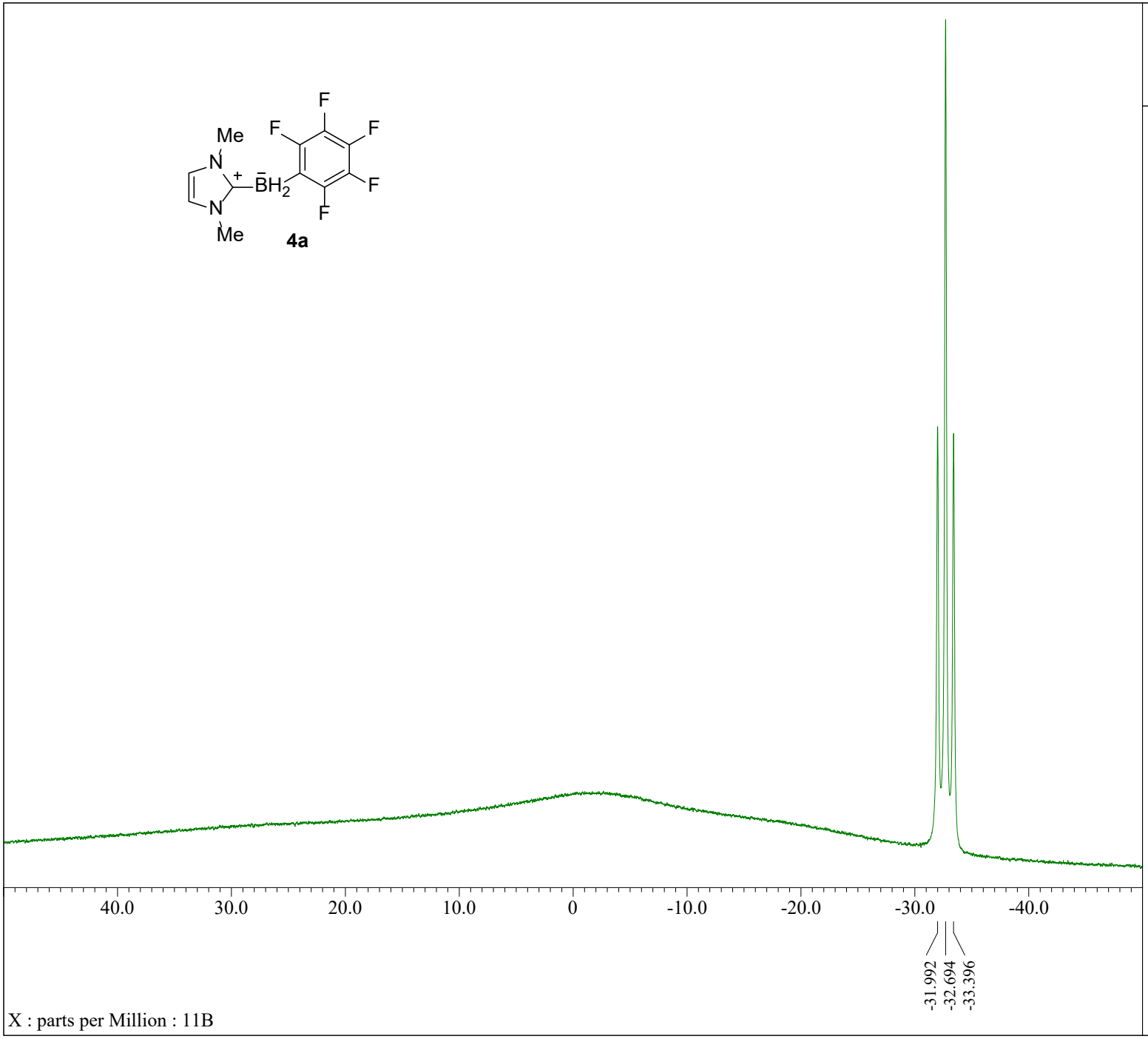

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Author           = nmrsu
Experiment       = zg
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Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 11B
X_Freq           = 128.37760517[MHz]
X_Freq_Flip      = TRUE
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X_Points         = 32768
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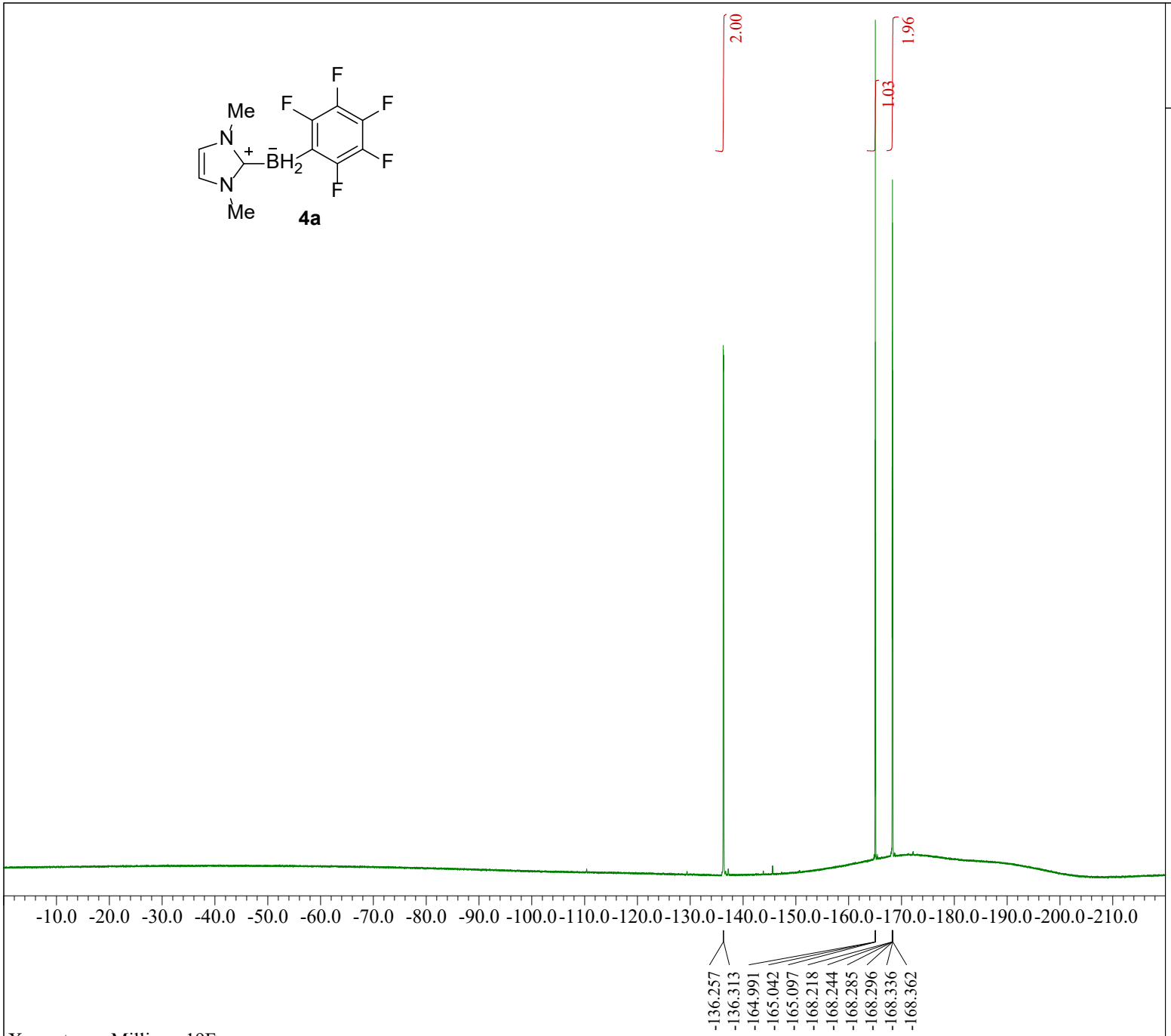

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Experiment       = zg
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Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 15:13:20
Revision_Time    =  5-FEB-2020 18:27:03
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Dim_Title        = 19F
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
X_Prescans       = 4
X_Sweep          = 90.90909091[kHz]
Scans            = 16

Temp_Get         = 301.1491[K]
Filter_Factor    = 220
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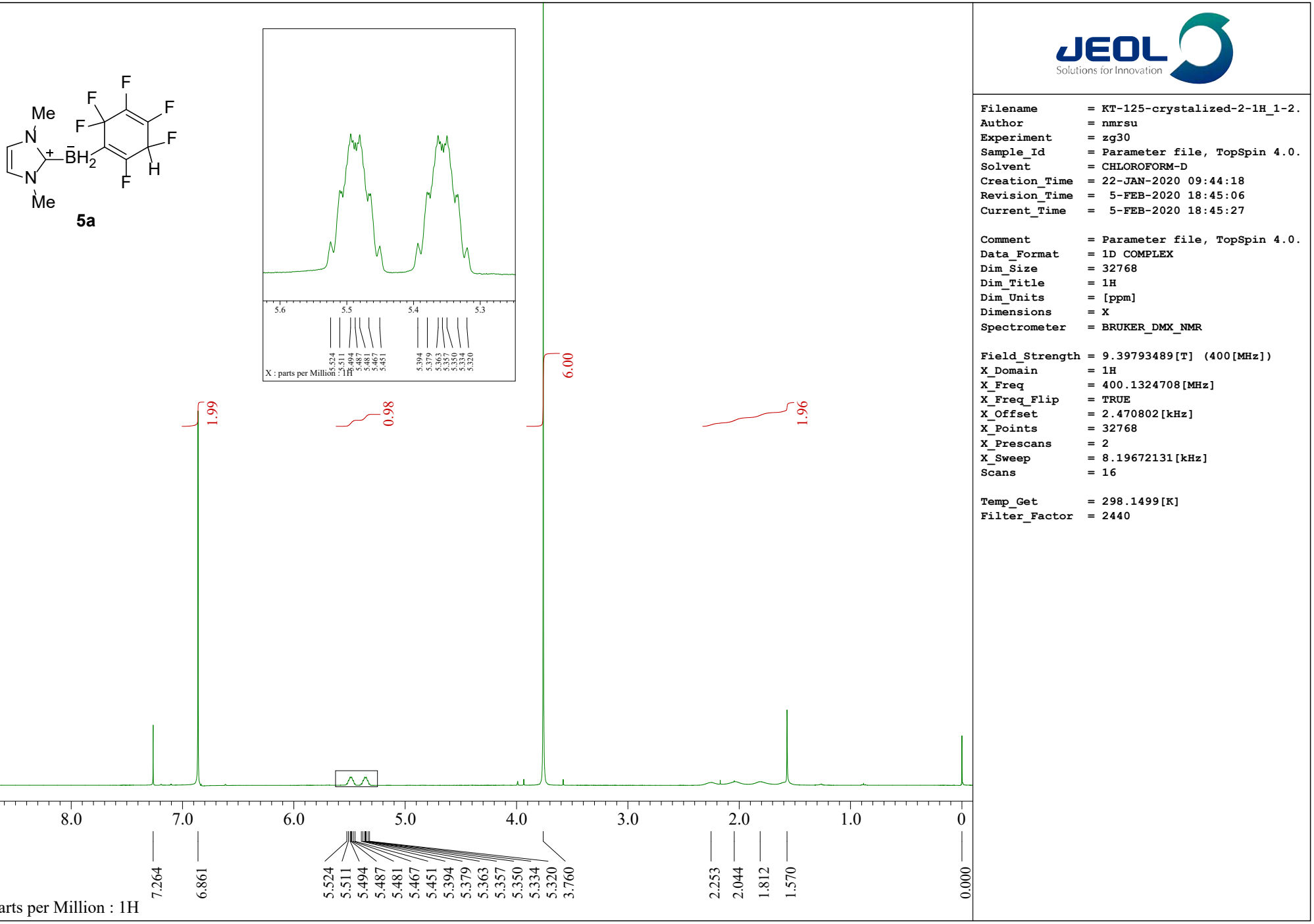

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Author          = nmrsu
Experiment      = zg30
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Revision_Time   =  5-FEB-2020 18:45:06
Current_Time    =  5-FEB-2020 18:45:27

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Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 1H
X_Freq          = 400.1324708[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 2.470802[kHz]
X_Points        = 32768
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Scans           = 16

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Filter_Factor   = 2440
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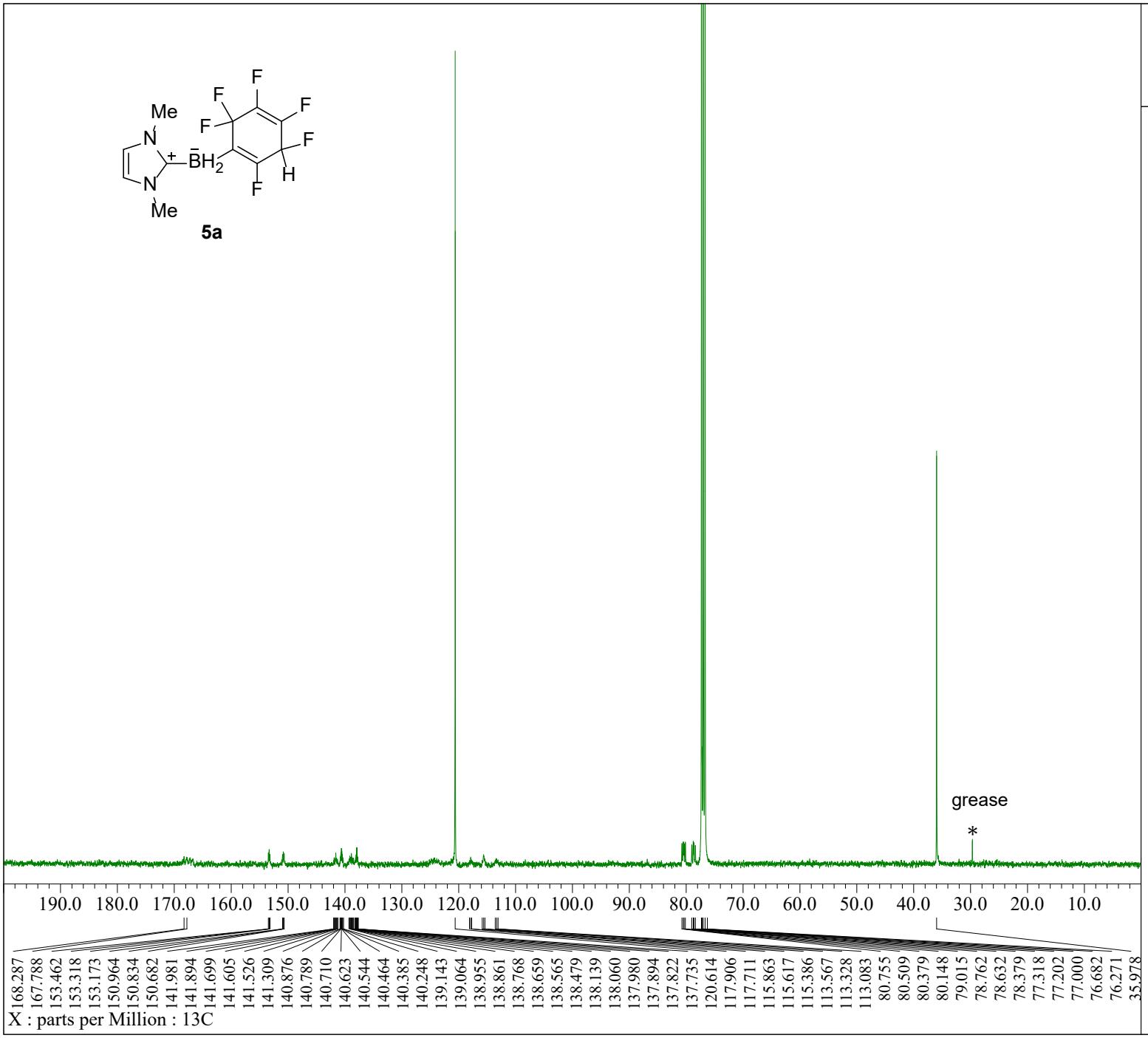

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Author          = nmrsu
Experiment      = zgpg30
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Dim_Title       = 13C
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 13C
X_Freq          = 100.6228298[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 10.061277[kHz]
X_Points        = 32768
X_Prescans      = 4
X_Sweep         = 23.80952381[kHz]
Scans           = 12958

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Filter_Factor   = 840
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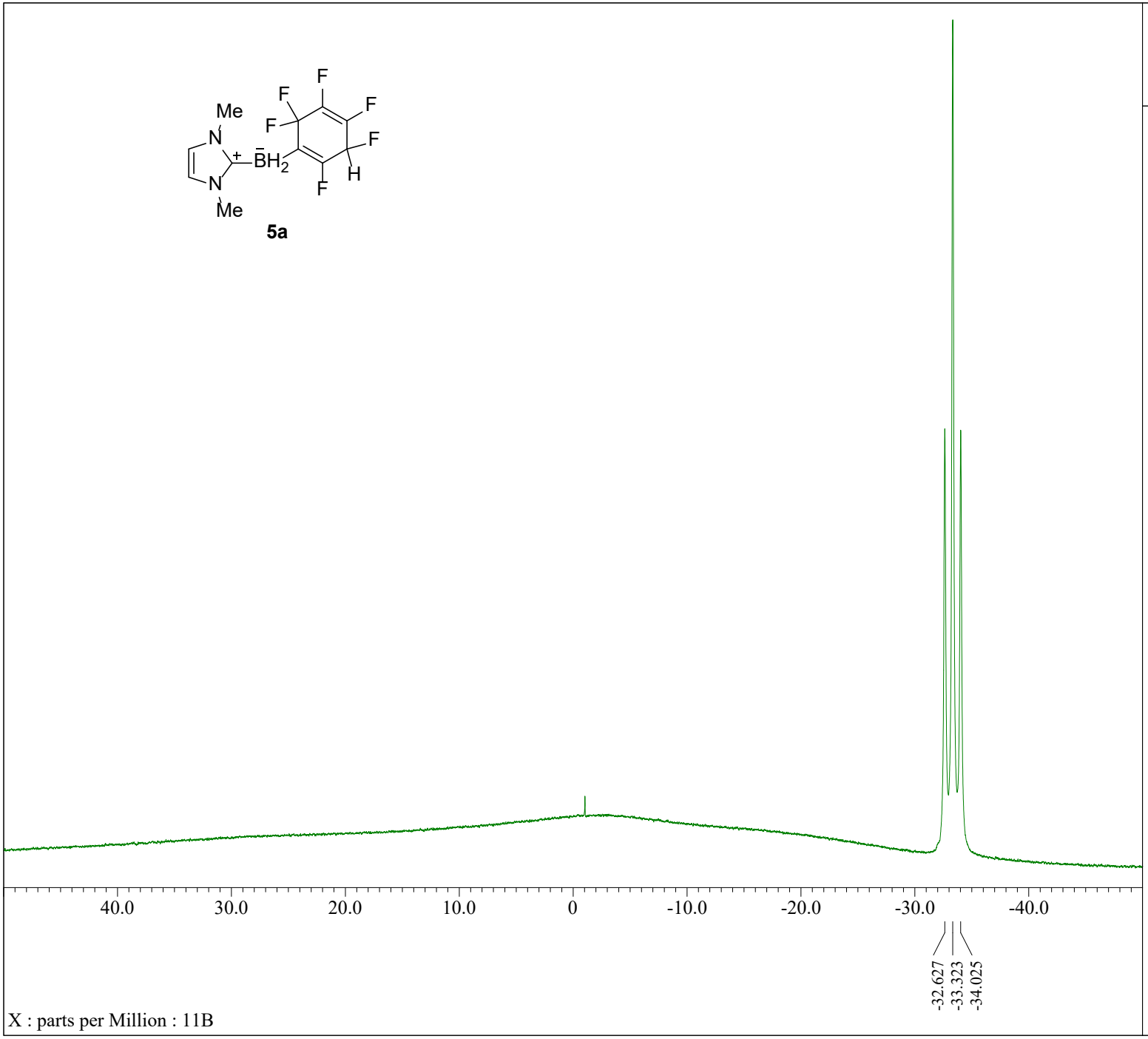

JEOL
Solutions for Innovation

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Experiment       = zg
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Revision_Time    = 29-JAN-2020 11:54:50
Current_Time     =  6-FEB-2020 09:40:42

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Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 11B
X_Freq           = 128.37760517[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 0[Hz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 25.90673575[kHz]
Scans            = 64

Temp_Get         = 298.1521[K]
Filter_Factor    = 772
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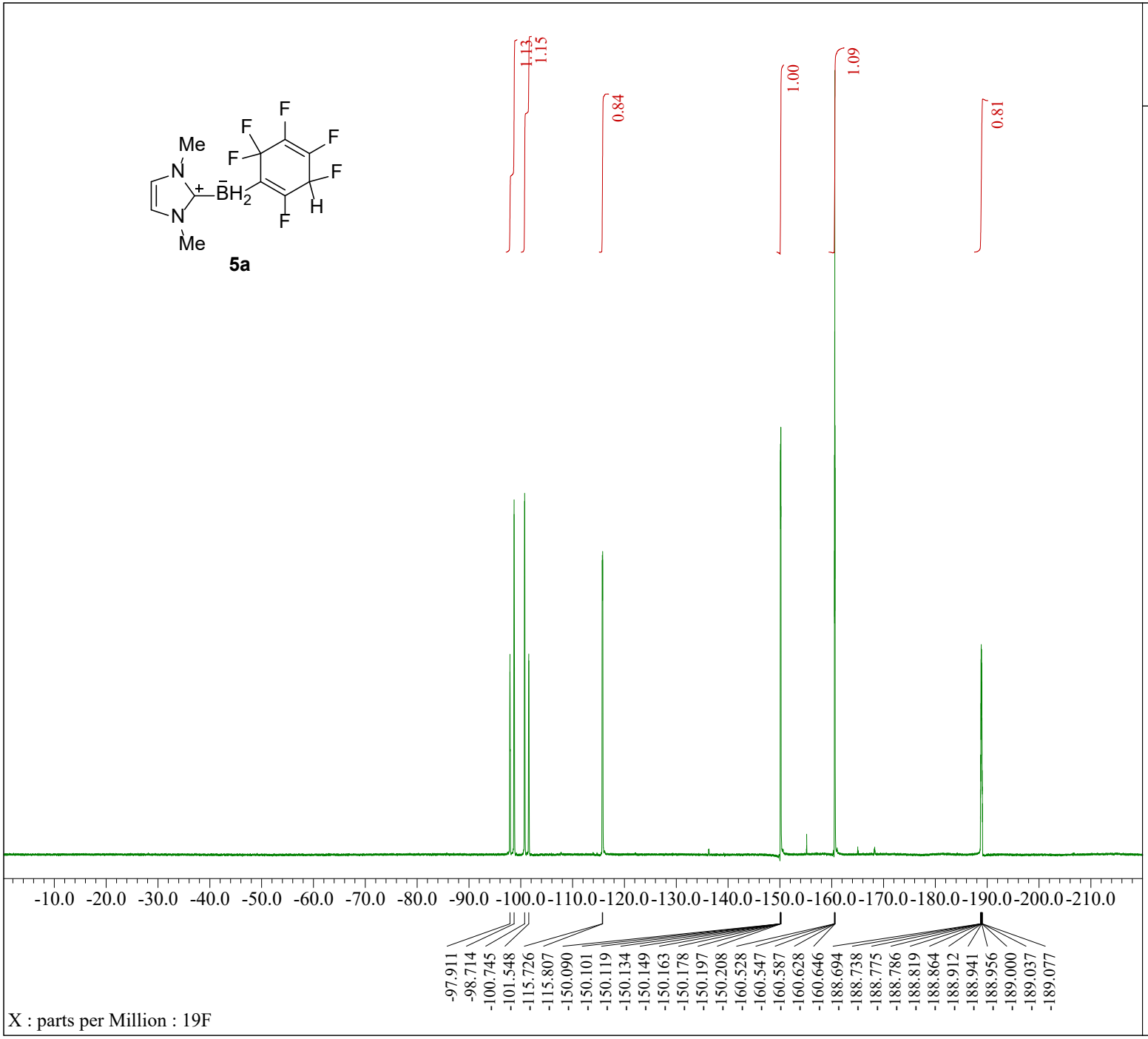

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Solvent          = CHLOROFORM-D
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Revision_Time    =  6-FEB-2020 09:51:20
Current_Time     =  6-FEB-2020 09:52:29

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Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
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X_Sweep          = 90.90909091[kHz]
Scans            = 16

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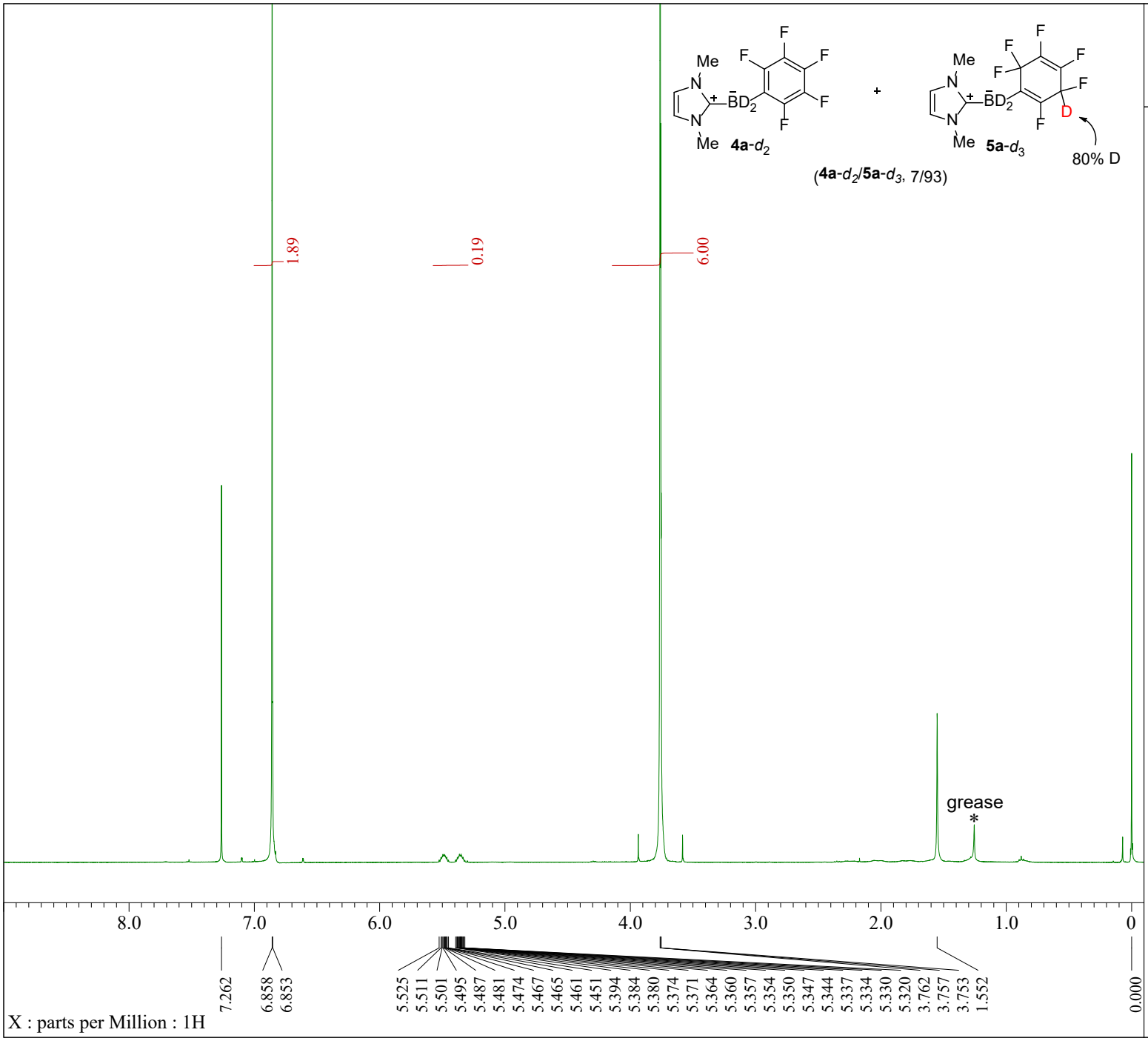

JEOL Solutions for Innovation

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Current_Time      = 14-FEB-2020 18:57:56

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Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = BRUKER_DMX_NMR

Field_Strength    = 9.39793489[T] (400[MHz])
X_Domain          = 1H
X_Freq            = 400.1324708[MHz]
X_Freq_Flip       = TRUE
X_Offset          = 2.470802[kHz]
X_Points          = 32768
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Scans             = 16

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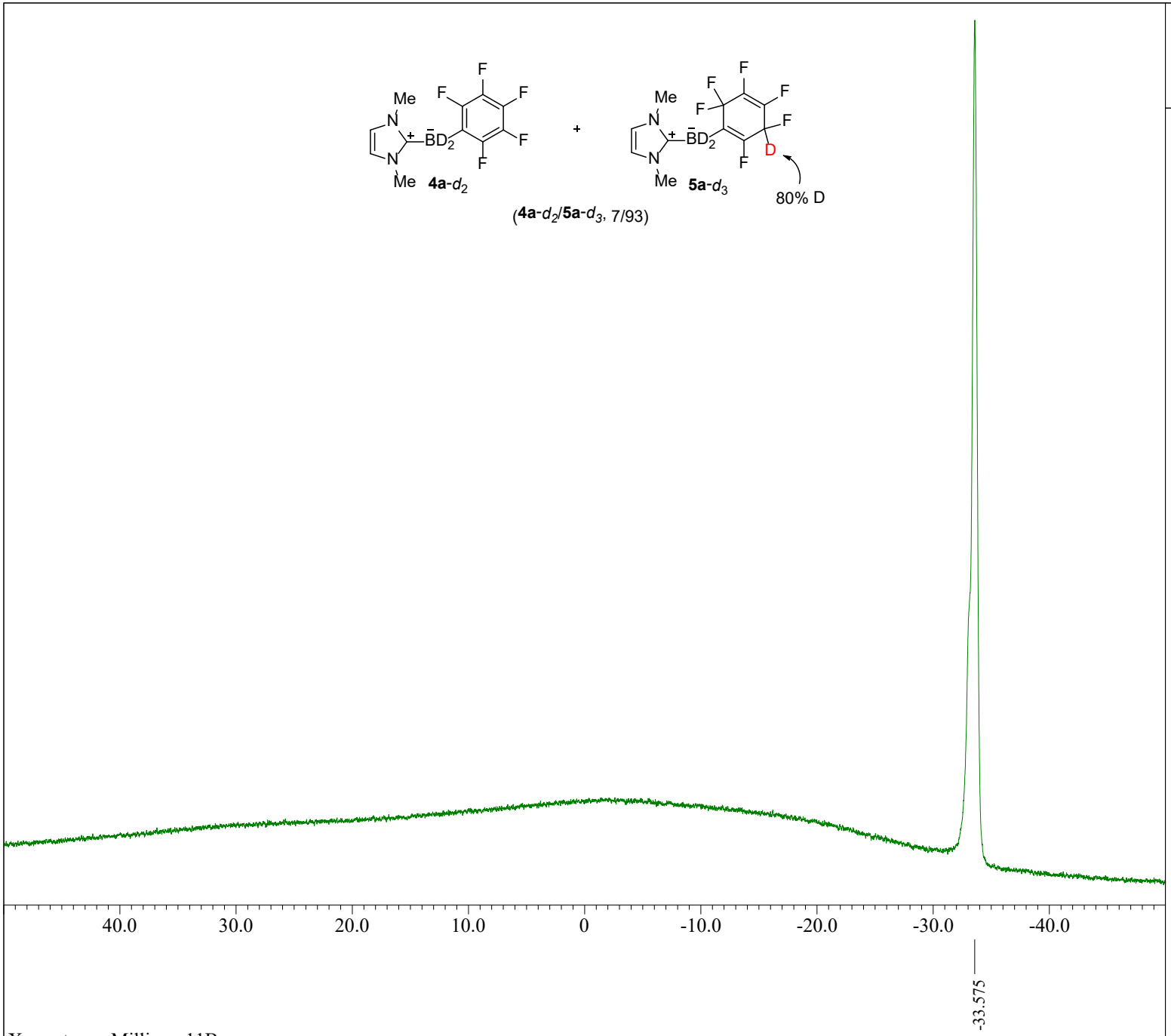

JEOL Solutions for Innovation

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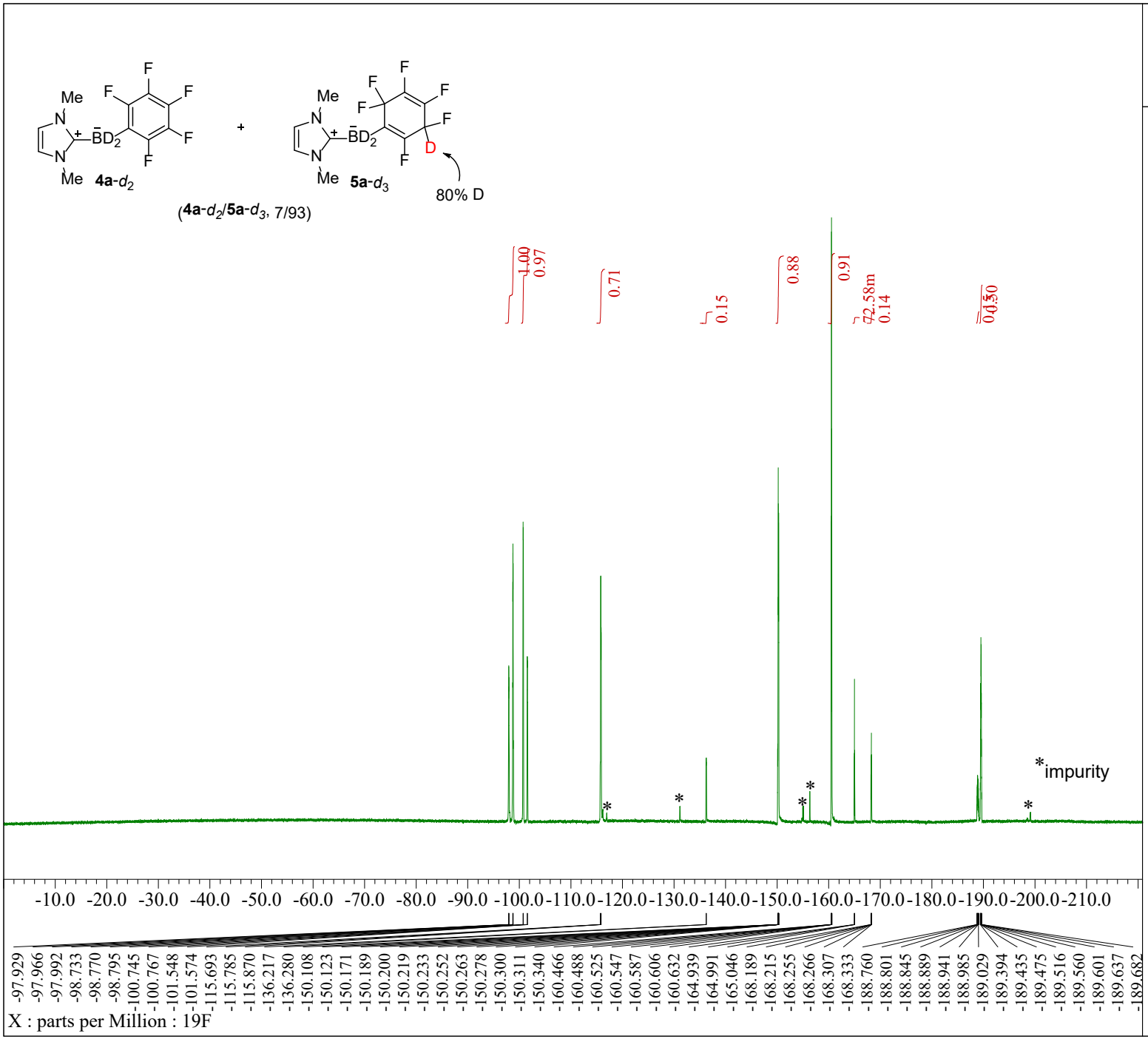
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 14-FEB-2020 10:57:15
Revision_Time    = 14-FEB-2020 18:58:53
Current_Time     = 14-FEB-2020 19:00:32

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 11B
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 11B
X_Freq           = 128.37760517[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 0[Hz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 25.90673575[kHz]
Scans            = 45

Temp_Get         = 298.1495[K]
Filter_Factor    = 772
```



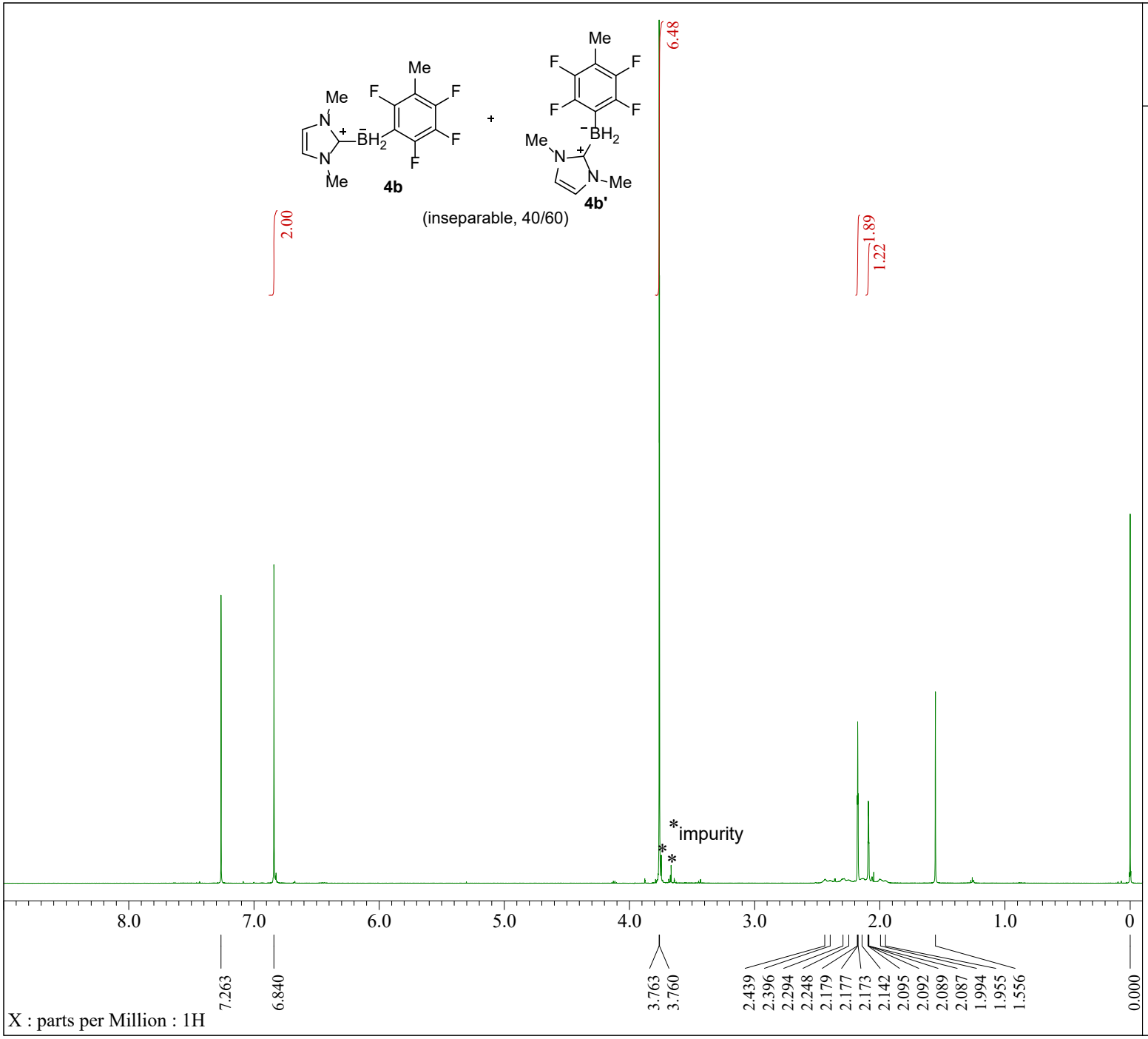
JEOL Solutions for Innovation

```
Filename         = KT-239-purified-19F_1-5.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 14-FEB-2020 11:09:06
Revision_Time    = 14-FEB-2020 19:56:20
Current_Time     = 14-FEB-2020 20:33:46

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 65536
Dim_Title        = 19F
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength   = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
X_Prescans       = 4
X_Sweep          = 90.90909091[kHz]
Scans            = 16

Temp_Get         = 298.1487[K]
Filter_Factor    = 220
```



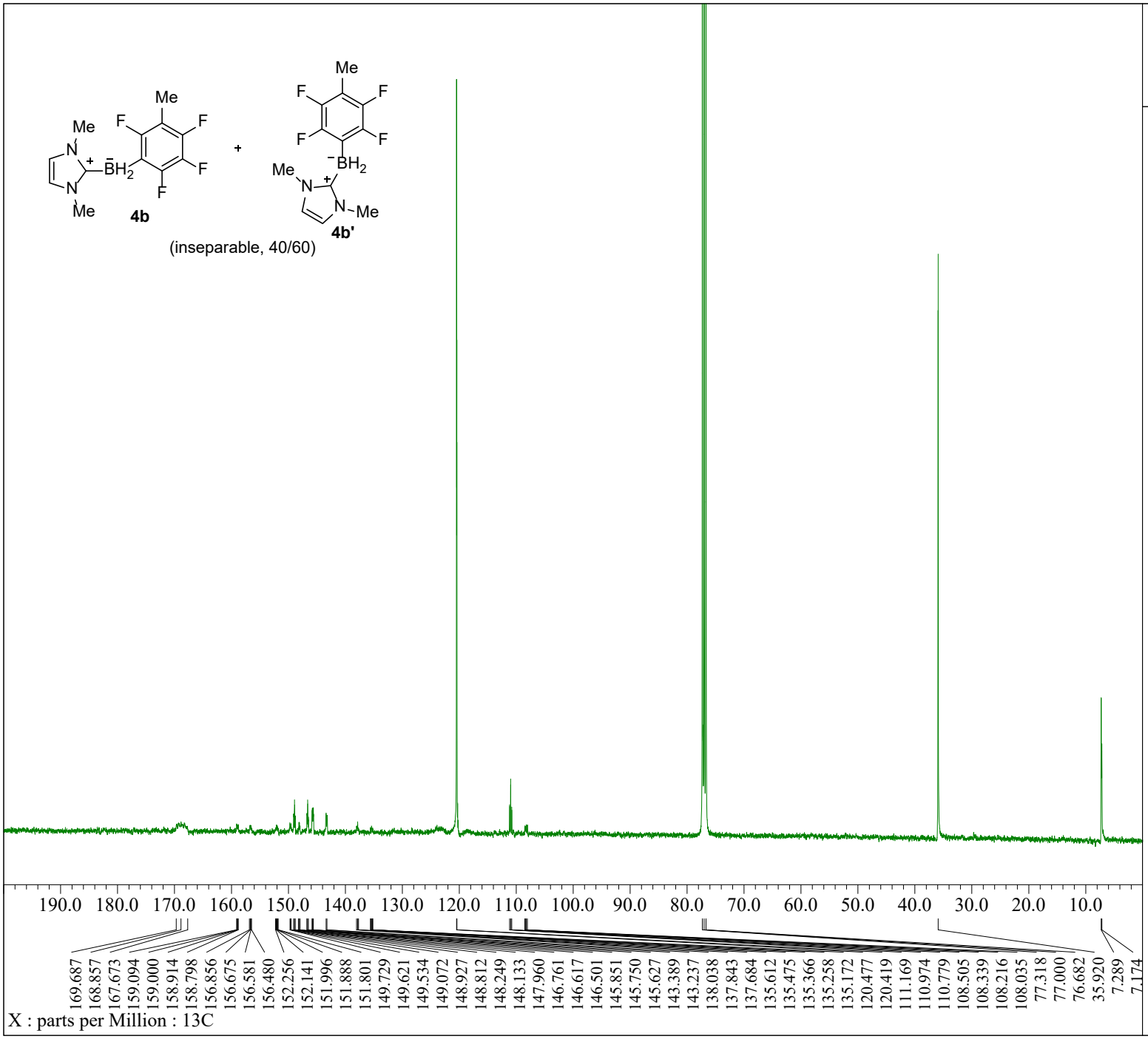
JEOL Solutions for Innovation

```
Filename          = KT-136-purified-1H-1.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#473304
Solvent           = CHLOROFORM-D
Creation_Time     = 15-MAR-2019 12:49:44
Revision_Time     = 29-JAN-2020 14:56:07
Current_Time      =  6-FEB-2020 09:58:05

Comment           = KT-136-purified-1H
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = JNM-ECA600

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.9097984[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.34366642[Hz]
X_Sweep           = 11.26126126[kHz]
Irr_Domain        = 1H
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 2[s]
Recvr_Gain        = 50
Temp_Get          = 19.4[dC]
X_90_Width        = 13.6[us]
X_Acq_Time        = 2.9097984[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.8[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 4.9097984[s]
```



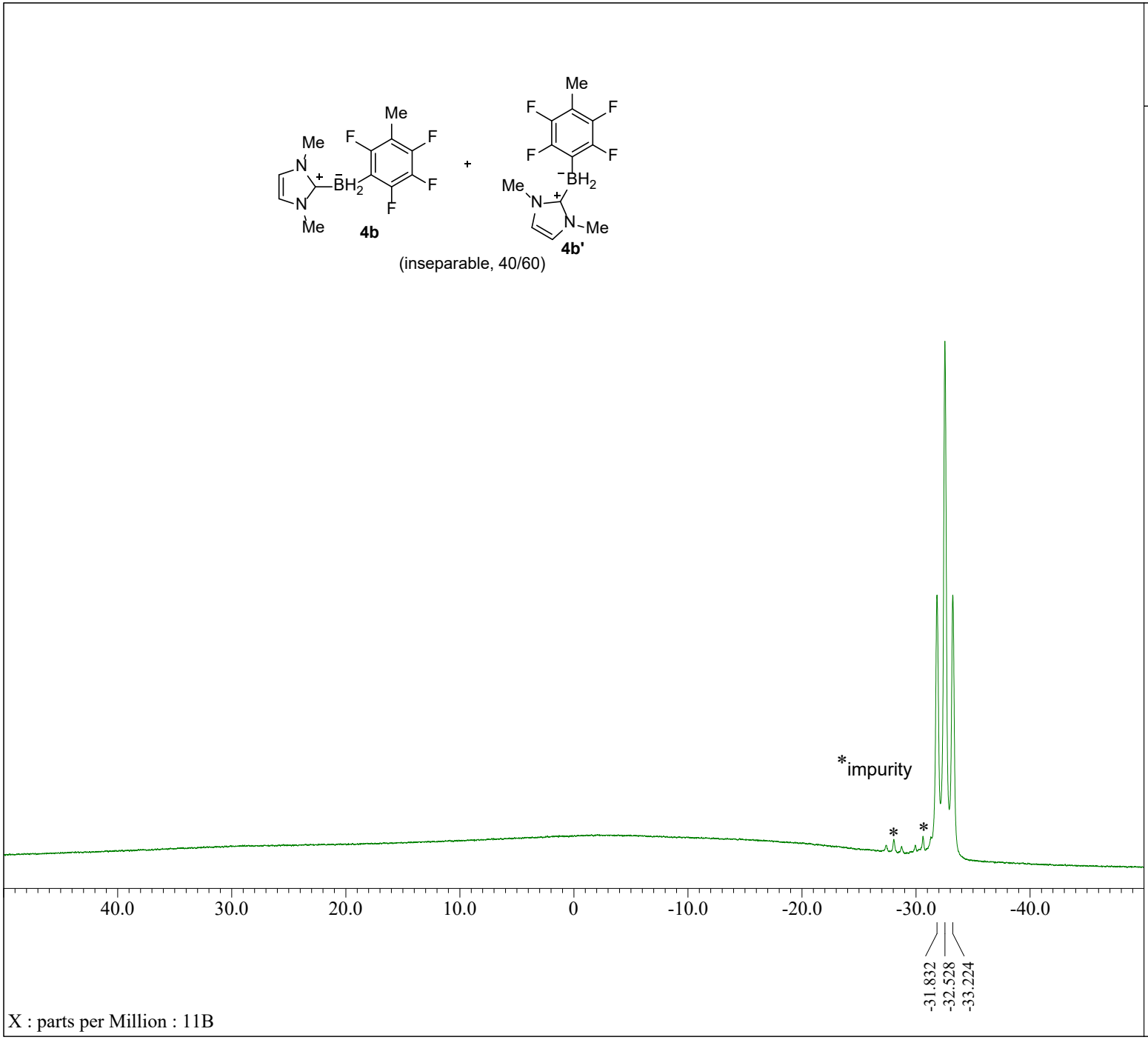
JEOL Solutions for Innovation

```
Filename         = KT-136-purified-13C-re_1-1.j
Author           = nmrsu
Experiment       = zgpg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 29-JAN-2020 14:58:18
Revision_Time    = 29-JAN-2020 18:30:34
Current_Time     =  6-FEB-2020 09:59:15

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 13C
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 13C
X_Freq           = 100.6228298[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 10.061277[kHz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 23.80952381[kHz]
Scans            = 13284

Temp_Get         = 298.1515[K]
Filter_Factor    = 840
```



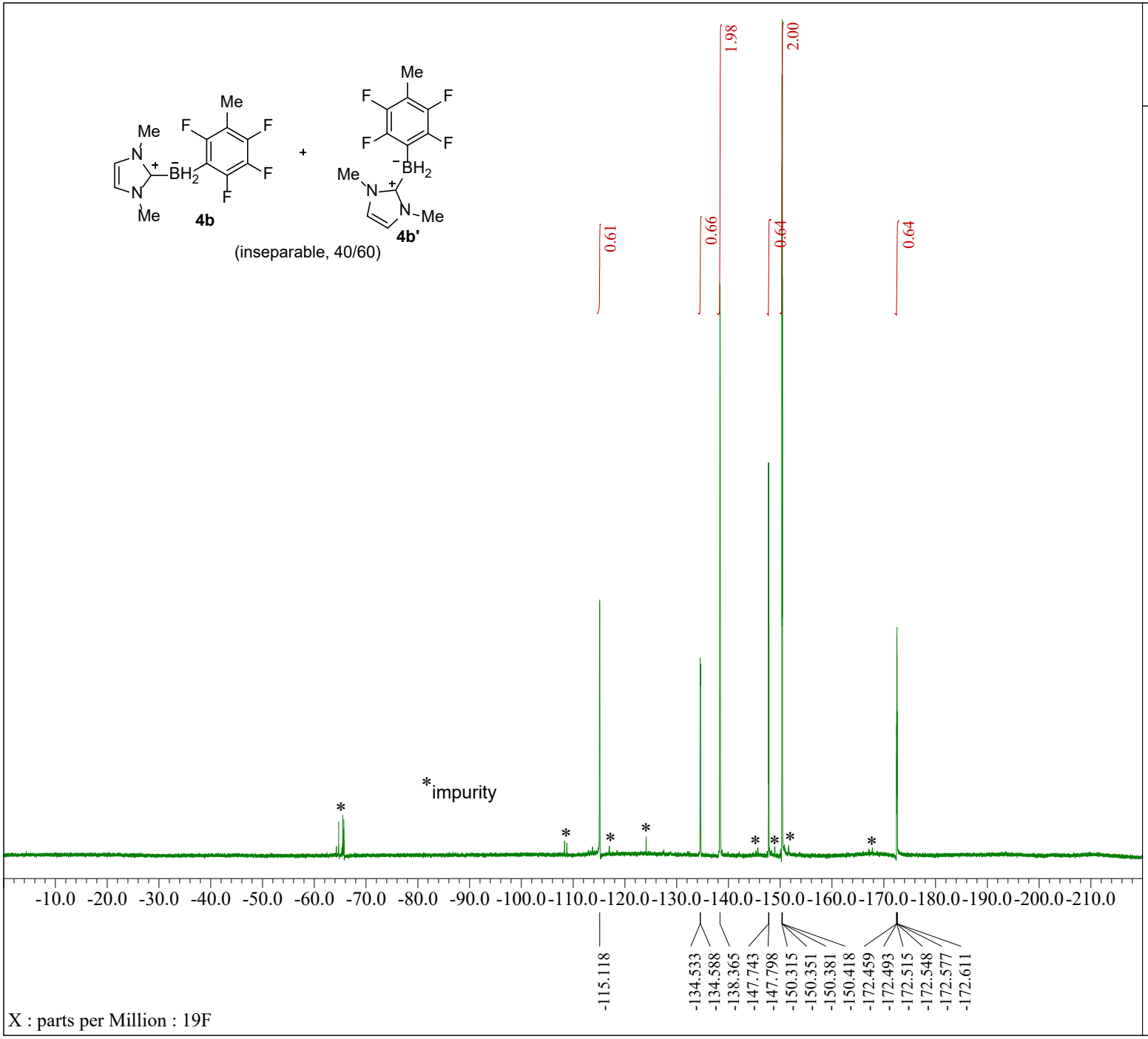
JEOL Solutions for Innovation

```
Filename         = KT-136-purified-11B-re_1-2.j
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 28-JAN-2020 13:35:38
Revision_Time    =  6-FEB-2020 10:00:01
Current_Time     =  6-FEB-2020 10:00:29

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 11B
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 11B
X_Freq           = 128.37760517[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 0[Hz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 25.90673575[kHz]
Scans            = 86

Temp_Get         = 298.1488[K]
Filter_Factor    = 772
```



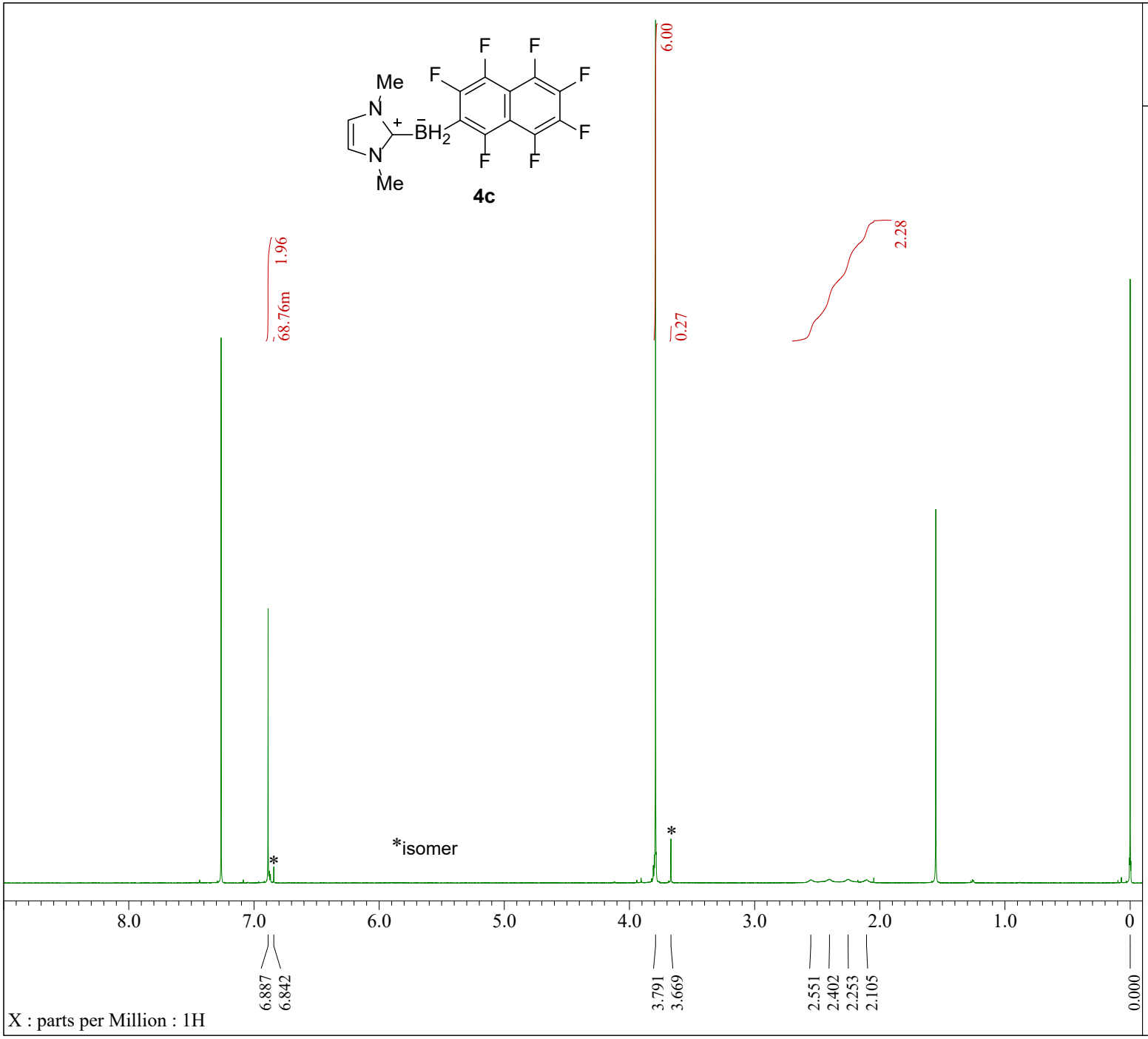
JEOL Solutions for Innovation

```
Filename         = KT-136-purified-19F-re_1-1.j
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 28-JAN-2020 13:19:05
Revision_Time    = 29-JAN-2020 18:46:57
Current_Time     =  6-FEB-2020 10:01:11

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 65536
Dim_Title        = 19F
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength   = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
X_Prescans       = 4
X_Sweep          = 90.90909091[kHz]
Scans            = 4

Temp_Get         = 298.1582[K]
Filter_Factor    = 220
```



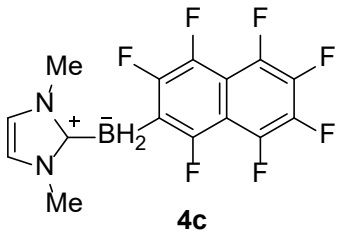
JEOL Solutions for Innovation

```
Filename          = KT-137-purified-1H-1.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#481398
Solvent           = CHLOROFORM-D
Creation_Time     = 15-MAR-2019 13:07:11
Revision_Time     = 27-JAN-2020 22:45:25
Current_Time      =  6-FEB-2020 10:03:10

Comment           = KT-137-purified-1H
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = JNM-ECA600

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.9097984[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.34366642[Hz]
X_Sweep           = 11.26126126[kHz]
Irr_Domain        = 1H
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 2[s]
Recvr_Gain        = 54
Temp_Get          = 19.5[dC]
X_90_Width        = 13.6[us]
X_Acq_Time        = 2.9097984[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.8[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 4.9097984[s]
```



4c

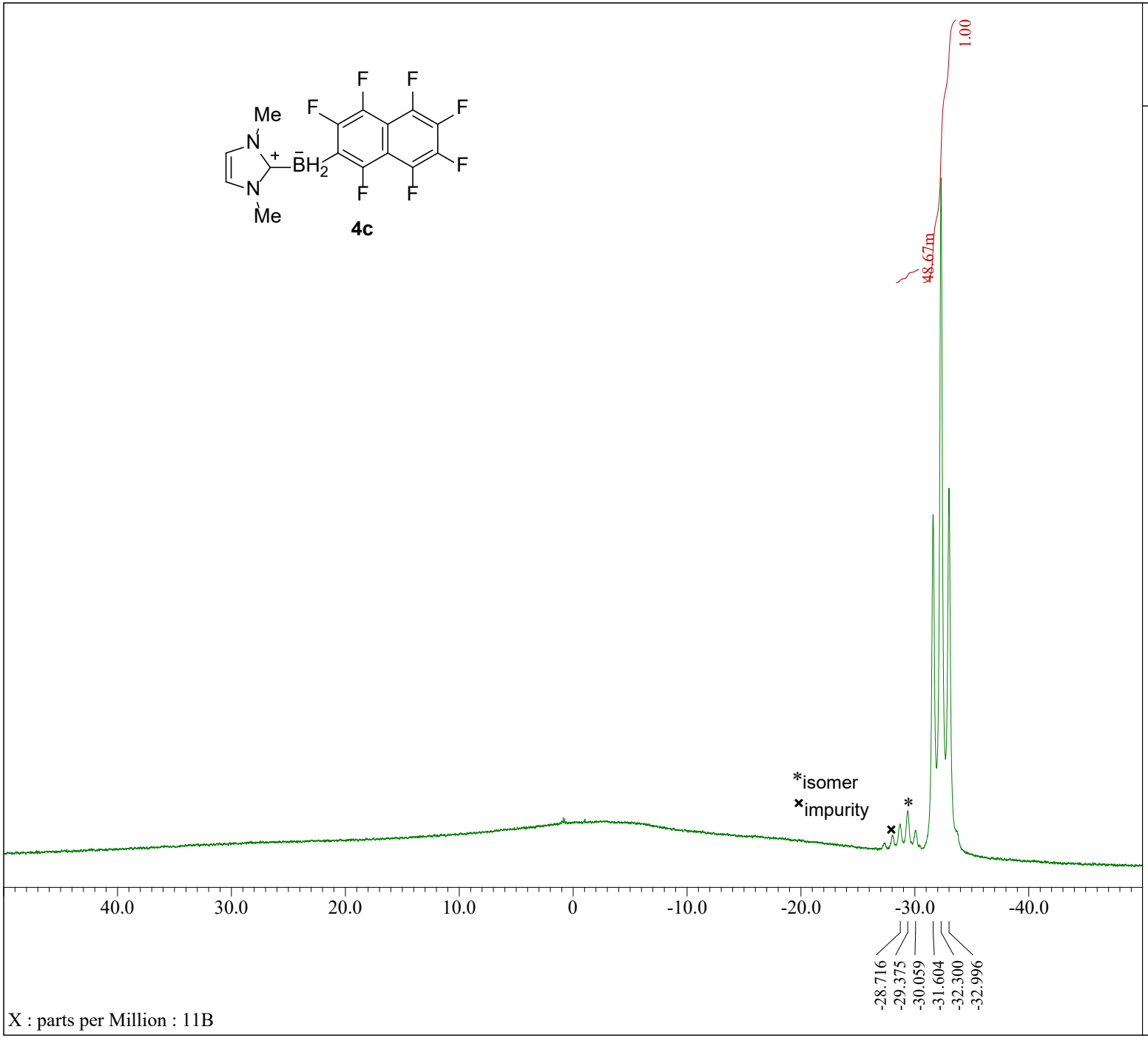
JEOL Solutions for Innovation

```
Filename         = KT-137-purified-13C-re2_1-4.
Author           = nmrsu
Experiment       = zgpg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 15-JAN-2020 16:11:37
Revision_Time    =  6-FEB-2020 10:12:36
Current_Time     =  6-FEB-2020 10:13:20

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 32768
Dim_Title        = 13C
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 13C
X_Freq           = 100.6228298[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 10.061277[kHz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 23.80952381[kHz]
Scans            = 11745

Temp_Get         = 298.1527[K]
Filter_Factor    = 840
```



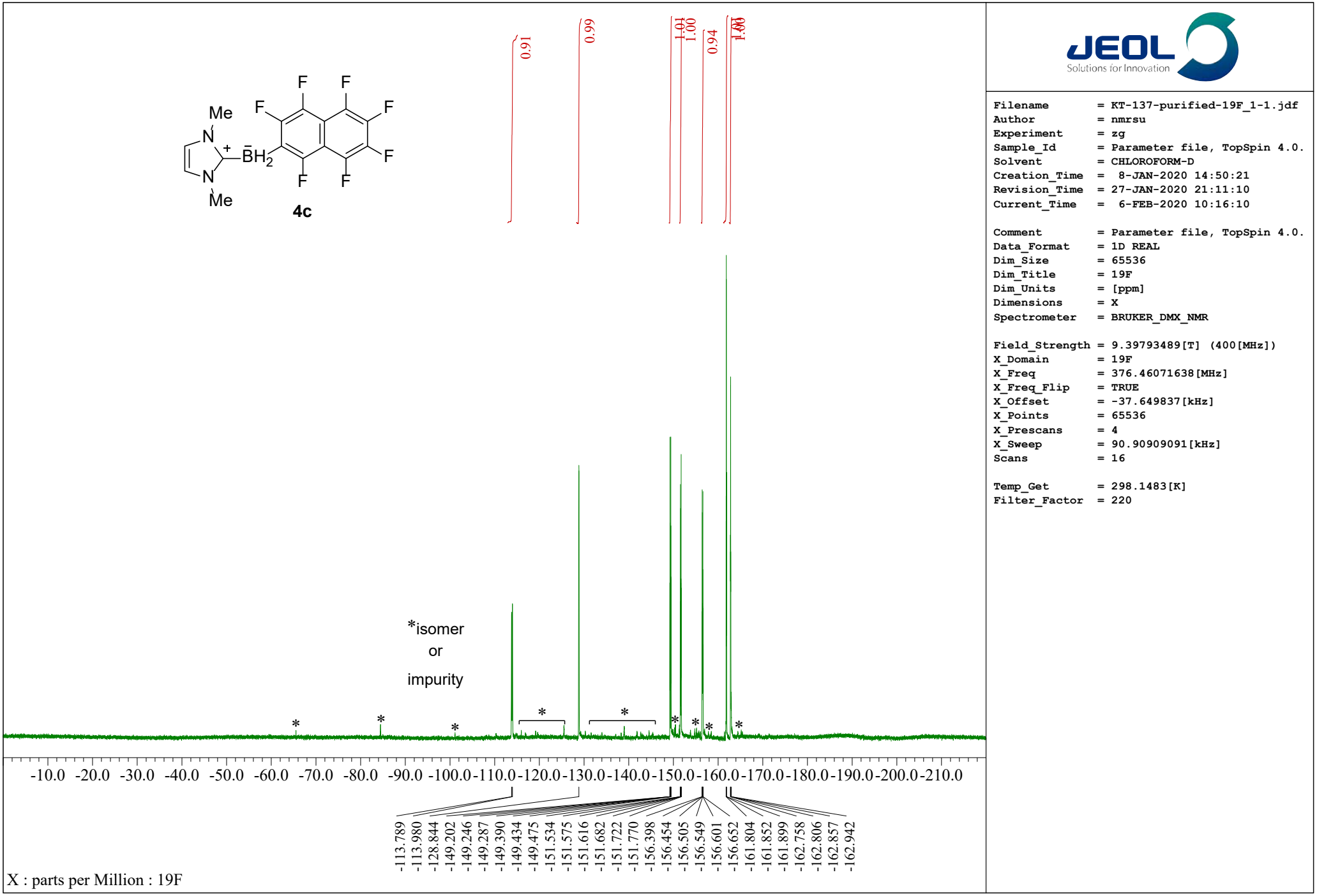
JEOL
Solutions for Innovation

```
Filename         = KT-137-purified-11B-re_1-2.j
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 28-JAN-2020 13:16:26
Revision_Time    =  6-FEB-2020 10:14:56
Current_Time     =  6-FEB-2020 10:15:01

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 11B
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 11B
X_Freq           = 128.37760517[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 0[Hz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 25.90673575[kHz]
Scans            = 39

Temp_Get         = 298.1409[K]
Filter_Factor    = 772
```



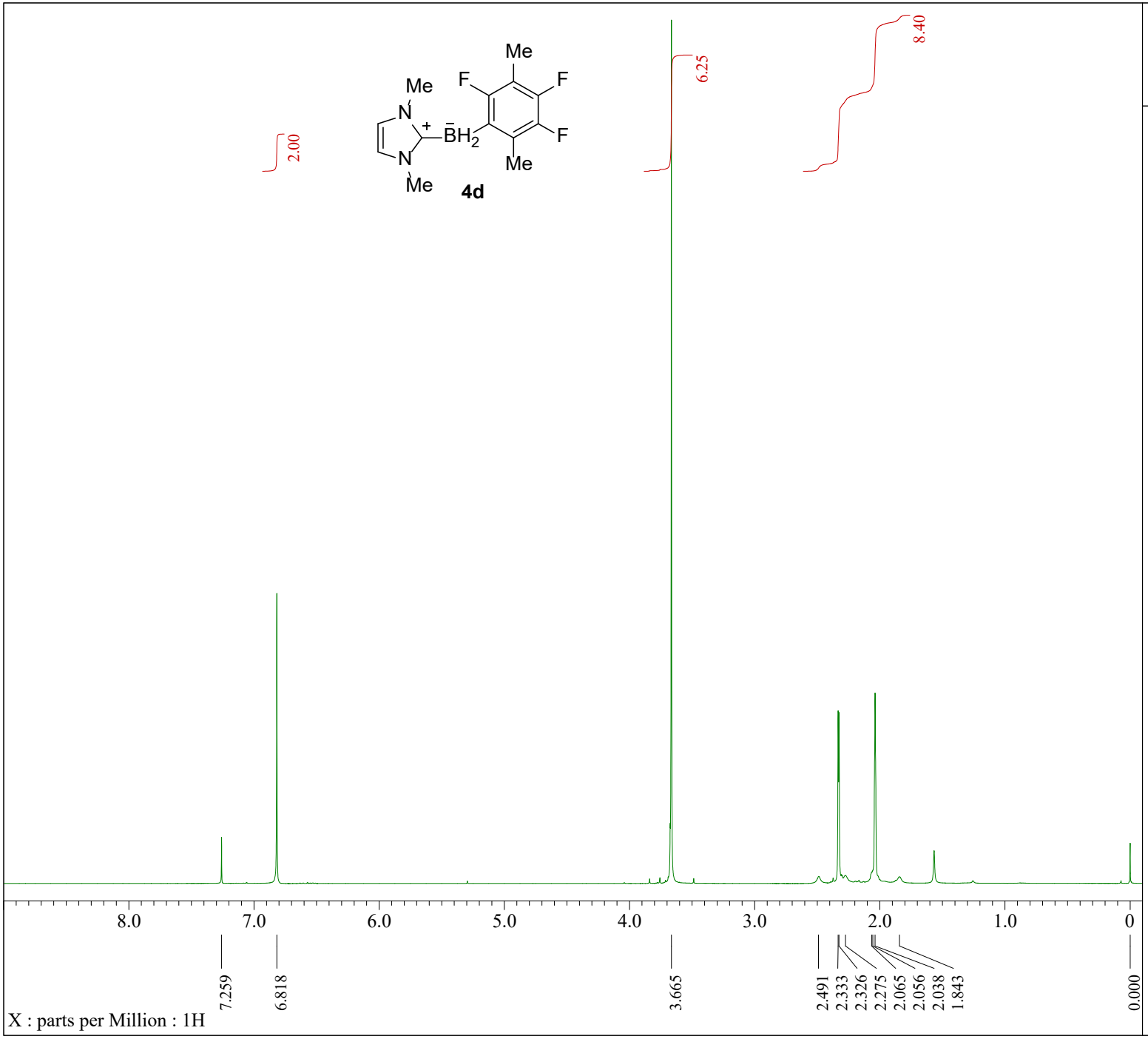
JEOL
Solutions for Innovation

```
Filename         = KT-137-purified-19F_1-1.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 14:50:21
Revision_Time    = 27-JAN-2020 21:11:10
Current_Time     =  6-FEB-2020 10:16:10

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 65536
Dim_Title        = 19F
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
X_Prescans       = 4
X_Sweep          = 90.90909091[kHz]
Scans            = 16

Temp_Get         = 298.1483[K]
Filter_Factor    = 220
```



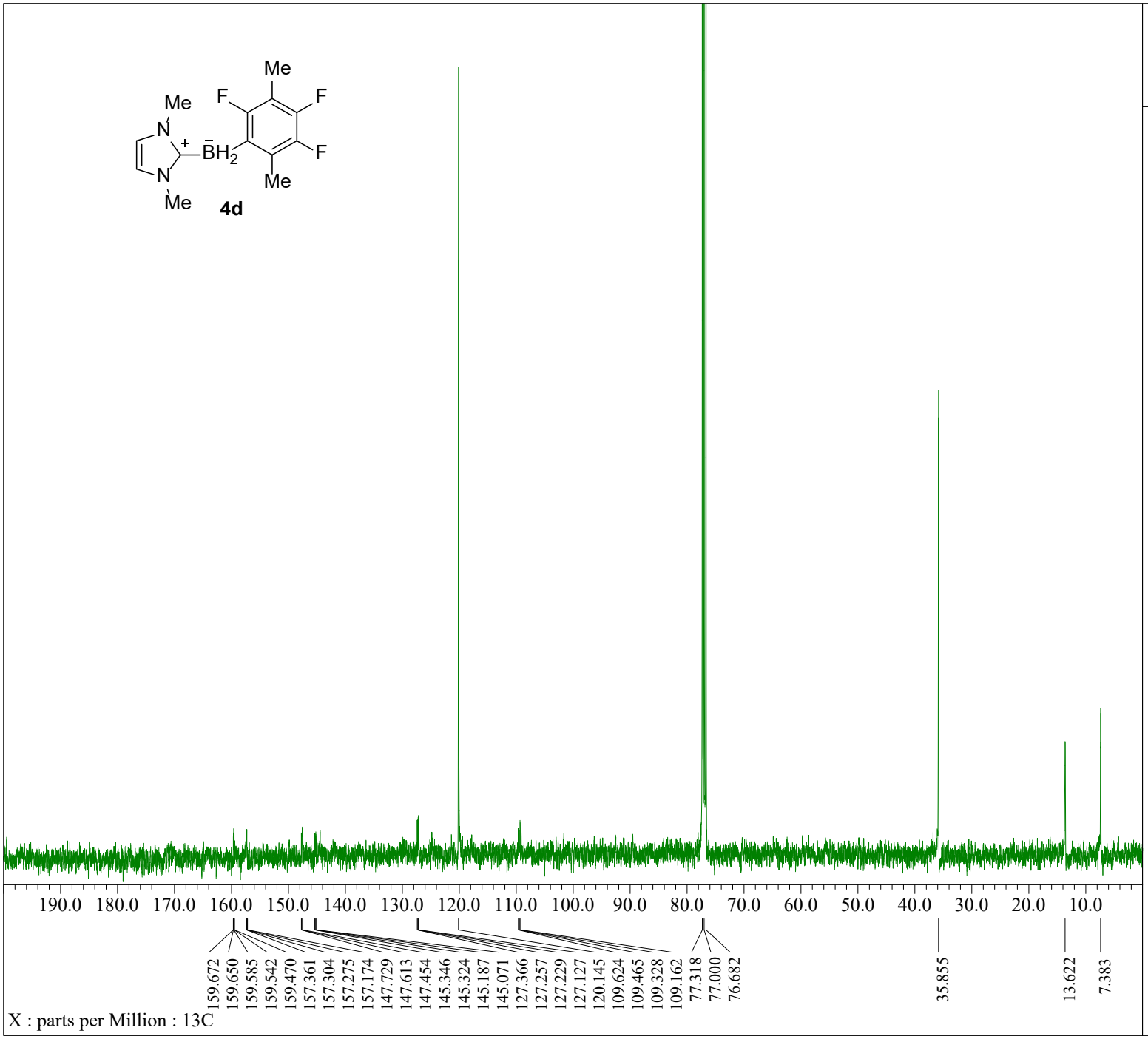
JEOL Solutions for Innovation

```
Filename         = ms-335-purified-1H_1-2.jdf
Author           = nmrsu
Experiment       = zg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 16:23:24
Revision_Time    = 29-JAN-2020 20:04:43
Current_Time     =  6-FEB-2020 10:17:19

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 1H
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 1H
X_Freq           = 400.1324708[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 2.470802[kHz]
X_Points         = 32768
X_Prescans       = 2
X_Sweep          = 8.19672131[kHz]
Scans            = 16

Temp_Get         = 301.15[K]
Filter_Factor    = 2440
```



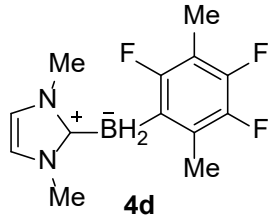
JEOL Solutions for Innovation

```
Filename        = ms-335-purified-13C_1-1.jdf
Author          = nmrsu
Experiment      = zgpg30
Sample_Id       = Parameter file, TopSpin 4.0.
Solvent         = CHLOROFORM-D
Creation_Time   = 29-JAN-2020 20:22:03
Revision_Time   = 29-JAN-2020 21:01:56
Current_Time    =  6-FEB-2020 10:18:15

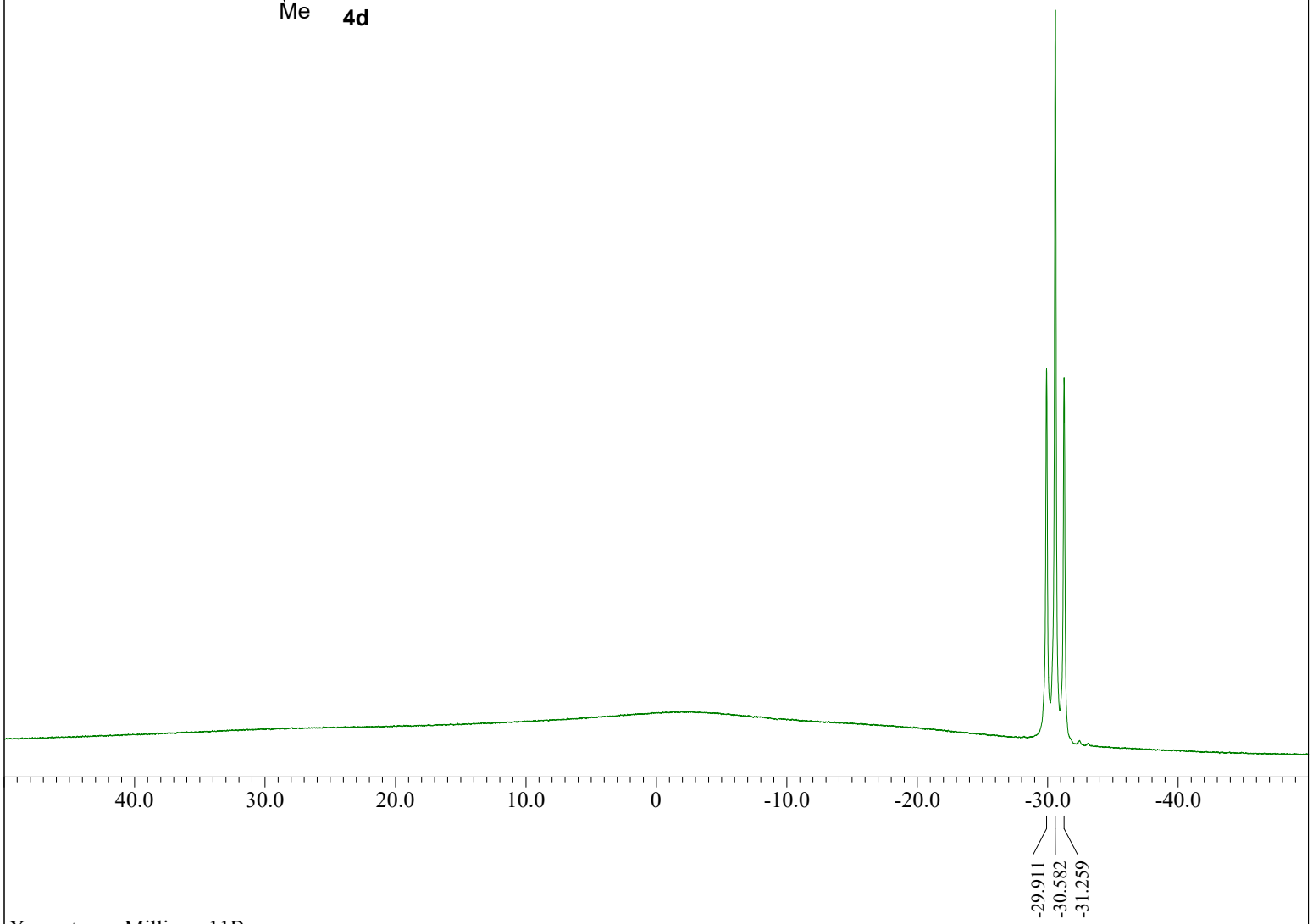
Comment         = Parameter file, TopSpin 4.0.
Data_Format     = 1D REAL
Dim_Size        = 32768
Dim_Title       = 13C
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 13C
X_Freq          = 100.6228298[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 10.061277[kHz]
X_Points        = 32768
X_Prescans      = 4
X_Sweep         = 23.80952381[kHz]
Scans           = 512

Temp_Get        = 301.1474[K]
Filter_Factor   = 840
```



4d



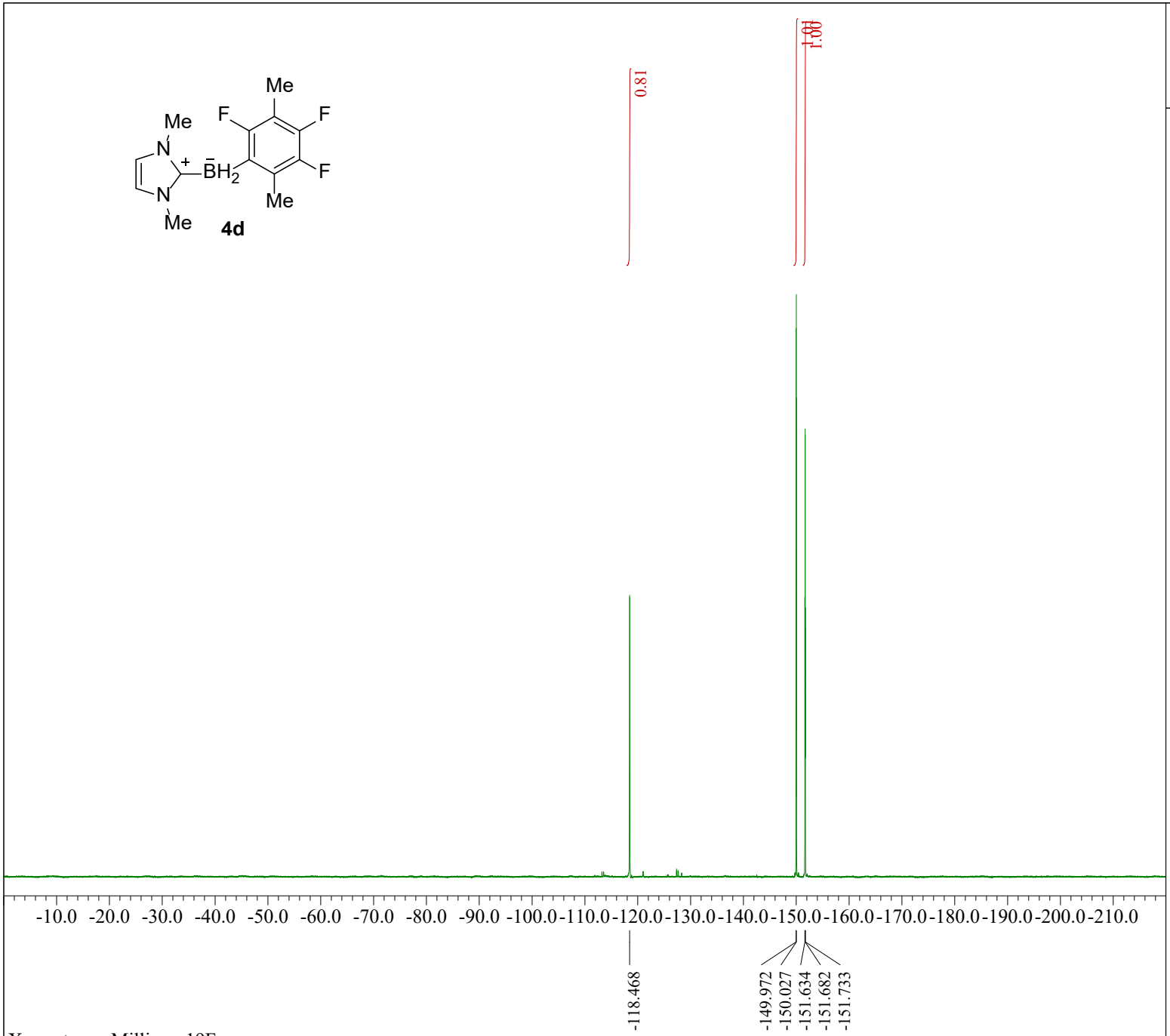
X : parts per Million : 11B

```
Filename        = ms-335-purified-11B_1-1.jdf
Author          = nmrsu
Experiment      = zg
Sample_Id       = Parameter file, TopSpin 4.0.
Solvent         = CHLOROFORM-D
Creation_Time   =  8-JAN-2020 16:24:18
Revision_Time   = 27-JAN-2020 12:33:34
Current_Time    =  6-FEB-2020 10:19:17

Comment         = Parameter file, TopSpin 4.0.
Data_Format     = 1D COMPLEX
Dim_Size        = 32768
Dim_Title       = 11B
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 11B
X_Freq          = 128.37760517[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 0[Hz]
X_Points        = 32768
X_Prescans      = 4
X_Sweep         = 25.90673575[kHz]
Scans           = 128

Temp_Get        = 301.1507[K]
Filter_Factor   = 772
```



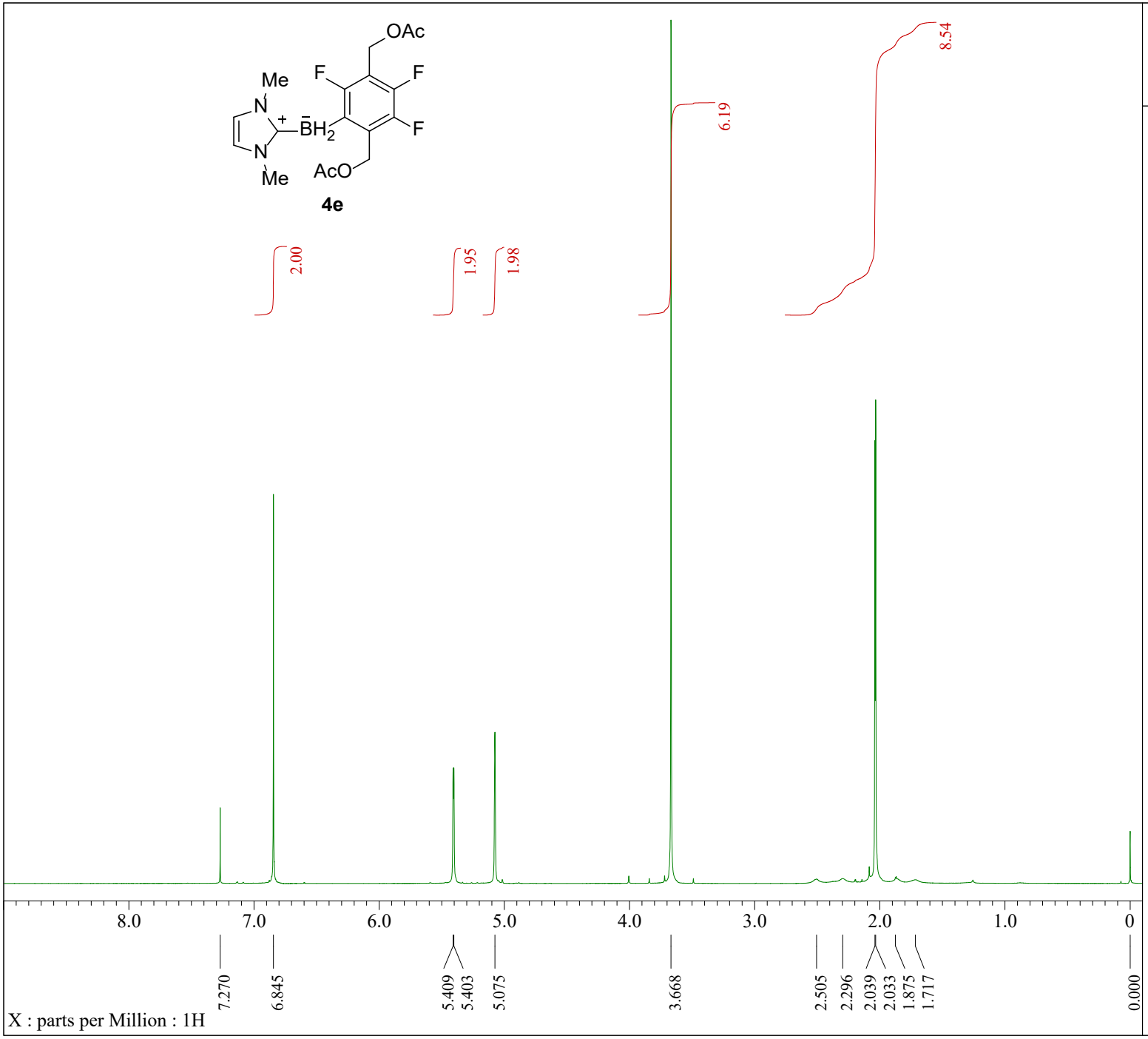
JEOL Solutions for Innovation

```
Filename         = ms-335-purified-19F_1-2.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 16:26:09
Revision_Time    =  6-FEB-2020 10:20:14
Current_Time     =  6-FEB-2020 10:20:20

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 65536
Dim_Title        = 19F
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
X_Prescans       = 4
X_Sweep          = 90.90909091[kHz]
Scans            = 16

Temp_Get         = 301.1485[K]
Filter_Factor    = 220
```



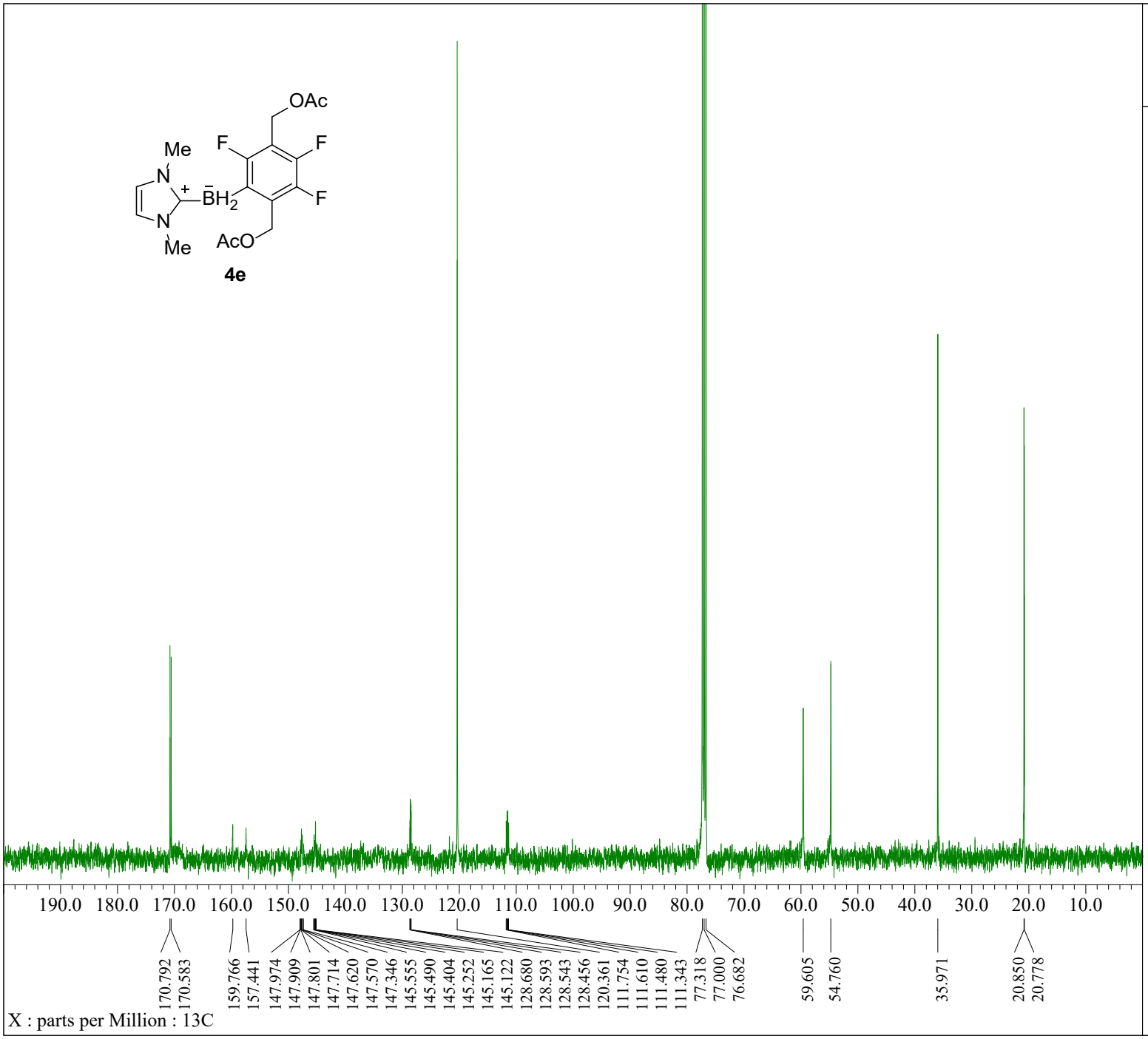
JEOL
Solutions for Innovation

```
Filename         = MS-380-1.jdf
Author           = nmrsu
Experiment       = zg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 30-JAN-2020 13:51:24
Revision_Time    = 30-JAN-2020 16:21:31
Current_Time     =  6-FEB-2020 10:23:22

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 1H
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 1H
X_Freq           = 400.1324708[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 2.470802[kHz]
X_Points         = 32768
X_Prescans       = 2
X_Sweep          = 8.19672131[kHz]
Scans            = 16

Temp_Get         = 298.1402[K]
Filter_Factor    = 2440
```



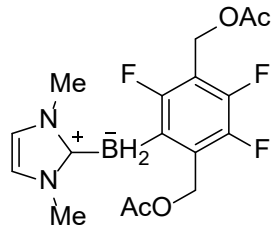
JEOL Solutions for Innovation

```
Filename         = MS-380-purified-13C-re_1-1.j
Author           = nmrsu
Experiment       = zgpg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 28-JAN-2020 13:11:25
Revision_Time    = 30-JAN-2020 17:12:17
Current_Time     =  6-FEB-2020 10:25:00

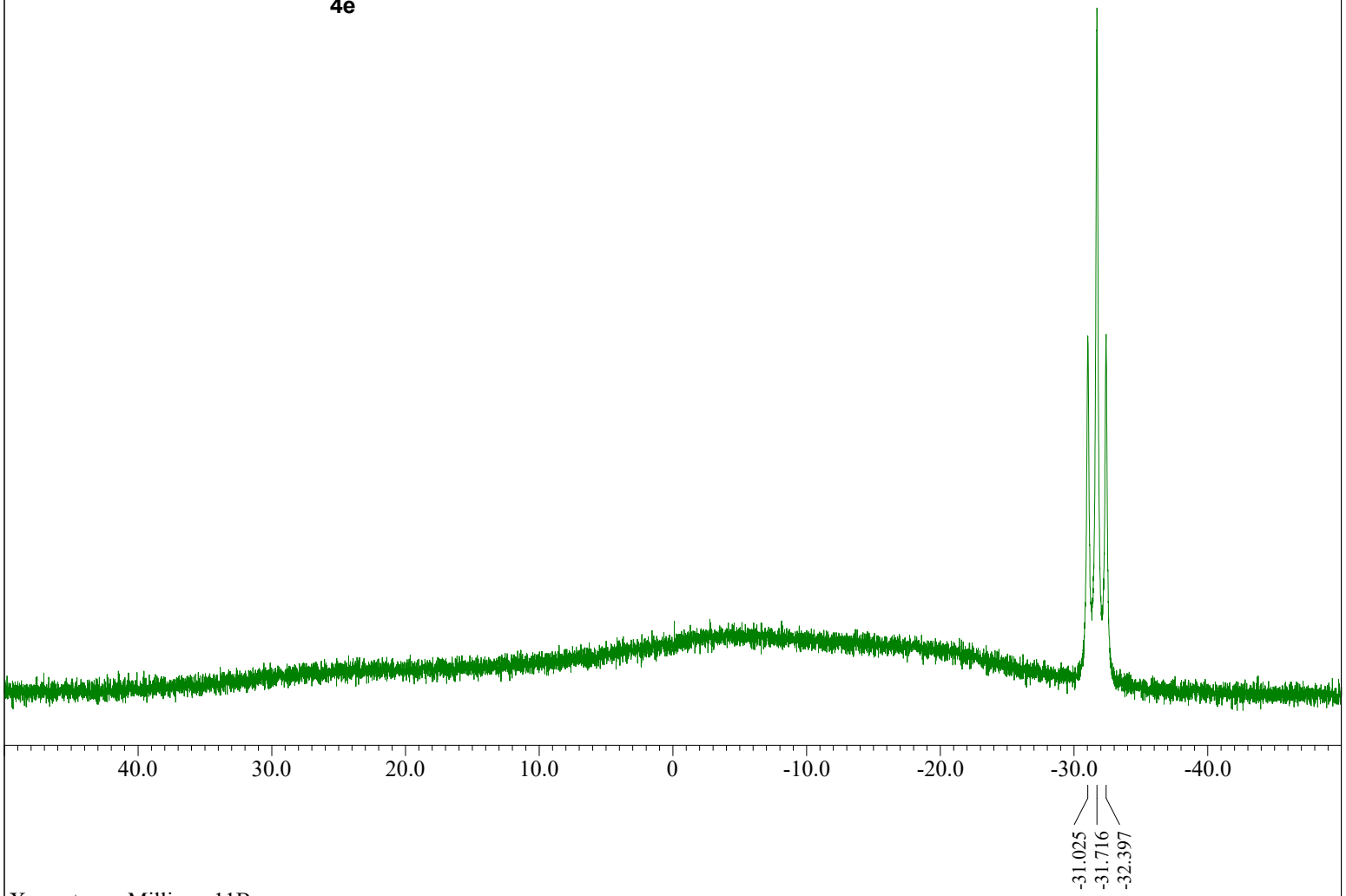
Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 32768
Dim_Title        = 13C
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 13C
X_Freq           = 100.6228298[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 10.061277[kHz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 23.80952381[kHz]
Scans            = 671

Temp_Get         = 298.1469[K]
Filter_Factor    = 840
```



4e



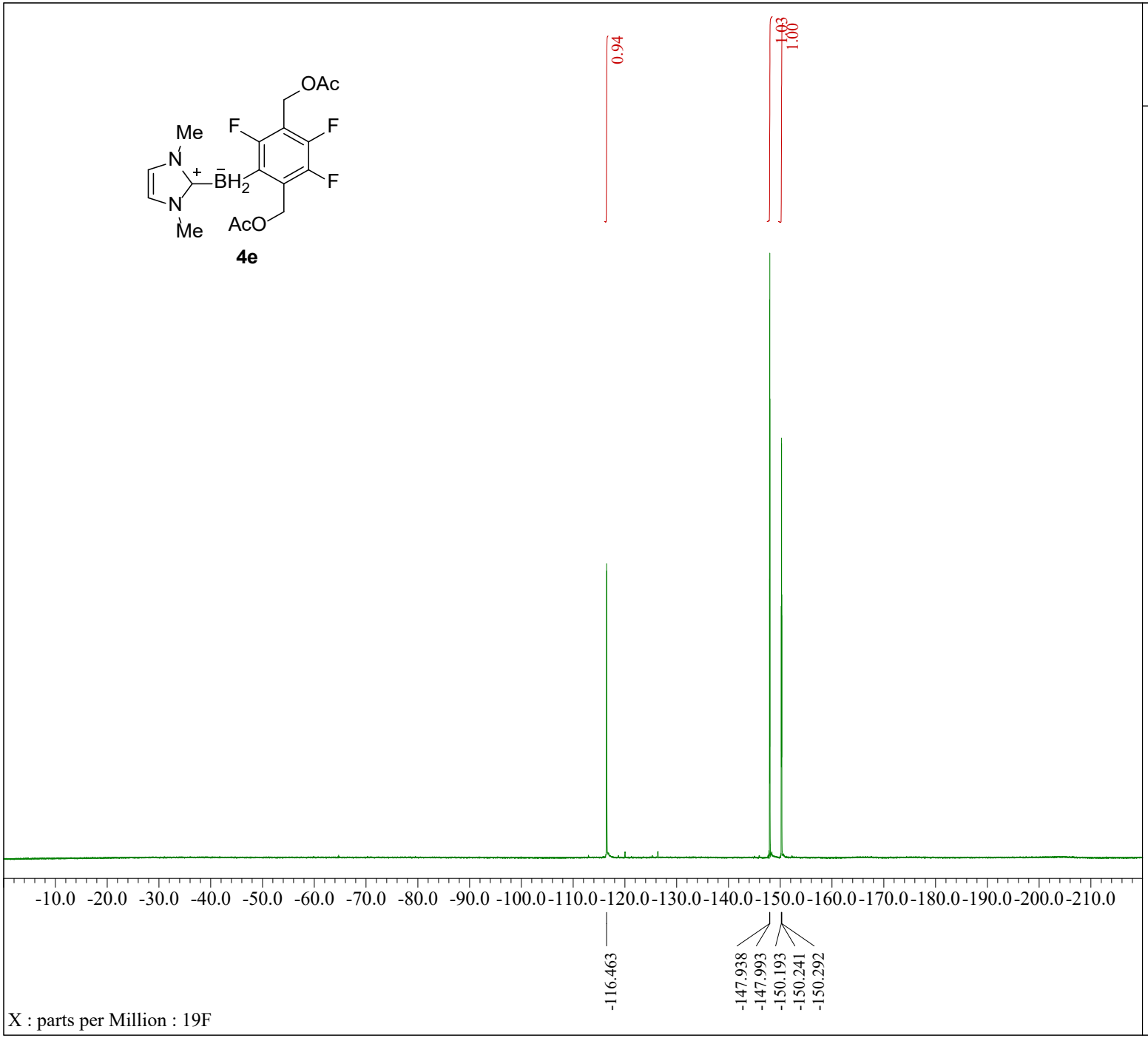
X : parts per Million : 11B

```
Filename          = ms-380-11B-1.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#400534
Solvent           = CHLOROFORM-D
Creation_Time     =  1-FEB-2019 11:12:11
Revision_Time     = 30-JAN-2020 17:17:25
Current_Time      =  6-FEB-2020 10:25:57

Comment           = ms-380-11B
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 11B
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 0.8126464[s]
X_Domain          = 11B
X_Freq            = 128.26613597[MHz]
X_Offset          = 0[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 1.23054751[Hz]
X_Sweep           = 40.32258065[kHz]
Irr_Domain        = 11B
Irr_Freq          = 128.26613597[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 11B
Tri_Freq          = 128.26613597[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 32
Total_Scans       = 32

Relaxation_Delay  = 2[s]
Recvr_Gain        = 46
Temp_Get          = 19.7[dC]
X_90_Width        = 22[us]
X_Acq_Time        = 0.8126464[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 11[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 2.8126464[s]
```



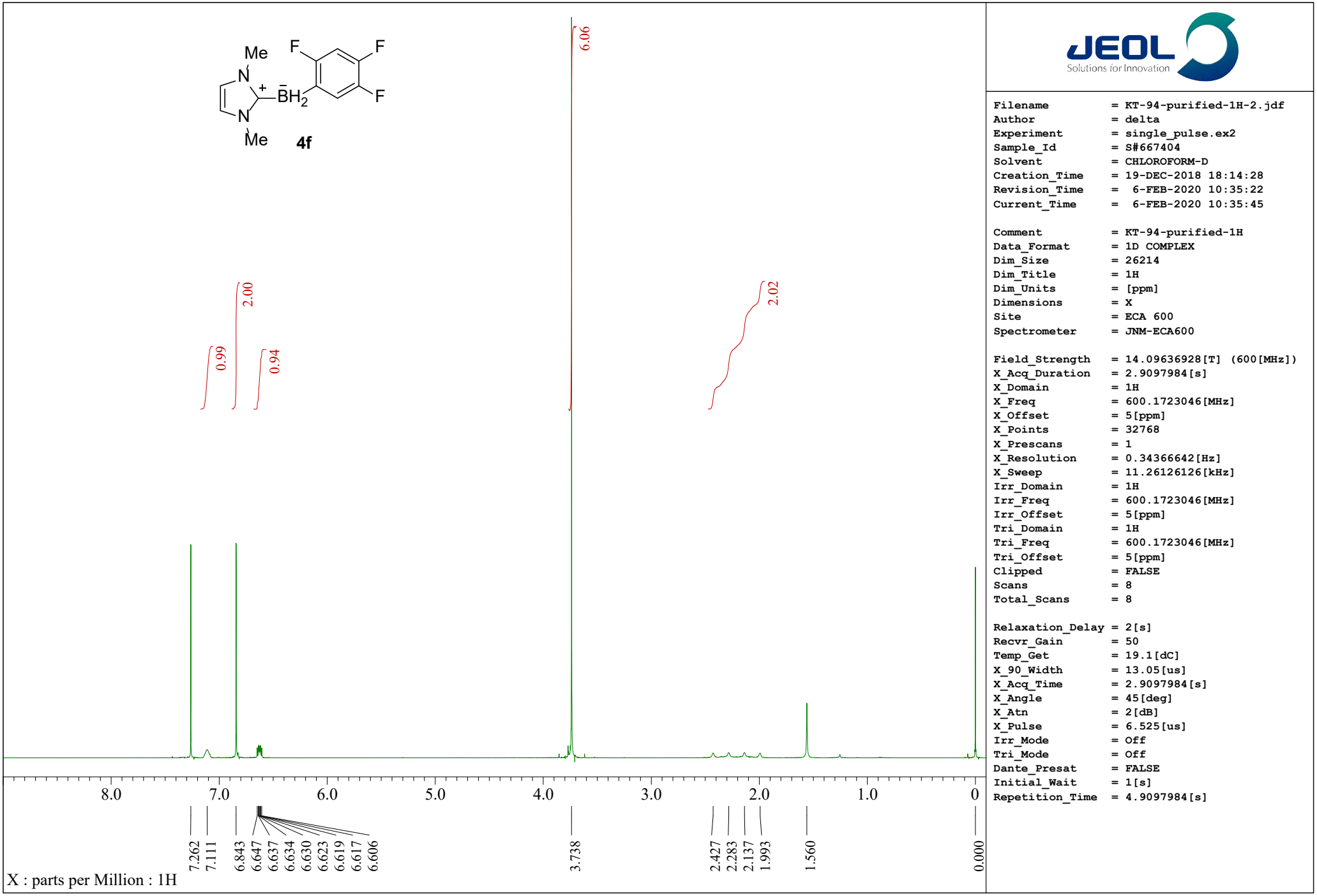
JEOL Solutions for Innovation

```
Filename         = MS-380-purified-19F_1-2.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 30-JAN-2020 17:40:53
Revision_Time    =  6-FEB-2020 10:34:07
Current_Time     =  6-FEB-2020 10:34:12

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 65536
Dim_Title        = 19F
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
X_Prescans       = 4
X_Sweep          = 90.90909091[kHz]
Scans            = 16

Temp_Get         = 301.1495[K]
Filter_Factor    = 220
```



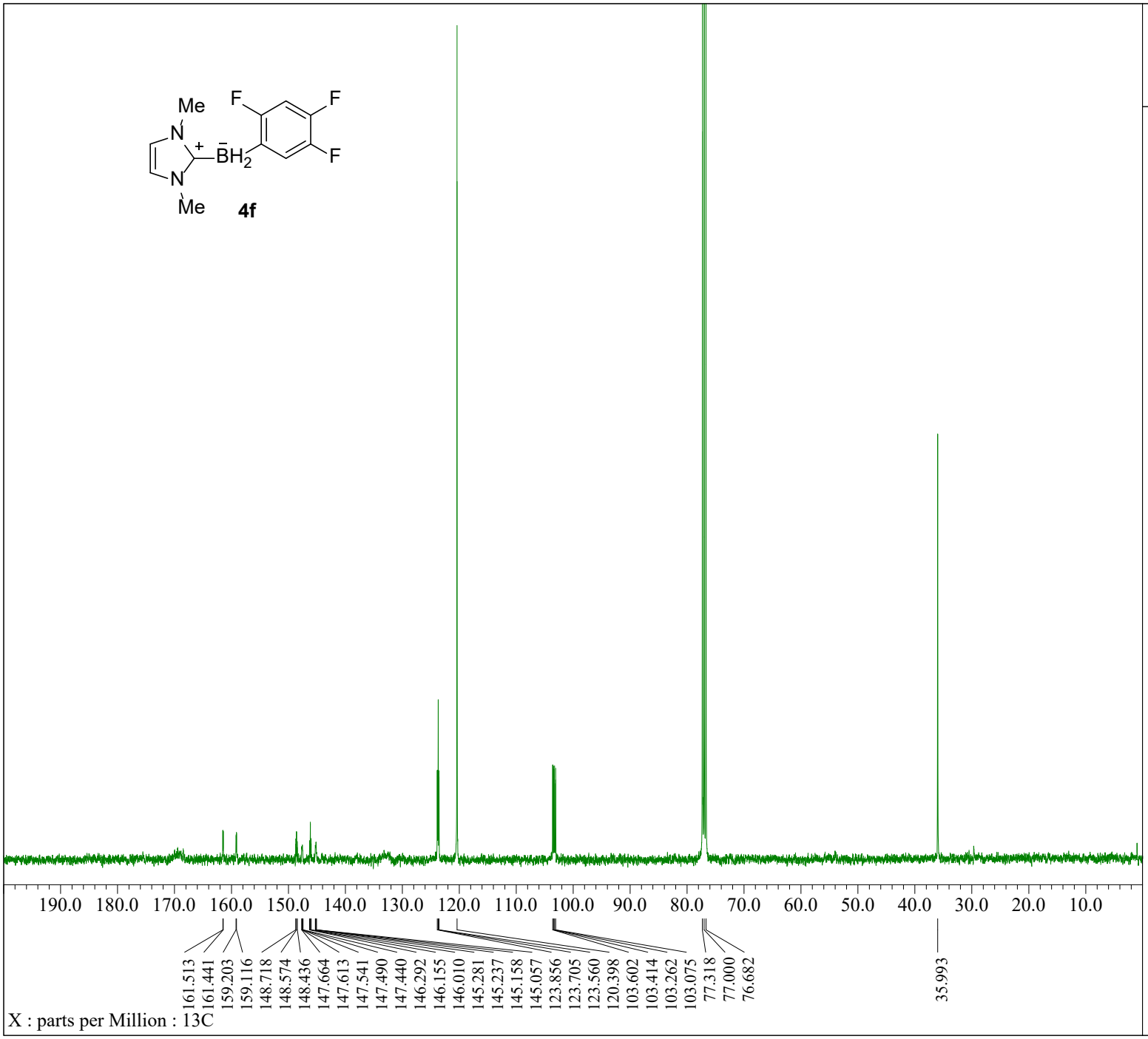
JEOL Solutions for Innovation

```
Filename          = KT-94-purified-1H-2.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#667404
Solvent           = CHLOROFORM-D
Creation_Time     = 19-DEC-2018 18:14:28
Revision_Time     =  6-FEB-2020 10:35:22
Current_Time      =  6-FEB-2020 10:35:45

Comment           = KT-94-purified-1H
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = JNM-ECA600

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.9097984[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.34366642[Hz]
X_Sweep           = 11.26126126[kHz]
Irr_Domain        = 1H
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 2[s]
Recvr_Gain        = 50
Temp_Get          = 19.1[dC]
X_90_Width        = 13.05[us]
X_Acq_Time        = 2.9097984[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.525[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 4.9097984[s]
```



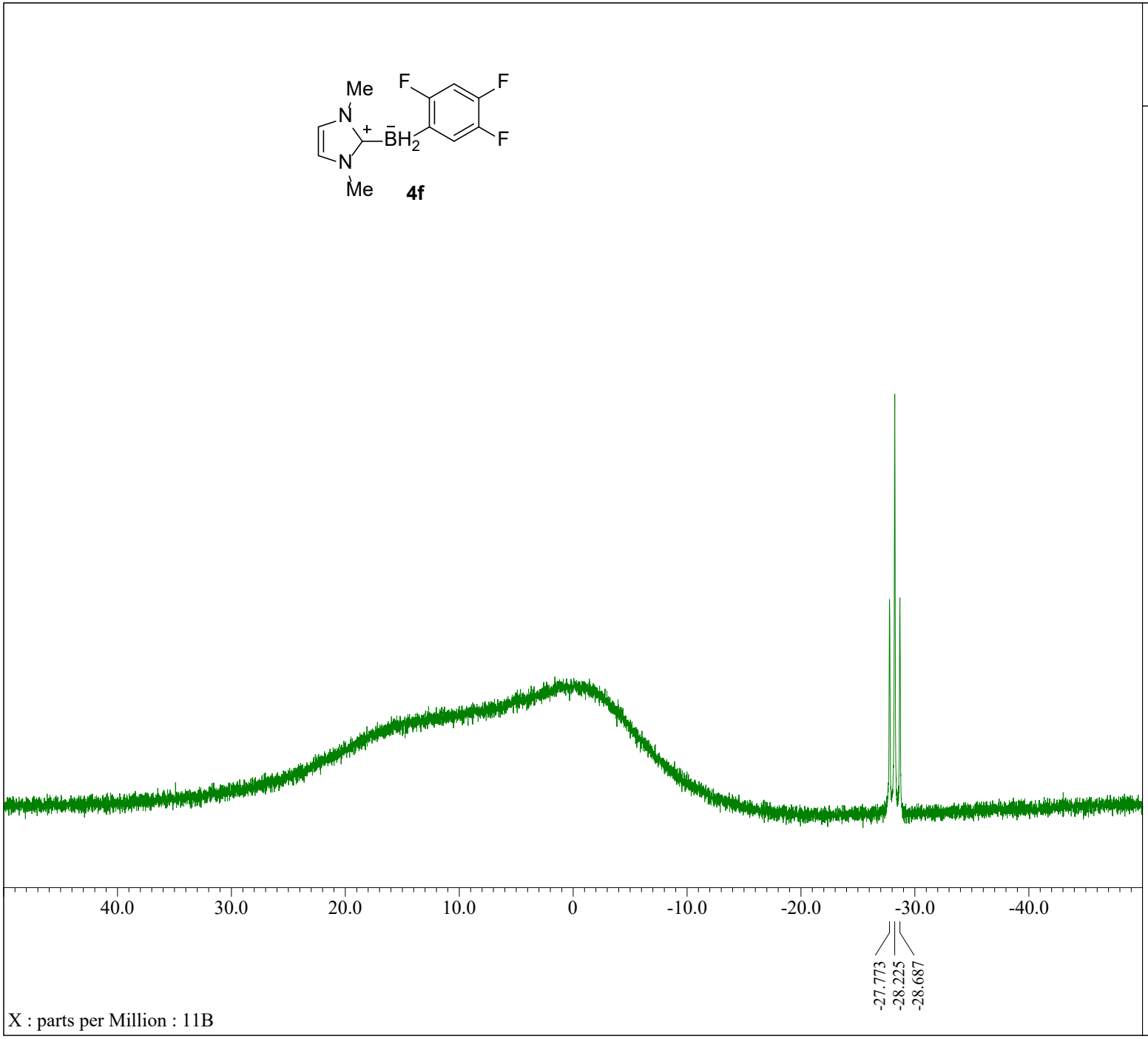
JEOL Solutions for Innovation

```
Filename         = KT-94-purified-13C_1-1.jdf
Author           = nmrsu
Experiment       = zgpg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 15:15:05
Revision_Time    = 31-JAN-2020 10:34:03
Current_Time     =  6-FEB-2020 10:37:02

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 32768
Dim_Title        = 13C
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 13C
X_Freq           = 100.6228298[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 10.061277[kHz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 23.80952381[kHz]
Scans            = 1024

Temp_Get         = 298.1392[K]
Filter_Factor    = 840
```



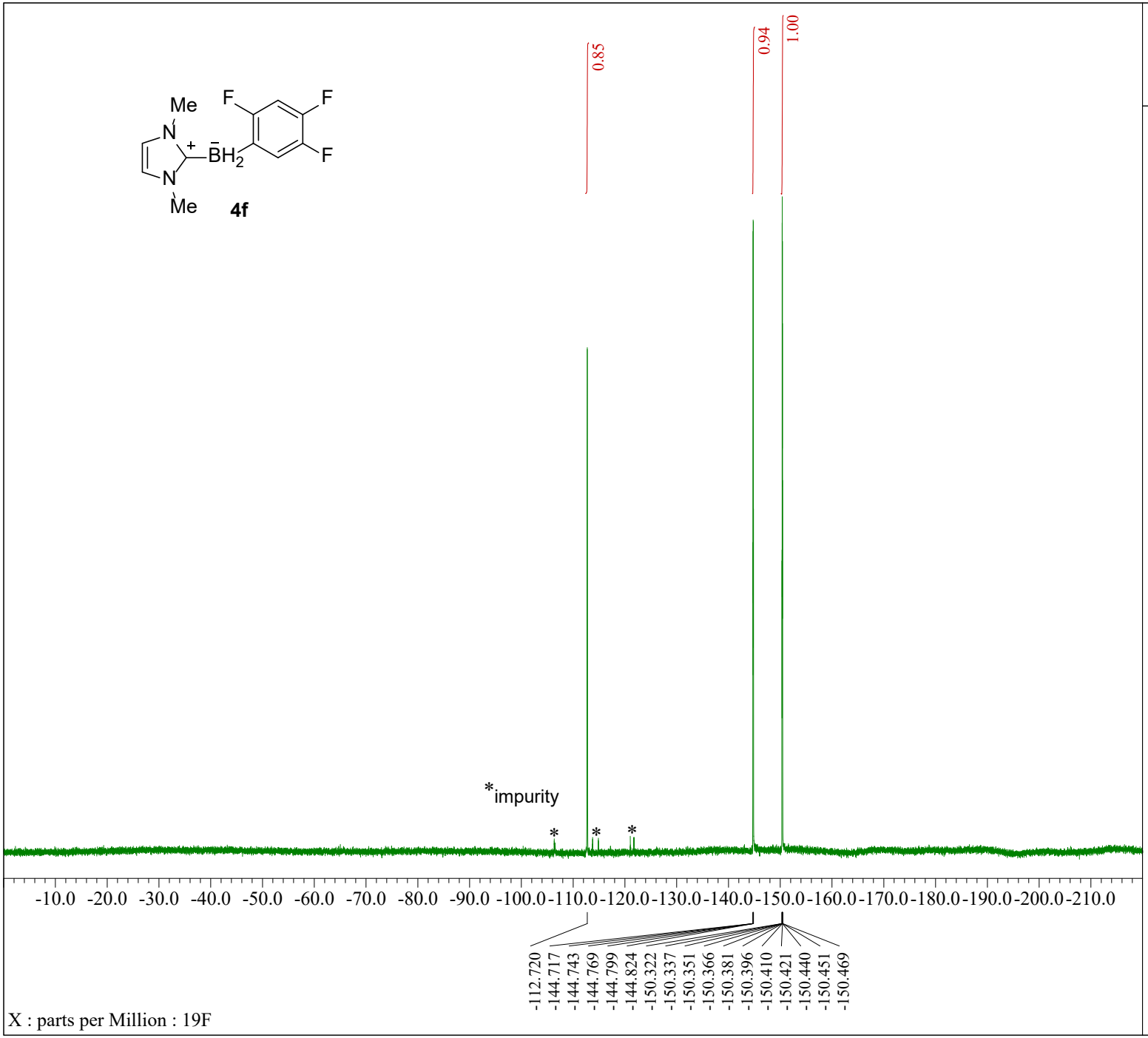
JEOL Solutions for Innovation

```
Filename          = KT-94-purified-11B-1.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#667474
Solvent           = CHLOROFORM-D
Creation_Time     = 19-DEC-2018 18:17:13
Revision_Time     = 31-JAN-2020 10:44:14
Current_Time      =  6-FEB-2020 10:37:55

Comment           = KT-94-purified-11B
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 11B
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = JNM-ECA600

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.54001664[s]
X_Domain          = 11B
X_Freq            = 192.5593054[MHz]
X_Offset          = 0[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 1.85179479[Hz]
X_Sweep           = 60.67961165[kHz]
Irr_Domain        = 11B
Irr_Freq          = 192.5593054[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 11B
Tri_Freq          = 192.5593054[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 32
Total_Scans       = 32

Relaxation_Delay  = 2[s]
Recvr_Gain        = 38
Temp_Get          = 19.2[dC]
X_90_Width        = 24[us]
X_Acq_Time        = 0.54001664[s]
X_Angle           = 45[deg]
X_Atn             = 6.9[dB]
X_Pulse           = 12[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 2.54001664[s]
```



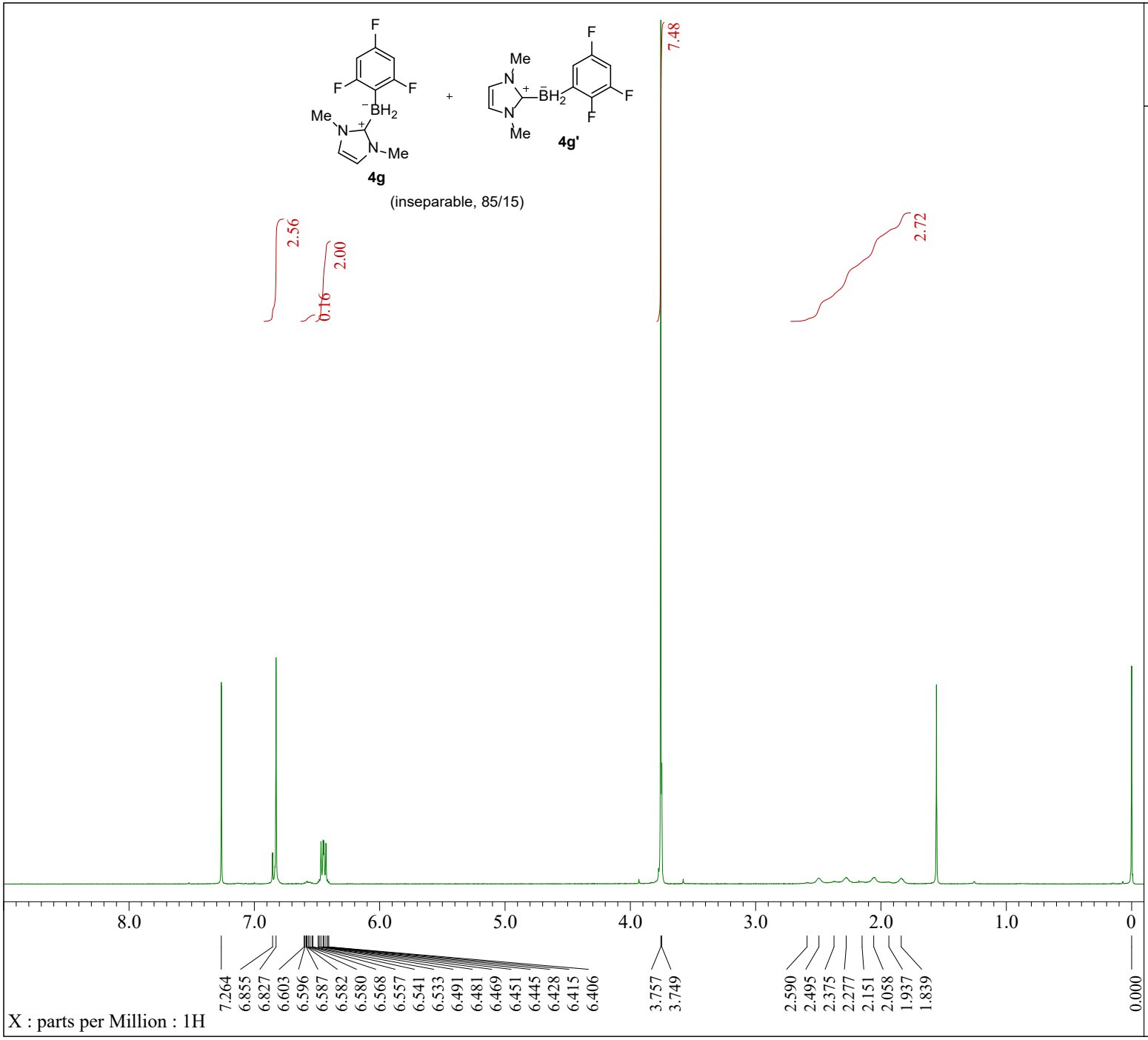
JEOL Solutions for Innovation

```
Filename         = KT-94-purified-19F_1-2.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 13:49:51
Revision_Time    =  6-FEB-2020 10:38:33
Current_Time     =  6-FEB-2020 10:39:21

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 65536
Dim_Title        = 19F
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
X_Prescans       = 4
X_Sweep          = 90.90909091[kHz]
Scans            = 16

Temp_Get         = 298.1339[K]
Filter_Factor    = 220
```



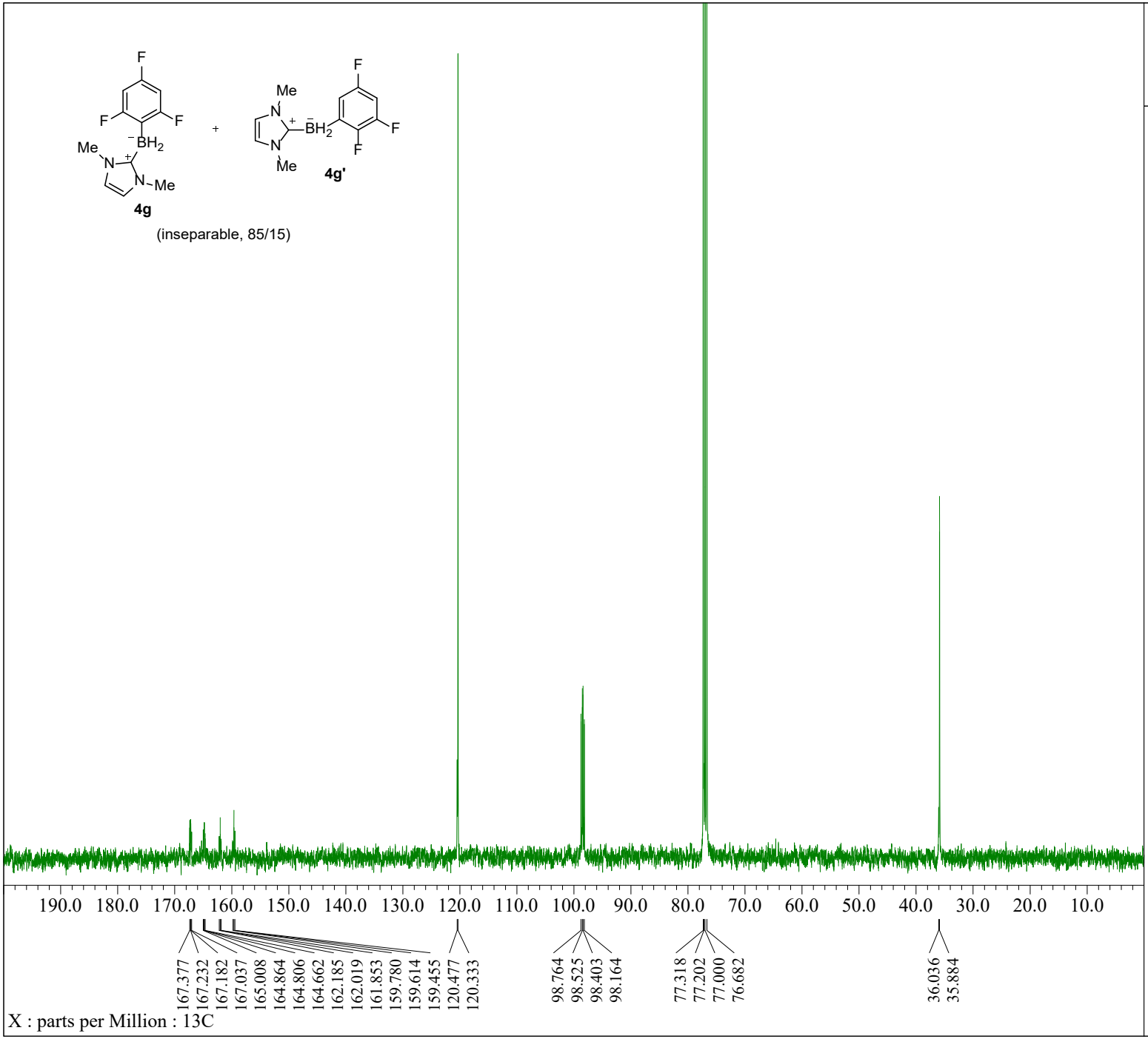
JEOL Solutions for Innovation

```
Filename          = KT-101-purified-1H-2.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#707502
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2019 19:39:41
Revision_Time     = 31-JAN-2020 11:20:06
Current_Time      =  6-FEB-2020 10:41:49

Comment           = KT-101-purified-1H
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 2[s]
Recvr_Gain        = 48
Temp_Get          = 20.2[dC]
X_90_Width        = 9.575[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 0.7[dB]
X_Pulse           = 4.7875[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 4.18365952[s]
```



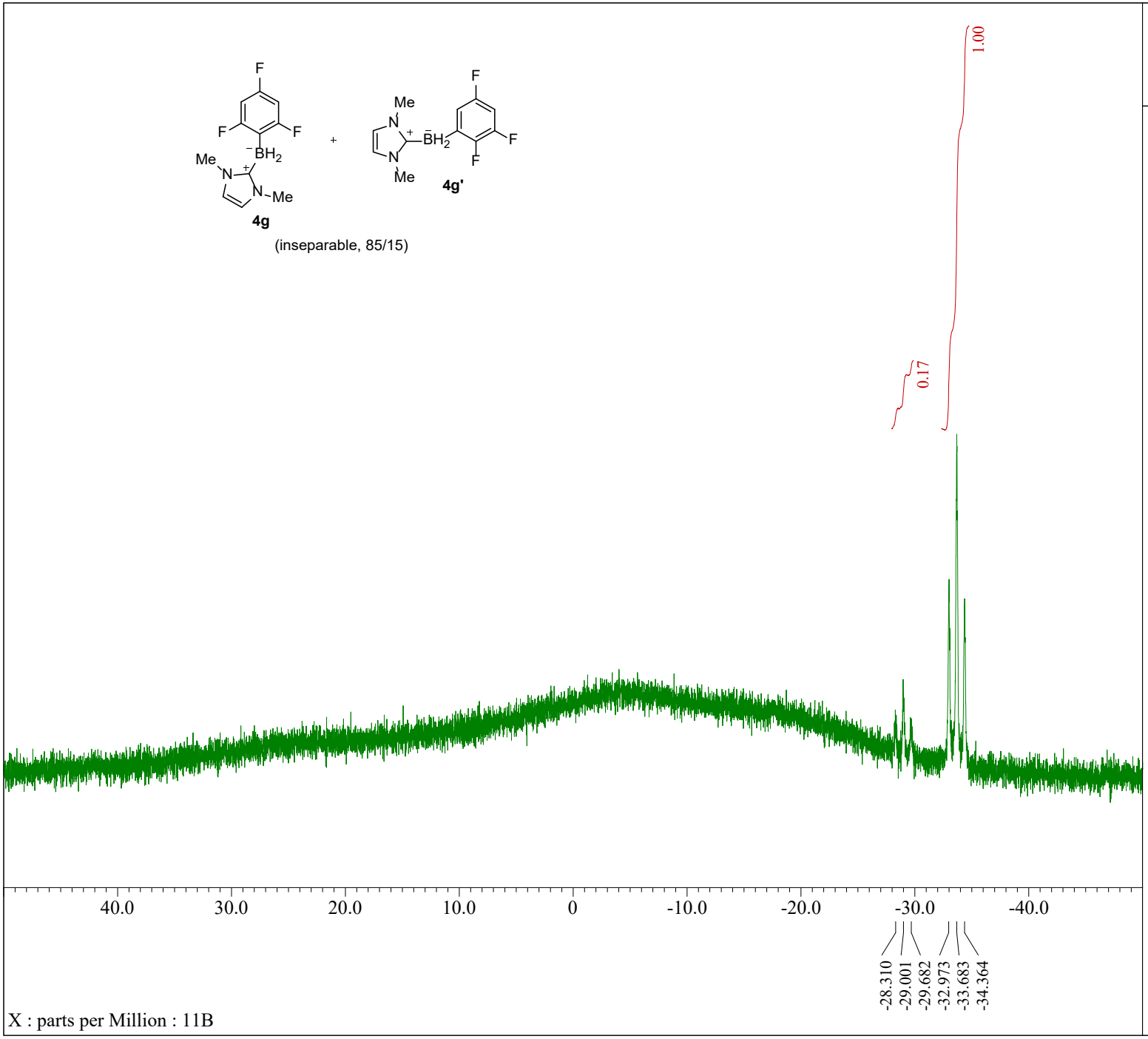
JEOL Solutions for Innovation

```
Filename        = KT-101-purified-13C_1-1.jdf
Author          = nmrsu
Experiment      = zgpg30
Sample_Id       = Parameter file, TopSpin 4.0.
Solvent         = CHLOROFORM-D
Creation_Time   =  8-JAN-2020 16:48:49
Revision_Time   = 31-JAN-2020 11:58:22
Current_Time    =  6-FEB-2020 10:42:43

Comment         = Parameter file, TopSpin 4.0.
Data_Format     = 1D REAL
Dim_Size        = 32768
Dim_Title       = 13C
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 13C
X_Freq          = 100.6228298[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 10.061277[kHz]
X_Points        = 32768
X_Prescans      = 4
X_Sweep         = 23.80952381[kHz]
Scans           = 512

Temp_Get        = 301.1496[K]
Filter_Factor   = 840
```



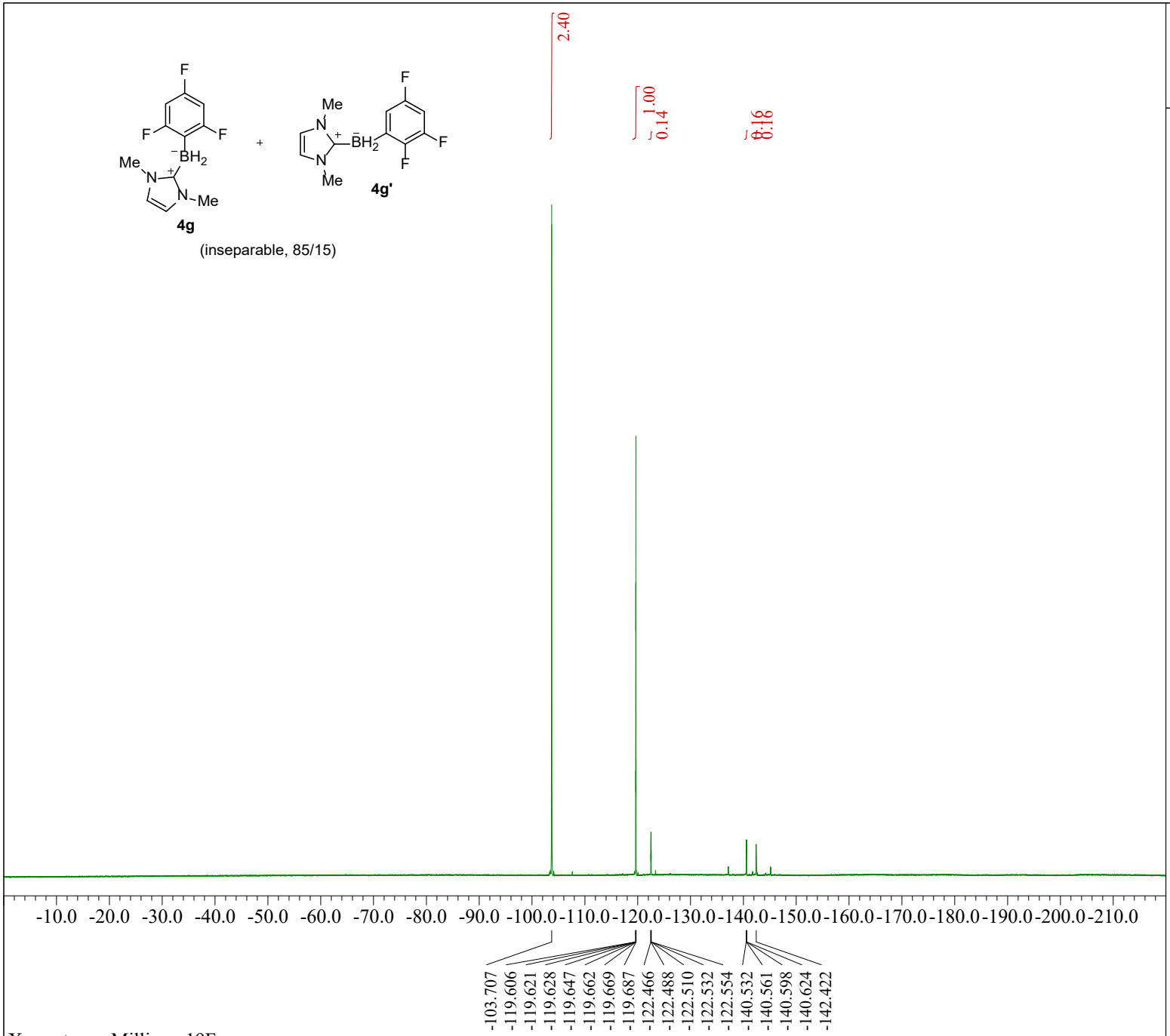
JEOL Solutions for Innovation

```
Filename          = KT-101-purified-11B-1.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#707595
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2019 19:42:54
Revision_Time     = 31-JAN-2020 12:03:41
Current_Time      =  6-FEB-2020 10:43:39

Comment           = KT-101-purified-11B
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 11B
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 0.8126464[s]
X_Domain          = 11B
X_Freq            = 128.26613597[MHz]
X_Offset          = 0[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 1.23054751[Hz]
X_Sweep           = 40.32258065[kHz]
Irr_Domain        = 11B
Irr_Freq          = 128.26613597[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 11B
Tri_Freq          = 128.26613597[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 32
Total_Scans       = 32

Relaxation_Delay  = 2[s]
Recvr_Gain        = 48
Temp_Get          = 20.3[dC]
X_90_Width        = 22[us]
X_Acq_Time        = 0.8126464[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 11[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 2.8126464[s]
```



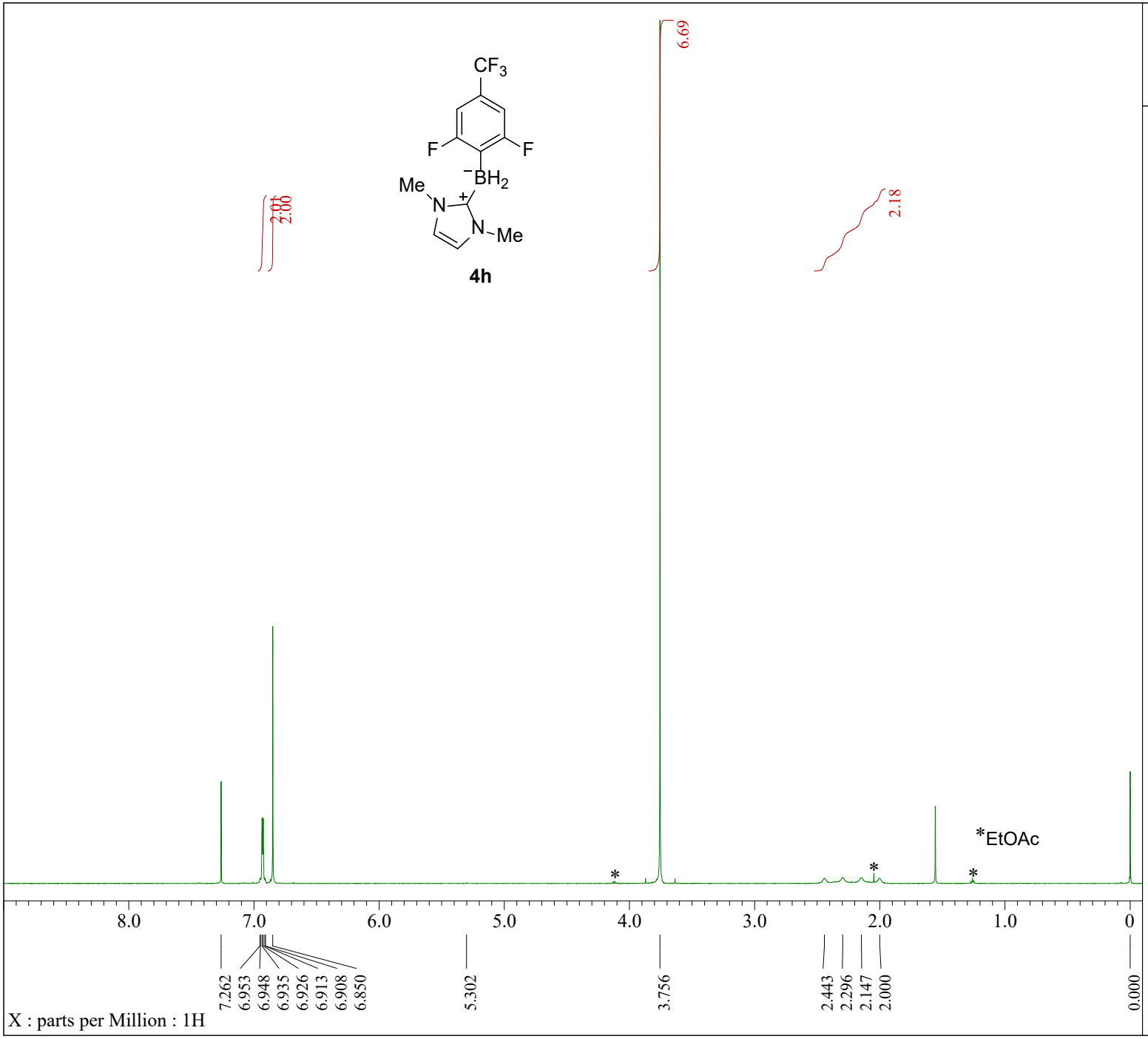
JEOL Solutions for Innovation

```
Filename         = KT-101-purified-19F_1-1.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 16:49:33
Revision_Time    = 31-JAN-2020 12:11:02
Current_Time     =  6-FEB-2020 10:44:31

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 65536
Dim_Title        = 19F
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
X_Prescans       = 4
X_Sweep          = 90.90909091[kHz]
Scans            = 16

Temp_Get         = 301.1496[K]
Filter_Factor    = 220
```



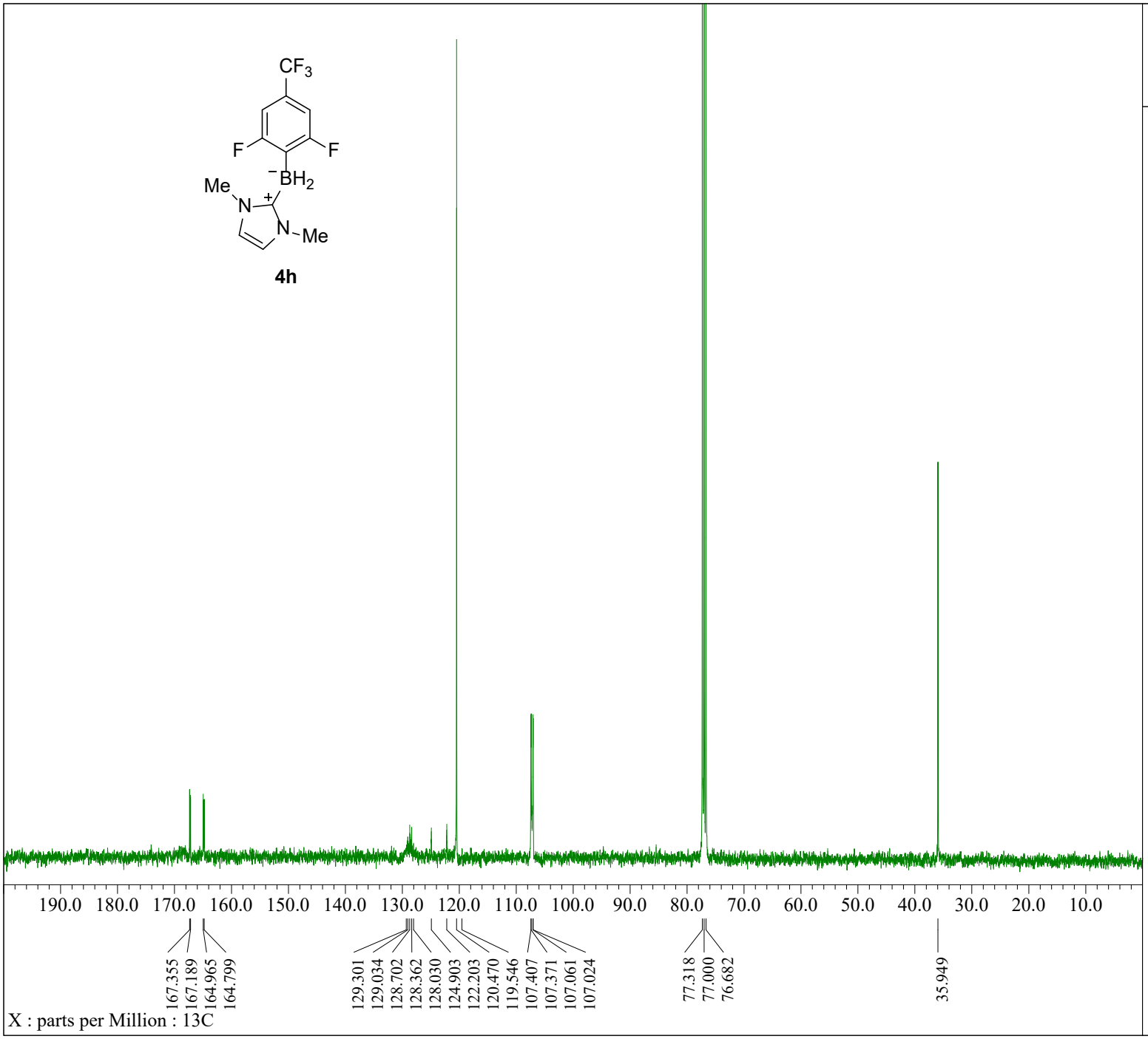
JEOL Solutions for Innovation

```
Filename          = KT-138-purified-1H-2.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#488116
Solvent           = CHLOROFORM-D
Creation_Time     = 15-MAR-2019 13:14:35
Revision_Time     =  6-FEB-2020 10:45:21
Current_Time      =  6-FEB-2020 10:45:41

Comment           = KT-138-purified-1H
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = JNM-ECA600

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.9097984[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.34366642[Hz]
X_Sweep           = 11.26126126[kHz]
Irr_Domain        = 1H
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 2[s]
Recvr_Gain        = 48
Temp_Get          = 19.5[dC]
X_90_Width        = 13.6[us]
X_Acq_Time        = 2.9097984[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.8[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 4.9097984[s]
```



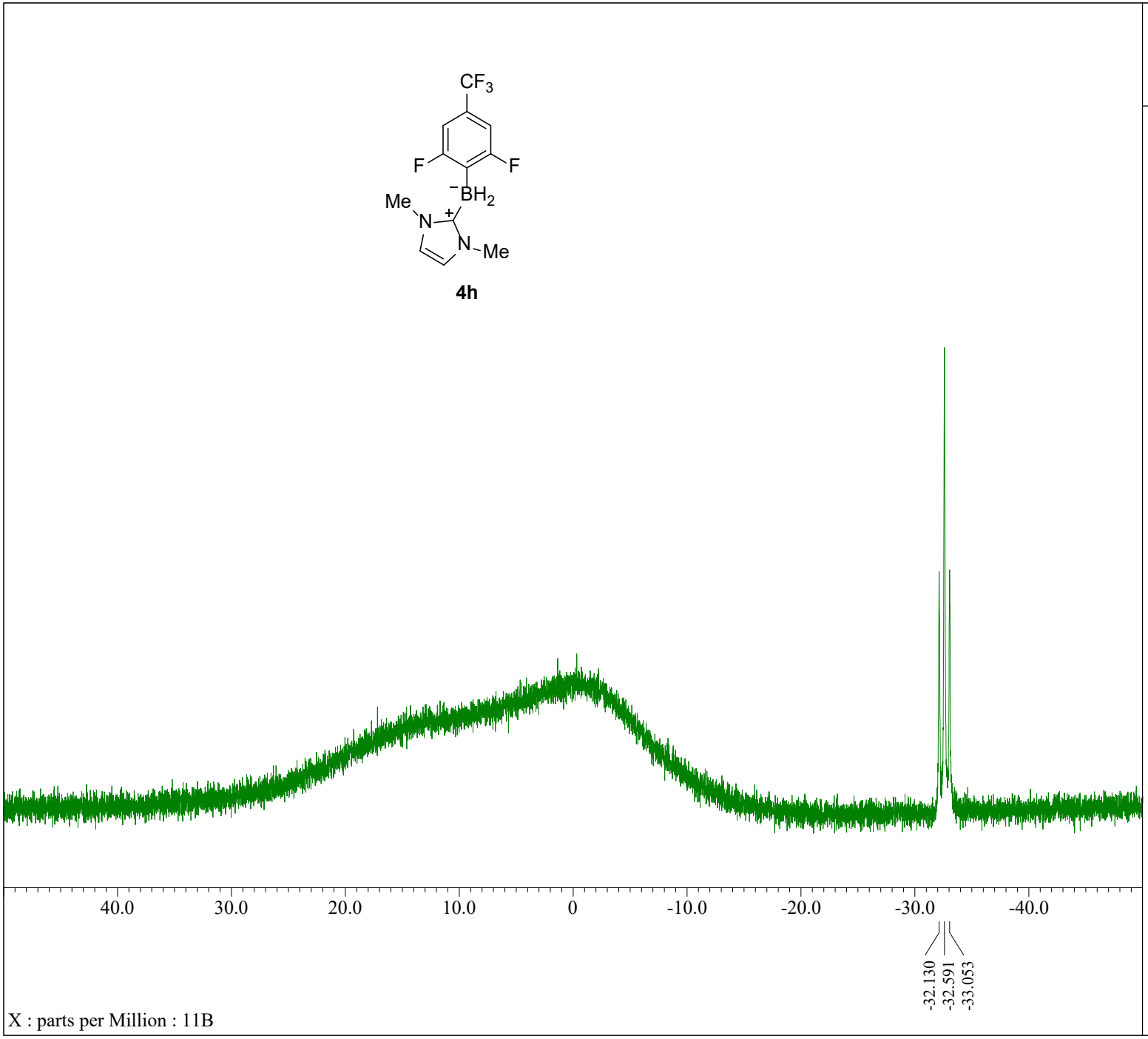
JEOL Solutions for Innovation

```
Filename         = KT-138-purified-13C_1-1.jdf
Author           = nmrsu
Experiment       = zgpg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 17:39:32
Revision_Time    = 31-JAN-2020 13:36:48
Current_Time     =  6-FEB-2020 10:46:32

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 32768
Dim_Title        = 13C
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 13C
X_Freq           = 100.6228298[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 10.061277[kHz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 23.80952381[kHz]
Scans            = 1024

Temp_Get         = 298.4157[K]
Filter_Factor    = 840
```



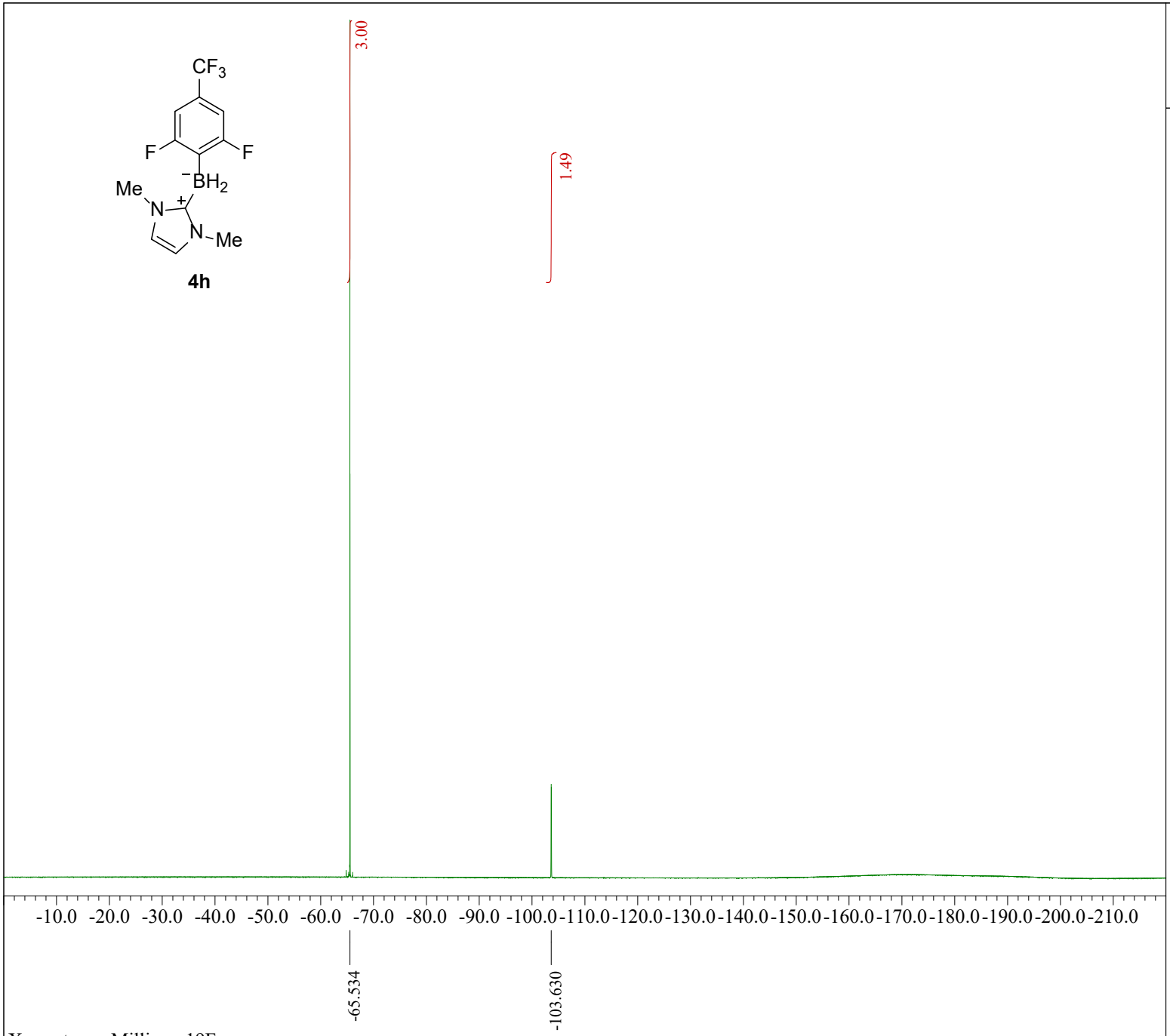
JEOL Solutions for Innovation

```
Filename          = KT-138-purified-11B-1.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#488178
Solvent           = CHLOROFORM-D
Creation_Time     = 15-MAR-2019 13:16:47
Revision_Time     = 31-JAN-2020 13:40:47
Current_Time      =  6-FEB-2020 11:05:48

Comment           = KT-138-purified-11B
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 11B
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = JNM-ECA600

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.54001664[s]
X_Domain          = 11B
X_Freq            = 192.5593054[MHz]
X_Offset          = 0[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 1.85179479[Hz]
X_Sweep           = 60.67961165[kHz]
Irr_Domain        = 11B
Irr_Freq          = 192.5593054[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 11B
Tri_Freq          = 192.5593054[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 2[s]
Recvr_Gain        = 36
Temp_Get          = 19.5[dC]
X_90_Width        = 24[us]
X_Acq_Time        = 0.54001664[s]
X_Angle           = 45[deg]
X_Atn             = 6.9[dB]
X_Pulse           = 12[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 2.54001664[s]
```



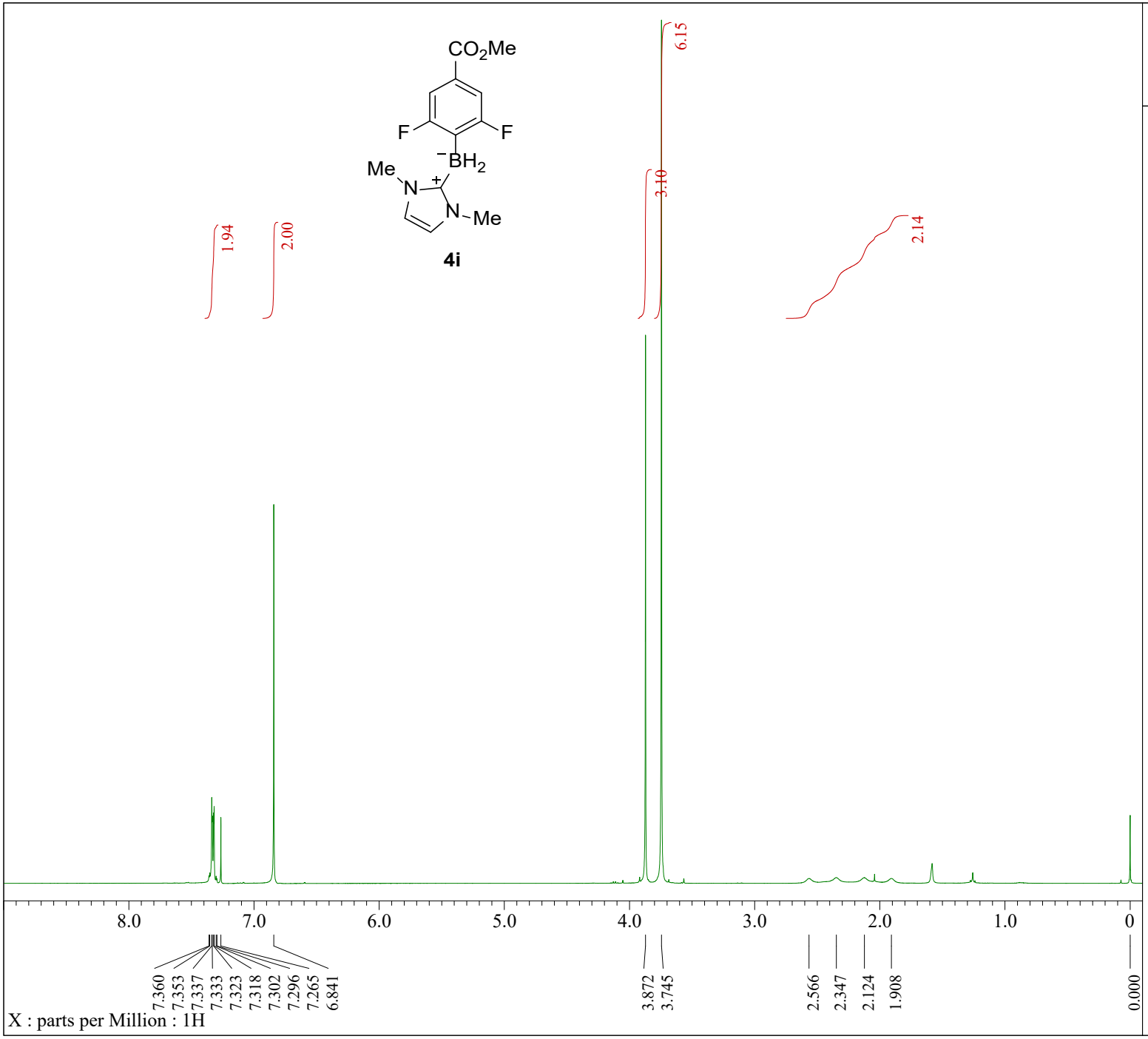
JEOL Solutions for Innovation

```
Filename         = KT-138-purified-19F_1-1.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 14:51:09
Revision_Time    = 31-JAN-2020 13:46:10
Current_Time     =  6-FEB-2020 11:06:53

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 65536
Dim_Title        = 19F
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
X_Prescans       = 4
X_Sweep          = 90.90909091[kHz]
Scans            = 16

Temp_Get         = 298.1476[K]
Filter_Factor    = 220
```



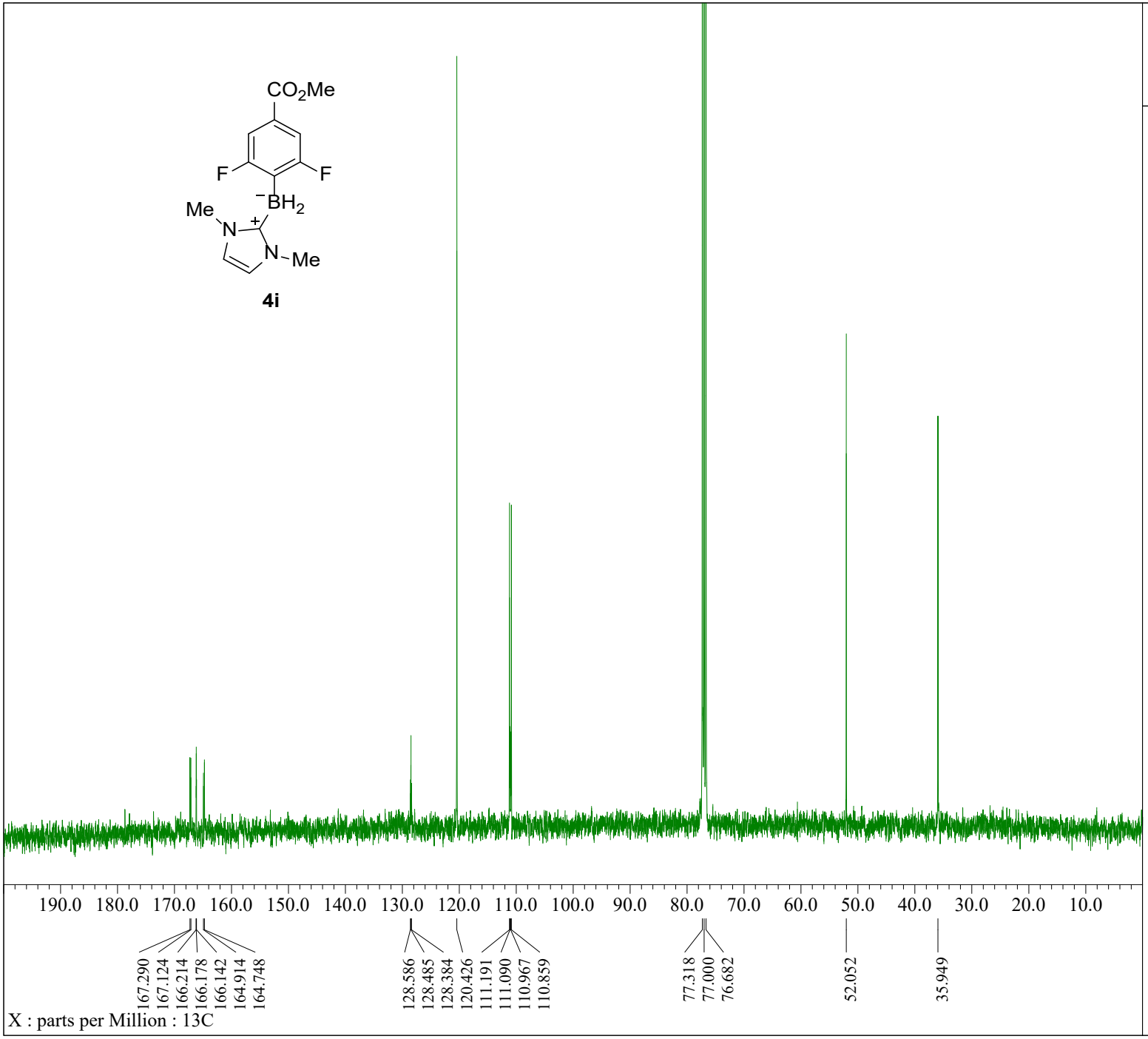
JEOL Solutions for Innovation

```
Filename         = KT-161-purified-1H_1-2.jdf
Author           = nmrsu
Experiment       = zg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 18:54:41
Revision_Time    =  6-FEB-2020 11:07:55
Current_Time     =  6-FEB-2020 11:08:20

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 1H
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 1H
X_Freq           = 400.1324708[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 2.470802[kHz]
X_Points         = 32768
X_Prescans       = 2
X_Sweep          = 8.19672131[kHz]
Scans            = 16

Temp_Get         = 301.1528[K]
Filter_Factor    = 2440
```



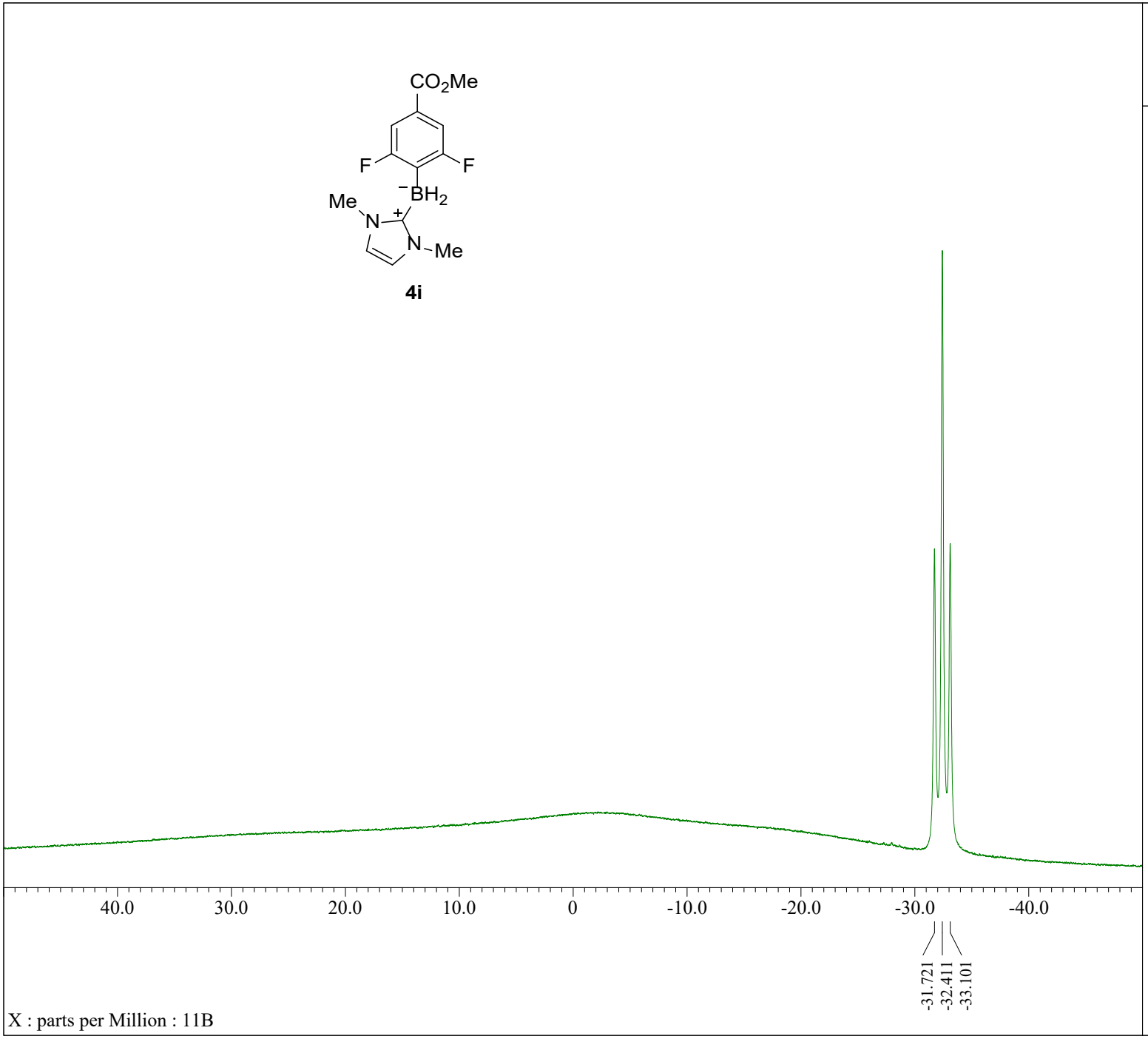
JEOL Solutions for Innovation

```
Filename         = KT-161-purified-13C_1-1.jdf
Author           = nmrsu
Experiment       = zgpg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 18:53:43
Revision_Time    = 31-JAN-2020 16:02:00
Current_Time     =  6-FEB-2020 11:09:31

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 13C
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 13C
X_Freq           = 100.6228298[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 10.061277[kHz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 23.80952381[kHz]
Scans            = 512

Temp_Get         = 301.1502[K]
Filter_Factor    = 840
```



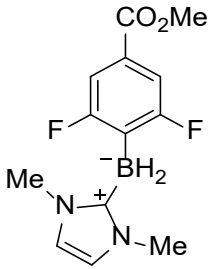
JEOL Solutions for Innovation

```
Filename        = KT-161-purified-11B_1-1.jdf
Author          = nmrsu
Experiment      = zg
Sample_Id       = Parameter file, TopSpin 4.0.
Solvent         = CHLOROFORM-D
Creation_Time   =  8-JAN-2020 18:53:19
Revision_Time   = 31-JAN-2020 16:11:02
Current_Time    =  6-FEB-2020 11:10:16

Comment         = Parameter file, TopSpin 4.0.
Data_Format     = 1D COMPLEX
Dim_Size        = 32768
Dim_Title       = 11B
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 11B
X_Freq          = 128.37760517[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 0[Hz]
X_Points        = 32768
X_Prescans      = 4
X_Sweep         = 25.90673575[kHz]
Scans           = 128

Temp_Get        = 301.1498[K]
Filter_Factor   = 772
```



4i

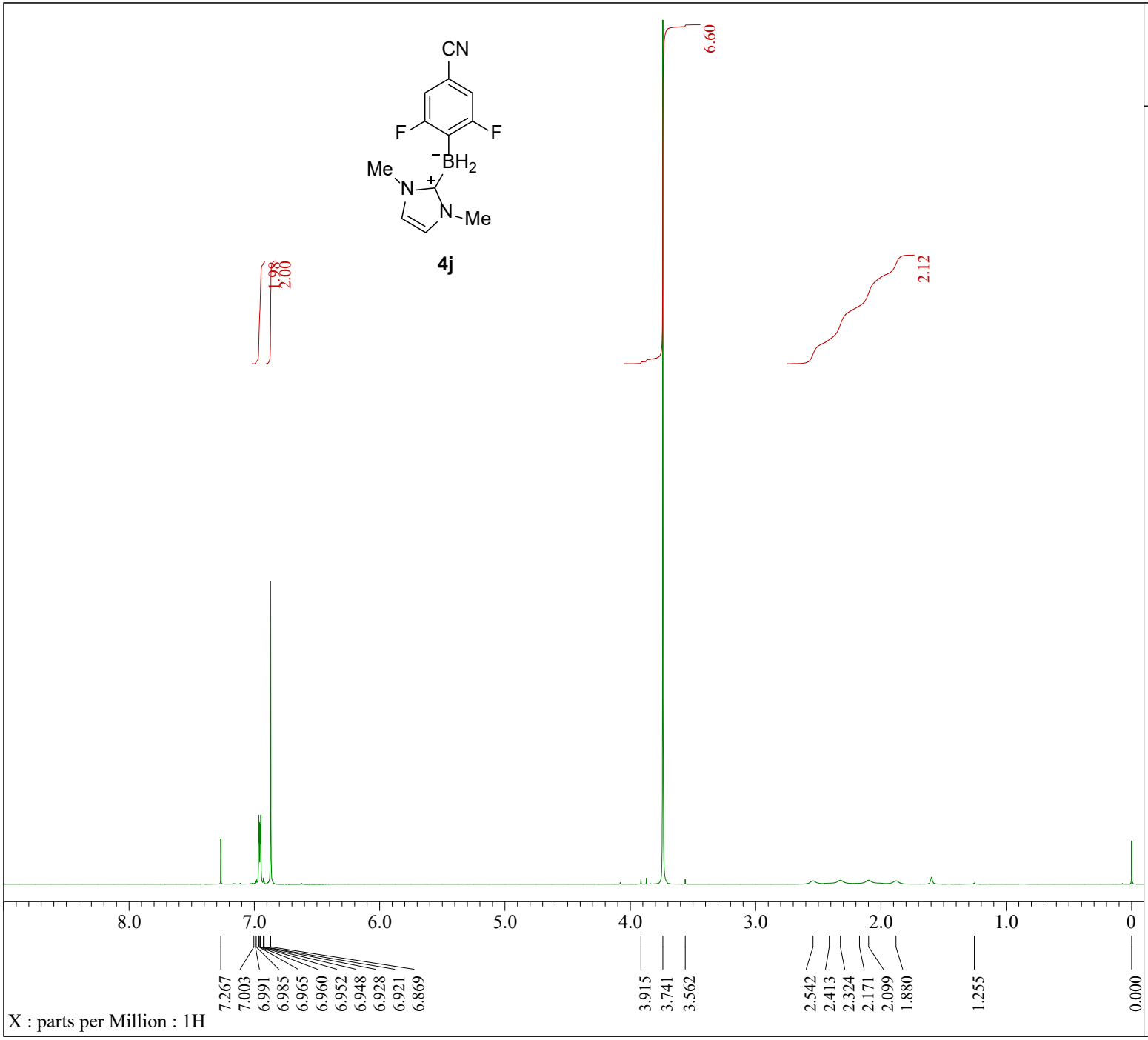
JEOL Solutions for Innovation

```
Filename         = KT-161-purified-19F_1-1.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 18:54:24
Revision_Time    = 31-JAN-2020 16:13:28
Current_Time     =  6-FEB-2020 11:11:11

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 65536
Dim_Title        = 19F
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
X_Prescans       = 4
X_Sweep          = 90.90909091[kHz]
Scans            = 16

Temp_Get         = 301.1495[K]
Filter_Factor    = 220
```



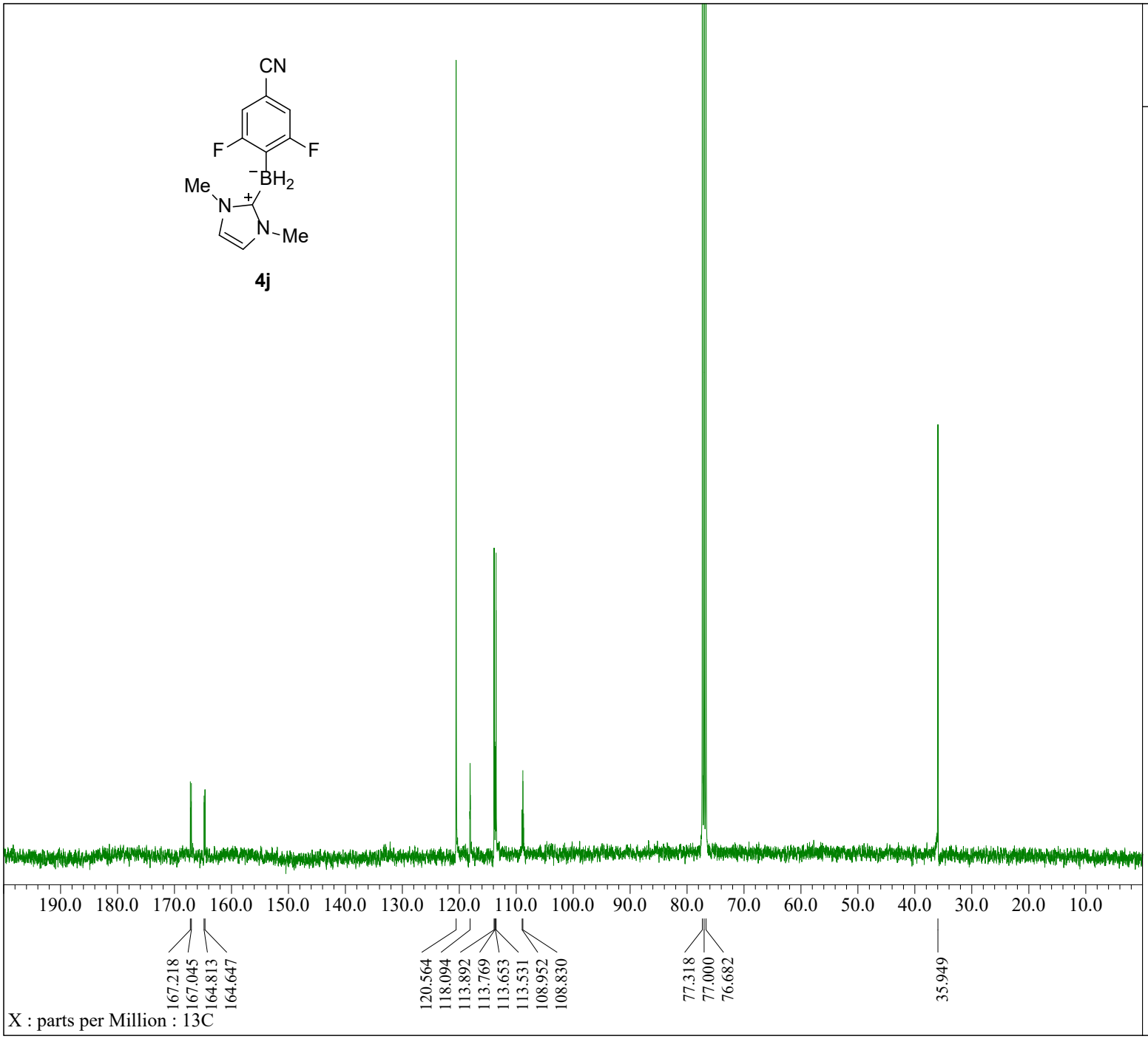
JEOL Solutions for Innovation

```
Filename         = KT-143-repurified-1H-re_1-1.
Author           = nmrsu
Experiment       = zg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  3-FEB-2020 14:18:01
Revision_Time    =  4-FEB-2020 09:24:05
Current_Time     =  6-FEB-2020 11:12:25

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 1H
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 1H
X_Freq           = 400.1324708[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 2.470802[kHz]
X_Points         = 32768
X_Prescans       = 2
X_Sweep          = 8.19672131[kHz]
Scans            = 16

Temp_Get         = 298.1462[K]
Filter_Factor    = 2440
```



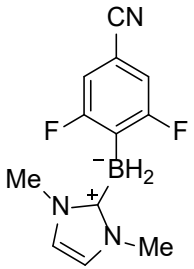
JEOL Solutions for Innovation

```
Filename         = KT-143-repurified-13C-re_1-1
Author           = nmrsu
Experiment       = zgpg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  3-FEB-2020 22:00:40
Revision_Time    =  4-FEB-2020 09:27:39
Current_Time     =  6-FEB-2020 11:13:23

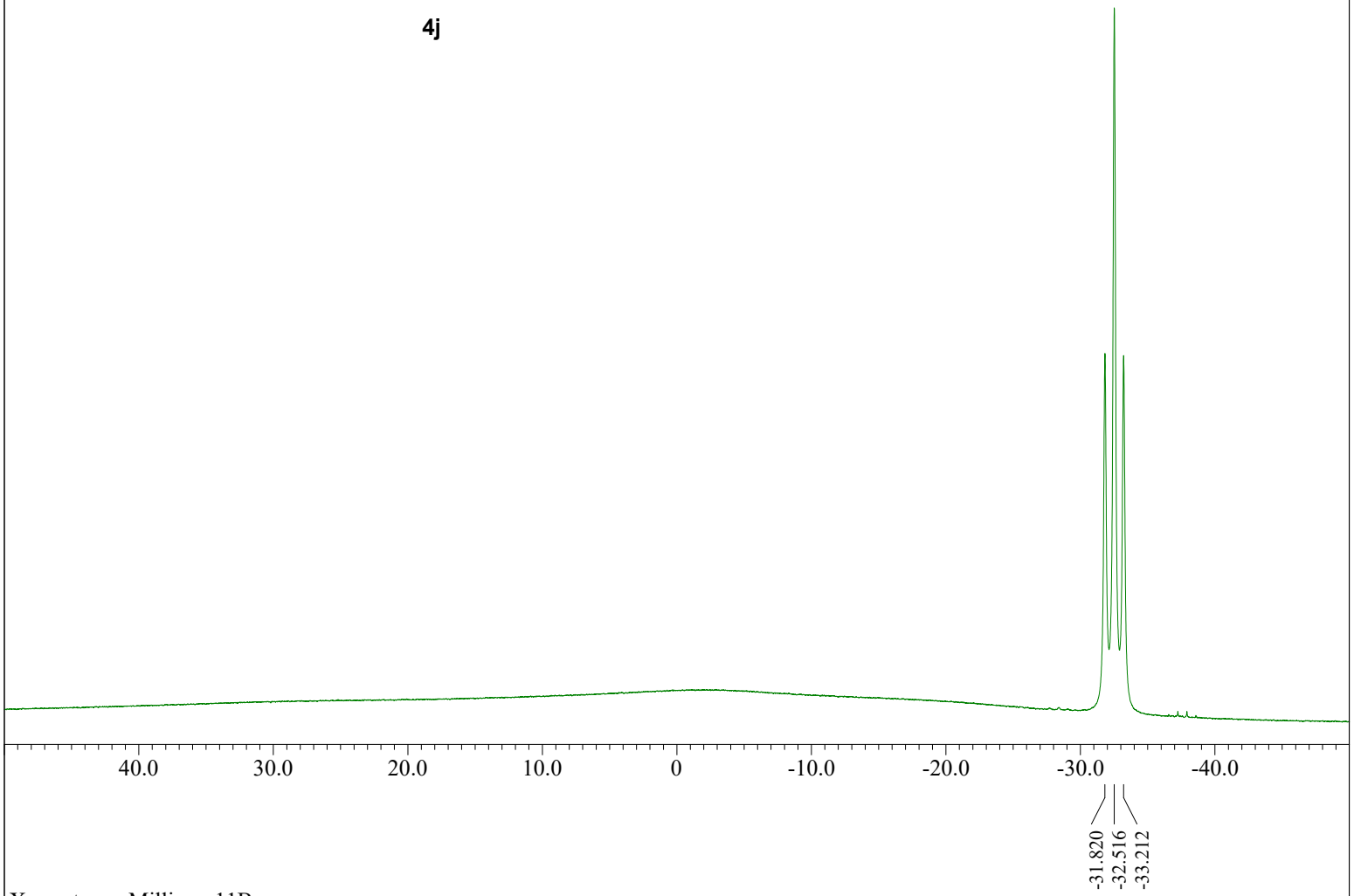
Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 32768
Dim_Title        = 13C
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 13C
X_Freq           = 100.6228298[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 10.061277[kHz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 23.80952381[kHz]
Scans            = 742

Temp_Get         = 298.1424[K]
Filter_Factor    = 840
```



4j



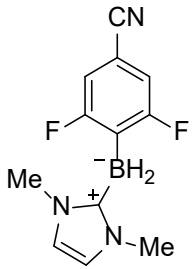
X : parts per Million : 11B

```
Filename        = KT-143-repurified-11B_1-1.jd
Author          = nmrsu
Experiment      = zg
Sample_Id       = Parameter file, TopSpin 4.0.
Solvent         = CHLOROFORM-D
Creation_Time   =  8-JAN-2020 18:30:59
Revision_Time   = 31-JAN-2020 17:26:04
Current_Time    =  6-FEB-2020 11:26:39

Comment         = Parameter file, TopSpin 4.0.
Data_Format     = 1D COMPLEX
Dim_Size        = 32768
Dim_Title       = 11B
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 11B
X_Freq          = 128.37760517[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 0[Hz]
X_Points        = 32768
X_Prescans      = 4
X_Sweep         = 25.90673575[kHz]
Scans           = 128

Temp_Get        = 298.1493[K]
Filter_Factor   = 772
```



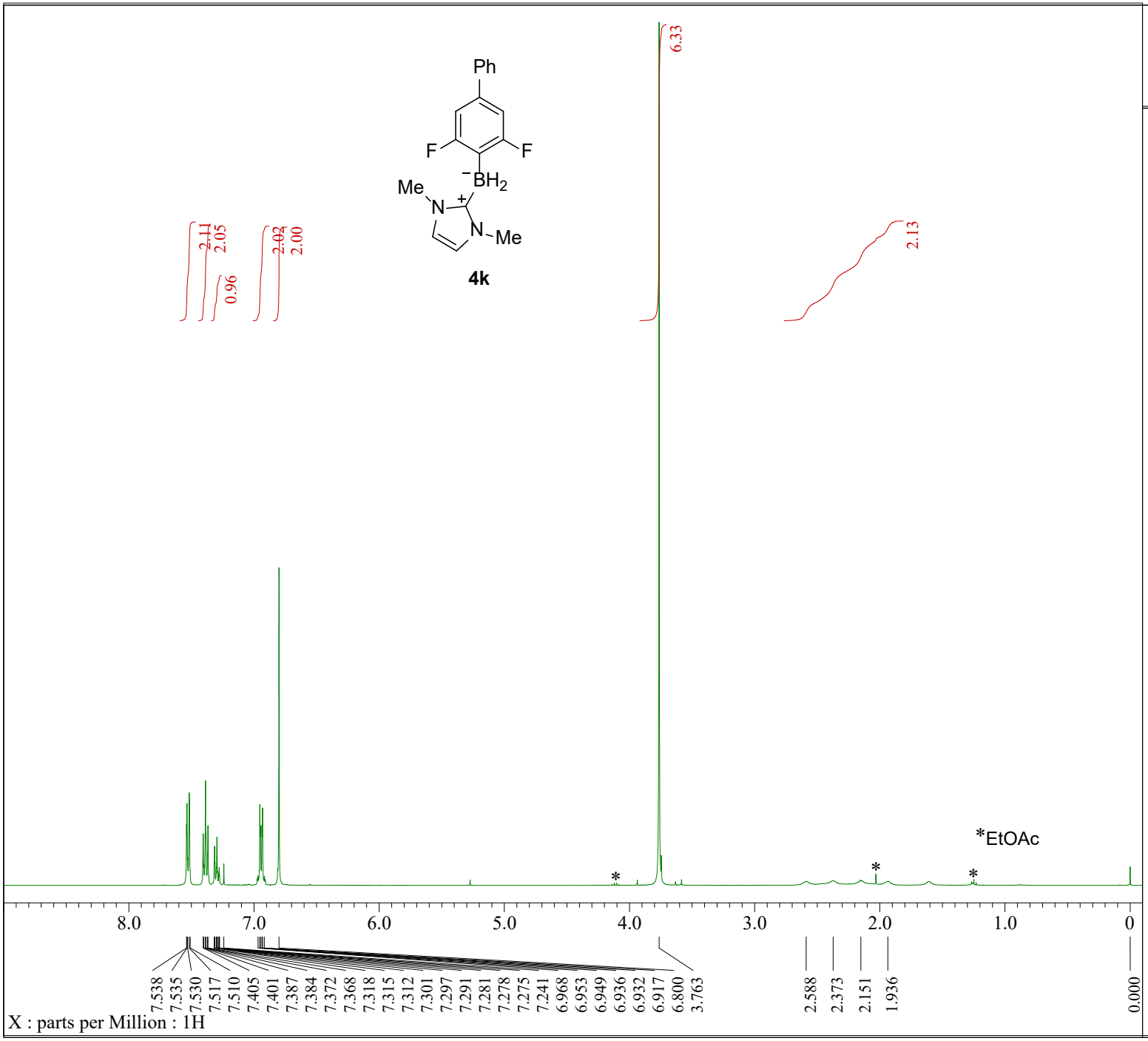
4j


```
Filename         = KT-143-repurified-19F_1-1.jd
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 18:33:09
Revision_Time    = 31-JAN-2020 17:27:55
Current_Time     =  6-FEB-2020 11:27:41

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 65536
Dim_Title        = 19F
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
X_Prescans       = 4
X_Sweep          = 90.90909091[kHz]
Scans            = 16

Temp_Get         = 298.1386[K]
Filter_Factor    = 220
```



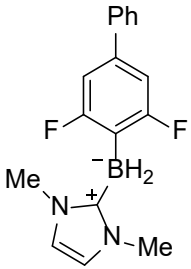
JEOL Solutions for Innovation

```
Filename         = KT-154-purified-1H_1-1.jdf
Author           = nmrsu
Experiment       = zg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 18:52:33
Revision_Time    = 31-JAN-2020 18:30:15
Current_Time     =  6-FEB-2020 13:06:33

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 1H
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength   = 9.39793489[T] (400[MHz])
X_Domain         = 1H
X_Freq           = 400.1324708[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 2.470802[kHz]
X_Points         = 32768
X_Prescans       = 2
X_Sweep          = 8.19672131[kHz]
Scans            = 16

Temp_Get         = 301.1497[K]
Filter_Factor    = 2440
```



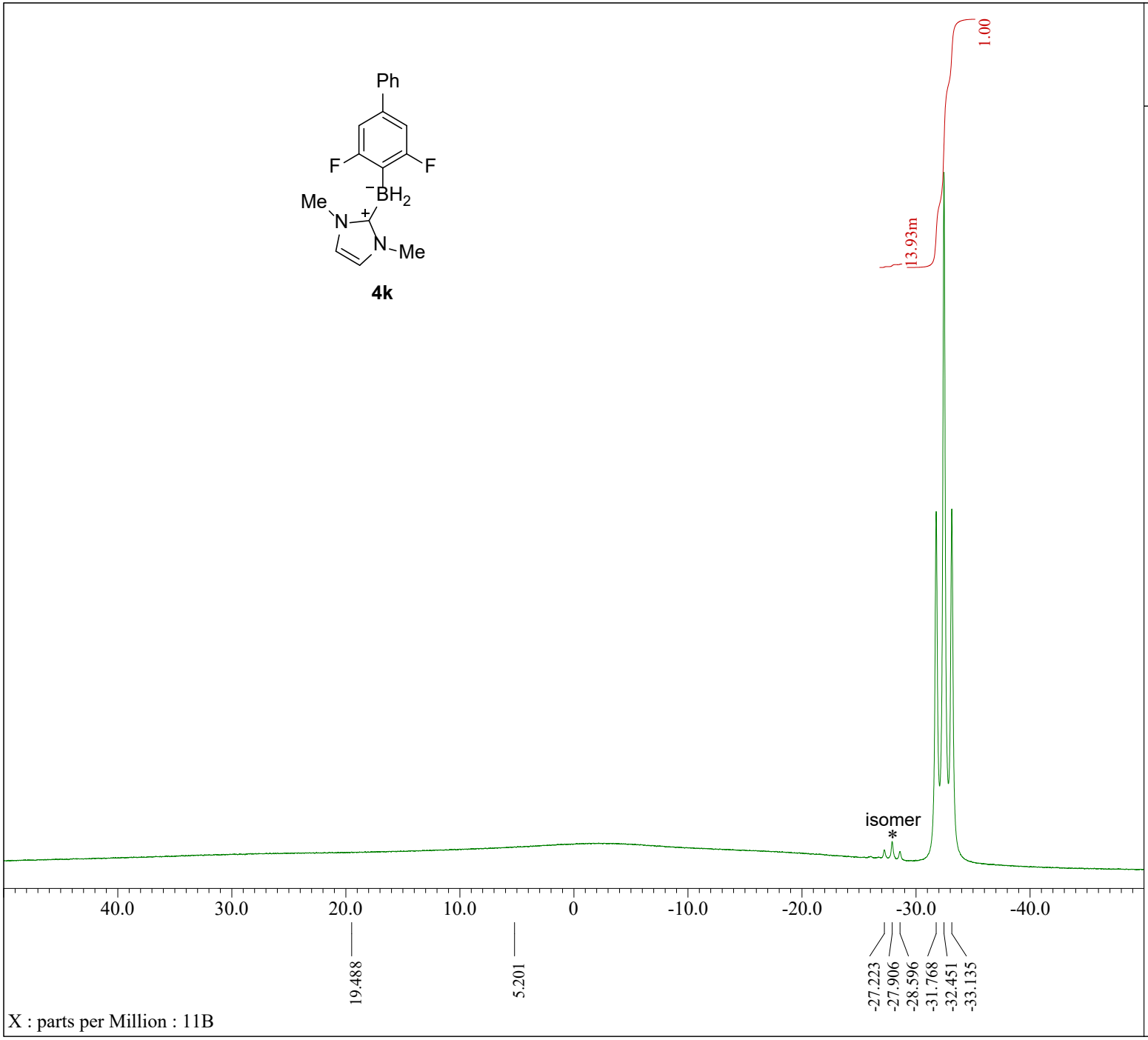
4k


```
Filename         = KT-154-purified-13C-re_1-1.j
Author           = nmrsu
Experiment       = zgpg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 18:49:38
Revision_Time    = 31-JAN-2020 18:37:12
Current_Time     =  6-FEB-2020 13:07:45

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 13C
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 13C
X_Freq           = 100.6228298[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 10.061277[kHz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 23.80952381[kHz]
Scans            = 459

Temp_Get         = 298.152[K]
Filter_Factor    = 840
```



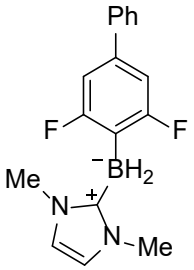
JEOL
Solutions for Innovation

```
Filename         = KT-154-purified-11B_1-2.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 18:48:50
Revision_Time    =  6-FEB-2020 13:08:56
Current_Time     =  6-FEB-2020 13:09:11

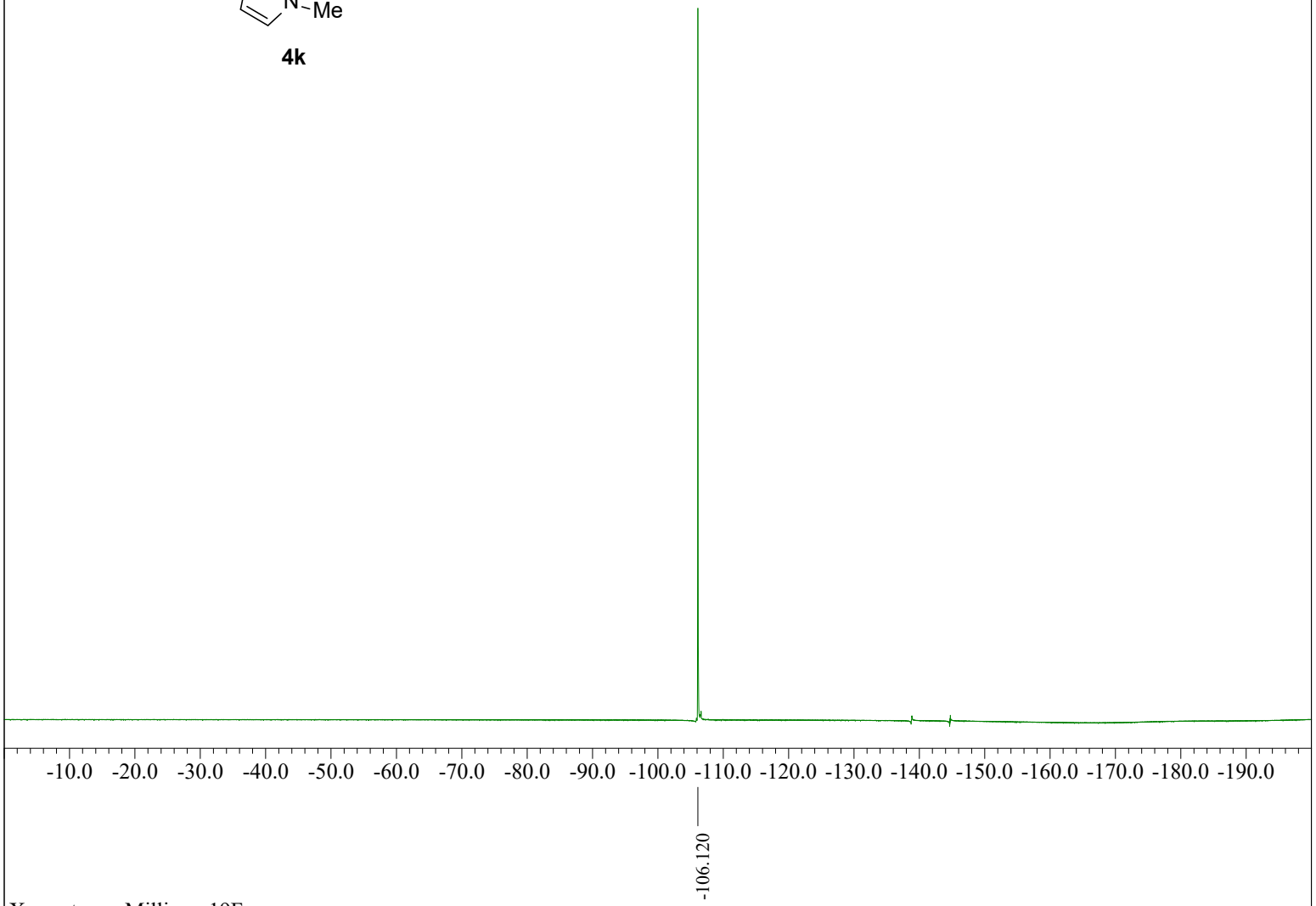
Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 11B
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 11B
X_Freq           = 128.37760517[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 0[Hz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 25.90673575[kHz]
Scans            = 128

Temp_Get         = 301.1503[K]
Filter_Factor    = 772
```



4k



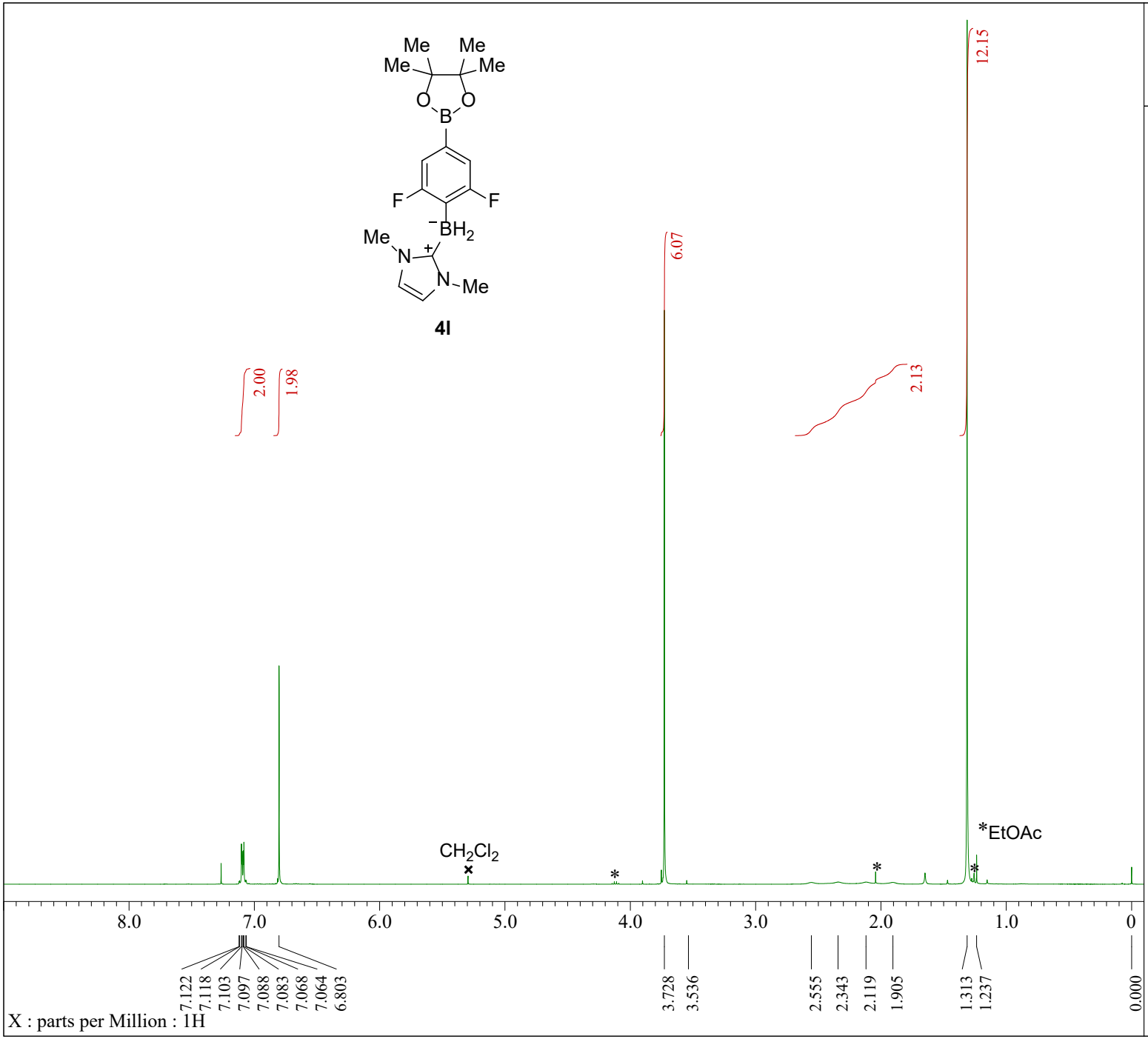
JEOL Solutions for Innovation

```
Filename         = KT-154-purified-19F_1-1.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 18:50:07
Revision_Time    = 31-JAN-2020 18:50:27
Current_Time     =  6-FEB-2020 13:10:08

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 65536
Dim_Title        = 19F
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
X_Prescans       = 4
X_Sweep          = 90.90909091[kHz]
Scans            = 16

Temp_Get         = 301.1476[K]
Filter_Factor    = 220
```



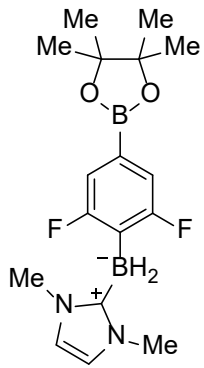
JEOL Solutions for Innovation

```
Filename         = KT-152-repurified-1H_1-1.jdf
Author           = nmrsu
Experiment       = zg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 18:44:07
Revision_Time    = 31-JAN-2020 19:17:23
Current_Time     =  6-FEB-2020 13:11:04

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 1H
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength   = 9.39793489[T] (400[MHz])
X_Domain         = 1H
X_Freq           = 400.1324708[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 2.470802[kHz]
X_Points         = 32768
X_Prescans       = 2
X_Sweep          = 8.19672131[kHz]
Scans            = 16

Temp_Get         = 298.1507[K]
Filter_Factor    = 2440
```



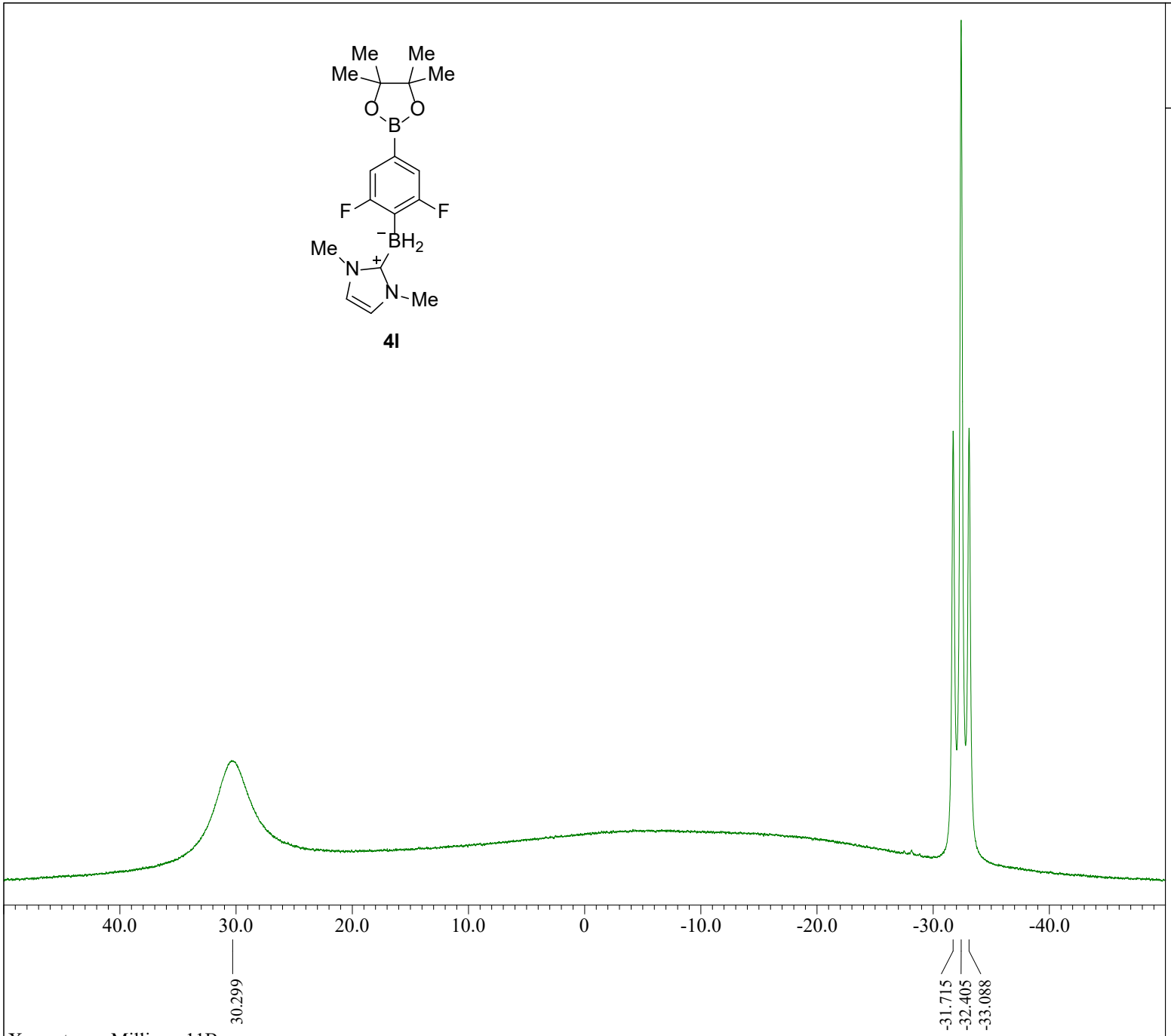
4l


```
Filename         = KT-152-repurified-13C_1-1.jd
Author           = nmrsu
Experiment       = zgpg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  8-JAN-2020 18:43:27
Revision_Time    = 31-JAN-2020 19:25:49
Current_Time     =  6-FEB-2020 13:12:14

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 13C
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 13C
X_Freq           = 100.6228298[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 10.061277[kHz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 23.80952381[kHz]
Scans            = 558

Temp_Get         = 298.1506[K]
Filter_Factor    = 840
```



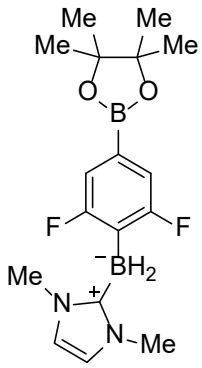
JEOL Solutions for Innovation

```
Filename        = KT-152-repurified-11B_1-1.jd
Author          = nmrsu
Experiment      = zg
Sample_Id       = Parameter file, TopSpin 4.0.
Solvent         = CHLOROFORM-D
Creation_Time   =  8-JAN-2020 18:42:48
Revision_Time   = 31-JAN-2020 19:29:45
Current_Time    =  6-FEB-2020 13:51:46

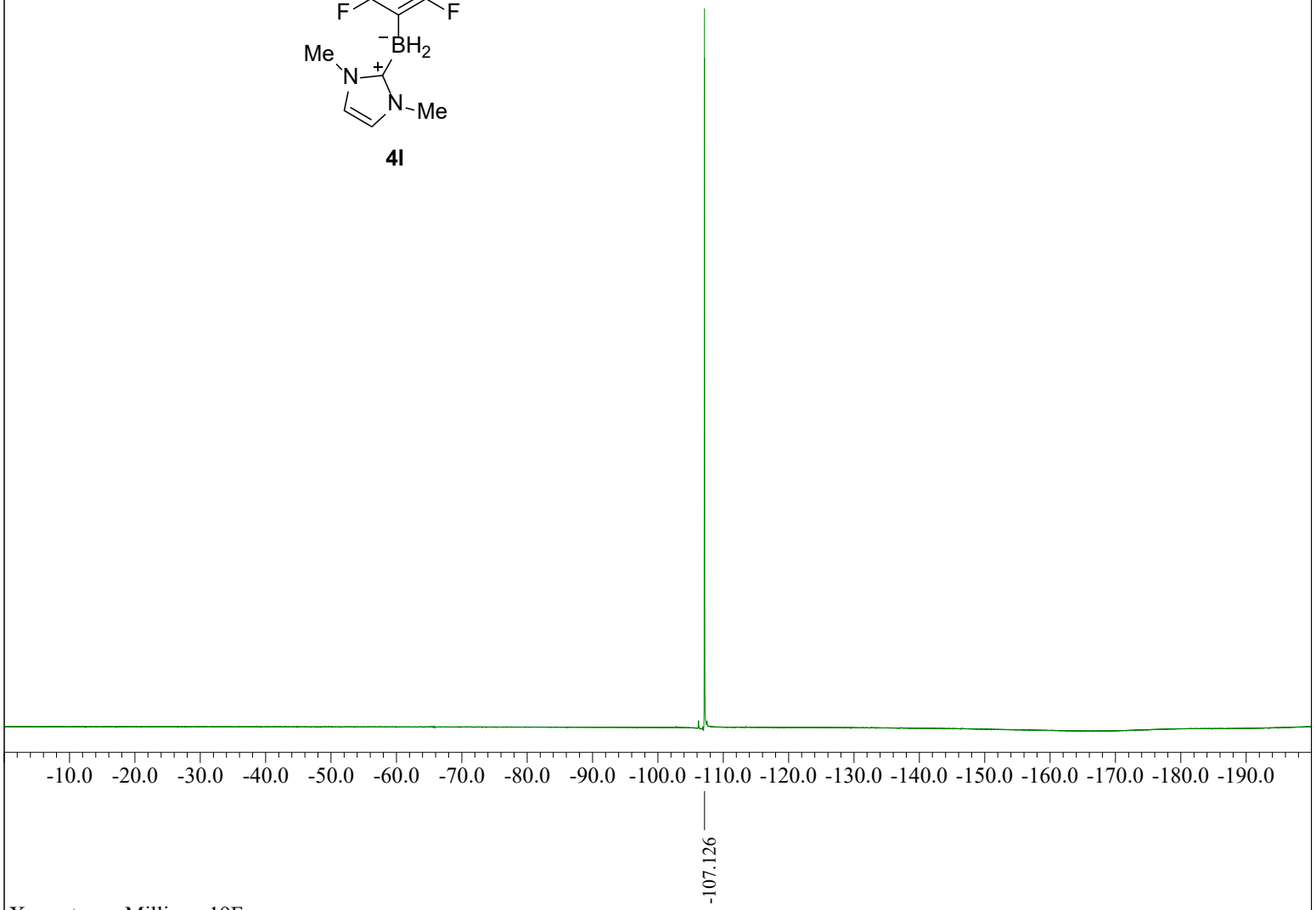
Comment         = Parameter file, TopSpin 4.0.
Data_Format     = 1D COMPLEX
Dim_Size        = 32768
Dim_Title       = 11B
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 11B
X_Freq          = 128.37760517[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 0[Hz]
X_Points        = 32768
X_Prescans      = 4
X_Sweep         = 25.90673575[kHz]
Scans           = 66

Temp_Get        = 298.1484[K]
Filter_Factor   = 772
```



4l

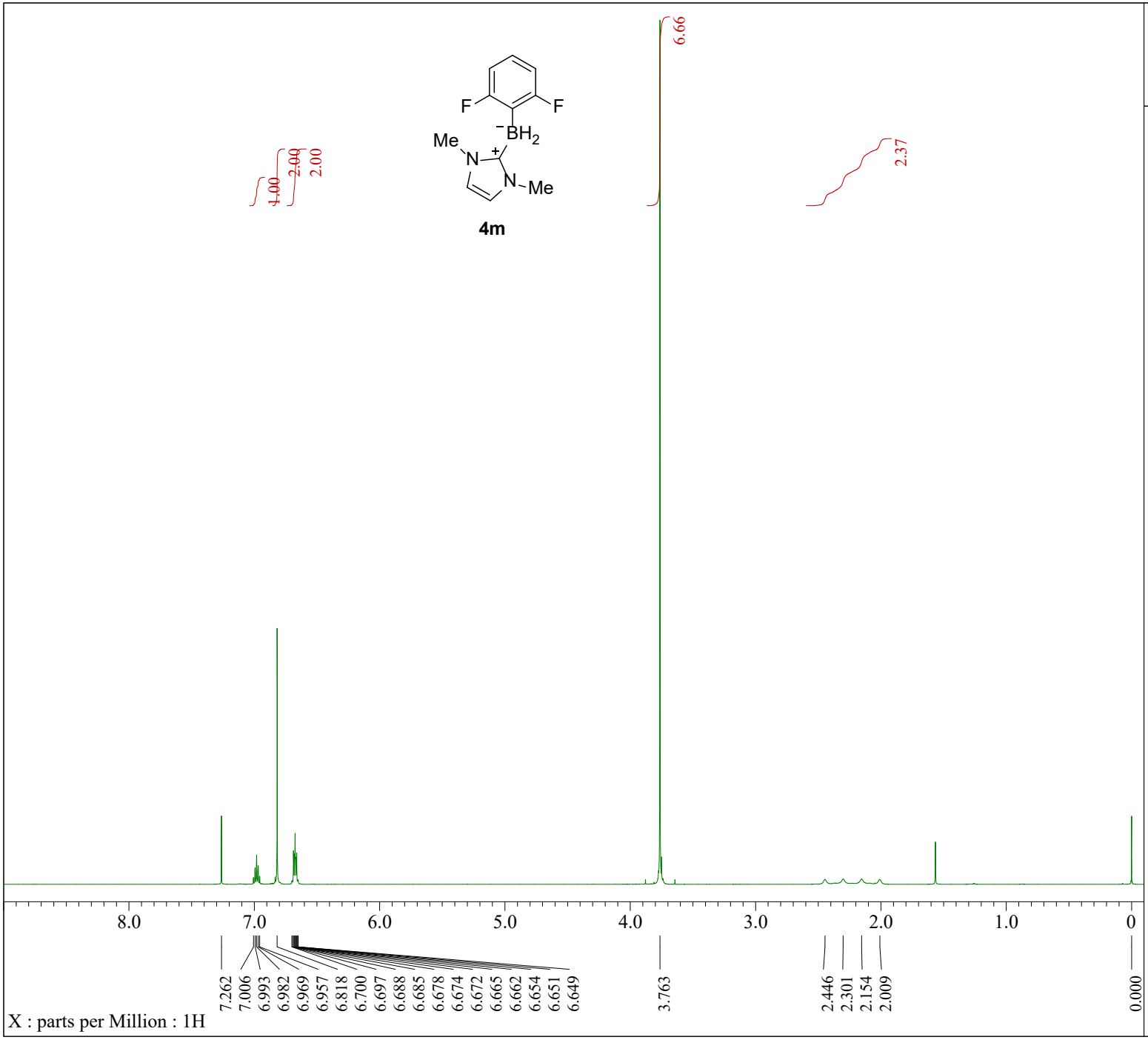



```
Filename        = KT-152-repurified-19F_1-1.jd
Author          = nmrsu
Experiment      = zg
Sample_Id       = Parameter file, TopSpin 4.0.
Solvent         = CHLOROFORM-D
Creation_Time   =  8-JAN-2020 18:43:51
Revision_Time   = 31-JAN-2020 19:31:37
Current_Time    =  6-FEB-2020 13:53:13

Comment         = Parameter file, TopSpin 4.0.
Data_Format     = 1D COMPLEX
Dim_Size        = 65536
Dim_Title       = 19F
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 19F
X_Freq          = 376.46071638[MHz]
X_Freq_Flip     = TRUE
X_Offset        = -37.649837[kHz]
X_Points        = 65536
X_Prescans      = 4
X_Sweep         = 90.90909091[kHz]
Scans           = 16

Temp_Get        = 298.1491[K]
Filter_Factor   = 220
```



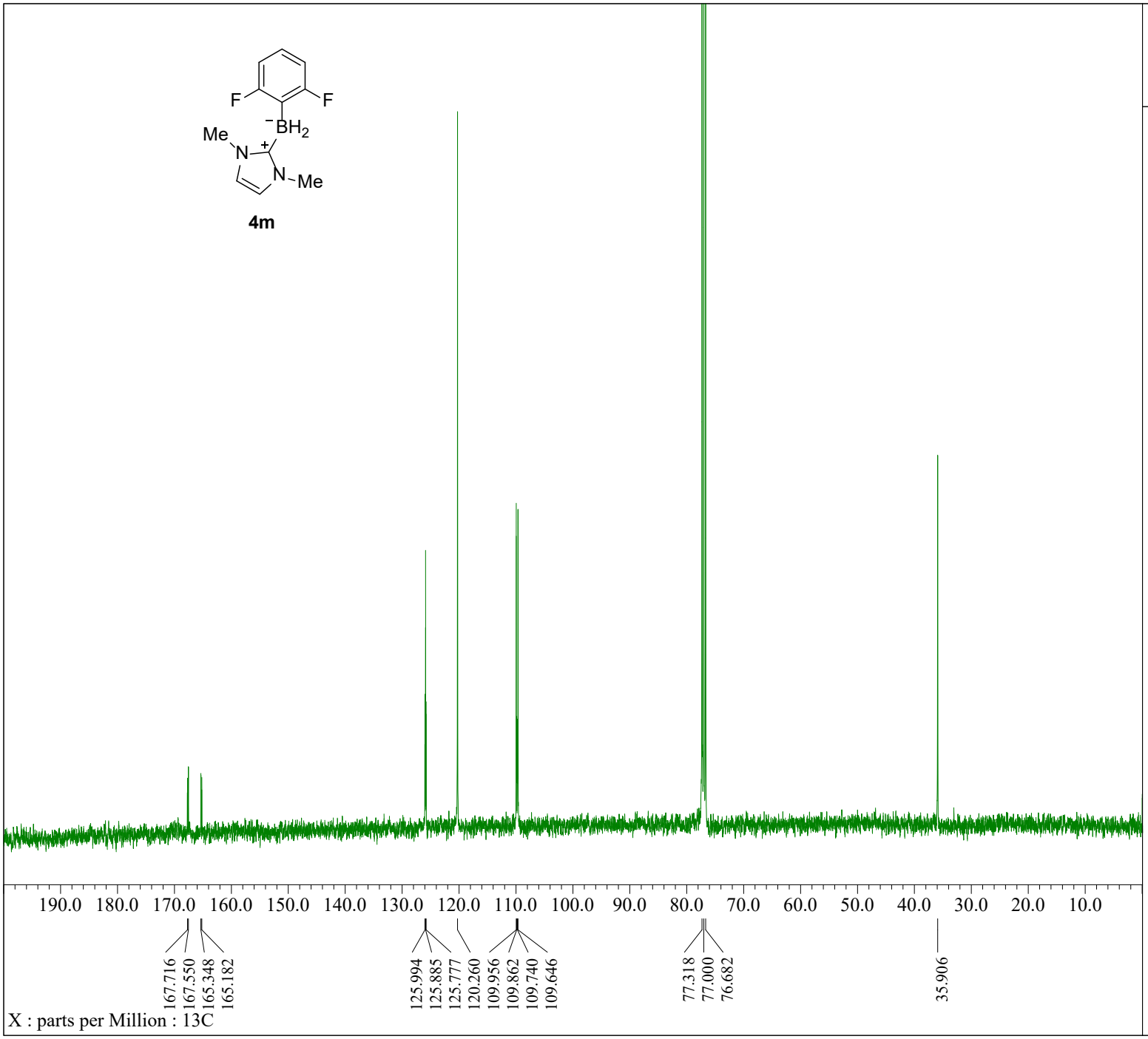
JEOL Solutions for Innovation

```
Filename          = KT-105-purified-1H-1.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#652973
Solvent           = CHLOROFORM-D
Creation_Time     = 29-JAN-2019 17:50:37
Revision_Time     = 31-JAN-2020 20:31:17
Current_Time      =  6-FEB-2020 14:00:04

Comment           = KT-105-purified-1H
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = JNM-ECA600

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.9097984[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.34366642[Hz]
X_Sweep           = 11.26126126[kHz]
Irr_Domain        = 1H
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 2[s]
Recvr_Gain        = 44
Temp_Get          = 19[dC]
X_90_Width        = 13.05[us]
X_Acq_Time        = 2.9097984[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.525[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 4.9097984[s]
```



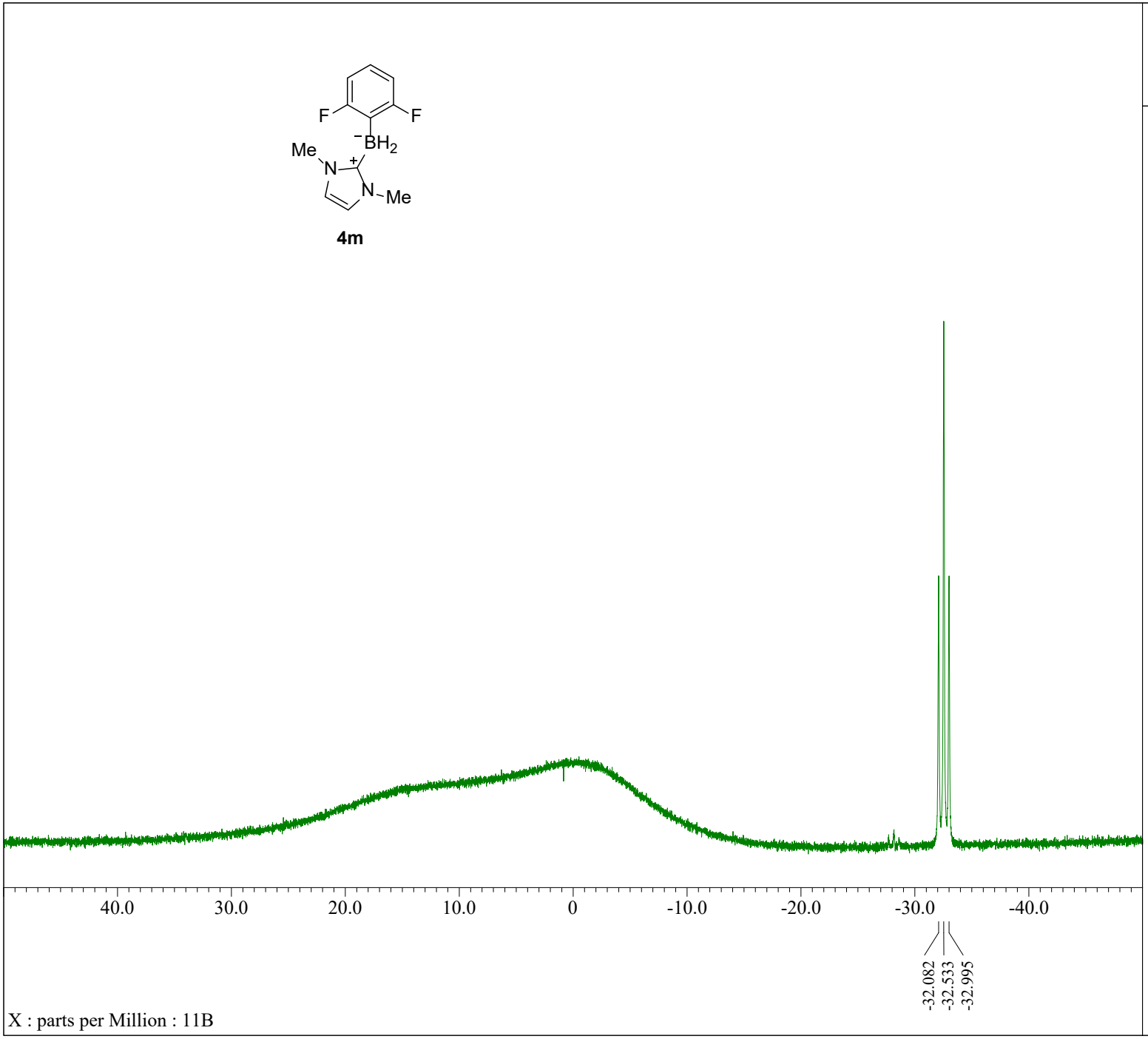
JEOL Solutions for Innovation

```
Filename        = KT-105-purified-13C-re2積算600
Author          = nmrsu
Experiment      = zgpg30
Sample_Id       = Parameter file, TopSpin 4.0.
Solvent         = CHLOROFORM-D
Creation_Time   =  8-JAN-2020 18:03:52
Revision_Time   = 31-JAN-2020 20:45:51
Current_Time    =  6-FEB-2020 14:01:13

Comment         = Parameter file, TopSpin 4.0.
Data_Format     = 1D COMPLEX
Dim_Size        = 32768
Dim_Title       = 13C
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 13C
X_Freq          = 100.6228298[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 10.061277[kHz]
X_Points        = 32768
X_Prescans      = 4
X_Sweep         = 23.80952381[kHz]
Scans           = 579

Temp_Get        = 298.1523[K]
Filter_Factor   = 840
```



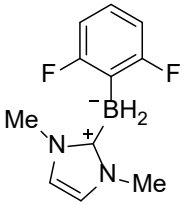
JEOL Solutions for Innovation

```
Filename          = KT-105-purified-11B-1.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#653088
Solvent           = CHLOROFORM-D
Creation_Time     = 29-JAN-2019 17:53:21
Revision_Time     = 31-JAN-2020 20:50:32
Current_Time      =  6-FEB-2020 14:02:02

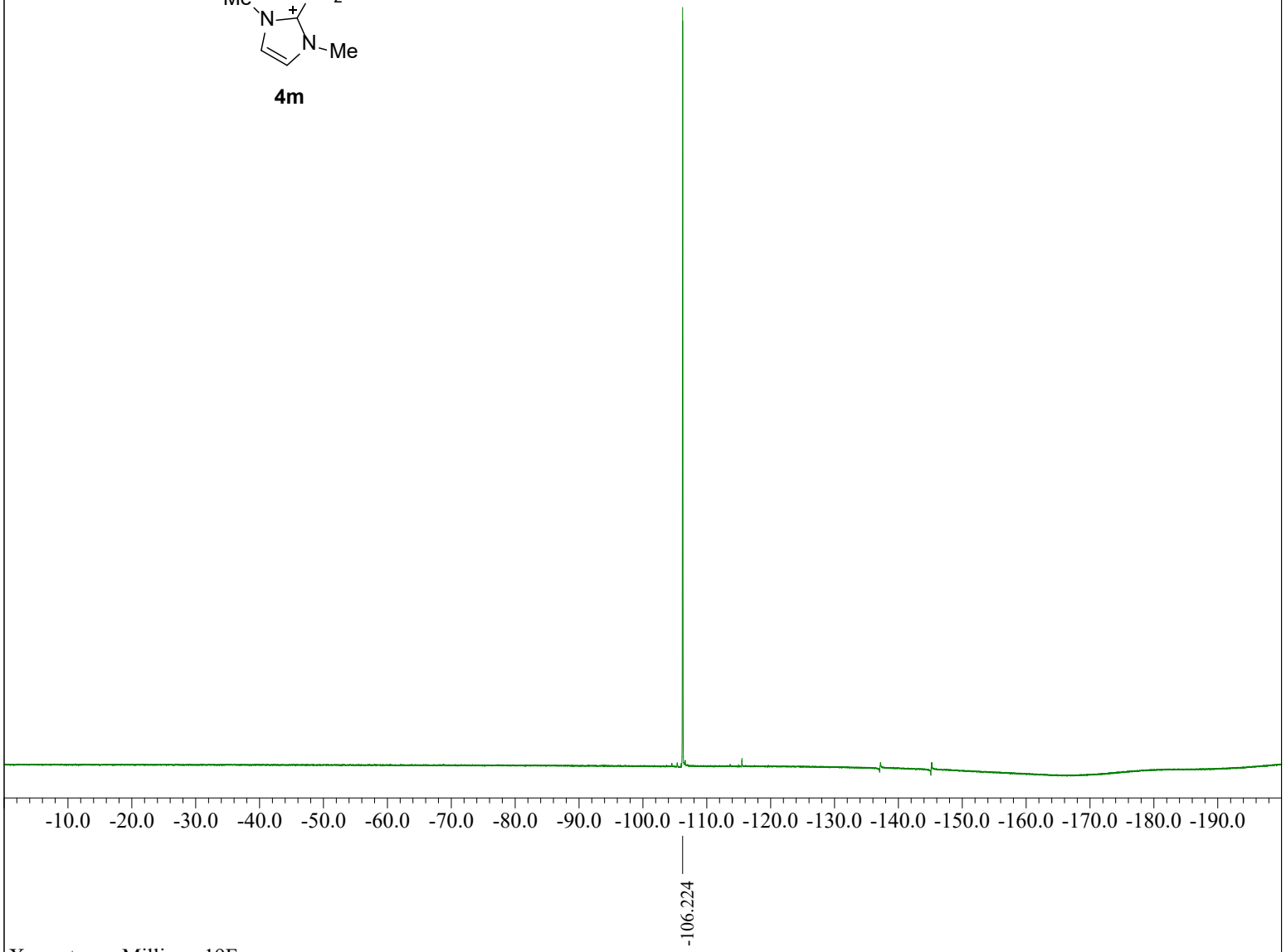
Comment           = KT-105-purified-11B
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 11B
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = JNM-ECA600

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.54001664[s]
X_Domain          = 11B
X_Freq            = 192.5593054[MHz]
X_Offset          = 0[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 1.85179479[Hz]
X_Sweep           = 60.67961165[kHz]
Irr_Domain        = 11B
Irr_Freq          = 192.5593054[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 11B
Tri_Freq          = 192.5593054[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 32
Total_Scans       = 32

Relaxation_Delay = 2[s]
Recvr_Gain        = 36
Temp_Get          = 19.1[dC]
X_90_Width        = 24[us]
X_Acq_Time        = 0.54001664[s]
X_Angle           = 45[deg]
X_Atn             = 6.9[dB]
X_Pulse           = 12[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 2.54001664[s]
```



4m



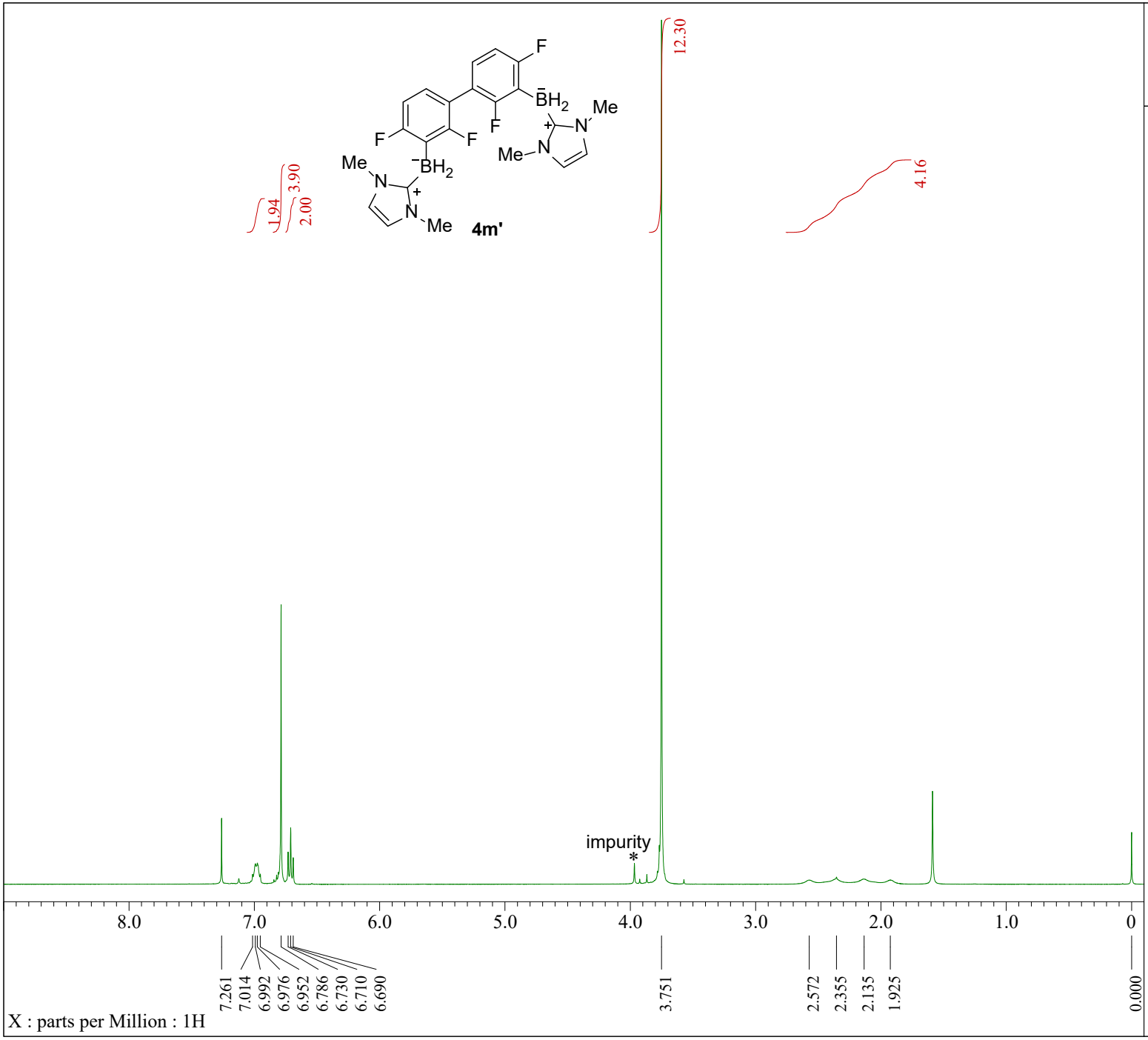
JEOL Solutions for Innovation

```
Filename        = KT-105-purified-19F-re_1-1.j
Author          = nmrsu
Experiment      = zg
Sample_Id       = Parameter file, TopSpin 4.0.
Solvent         = CHLOROFORM-D
Creation_Time   =  8-JAN-2020 15:32:27
Revision_Time   = 31-JAN-2020 20:52:03
Current_Time    =  6-FEB-2020 14:02:45

Comment         = Parameter file, TopSpin 4.0.
Data_Format     = 1D COMPLEX
Dim_Size        = 65536
Dim_Title       = 19F
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 19F
X_Freq          = 376.46071638[MHz]
X_Freq_Flip     = TRUE
X_Offset        = -37.649837[kHz]
X_Points        = 65536
X_Prescans      = 4
X_Sweep         = 90.90909091[kHz]
Scans           = 16

Temp_Get        = 298.1488[K]
Filter_Factor   = 220
```



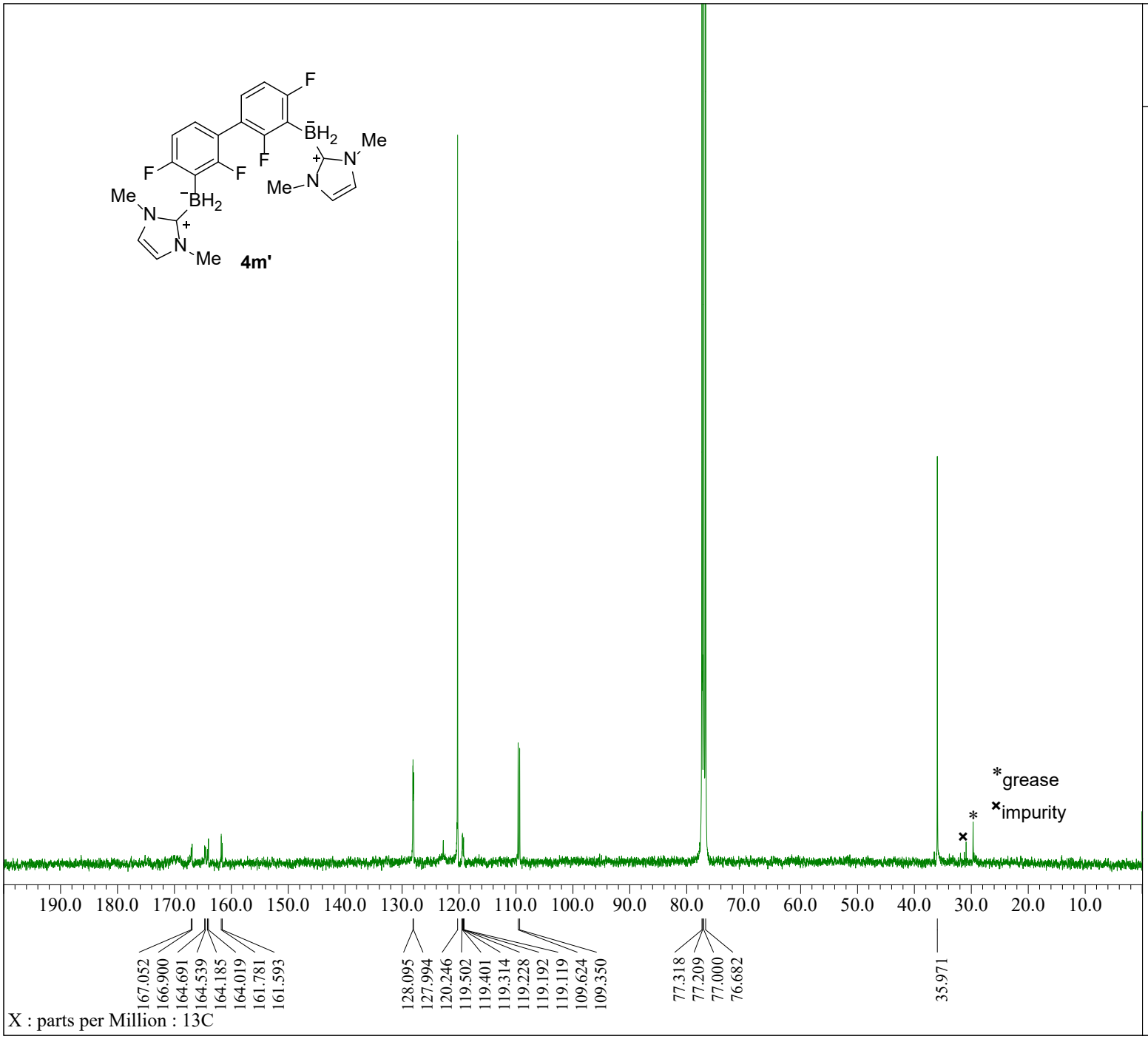
JEOL Solutions for Innovation

```
Filename         = KT-111-recrystalized-dimer-1
Author           = nmrsu
Experiment       = zg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 09:36:32
Revision_Time    =  6-FEB-2020 14:04:03
Current_Time     =  6-FEB-2020 14:04:09

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 1H
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 1H
X_Freq           = 400.1324708[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 2.470802[kHz]
X_Points         = 32768
X_Prescans       = 2
X_Sweep          = 8.19672131[kHz]
Scans            = 16

Temp_Get         = 298.1499[K]
Filter_Factor    = 2440
```



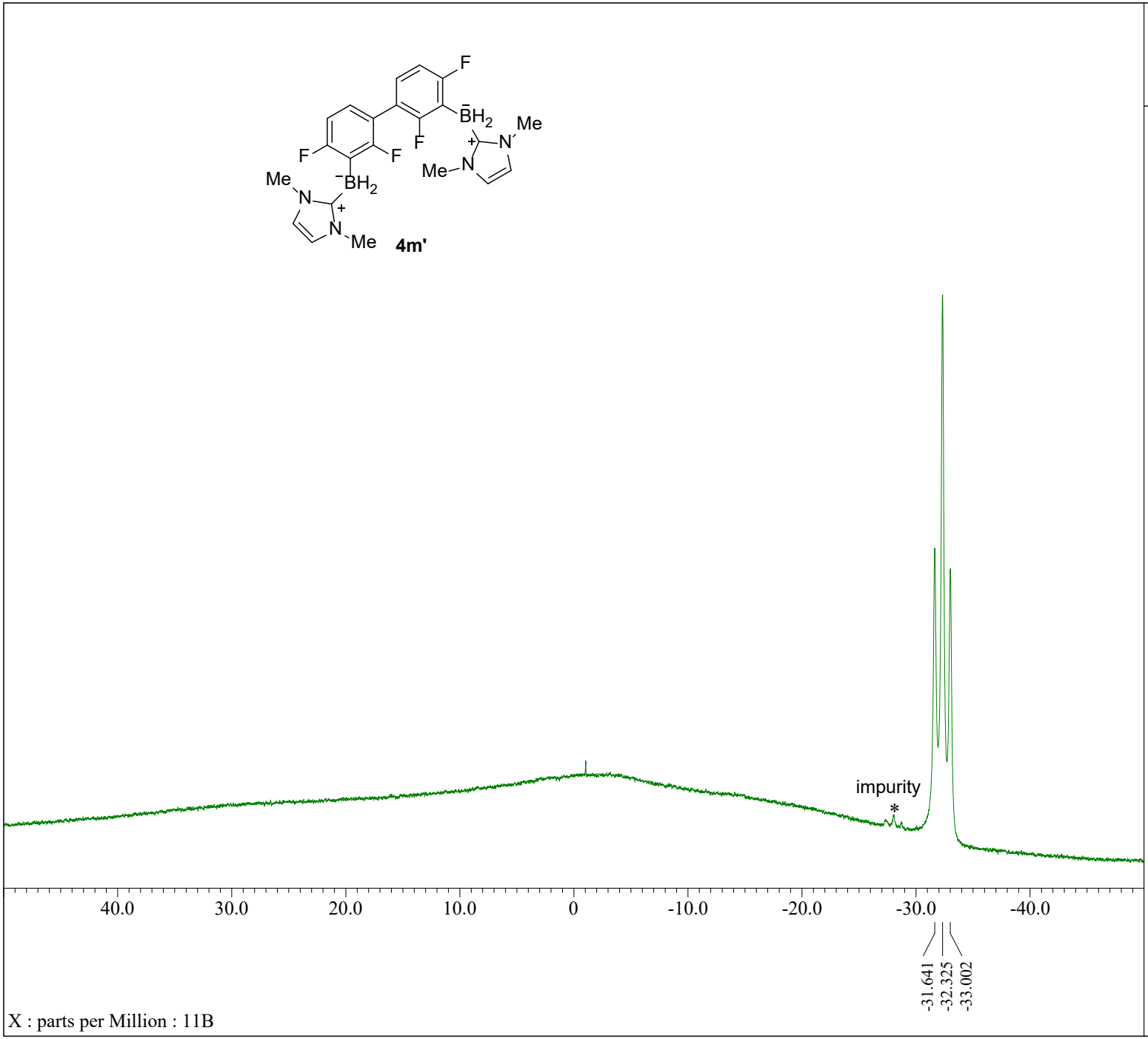
JEOL Solutions for Innovation

```
Filename         = KT-111-recrystalized-dimer-1
Author           = nmrsu
Experiment       = zgpg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 15-JAN-2020 16:53:21
Revision_Time    = 31-JAN-2020 21:31:54
Current_Time     =  6-FEB-2020 14:05:01

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 32768
Dim_Title        = 13C
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 13C
X_Freq           = 100.6228298[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 10.061277[kHz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 23.80952381[kHz]
Scans            = 11655

Temp_Get         = 298.1469[K]
Filter_Factor    = 840
```



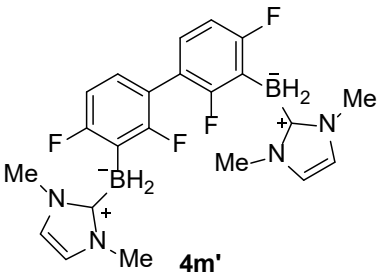
JEOL Solutions for Innovation

```
Filename         = KT-111-recrystalized-dimer-1
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 09:37:53
Revision_Time    = 31-JAN-2020 21:46:26
Current_Time     =  6-FEB-2020 14:05:46

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 11B
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 11B
X_Freq           = 128.37760517[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 0[Hz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 25.90673575[kHz]
Scans            = 70

Temp_Get         = 298.1477[K]
Filter_Factor    = 772
```



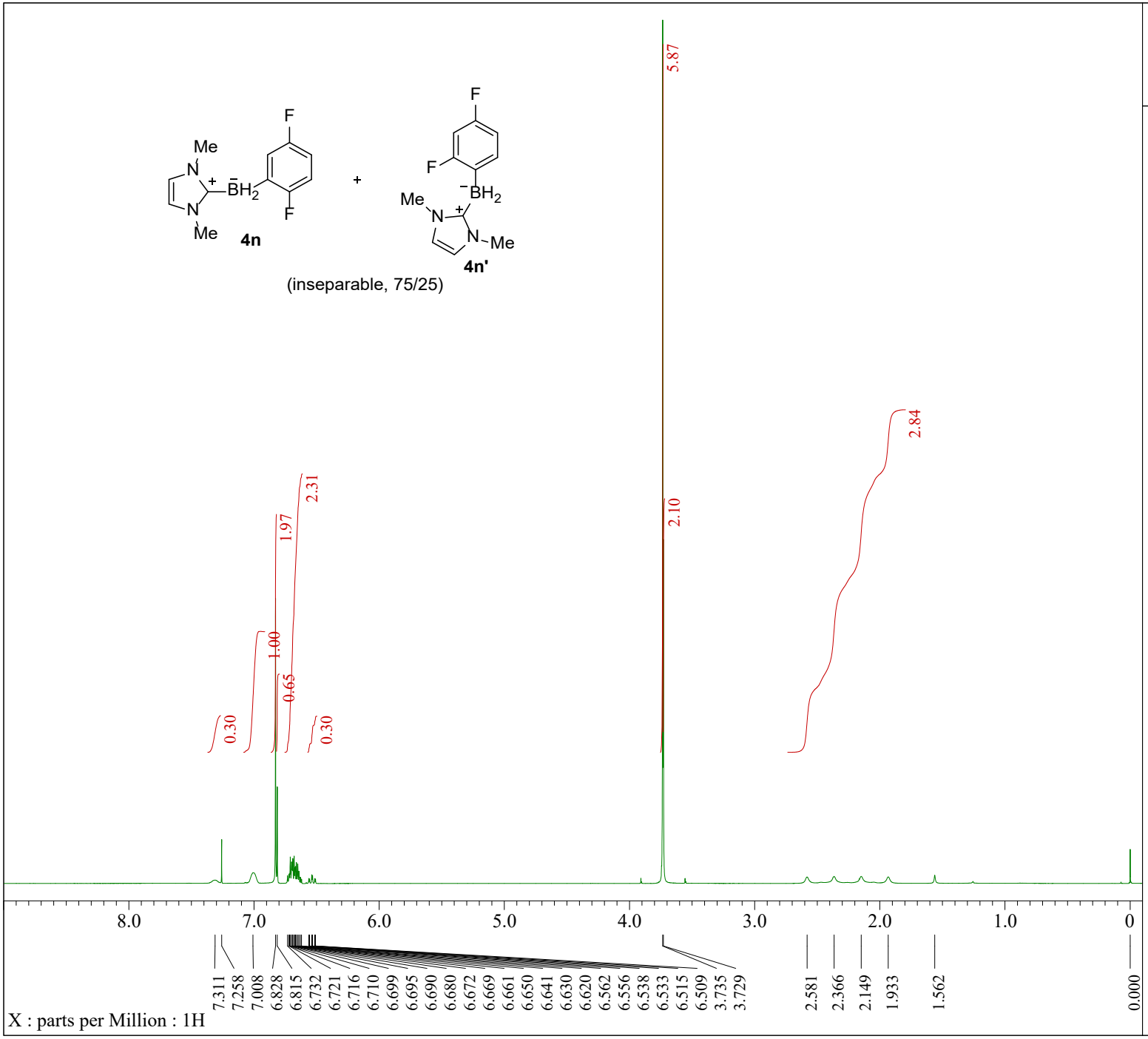
4m'


```
Filename         = KT-111-recrystalized-dimer-1
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 09:40:51
Revision_Time    =  6-FEB-2020 14:06:54
Current_Time     =  6-FEB-2020 14:07:14

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 65536
Dim_Title        = 19F
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
X_Prescans       = 4
X_Sweep          = 90.90909091[kHz]
Scans            = 16

Temp_Get         = 298.1479[K]
Filter_Factor    = 220
```



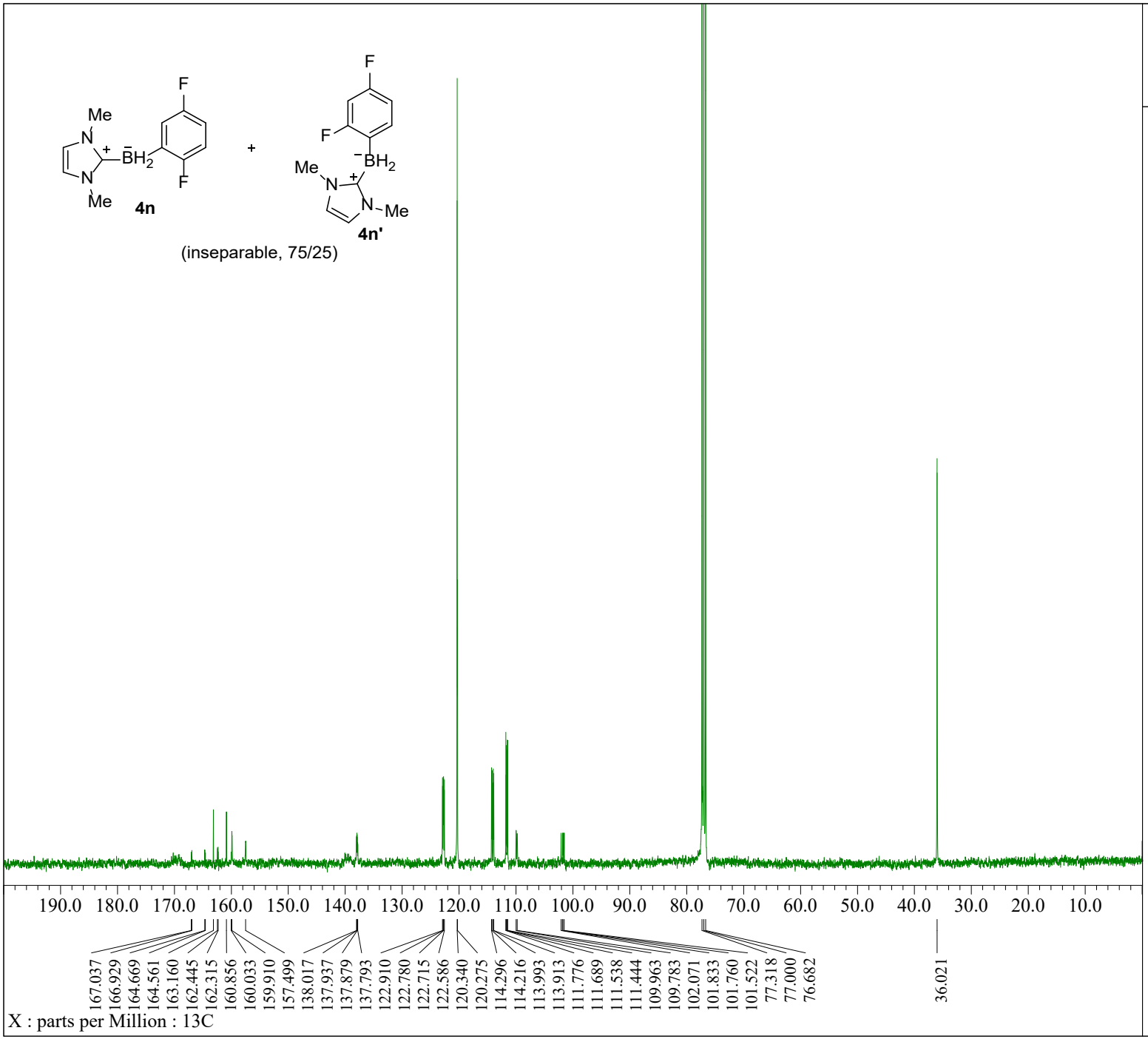
JEOL Solutions for Innovation

```
Filename         = KT-234-purified-1H_1-1.jdf
Author           = nmrsu
Experiment       = zg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 20-JAN-2020 19:40:14
Revision_Time    = 31-JAN-2020 22:43:24
Current_Time     =  6-FEB-2020 14:11:00

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 1H
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 1H
X_Freq           = 400.1324708[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 2.470802[kHz]
X_Points         = 32768
X_Prescans       = 2
X_Sweep          = 8.19672131[kHz]
Scans            = 16

Temp_Get         = 298.1522[K]
Filter_Factor    = 2440
```



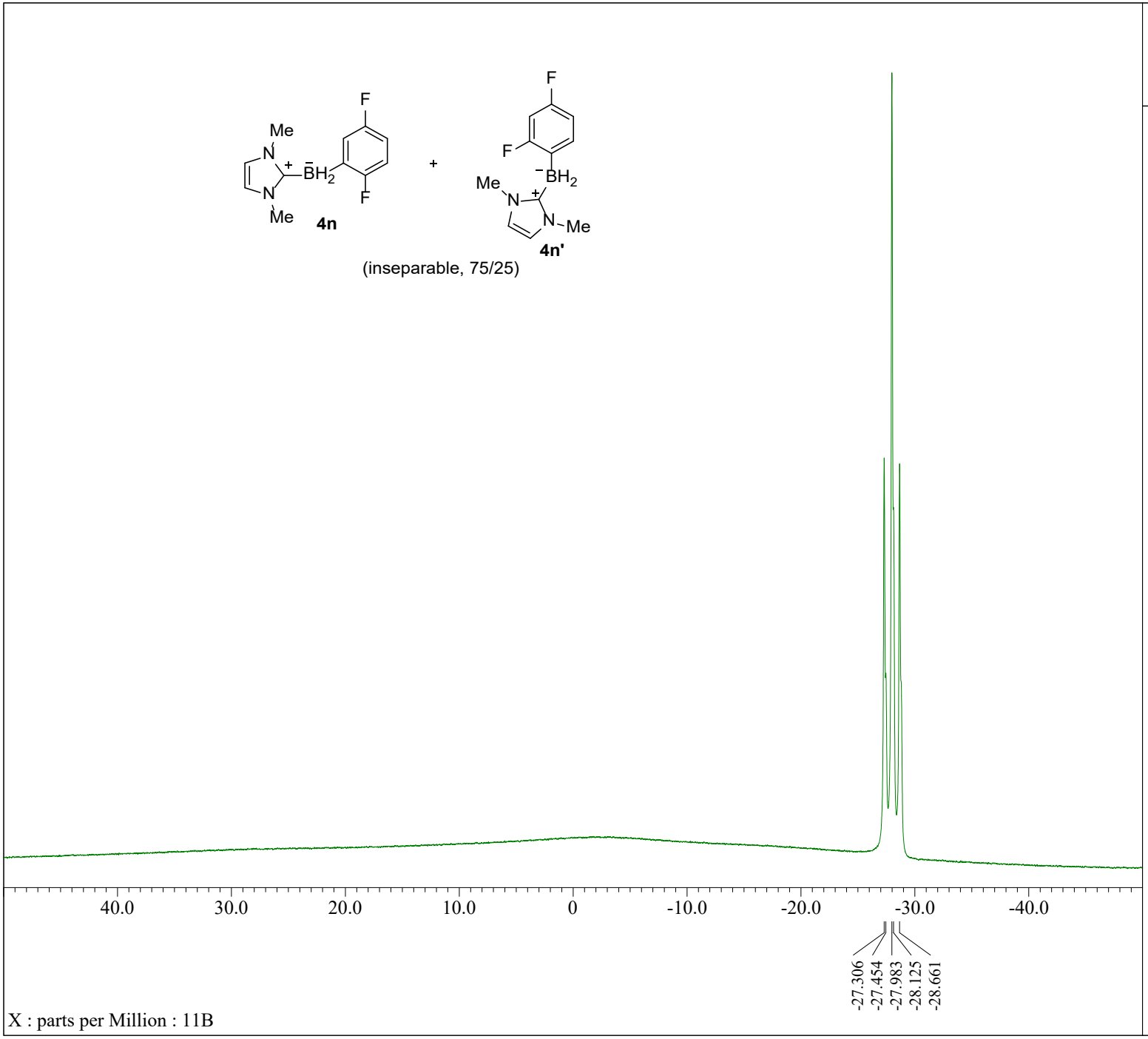
JEOL Solutions for Innovation

```
Filename         = KT-234-purified-13C_1-1.jdf
Author           = nmrsu
Experiment       = zgpg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 31-JAN-2020 22:15:10
Revision_Time    = 31-JAN-2020 22:31:12
Current_Time     =  6-FEB-2020 14:20:07

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 32768
Dim_Title        = 13C
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 13C
X_Freq           = 100.6228298[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 10.061277[kHz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 23.80952381[kHz]
Scans            = 2048

Temp_Get         = 298.1468[K]
Filter_Factor    = 840
```



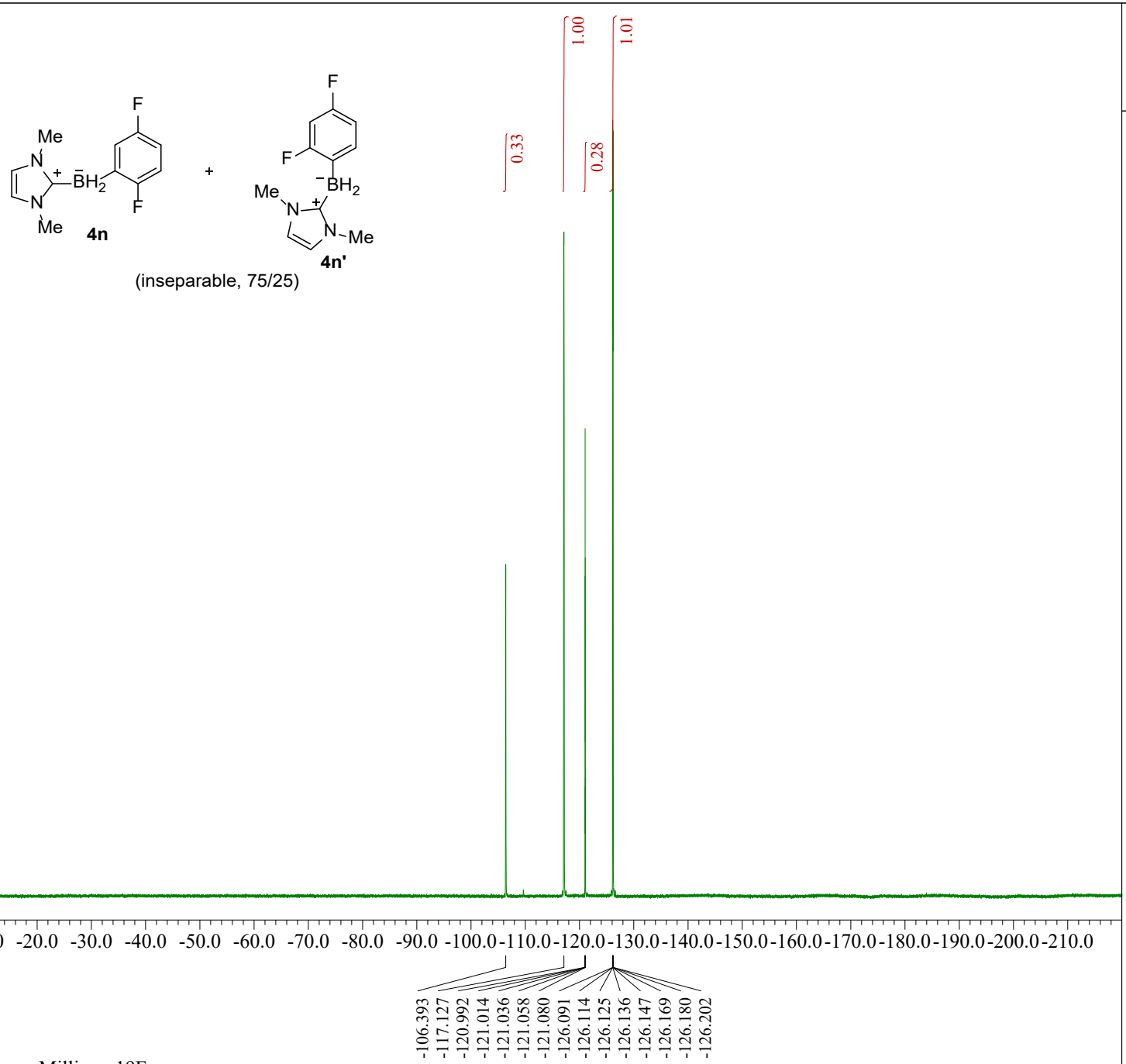
JEOL Solutions for Innovation

```
Filename         = KT-234-purified-11B_1-3.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 20-JAN-2020 19:39:11
Revision_Time    =  6-FEB-2020 14:09:31
Current_Time     =  6-FEB-2020 14:10:08

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 11B
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 11B
X_Freq           = 128.37760517[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 0[Hz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 25.90673575[kHz]
Scans            = 64

Temp_Get         = 298.1465[K]
Filter_Factor    = 772
```



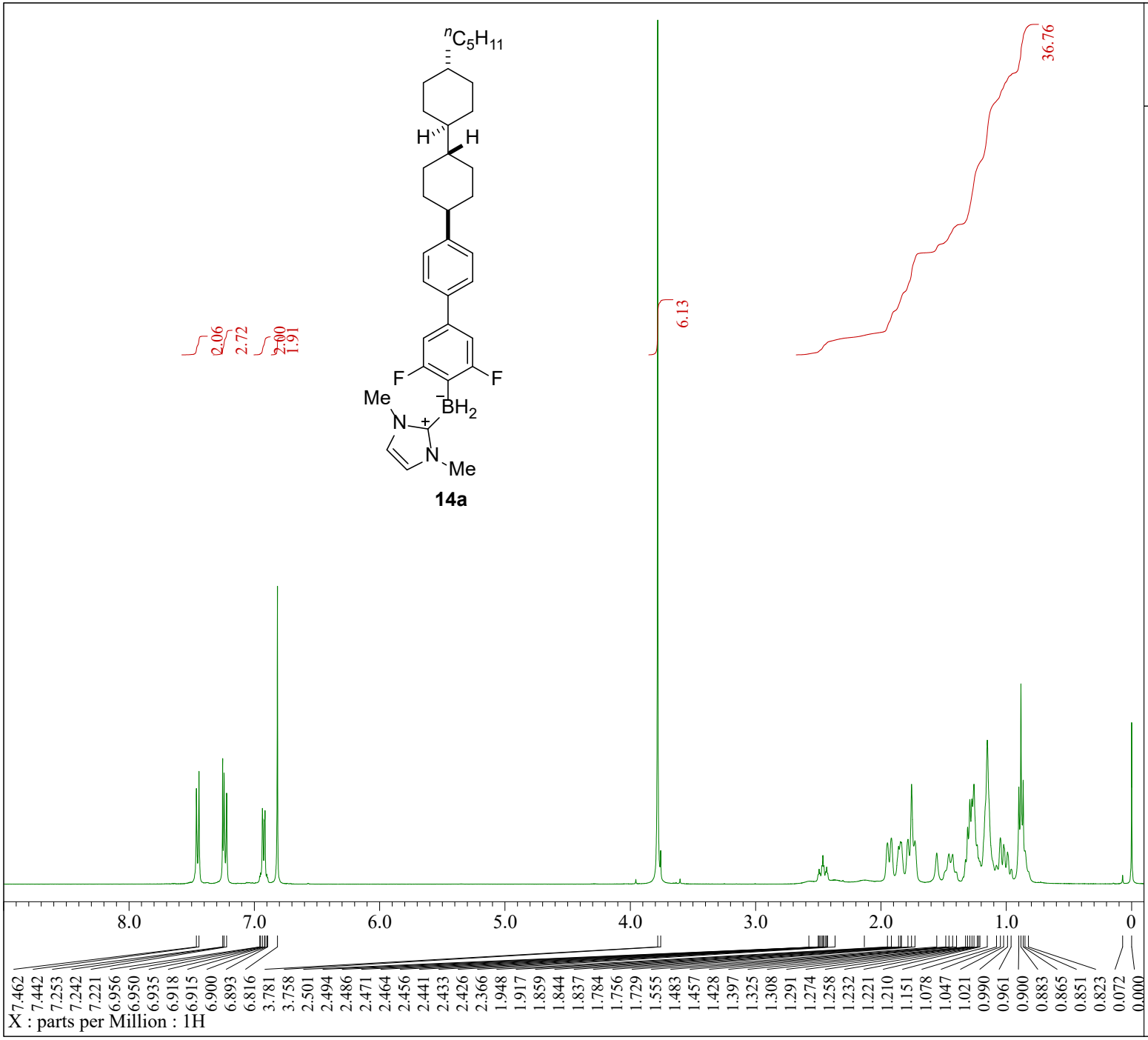
JEOL Solutions for Innovation

```
Filename         = KT-234-purified-19F_1-2.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 20-JAN-2020 19:31:04
Revision_Time    =  6-FEB-2020 14:20:49
Current_Time     =  6-FEB-2020 14:21:14

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 65536
Dim_Title        = 19F
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
X_Prescans       = 4
X_Sweep          = 90.90909091[kHz]
Scans            = 16

Temp_Get         = 298.1534[K]
Filter_Factor    = 220
```



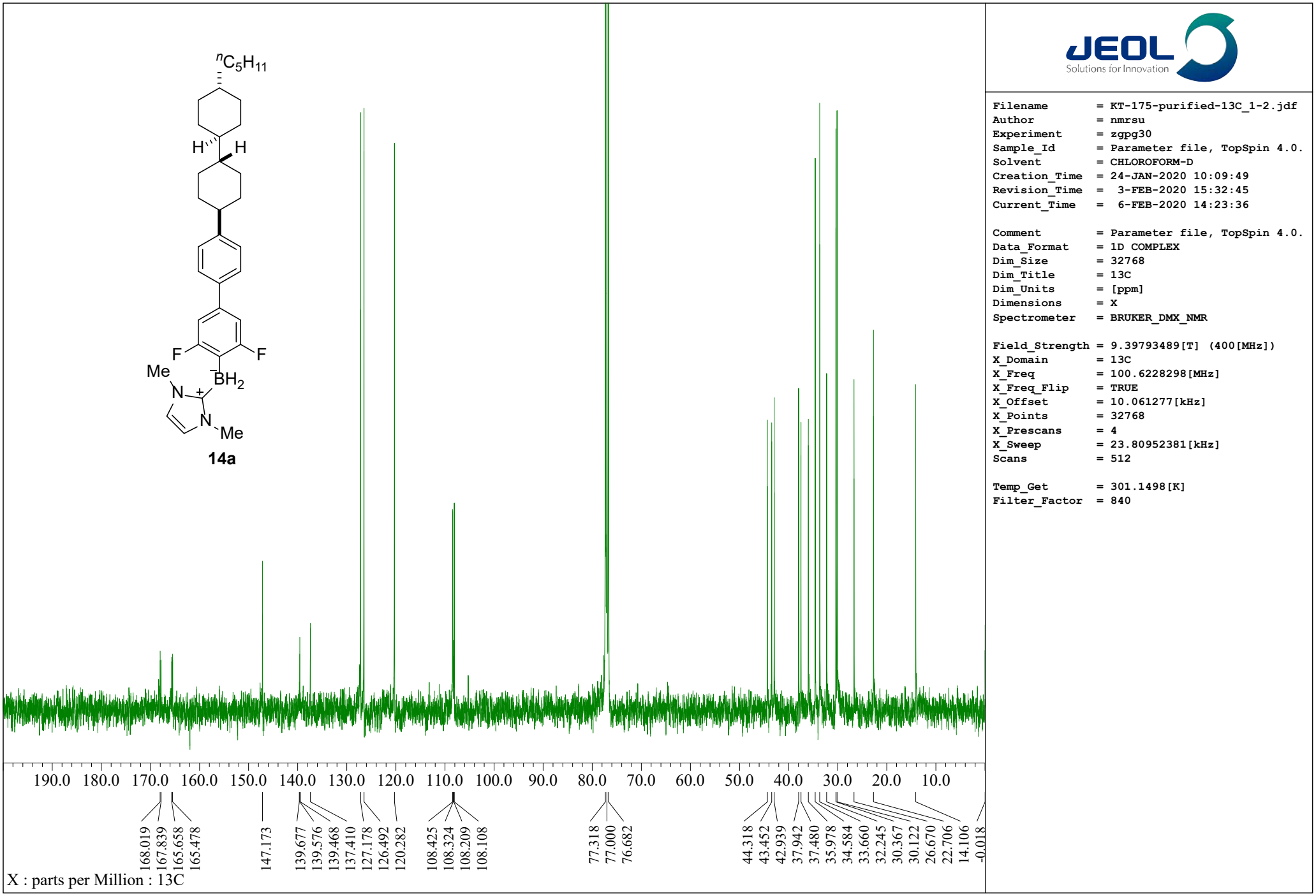
JEOL
Solutions for Innovation

```
Filename         = KT-175-purified-1H_1-1.jdf
Author           = nmrsu
Experiment       = zg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 24-JAN-2020 10:11:53
Revision_Time    =  3-FEB-2020 09:51:31
Current_Time     =  6-FEB-2020 14:22:38

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 1H
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength   = 9.39793489[T] (400[MHz])
X_Domain         = 1H
X_Freq           = 400.1324708[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 2.470802[kHz]
X_Points         = 32768
X_Prescans       = 2
X_Sweep          = 8.19672131[kHz]
Scans            = 16

Temp_Get         = 301.1499[K]
Filter_Factor    = 2440
```



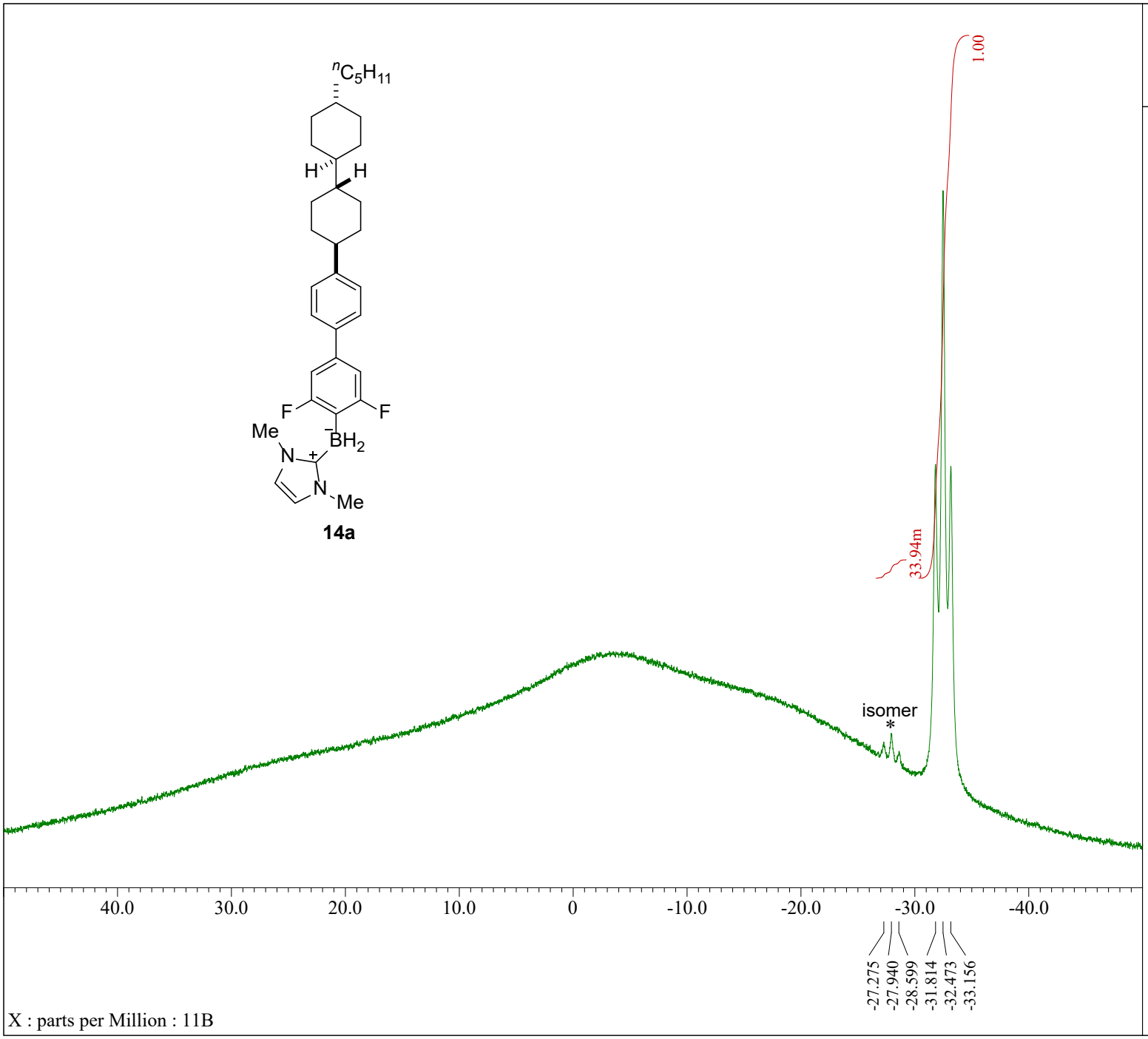
JEOL Solutions for Innovation

```
Filename         = KT-175-purified-13C_1-2.jdf
Author           = nmrsu
Experiment       = zgpg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 24-JAN-2020 10:09:49
Revision_Time    =  3-FEB-2020 15:32:45
Current_Time     =  6-FEB-2020 14:23:36

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 13C
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 13C
X_Freq           = 100.6228298[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 10.061277[kHz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 23.80952381[kHz]
Scans            = 512

Temp_Get         = 301.1498[K]
Filter_Factor    = 840
```



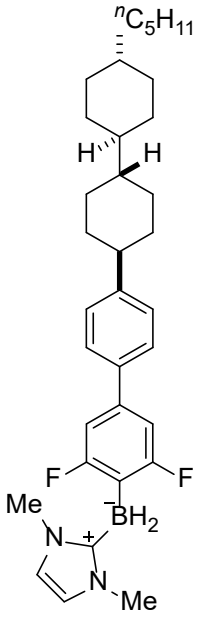
JEOL Solutions for Innovation

```
Filename         = KT-175-purified-11B_1-1.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 24-JAN-2020 10:10:26
Revision_Time    =  3-FEB-2020 13:45:45
Current_Time     =  6-FEB-2020 14:24:24

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 11B
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 11B
X_Freq           = 128.37760517[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 0[Hz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 25.90673575[kHz]
Scans            = 128

Temp_Get         = 301.1498[K]
Filter_Factor    = 772
```



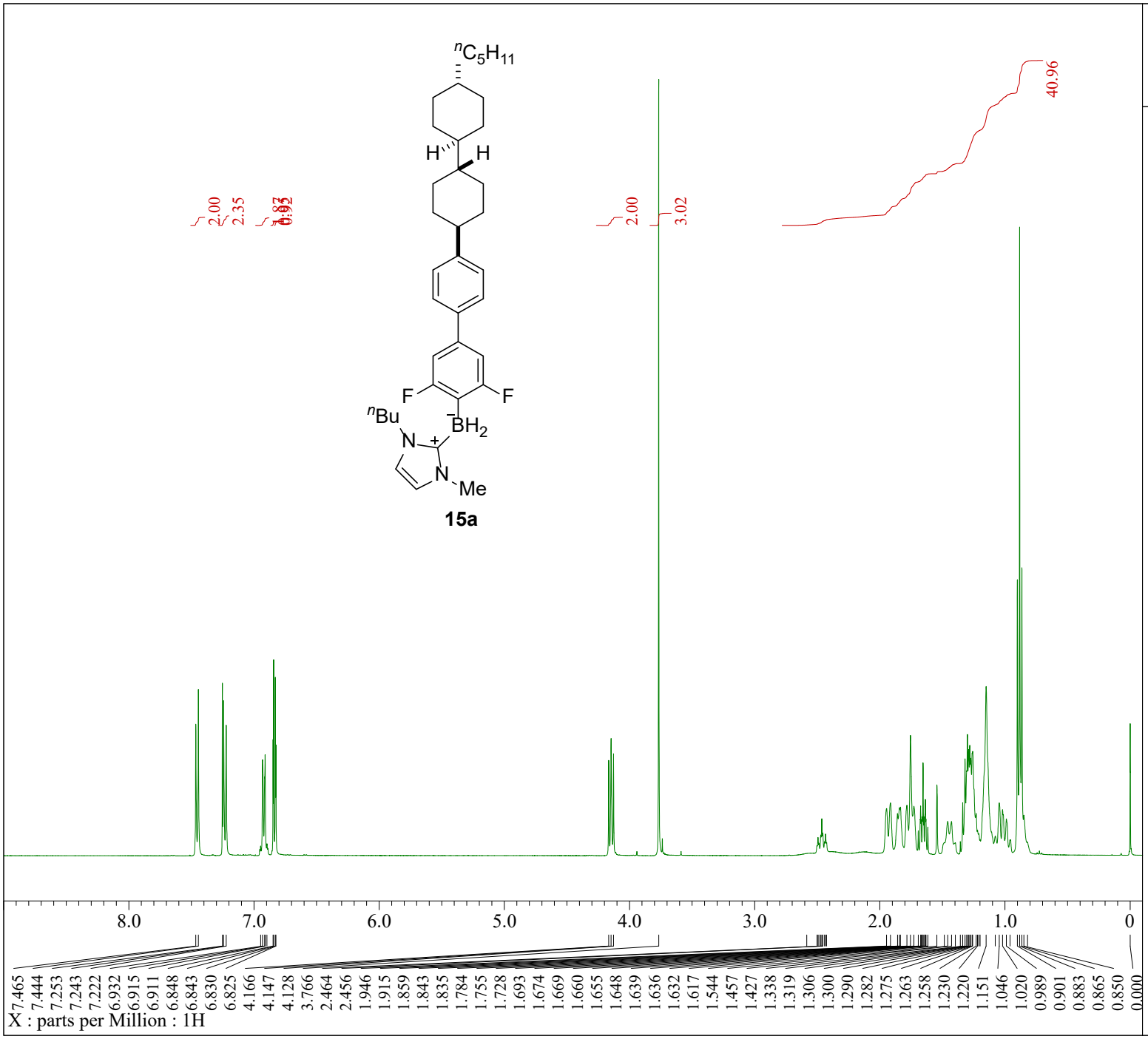
14a


```
Filename        = KT-175-purified-19F_1-1.jdf
Author          = nmrsu
Experiment      = zg
Sample_Id       = Parameter file, TopSpin 4.0.
Solvent         = CHLOROFORM-D
Creation_Time   = 24-JAN-2020 10:11:24
Revision_Time   =  3-FEB-2020 11:02:52
Current_Time    =  6-FEB-2020 14:25:05

Comment         = Parameter file, TopSpin 4.0.
Data_Format     = 1D COMPLEX
Dim_Size        = 65536
Dim_Title       = 19F
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 19F
X_Freq          = 376.46071638[MHz]
X_Freq_Flip     = TRUE
X_Offset        = -37.649837[kHz]
X_Points        = 65536
X_Prescans      = 4
X_Sweep         = 90.90909091[kHz]
Scans           = 16

Temp_Get        = 301.149[K]
Filter_Factor   = 220
```



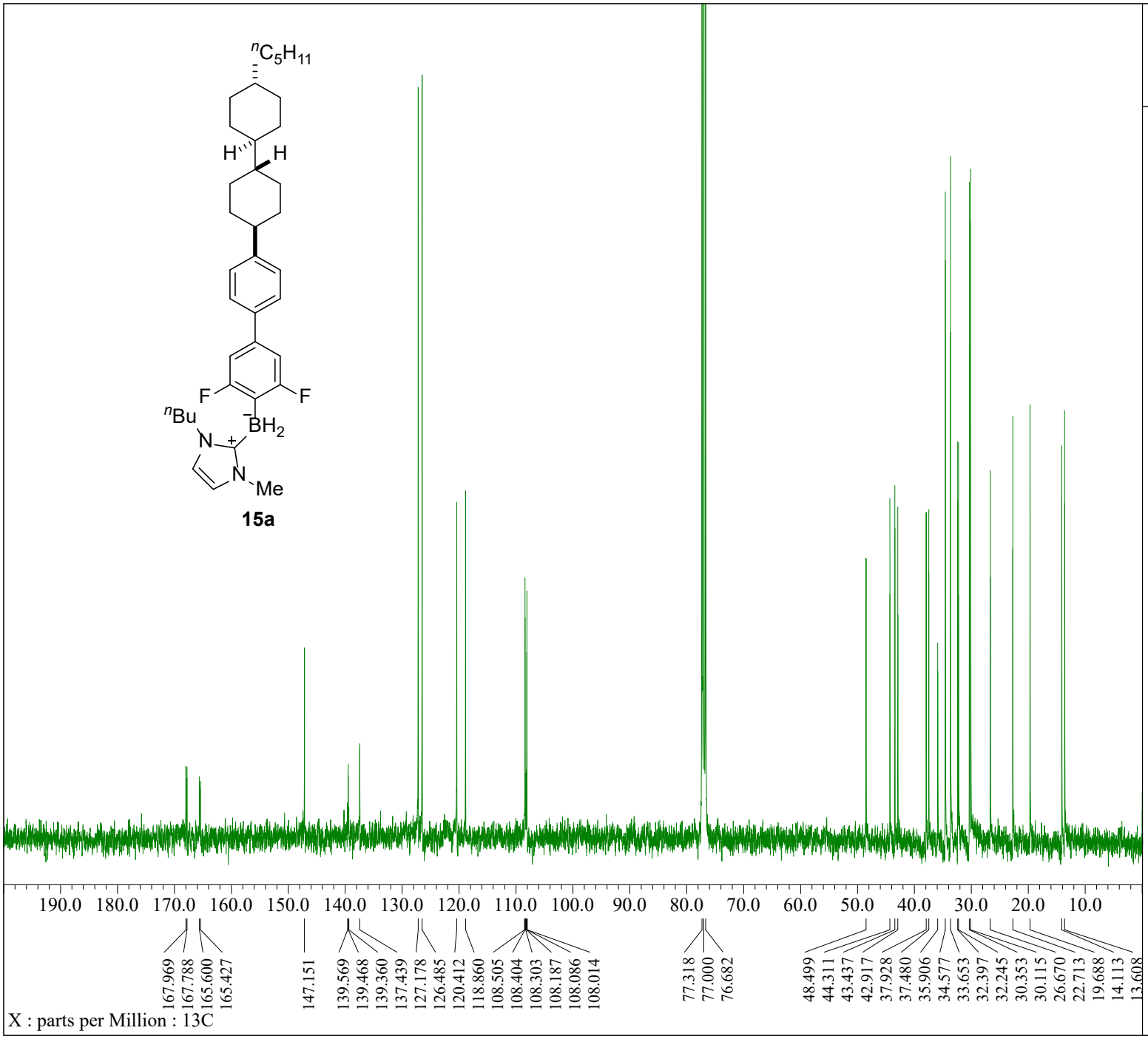
JEOL Solutions for Innovation

```
Filename         = KT-181-purified-1H-re_1-2.jd
Author           = nmrsu
Experiment       = zg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  3-FEB-2020 21:52:29
Revision_Time    =  6-FEB-2020 16:17:49
Current_Time     =  6-FEB-2020 16:18:33

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 1H
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 1H
X_Freq           = 400.1324708[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 2.470802[kHz]
X_Points         = 32768
X_Prescans       = 2
X_Sweep          = 8.19672131[kHz]
Scans            = 16

Temp_Get         = 298.144[K]
Filter_Factor    = 2440
```



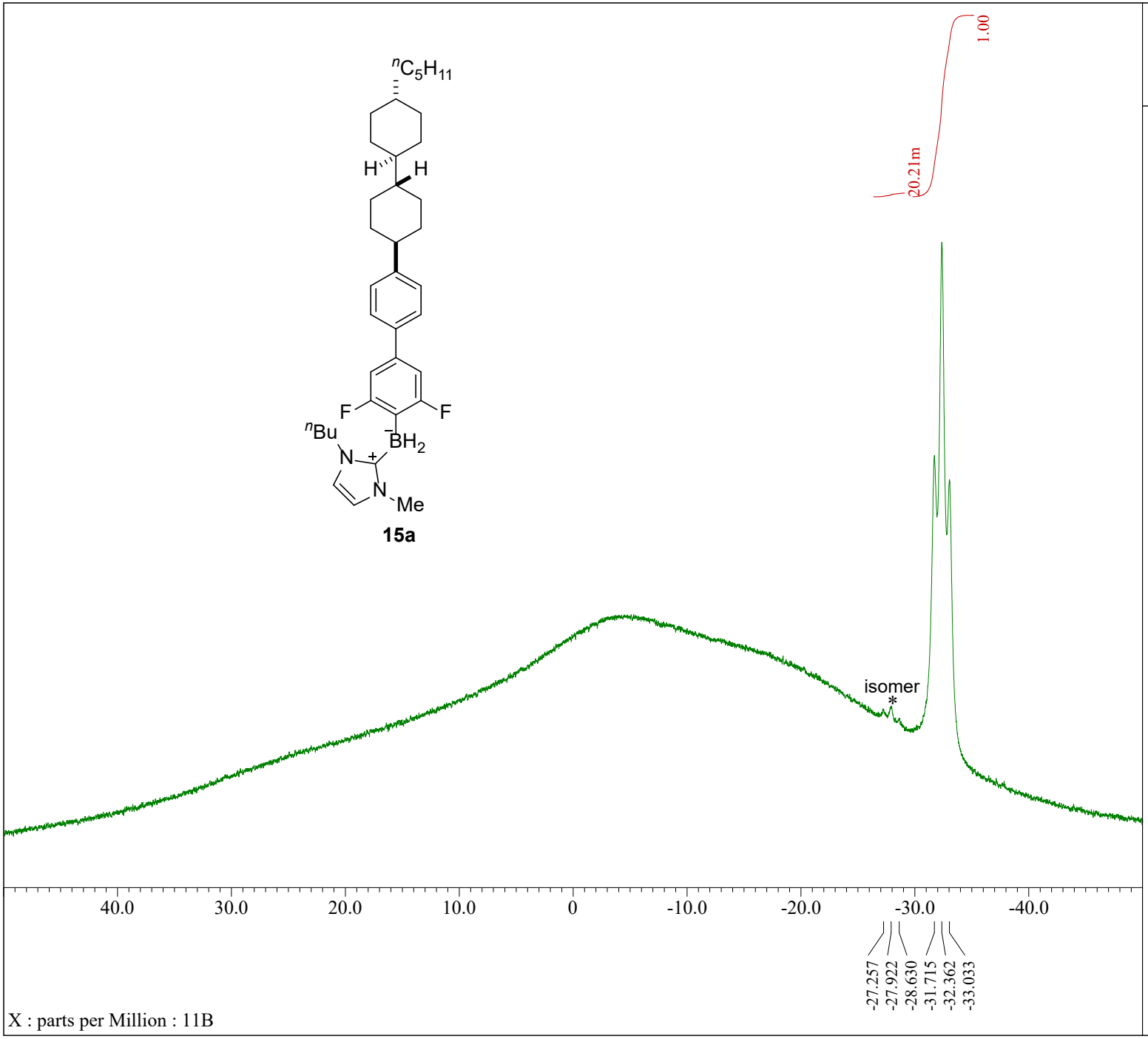
JEOL Solutions for Innovation

```
Filename         = KT-181-purified-13C-re_1-3.j
Author           = nmrsu
Experiment       = zgpg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    =  3-FEB-2020 21:53:47
Revision_Time    =  6-FEB-2020 16:20:19
Current_Time     =  6-FEB-2020 16:20:56

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 32768
Dim_Title        = 13C
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 13C
X_Freq           = 100.6228298[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 10.061277[kHz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 23.80952381[kHz]
Scans            = 800

Temp_Get         = 298.1488[K]
Filter_Factor    = 840
```



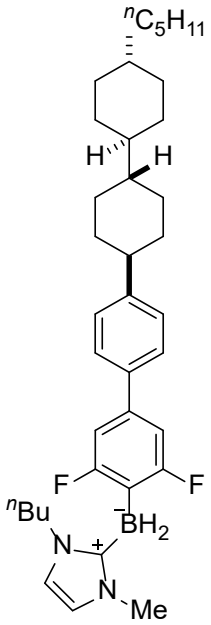
JEOL Solutions for Innovation

```
Filename         = KT-181-purified-11B_1-1.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 10:05:53
Revision_Time    =  3-FEB-2020 13:52:23
Current_Time     =  6-FEB-2020 16:22:02

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 11B
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 11B
X_Freq           = 128.37760517[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 0[Hz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 25.90673575[kHz]
Scans            = 128

Temp_Get         = 298.1487[K]
Filter_Factor    = 772
```



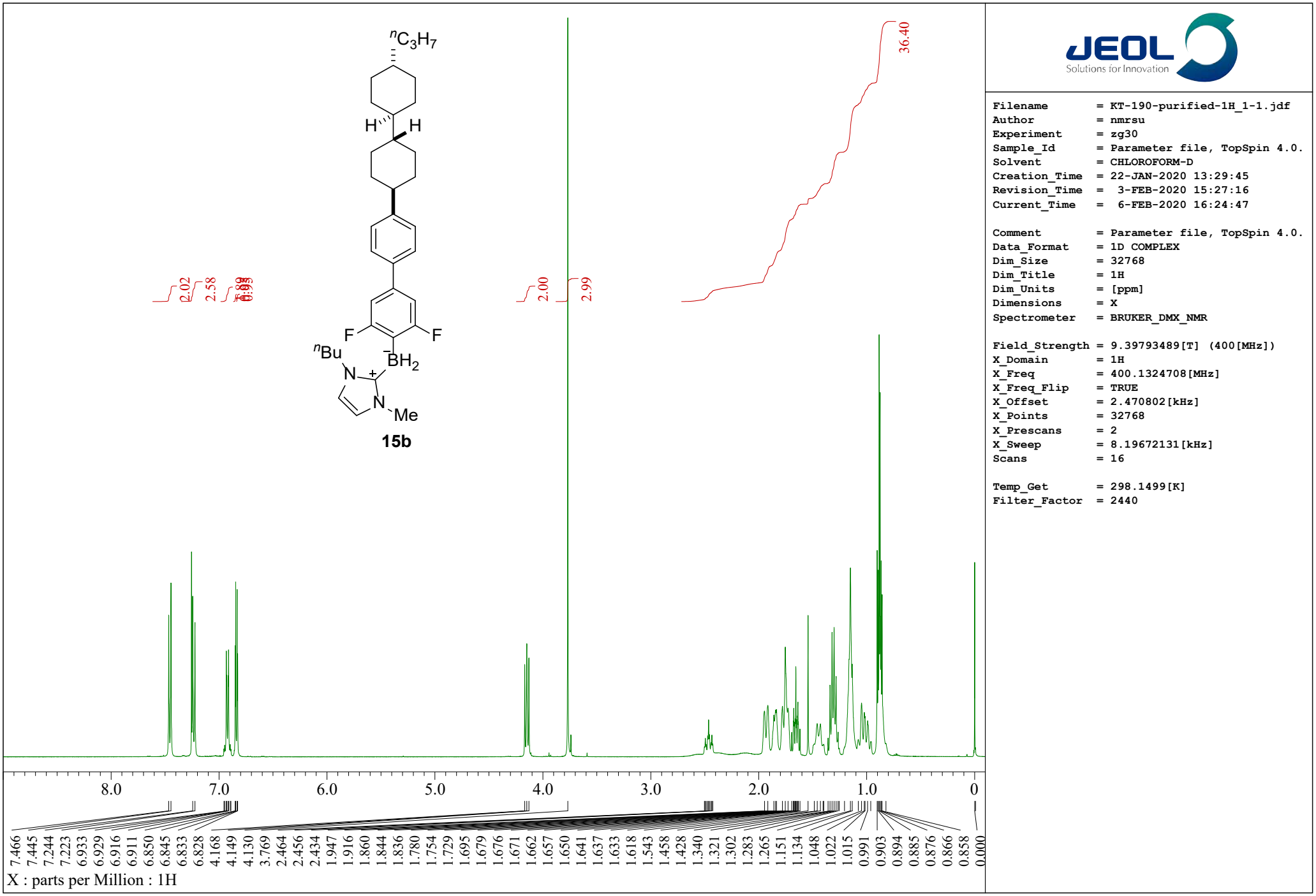
15a


```
Filename         = KT-181-purified-19F_1-1.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 10:22:16
Revision_Time    =  3-FEB-2020 13:54:47
Current_Time     =  6-FEB-2020 16:23:37

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 65536
Dim_Title        = 19F
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
X_Prescans       = 4
X_Sweep          = 90.90909091[kHz]
Scans            = 16

Temp_Get         = 298.1436[K]
Filter_Factor    = 220
```



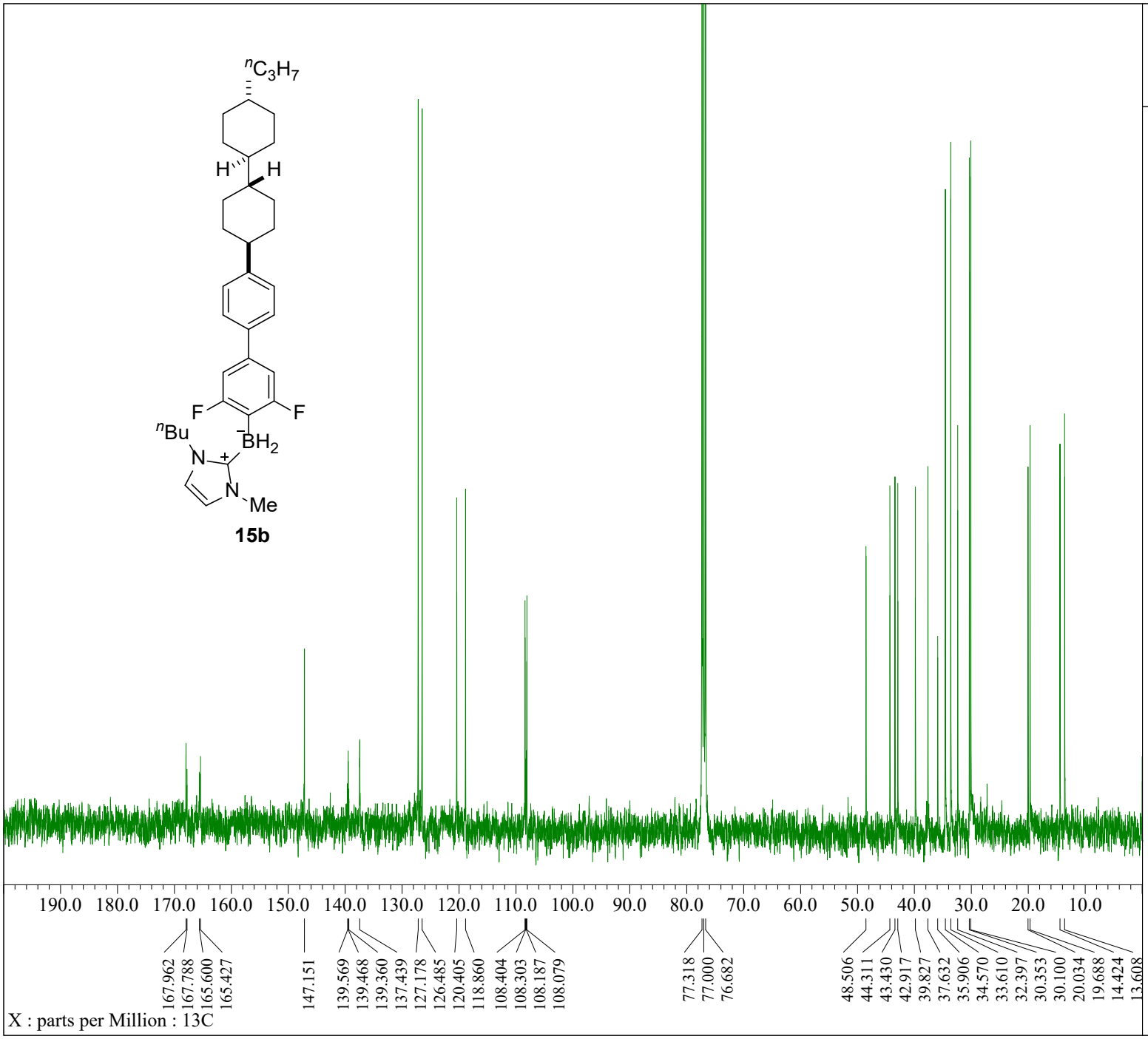
JEOL
Solutions for Innovation

```
Filename         = KT-190-purified-1H_1-1.jdf
Author           = nmrsu
Experiment       = zg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 13:29:45
Revision_Time    =  3-FEB-2020 15:27:16
Current_Time     =  6-FEB-2020 16:24:47

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 1H
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength   = 9.39793489[T] (400[MHz])
X_Domain         = 1H
X_Freq           = 400.1324708[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 2.470802[kHz]
X_Points         = 32768
X_Prescans       = 2
X_Sweep          = 8.19672131[kHz]
Scans            = 16

Temp_Get         = 298.1499[K]
Filter_Factor    = 2440
```



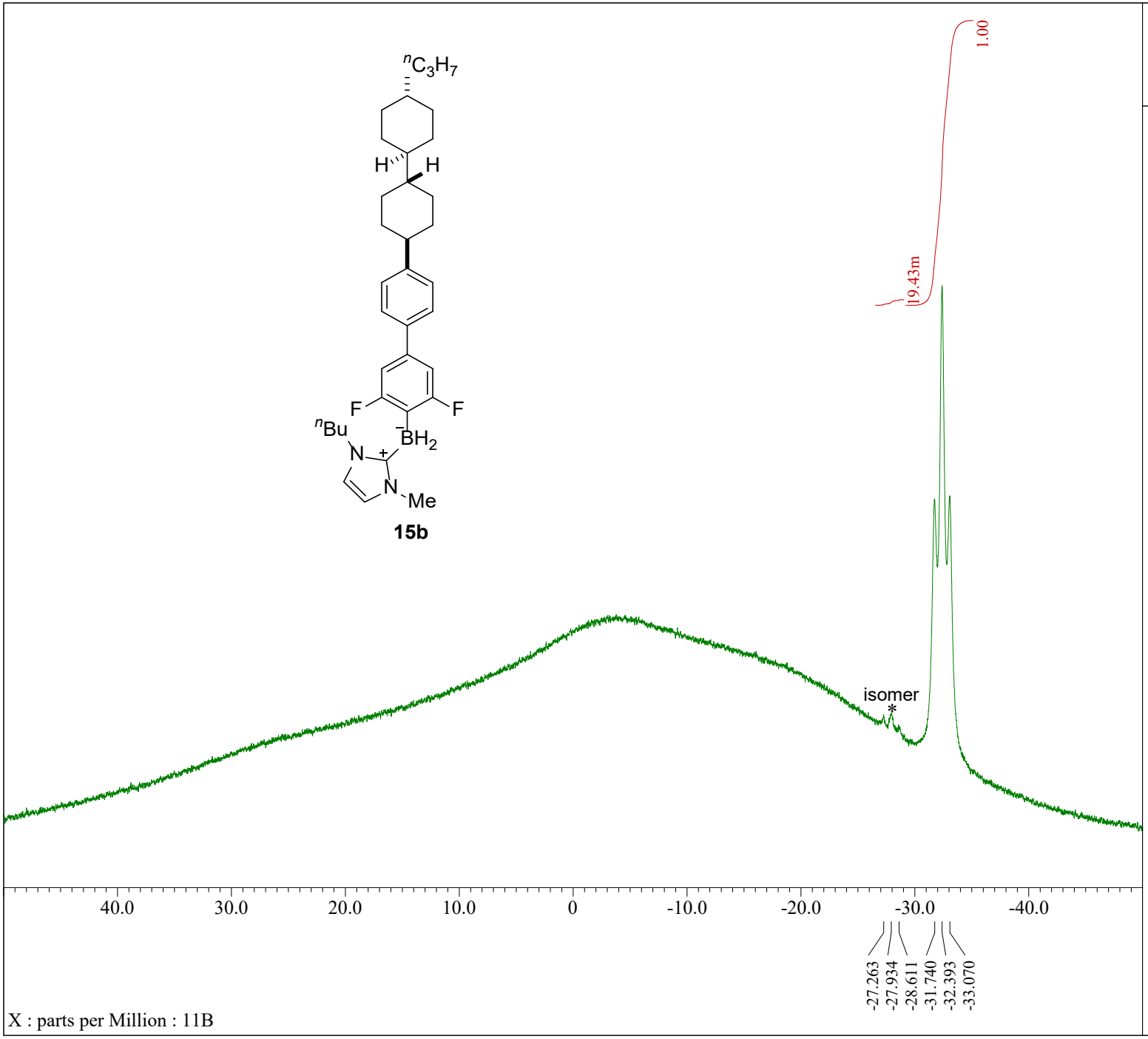
JEOL
Solutions for Innovation

```
Filename         = KT-190-purified-13C_1-1.jdf
Author           = nmrsu
Experiment       = zgpg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 13:27:59
Revision_Time    =  3-FEB-2020 15:38:41
Current_Time     =  6-FEB-2020 16:26:37

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 13C
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 13C
X_Freq           = 100.6228298[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 10.061277[kHz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 23.80952381[kHz]
Scans            = 587

Temp_Get         = 298.1506[K]
Filter_Factor    = 840
```



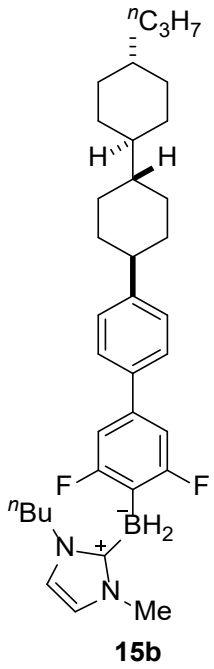
JEOL Solutions for Innovation

```
Filename         = KT-190-purified-11B_1-2.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 13:27:37
Revision_Time    =  6-FEB-2020 16:27:07
Current_Time     =  6-FEB-2020 16:27:25

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 11B
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 11B
X_Freq           = 128.37760517[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 0[Hz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 25.90673575[kHz]
Scans            = 128

Temp_Get         = 298.1508[K]
Filter_Factor    = 772
```



15b

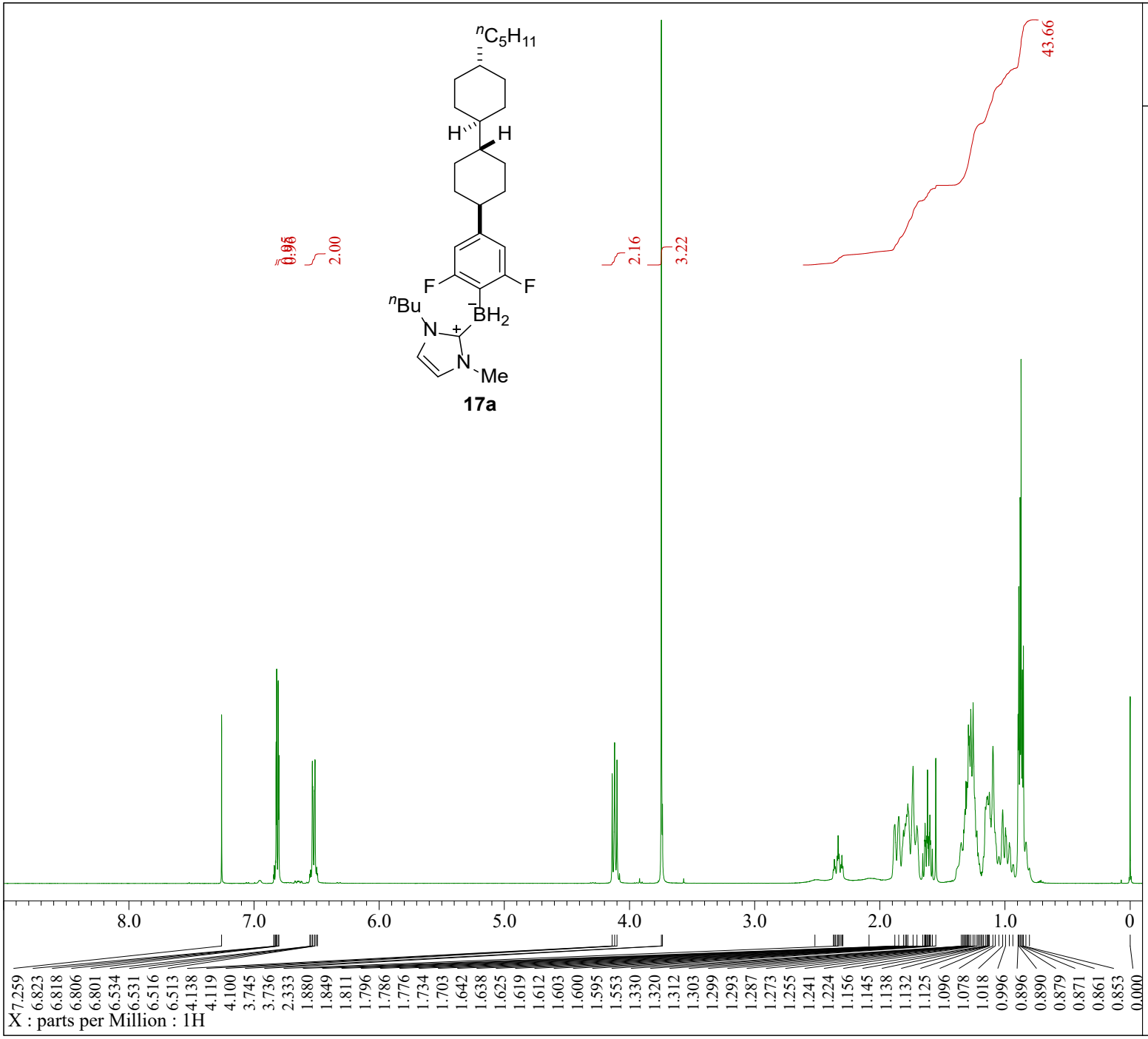
JEOL Solutions for Innovation

```
Filename         = KT-190-purified-19F_1-1.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 13:29:09
Revision_Time    =  3-FEB-2020 16:37:30
Current_Time     =  6-FEB-2020 16:28:54

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 65536
Dim_Title        = 19F
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
X_Prescans       = 4
X_Sweep          = 90.90909091[kHz]
Scans            = 16

Temp_Get         = 298.1483[K]
Filter_Factor    = 220
```



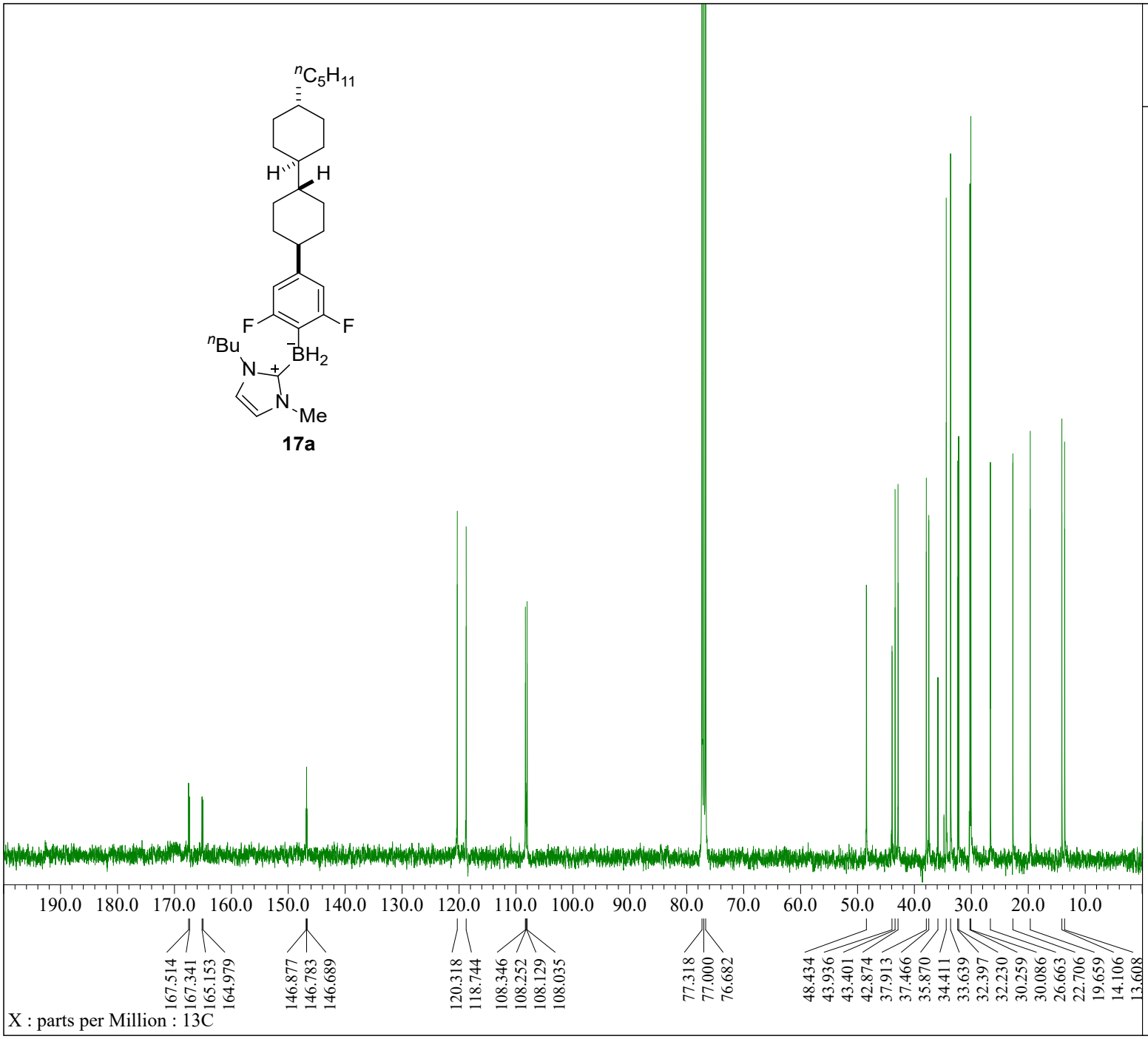
JEOL
Solutions for Innovation

```
Filename        = KT-189-purified-1H_1-1.jdf
Author          = nmrsu
Experiment      = zg30
Sample_Id       = Parameter file, TopSpin 4.0.
Solvent         = CHLOROFORM-D
Creation_Time   = 22-JAN-2020 13:25:25
Revision_Time   =  3-FEB-2020 16:56:20
Current_Time    =  6-FEB-2020 16:30:09

Comment         = Parameter file, TopSpin 4.0.
Data_Format     = 1D COMPLEX
Dim_Size        = 32768
Dim_Title       = 1H
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 1H
X_Freq          = 400.1324708[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 2.470802[kHz]
X_Points        = 32768
X_Prescans      = 2
X_Sweep         = 8.19672131[kHz]
Scans           = 16

Temp_Get        = 298.1472[K]
Filter_Factor   = 2440
```



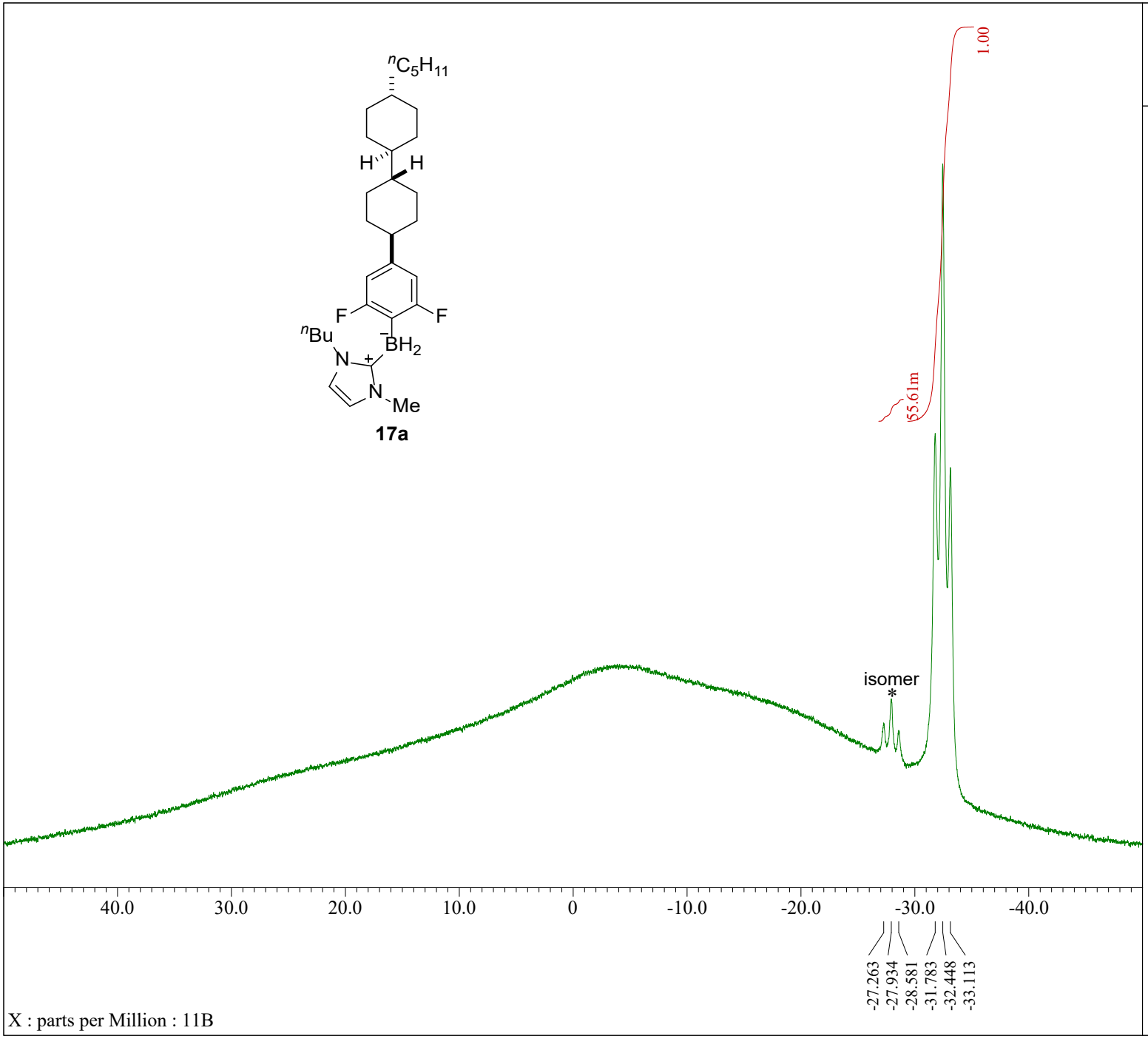
JEOL Solutions for Innovation

```
Filename        = KT-189-purified-13C_1-1.jdf
Author          = nmrsu
Experiment      = zgpg30
Sample_Id       = Parameter file, TopSpin 4.0.
Solvent         = CHLOROFORM-D
Creation_Time   = 22-JAN-2020 13:22:06
Revision_Time   =  3-FEB-2020 17:01:25
Current_Time    =  6-FEB-2020 16:31:19

Comment         = Parameter file, TopSpin 4.0.
Data_Format     = 1D COMPLEX
Dim_Size        = 32768
Dim_Title       = 13C
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 13C
X_Freq          = 100.6228298[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 10.061277[kHz]
X_Points        = 32768
X_Prescans      = 4
X_Sweep         = 23.80952381[kHz]
Scans           = 1024

Temp_Get        = 298.152[K]
Filter_Factor   = 840
```



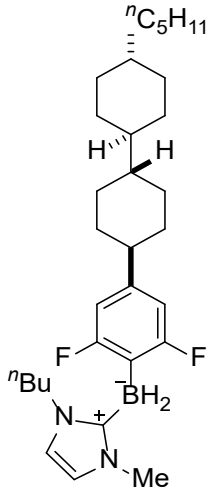
JEOL Solutions for Innovation

```
Filename         = KT-189-purified-11B_1-2.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 13:21:20
Revision_Time    =  6-FEB-2020 16:31:52
Current_Time     =  6-FEB-2020 16:32:18

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 11B
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 11B
X_Freq           = 128.37760517[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 0[Hz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 25.90673575[kHz]
Scans            = 128

Temp_Get         = 298.1497[K]
Filter_Factor    = 772
```



17a

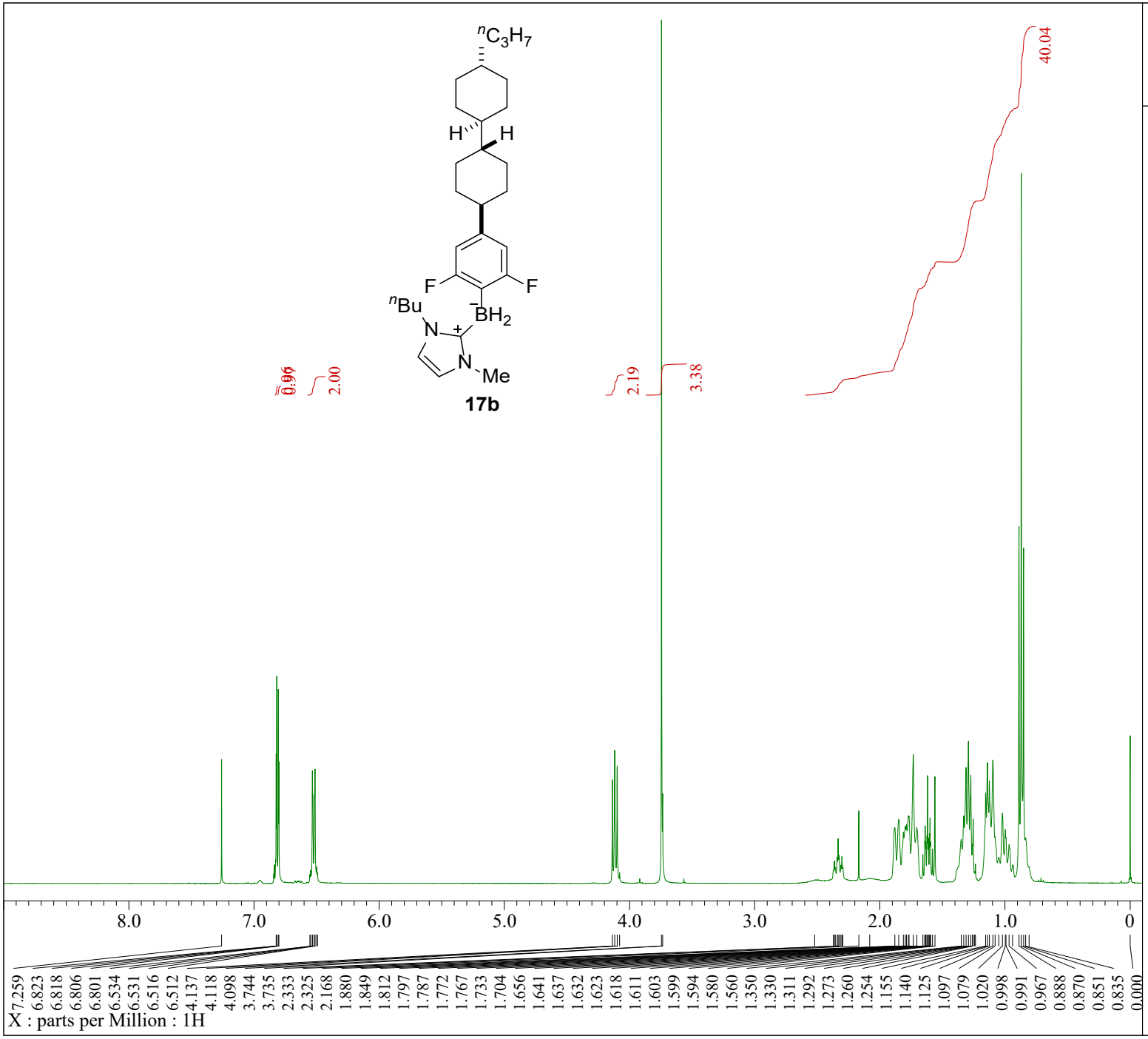
JEOL Solutions for Innovation

```
Filename        = KT-189-purified-19F_1-1.jdf
Author          = nmrsu
Experiment      = zg
Sample_Id       = Parameter file, TopSpin 4.0.
Solvent         = CHLOROFORM-D
Creation_Time   = 22-JAN-2020 13:22:30
Revision_Time   =  3-FEB-2020 18:03:50
Current_Time    =  6-FEB-2020 16:33:09

Comment         = Parameter file, TopSpin 4.0.
Data_Format     = 1D REAL
Dim_Size        = 65536
Dim_Title       = 19F
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 19F
X_Freq          = 376.46071638[MHz]
X_Freq_Flip     = TRUE
X_Offset        = -37.649837[kHz]
X_Points        = 65536
X_Prescans      = 4
X_Sweep         = 90.90909091[kHz]
Scans           = 16

Temp_Get        = 298.145[K]
Filter_Factor   = 220
```



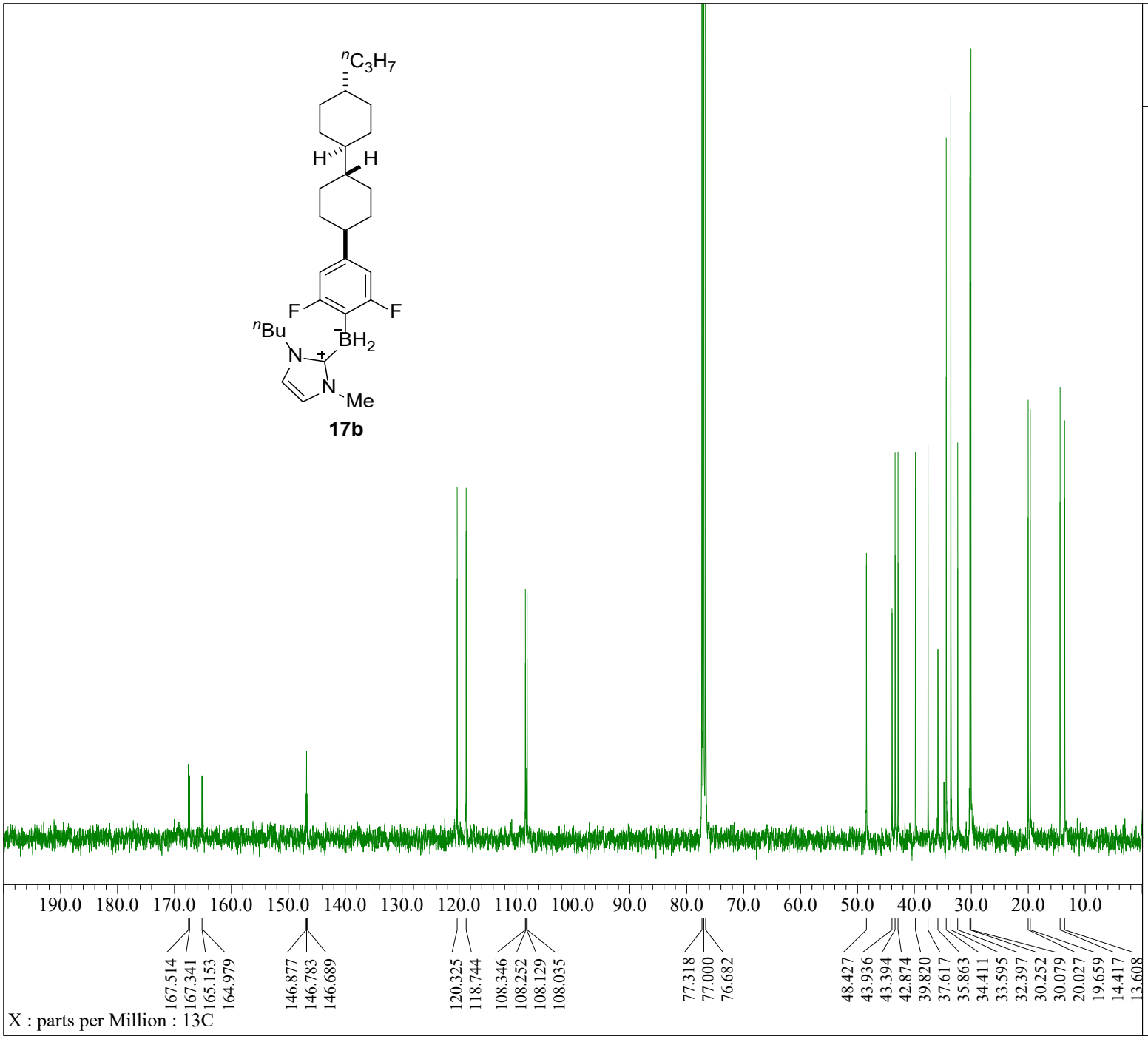
JEOL Solutions for Innovation

```
Filename         = KT-186-purified-1H_1-1.jdf
Author           = nmrsu
Experiment       = zg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 13:20:55
Revision_Time    =  3-FEB-2020 18:15:57
Current_Time     =  6-FEB-2020 16:34:11

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 1H
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength   = 9.39793489[T] (400[MHz])
X_Domain         = 1H
X_Freq           = 400.1324708[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 2.470802[kHz]
X_Points         = 32768
X_Prescans       = 2
X_Sweep          = 8.19672131[kHz]
Scans            = 16

Temp_Get         = 298.1479[K]
Filter_Factor    = 2440
```



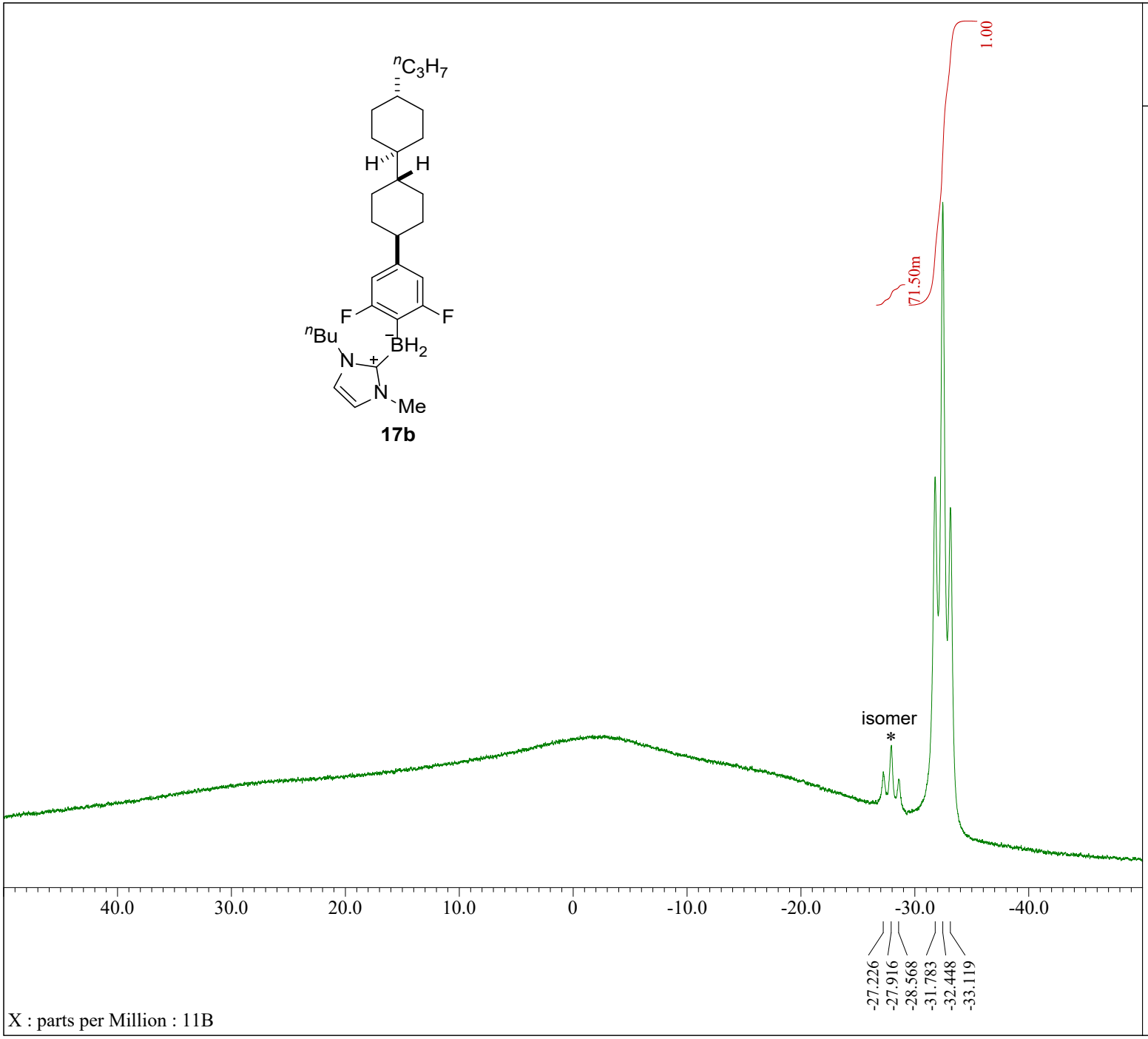
JEOL
Solutions for Innovation

```
Filename        = KT-186-purified-13C_1-1.jdf
Author          = nmrsu
Experiment      = zgpg30
Sample_Id       = Parameter file, TopSpin 4.0.
Solvent         = CHLOROFORM-D
Creation_Time   = 22-JAN-2020 13:19:24
Revision_Time   =  3-FEB-2020 18:20:53
Current_Time    =  6-FEB-2020 16:35:07

Comment         = Parameter file, TopSpin 4.0.
Data_Format     = 1D COMPLEX
Dim_Size        = 32768
Dim_Title       = 13C
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 13C
X_Freq          = 100.6228298[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 10.061277[kHz]
X_Points        = 32768
X_Prescans      = 4
X_Sweep         = 23.80952381[kHz]
Scans           = 527

Temp_Get        = 298.1494[K]
Filter_Factor   = 840
```



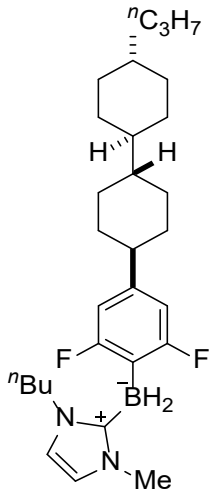
JEOL Solutions for Innovation

```
Filename         = KT-186-purified-11B_1-2.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 13:17:23
Revision_Time    =  6-FEB-2020 16:35:46
Current_Time     =  6-FEB-2020 16:36:31

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 11B
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 11B
X_Freq           = 128.37760517[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 0[Hz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 25.90673575[kHz]
Scans            = 128

Temp_Get         = 298.1525[K]
Filter_Factor    = 772
```



17b

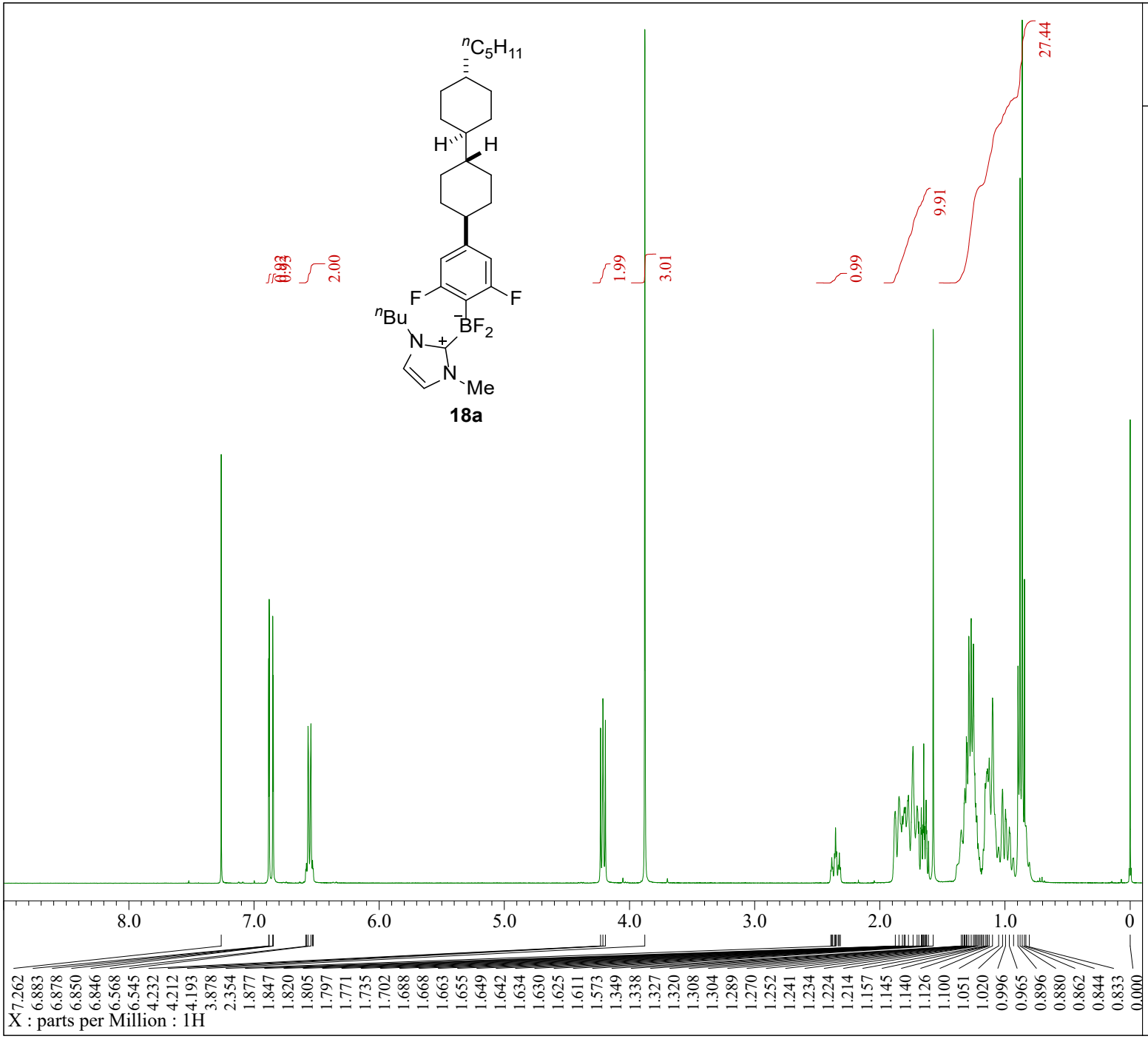
JEOL Solutions for Innovation

```
Filename        = KT-186-purified-19F_1-1.jdf
Author          = nmrsu
Experiment      = zg
Sample_Id       = Parameter file, TopSpin 4.0.
Solvent         = CHLOROFORM-D
Creation_Time   = 22-JAN-2020 13:19:46
Revision_Time   =  3-FEB-2020 18:33:46
Current_Time    =  6-FEB-2020 16:37:13

Comment         = Parameter file, TopSpin 4.0.
Data_Format     = 1D REAL
Dim_Size        = 65536
Dim_Title       = 19F
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 19F
X_Freq          = 376.46071638[MHz]
X_Freq_Flip     = TRUE
X_Offset        = -37.649837[kHz]
X_Points        = 65536
X_Prescans      = 4
X_Sweep         = 90.90909091[kHz]
Scans           = 16

Temp_Get        = 298.1417[K]
Filter_Factor   = 220
```



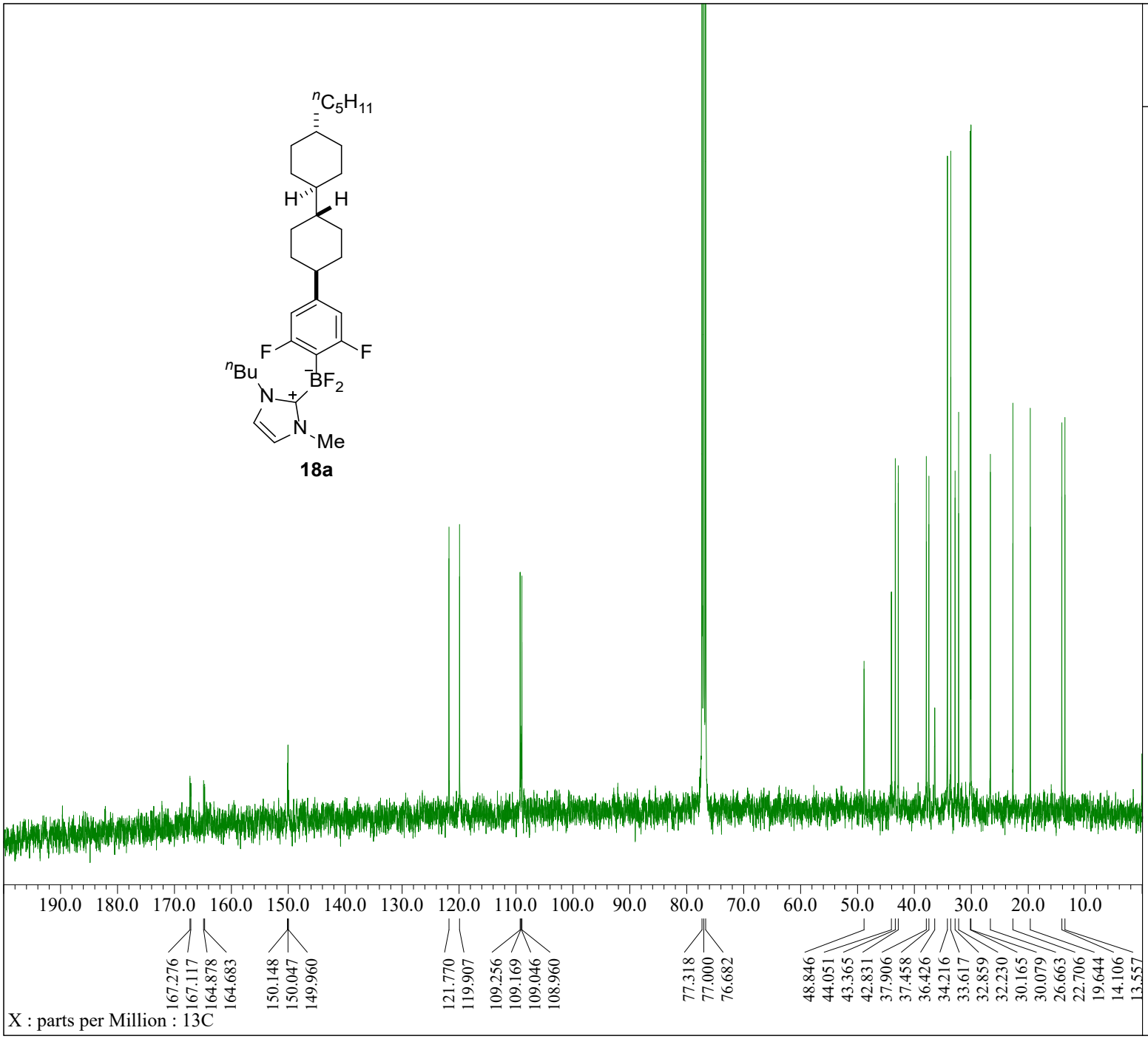
JEOL Solutions for Innovation

```
Filename         = KT-191-purified-1H_1-1.jdf
Author           = nmrsu
Experiment       = zg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 14:09:29
Revision_Time    =  3-FEB-2020 18:48:12
Current_Time     =  6-FEB-2020 16:38:07

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 1H
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength   = 9.39793489[T] (400[MHz])
X_Domain         = 1H
X_Freq           = 400.1324708[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 2.470802[kHz]
X_Points         = 32768
X_Prescans       = 2
X_Sweep          = 8.19672131[kHz]
Scans            = 16

Temp_Get         = 298.147[K]
Filter_Factor    = 2440
```



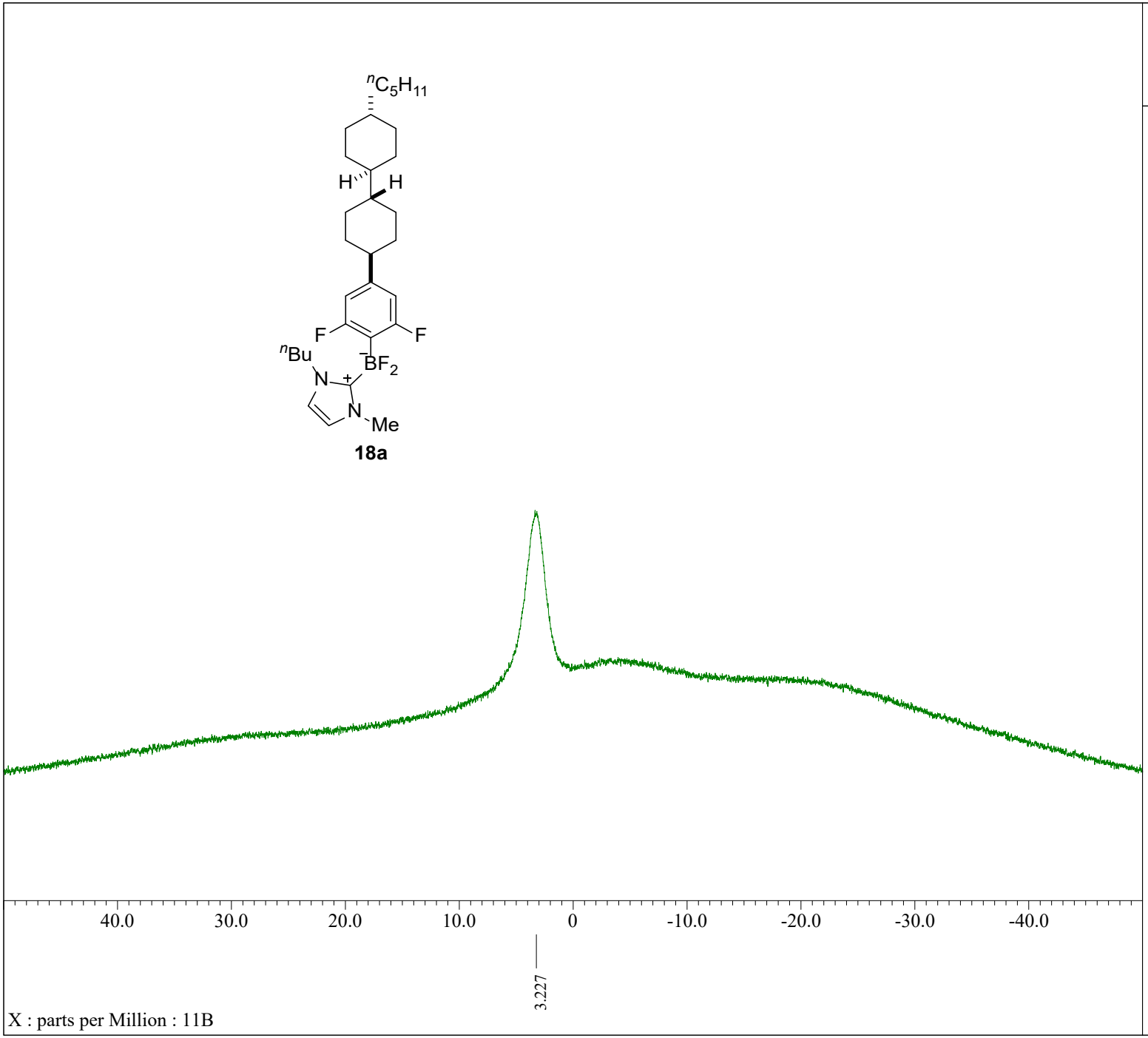
JEOL Solutions for Innovation

```
Filename        = KT-191-purified-13C_1-1.jdf
Author          = nmrsu
Experiment      = zgpg30
Sample_Id       = Parameter file, TopSpin 4.0.
Solvent         = CHLOROFORM-D
Creation_Time   = 22-JAN-2020 14:07:48
Revision_Time   =  3-FEB-2020 18:54:36
Current_Time    =  6-FEB-2020 16:39:08

Comment         = Parameter file, TopSpin 4.0.
Data_Format     = 1D COMPLEX
Dim_Size        = 32768
Dim_Title       = 13C
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 13C
X_Freq          = 100.6228298[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 10.061277[kHz]
X_Points        = 32768
X_Prescans      = 4
X_Sweep         = 23.80952381[kHz]
Scans           = 1024

Temp_Get        = 298.1489[K]
Filter_Factor   = 840
```



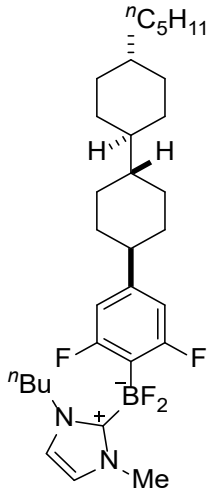
JEOL Solutions for Innovation

```
Filename         = KT-191-purified-11B_1-1.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 13:30:44
Revision_Time    =  3-FEB-2020 19:01:26
Current_Time     =  6-FEB-2020 16:39:53

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 11B
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 11B
X_Freq           = 128.37760517[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 0[Hz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 25.90673575[kHz]
Scans            = 128

Temp_Get         = 298.1516[K]
Filter_Factor    = 772
```



18a

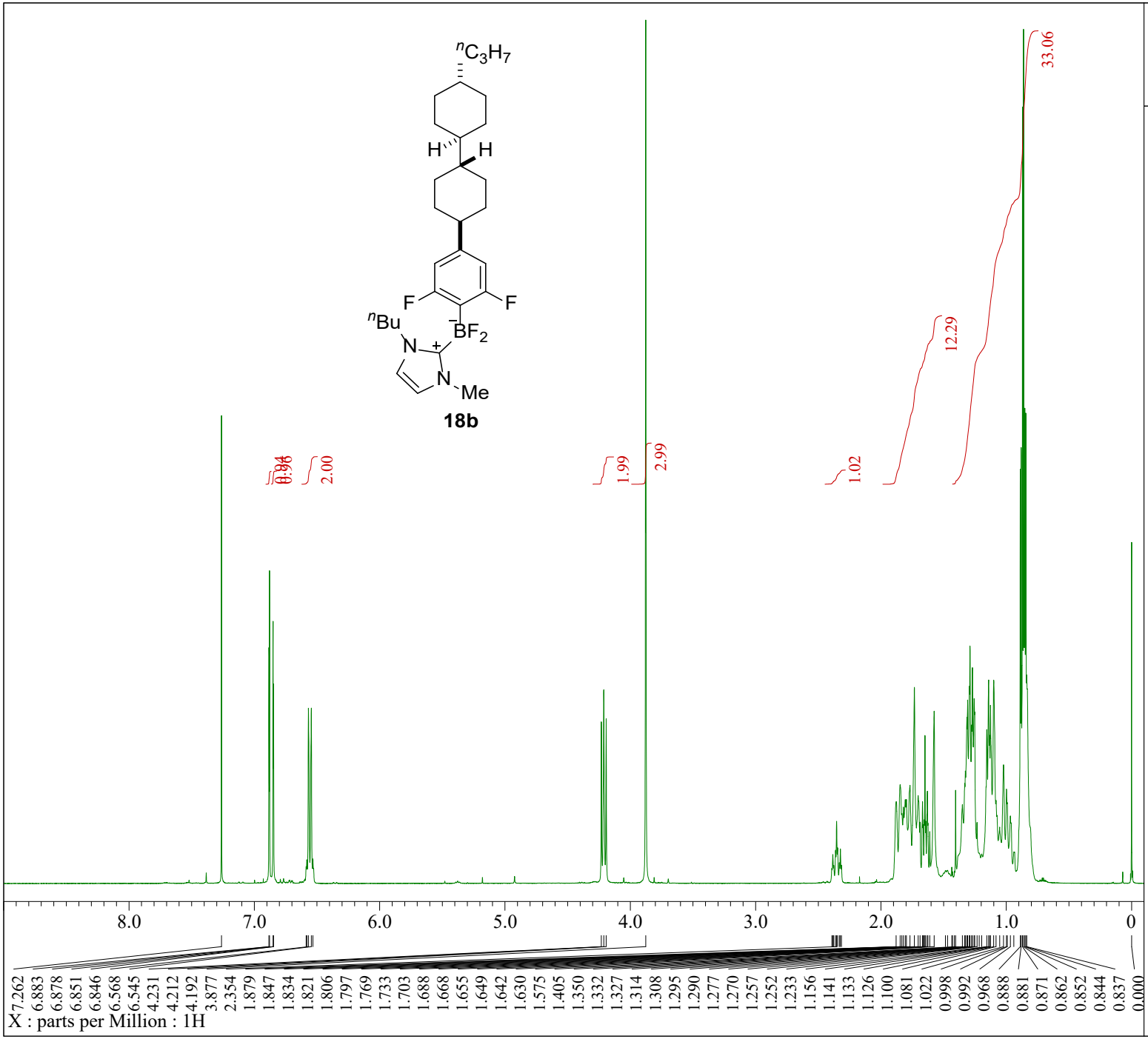
JEOL Solutions for Innovation

```
Filename         = KT-191-purified-19F_1-2.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 14:08:09
Revision_Time    =  6-FEB-2020 16:40:24
Current_Time     =  6-FEB-2020 16:40:43

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 65536
Dim_Title        = 19F
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
X_Prescans       = 4
X_Sweep          = 90.90909091[kHz]
Scans            = 16

Temp_Get         = 298.1544[K]
Filter_Factor    = 220
```



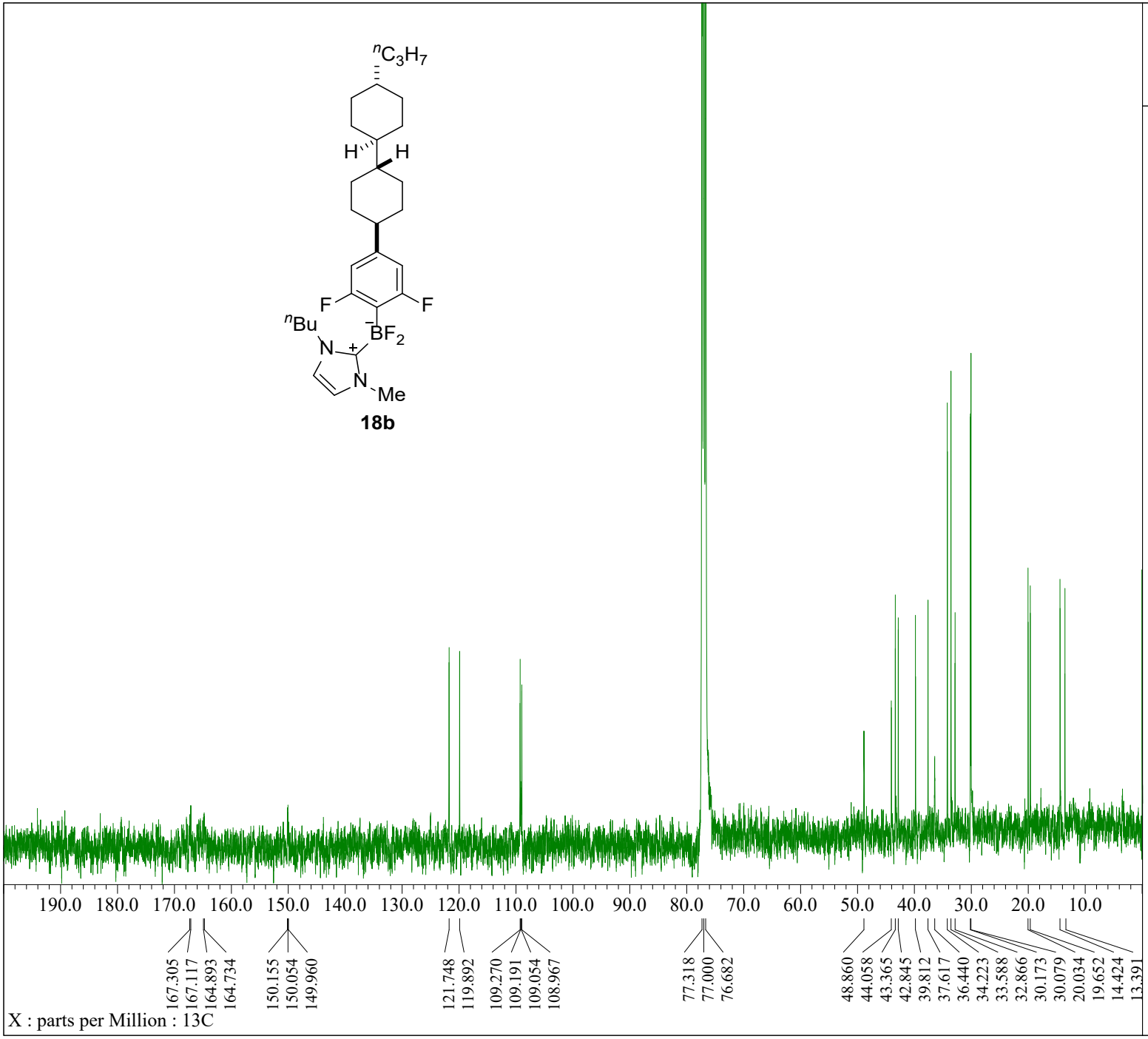
JEOL Solutions for Innovation

```
Filename         = KT-193-purified-1H_1-1.jdf
Author           = nmrsu
Experiment       = zg30
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 14:17:09
Revision_Time    =  3-FEB-2020 19:41:48
Current_Time     =  6-FEB-2020 16:41:32

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 1H
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength   = 9.39793489[T] (400[MHz])
X_Domain         = 1H
X_Freq           = 400.1324708[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 2.470802[kHz]
X_Points         = 32768
X_Prescans       = 2
X_Sweep          = 8.19672131[kHz]
Scans            = 16

Temp_Get         = 298.1467[K]
Filter_Factor    = 2440
```



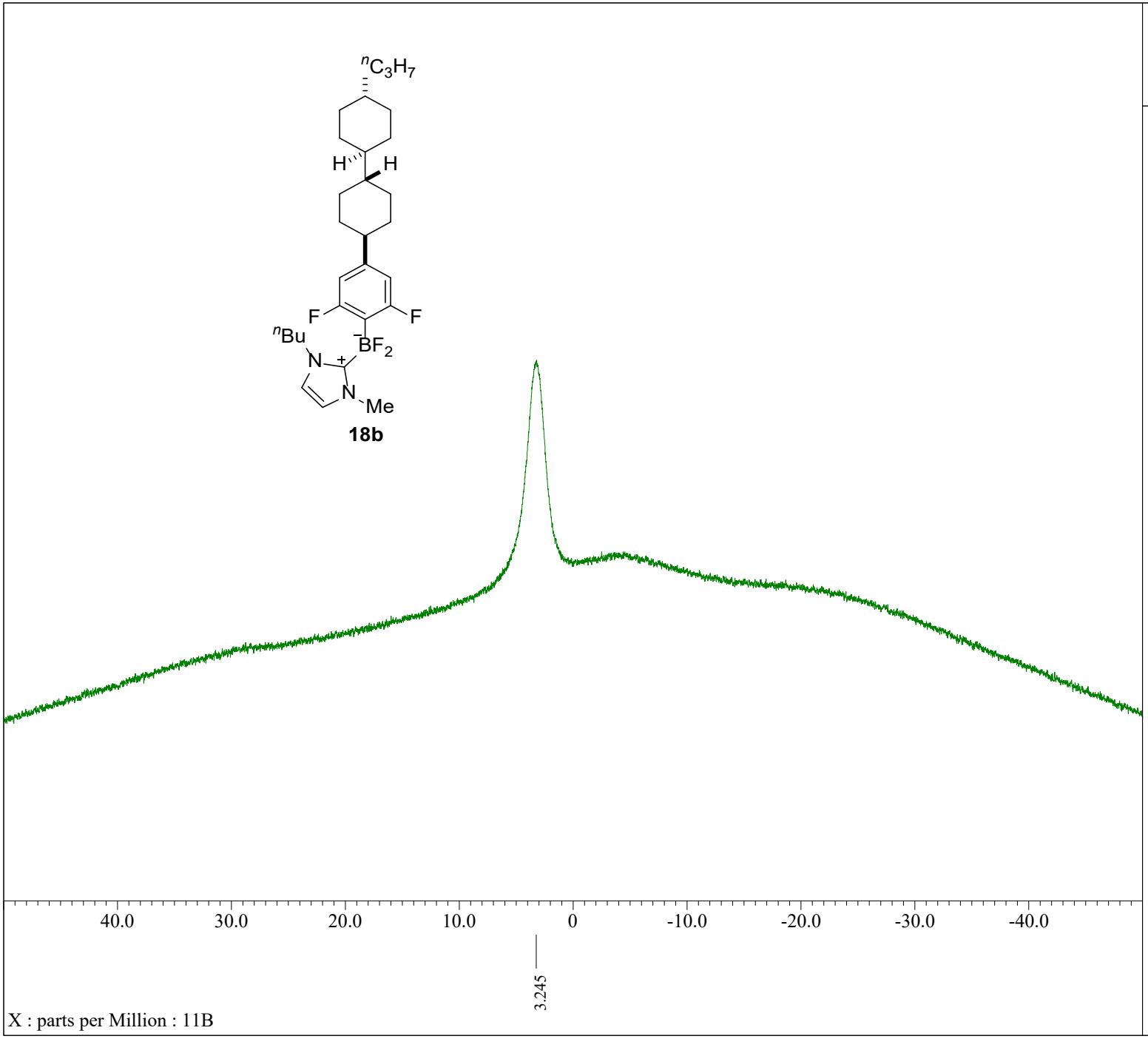
JEOL Solutions for Innovation

```
Filename        = KT-193-purified-13C-re_1-1.j
Author          = nmrsu
Experiment      = zgpg30
Sample_Id       = Parameter file, TopSpin 4.0.
Solvent         = CHLOROFORM-D
Creation_Time   =  5-FEB-2020 09:57:49
Revision_Time   =  5-FEB-2020 10:23:12
Current_Time    =  6-FEB-2020 16:43:07

Comment         = Parameter file, TopSpin 4.0.
Data_Format     = 1D COMPLEX
Dim_Size        = 32768
Dim_Title       = 13C
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain        = 13C
X_Freq          = 100.6228298[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 10.061277[kHz]
X_Points        = 32768
X_Prescans      = 4
X_Sweep         = 23.80952381[kHz]
Scans           = 11814

Temp_Get        = 298.1495[K]
Filter_Factor   = 840
```



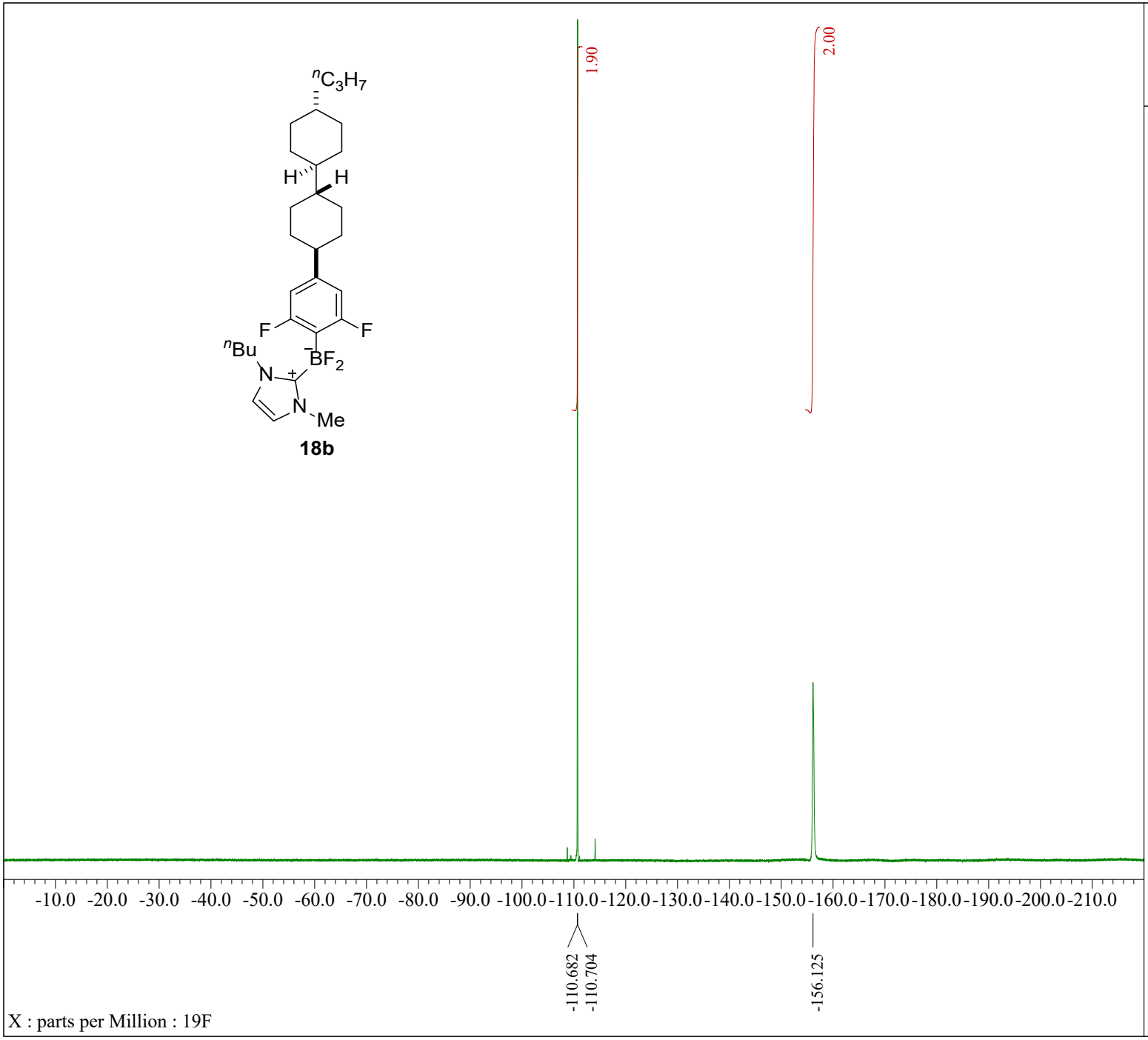
JEOL Solutions for Innovation

```
Filename         = KT-193-purified-11B_1-1.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 14:10:52
Revision_Time    =  3-FEB-2020 20:31:04
Current_Time     =  6-FEB-2020 16:44:17

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
Dim_Title        = 11B
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 11B
X_Freq           = 128.37760517[MHz]
X_Freq_Flip      = TRUE
X_Offset         = 0[Hz]
X_Points         = 32768
X_Prescans       = 4
X_Sweep          = 25.90673575[kHz]
Scans            = 128

Temp_Get         = 298.1522[K]
Filter_Factor    = 772
```



JEOL Solutions for Innovation

```
Filename         = KT-193-purified-19F_1-1.jdf
Author           = nmrsu
Experiment       = zg
Sample_Id        = Parameter file, TopSpin 4.0.
Solvent          = CHLOROFORM-D
Creation_Time    = 22-JAN-2020 14:15:19
Revision_Time    =  3-FEB-2020 20:49:38
Current_Time     =  6-FEB-2020 16:45:04

Comment          = Parameter file, TopSpin 4.0.
Data_Format      = 1D REAL
Dim_Size         = 65536
Dim_Title        = 19F
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 19F
X_Freq           = 376.46071638[MHz]
X_Freq_Flip      = TRUE
X_Offset         = -37.649837[kHz]
X_Points         = 65536
X_Prescans       = 4
X_Sweep          = 90.90909091[kHz]
Scans            = 16

Temp_Get         = 298.1463[K]
Filter_Factor    = 220
```