

Supporting Information

Palladium-Catalyzed Direct Arylation of Polyfluoroarenes with Aryl Tosylates and Mesylates

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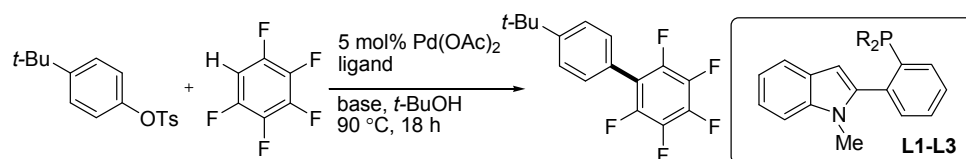
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1. General considerations

Unless otherwise noted, all reagents were purchased from commercial suppliers and used without purification. All arylation reactions were performed in Rotaflo® (England) resealable screw cap Schlenk flask (approx. 8 mL volume) in the presence of Teflon coated magnetic stirrer bar (3 mm × 10 mm). Toluene and dioxane were distilled from sodium under nitrogen. *N,N*-Dimethylformamide (DMF) was distilled from calcium hydride under reduced pressure. *tert*-Butanol was distilled from sodium under nitrogen and stored with calcium hydride.^[1] K₂CO₃ was purchased from Fluka. KOAc and Na₂CO₃ were purchased from Aldrich. CM-phos ligand was developed and prepared by our group.^[2] All aryl/heteroaryl tosylates and mesylates used were generated from the corresponding phenols according to the literature.³ Thin layer chromatography was performed on Merck precoated silica gel 60 F₂₅₄ plates. Silica gel (Merck, 70-230 and 230-400 mesh) was used for column chromatography. ¹H NMR spectra were recorded on a Bruker (400 MHz) or Varian (500 MHz) spectrometer. Spectra were referenced internally to the residual proton resonance in CDCl₃ (δ 7.26 ppm), or with tetramethylsilane (TMS, δ 0.00 ppm) as the internal standard. Chemical shifts (δ) were reported as part per million (ppm) in δ scale downfield from TMS. ¹³C NMR spectra were referenced to CDCl₃ (δ 77.0 ppm, the middle peak). Coupling constants (*J*) were reported in Hertz (Hz). Mass spectra (EI-MS and ES-MS) were recorded on a HP 5989B Mass Spectrometer. High-resolution mass spectra (HRMS) were obtained on a Bruker APEX 47e FT-ICR mass spectrometer (ESIMS). GC-MS analysis was conducted on a HP 5973 GCD system using a HP5MS column (30 m × 0.25 mm). The products described in GC yield were accorded to the authentic samples/dodecane calibration standard from HP 6890 GC-FID system.

2. General procedures for initial ligand and reaction conditions screening

General Procedure for reaction condition screenings: Pd(OAc)₂ (3.4 mg, 0.015 mmol) and ligand (Pd:L = 1:4) were loaded into a Schlenk tube equipped with a Teflon-coated magnetic stir bar. The tubes were evacuated and backfilled with nitrogen (3 cycles). Precomplexation was applied by adding freshly distilled dichloromethane (1.0 mL) and Et₃N (0.05 mL) into the tube. The palladium complex stock solution was stirred and warmed using a hair drier for 1 to 2 minutes until the solvent started boiling. The solvent was then evaporated under high vacuum. 4-*tert*-Butylphenyl tosylate (0.3 mmol) and base (0.45 mmol) were loaded into the tube, and the system was further evacuated and flushed with nitrogen for three times. The solvent (1.0 mL) was then added with stirring at room temperature for several minutes and pentafluorobenzene (0.6 mmol) was then loaded into the tube. The tube was then placed into a preheated oil bath (90 °C) and stirred for 18 hours. After completion of reaction, the reaction tube was allowed to cool to room temperature and quenched with water and diluted with ethyl acetate. Dodecane (68 μL, internal standard) was then added. The organic layer was subjected to GC analysis. The GC yield obtained was previously calibrated by authentic sample/dodecane calibration curve.



| Entry ^[a] | Pd Source | Ligand | Base | Additives | Yield ^[b] |
|----------------------|----------------------|-------------------|---------------------------------|----------------------|----------------------|
| 1 | Pd(OAc) ₂ | L1 (R=Cy) | K ₂ CO ₃ | -- | 76 |
| 2 ^[c] | Pd(OAc) ₂ | L1 | K ₂ CO ₃ | -- | 72 |
| 3 ^[d] | Pd(OAc) ₂ | L1 | K ₂ CO ₃ | -- | 43 |
| 4 | Pd(OAc) ₂ | L2 (R=Ph) | K ₂ CO ₃ | -- | 0 |
| 5 | Pd(OAc) ₂ | L3 (R=iPr) | K ₂ CO ₃ | -- | 33 |
| 6 | Pd(TFA) ₂ | L1 | K ₂ CO ₃ | -- | 26 |
| 7 | PdCl ₂ | L1 | K ₂ CO ₃ | -- | 10 |
| 8 | Pd(OAc) ₂ | L1 | K ₂ CO ₃ | CF ₃ COOH | 70 |
| 9 | Pd(OAc) ₂ | L1 | K ₂ CO ₃ | AcOH | 82 |
| 10 | Pd(OAc) ₂ | L1 | K ₂ CO ₃ | PivOH | 87 |
| 11 | Pd(OAc) ₂ | L1 | K ₂ CO ₃ | PhCOOH | 80 |
| 12 | Pd(OAc) ₂ | L1 | Na ₂ CO ₃ | -- | 90 |
| 13 | Pd(OAc) ₂ | L1 | Na ₂ CO ₃ | PivOH | 84 |
| 14 | Pd(OAc) ₂ | L1 | KOAc | -- | 90 |
| 15 | Pd(OAc) ₂ | L1 | KOAc | PivOH | 92 |
| 16 | Pd(OAc) ₂ | L1 | NaOAc | -- | 73 |

[a]Reaction conditions: C₁₀H₁₃OTs (0.3 mmol), C₆HF₅ (0.6 mmol), base (0.45 mmol), Pd/L = 1:4, solvent (1.0 mL), additives (10 mol%) were stirred for 18 h at 90 °C under nitrogen. [b]Calibrated GC yields were reported using dodecane as the internal standard. [c]Reaction was conducted at 110 °C. [d]2.0 mol% of Pd(OAc)₂ was used.

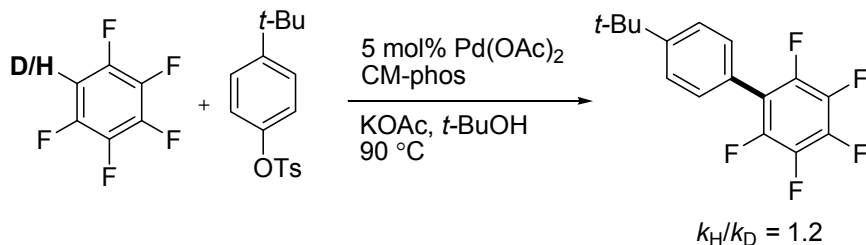
3. General procedures for direct arylation of polyfluoroarenes with aryl sulfonates

General procedures for direct arylation of polyfluoroarenes with aryl sulfonates: Pd(OAc)₂ (3.4 mg, 0.015 mmol) and ligand (Pd:L = 1:4) were loaded into a Schlenk tube equipped with a Teflon-coated magnetic stir bar. The tubes were evacuated and backfilled with nitrogen (3 cycles). Precomplexation was applied by adding freshly distilled dichloromethane (1.0 mL) and Et₃N (0.05 mL) into the tube. The palladium complex stock solution was stirred and warmed using a hair drier for 1 to 2 minutes until the solvent started boiling. The solvent was then evaporated under high vacuum. Aryl tosylates/ mesylates (0.3 mmol) and base (0.45 mmol) were loaded into the tube, and the system was further evacuated and flushed with nitrogen for three times. The solvent (1.0 mL) was then added with stirring at room temperature for several minutes and polyfluoroarenes (0.6 mmol) was then loaded into the tube. The tube was then placed into a preheated oil bath (90 °C) and stirred for the time as indicated. After completion of reaction, the reaction tube was allowed to cool to room temperature and quenched with water and diluted with ethyl acetate (EtOAc). The organic layer was separated and the aqueous layer was washed with EtOAc. The filtrate was concentrated under reduced pressure. The crude products were purified by flash column chromatography on silica gel (230-400 mesh) to afford the desired product.

4. General procedures for sequential arylation of aryl tosylates

General procedures for one-pot stepwise sequential arylation of aryl tosylates: Pd(OAc)₂ (3.4 mg, 0.015 mmol) and ligand (Pd:L = 1:4) were loaded into a Schlenk tube equipped with a Teflon-coated magnetic stir bar. The tubes were evacuated and backfilled with nitrogen (3 cycles). Precomplexation was applied by adding freshly distilled dichloromethane (1.0 mL) and Et₃N (0.05 mL) into the tube. The palladium complex stock solution was stirred and warmed using a hair drier for 1 to 2 minutes until the solvent started boiling. The solvent was then evaporated under high vacuum. 3-Aminophenyl tosylate (0.3 mmol) and KOAc (0.45 mmol) were loaded into the tube, and the system was further evacuated and flushed with nitrogen for three times. The solvent *tert*-butanol (1.0 mL) was then added with stirring at room temperature for several minutes and pentafluorobenzene (0.6 mmol) was then loaded into the tube. The tube was then placed into a preheated oil bath (90 °C) and stirred for 18 hours. After completion of reaction, the reaction tube was allowed to cool to room temperature. The system was flushed with nitrogen while 4-cyanophenyl tosylate (0.158 mmol) and K₂CO₃ (0.395 mmol) were loaded into the tube. Another 1.0 mL solvent of *tert*-butanol was then added to the system. The tube was then placed into a preheated oil bath (110 °C) and stirred for another 24 hours. After the completion of reaction, the tube was allowed to cool to room temperature. The reaction was then quenched with water and diluted with EtOAc and judged as GC. The organic layer was separated and the aqueous layer was washed with EtOAc. The filtrate was concentrated under reduced pressure. The crude products were purified by flash column chromatography on silica gel (230-400 mesh) to afford the desired product.

5. Kinetic Isotope Experiment

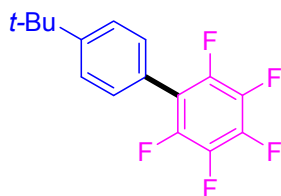


Deuteropentafluorobenzene used was synthesized from bromopentafluorobenzene. The bromopentafluorobenzene was refluxed with Mg in anhydrous diethyl ether for 1 h and was quenched by deuterium oxide according to the literature.^{4, 5}

Pd(OAc)₂ (3.4 mg, 0.015 mmol) and ligand (Pd:L = 1:4) were loaded into a Schlenk tube equipped with a Teflon-coated magnetic stir bar. The tubes were evacuated and backfilled with nitrogen (3 cycles). Precomplexation was applied by adding freshly distilled dichloromethane (1.0 mL) and Et₃N (0.05 mL) into the tube. The palladium complex stock solution was stirred and warmed using a hair drier for 1 to 2 minutes until the solvent started boiling. The solvent was then evaporated under high vacuum. 4-*tert*-Butylphenyl tosylate (0.3 mmol) and KOAc (0.45 mmol) were loaded into the tube, and the system was further evacuated and flushed with nitrogen for three times. *tert*-Butanol (1.0 mL) was then added with stirring at room temperature for several minutes and pentafluorobenzene or deuteropentafluorobenzene (0.6 mmol) was then loaded into the tube. The tube was then placed into a preheated oil bath (90 °C) and stirred for assigned time intervals. The reaction tube was allowed to cool to room temperature and quenched with water and diluted with ethyl acetate (EtOAc). Dodecane (68 μL, internal standard) was then added. The organic layer was subjected to GC analysis. The GC yield obtained was previously calibrated by authentic sample/dodecane calibration curve.

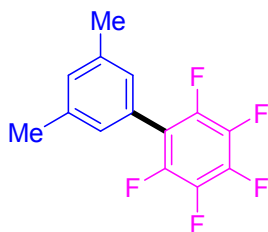
6. Characterization data of coupling products

2,3,4,5,6-Pentafluoro-4'-*tert*-butylbiphenyl (Compound 3aa)⁶



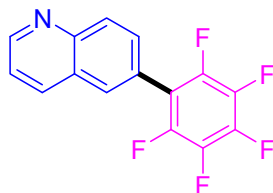
Pure Hexane, $R_f = 0.4$; m.p. 81-83.2 °C; ^1H NMR (400 MHz, CDCl_3) δ 1.41 (s, 9H), 7.39-7.42 (m, 2H), 7.53-7.57 (m, 2H); ^{13}C NMR (100MHz, CDCl_3) δ 31.2, 34.7, 115.9 (m), 123.4, 125.7, 129.8, 136.6 (m), 139.1 (m), 141.4 (m), 143.12 (m) 145.4 (m), 152.4; ^{19}F NMR (400 MHz, CDCl_3) δ -162.5 (m, 2F), -156.2 (t, 1F), -143.3 (dd, 2F).

2,3,4,5,6-Pentafluoro-3',5'-dimethyl-1,1'-biphenyl (Compound 3ab)¹¹



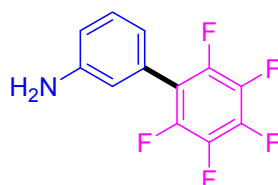
Pure Hexane, $R_f = 0.7$; m. p. 83-84.1 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.42 (s, 3H), 7.06 (s, 2H), 7.14 (s, 1H); ^{13}C NMR (100MHz, CDCl_3) δ 21.2, 116.3 (m), 126.1, 127.8, 130.9, 136.5 (m), 138.3, 139.0 (m), 141.4 (m), 142.9 (m), 145.4 (m); ^{19}F NMR (400 MHz, CDCl_3) δ -162.6 (m, 2F), -156.2 (t, 1F), -143.0 (dd, 2F).

6'-(2,3,4,5,6-Pentafluorophenyl)quinoline (Compound 3ac)



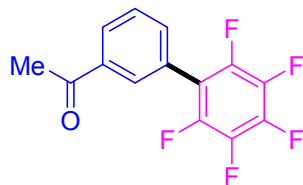
Hexane: EA = 4:1, R_f = 0.45; m. p. 172-173.6 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.48 (q, $J=4.4\text{Hz}$, 1H), 7.75 (dd, $J=7.2$, 1.6Hz, 1H), 7.93 (s, 1H), 8.21-8.25 (m, 2H), 9.01 (d, $J=2.8\text{Hz}$, 1H); ^{13}C NMR (100MHz, CDCl_3) δ 121.8, 124.6, 127.9, 129.9, 130.0, 130.6, 136.3, 147.9, 151.6, 192.2; ^{19}F NMR (400 MHz, CDCl_3) δ -161.7 (m, 2F), -154.5 (t, 1F), -142.9 (dd, 2F); HRMS: calcd. for $\text{C}_{15}\text{H}_7\text{NF}_5^+$: 296.0499, found 296.0495.

2,3,4,5,6-Pentafluoro-3'-aminobiphenyl (Compound 3ad)⁷



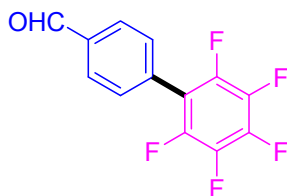
Hexane: EA = 10:1, R_f = 0.2; m. p. 100-101.2 °C; ^1H NMR (400 MHz, CDCl_3) δ 3.81 (s, 2H), 6.73 (s, 1H), 6.78-6.82 (m, 2H), 7.29 (dd, $J=5.2$, 2.8Hz, 1H); ^{13}C NMR (100MHz, CDCl_3) δ 115.9, 116.2, 116.5, 120.3, 127.2, 129.6, 136.4 (m), 139.0 (m), 141.5 (m), 142.9 (m), 145.3 (m), 146.6; ^{19}F NMR (400 MHz, CDCl_3) δ -162.5 (m, 2F), -156.0 (t, 1F), -142.7 (dd, 2F).

3'-(2,3,4,5,6-Pentafluorophenyl)acetophenone (Compound 3ae)



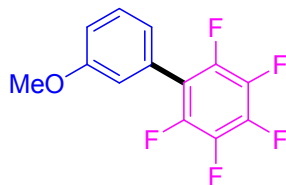
Hexane: EA = 9:1, R_f = 0.4; m. p. 106-108 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.65 (s, 3H), 7.62 (d, J =3.6Hz, 2H), 8.03-8.07 (m, 2H); ^{13}C NMR (100MHz, CDCl_3) δ 26.5, 114.9 (m), 126.9, 129.0, 130.0, 134.4, 136.6 (m), 137.5, 139.1 (m), 142.8 (m), 145.3 (m), 197.0; ^{19}F NMR (400 MHz, CDCl_3) δ -161.8 (m, 2F), -154.5 (t, 1F), -143.1 (dd, 2F); HRMS: calcd. for $\text{C}_{14}\text{H}_8\text{OF}_5^+$: 287.0495, found 287.0485.

2',3',4',5',6'-Pentafluoro-[1,1'-biphenyl]-4-carboxaldehyde (Compound 3af)⁸



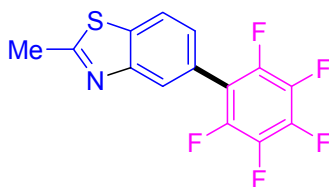
Hexane: EA = 9:1, R_f = 0.4; m. p. 75.8-78.2 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.63 (q, J =6.8, 1.2Hz, 2H), 8.01-8.04 (m, 2H), 10.1 (s, 1H); ^{13}C NMR (100MHz, CDCl_3) δ 129.7, 130.9, 132.3, 134.3 (m), 136.8 (d), 139.5 (d), 141.4 (m), 142.8 (m), 145.3 (m), 191.4; ^{19}F NMR (400 MHz, CDCl_3) δ -161.4 (m, 2F), -153.6 (t, 1F), -142.7 (dd, 2F).

2,3,4,5,6-Pentafluoro-3'-methoxybiphenyl (Compound 3ag)¹¹



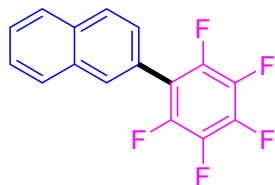
Hexane: EA = 20:1, $R_f = 0.45$; m. p. 33.8-34.7 °C; ^1H NMR (400 MHz, CDCl_3) δ 3.87 (s, 3H), 6.97 (s, 1H), 7.02-7.05 (m, 2H), 7.43 (t, $J=7.6\text{Hz}$, 1H); ^{13}C NMR (100MHz, CDCl_3) δ 55.3, 114.8, 115.8, 122.4, 127.4, 129.7, 134.1 (m), 136.5 (m), 139.0 (m), 141.6 (m), 142.8 (m), 145.5 (m), 159.6; ^{19}F NMR (400 MHz, CDCl_3) δ -162.3 (m, 2F), -155.6 (t, 1F), -142.8 (dd, 2F).

2-Methyl-5-(pentafluorophenyl)benzo[d]thiazole (Compound 3ah)



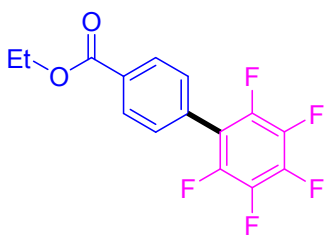
Hexane: EA = 20:1, $R_f = 0.25$; m. p. 152.6-154.7 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.88 (s, 3H), 7.39 (dd, $J=6.8, 1.6$, 1H), 7.94 (d, $J=8.4\text{Hz}$, 1H), 8.03 (s, 1H); ^{13}C NMR (100MHz, CDCl_3) δ 20.1, 121.7, 124.0, 124.1, 126.2, 136.8, 153.5, 168.3; ^{19}F NMR (400 MHz, CDCl_3) δ -161.9 (m, 2F), -155.1 (t, 1F), -143.0 (dd, 2F); HRMS: calcd. for $\text{C}_{14}\text{H}_7\text{NSF}_5^+$: 316.0219, found 316.0207.

2-(2,3,4,5,6-pentafluorophenyl)naphthalene (Compound 3ai)⁹



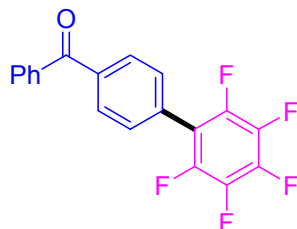
Hexane: EA = 9:1, R_f = 0.7; m. p. 168-169.8 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.51-7.62 (m, 3H), 7.91-7.99 (m, 4H); ^{13}C NMR (100MHz, CDCl_3) δ 123.7, 126.6, 127.0, 127.1, 127.7, 128.2, 128.4, 130.1, 133.0, 133.2; ^{19}F NMR (400 MHz, CDCl_3) δ -162.1 (m, 2F), -155.4 (t, 1F), -142.9 (dd, 2F).

[1,1'-Biphenyl]-4-carboxylic acid, 2',3',4',5',6'-pentafluoro-, ethyl ester (Compound 3aj)



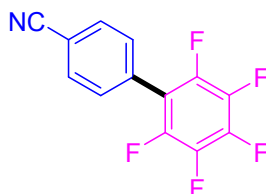
Hexane: EA = 20:1, R_f = 0.25; m. p. 147-149.1 °C; ^1H NMR (400 MHz, CDCl_3) δ 1.43 (t, $J=7.2\text{Hz}$, 3H), 4.43 (q, $J=7.2\text{Hz}$, 2H), 7.51-7.54 (m, 2H), 8.16-8.19 (m, 2H); ^{13}C NMR (100MHz, CDCl_3) δ 14.2, 61.2, 115.0, 129.8, 130.1, 130.7, 131.2, 136.6 (m), 139.2 (m), 139.6 (m), 142.0 (m), 142.8 (m), 145.3 (m), 165.8; ^{19}F NMR (400 MHz, CDCl_3) δ -161.7 (m, 2F), -154.3 (t, 1F), -142.8 (dd, 2F); HRMS: calcd. for $\text{C}_{15}\text{H}_9\text{O}_2\text{F}_5^+$: 316.0523, found 316.0533.

(2',3',4',5',6'-Pentafluoro[1,1'-biphenyl]-4-yl)phenylmethanone (Compound 3ak)¹⁰



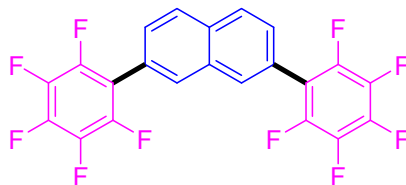
Hexane: EA = 9:1, $R_f = 0.5$; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.51-7.66 (m, 5H), 7.85-7.93 (m, 4H); $^{13}\text{C NMR}$ (100MHz, CDCl_3) δ 128.2, 128.3, 130.0, 130.1, 130.3, 132.3, 132.7, 136.6 (m), 137.1, 138.1, 138.1 (m), 138.9 (m), 139.6 (m), 142.8, 145.4 (m), 195.8; $^{19}\text{F NMR}$ (400 MHz, CDCl_3) δ -161.5 (m, 2F), -154.1 (t, 1F), -142.7 (dd, 2F).

2,3,4,5,6-Pentafluoro-4'-cyanobiphenyl (Compound 3al)¹¹



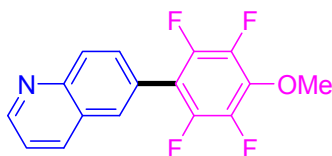
Hexane: EA = 10:1, $R_f = 0.4$; m. p. 126-128.8 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.59 (d, $J=8.4\text{Hz}$, 2H), 7.80-7.83 (m, 2H); $^{13}\text{C NMR}$ (100MHz, CDCl_3) δ 113.3, 118.0, 130.9, 131.1, 132.4, 136.7 (m), 139.1 (m), 142.7 (m), 145.2 (m); $^{19}\text{F NMR}$ (400 MHz, CDCl_3) δ -161.0 (m, 2F), -152.9 (t, 1F), -142.8 (dd, 2F).

2,7-Bis(pentafluorophenyl)naphthalene (Compound 3am)



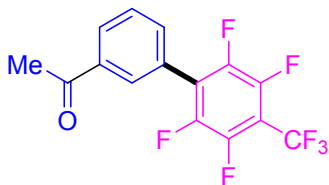
Hexane: EA = 9:1, R_f = 0.8; m. p. 140.7-143.8 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.61 (dd, J =1.2, 7.6Hz, 2H), 8.03 (t, J =8.8, 10Hz, 3H); ^{13}C NMR (100MHz, CDCl_3) δ 124.7, 128.3, 130.4, 132.7, 133.0; ^{19}F NMR (400 MHz, CDCl_3) δ -161.8 (m, 4F), -154.8 (t, 2F), -142.9 (dd, 4F); HRMS: calcd. for $\text{C}_{22}\text{H}_6\text{F}_{10}^+$: 460.0310, found 460.0313.

6'-(2,3,5,6-Tetrafluoro-4-methoxyphenyl)quinoline (Compound 3bc)



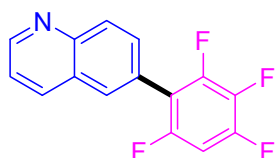
Hexane: EA = 9:1, R_f = 0.15; m. p. 162.8-165.1 °C; ^1H NMR (400 MHz, CDCl_3) δ 4.13 (s, 3H), 7.42 (dd, J =4.4, 4.0Hz, 1H), 7.75 (d, J =8.8Hz, 1H), 7.90 (s, 1H), 8.18 (dd, J =9.2, 2.0Hz, 2H), 8.96 (d, J =3.2Hz, 1H); ^{13}C NMR (100MHz, CDCl_3) δ 62.07, 113.2, 121.6, 125.4, 127.9, 129.7, 130.8, 136.2, 137.8 (m), 139.9 (m), 142.4 (m), 143.0 (m), 145.6 (m), 147.7, 151.2; ^{19}F NMR (400 MHz, CDCl_3) δ -157.8 (dd, 2F), -144.7 (m, 2F); HRMS: calcd. for $\text{C}_{16}\text{H}_{10}\text{NOF}_4^+$: 308.0699, found 308.0706.

3'-(2,3,5,6-Tetrafluoro-4-(trifluoromethyl)phenyl)acetophenone (Compound 3ce)



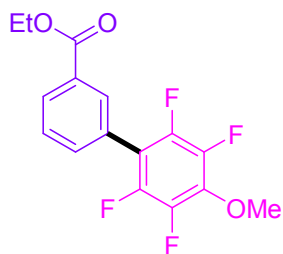
Hexane: EA = 9:1, R_f = 0.25; m. p. 86.3-88.3 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.661 (s, 3H), 7.66 (d, $J=7.2\text{Hz}$, 2H), 8.10 (dd, 4.4, 4.8Hz, 2H); ^{13}C NMR (100MHz, CDCl_3) δ 26.4, 119.3, 123.8, 126.5, 129.2, 129.6, 129.7, 134.1, 137.6, 142.9 (m), 145.4 (m), 196.8; ^{19}F NMR (400 MHz, CDCl_3) δ -141.3 (m, 2F), -140.3 (m, 3F), -56.4 (t, 2F); HRMS: calcd. for $\text{C}_{15}\text{H}_7\text{OF}_7^+$: 336.0385, found 336.0375.

6-(2,3,4,6-Tetrafluorophenyl)quinoline (Compound 3dc)



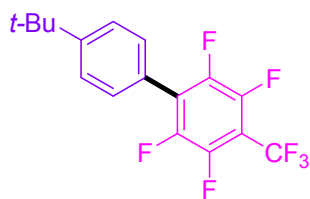
Hexane: EA = 4:1, R_f = 0.25; m. p. 130-132.3 °C; ^1H NMR (400 MHz, CDCl_3) δ 6.90-6.97 (m, 1H), 7.46 (dd, J = 4.4, 4.0 Hz, 1H), 7.76 (dd, J = 1.2, 7.6 Hz, 1H), 7.92 (s, 1H), 8.21 (t, J = 8.8 Hz, 2H), 8.98 (s, 1H); ^{13}C NMR (100MHz, CDCl_3) δ 101.0 (m), 121.6, 125.7, 127.9, 129.6, 129.7, 130.9, 136.3, 147.7, 151.2; ^{19}F NMR (400 MHz, CDCl_3) δ -164.3 (m, 1F), -135.2 (dd, 1F), -132.3 (dd, 1F), -117.8 (d, 1F); HRMS: calcd. for $\text{C}_{15}\text{H}_8\text{NF}_4^+$: 278.0593, found 278.0605.

**[1,1'-Biphenyl]-3-carboxylic acid, 2',3',5',6'-tetrafluoro-4'-methoxy-, ethyl ester
(Compound 3bj)**



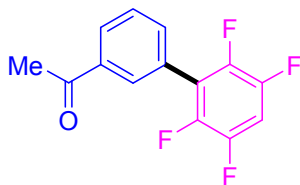
Hexane: EA = 9:1, R_f = 0.5; m. p. 87.3-88.8 °C; ^1H NMR (400 MHz, CDCl_3) δ 1.42 (t, J = 7.2 Hz, 3H), 4.14 (s, 3H), 4.42 (dd, J = 7.2, 6.8 Hz, 2H), 7.54-7.63 (m, 2H), 8.13 (d, J = 7.6 Hz, 2H); ^{13}C NMR (100MHz, CDCl_3) δ 14.2, 61.2, 62.1, 113.1, 127.5, 128.6, 129.9, 131.0, 131.2, 134.3, 139.8 (m), 142.4 (m), 143.0 (m), 145.5 (m), 165.9; ^{19}F NMR (400 MHz, CDCl_3) δ -157.9 (dd, 2F), -145.1 (dd, 2F); HRMS: calcd. for $\text{C}_{16}\text{H}_{12}\text{O}_3\text{F}_4^+$: 328.0723, found 328.0722.

2,3,5,6-Tetrafluoro-4'-tert-butyl-4-(trifluoromethyl)-1,1'-biphenyl (Compound 3ca)



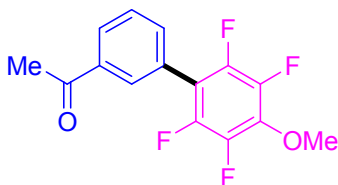
Pure hexane, R_f = 0.7; m. p. 67-68.8 °C; ^1H NMR (400 MHz, CDCl_3) δ 1.42 (s, 9H), 7.46 (d, J = 8.4 Hz, 2H), 7.59 (d, J = 8.8 Hz, 2H); ^{13}C NMR (100MHz, CDCl_3) δ 31.1, 34.8, 123.1, 124.9 (m), 125.8, 129.6, 143.0 (m), 145.6 (m), 153.3; ^{19}F NMR (400 MHz, CDCl_3) δ -141.7 (m, 3F), -141.0 (m, 2F), -56.2 (t, 2F); HRMS: calcd. for $\text{C}_{17}\text{H}_{13}\text{F}_7^+$: 350.0905, found 350.0906.

3'-(2,3,5,6-Tetrafluorophenyl)acetophenone (Compound 3ee)



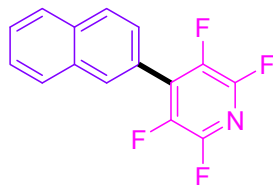
Hexane: EA = 9:1, R_f = 0.48; m. p. 104.9-107 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.66 (s, 3H), 7.08-7.17 (m, 1H), 7.60-7.68 (m, 2H), 8.06-8.07 (t, $J=5.2, 1.6\text{Hz}$, 2H); ^{13}C NMR (100MHz, CDCl_3) δ 26.5, 105.4 (t), 120.6, 127.9, 128.9, 130.0, 134.5, 137.4, 142.5, 144.9, 147.5 (m), 150.9 (m), 197.2; ^{19}F NMR (400 MHz, CDCl_3) δ -143.7 (dd, 2F), -138.6 (dd, 2F); HRMS: calcd. for $\text{C}_{14}\text{H}_9\text{OF}_4^+$: 269.0590, found 269.0582.

3'-(2,3,5,6-Tetrafluoro-4-methoxyphenyl)acetophenone (Compound 3be)



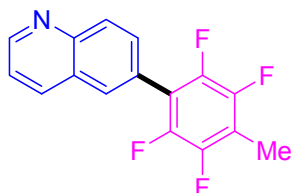
Hexane: EA = 9:1, R_f = 0.35; m. p. 97.8-99.9 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.65 (s, 3H), 4.14 (s, 3H), 7.58-7.65 (m, 2H), 8.03 (t, $J=4.8, 1.6\text{Hz}$, 2H); ^{13}C NMR (100MHz, CDCl_3) δ 26.6, 62.1, 113.1, 127.8, 128.6 (d), 130.1, 134.6, 137.4 (m), 139.9 (d), 142.3 (m), 142.4 (m), 143.0 (m), 145.5 (m), 197.3; ^{19}F NMR (400 MHz, CDCl_3) δ -157.8 (dd, 2F), -145.1 (dd, 2F).

2,3,5,6-Tetrafluoro-4-(naphthalene-2-yl)pyridine (Compound 3fi)



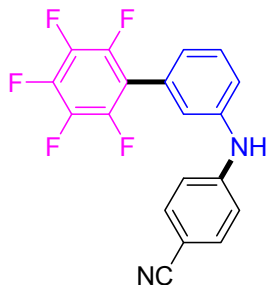
Hexane: EA = 9:1, R_f = 0.65; m. p. 139.8-141.9 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.59-7.66 (m, 3H), 7.94-8.08 (m, 4H); ^{13}C NMR (100MHz, CDCl_3) δ 125.9, 127.0, 127.8, 127.0, 128.6, 128.7, 1303., 132.8, 133.7, 201.2; ^{19}F NMR (400 MHz, CDCl_3) δ -144.9 (dd, 2F), -90.7 (dd, 2F); HRMS: calcd. for $\text{C}_{15}\text{H}_{17}\text{NF}_4^+$: 277.0515, found 277.0514.

6-(2,3,5,6-Tetrafluoro-4-methylphenyl)quinoline (Compound 3gc)



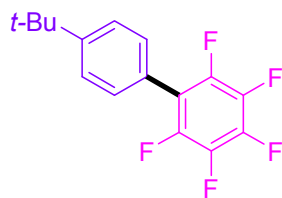
Hexane: EA = 4:1, R_f = 0.3; m. p. 197-198.4 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.36 (s, 3H), 7.46 (dd, J = 4.4, 4.0 Hz, 1H), 7.79 (d, J = 7.6 Hz, 1H), 7.95 (s, 1H), 8.22 (t, J = 4.8, 4.0 Hz, 2H), 8.99 (d, J = 2.4 Hz, 1H); ^{13}C NMR (100MHz, CDCl_3) δ 7.6, 121.6, 127.9, 129.7, 130.9, 136.3, 147.9, 151.3; ^{19}F NMR (400 MHz, CDCl_3) δ -145.4 (dd, 2F), -143.6 (dd, 2F); HRMS: calcd. for $\text{C}_{16}\text{H}_{10}\text{NF}_4^+$: 292.0749, found 292.0737.

4-((3-(Pentafluorophenyl)phenyl)amino)benzonitrile

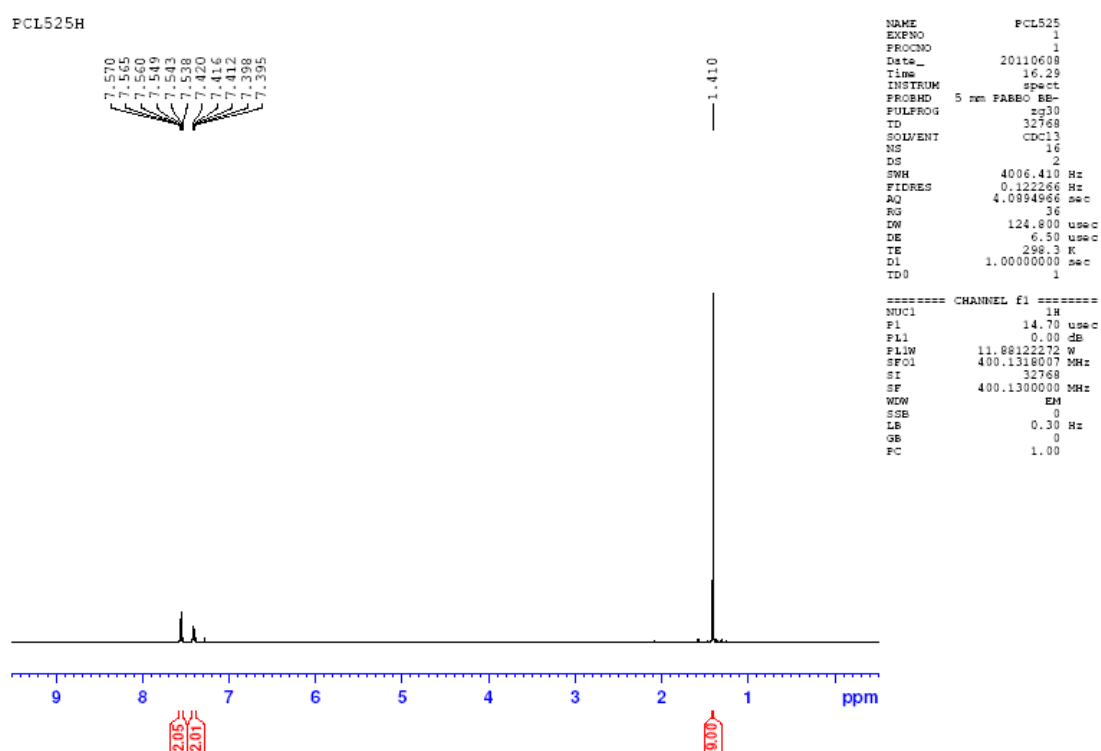


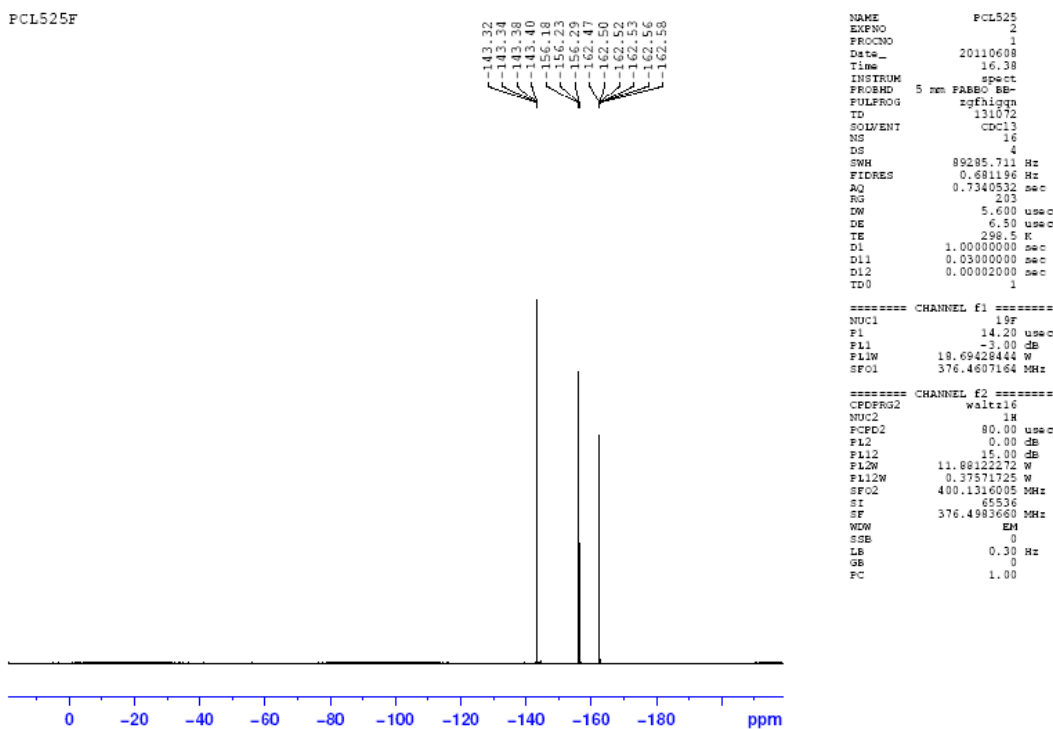
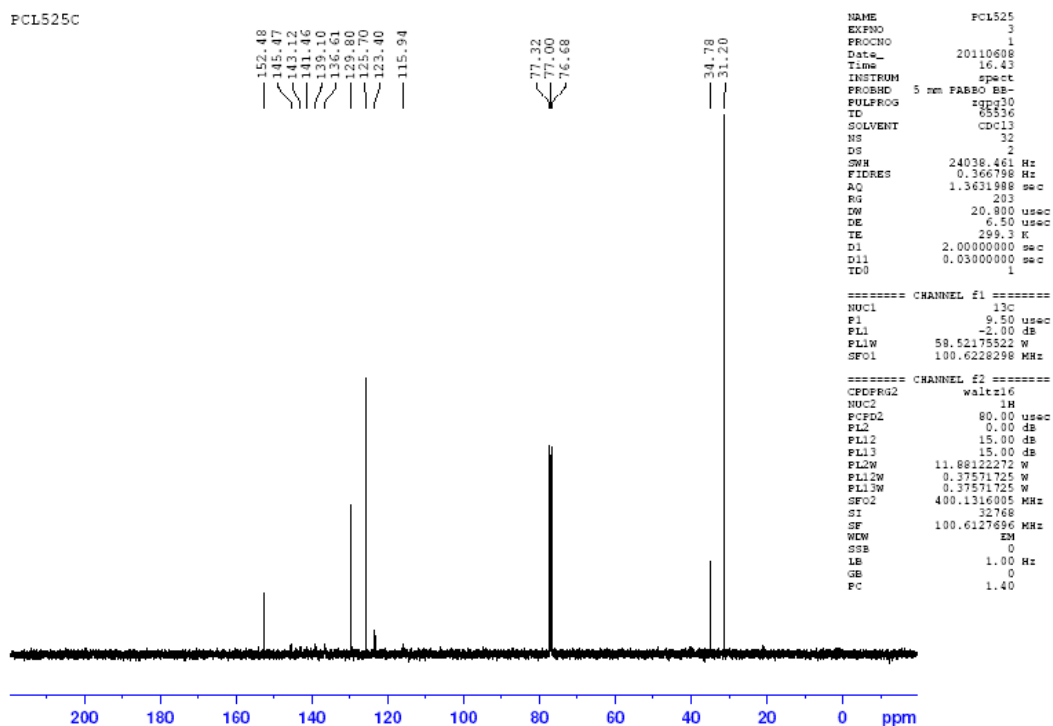
Hexane: EA = 9:1, R_f = 0.15; m. p. 127-128.1 °C; ^1H NMR (400 MHz, CDCl_3) δ 6.37 (s, 1H), 7.06 (d, J = 8.4 Hz, 2H), 7.17 (d, J = 7.6 Hz, 1H), 7.26 (d, J = 2.8 Hz, 2H), 7.49 (dd, J = 8.0, 6.4 Hz, 3H); ^{13}C NMR (100MHz, CDCl_3) δ 14.1, 102.2, 115.3, 115.4, 119.7, 121.4, 122.2, 125.3, 127.7, 130.0, 133.8, 140.7, 147.4; ^{19}F NMR (400 MHz, CDCl_3) δ -162.4 (m, 2F), -156.0 (t, 2F), -142.7 (dd, 1F); HRMS: calcd. for $\text{C}_{19}\text{H}_{10}\text{N}_2\text{F}_5^+$: 361.0764, found 361.0762.

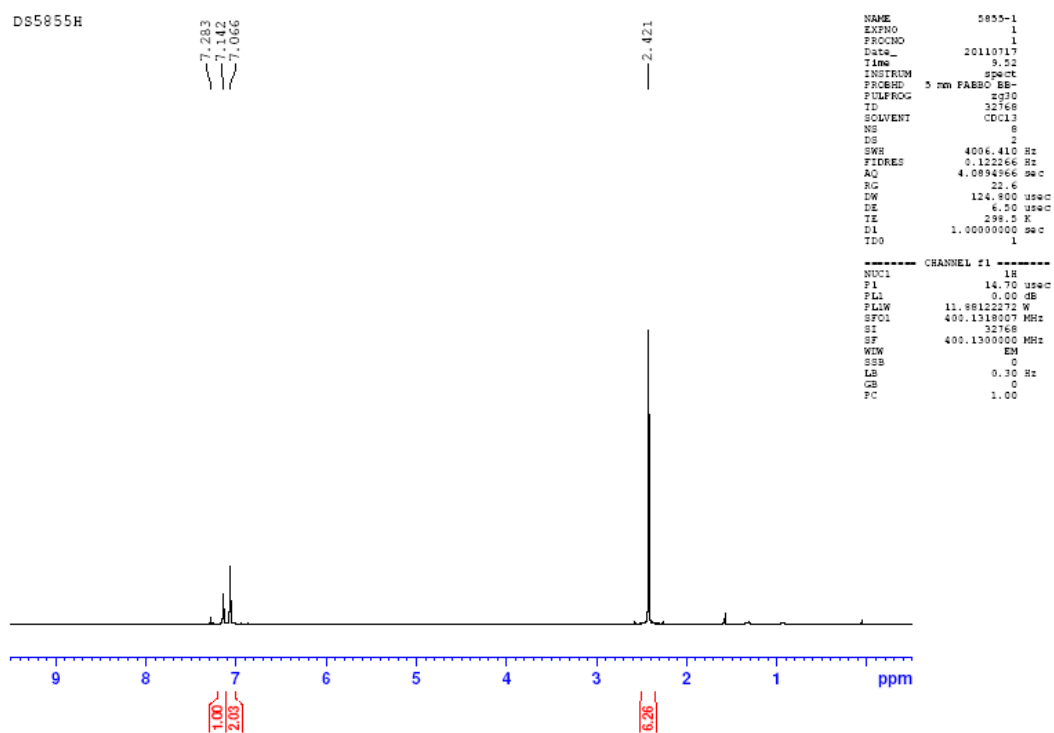
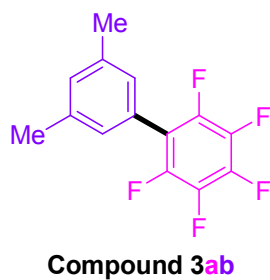
7. ^1H , ^{13}C , MS and HRMS spectra

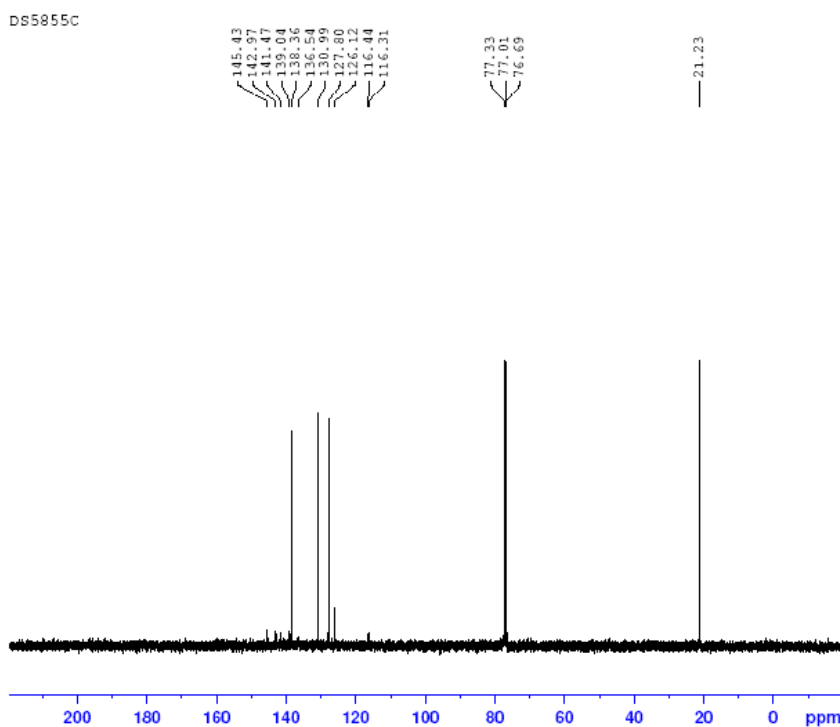


Compound **3aa**







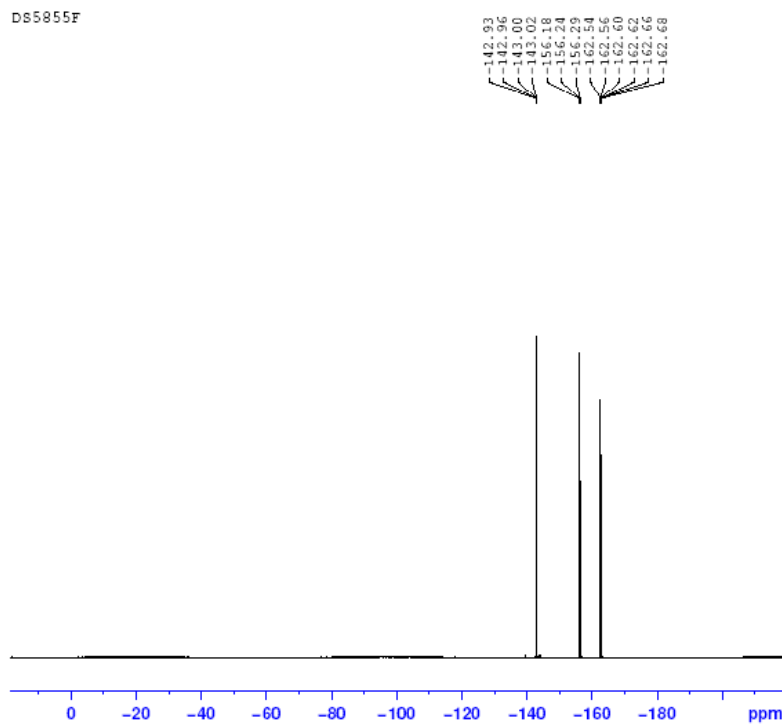


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NAME          5855-1-C
EXPNO         1
PROCNO        1
Date_         20110717
Time          9.56
INSTRUM       spect
PROBHD        5 mm FAPBO BB-
PULPROG       zgpg30
TD            85536
SOLVENT       CDCl3
NS            32
DS            2
SWH           24039.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            203
DW            20.800 usec
DE            6.50 usec
TE            299.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           2.00 dB
PL1W         58.5217552 W
SFO1         100.6228298 MHz

===== CHANNEL f2 =====
CFPRG2       waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           0.00 dB
PL12         15.00 dB
PL13         15.00 dB
PL2W         11.88122272 W
PL12W        0.37571725 W
PL13W        0.37571725 W
SFO2         400.1316005 MHz
SI           32768
SF           100.6127690 MHz
WDW          EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

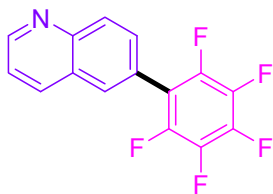


```

NAME          5855-1-F
EXPNO         1
PROCNO        1
Date_         20110717
Time          9.58
INSTRUM       spect
PROBHD        5 mm FAPBO BB-
PULPROG       zgpg30
TD            131072
SOLVENT       CDCl3
NS            16
DS            4
SWH           89295.711 Hz
FIDRES        0.681196 Hz
AQ            0.7340532 sec
RG            203
DW            5.600 usec
DE            6.50 usec
TE            298.8 K
D1            1.00000000 sec
D11           0.03000000 sec
D12           0.00020000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13F
P1            14.20 usec
PL1           -3.00 dB
PL1W         19.69428444 W
SFO1         376.4607164 MHz

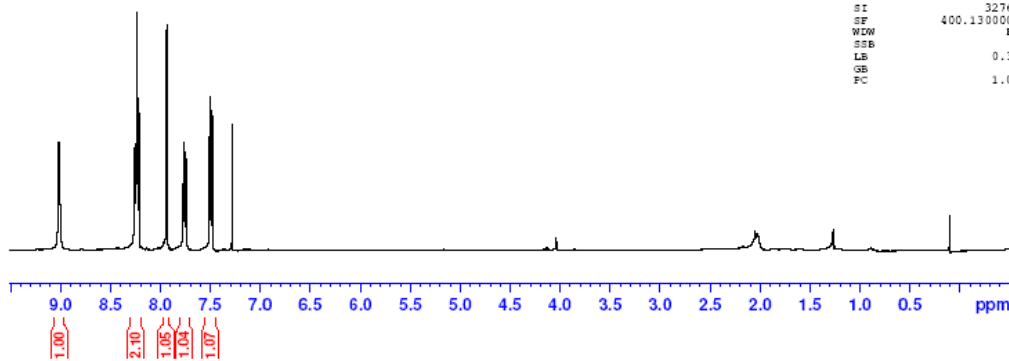
===== CHANNEL f2 =====
CFPRG2       waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           0.00 dB
PL12         15.00 dB
PL13         15.00 dB
PL2W         11.88122272 W
PL12W        0.37571725 W
PL13W        0.37571725 W
SFO2         400.1316005 MHz
SI           85536
SF           376.4983660 MHz
WDW          EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
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Compound 3ac

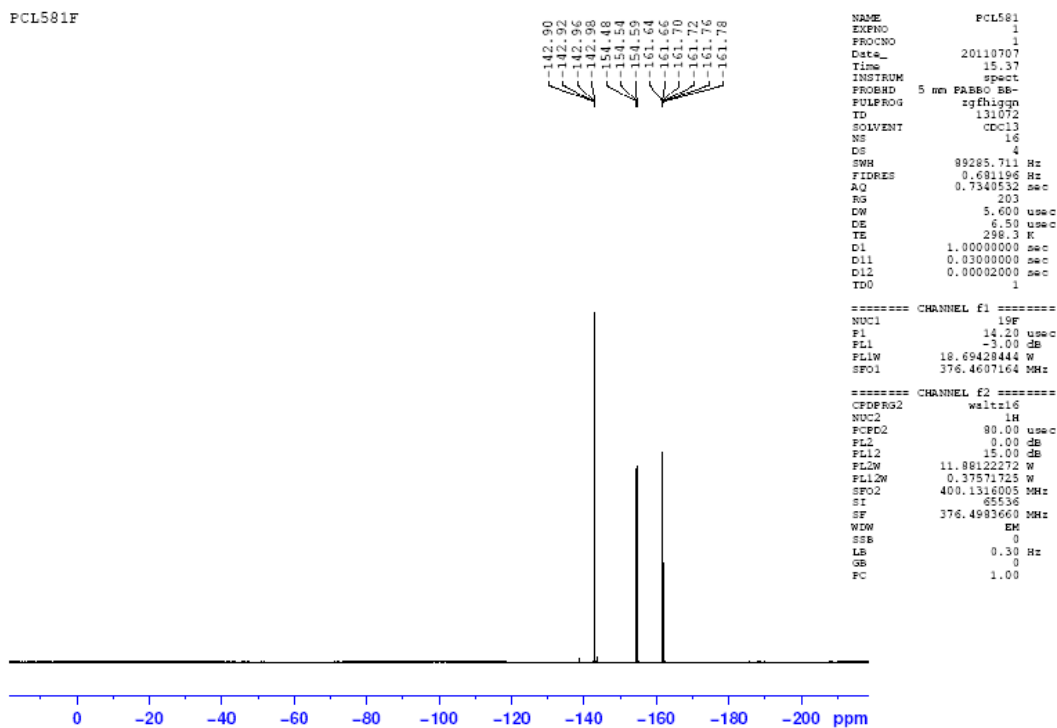
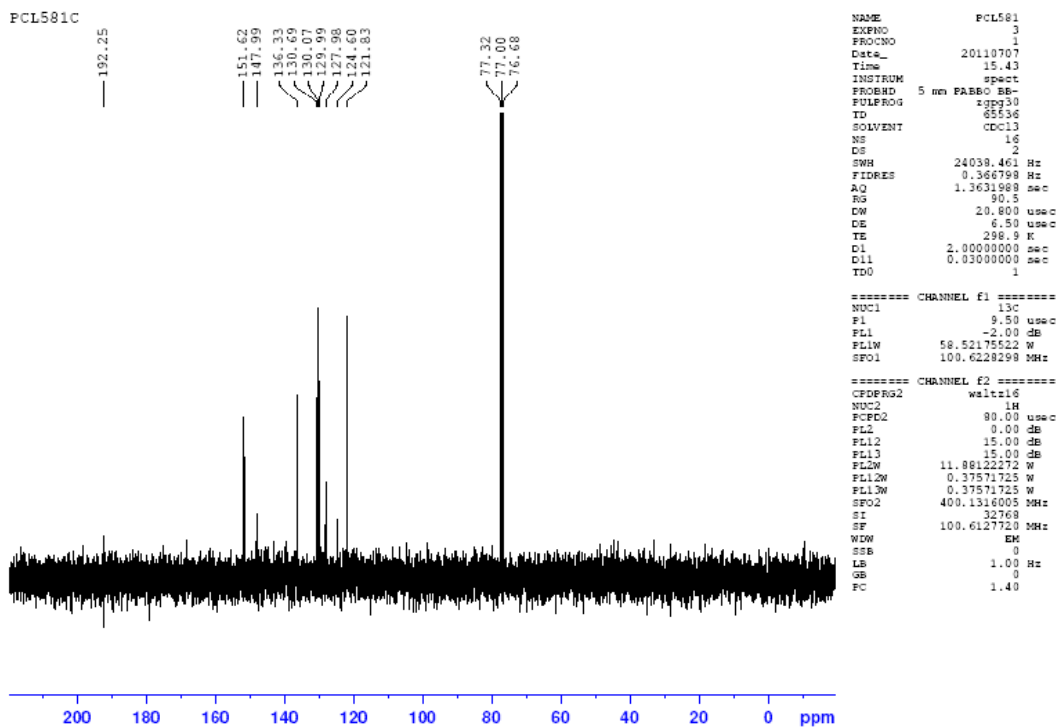
PCL581H

9.017
8.910
8.255
8.238
8.235
8.219
8.215
7.968
7.937
7.768
7.745
7.743
7.508
7.497
7.487
7.476
7.284



```
NAME          PCL581
EXPNO         2
PROCNO        1
Date_         20110707
Time          15.40
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            16
DS            2
SWH           4006.410 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG            48.2
CW           124.800 usec
DE            6.50 usec
TE            298.2 K
DL            1.0000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.70 usec
PL1           0.00 db
PL1W          11.98122272 W
SFO1          400.1318007 MHz
SI            32768
SF            400.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
```

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions

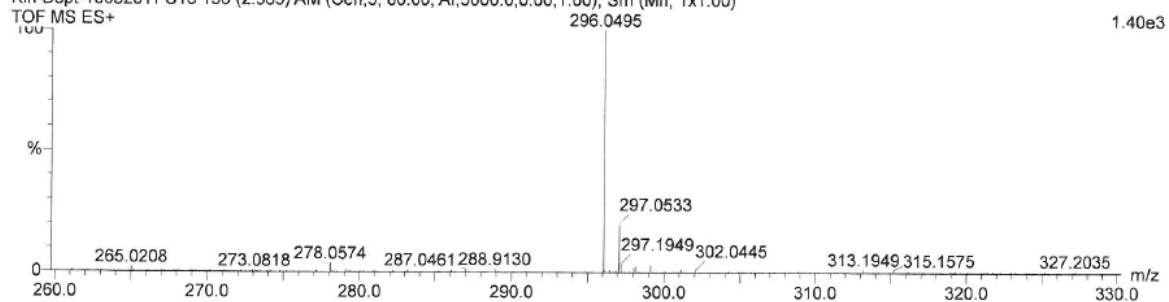
32 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-15 H: 0-7 N: 0-3 F: 0-5 Na: 0-1

Kin-Dept-10082011 S16 136 (2.565) AM (Cen,5, 80.00, Ar,5000.0,0.00,1.00); Sm (Mn, 1x1.00)

TOF MS ES+

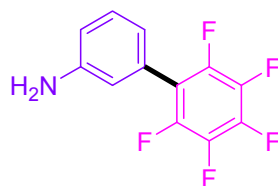


Minimum:

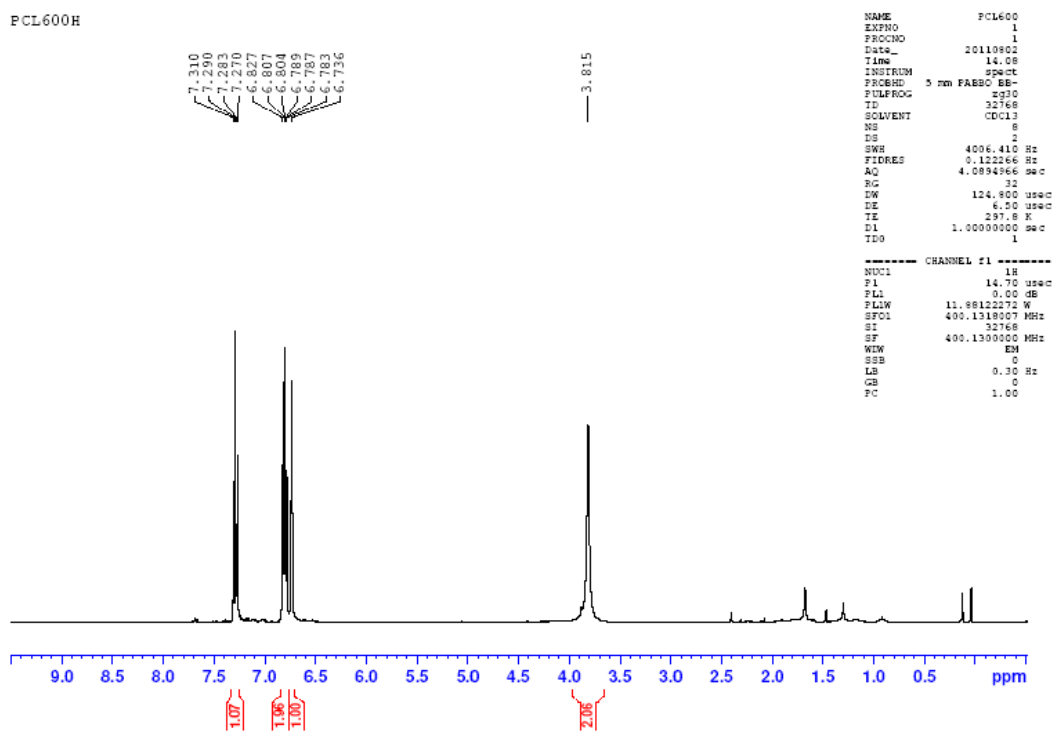
Maximum: 5.0 10.0 -1.5

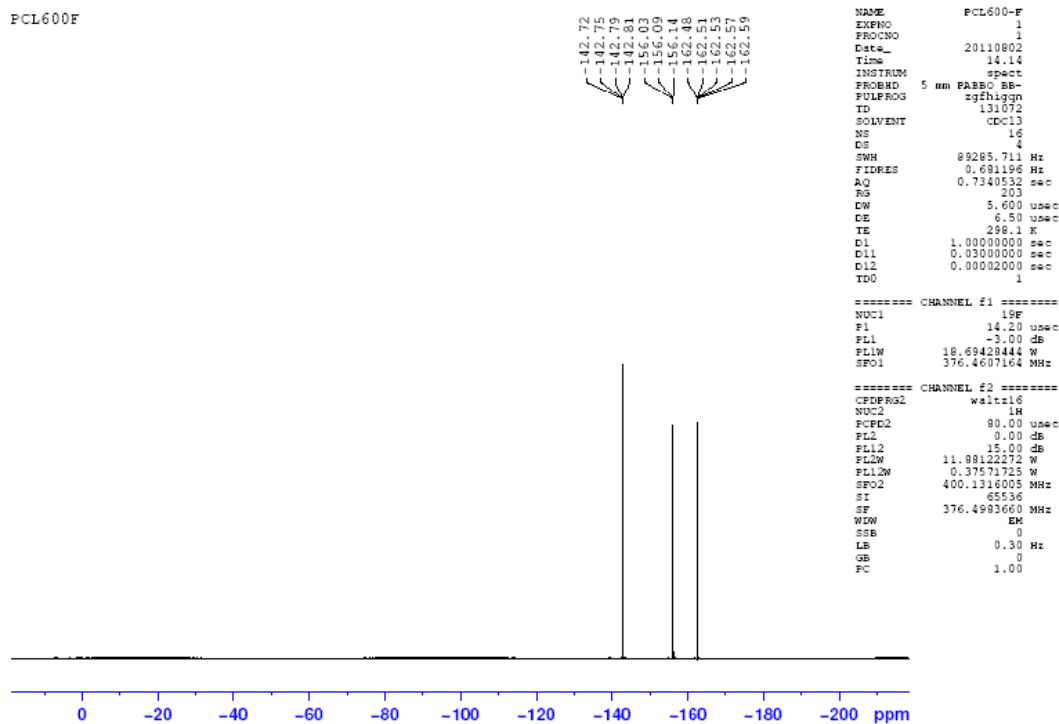
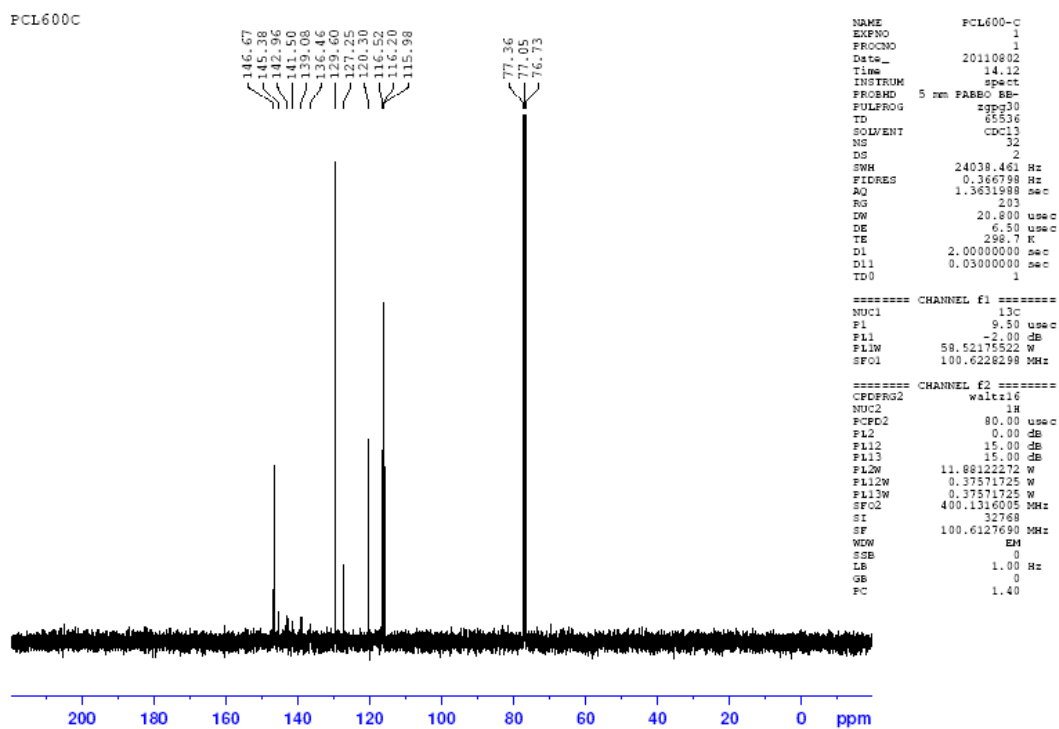
50.0

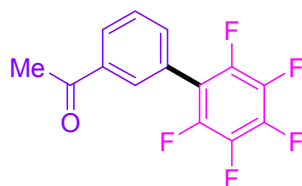
| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
|----------|------------|------|------|------|-------|-------------|
| 296.0495 | 296.0499 | -0.4 | -1.4 | 10.5 | 3.1 | C15 H7 N F5 |



Compound 3ad

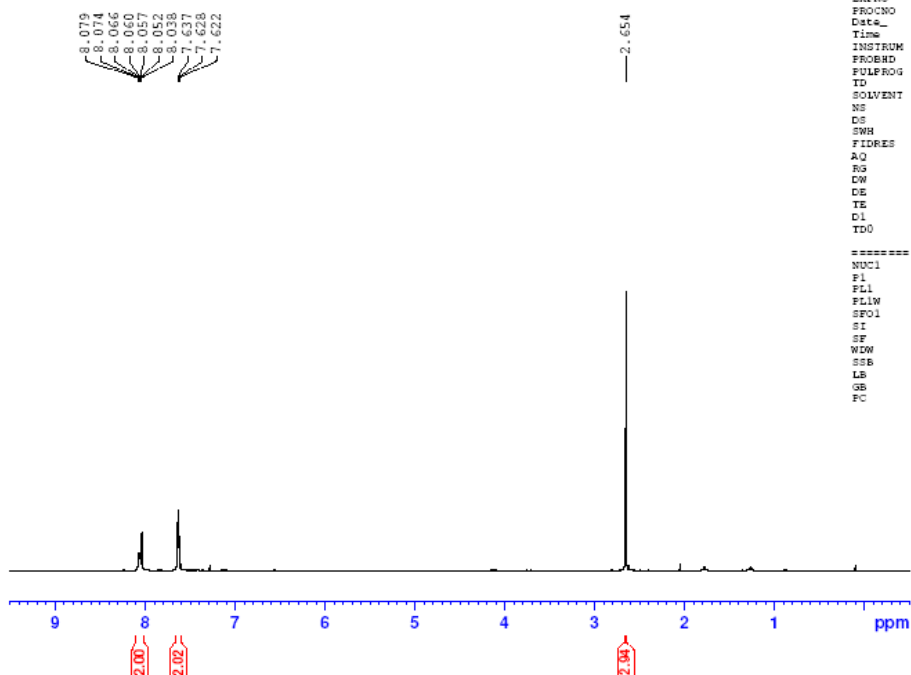






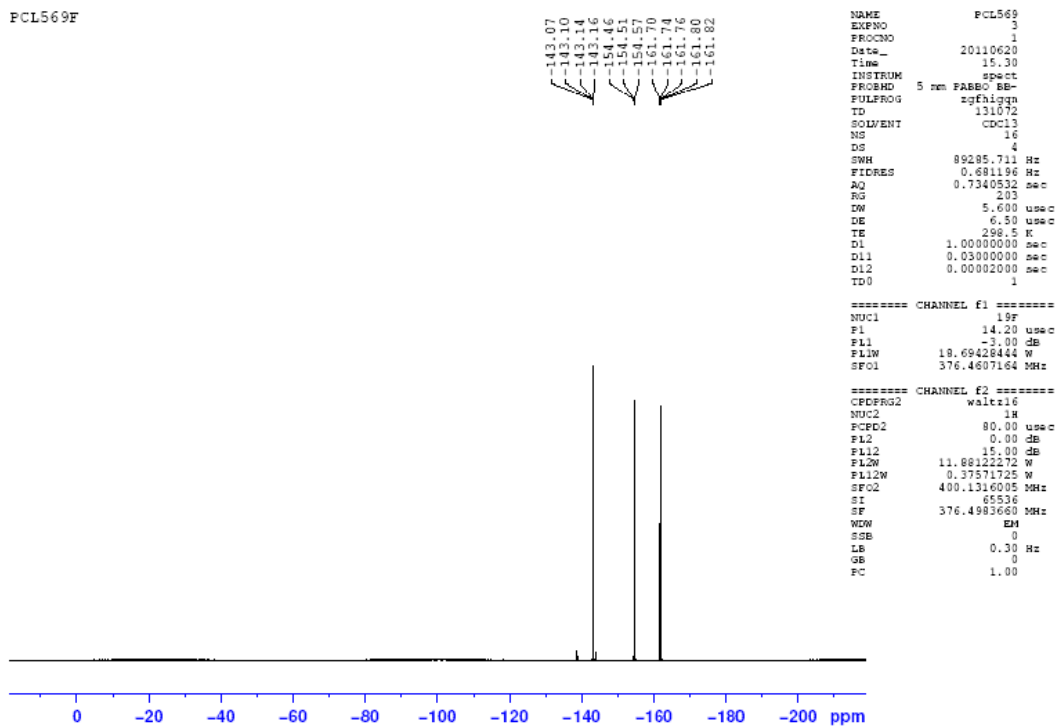
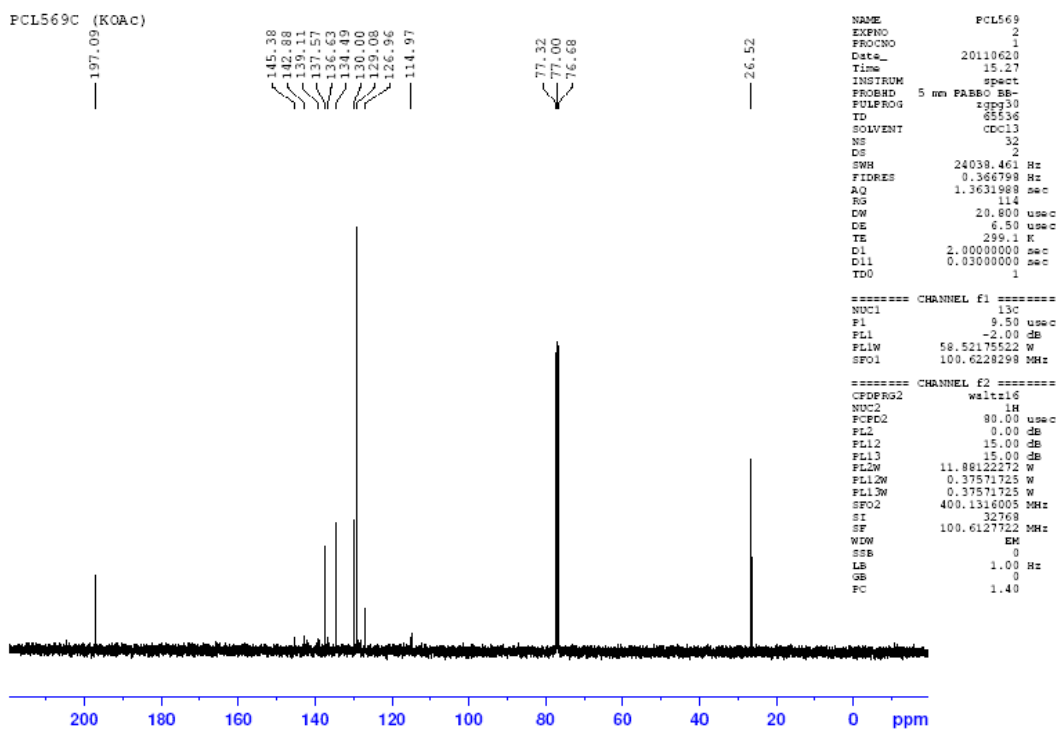
Compound 3ae

PCL569H (K0Ac)



```
NAME          PCL569
EXPNO         1
PROCNO        1
Date_         20110620
Time         15.32
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            16
DS            2
SWH           5597.015 Hz
FIDRES        0.170807 Hz
AQ            2.9273248 sec
RG            40.3
DE            89.333 usec
TE            298.3 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.70 usec
PL1           0.00 dB
PL1W         11.88122272 W
SFO1          400.1324009 MHz
SI            32768
SF            400.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
```



Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions

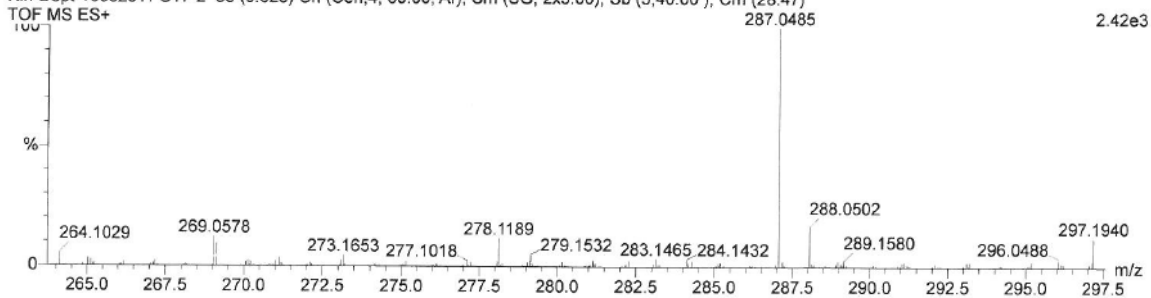
39 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-15 H: 0-8 O: 0-4 F: 0-5 Na: 0-1

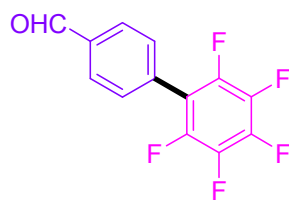
Kin-Dept-10082011 S17-2 33 (0.628) Cn (Cen,4, 80.00, Ar); Sm (SG, 2x3.00); Sb (5,40.00); Cm (28.47)

TOF MS ES+



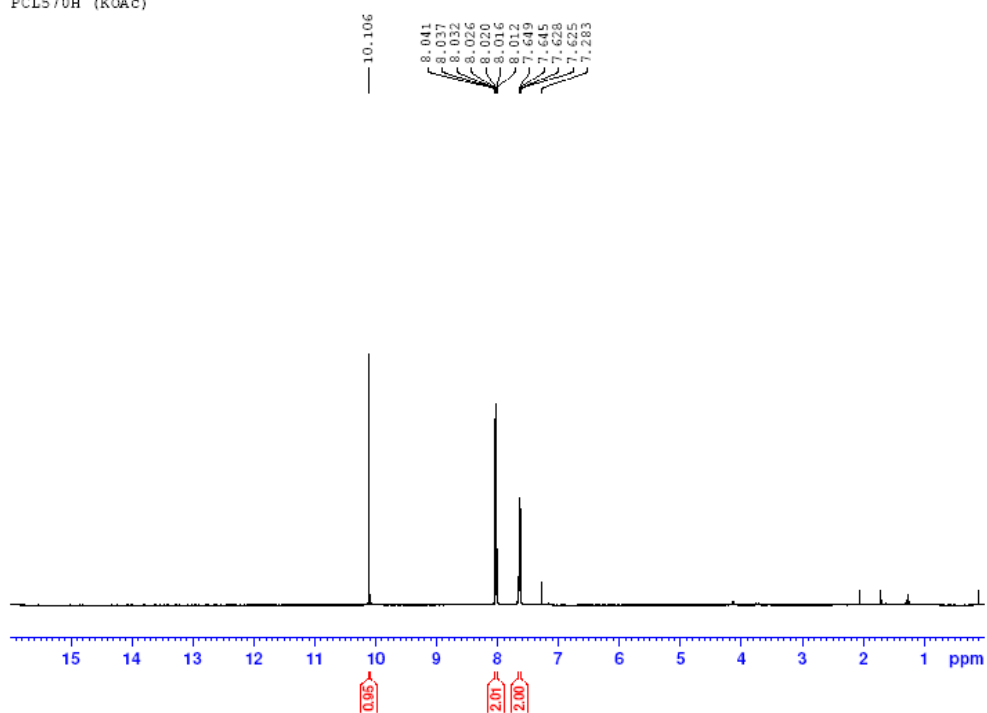
Minimum: -1.5
Maximum: 5.0 5.0 50.0

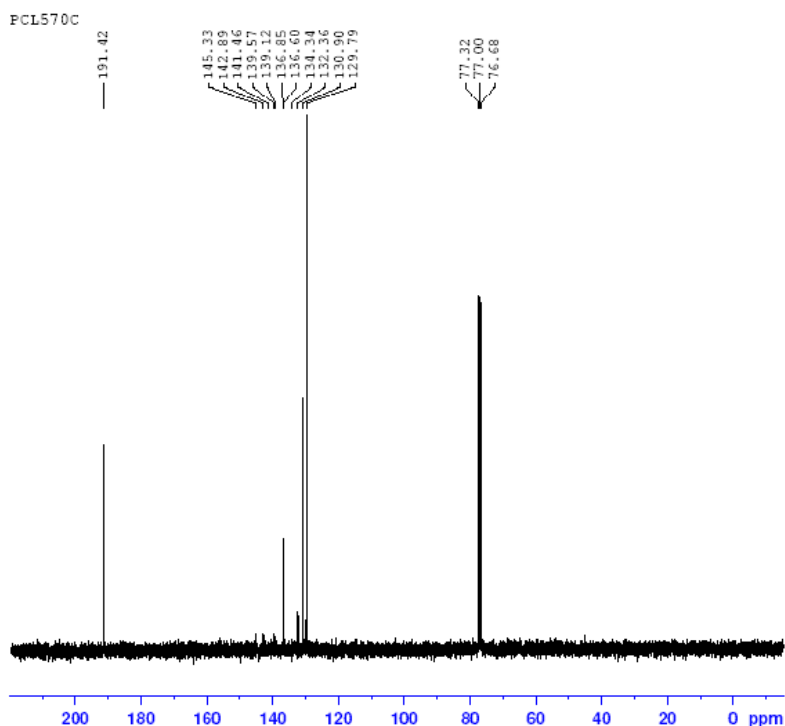
| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
|----------|------------|------|------|-----|-------|-------------|
| 287.0485 | 287.0495 | -1.0 | -3.5 | 8.5 | 0.8 | C14 H8 O F5 |



Compound 3af

PCL570H (KOC)



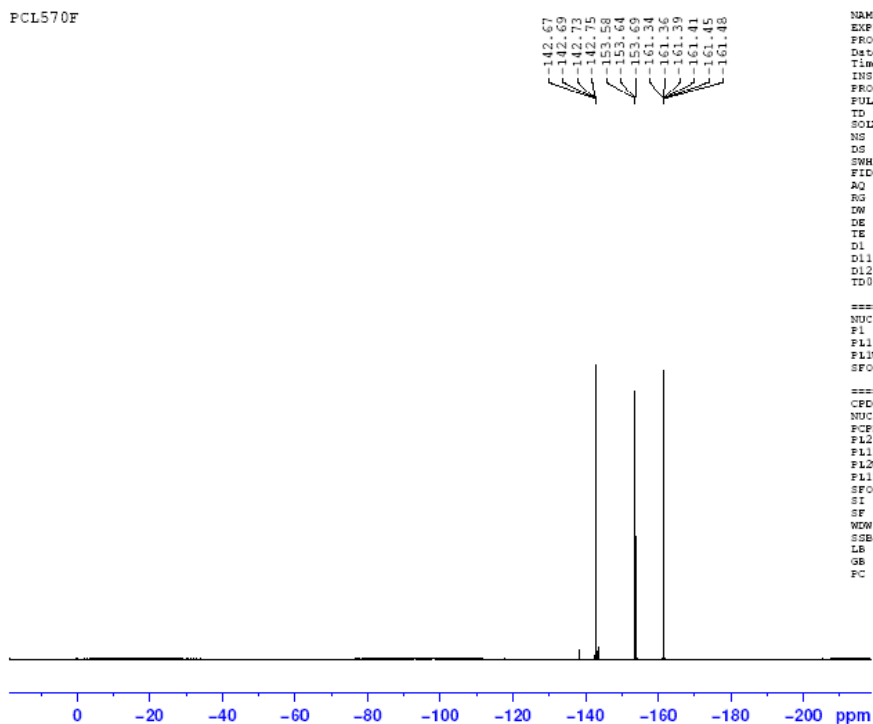


```

NAME          PCL570
EXPNO         3
PROCNO        1
Date_         20110625
Time         16.46
INSTRUM       spect
PROBHD        5 mm FAPBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           24039.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631989 sec
RG            203
LW           20.800 usec
DE            6.50 usec
TE            298.8 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           2.00 dB
PL1W         58.5217552 W
SFO1         100.6228298 MHz

===== CHANNEL f2 =====
CFPRG2       waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           0.00 dB
PL2W         15.00 dB
PL13         15.00 dB
PL2W         11.88122272 W
PL12W        0.37571725 W
SFO2         400.1316005 MHz
SI           32768
SF           100.6127715 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

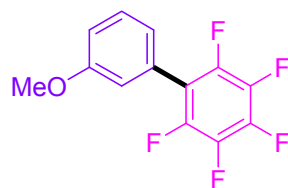


```

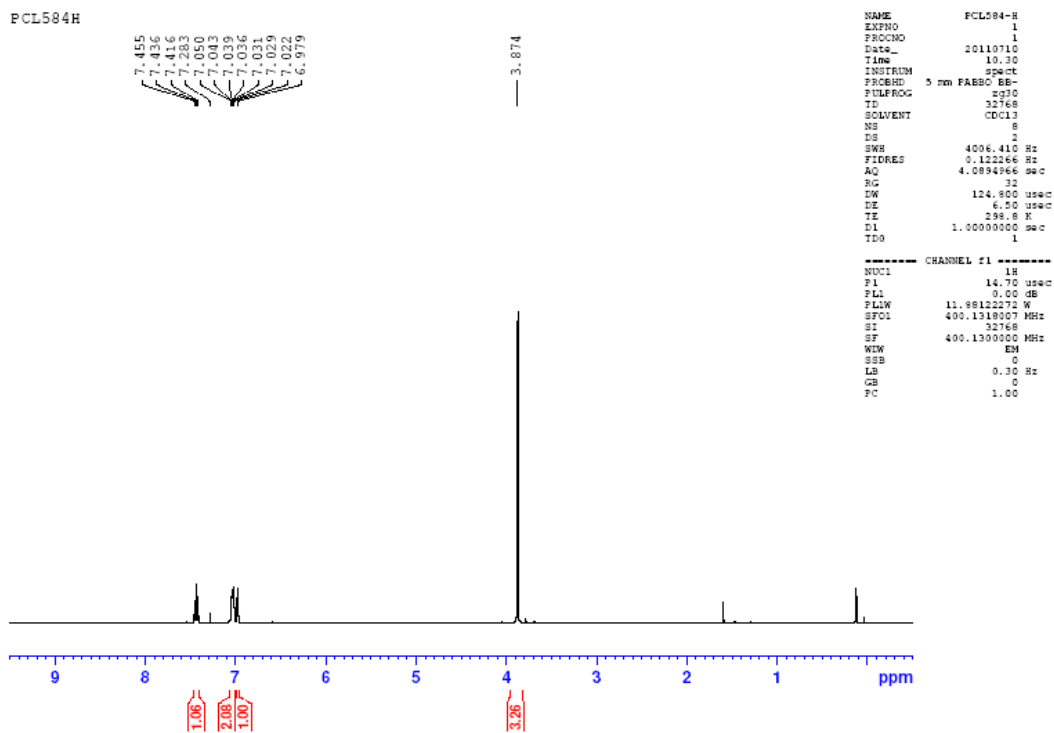
NAME          PCL570
EXPNO         1
PROCNO        1
Date_         20110625
Time         16.37
INSTRUM       spect
PROBHD        5 mm FAPBO BB-
PULPROG       zgpg11gqn
TD            131072
SOLVENT       CDCl3
NS            16
DS            4
SWH           89285.711 Hz
FIDRES        0.681196 Hz
AQ            0.7340532 sec
RG            203
LW           5.600 usec
DE            6.50 usec
TE            298.4 K
D1            1.0000000 sec
D11           0.0300000 sec
D12           0.0000200 sec
TD0           1

===== CHANNEL f1 =====
NUC1          19F
P1           14.20 usec
PL1           -3.00 dB
PL1W         18.69428444 W
SFO1         376.4607164 MHz

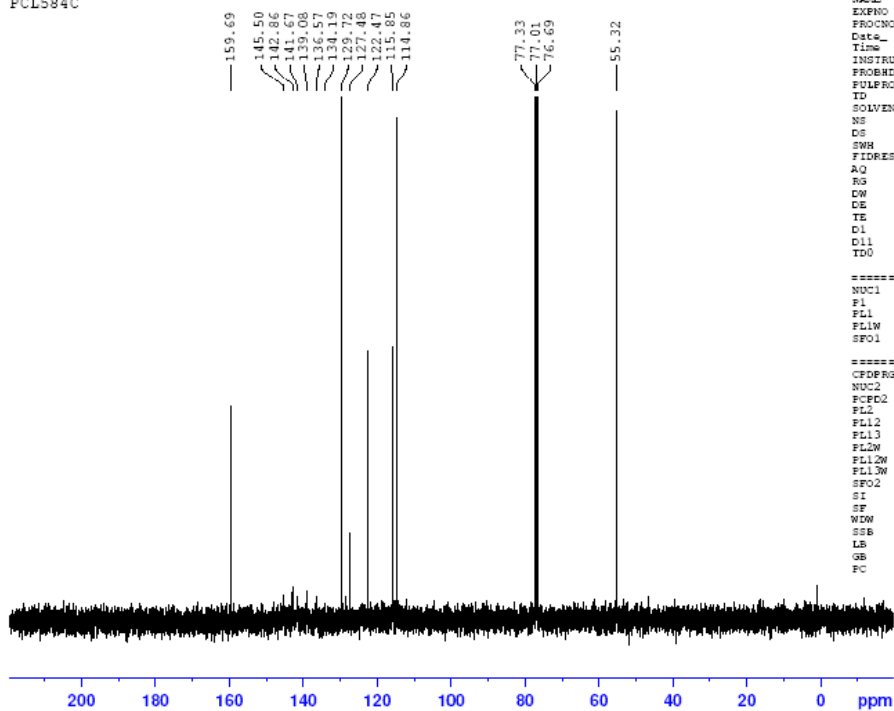
===== CHANNEL f2 =====
CFPRG2       waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           0.00 dB
PL2W         15.00 dB
PL12W        11.88122272 W
PL12W        0.37571725 W
SFO2         400.1316005 MHz
SI           65536
SF           376.4883669 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```



Compound 3ag



PCL584C



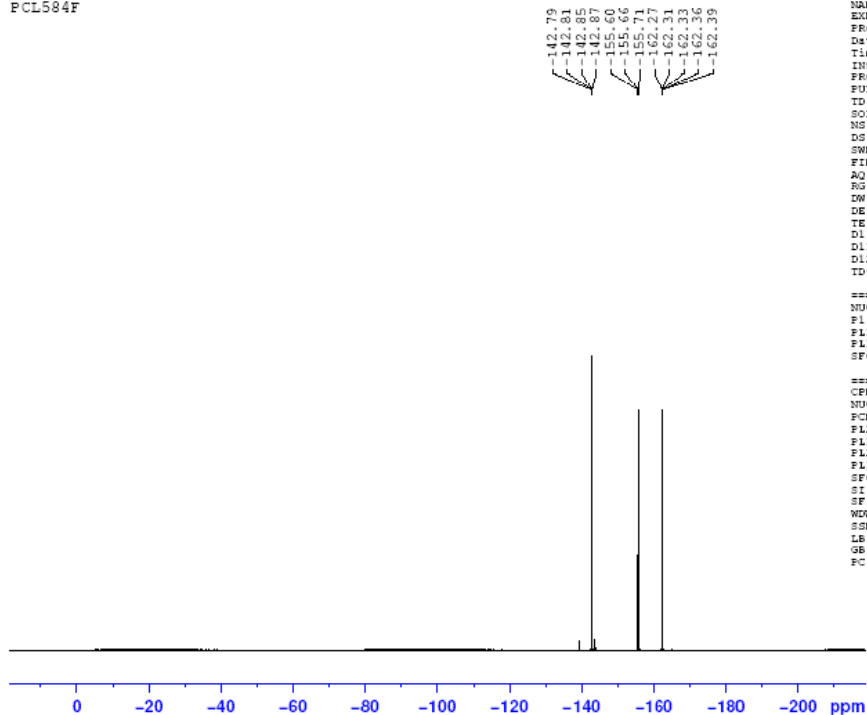
```

NAME          PCL584-C
EXPNO         1
PROCNO        1
Date_         20110710
Time          10.33
INSTRUM       spect
PROBHD        5 mm FAPBO BB-
PULPROG       zgpg30
TD            85536
SOLVENT       CDCl3
NS            32
DS            2
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            203
LW           20.800 usec
DE            6.50 usec
TE            299.7 K
DL            2.0000000 sec
Dl1           0.0300000 sec
Dl2           0.0300000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -2.00 dB
PL1W          58.5217552 W
SF01          100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12          15.00 dB
PL13          15.00 dB
PL2W          11.88122272 W
PL12W         0.37571725 W
PL13W         0.37571725 W
SF02          400.1316005 MHz
SI            32768
SF           100.6127690 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

PCL584F

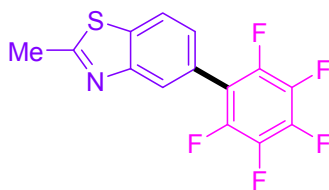


```

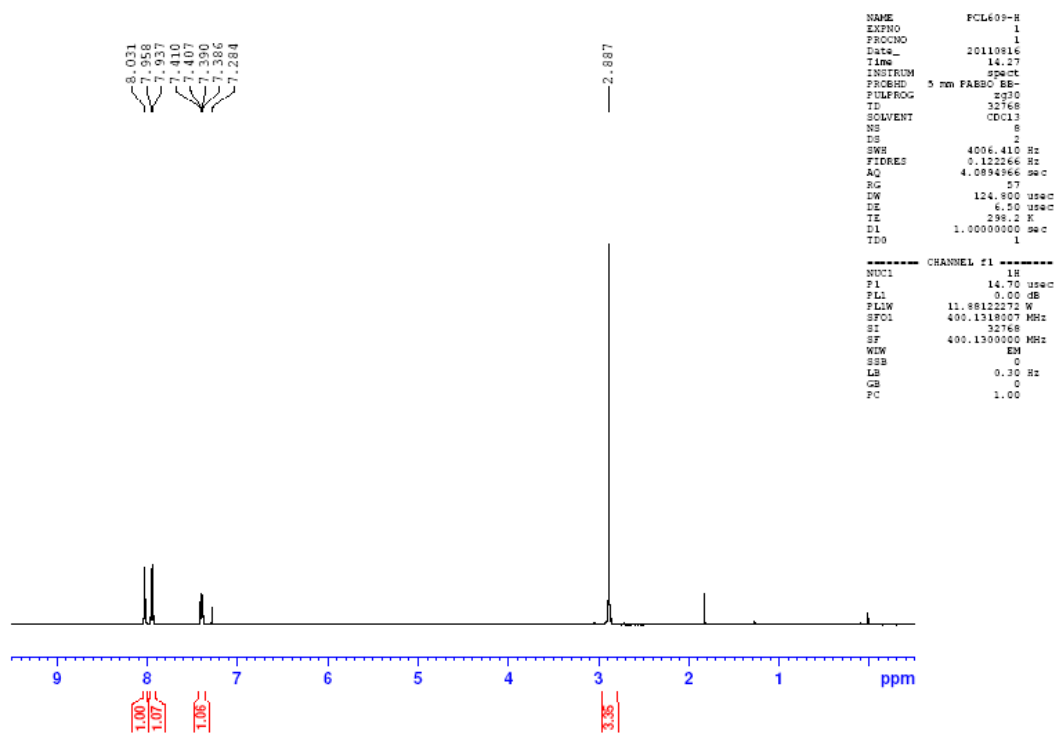
NAME          PCL584-F
EXPNO         1
PROCNO        1
Date_         20110710
Time          10.36
INSTRUM       spect
PROBHD        5 mm FAPBO BB-
PULPROG       zgpg30
TD            131072
SOLVENT       CDCl3
NS            16
DS            4
SWH           89285.711 Hz
FIDRES        0.681196 Hz
AQ            0.7340532 sec
RG            203
LW            5.600 usec
DE            6.50 usec
TE            299.0 K
DL            1.0000000 sec
Dl1           0.0300000 sec
Dl2           0.0002000 sec
TD0           1

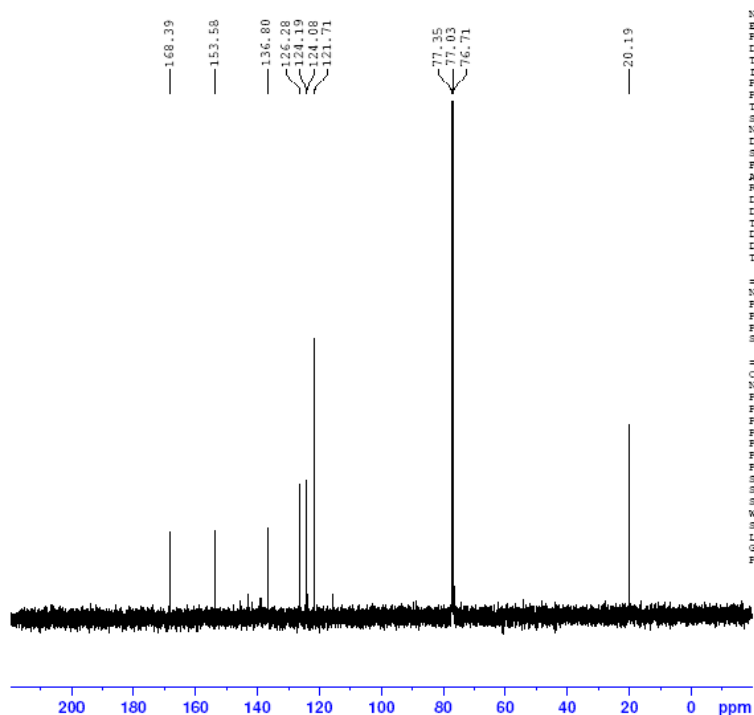
===== CHANNEL f1 =====
NUC1          19F
P1            14.20 usec
PL1           -3.00 dB
PL1W          18.69428444 W
SF01          376.4607164 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12          15.00 dB
PL13          15.00 dB
PL2W          11.88122272 W
PL12W         0.37571725 W
PL13W         0.37571725 W
SF02          400.1316005 MHz
SI            85536
SF           376.4983660 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```



Compound 3ah





```

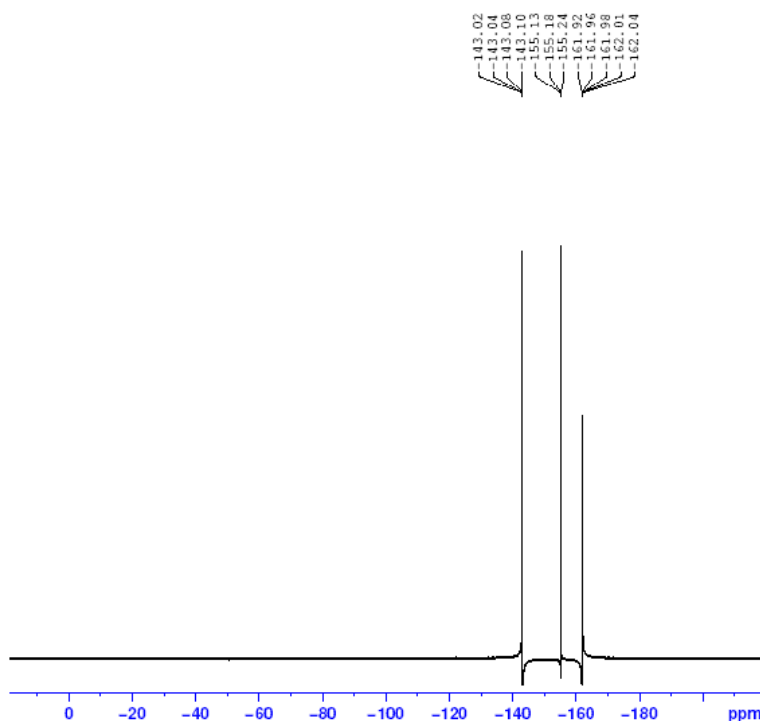
NAME          PCL609-C
EXPNO         1
PROCNO        1
Date_         20110816
Time          14.30
INSTRUM       spect
PROBHD        5 mm FAPBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       cdcl3
NS            32
DS            2
SWH           24030.461 Hz
FIDRES        0.360798 Hz
AQ            1.3631998 sec
RG            203
IN           20.000 usec
DE            6.50 usec
TE            299.1 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1
    
```

```

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -2.00 dB
PL1W         59.5217522 W
SF01         100.6228298 MHz
    
```

```

===== CHANNEL f2 =====
CFDPRG2      waltr16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12         15.00 dB
PL13         15.00 dB
PL2W         11.68122272 W
PL12W        0.37571725 W
PL13W        0.37571725 W
SF02         400.1316005 MHz
SI            32768
SF           100.6127699 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```



```

NAME          PCL609-F
EXPNO         1
PROCNO        1
Date_         20110816
Time          14.37
INSTRUM       spect
PROBHD        5 mm FAPBO BB-
PULPROG       zgzhigpn
TD            131072
SOLVENT       cdcl3
NS            16
DS            4
SWH           89295.711 Hz
FIDRES        0.681196 Hz
AQ            0.7340532 sec
RG            203
IN           5.600 usec
DE            6.50 usec
TE            299.6 K
D1            1.0000000 sec
D11           0.0300000 sec
D12           0.00002000 sec
TD0           1
    
```

```

===== CHANNEL f1 =====
NUC1          19F
P1            14.20 usec
PL1           -3.00 dB
PL1W         19.69428444 W
SF01         376.4607164 MHz
    
```

```

===== CHANNEL f2 =====
CFDPRG2      waltr16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12         15.00 dB
PL2W         11.68122272 W
PL12W        0.37571725 W
SF02         400.1316005 MHz
SI            65536
SF           376.4983660 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 20.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions

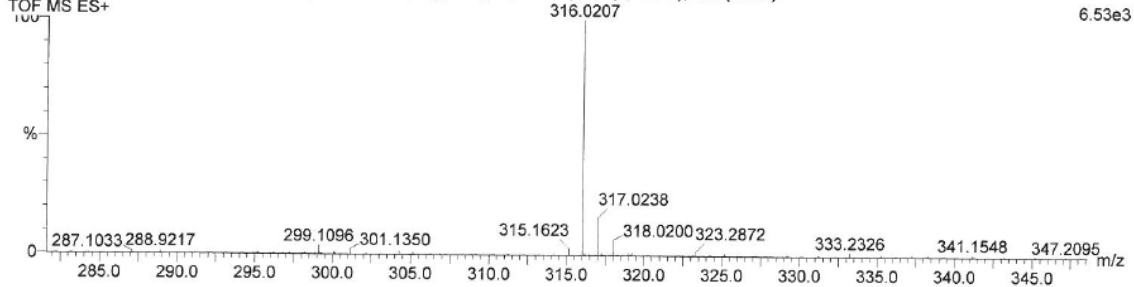
105 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-14 H: 0-7 N: 0-2 Na: 0-1 S: 0-1 39K: 0-1 F: 0-5

Kin-Dept-25082011 S10 49 (0.917) Cn (Cen,4, 80.00, Ar); Sm (SG, 2x3.00); Sb (5,40.00); Cm (41:52)

TOF MS ES+

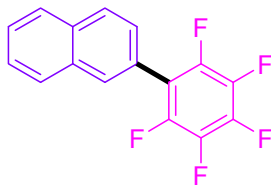


Minimum:

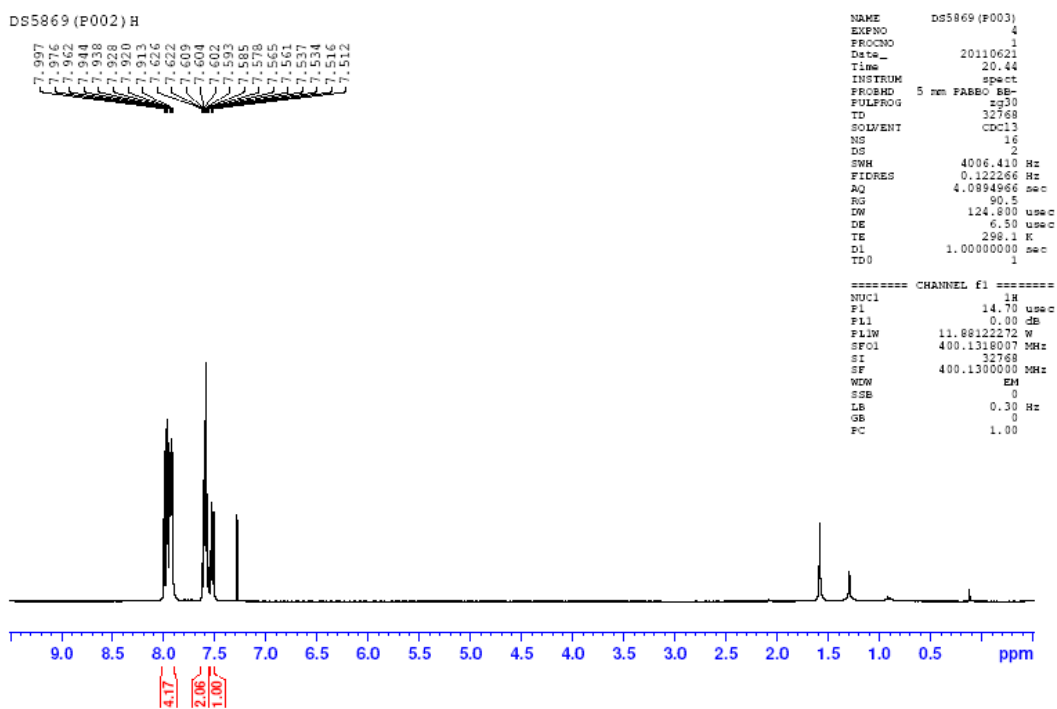
Maximum: 5.0 20.0 -1.5

50.0

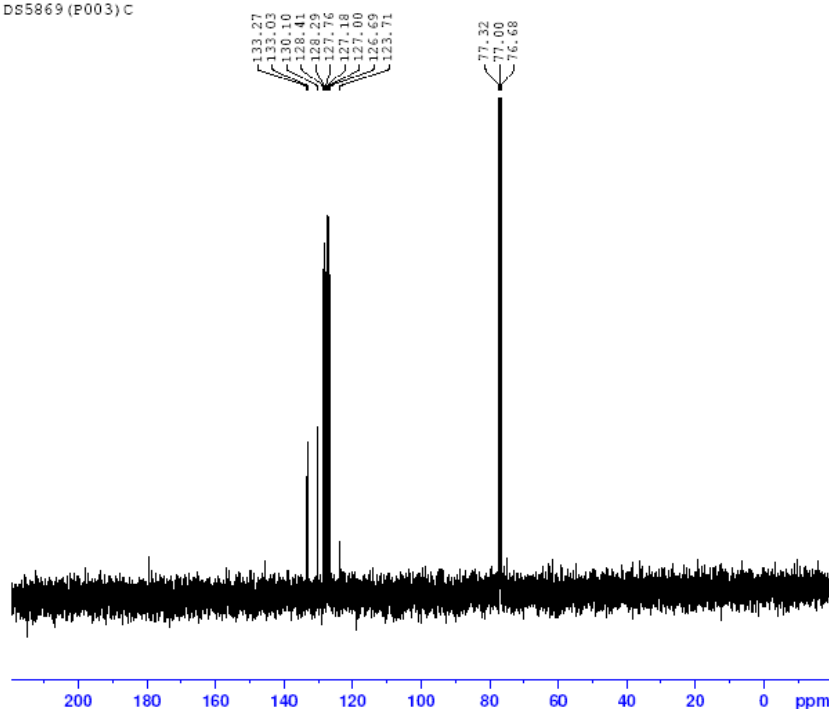
| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
|----------|------------|------|------|-----|-------|---------------|
| 316.0207 | 316.0219 | -1.2 | -3.8 | 9.5 | 5.0 | C14 H7 N S F5 |



Compound 3ai



D55869 (P003) C



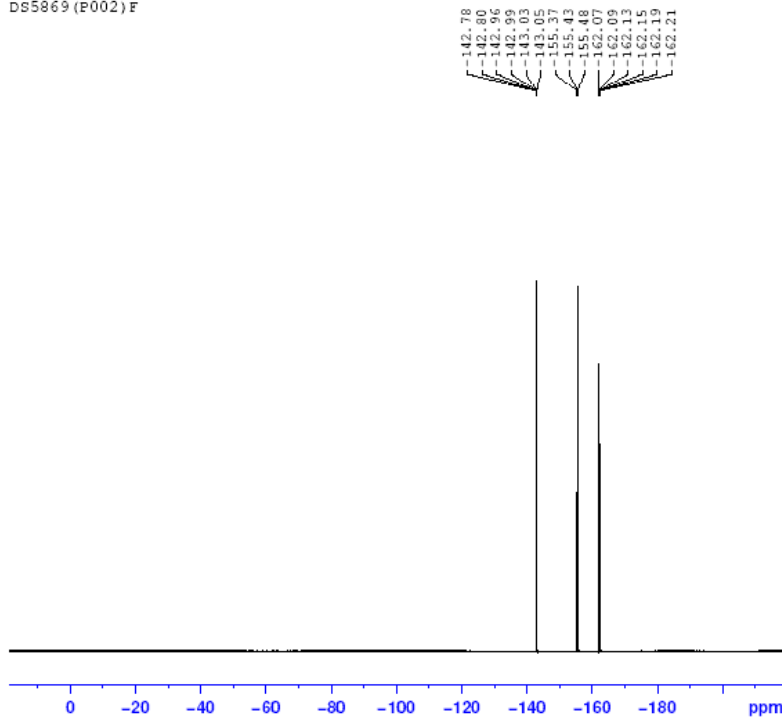
```

NAME      D55869(P003)
EXPNO    3
PROCNO   1
Date_    20110621
Time     18.56
INSTRUM  spect
PROBHD   5 mm F4BBO BB-
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        32
DS        2
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ        1.3631988 sec
RG        203
DW        20.800 usec
DE        6.50 usec
TE        299.1 K
D1        2.0000000 sec
D11       0.0300000 sec
TD0       1

===== CHANNEL f1 =====
NUC1      13C
P1        9.50 usec
PL1       -2.00 dB
PL1W      58.52175522 W
SFO1      100.6228298 MHz

===== CHANNEL f2 =====
CFDPFG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2        0.00 dB
PL12       15.00 dB
PL13       15.00 dB
PL12W     11.88122272 W
PL12W     0.37571725 W
PL13W     0.37571725 W
SFO2      400.1316005 MHz
SI         32768
SF        100.6127709 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```

D55869 (P002) F

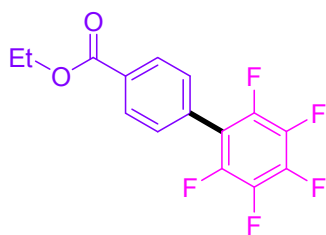


```

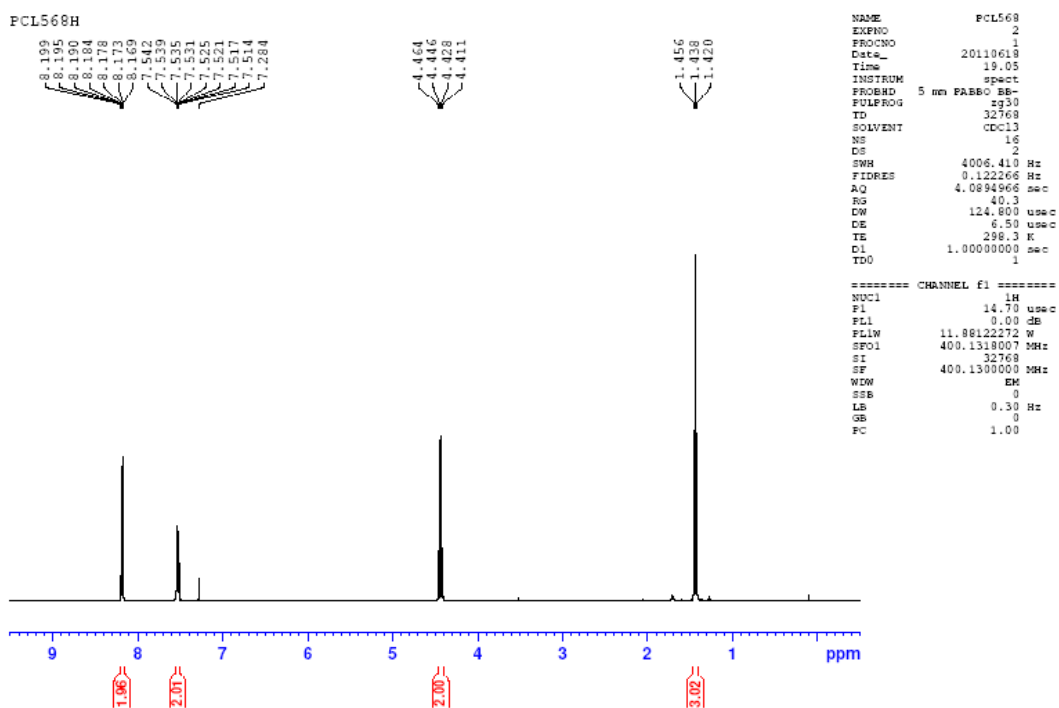
NAME      D55869(P003)
EXPNO    1
PROCNO   1
Date_    20110621
Time     18.50
INSTRUM  spect
PROBHD   5 mm F4BBO BB-
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        4
SWH      89285.711 Hz
FIDRES   0.691196 Hz
AQ        0.7340532 sec
RG        203
DW        5.600 usec
DE        6.50 usec
TE        298.2 K
D1        1.0000000 sec
D11       0.0300000 sec
D12       0.0002000 sec
TD0       1

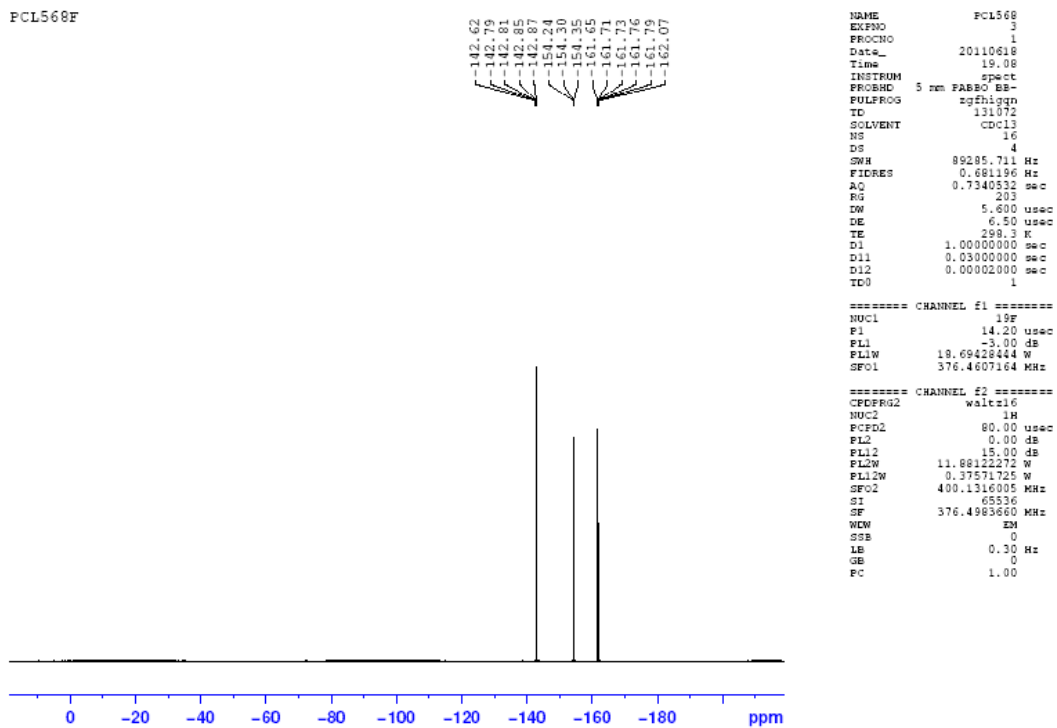
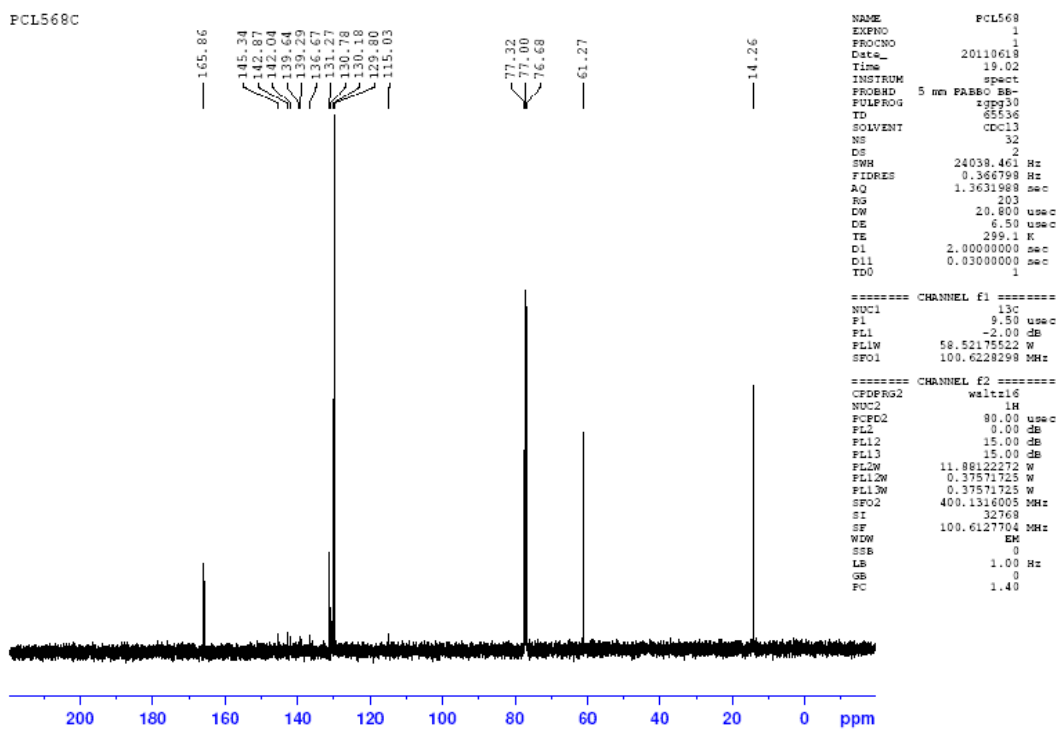
===== CHANNEL f1 =====
NUC1      19F
P1        14.20 usec
PL1       -3.00 dB
PL1W      18.69428444 W
SFO1      376.4607164 MHz

===== CHANNEL f2 =====
CFDPFG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2        0.00 dB
PL12       15.00 dB
PL13       15.00 dB
PL12W     11.88122272 W
PL12W     0.37571725 W
PL13W     0.37571725 W
SFO2      400.1316005 MHz
SI         65536
SF        376.4993660 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```

Compound 3aj





Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

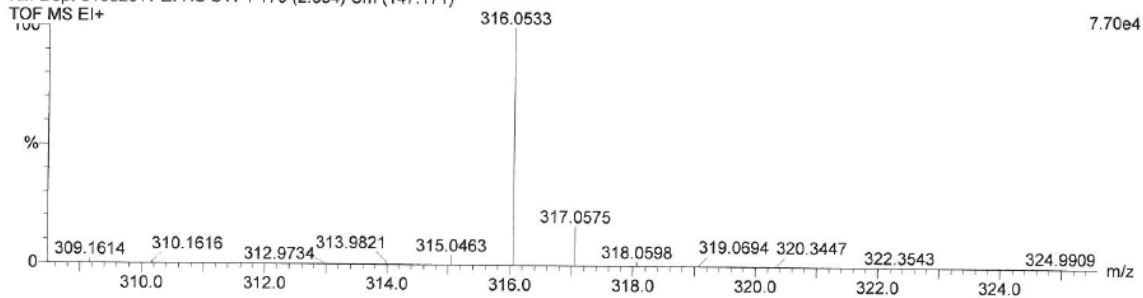
55 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

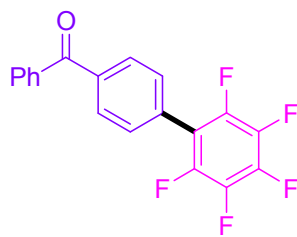
C: 0-15 H: 0-9 O: 0-2 F: 0-5 Na: 0-1 39K: 0-1

Kin-Dept-31082011 EI HS S11 1 170 (2.834) Cm (147:171)

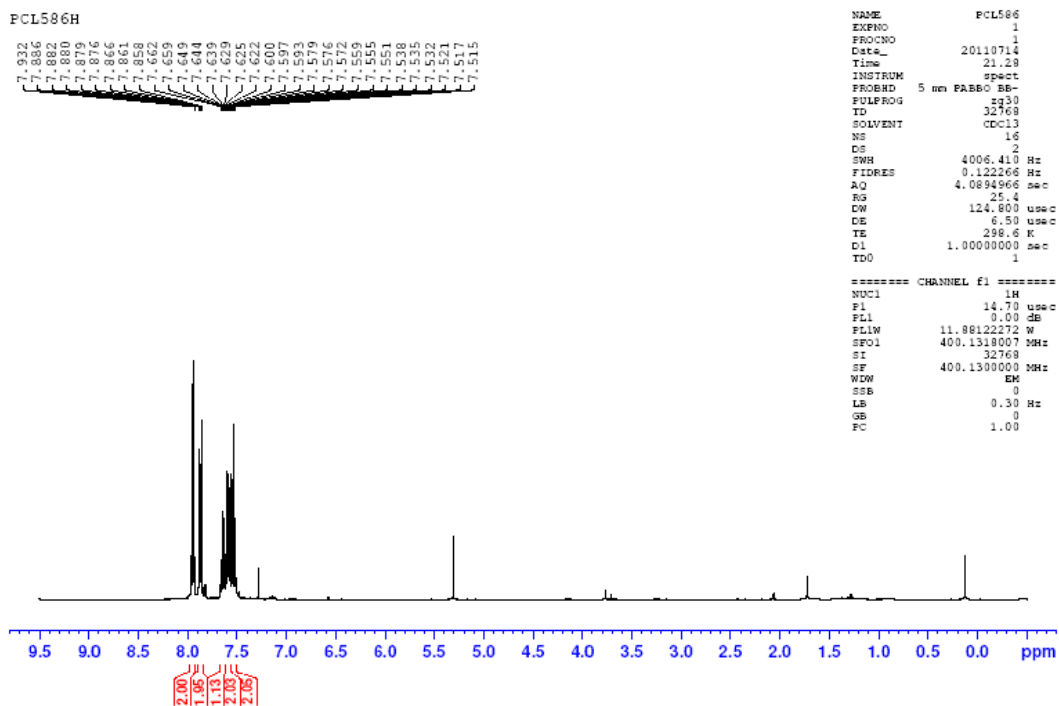
TOF MS EI+

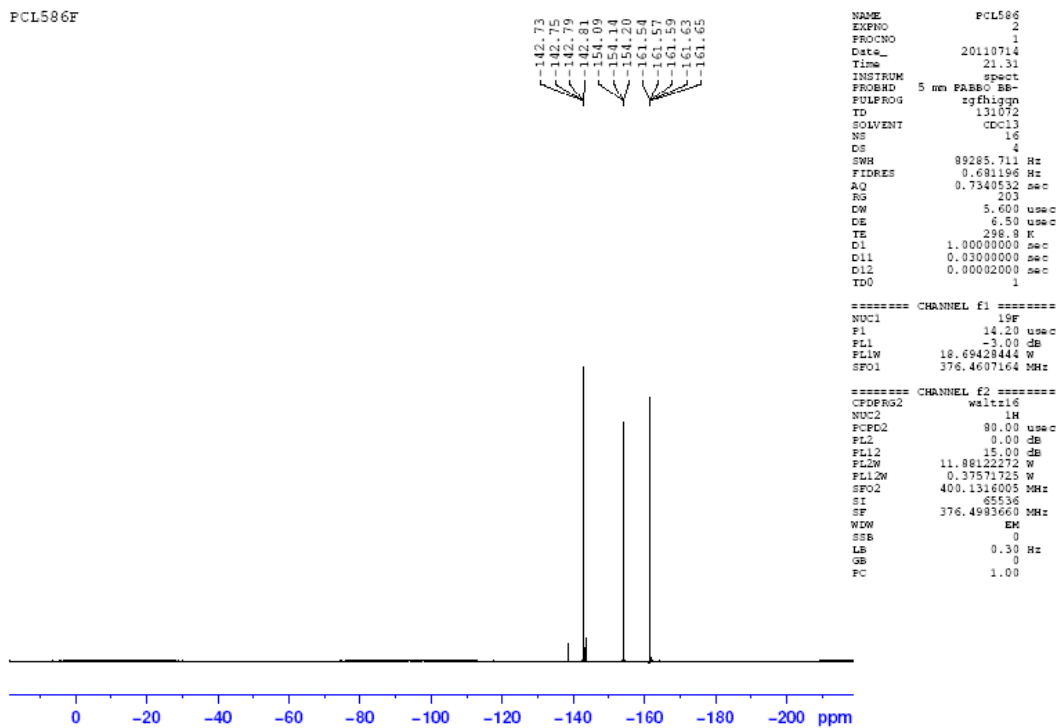
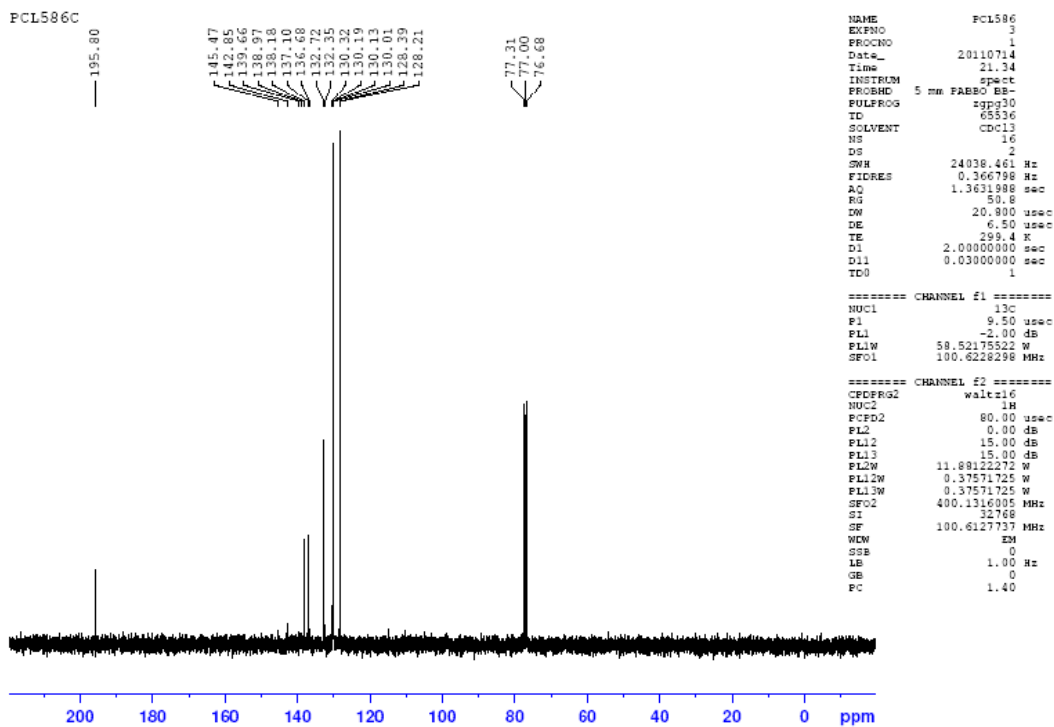


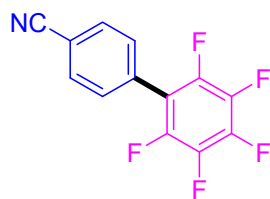
| | | | | | | |
|----------|------------|-----|-----|------|-------|--------------|
| Minimum: | | | | -1.5 | | |
| Maximum: | | 5.0 | 5.0 | 50.0 | | |
| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
| 316.0533 | 316.0523 | 1.0 | 3.2 | 9.0 | 9.4 | C15 H9 O2 F5 |



Compound 3ak

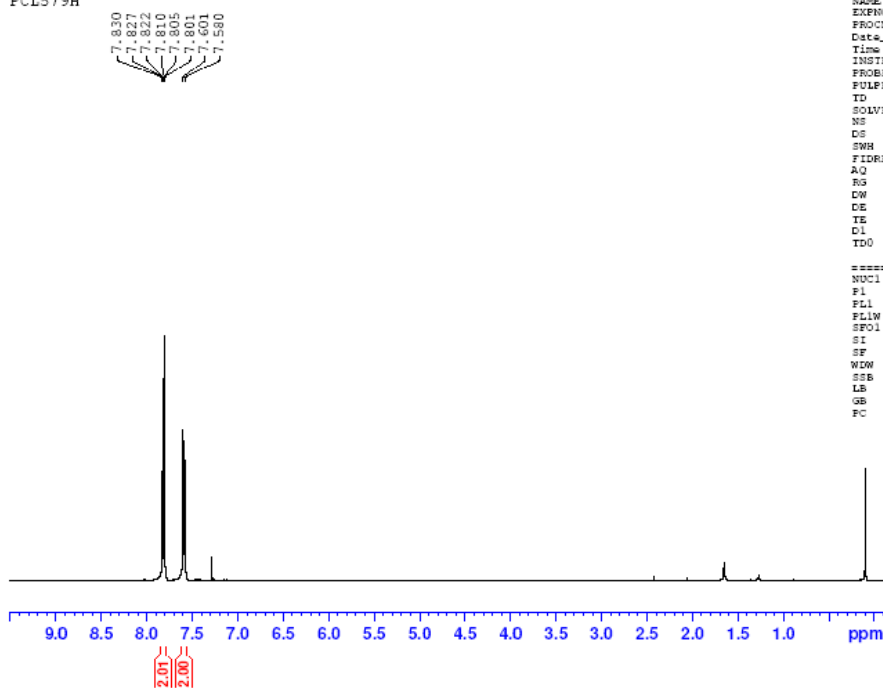






Compound 3al

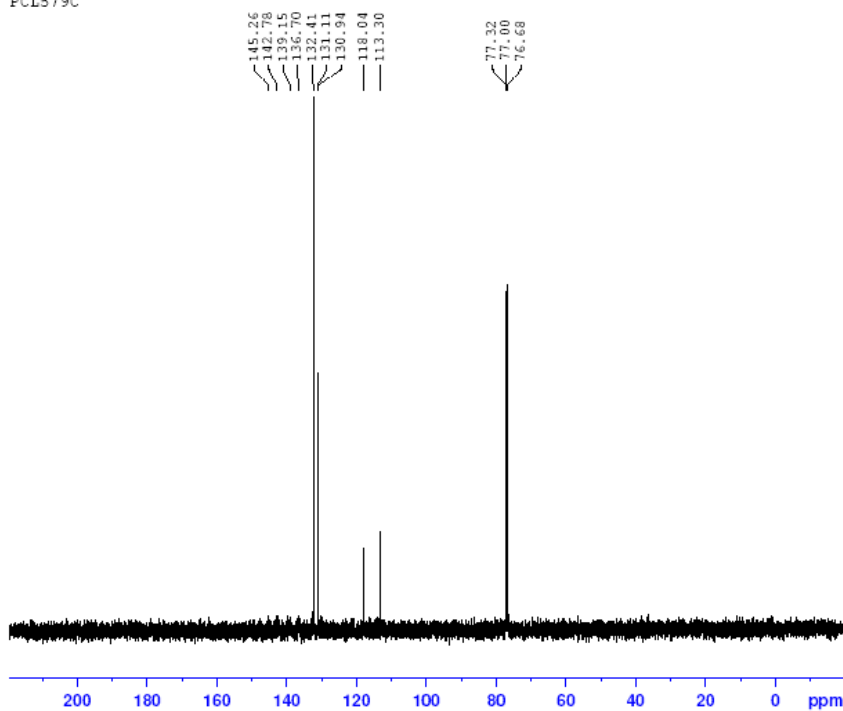
PCL579H



```
NAME          PCL579
EXPNO         2
PROCNO        1
Date_         20110630
Time          17.45
INSTRUM       spect
PROBHD        5 mm F4BBO BB-
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            16
DS            2
SWH           4006.410 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG            45.2
DW            124.800 usec
DE            6.50 usec
TE            298.2 K
D1            1.00000000 sec
D11           1
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.70 usec
PL1           0.00 dB
PL1W          11.88122272 W
SFO1          400.1318007 MHz
SI            32768
SF            400.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
```

PCL579C



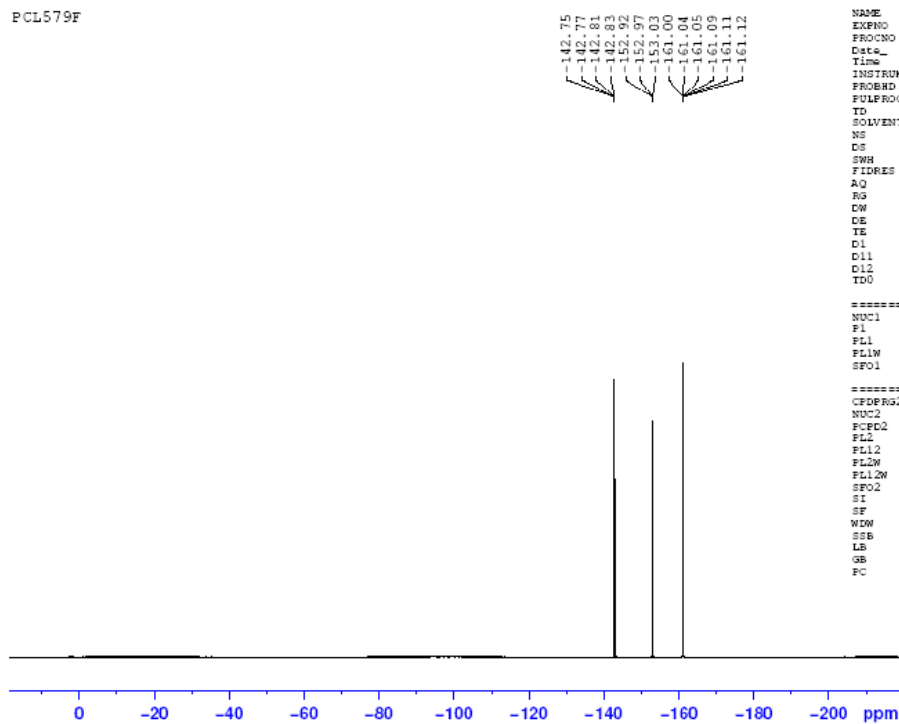
```

NAME          PCL579
EXPNO         3
PROCNO        1
Date_         20110630
Time_         17.49
INSTRUM       spect
PROBHD        5 mm FAPBBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           24039.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            203
DW            20.800 usec
DE            6.50 usec
TE            298.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -2.00 dB
PL1W          58.52175522 W
SF01          100.6228298 MHz

===== CHANNEL f2 =====
CFDPRG2       waltz16
NUC2          1H
PCPD2         90.00 usec
PL2           0.00 dB
PL12          15.00 dB
PL13          15.00 dB
PL2W          11.98122272 W
PL12W         0.37571725 W
PL13W         0.37571725 W
SF02          400.1316005 MHz
SI            32768
SF            100.6127736 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

PCL579F

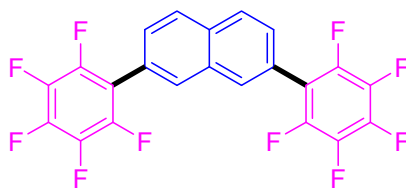


```

NAME          PCL579
EXPNO         1
PROCNO        1
Date_         20110630
Time_         17.42
INSTRUM       spect
PROBHD        5 mm FAPBBO BB-
PULPROG       zgfg30
TD            131072
SOLVENT       CDCl3
NS            16
DS            4
SWH           99285.711 Hz
FIDRES        0.691196 Hz
AQ            0.7340532 sec
RG            203
DW            5.600 usec
DE            6.50 usec
TE            298.3 K
D1            1.00000000 sec
D11           0.03000000 sec
D12           0.00020000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          19F
P1            14.20 usec
PL1           -3.00 dB
PL1W          18.69428444 W
SF01          376.4607164 MHz

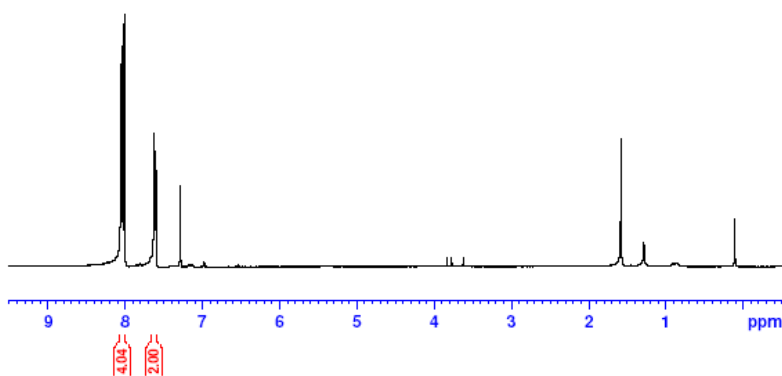
===== CHANNEL f2 =====
CFDPRG2       waltz16
NUC2          1H
PCPD2         90.00 usec
PL2           0.00 dB
PL12          15.00 dB
PL13          15.00 dB
PL2W          11.98122272 W
PL12W         0.37571725 W
PL13W         0.37571725 W
SF02          400.1316005 MHz
SI            65536
SF            376.4993660 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```



Compound 3am

FCL597H

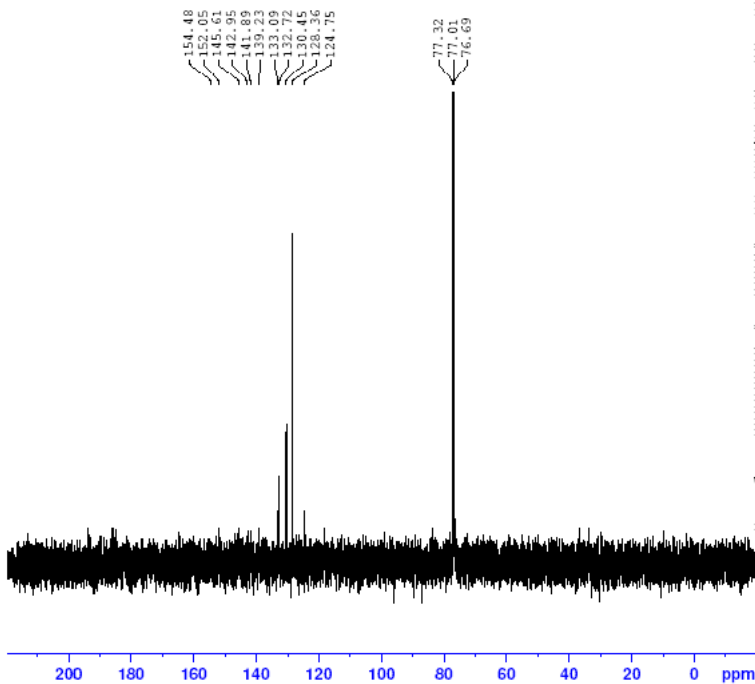
8.058
8.036
8.011
7.627
7.624
7.605
7.603



```
NAME          FCL597
EXPNO         6
PROCNO        1
Date_         20110818
Time         12.05
INSTRUM       spect
PROBHD        5 mm FASBO BB-
PULPROG       zg30
TD            32768
SOLVENT       cdcl3
NS            16
DS            2
SWH           4006.410 Hz
FIDRES       0.122266 Hz
AQ           4.0394966 sec
RG            128
DW           124.800 usec
DE            6.50 usec
TE           297.8 K
D1            1.00000000 sec
TD0           1
```

```
===== CHANNEL f1 =====
NUC1          1H
P1            14.70 usec
PL1           0.00 dB
PL12         11.68122272 W
SFO1         400.1316007 MHz
SI            32768
SF           400.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
```


PCL597C



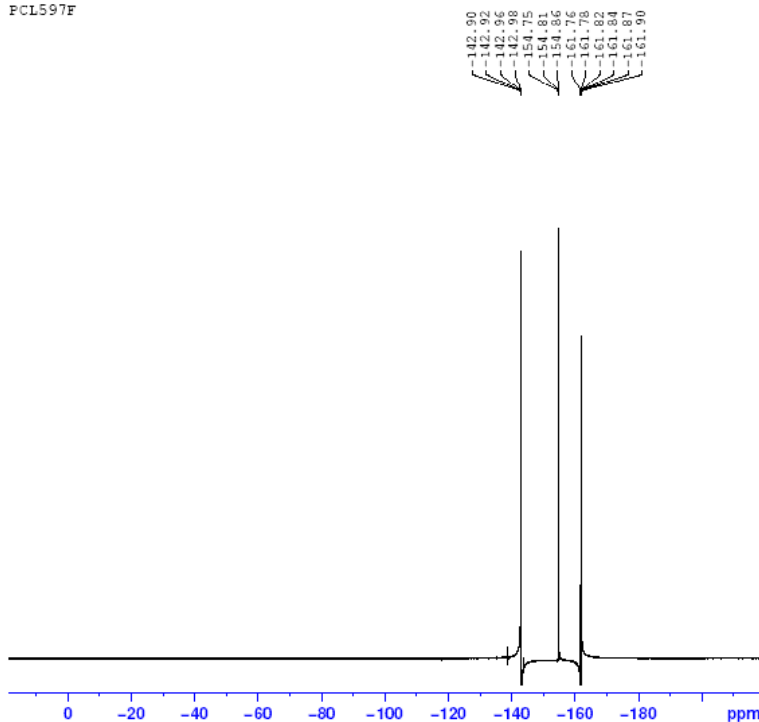
```

NAME          PCL597
EXPNO         8
PROCNO        1
Data_         20110818
Time          12.11
INSTRUM       spect
PROBHD        5 mm PABBO EB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            101
DW            20.800 usec
DE            6.50 usec
TE            299.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -2.00 dB
PL1W          58.52175522 W
SFOL          100.6229298 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           0.00 dB
PL12         15.00 dB
PL13         15.00 dB
PL2W         11.88122272 W
PL12W        0.37571725 W
PL13W        0.37571725 W
SFO2         400.1316005 MHz
SI            32768
SF           100.6127690 MHz
WEN          EM
SFB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

PCL597F



```

NAME          PCL597
EXPNO         7
PROCNO        1
Data_         20110818
Time          12.07
INSTRUM       spect
PROBHD        5 mm PABBO EB-
PULPROG       zgHigpm
TD            131072
SOLVENT       CDCl3
NS            16
DS            4
SWH           89285.711 Hz
FIDRES        0.3681196 Hz
AQ            0.7340532 sec
RG            203
DW            5.600 usec
DE            6.50 usec
TE            299.0 K
D1            1.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          19F
P1            14.20 usec
PL1           -3.00 dB
PL1W          18.69429444 W
SFOL          376.4607164 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           0.00 dB
PL12         15.00 dB
PL13         15.00 dB
PL2W         11.88122272 W
PL12W        0.37571725 W
PL13W        0.37571725 W
SFO2         400.1316005 MHz
SI            65536
SF           376.4983660 MHz
WEN          EM
SFB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

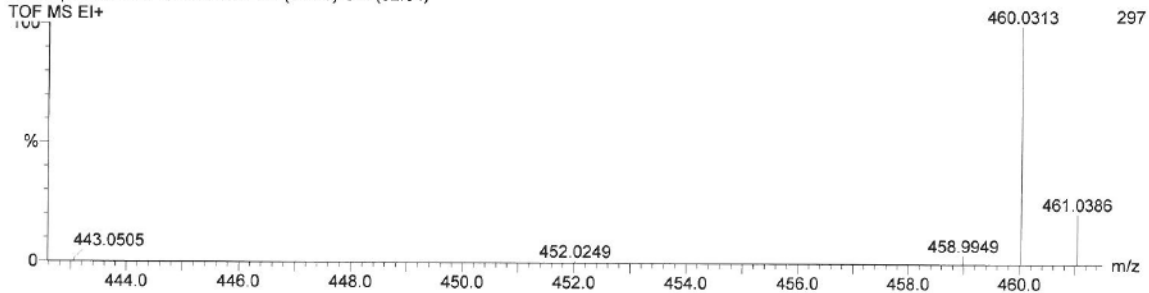
40 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

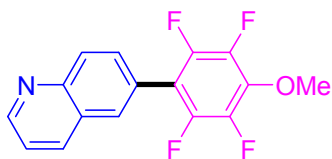
C: 0-22 H: 0-15 F: 0-10 Na: 0-1 39K: 0-1

Kin-Dept-31082011 EI HS S14 1 54 (0.900) Cm (52:54)

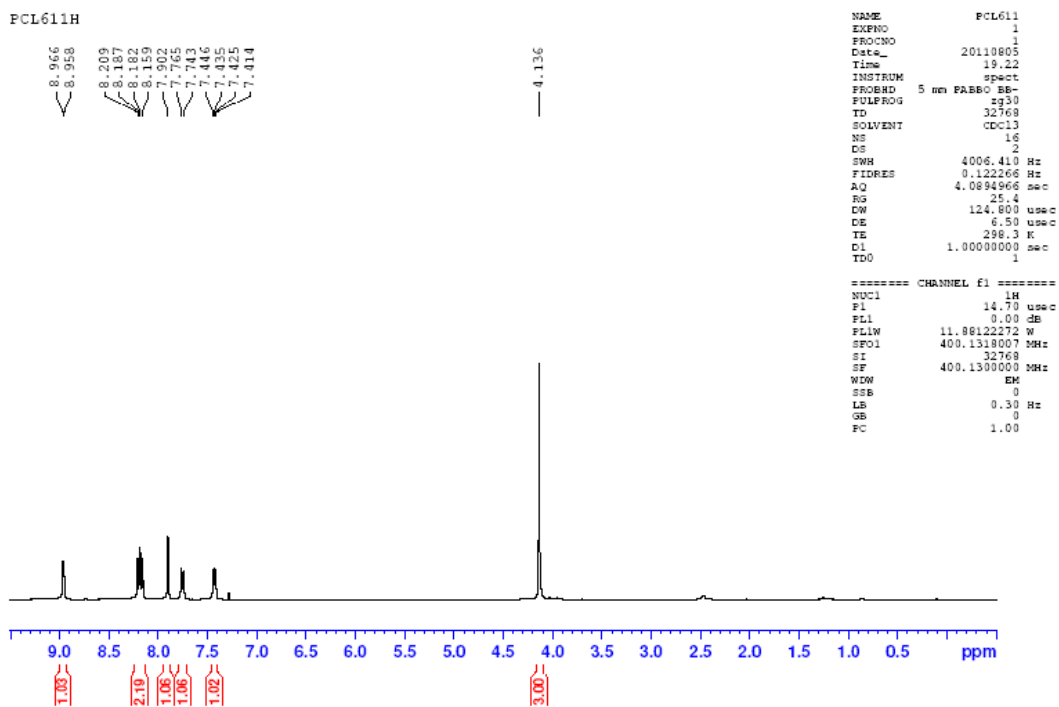
TOF MS EI+



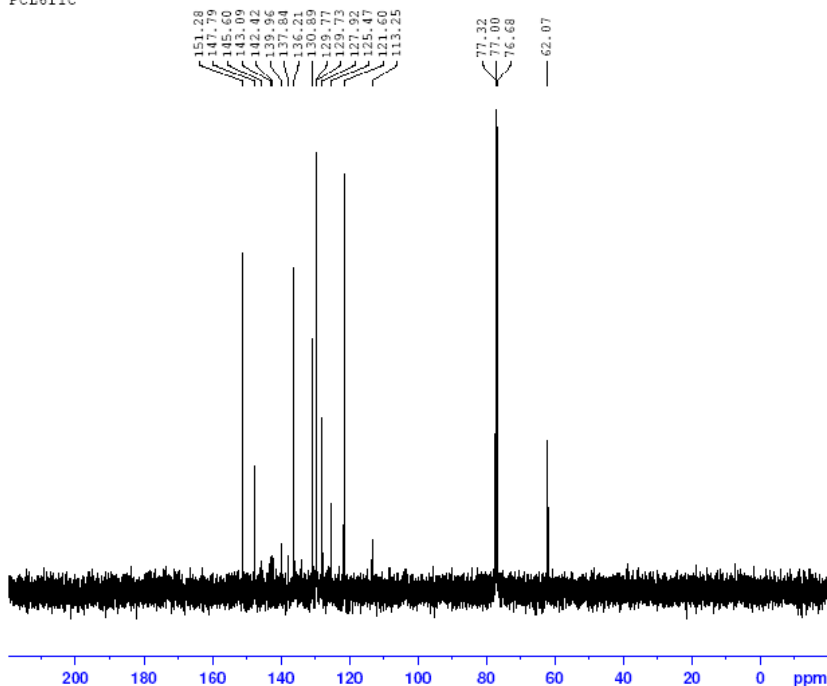
| | | | | | | |
|----------|------------|-----|-----|------|-----------|------------|
| Minimum: | | | | -1.5 | | |
| Maximum: | | 5.0 | 5.0 | 50.0 | | |
| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
| 460.0313 | 460.0310 | 0.3 | 0.7 | 15.0 | 2773035.5 | C22 H6 F10 |



Compound 3bc



PCL611C



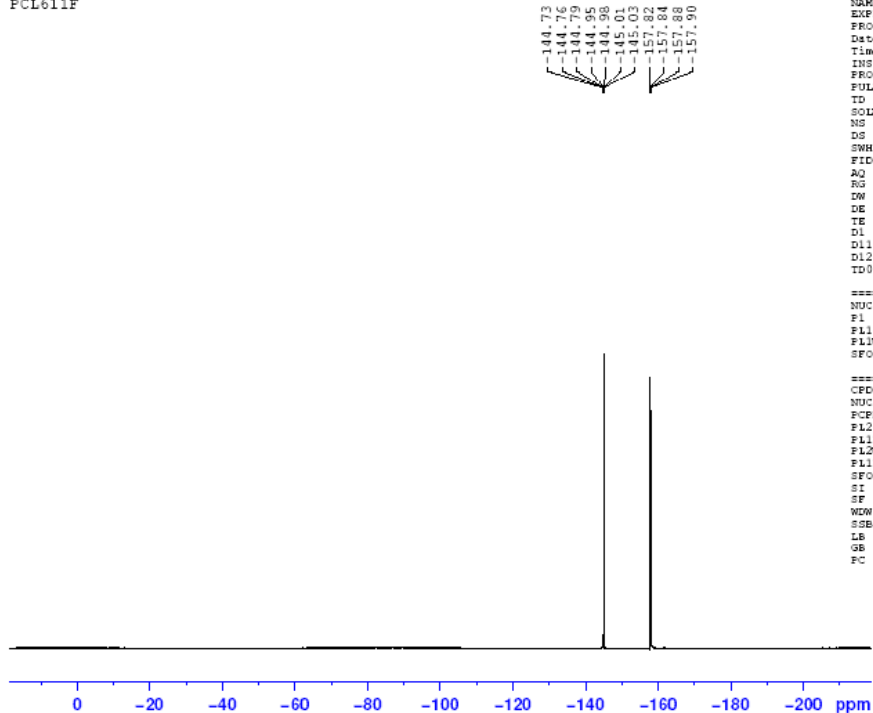
```

NAME          PCL611
EXPNO         3
PROCNO       20110805
Date_        19.32
INSTRUM      spect
PROBHD       5 mm F4BBO BB-
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           16
DS           2
SWH          24038.461 Hz
FIDRES       0.366798 Hz
AQ           1.3631988 sec
RG           50.8
DW           20.800 usec
DE           6.50 usec
TE           299.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0          1

===== CHANNEL f1 =====
NUC1         13C
P1           8.00 usec
PL1         -2.00 dB
PL1W        58.52175522 W
SFO1        100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2     waitt16
NUC2        1H
PCPD2       80.00 usec
PL2         0.00 dB
PL12        15.00 dB
PL13        15.00 dB
PL1W        11.88122272 W
PL12W       0.37571725 W
SFO2        400.1316005 MHz
SI          32768
SF          100.6127759 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
    
```

PCL611F



```

NAME          PCL611
EXPNO         2
PROCNO       20110805
Date_        19.26
INSTRUM      spect
PROBHD       5 mm F4BBO BB-
PULPROG      zgfg13qqa
TD           131072
SOLVENT      CDCl3
NS           16
DS           4
SWH          89295.711 Hz
FIDRES       0.681196 Hz
AQ           0.7340532 sec
RG           203
DW           5.600 usec
DE           6.50 usec
TE           298.5 K
D1           1.00000000 sec
D11          0.03000000 sec
D12          0.00020000 sec
TD0          1

===== CHANNEL f1 =====
NUC1         15P
P1           14.20 usec
PL1         -3.00 dB
PL1W       19.69428444 W
SFO1       376.4607164 MHz

===== CHANNEL f2 =====
CPDPRG2     waitt16
NUC2        1H
PCPD2       80.00 usec
PL2         0.00 dB
PL12        15.00 dB
PL13        15.00 dB
PL1W        11.88122272 W
PL12W       0.37571725 W
SFO2        400.1316005 MHz
SI          65536
SF          376.4983669 MHz
WDW         EM
SSB         0
LB          0.30 Hz
GB          0
PC          1.00
    
```

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 20.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions

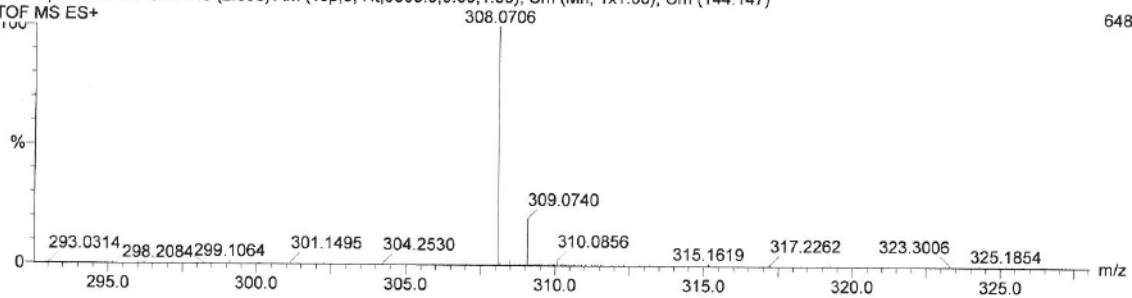
345 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-16 H: 0-10 N: 0-2 O: 0-2 F: 0-5 Na: 0-1 S: 0-1 39K: 0-1

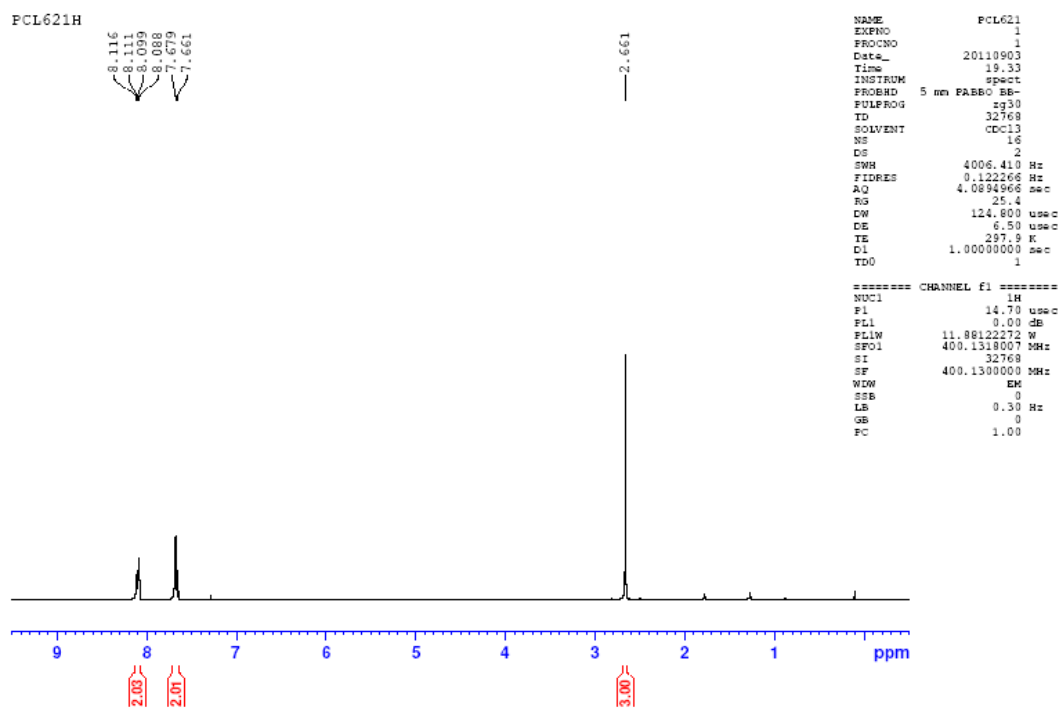
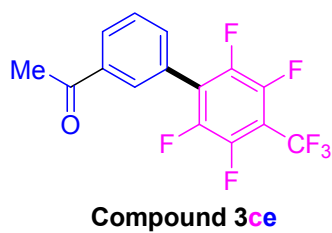
Kin-Dept-25082011 S12 145 (2.698) AM (Top,5, Ht,5000.0,0.00,1.00); Sm (Mn, 1x1.00); Cm (144:147)

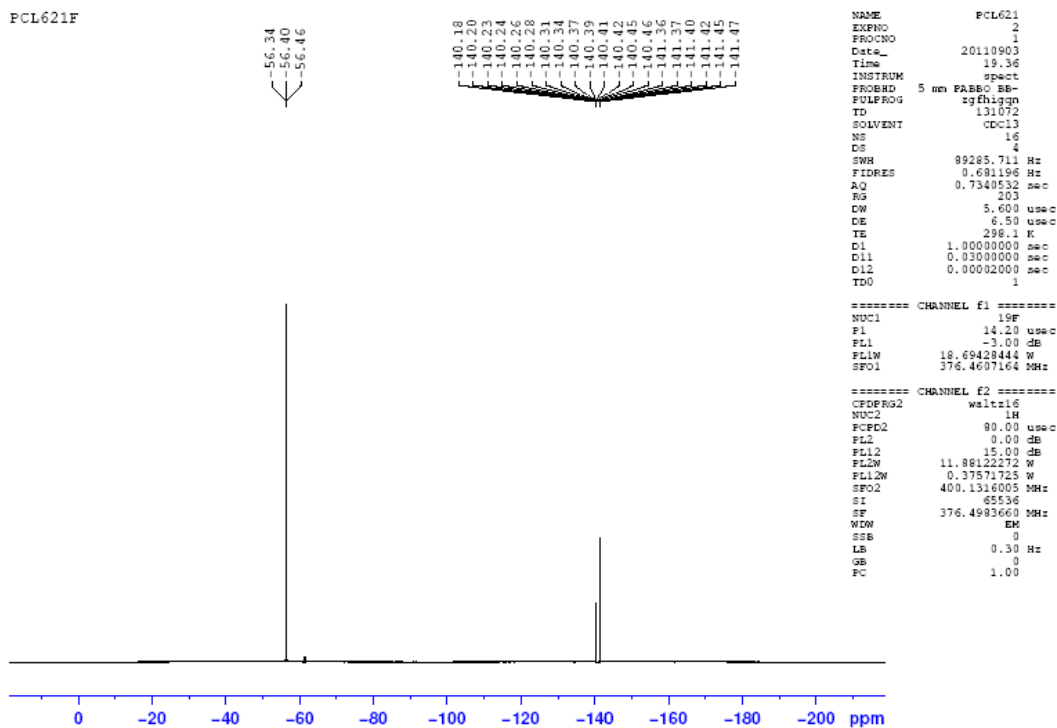
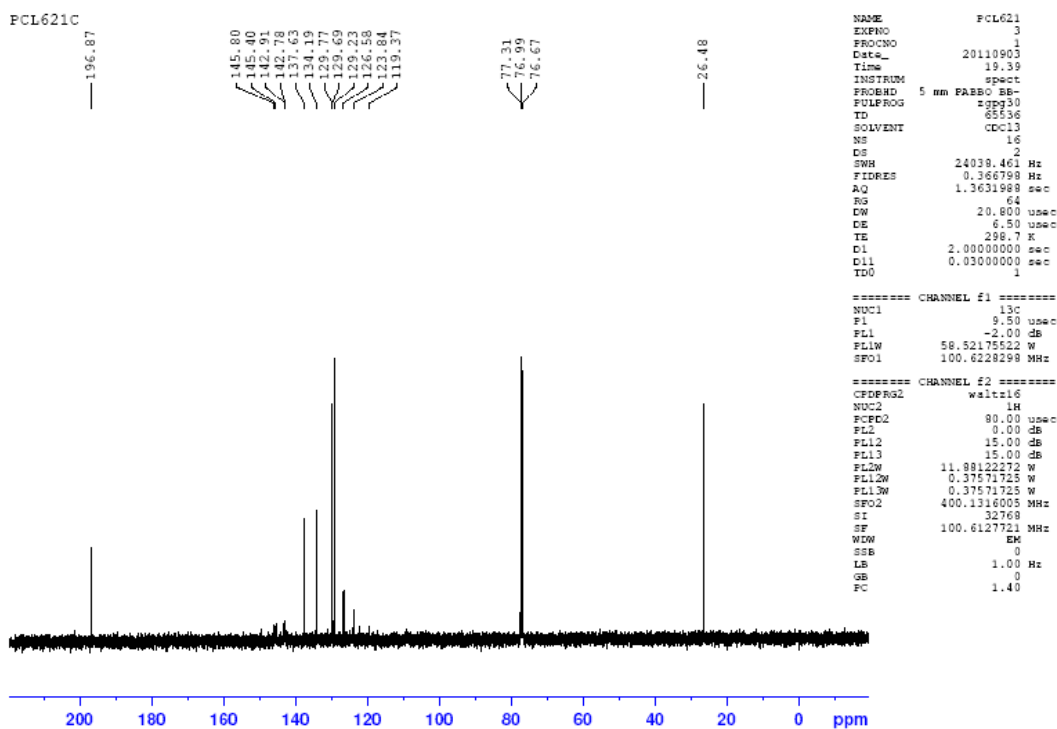
TOF MS ES+



Minimum: -1.5
Maximum: 50.0

| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
|----------|------------|-----|-----|------|-------|----------------|
| 308.0706 | 308.0699 | 0.7 | 2.3 | 10.5 | 0.3 | C16 H10 N 0 F4 |





Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

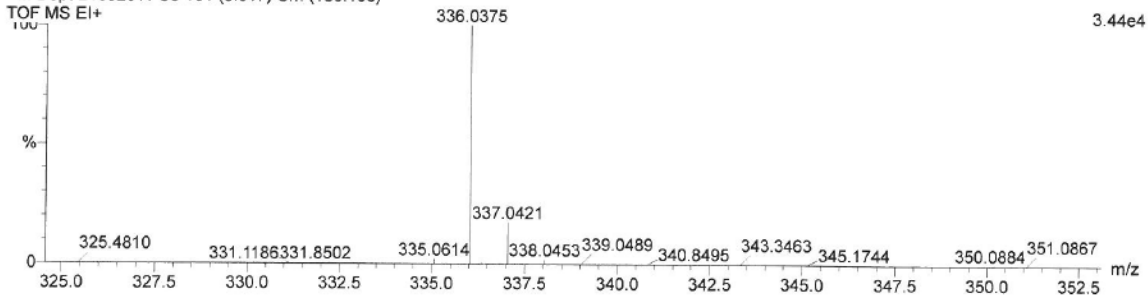
116 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-15 H: 0-7 O: 0-4 F: 0-7 Na: 0-1 39K: 0-1

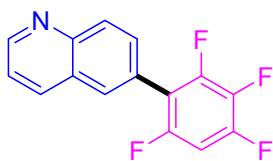
Kin-Dept-21092011 S5 181 (3.017) Cm (180:196)

TOF MS EI+



Minimum: -1.5
Maximum: 5.0 5.0 50.0

| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
|----------|------------|------|------|-----|-------|-------------|
| 336.0375 | 336.0385 | -1.0 | -3.0 | 9.0 | 3.4 | C15 H7 O F7 |



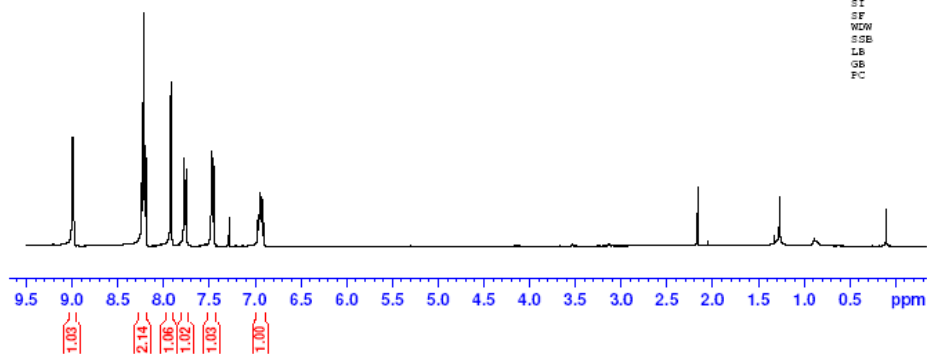
Compound 3dc

PCL626aH

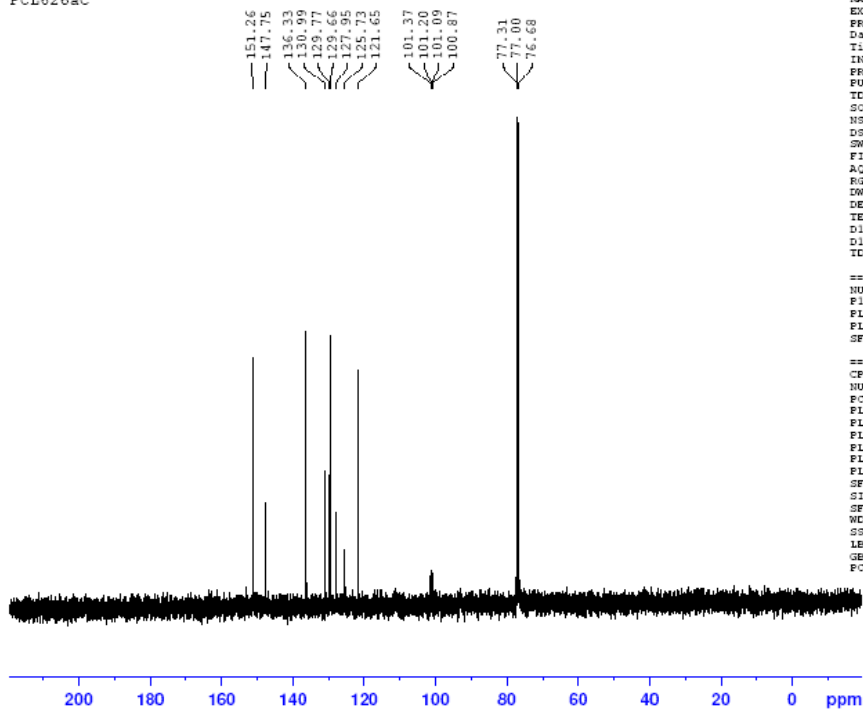
8.095
8.086
8.234
8.214
8.197
7.920
7.773
7.754
7.751
7.480
7.470
7.460
7.449
6.972
6.966
6.957
6.948
6.942
6.932
6.926
6.917
6.908
6.902

```
NAME          PCL626a
EXPNO         1
PROCNO        1
Date_         20110904
Time          19.45
INSTRUM       spect
PROBHD        5 mm FAPBO BB-
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            16
DS            2
SWH           4006.410 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG            45.2
DN            124.800 usec
DE            6.50 usec
TE            298.1 K
D1            1.0000000 sec
TDO           1
```

```
===== CHANNEL f1 =====
NUC1          1H
P1            14.70 usec
PL1           0.00 dB
PL1W          11.88122272 W
SFO1          400.1318007 MHz
SI            32768
SF            400.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
```



PCL626aC



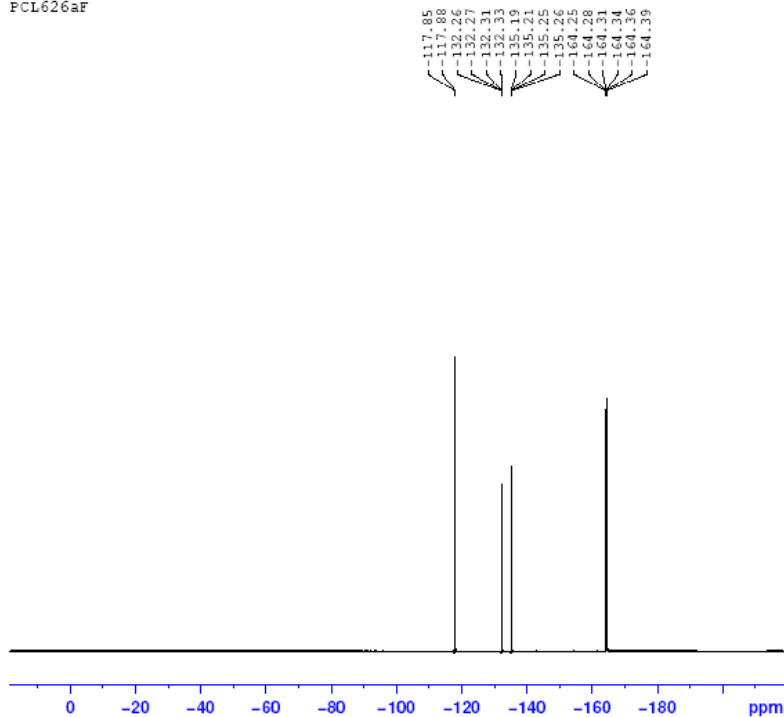
```

NAME          PCL626a
EXPNO         2
PROCNO        1
Date_         20110904
Time          19.47
INSTRUM       spect
PROBHD        5 mm FAPBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631998 sec
RG            114
DW            20.800 usec
DE            6.50 usec
TE            298.6 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -2.00 dB
PL1W          59.5217532 W
SFO1          100.6228258 MHz

===== CHANNEL f2 =====
CFDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           0.00 dB
PL12         15.00 dB
PL13         15.00 dB
PL12W        11.88122272 W
PL12W        0.37571725 W
PL13W        0.37571725 W
SFO2          400.1316005 MHz
SI            32768
SF            100.6127738 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

PCL626aF



```

NAME          PCL626a
EXPNO         3
PROCNO        1
Date_         20110904
Time          19.50
INSTRUM       spect
PROBHD        5 mm FAPBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            4
SWH           89285.711 Hz
FIDRES        0.681196 Hz
AQ            0.7340532 sec
RG            203
DW            5.600 usec
DE            6.50 usec
TE            298.2 K
D1            1.0000000 sec
D11           0.0300000 sec
D12           0.0002000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          19F
P1            14.20 usec
PL1           -3.00 dB
PL1W          18.69428444 W
SFO1          376.4607164 MHz

===== CHANNEL f2 =====
CFDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           0.00 dB
PL12         15.00 dB
PL13         15.00 dB
PL12W        11.88122272 W
PL12W        0.37571725 W
SFO2          400.1316005 MHz
SI            65536
SF            376.4993660 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

Elemental Composition Report

Page 1

Single Mass Analysis (displaying only valid results)

Tolerance = 10.0 PPM / DBE: min = -100.0, max = 1000.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions

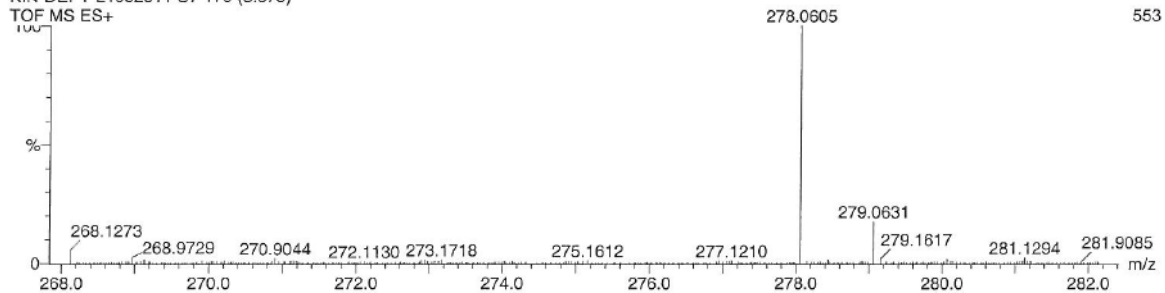
6 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-16 H: 5-10 N: 0-1 F: 0-4

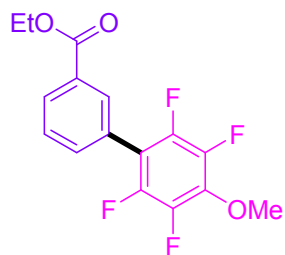
KIN-DEPT-21092011 S7 179 (3.375)

TOF MS ES+



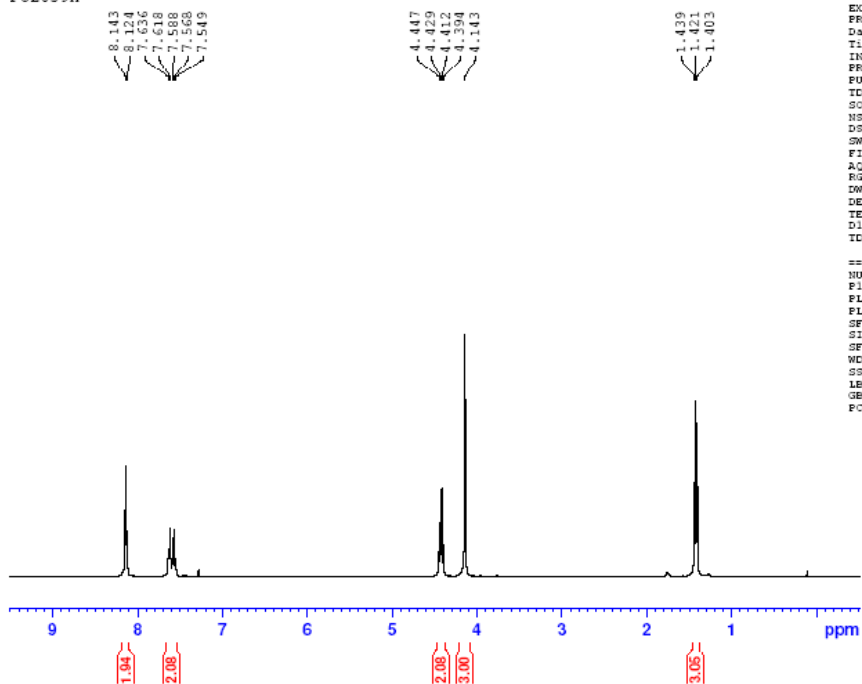
Minimum: -100.0
Maximum: 5.0 10.0 1000.0

| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
|----------|------------|-----|-----|------|-------|-------------|
| 278.0605 | 278.0593 | 1.2 | 4.3 | 10.5 | 0.3 | C15 H8 N F4 |



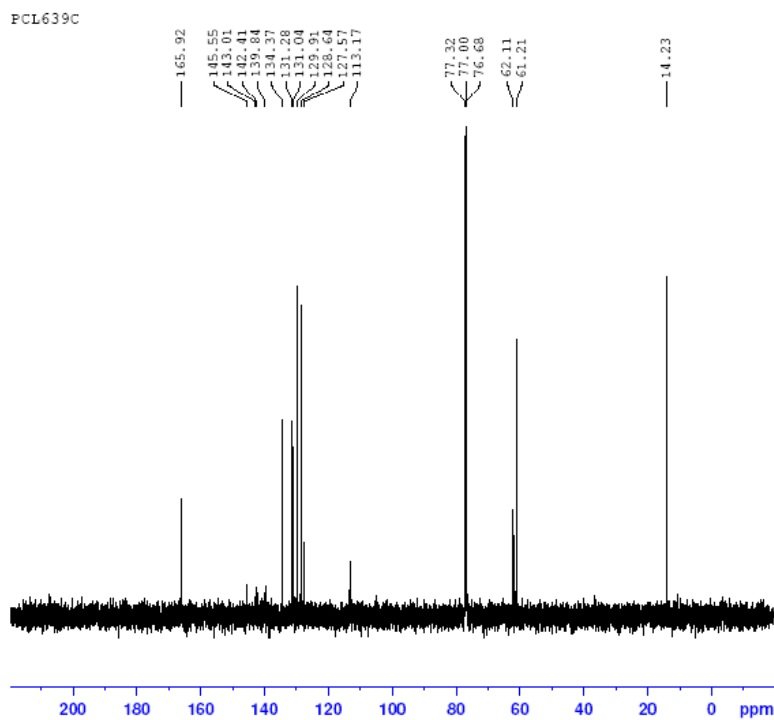
Compound 3bj

PCL639H



```
NAME          PCL639
EXPNO         1
PROCNO        1
Date_         20110906
Time          22.57
INSTRUM       spect
PROBHD        5 mm FAPBBO BB-
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            16
DS            2
SWH           4006.410 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG            25.4
EW           124.800 usec
DE            6.50 usec
TE            299.9 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.70 usec
PL1           0.00 dB
PLLW         11.89122272 W
SFO1         400.1318007 MHz
SI            32768
SF           400.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
```

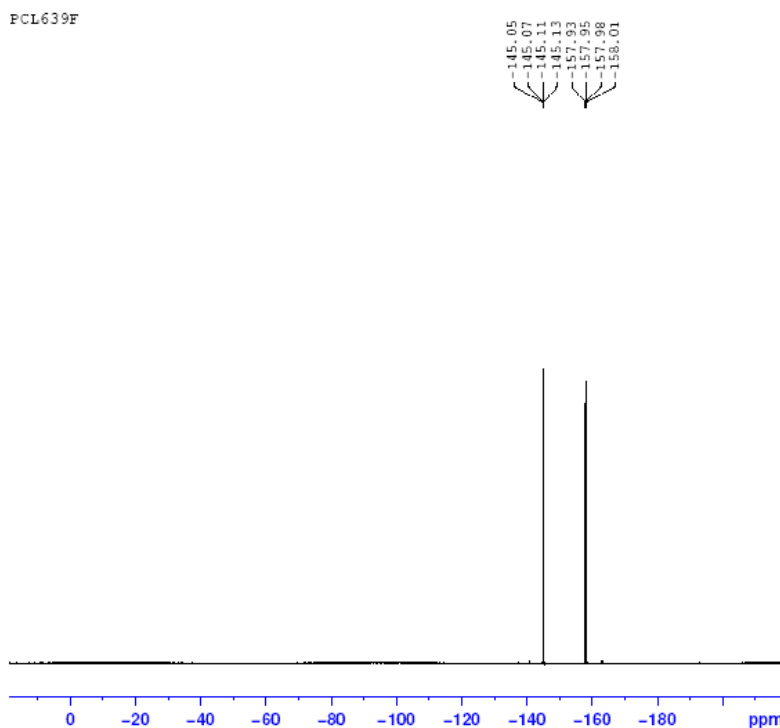


```

NAME          PCL639
EXPNO         2
PROCNO        1
Date_         20110906
Time          22.58
INSTRUM       spect
PROBRD        5 mm F4BBO BB-
PULPROG       zgpg30
TD            85536
SOLVENT       CDCl3
NS            16
DS            2
SWH           24039.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631998 sec
RG            203
LW           20.800 usec
DE            6.50 usec
TE            300.4 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -2.00 dB
PL1W         58.52175522 W
SF01         100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           0.00 dB
PL12         15.00 dB
PL13         15.00 dB
PL2W         11.88122272 W
PL12W        0.37571725 W
PL13W        0.37571725 W
SF02         400.1316005 MHz
SI           32769
SF           100.6127720 MHz
WDW          EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```



```

NAME          PCL639
EXPNO         3
PROCNO        1
Date_         20110906
Time          23.01
INSTRUM       spect
PROBRD        5 mm F4BBO BB-
PULPROG       zgpg30
TD            131072
SOLVENT       CDCl3
NS            16
DS            4
SWH           89285.711 Hz
FIDRES        0.681196 Hz
AQ            0.7349532 sec
RG            203
LW            5.600 usec
DE            6.50 usec
TE            299.9 K
D1            1.0000000 sec
D11           0.0300000 sec
D12           0.0002000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          19F
P1            14.20 usec
PL1           -3.00 dB
PL1W         18.49428444 W
SF01         376.4607164 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           0.00 dB
PL12         15.00 dB
PL13         15.00 dB
PL2W         11.88122272 W
PL12W        0.37571725 W
SF02         400.1316005 MHz
SI           85536
SF           376.4983660 MHz
WDW          EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

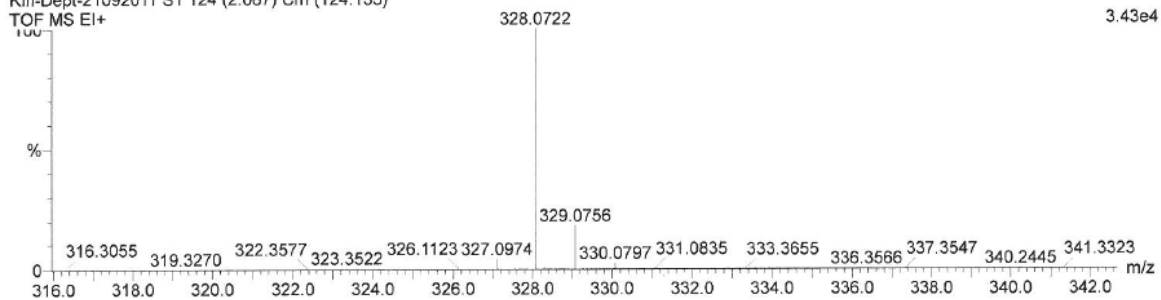
78 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-16 H: 0-12 O: 0-4 Na: 0-1 39K: 0-1 F: 0-4

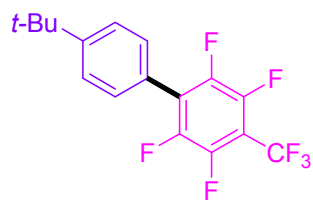
Kin-Dept-21092011 S1 124 (2.067) Cm (124:135)

TOF MS EI+

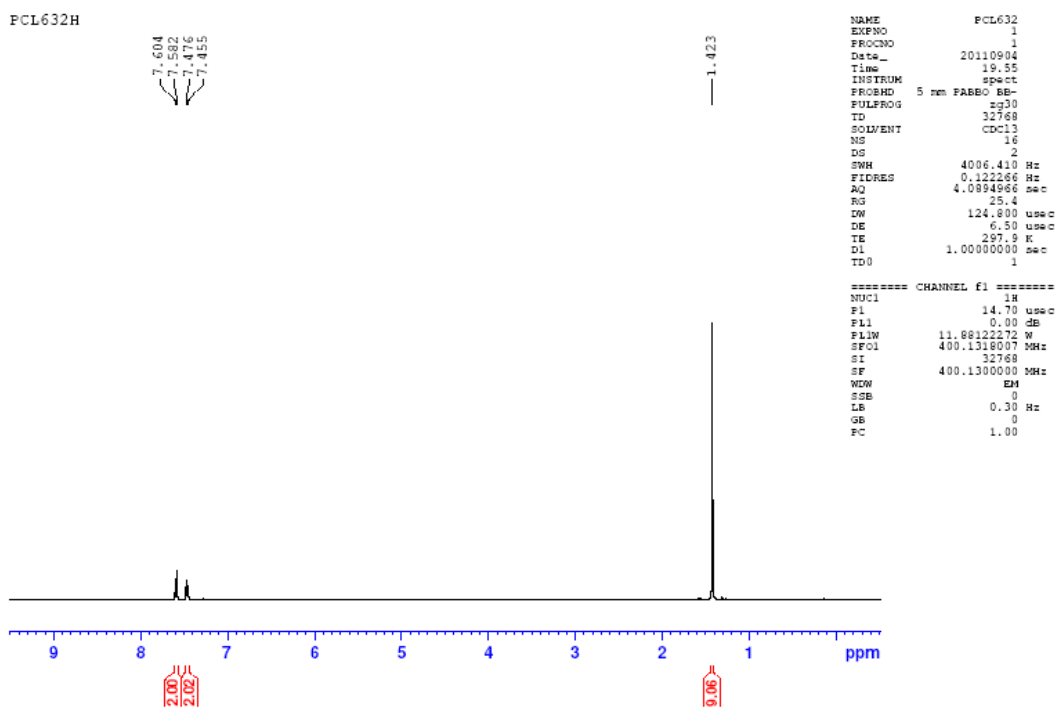


Minimum: -1.5
Maximum: 5.0 5.0 50.0

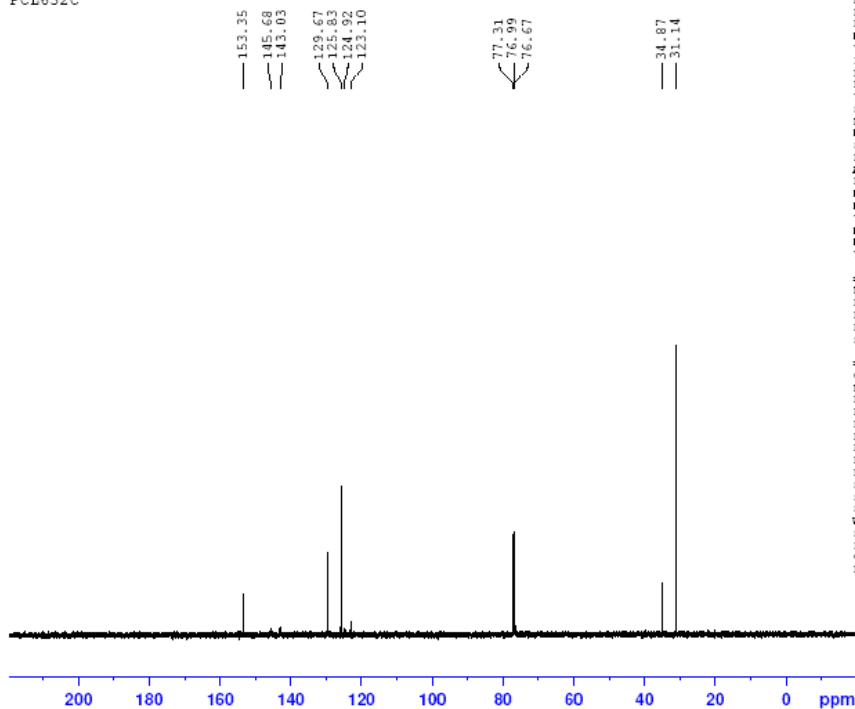
| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
|----------|------------|------|------|-----|-------|---------------|
| 328.0722 | 328.0723 | -0.1 | -0.3 | 9.0 | 5.0 | C16 H12 O3 F4 |



Compound 3ca



PCL632C



```

NAME          PCL632
EXPNO         3
PROCNO        1
Date_         20110904
Time         19.59
INSTRUM       spect
PROBHD        5 mm FAPBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            71.8
CW            20.800 usec
DE            6.50 usec
TE            298.5 K
D1            2.0000000 sec
d11           0.0300000 sec
TD0           1
    
```

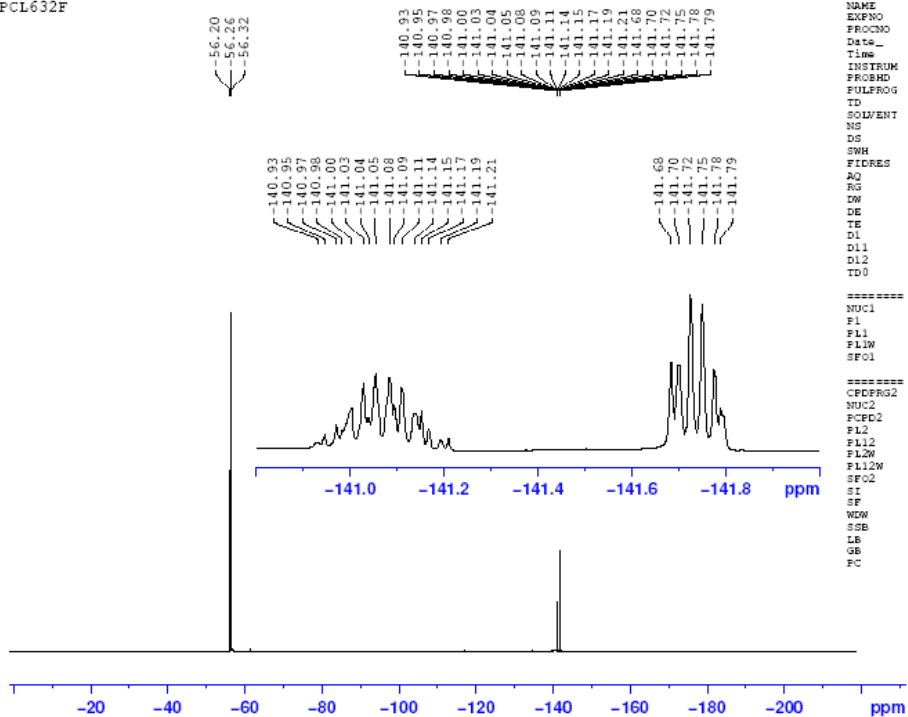
```

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -2.00 dB
PL1W          58.52175522 W
SFO1          100.6228299 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12          15.00 dB
PL13          15.00 dB
PL1W          11.88122272 W
PL12W         0.37571725 W
PL13W         0.37571725 W
SFO2          400.1316005 MHz
SI            32769
SF            100.6127692 MHz
WZM           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

PCL632F



```

NAME          PCL632
EXPNO         2
PROCNO        1
Date_         20110904
Time         19.56
INSTRUM       spect
PROBHD        5 mm FAPBO BB-
PULPROG       zgpg30
TD            131072
SOLVENT       CDCl3
NS            16
DS            4
SWH           89285.711 Hz
FIDRES        0.681196 Hz
AQ            0.7340532 sec
RG            2.03
CW            5.600 usec
DE            6.50 usec
TE            298.0 K
D1            1.0000000 sec
d11           0.0300000 sec
D12           0.0000200 sec
TD0           1
    
```

```

===== CHANNEL f1 =====
NUC1          19F
P1            14.20 usec
PL1           -3.00 dB
PL1W          18.69428444 W
SFO1          376.4607164 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12          15.00 dB
PL13          15.00 dB
PL1W          11.88122272 W
PL12W         0.37571725 W
PL13W         0.37571725 W
SFO2          400.1316005 MHz
SI            65536
SF            376.4982680 MHz
WZM           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```


Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

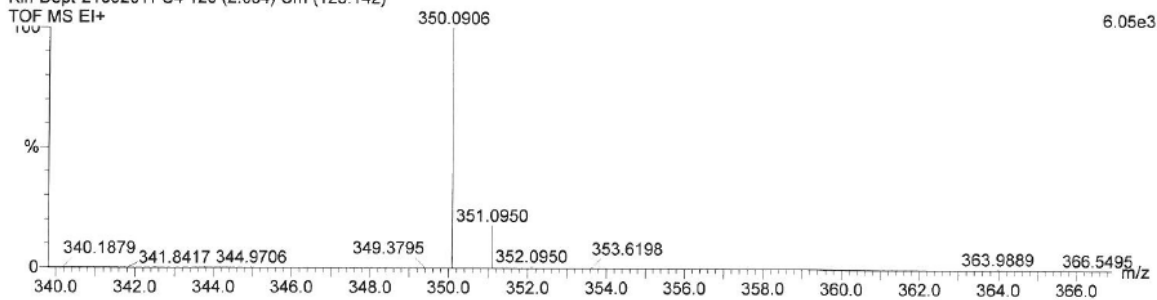
30 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-17 H: 0-13 F: 0-7 Na: 0-1 39K: 0-1

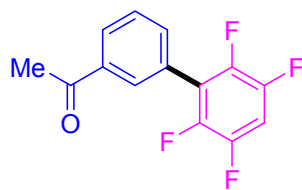
Kin-Dept-21092011 S4 125 (2.084) Cm (125:142)

TOF MS EI+

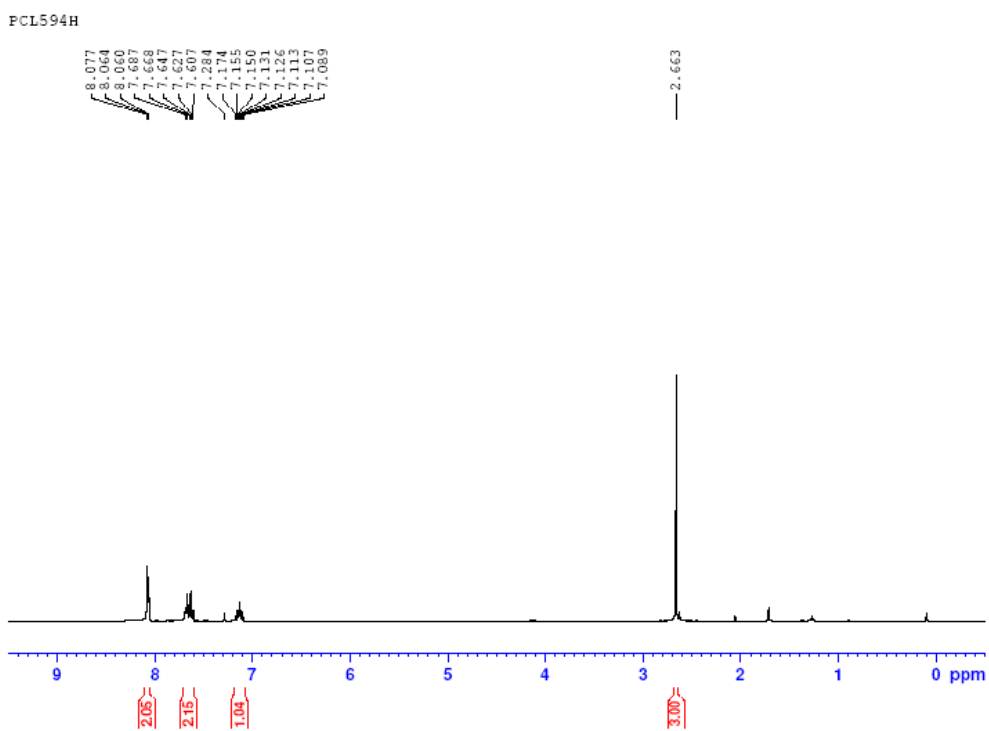


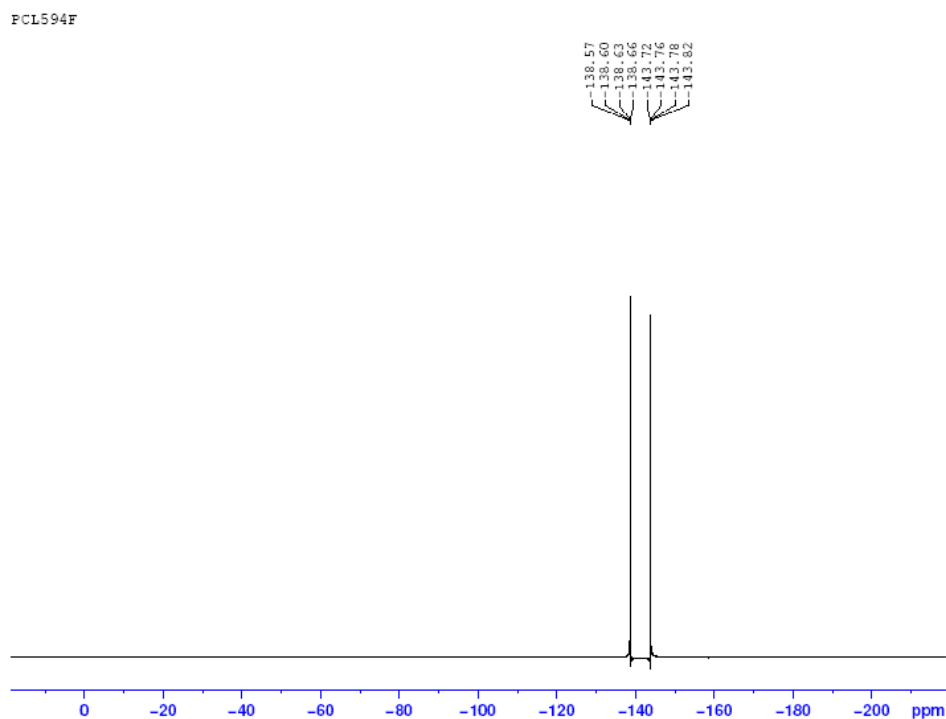
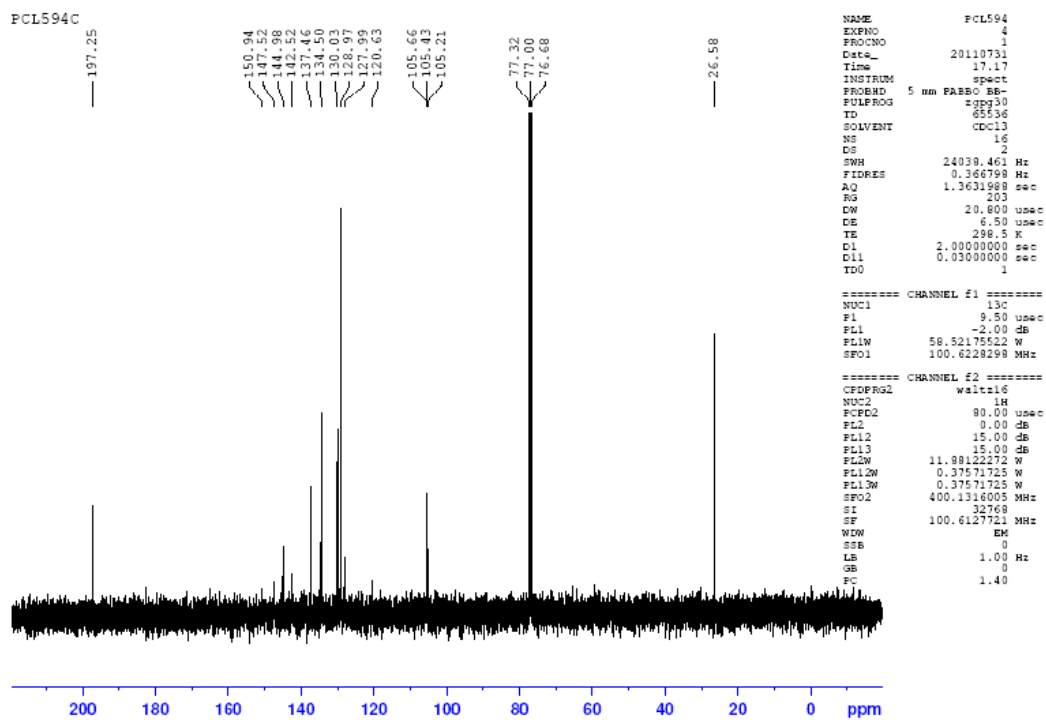
Minimum: -1.5
Maximum: 50.0

| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
|----------|------------|-----|-----|-----|-------|------------|
| 350.0906 | 350.0905 | 0.1 | 0.3 | 8.0 | 43.8 | C17 H13 F7 |



Compound 3ee





Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions

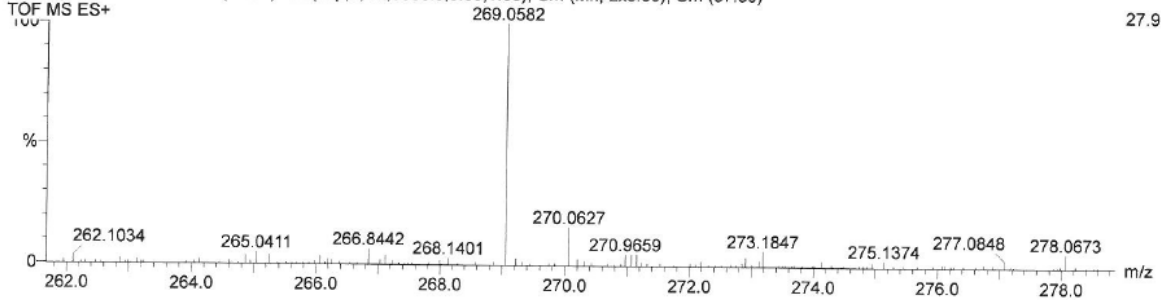
395 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-16 H: 0-10 N: 0-2 O: 0-2 F: 0-5 Na: 0-1 S: 0-1 39K: 0-1

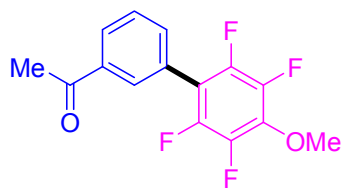
Kin-Dept-25082011 S15 37 (0.694) AM (Top,5, Ht,5000.0,0.00,1.00); Sm (Mn, 2x3.00); Cm (37:39)

TOF MS ES+



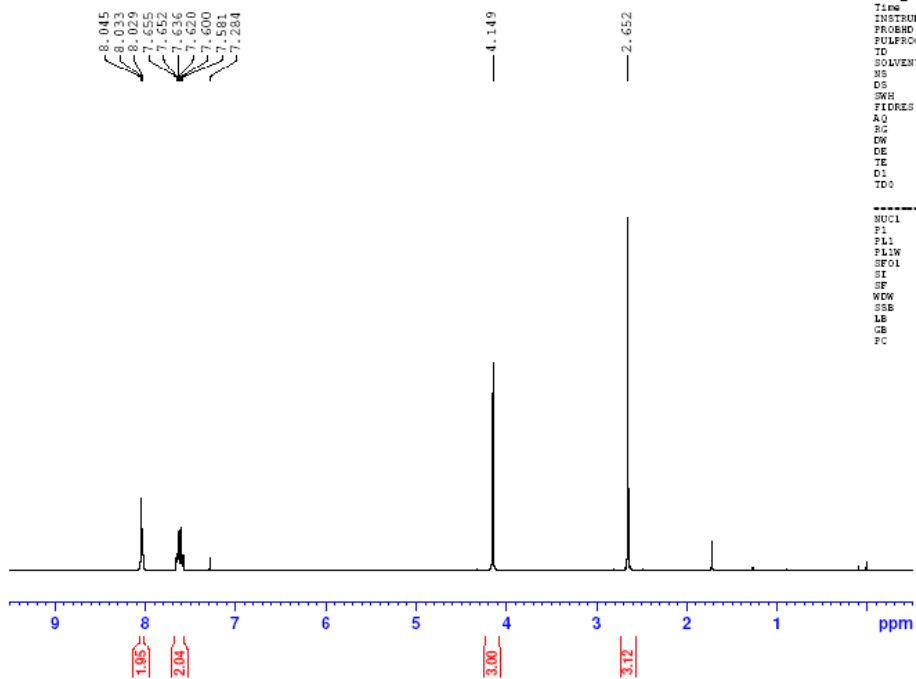
Minimum: -1.5
Maximum: 50.0

| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
|----------|------------|------|------|-----|-------|-------------|
| 269.0582 | 269.0590 | -0.8 | -3.0 | 8.5 | 0.3 | C14 H9 O F4 |



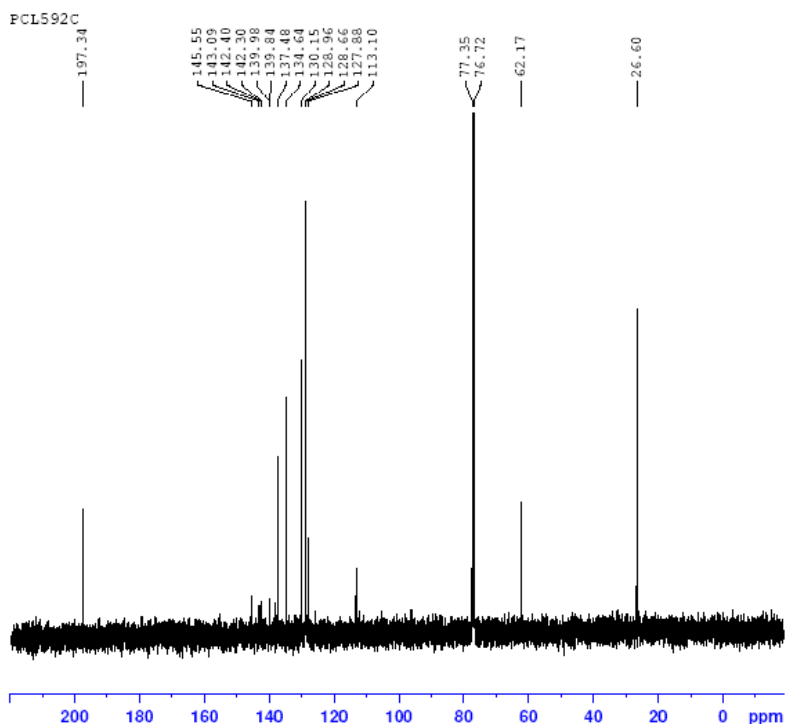
Compound 3be

PCL592H



```
NAME          PCL592
EXPNO         1
PROCNO        1
Date_         20110716
Time         13.44
INSTRUM       spect
PROBHD        5 mm F4BBO BB-
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            8
DS            2
SWH           4006.410 Hz
FIDRES       0.122166 Hz
AQ           4.0894966 sec
RG           20.2
DW           124.800 usec
DE           6.50 usec
TE           297.3 K
D1           1.00000000 sec
TD0          1

----- CHANNEL f1 -----
NUC1          13
P1           14.70 usec
PL1          0.00 dB
PL12         11.98122272 W
SFO1         400.1318007 MHz
SI           32768
SF           400.1300000 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
```

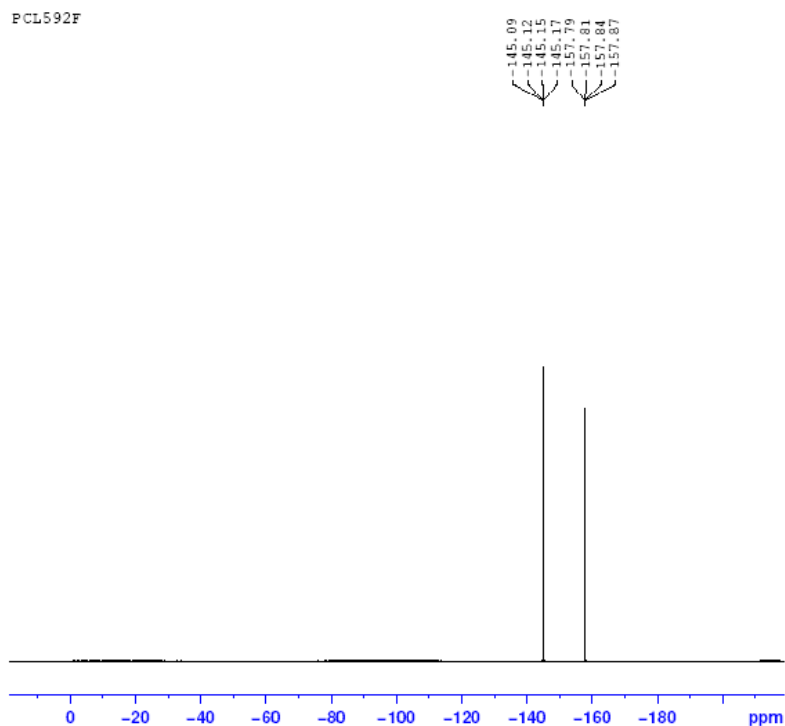


```

NAME          PCL592-C
EXPNO         1
PROCNO        1
Date_         20110716
Time          13.48
INSTRUM       spect
PROBHD        5 mm FASBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            32
DS            2
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631998 sec
RG            203
DW            20.800 usec
DE            6.50 usec
TE            298.9 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -2.00 dB
PL1W          59.52175522 W
SF01          100.6228259 MHz

===== CHANNEL f2 =====
PCPD2         wait116
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12          15.00 dB
PL13          15.00 dB
PL12W         11.88122272 W
PL12W         0.37571725 W
PL13W         0.37571725 W
SF02          400.1316005 MHz
SI            32768
SF            100.6127690 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

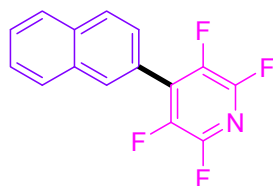


```

NAME          PCL592-F
EXPNO         1
PROCNO        1
Date_         20110716
Time          13.51
INSTRUM       spect
PROBHD        5 mm FASBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            4
SWH           89285.711 Hz
FIDRES        0.691196 Hz
AQ            0.7340532 sec
RG            203
EW            5.600 usec
DE            6.50 usec
TE            298.3 K
D1            1.0000000 sec
D11           0.0300000 sec
D12           0.0002000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          19F
P1            14.20 usec
PL1           -3.00 dB
PL1W          18.69428444 W
SF01          376.4607164 MHz

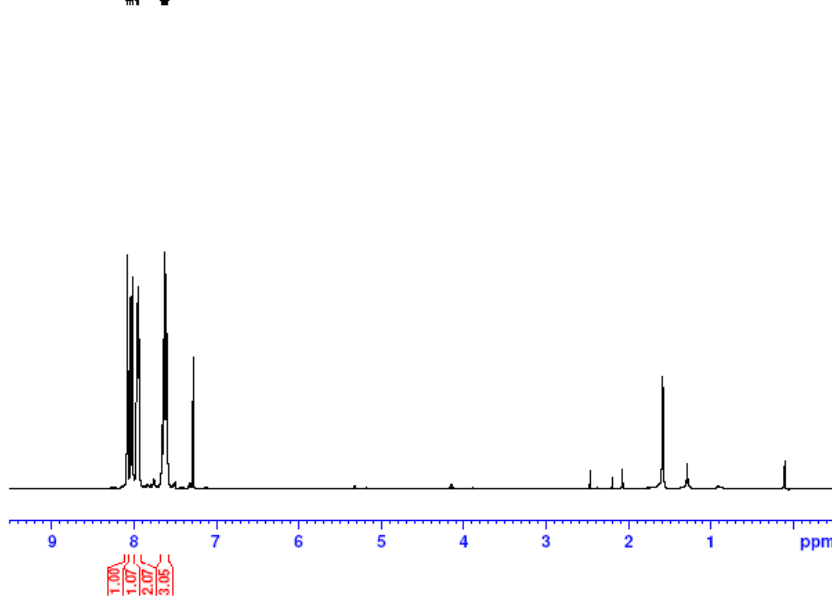
===== CHANNEL f2 =====
PCPD2         wait116
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12          15.00 dB
PL12W         11.88122272 W
PL12W         0.37571725 W
SF02          400.1316005 MHz
SI            65536
SF            376.4983660 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```



Compound 3fi

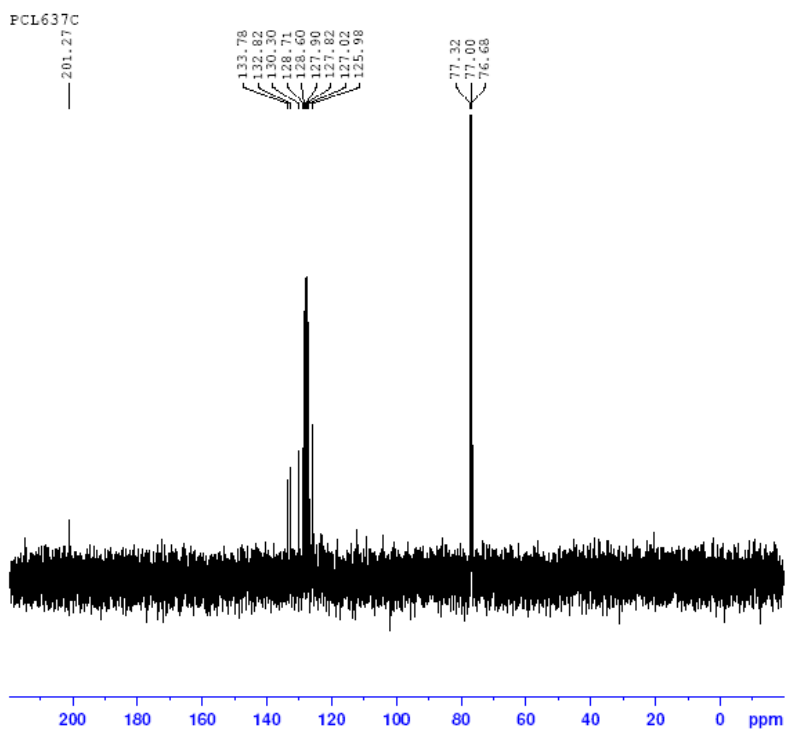
PCL637H

8.080
8.070
8.019
7.964
7.947
7.942
7.667
7.664
7.650
7.647
7.632
7.629
7.621
7.599
7.596



```
NAME          PCL637
EXPNO         1
PROCNO        1
Date_         20110905
Time          11.44
INSTRUM       spect
PROBHD        5 mm F4BBO BB-
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            16
DS            2
SWH           4006.410 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG            90.5
DW            124.800 usec
DE            6.50 usec
TE            296.1 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.70 usec
PL1           0.00 dB
PL12          11.88122272
SFO1          400.1318007 MHz
SI            32768
SF            400.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
```

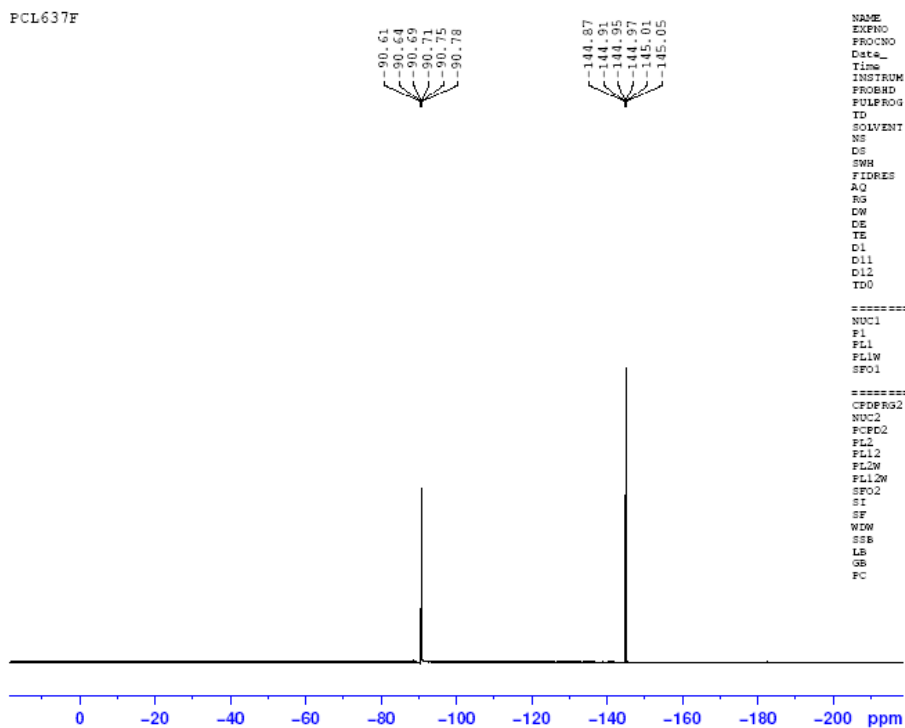


```

NAME          PCL637
EXPNO         2
PROCNO        1
Date_         20110905
Time          11.47
INSTRUM       spect
PROBHD        5 mm FAPBO BB-
PULPROG       zgpg30
TD             65536
SOLVENT       CDCl3
NS             16
DS             2
SWH            24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG             203
CW             20.800 usec
DE             6.50 usec
TE             298.7 K
DL             2.0000000 sec
D11            0.0300000 sec
TD0            1

===== CHANNEL f1 =====
NUC1           13C
P1              9.50 usec
PL1             -2.00 db
PL1W           58.52175522 W
SFO1           100.6228298 MHz

===== CHANNEL f2 =====
CFPRG2         waltz16
NUC2            1H
PCPD2           80.00 usec
PL2              0.00 db
PL12            15.00 db
PL13            15.00 db
PL2W           11.88122272 W
PL12W           0.37571725 W
PL13W           0.37571725 W
SFO2           400.1316005 MHz
SI              32769
SF             100.6127713 MHz
WDW             EM
SSB             0
LB              1.00 Hz
GB              0
PC              1.40
    
```



```

NAME          PCL637
EXPNO         3
PROCNO        1
Date_         20110905
Time          11.50
INSTRUM       spect
PROBHD        5 mm FAPBO BB-
PULPROG       zgfg19gn
TD             131072
SOLVENT       CDCl3
NS             16
DS             4
SWH            89285.711 Hz
FIDRES         0.691196 Hz
AQ             0.7340532 sec
RG             203
CW             5.600 usec
DE             6.50 usec
TE             298.3 K
DL             1.0000000 sec
D11            0.0300000 sec
D12            0.0002000 sec
TD0            1

===== CHANNEL f1 =====
NUC1           19F
P1             14.20 usec
PL1             -3.00 db
PL1W           18.69428444 W
SFO1           376.4607164 MHz

===== CHANNEL f2 =====
CFPRG2         waltz16
NUC2            1H
PCPD2           80.00 usec
PL2              0.00 db
PL12            15.00 db
PL13            15.00 db
PL2W           11.88122272 W
PL12W           0.37571725 W
PL13W           0.37571725 W
SFO2           400.1316005 MHz
SI              65536
SF             376.4993660 MHz
WDW             EM
SSB             0
LB              0.30 Hz
GB              0
PC              1.00
    
```


Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

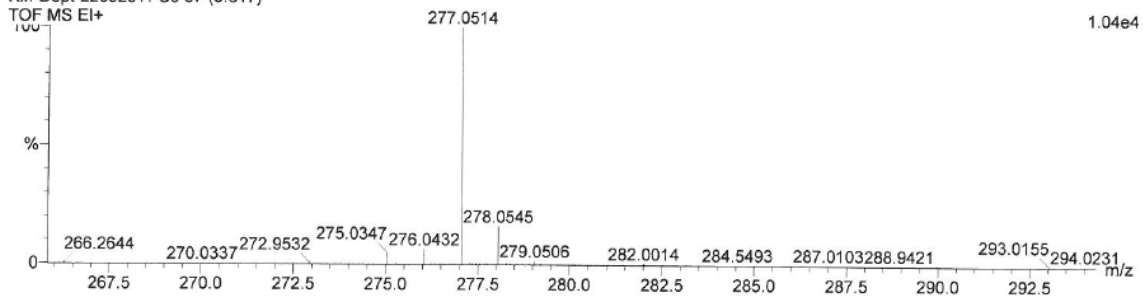
82 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-15 H: 0-7 N: 0-3 F: 0-7 Na: 0-1 39K: 0-1

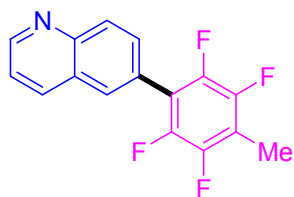
Kin-Dept-22092011 S6 37 (0.617)

TOF MS EI+



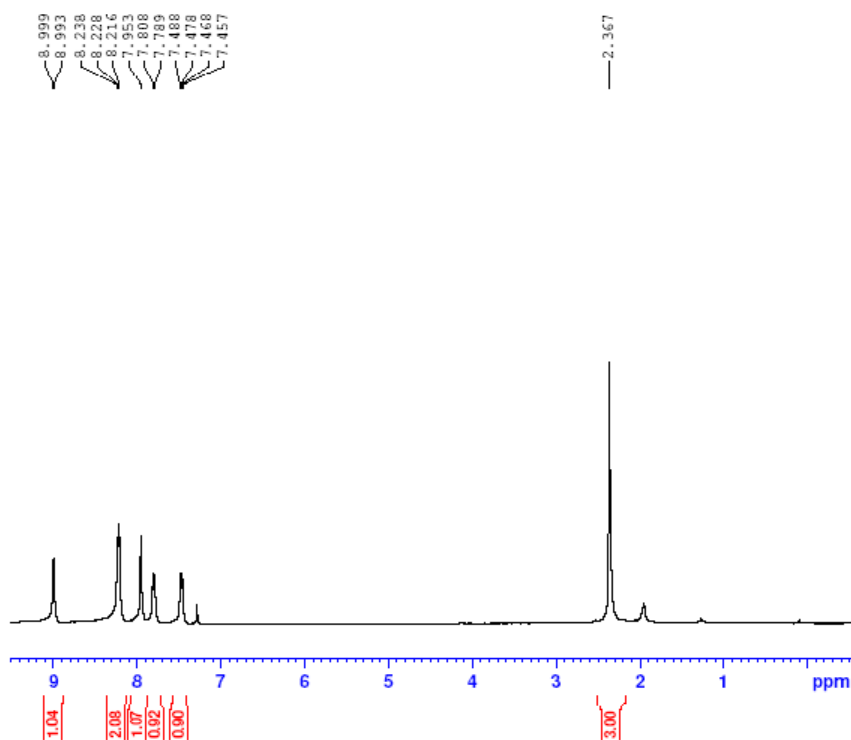
Minimum: -1.5
Maximum: 5.0 10.0 50.0

| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
|----------|------------|------|------|------|-------|-------------|
| 277.0514 | 277.0515 | -0.1 | -0.4 | 11.0 | 3.7 | C15 H7 N F4 |



Compound 3gc

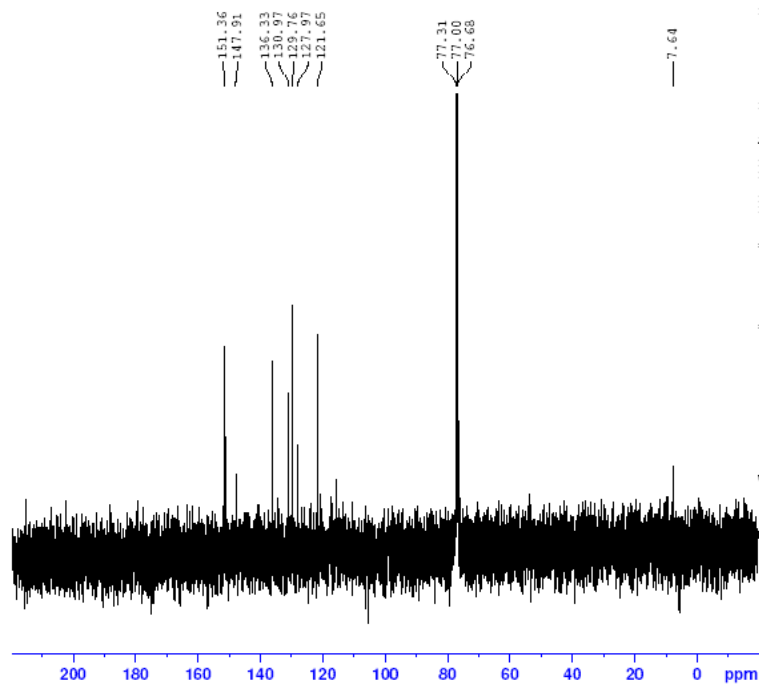
PCL638H



```
NAME          PCL638
EXPNO         1
PROCNO        1
Date_         20110914
Time          23.07
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            16
DS            2
SWH           4006.410 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG            48.2
DW            124.800 usec
DE            6.50 usec
TE            299.8 K
D1            1.0000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.70 usec
PL1           0.00 dB
PL1W         11.98122272 W
SFO1          400.1318007 MHz
SI            32768
SF            400.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
```

PCL638C

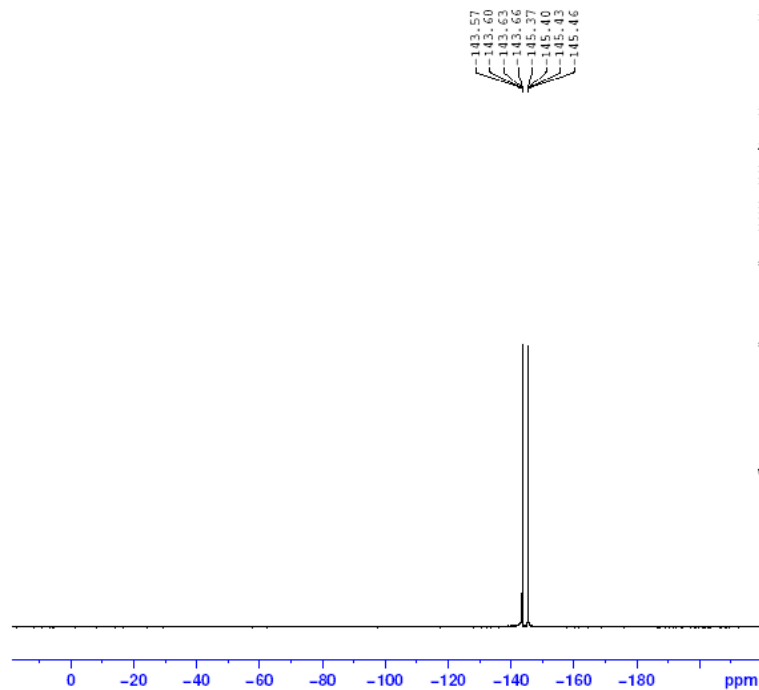


```
NAME          PCL638
EXPNO         3
PROCNO        1
Data_         20110914
Time          23.11
INSTRUM       spect
PROBHD        5 mm FAPBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            203
DW            20.800 usec
DE            6.50 usec
TE            301.0 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1
```

```
===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -2.00 dB
PL1W          58.5217522 W
SFO1          100.6228258 MHz
```

```
===== CHANNEL F2 =====
CFDPFG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           0.00 dB
PL2W          0.0000000 W
SFO2          400.1316005 MHz
SI            32768
SF            100.6127714 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
FC            1.40
```

PCL638F



```
NAME          PCL638
EXPNO         2
PROCNO        1
Data_         20110914
Time          23.08
INSTRUM       spect
PROBHD        5 mm FAPBO BB-
PULPROG       zgpg30
TD            131072
SOLVENT       CDCl3
NS            16
DS            4
SWH           89285.711 Hz
FIDRES        0.691196 Hz
AQ            0.7340532 sec
RG            203
DW            5.600 usec
DE            6.50 usec
TE            300.1 K
D1            1.0000000 sec
D11           0.0300000 sec
D12           0.0002000 sec
TD0           1
```

```
===== CHANNEL f1 =====
NUC1          19F
P1            14.20 usec
PL1           -3.00 dB
PL1W          18.69428444 W
SFO1          376.4607164 MHz
```

```
===== CHANNEL F2 =====
CFDPFG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           0.00 dB
PL2W          0.0000000 W
SFO2          400.1316005 MHz
SI            65536
SF            376.4993660 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
FC            1.00
```

Elemental Composition Report

Page 1

Single Mass Analysis (displaying only valid results)

Tolerance = 10.0 PPM / DBE: min = -100.0, max = 1000.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions

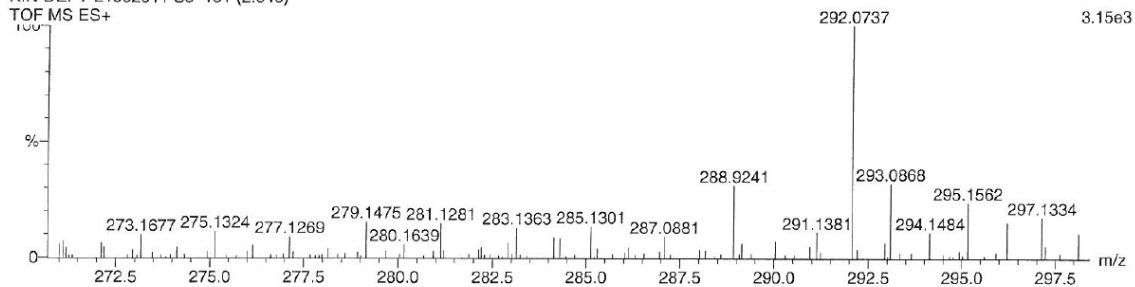
6 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-16 H: 5-10 N: 0-1 F: 0-4

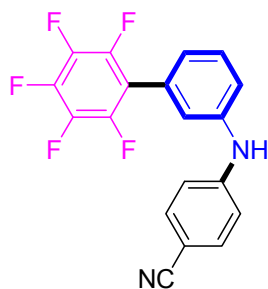
KIN-DEPT-21092011 S5 151 (2.848)

TOF MS ES+



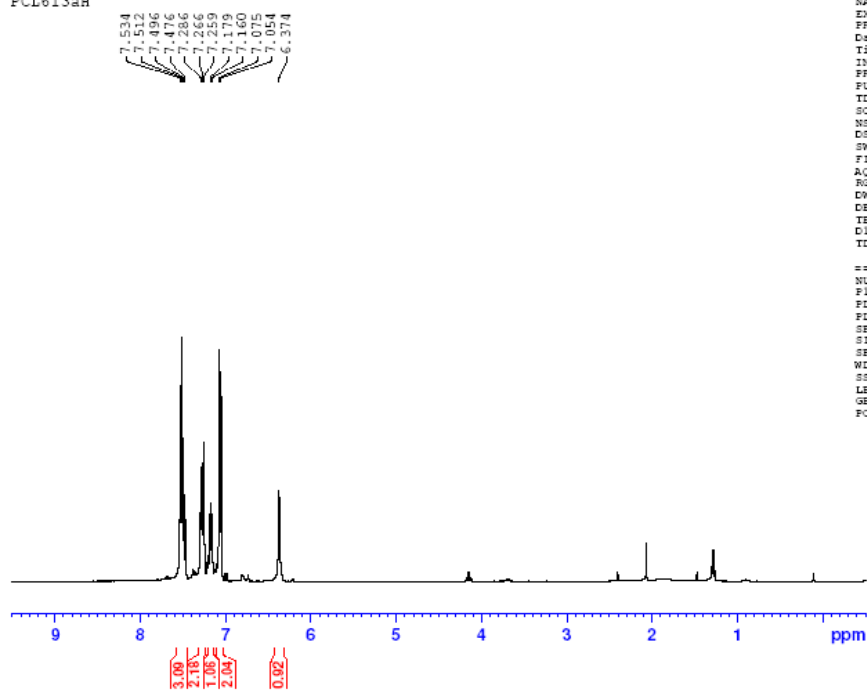
Minimum: -100.0
Maximum: 5.0 10.0 1000.0

| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
|----------|------------|------|------|------|-----------|--------------|
| 292.0737 | 292.0749 | -1.2 | -4.1 | 10.5 | 2773895.0 | C16 H10 N F4 |



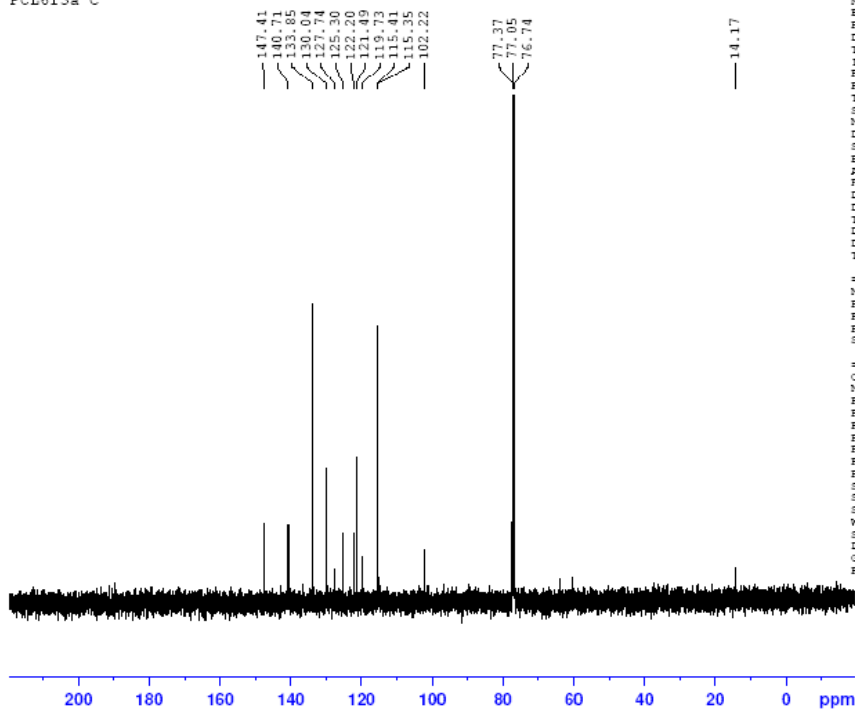
Compound 4ad

PCL613aH



```
NAME          PCL613a
EXPNO         11
PROCNO        1
Date_         20110816
Time         20.59
INSTRUM       spect
PROBRD        5 mm FABSQ BB-
PULPROG       zg30
TD            32768
FIDRES        0.122266 Hz
AQ            4.0694966 sec
RG            45.2
EW            124.800 usec
DE            6.50 usec
TE            298.0 K
DL            1.0000000 sec
TD0           1
===== CHANNEL f1 =====
NUC1          1H
P1            14.70 usec
PL1           0.00 dB
PL1W          11.98122272 W
SFO1          400.1318007 MHz
SI            32768
SF            400.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
```

PCL613a C



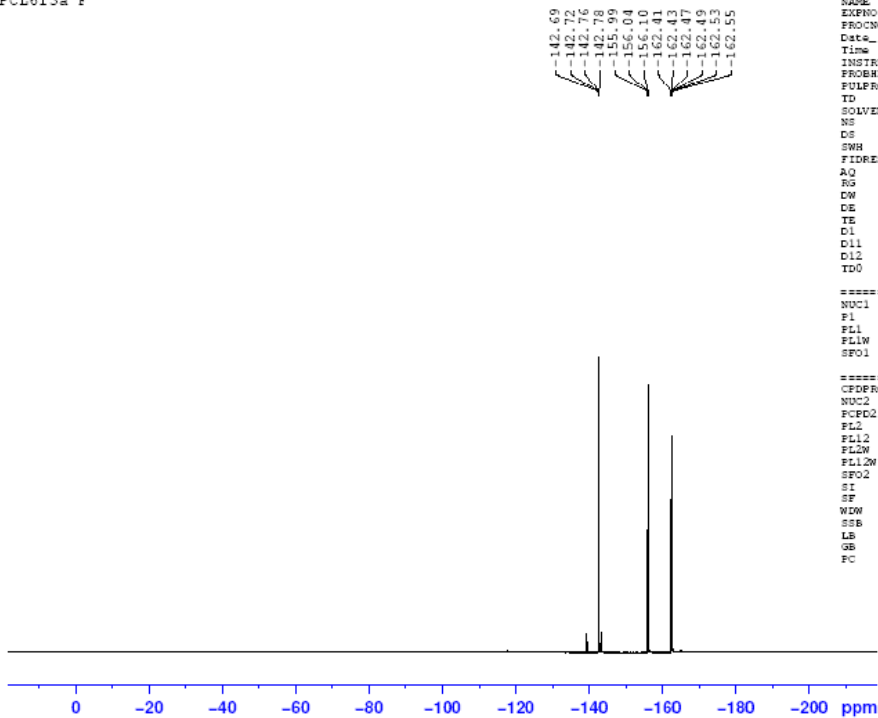
```

NAME          PCL613a
EXPNO         10
PROCNO        1
Date_         20110816
Time         20.52
INSTRUM       spect
PROBHD        5 mm F4BBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ           1.3631988 sec
RG           114
EW           20.800 usec
DE           6.50 usec
TE           298.9 K
DL           2.0000000 sec
D11          0.0300000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1          -2.00 db
PL1W         58.52175522 W
SF01         100.6228259 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 db
PL12         15.00 db
PL13         15.00 db
PL1W         11.88122272 W
PL12W        0.37571725 W
PL13W        0.37571725 W
SFO2         400.1316005 MHz
SI           32768
SF           100.6127690 MHz
WDW           EM
SSB           0
LB           1.00 Hz
GB           0
PC           1.40
    
```

PCL613a F



```

NAME          PCL613a
EXPNO         2
PROCNO        1
Date_         20110816
Time         11.52
INSTRUM       spect
PROBHD        5 mm F4BBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            4
SWH           89285.711 Hz
FIDRES        0.691196 Hz
AQ           0.7340532 sec
RG           203
EW           5.600 usec
DE           6.50 usec
TE           298.1 K
DL           1.0000000 sec
D11          0.0300000 sec
D12          0.0002000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          19F
P1           14.20 usec
PL1          -3.00 db
PL1W         18.69429444 W
SF01         376.4607164 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 db
PL12         15.00 db
PL1W         11.88122272 W
PL12W        0.37571725 W
SFO2         400.1316005 MHz
SI           65536
SF           376.4983660 MHz
WDW           EM
SSB           0
LB           0.30 Hz
GB           0
PC           1.00
    
```

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions

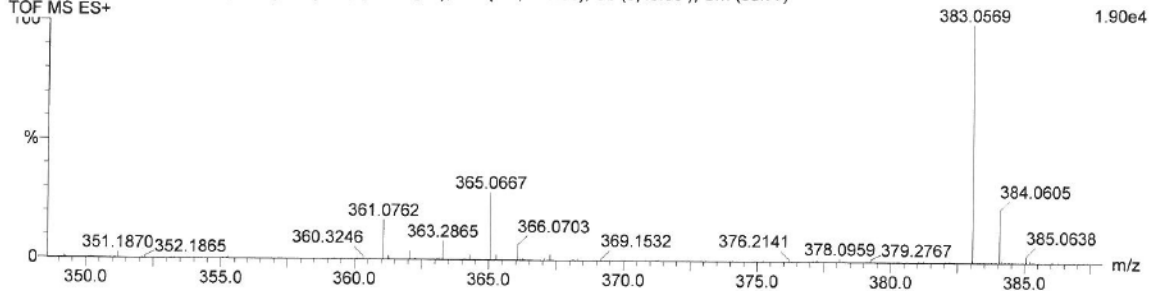
49 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-19 H: 0-10 N: 0-2 F: 0-5 Na: 0-1 39K: 0-1

Kin-Dept-25082011 S19 43 (0.805) Cn (Cen,4, 80.00, Ar); Sm (SG, 2x3.00); Sb (5,40.00); Cm (38:77)

TOF MS ES+



Minimum: -1.5
Maximum: 5.0 10.0 50.0

| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
|----------|------------|------|------|------|-------|---------------|
| 361.0762 | 361.0764 | -0.2 | -0.6 | 13.5 | 5.5 | C19 H10 N2 F5 |

8. References

-
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 - 2 C. M. So, Z. Zhou, C. P. Lau and F. Y. Kwong, *Angew. Chem. Int. Ed.* 2008, **47**, 6402.
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