Electronic Supplementary Information for Dalton Transactions

Frustrated Lewis pairs: Reactivities of TMS protected amines and phosphines in the presence of $B(C_6F_5)_3$

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SUPPLEMENTARY INFORMATION

Contents

Materials and Methods:

All experiments were performed on double-manifold H_2(Ar)/vacuum lines or in an argon glove box (MBraun Labmaster 130). Solvents were dried by an MBraun solvent purification system (MB SPS-800). Hydrogen gas was purchased from AGA Ab and passed through a drying unit prior to use. All organic reagents were purchased from Acros Organics, Sigma-Aldrich or Strem and purified by conventional methods. NMR experiments were performed on a Bruker ARX-300 spectrometer (^{1}H, ^{11}B, ^{13}C, ^{19}F), a Varian Gemini 300 (^{1}H, ^{13}C, ^{19}F), or on a Varian INOVA 500 (^{31}P). ^{1}H, ^{13}C NMR spectra are referenced to $SiMe_4$ by referencing the residual solvent peak. ^{11}B, ^{19}F, and ^{31}P NMR spectra are referenced externally to BF_3*Et_2O at 0 ppm, CF_3CO_2H at -78.5 ppm relative to $CFCl_3$ at 0 ppm, and to 85% H_3PO_4 at 0 ppm, respectively. 9-Trimethylsilyl-9*H*-carbazole[1] (**1**), *N*-trimethylsilyldiphenylamine[1] (**2**), *N*-trimethylsilyl-*t*-butylamine[2] (**4a**), and *N*-trimethylsilyldi-*t*-butylphosphine[3] (**6a**) were prepared by literature methods.

***N*-trimethylsilyl-2,4,6-trimethylaniline (3a)**[4]

In a 50 mL Schlenk tube a solution of 2,4,6-aniline (3.16 g, 23.4 mmol) in dry THF (10 mL) was cooled to -20 °C and *n*-BuLi (2.5 M solution in hexanes, 9.8 mL, 24.5 mmol) was added dropwise. The reaction mixture was allowed to warm to room temperature and stirred for 30 min. After cooling it to -78 °C a solution of trimethylsilylchloride (2.67 g, 24.6 mmol) in THF (5 mL) was added over 30 min, the mixture was allowed to warm to room temperature, and then stirred for 18 h. The THF, hexane and residual trimethylsilylchloride were removed *via* distillation, dry pentane (20 mL) was added and the contents of the Schlenk tube filtered. Further portions of dry pentane (2 * 10 mL) were used to wash the LiCl and the filtrates were combined. The pentane was removed *via* distillation under argon. The residue was distilled under reduced pressure (1.3 mbar, 75 °C) yielding **3a** (4.46 g, 92%) as a colorless liquid.

^{1}H NMR (C_6D_6, 300 MHz): δ 6.82 (s, 2H, C_6H_2), δ 2.19 (s, 3H, 4-$C_6H_2CH_3$), δ 2.18 (s, 6H, 2,6-$C_6H_2(CH_3)_2$), δ 1.96 (s, 1H, NH_2), δ 0.09 (s, 9H, $Si(CH_3)_3$).

^{13}C NMR (C_6D_6, 75 MHz): δ 141.20 (s, 1-C_6H_2), δ 132.59 (s, 2,6-C_6H_2), δ 131.43 (s, 4-C_6H_2), δ 129.69 (s, 3,5-C_6H_2), δ 21.18 (s, 4-$C_6H_2CH_3$), δ 20.14 (s, 2,6-$C_6H_2(CH_3)_2$), δ 1.47 (s, $Si(CH_3)_3$).

***N*-trimethylsilyldiisopropylamine (5a)**

In a 100 mL Schlenk tube a solution of diisopropylamine (10.12 g, 0.1 mol) in dry Et_2O (35 mL) was cooled to -20 °C and *n*-BuLi (2.5 M solution in hexanes, 42 mL, 0.105 mol) was added dropwise. The reaction mixture was allowed to warm to room temperature and was stirred for 30 min. After cooling it to -78 °C a solution of trimethylsilylchloride (11.41 g, 0.105 mol) in Et_2O (15 mL) was added over 30 min, the mixture was allowed to warm to room temperature, and then stirred for 18 h. The Et_2O, hexane and residual trimethylsilylchloride were removed *via* distillation, dry pentane (40 mL) was added, and the contents of the Schlenk tube filtered. Further portions of dry pentane (2 * 10 mL) were used to wash the LiCl, and the filtrates were combined. The pentane was removed *via* distillation under argon. The residue was distilled twice (157 °C) yielding **5a** (1.21 g, 7%) as a colorless liquid.

^{1}H NMR (Tol-D_8, 300 MHz): δ 3.11 (spt, 2H, $^{3}J_{HH}$ = 6.8 Hz, C*H*), δ 1.03 (d, 12H, $CH(CH_3)_2$), δ 0.14 (s, 9H, $Si(CH_3)_3$).

^{13}C NMR (Tol-D_8, 75 MHz): δ 45.52 (s, *C*H), δ 24.55 (s, $CH(CH_3)_2$), δ 2.64 (s, $Si(CH_3)_3$).

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[1] Smith, C. J.; Tsang, M. W. S.; Holmes, A. B.; Danheiser, R. L.; Tester, J. W. *Org. Biomol. Chem.* **2005**, *3*, 3767-3781.
[2] Courtois, G.; Miginiac, L. *J. Organomet. Chem.* **1988**, *340*, 127-141.
[3] Wolfsberger, W.; Burkart, W.; Bauer, S.; Hampp, A.; Wolf, J.; Werner, H. *Z. Naturforsch. B* **1994**, *49*, 1659-1673.
[4] cf.: Murugavel, R.; Chandrasekhar, V.; Voigt, A.; Roesky, H. W.; Schmidt, H. G.; Noltemeyer, M. *Organometallics* **1995**, *14*, 5298-5301.

Adduct of 2,4,6-trimethylaniline with $B(C_6F_5)_3$ (3c)

In a glove box, a 25 mL flame-dried Schlenk tube equipped with a stir bar, a Teflon stopcock and a glass stopper (Glindemann®-sealing rings were used for conical joints instead of grease) was charged with **3a** (20.7 mg, 0.1 mmol), $B(C_6F_5)_3$ (51.2 mg, 0.1 mmol), and dry benzene (1.0 mL). The reaction was degassed once with a freeze-pump-thaw cycle and refilled with H_2 (1.5 bar). It was stirred at 1000 rpm at room temperature for 15 h. All volatiles were removed *in vacuo* and the residue was washed with dry chloroform to give **3c** as a white solid (55.7 mg; 86%).

^{1}H NMR (DMSO-D_6, 300 MHz): δ 6.60 (s, 2H, C_6H_2), δ 4.40 (s, 2H, NH_2), δ 2.08 (s, 3H, 4-$C_6H_2CH_3$), δ 2.05 (s, 6H, 2,6-$C_6H_2(CH_3)_2$).

^{11}B NMR (DMSO-D_6, 96 MHz): δ -0.98 (s).

^{13}C NMR (DMSO-D_6, 75 MHz): δ 147.28 (dm, $^1J_{CF}$ = 243 Hz, *o*-C_6F_5), δ 141.21 (s, 1-C_6H_2), δ 139.16 (dm, $^1J_{CF}$ = 249 Hz, *p*-C_6F_5), δ 136.38 (dm, $^1J_{CF}$ = 248 Hz, *m*-C_6F_5), δ 128.31 (s, 3,5-C_6H_2), δ 124.30 (s, 4-C_6H_2), δ 120.92 (s, 2,6-C_6H_2), δ 118.97 (s, quarternary C of C_6F_5), δ 19.89 (s, 4-$C_6H_2CH_3$), δ 17.60 (s, 2,6-$C_6H_2(CH_3)_2$).

^{19}F NMR (DMSO-D_6, 282 MHz): δ -133.60 (d, 6F, $^3J_{FF}$ = 18 Hz, *o*-C_6F_5), δ -158.33 (t, 3F, $^3J_{FF}$ = 21 Hz, *p*-C_6F_5), δ -164.76 (m, 6F, *m*-C_6F_5).

Adduct of *t*-butylamine with $B(C_6F_5)_3$ (4c)

In a glove box, a 25 mL flame-dried Schlenk tube equipped with a stir bar, a Teflon stopcock and a glass stopper (Glindemann®-sealing rings were used for conical joints instead of grease) was charged with 4**a** (14.5 mg, 0.1 mmol), $B(C_6F_5)_3$ (51.2 mg, 0.1 mmol), and dry benzene (1.0 mL). The reaction was degassed once with a freeze-pump-thaw cycle and refilled with H_2 (1.5 bar). It was stirred at 1000 rpm at room temperature for 15 h. All volatiles were removed *in vacuo* and the residue was washed with dry hexane to give **4c** as a white solid (53.2 mg; 91%).[5]

^{1}H NMR (C_6D_6, 300 MHz): δ 4.38 (s, 2H, NH_2), δ 0.55 (s, 9H, $C(CH_3)_3$).

^{13}C NMR (C_6D_6, 75 MHz): δ 148.30 (dm, $^1J_{CF}$ = 237 Hz, *o*-C_6F_5), δ 140.67 (dm, $^1J_{CF}$ = 246 Hz, *p*-C_6F_5), δ 137.70 (dm, $^1J_{CF}$ = 249 Hz, *m*-C_6F_5), δ 117.20 (s, quarternary C of C_6F_5), δ 57.61 (s, $C(CH_3)_3$), δ 28.17 (s, $C(CH_3)_3$).

^{19}F NMR (C_6D_6, 282 MHz): δ -132.65 (s, 6F, *o*-C_6F_5), δ -156.01 (t, 3F, $^3J_{FF}$ = 21 Hz, *p*-C_6F_5), δ -163.05 (m, 6F, *m*-C_6F_5).

Diisopropylammonium tris(pentafluorophenyl)hydridoborate (5c)

In a glove box, a 25 mL flame-dried Schlenk tube equipped with a stir bar, a Teflon stopcock and a glass stopper (Glindemann®-sealing rings were used for conical joints instead of grease) was charged with **5a** (17.3 mg, 0.1 mmol), $B(C_6F_5)_3$ (51.2 mg, 0.1 mmol), and dry toluene (1.0 mL). The reaction was degassed once with a freeze-pump-thaw cycle and refilled with H_2 (1.5 bar). It was stirred at 1000 rpm at 110 °C for 15 h. All volatiles were removed *in vacuo* and the residue was washed with dry chloroform to give **5c** as a white solid (54.8 mg; 89%).[6]

^{1}H NMR (C_6D_6, 300 MHz): δ 4.30 (br t (1:1:1), 2H, $^1J_{HN}$ = 43 Hz, NH_2), δ 3.62 (br q, 1H, $^1J_{HB}$ = 92 Hz, B*H*), δ 2.34 (m, 2H, $CH(CH_3)_2$), δ 0.47 (d, 12H, $^3J_{HH}$ = 7 Hz, $CH(CH_3)_2$).

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[5] ^{1}H, ^{13}C, and ^{19}F NMR data are in agreement with those already reported for this adduct: Lancaster, S. J.; Mountford, A. J.; Hughes, D. L.; Schormann, M.; Bochmann, M. *J. Organomet. Chem.* **2003**, *680*, 193-205.

[6] ^{1}H, and ^{19}F NMR data are in agreement with those already reported for this salt: Sumerin, V.; Schulz, F.; Nieger, M.; Leskelä, M.; Repo, T.; Rieger, B. *Angew. Chem. Int. Ed.* **2008**, *47*, 6001-6003.

^{13}C NMR (C_6D_6, 75 MHz): δ 148.84 (dm, $^1J_{CF}$ = 236 Hz, *o*-C_6F_5), δ 138.96 (dm, $^1J_{CF}$ = 244 Hz, *p*-C_6F_5), δ 137.40 (dm, $^1J_{CF}$ = 256 Hz, *m*-C_6F_5), δ 50.22 (s, $CH(CH_3)_2$), δ 18.76 (s, $CH(CH_3)_2$). No signals could be detected for the quarternary carbon atoms of the C_6F_5–fragments.

^{19}F NMR (C_6D_6, 282 MHz): δ -133.98 (d, 6F, $^3J_{FF}$ = 21 Hz, *o*-C_6F_5), δ -162.20 (t, 3F, $^3J_{FF}$ = 21 Hz, *p*-C_6F_5), δ -166.10 (m, 6F, *m*-C_6F_5).

***t*-$Bu_2P(C_6F_4)B(C_6F_5)_2$ (6b)**

6a (21.8 mg, 0.1 mmol) in benzene (0.5 mL) was added dropwise to a solution of $B(C_6F_5)_3$ (51.2 mg, 0.1 mmol) in benzene (1.0 mL) over a period of 5 min. Immediately, intense bright orange coloration indicated the formation of the product. The reaction mixture was stirred over night at room temperature and the solvent was removed *in vacuo* to give **6b** (63.8 mg, 100%) as a yellow solid.[7]

^{1}H NMR (C_6D_6, 300 MHz): δ 1.14 (dd, $^3J_{HP}$ = 13 Hz, *J* = 7 Hz, $C(CH_3)_3$).

^{13}C NMR (C_6D_6, 75 MHz): δ 150.22 (dm, $^1J_{CF}$ = 246 Hz, C_6F_4), δ 148.07 (dm, $^1J_{CF}$ = 249 Hz, *o*-C_6F_5), δ 148.11 (dm, $^1J_{CF}$ = 246 Hz, *p*-C_6F_5), δ 145.58 (dm, $^1J_{CF}$ = 255 Hz, C_6F_4), δ 138.08 (dm, $^1J_{CF}$ = 255 Hz, *m*-C_6F_5), δ 120.93 (m, quarternary C of B(C_6F_4)), δ 113.82 (m, quarternary C of B(C_6F_5)$_2$), δ 33.55 (ddd, $^1J_{CP}$ = 27 Hz, *J* = 4 Hz, *J* = 2 Hz, $C(CH_3)_3$), δ 30.62 (dt, $^2J_{CP}$ = 17 Hz, *J* = 4 Hz, $C(CH_3)_3$). No signal could be detected for the quarternary carbon atom of P(C_6F_4).

^{19}F NMR (C_6D_6, 282 MHz): δ -120.14 (m, 1F, *o*-PC_6F_4), δ -125.10 (dm, 1F, $^3J_{FP}$ = 113 Hz, *o*-PC_6F_4), δ -128.88 (m, 4F, *o*-C_6F_5), δ -129.56 (dm, 1F, *J* = 55 Hz, *o*-BC_6F_4), δ -130.36 (dm, 1F, *J* = 85 Hz, *o*-BC_6F_4), δ -142.19 (s, 2F, *p*-C_6F_5), δ -160.49 (s, 4F, *m*-C_6F_5).

^{31}P NMR (C_6D_6, 202 MHz): δ 26.51 (dt, $^3J_{PF}$ = 111 Hz, *J* = 20 Hz).

[7] ^{1}H, ^{13}C, ^{19}F, and ^{31}P NMR data are in agreement with those already reported for this compound: Welch, G. C.; Cabrera, L.; Chase, P. A.; Hollink, E.; Masuda, J. D.; Wei, P. R.; Stephan, D. W. *Dalton Trans.* **2007**, 3407-3414.

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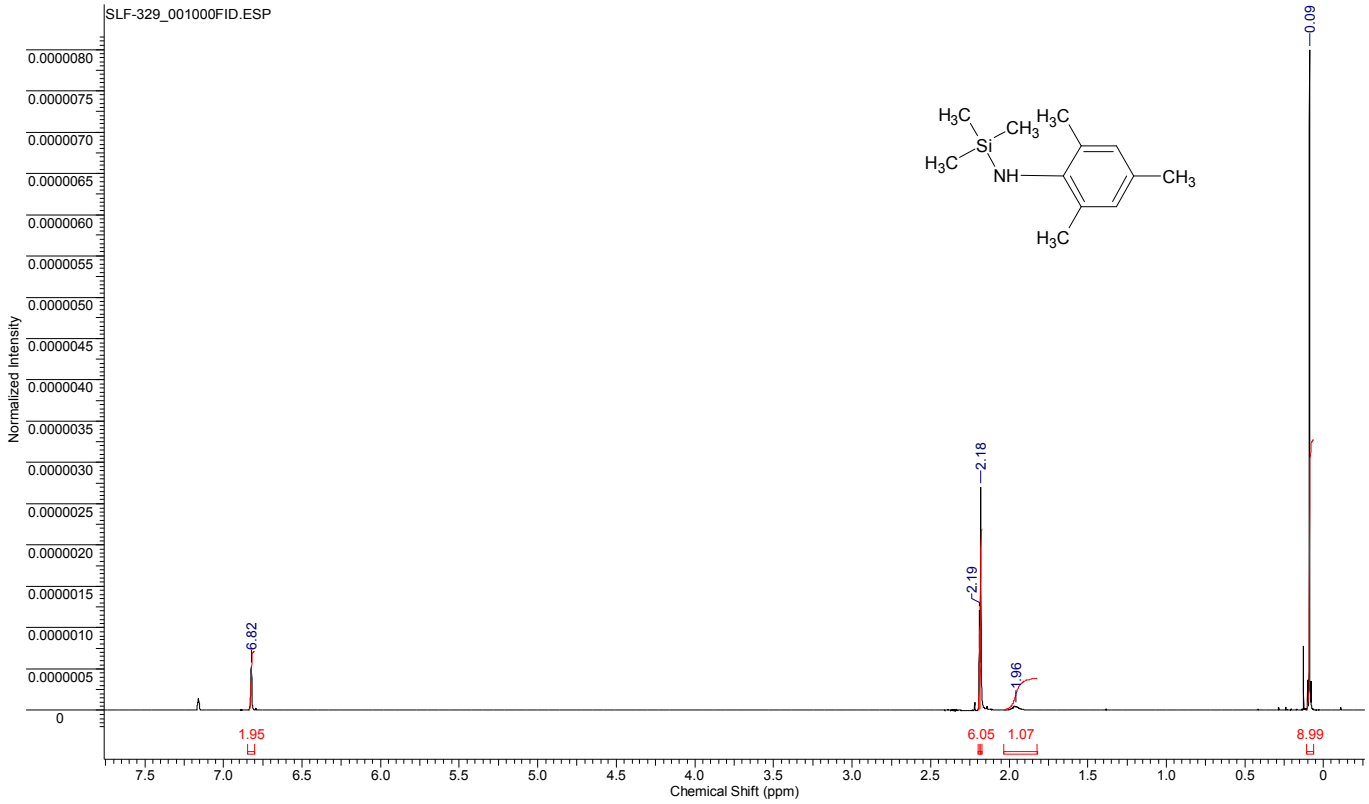

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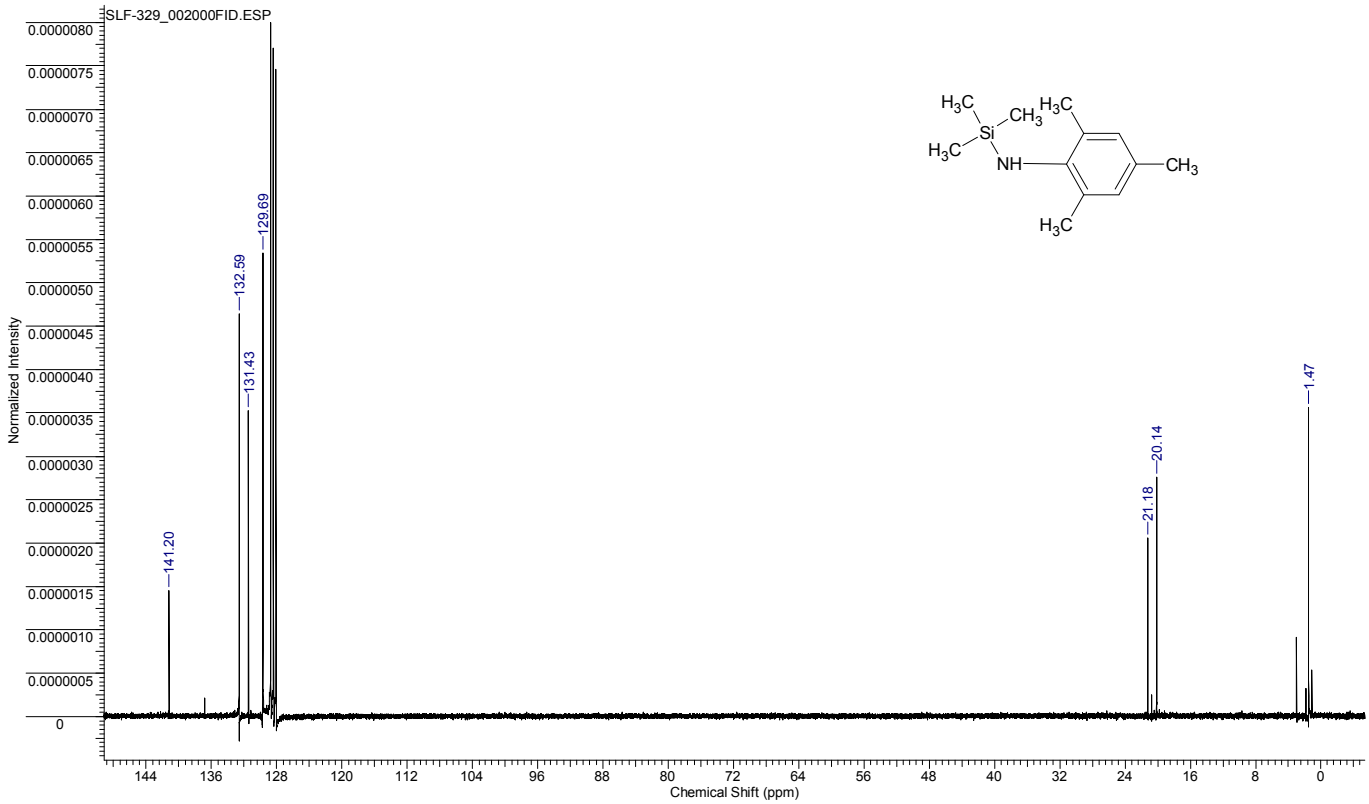

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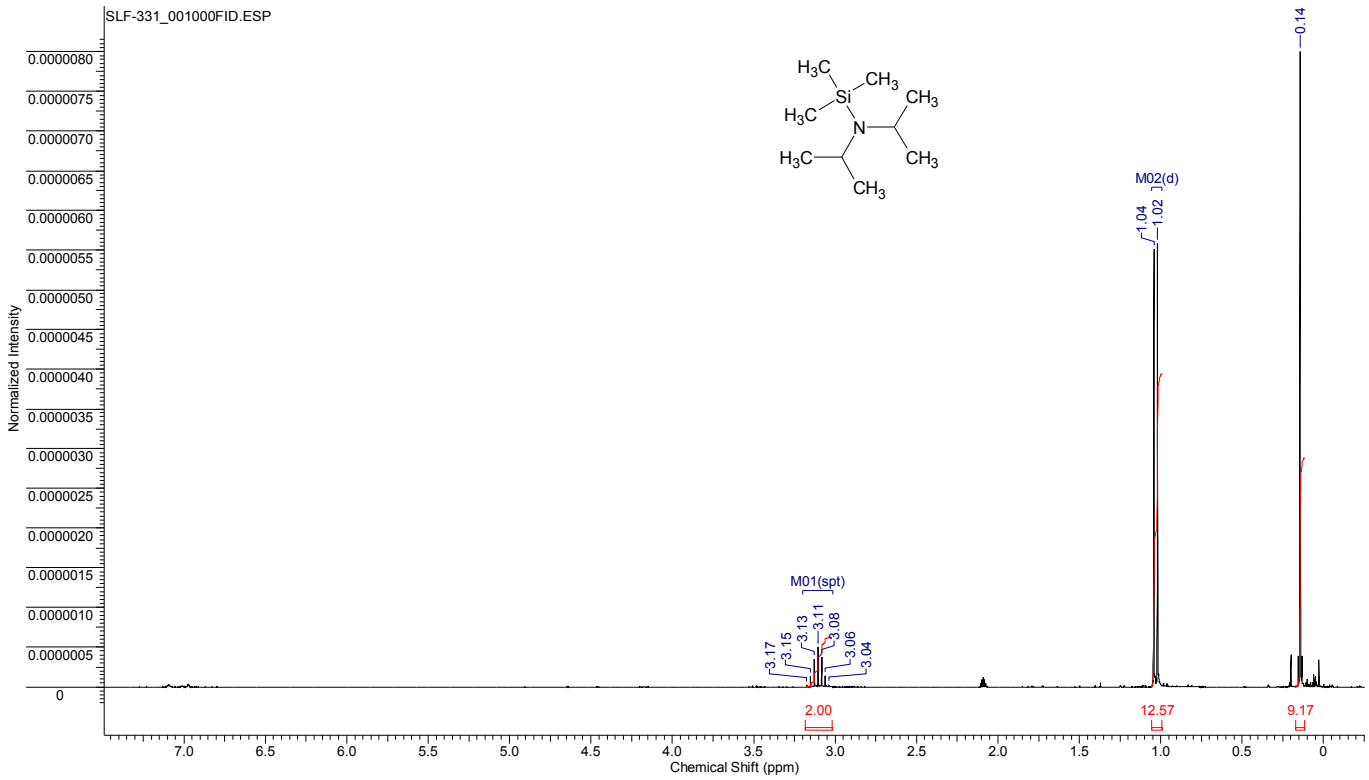

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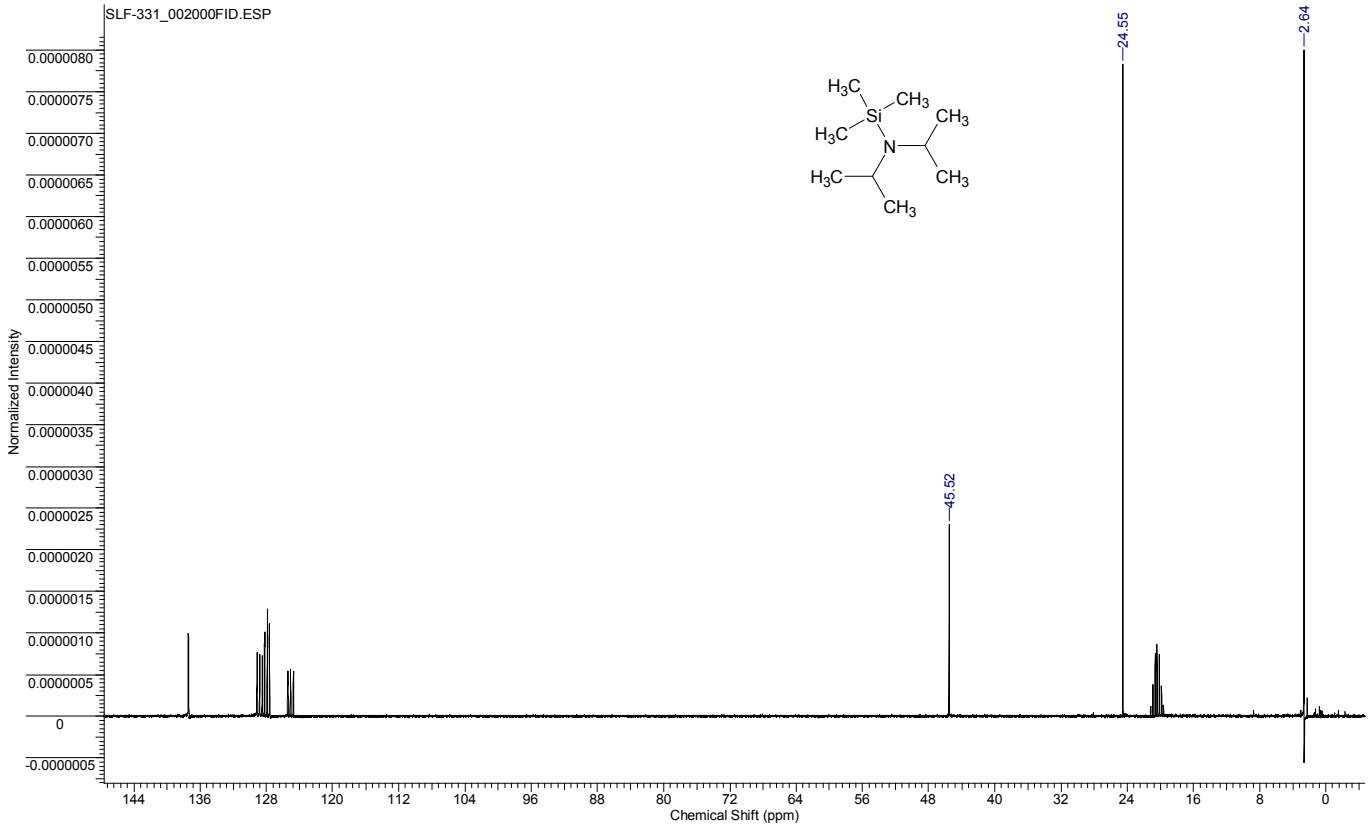

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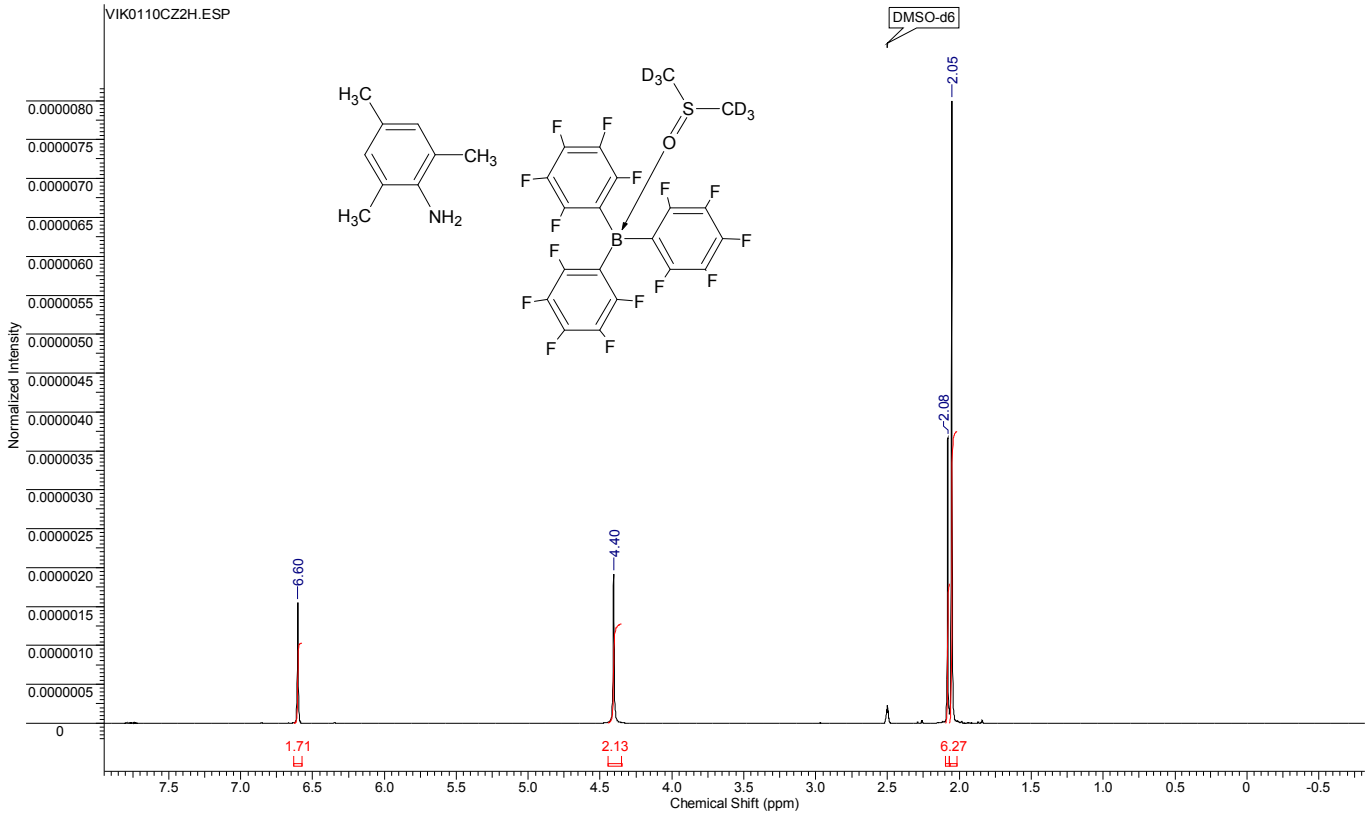

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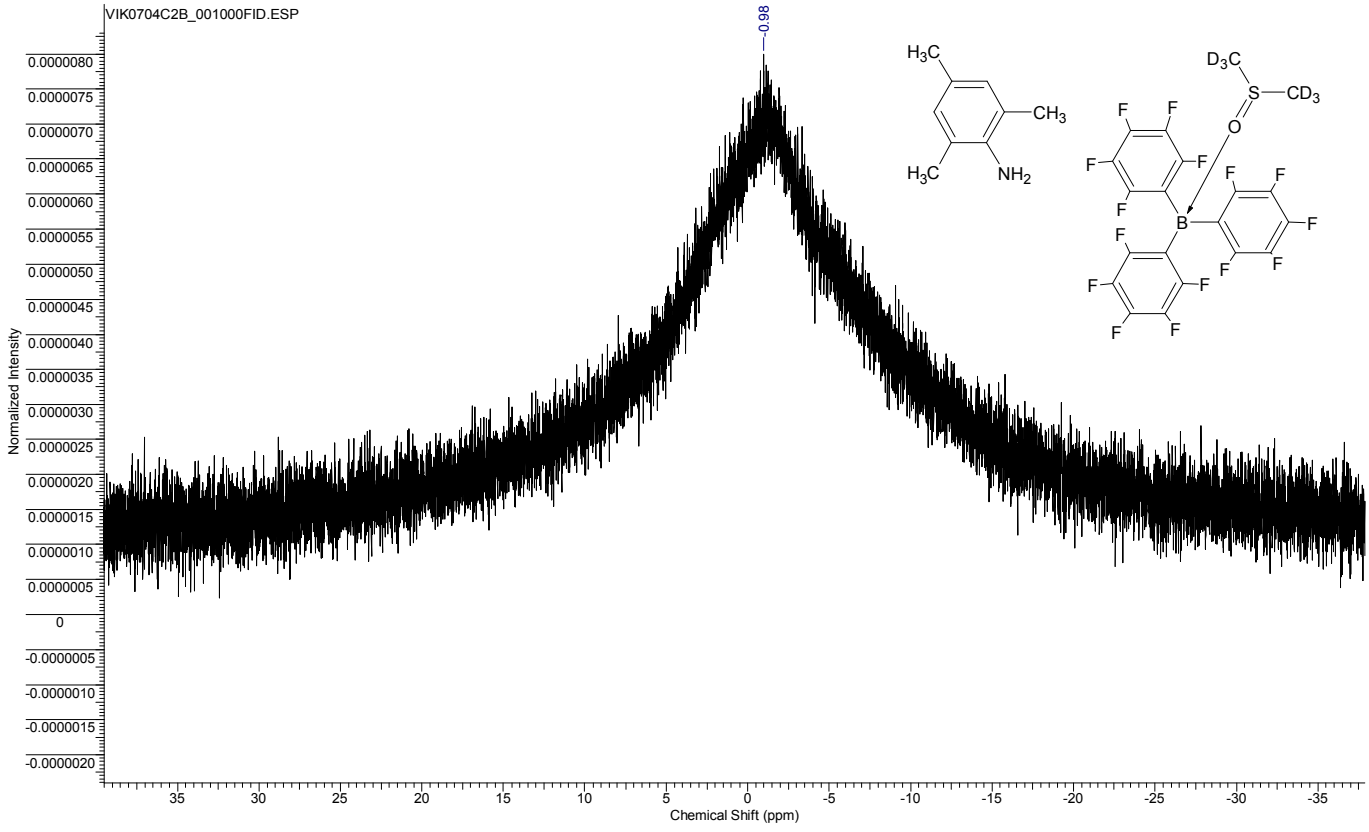

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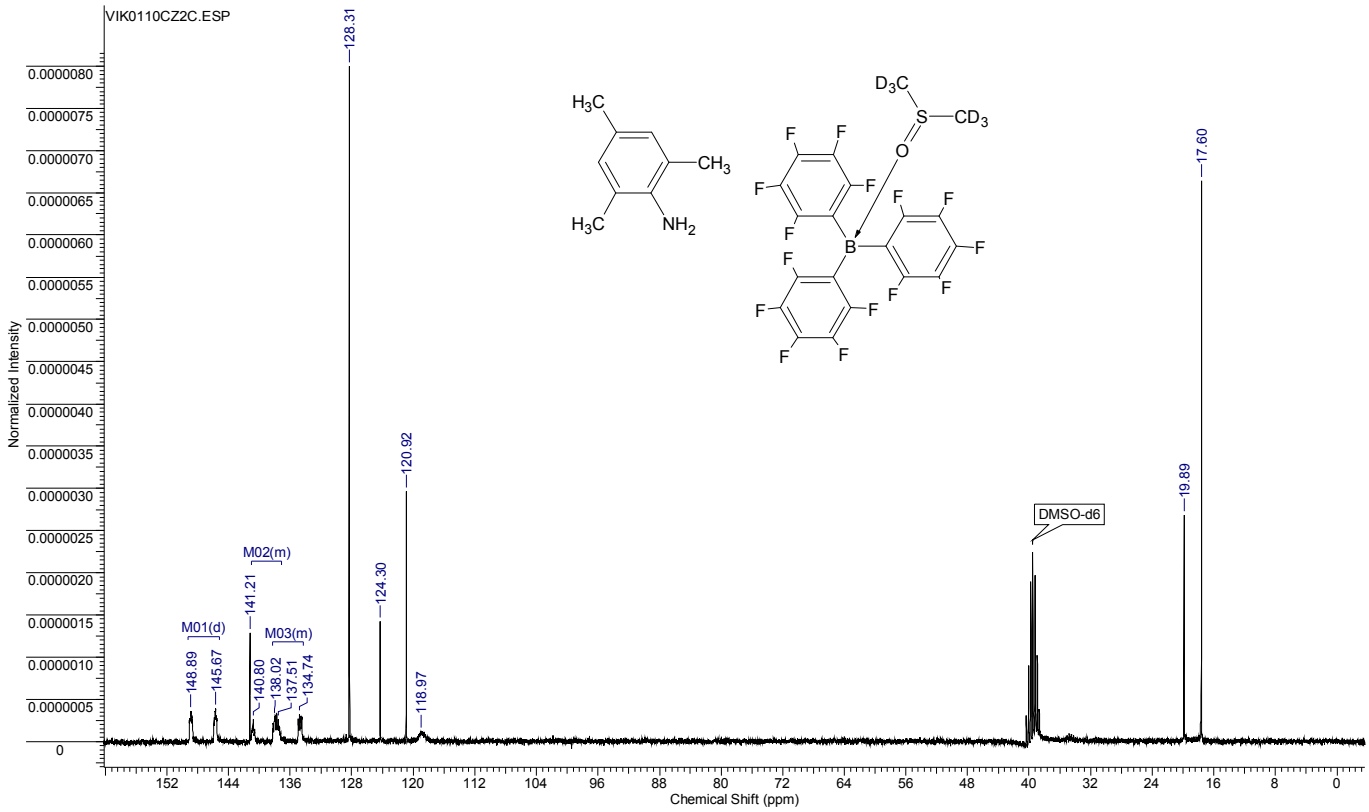

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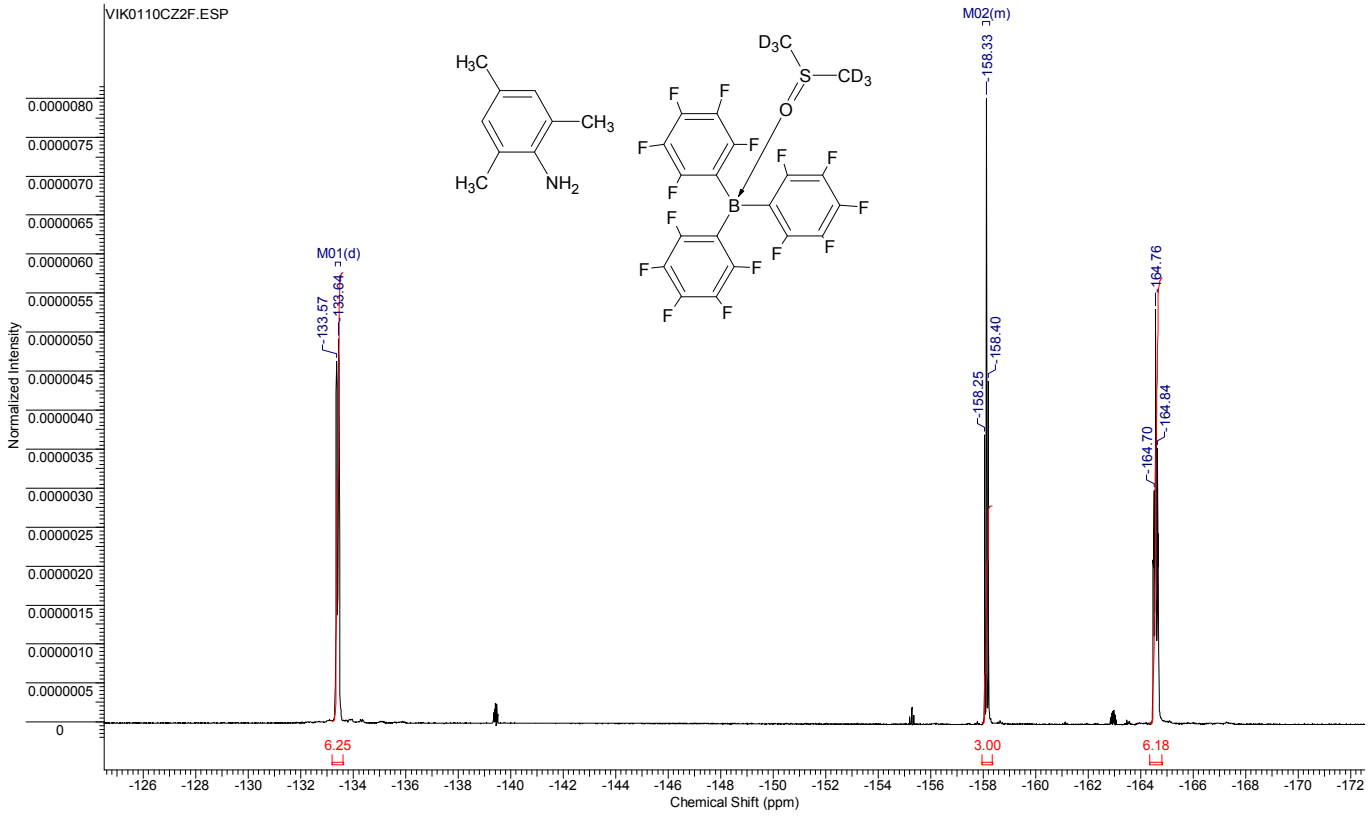

Formula $C_{22}H_{11}BF_{15}N$	FW	585.1165

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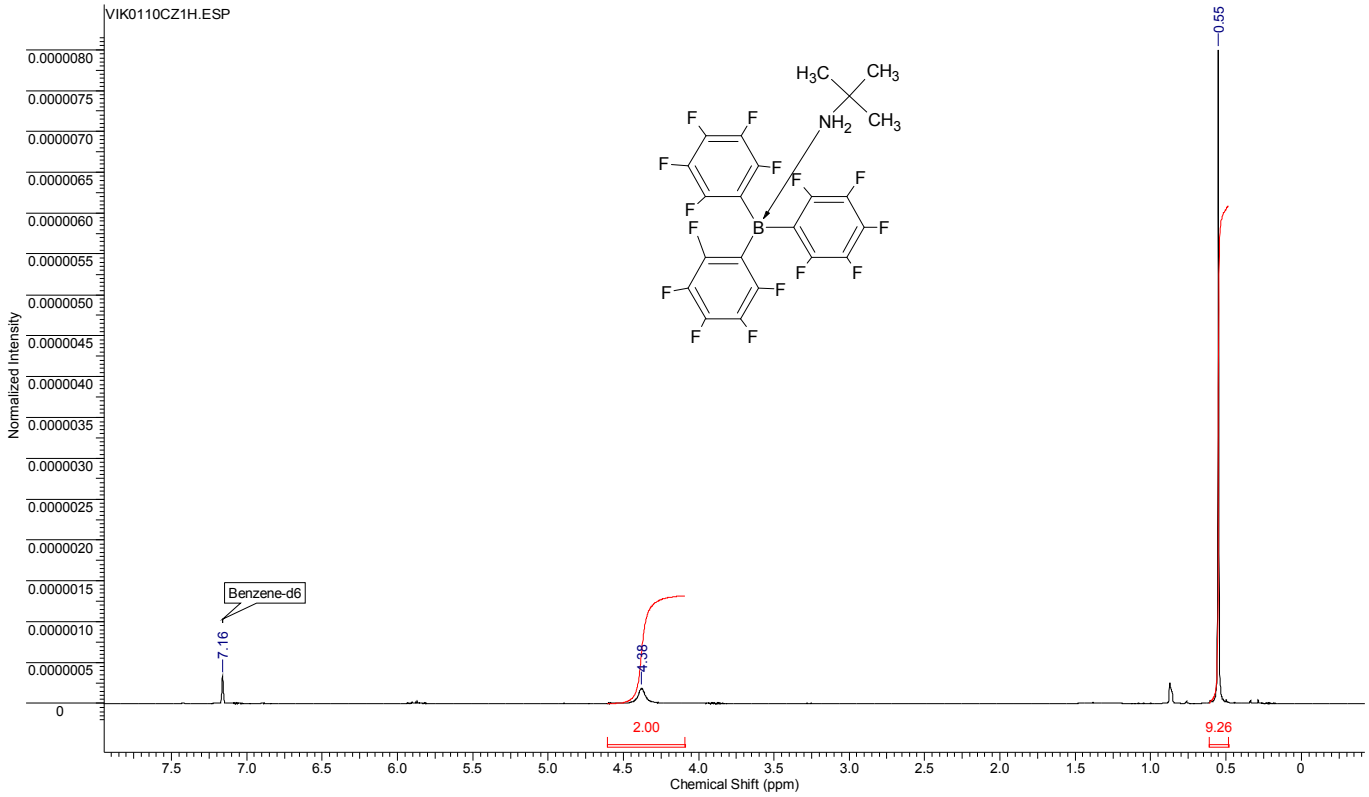

Formula $C_{22}H_{11}BF_{15}N$	FW	585.1165

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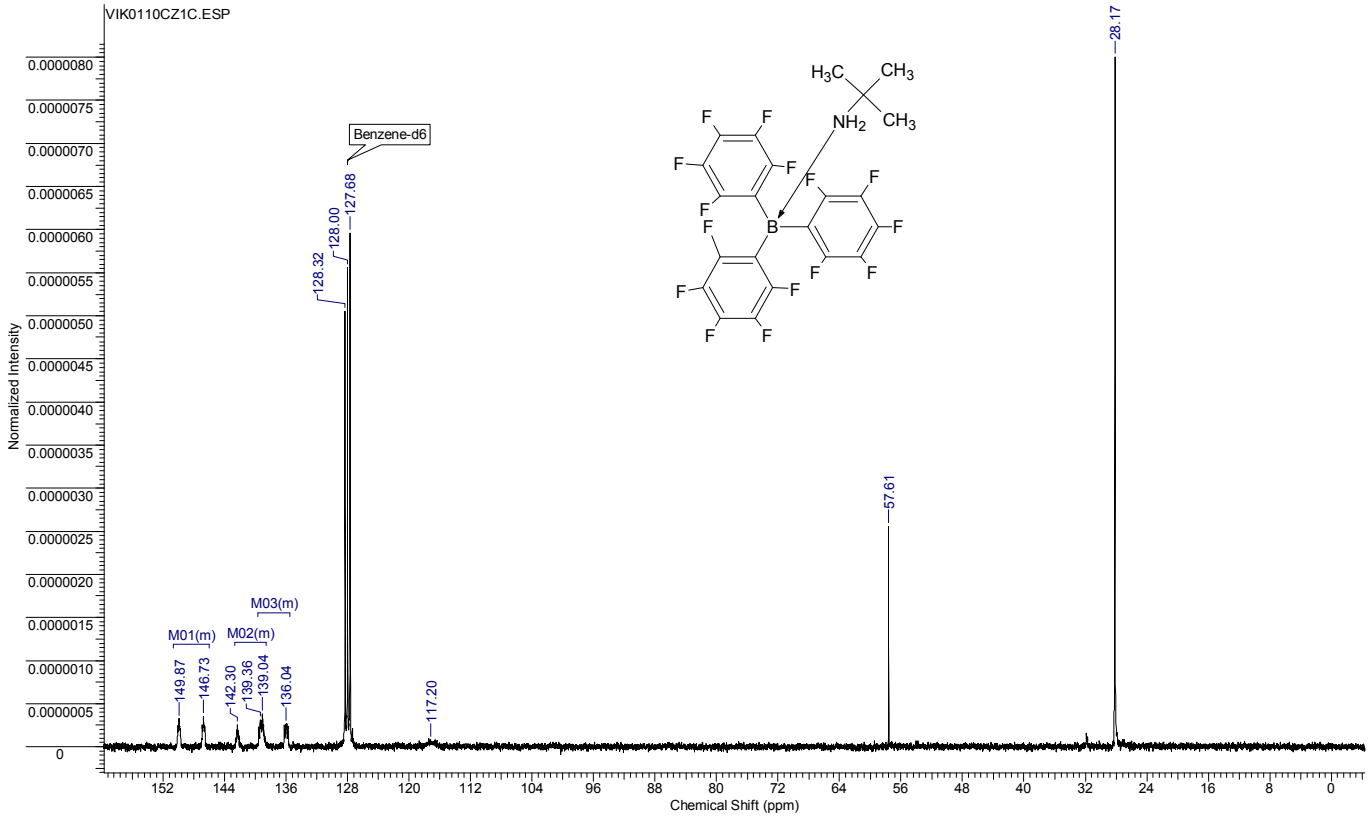

Formula	$C_{22}H_{11}BF_{15}N$	FW	585.1165

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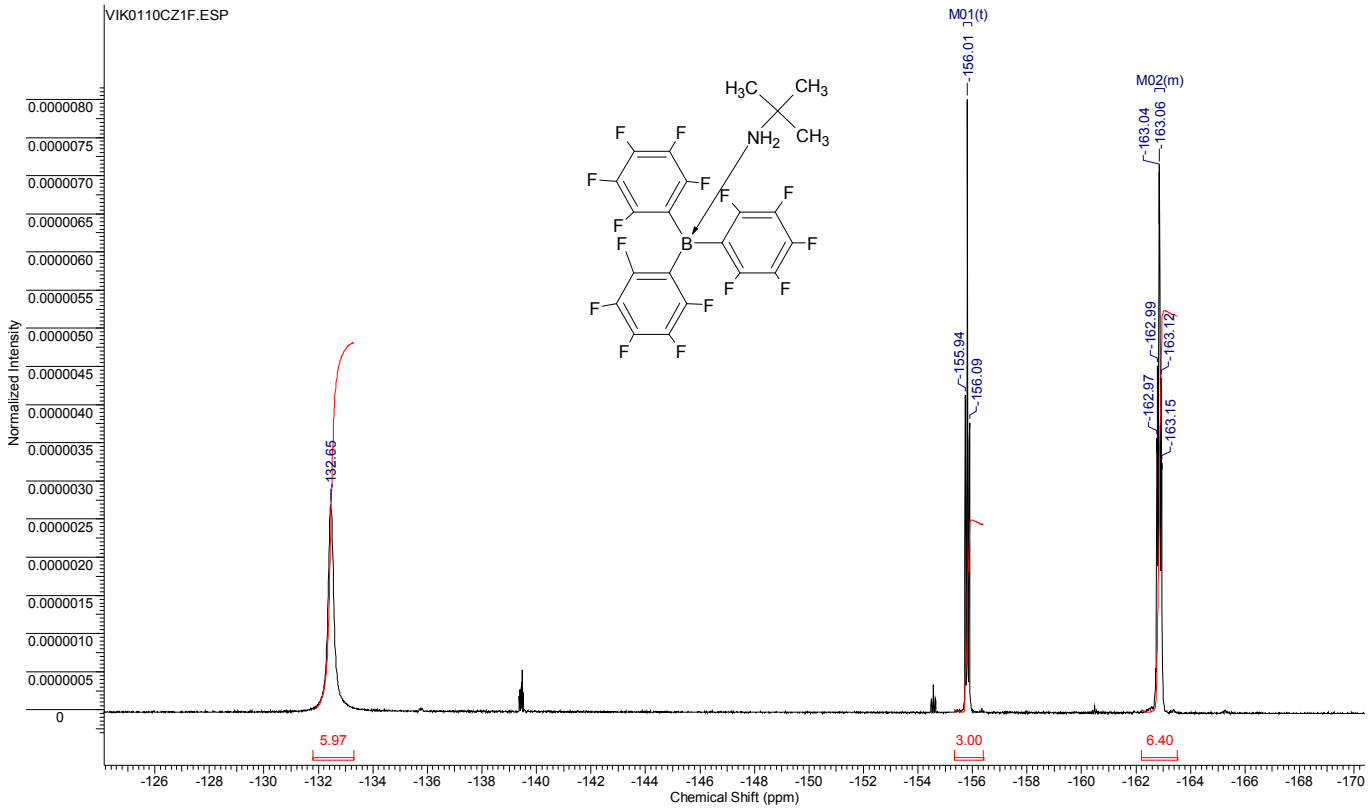

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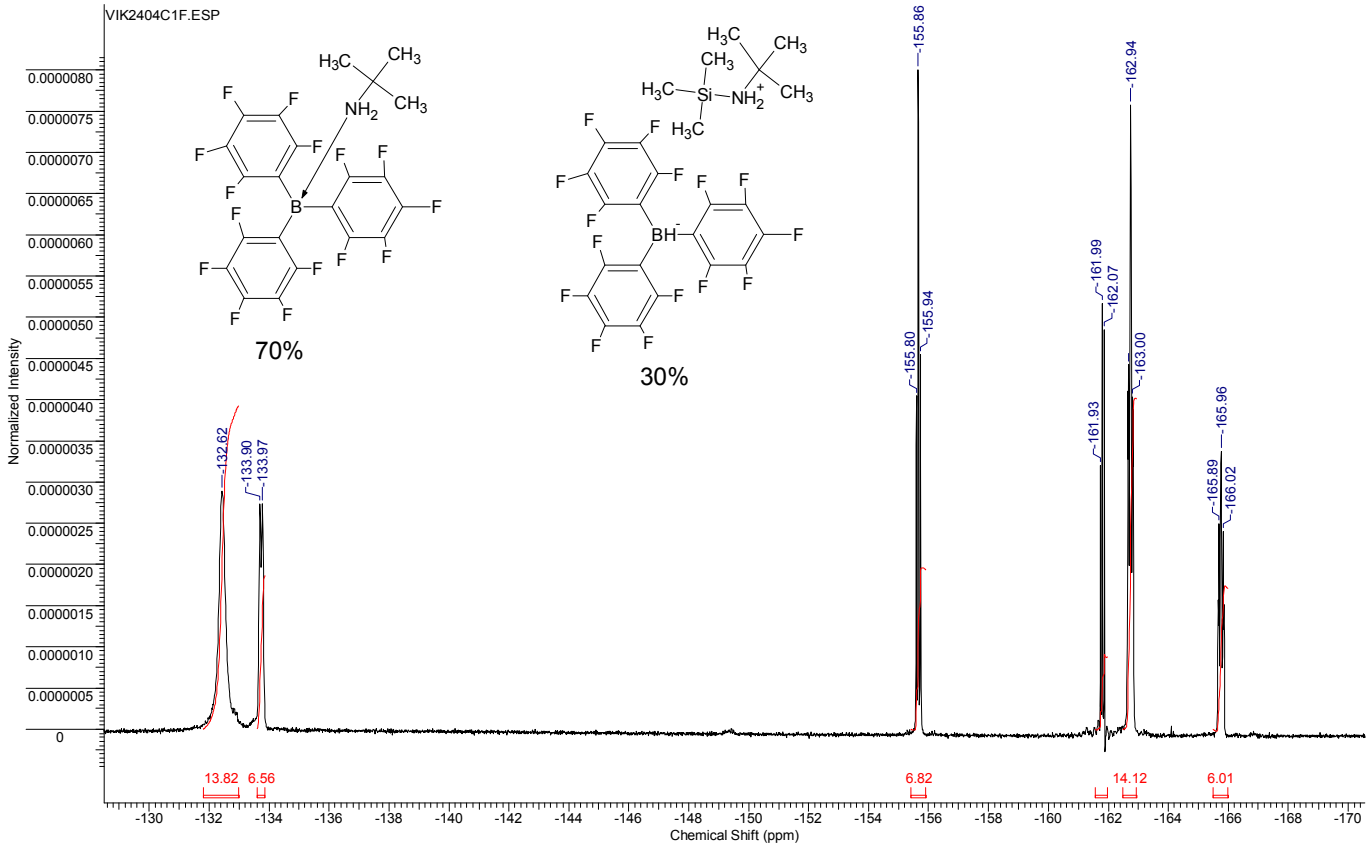

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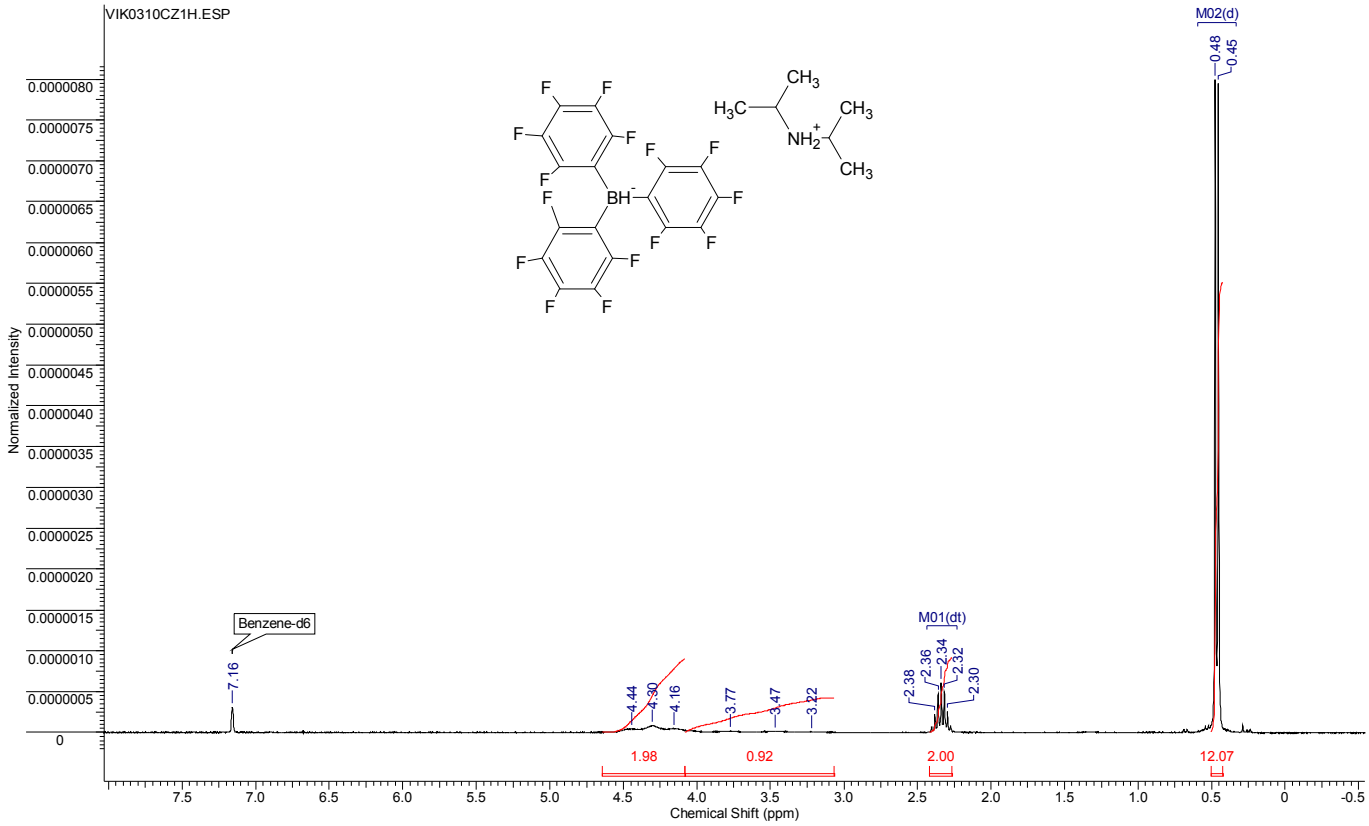
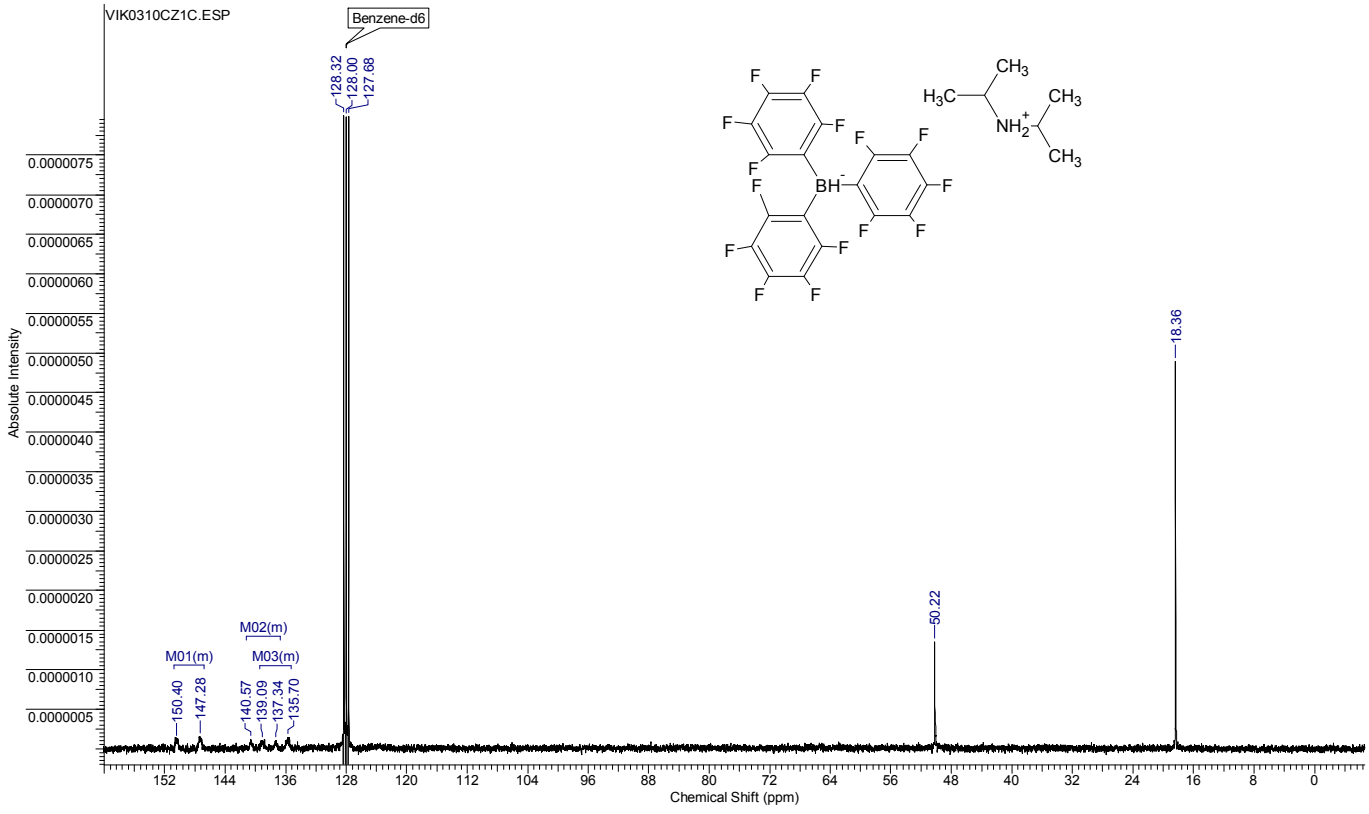
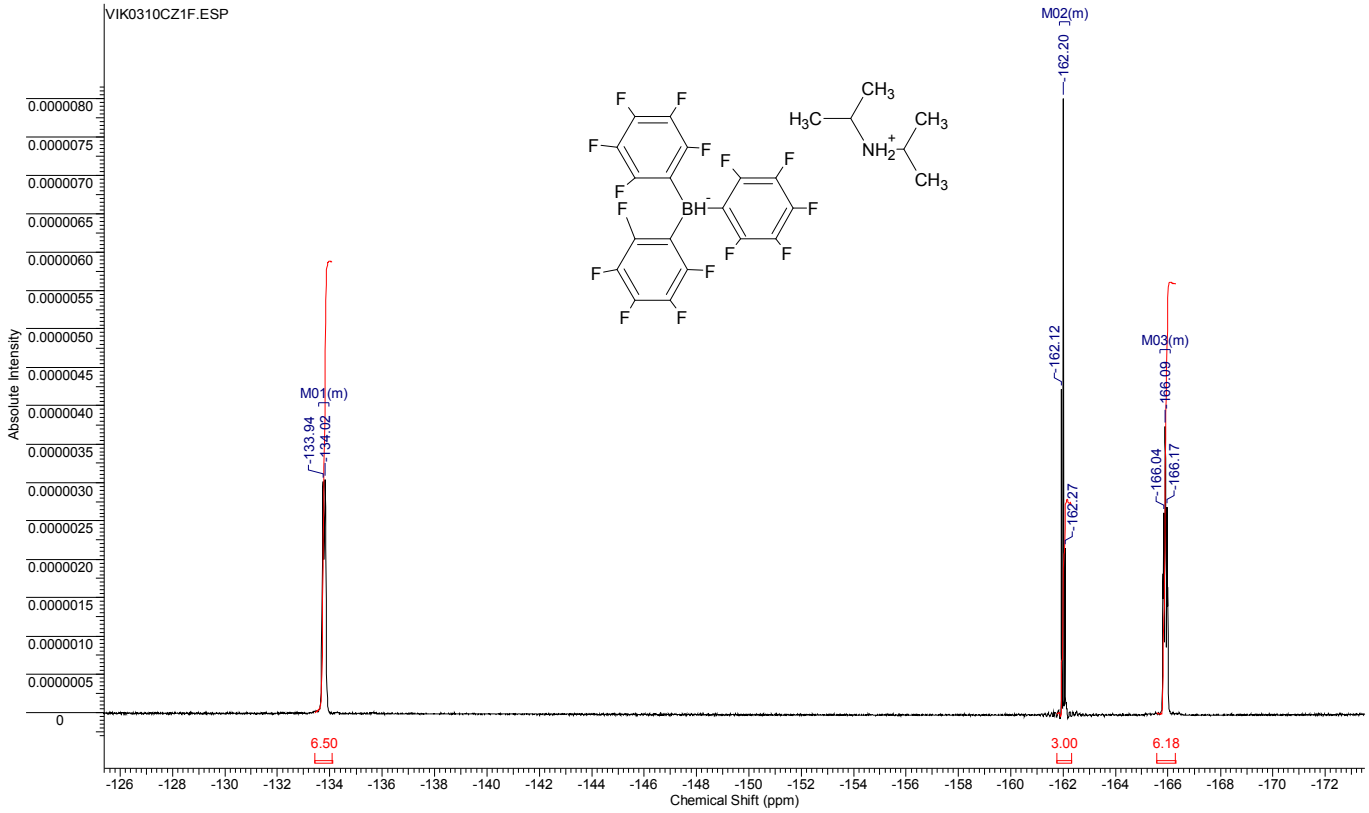
		Nucleus	1H	***Number of Transients***	4	***Original Points Count***	12500	***Points Count***	16384
Pulse Sequence	s2pul	***Solvent***	BENZENE-d6	***Spectrum Offset (Hz)***	1455.9307	***Sweep Width (Hz)***	5000.00	***Temperature (degree C)***	27.000



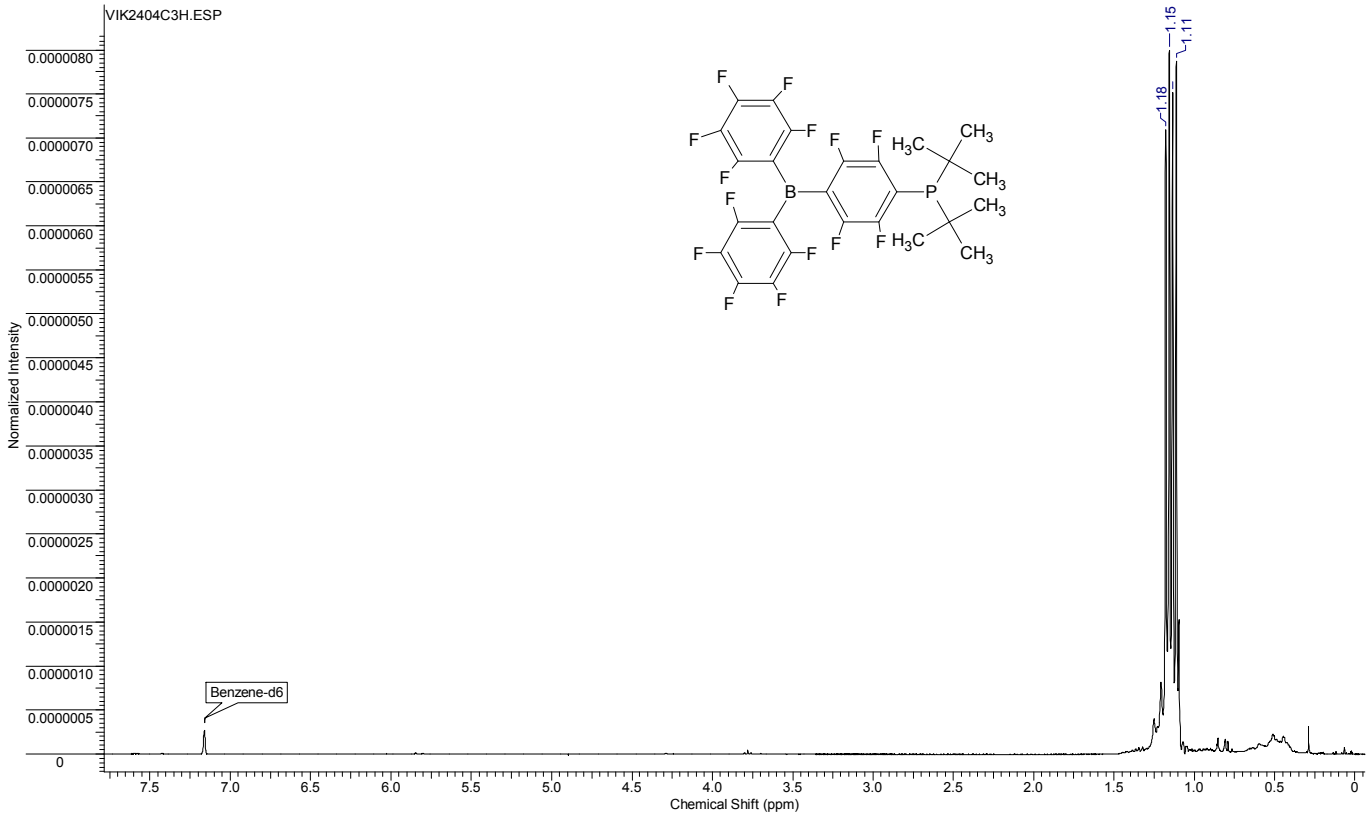
Formula $C_{24}H_{17}BF_{15}N$		***FW***	615.1855 (512.9881+102.1974)						
Acquisition Time (sec)	1.8150	***Comment***	13C OBSERVE						
								Nucleus	13C
Number of Transients	10000	***Original Points Count***	34053	***Points Count***	65536	***Pulse Sequence***	s2pul	***Solvent***	BENZENE-d6
Spectrum Offset (Hz)	7560.4385	***Sweep Width (Hz)***	18761.73	***Temperature (degree C)***	27.000				



Formula $C_{24}H_{17}BF_{15}N$		***FW***	615.1855 (512.9881+102.1974)				
Acquisition Time (sec)	0.2995	***Comment***	19F OBSERVE				
						Nucleus	19F
Number of Transients	12	***Original Points Count***	14976	***Points Count***	16384	***Pulse Sequence*** s2pul	***Solvent*** BENZENE-d6
Spectrum Offset (Hz)	-29410.2344	***Sweep Width (Hz)***	50000.00	***Temperature (degree C)***	27.000		



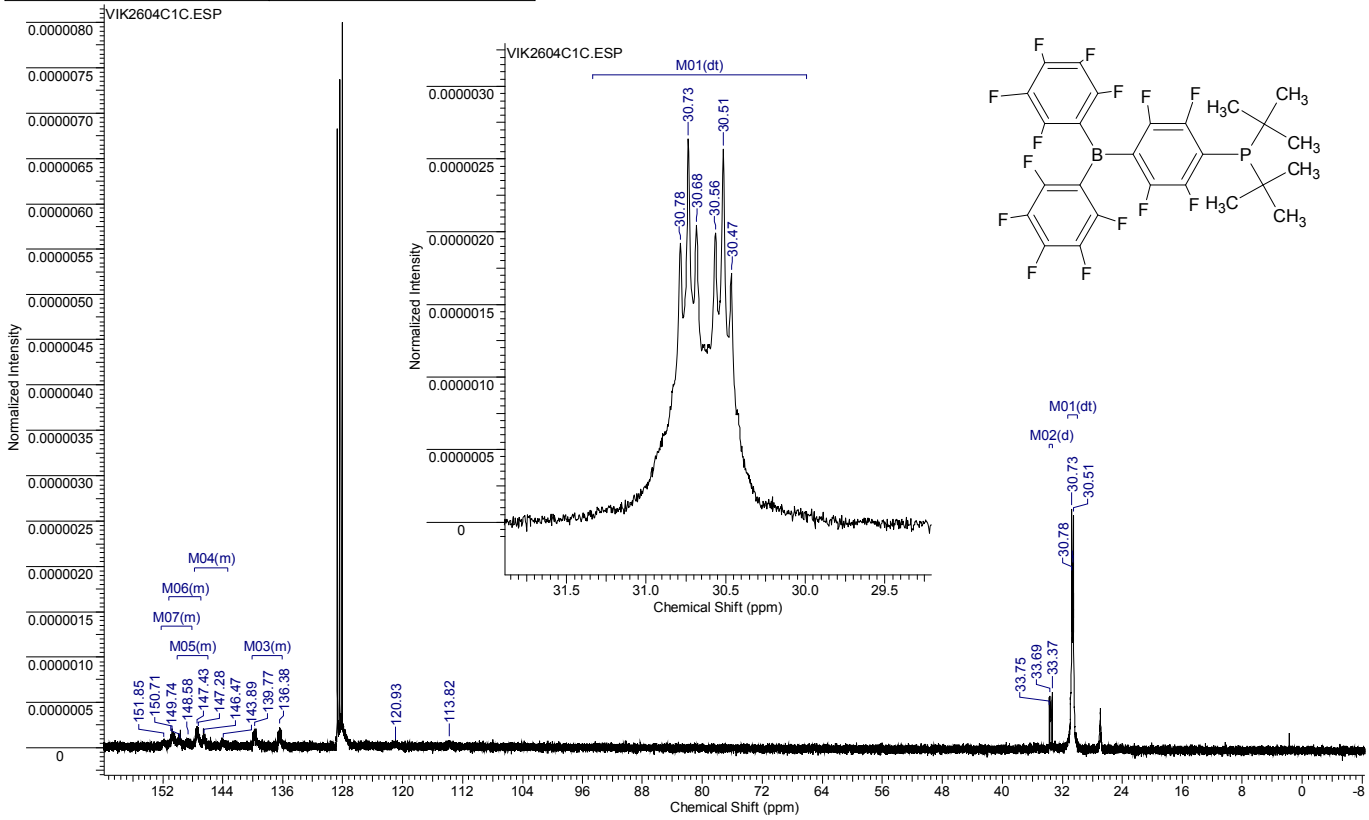
Formula $C_{26}H_{18}BF_{14}P$		***FW***	638.1835				
Acquisition Time (sec)	1.9980	***Comment***	STANDARD 1H OBSERVE			***Date***	
				Frequency (MHz)	299.95	***Nucleus*** 1H	***Number of Transients*** 12
Original Points Count	8992	***Points Count***	16384	***Pulse Sequence***	s2pul	***Solvent*** BENZENE-d6	***Spectrum Offset (Hz)*** 1467.9022
Sweep Width (Hz)	4500.45	***Temperature (degree C)***	27.000				



Formula $C_{26}H_{18}BF_{14}P$	FW	638.1835

Acquisition Time (sec)	1.8150	Comment	13C OBSERVE				
				Frequency (MHz)	75.43	Nucleus	13C
Original Points Count	34053	Points Count	65536	Pulse Sequence	s2pul	Solvent	BENZENE-d6
Sweep Width (Hz)	18761.73	Temperature (degree C)	27.000				

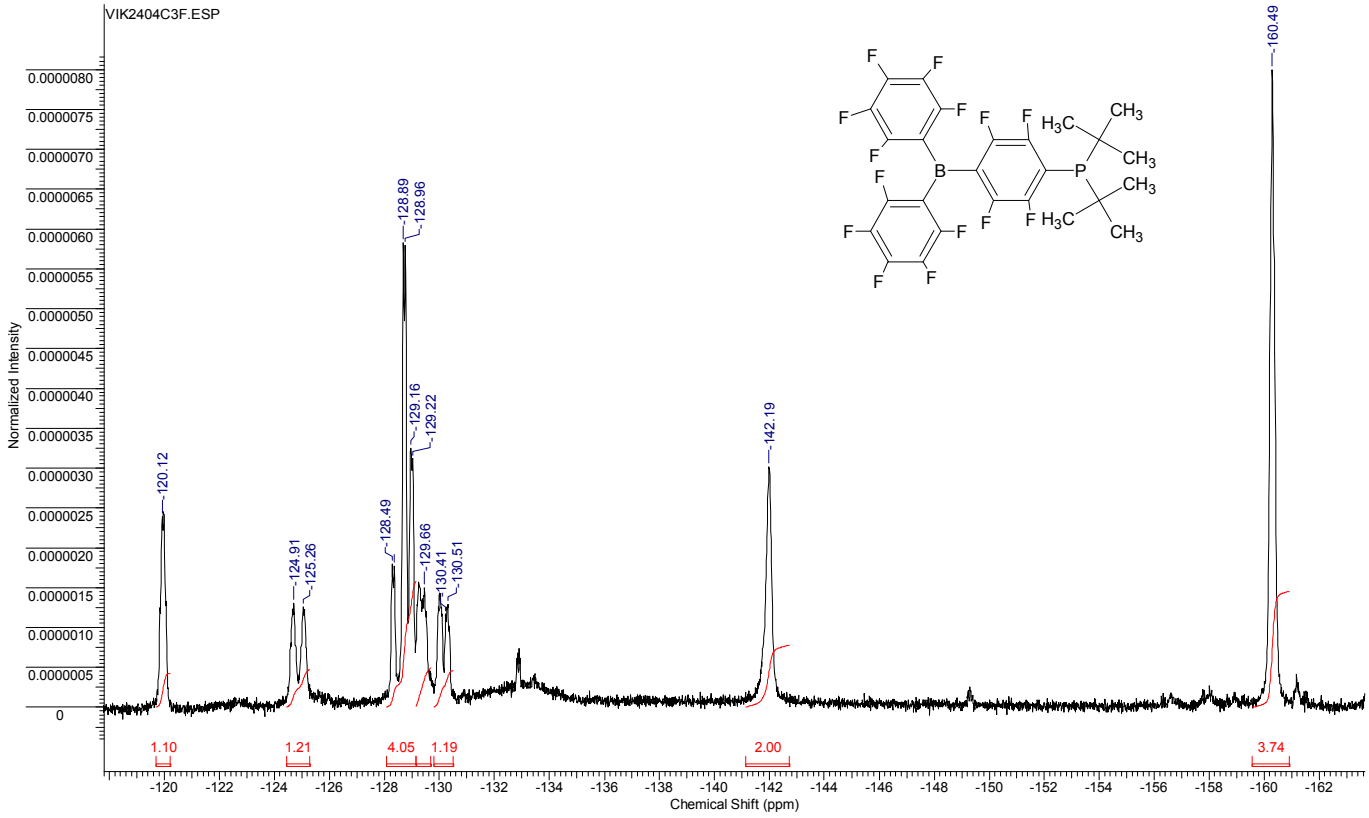
Number of Transients	30000
Spectrum Offset (Hz)	7589.2842



Formula $C_{26}H_{18}BF_{14}P$	FW	638.1835

Acquisition Time (sec)	0.2995	Comment	19F OBSERVE				
				Frequency (MHz)	282.21	Nucleus	19F
Original Points Count	14976	Points Count	16384	Pulse Sequence	s2pul	Solvent	BENZENE-d6
Sweep Width (Hz)	50000.00	Temperature (degree C)	27.000				

Number of Transients	12
Spectrum Offset (Hz)	-29410.2344



Acquisition Time (sec)	2.0000	***Comment***	P31 TRIPHENYLPHOSPHATE PARAMETERS				
				Frequency (MHz)	202.33	***Nucleus***	31P
Number of Transients	64	***Original Points Count***	200000	***Points Count***	262144	***Pulse Sequence***	s2pul
Solvent	BENZENE-d6	***Spectrum Offset (Hz)***	430.3750	***Sweep Width (Hz)***	100000.00	***Temperature (degree C)***	27.000