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Pd(NHC)-Catalyzed Alkylsulfonylation of Boronic Acids: A General and Efficient Approach for Sulfone Synthesis

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1. General

All commercial reagents were used directly without further purification, unless otherwise stated. Dry dimethylsulfoxide (DMSO) was purchased from J & K chemical, stored over 4 Å molecular sieves and handled under N_2. Anhydrous methanol (MeOH) was distilled from anhydrous calcium chloride, Dioxane, Tetrahydrofuran (THF) and toluene were distilled from sodium/benzophenone, 1,2-Dichloroethane (DCE) was distilled from calcium hydride prior to use. All schlenk tubes and sealed vessels (50 mL) were purchased from Beijing Synthware Glass. $CDCl_3$ was purchased from Cambridge Isotope Laboratories.^{1}H NMR and ^{13}C NMR spectra were recorded on Jeol ECA-400 and Bruker 400 DRX spectrometers. ^{13}C NMR spectra were referenced to the carbon signal of $CDCl_3$ (77.0 ppm). GC-MS spectra were recorded on Agilent Technologies 1890A GC system and 5975C inert MSD with Triple-Axis Detector.

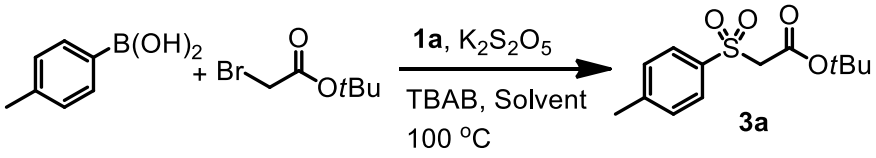
2. Experimental sections

2.1 General procedure for sulfones.

To a 50 mL schlenk tube containing boronic acid (0.5 mmol), $K_2S_2O_5$ (1.0 equiv), TBAB (1.0 equiv), and NHC palladium complex **1a** (5 mol%) were added and the tube was purged with N_2 for 3 times, followed by 2 mL of Dioxane and *tert*-butyl bromoacetate (2.0 equiv). The resulted reaction mixture was allowed to stir for 24h at 100 ^{o}C under the atmosphere of nitrogen. After the completion of the reaction, the resulting mixture was concentrated under the vacuum and directly purified by flash chromatography to give the desired product.

2.2 Optimization of reaction conditions

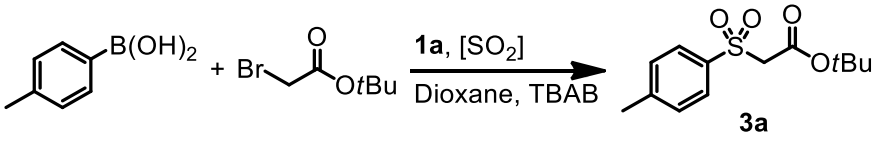
Table S1. Solvent effects (excluded data in the Table 1)[a]



Entry	Solvent	Yield[b](%)
1	MeOH	NR
2	DMF	43
3	DMSO	NR
4	MeCN	52

[a] Conditions: 4-tolylboronic acid (0.5 mmol, 1.0 equiv), TBAB, $K_2S_2O_5$ (2.0 equiv), **1a** (5 mol%) were stirred in Dioxane (2 mL) about 24 h at 100 °C under atmosphere of N_2. [b] Isolated yield based on 4-tolylboronic acid.

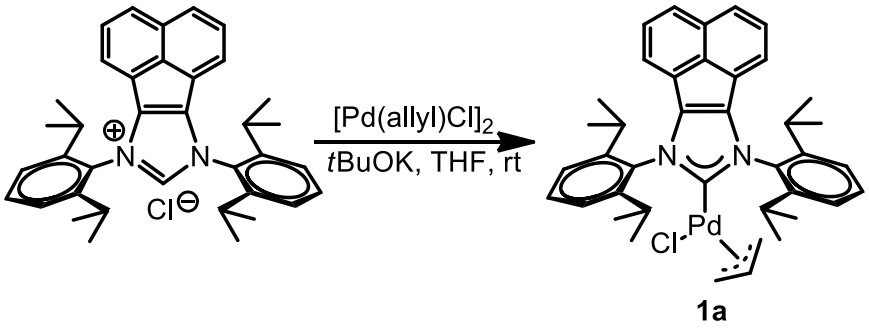
Table S2. The loading of TBAB and the source of sulfur dioxide (excluded data in the Table 1)[a]



Entry	TBAB (equiv)	$[SO_2]$	Yield[b](%)
1	1.1	DABSO	NR
2	1.1	$Na_2S_2O_5$	96
3	1.1	$K_2S_2O_5$	>99
4	0.5	$K_2S_2O_5$	86
5	0.2	$K_2S_2O_5$	54
6	0.1	$K_2S_2O_5$	44
7	/	$K_2S_2O_5$	Trace

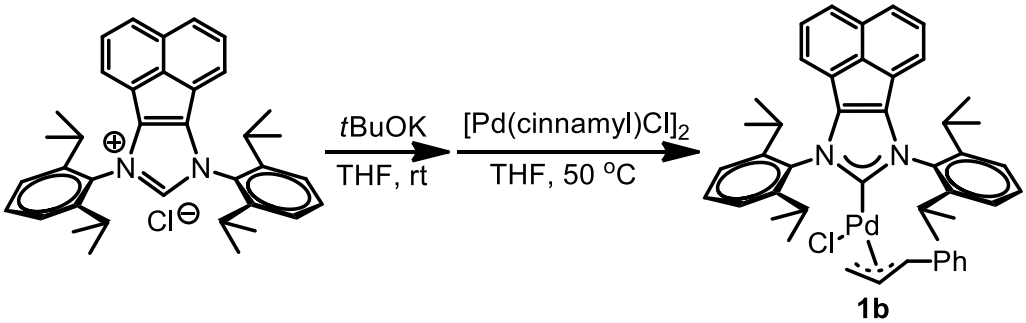
[a] Conditions: 4-tolylboronic acid (0.5 mmol, 1.0 equiv), TBAB, sulfur dioxide surrogate (2.0 equiv), **1a** (5 mol%) were stirred in Dioxane (2 mL) about 24 h at 100 °C under atmosphere of N_2. [b] Isolated yield based on 4-tolylboronic acid.

2.3 Synthesis of catalyst.



Scheme S1. Synthesis of Pd-NHC complex **1a**.

Pd-NHC complex 1a:[S1] To a schlenk tube containing IPr(BIAN) imidazolium chloride (315 mg, 0.57 mmol), $[Pd(allyl)Cl]_2$ (100 mg, 0.24 mmol), *t*BuOK (76.7 mg, 0.68 mmol) and a stirrer bar, THF (6 mL) was added. The reaction mixture was allowed to stir at room temperature for 24 hours under a nitrogen atmosphere. The reaction mixture was loaded on a plug of silica gel and eluted with DCM. A small amount of silica gel was added and the solvent was removed in vacuo. The product was loaded directly on a silica gel column and purified by flash chromatography to give a yellow solid. Yield: 340 mg, 85%. ^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.70 (d, *J* = 8.0 Hz, 2H), 7.54 (t, *J* = 8.0 Hz, 2H), 7.39-7.32 (m, 6H), 6.84 (d, *J* = 6.8Hz, 2H), 4.97-4.87 (m, 1H), 3.98 (dd, *J* = 7.2 Hz, *J* = 1.2 Hz, 1H), 3.37-3.27 (m, 3H), 3.16-3.10 (m, 2H), 2.90 (d, *J* = 13.2 Hz, 1H), 1.86 (d, *J* = 12.0 Hz, 1H), 1.37 (dd, *J* = 6.0 Hz, J = 1.6 Hz, 12H), 0.99-0.94 (m,12H); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm) *δ* = 192.95, 146.36, 146.20, 140.34, 134.61, 130.19, 129.87, 129.62, 127.75, 127.29, 126.25, 124.42, 124.16, 121.58, 114.44, 73.56, 50.26, 28.69, 28.65, 25.59, 25.31, 23.79, 23.28.



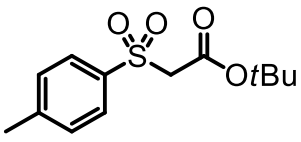
Scheme S2. Synthesis of Pd-NHC complex **1b.**

Pd-NHC complex 1b:[S2] To a schlenk tube containing IPr(BIAN) imidazolium chloride (346 mg, 0.63 mmol), *t*BuOK (85 mg, 0.76 mmol) and a stirrer bar, THF (20 mL) was added and the reaction was stirred at room temperature 12 h. $[Pd(cinnamyl)Cl]_2$ (147.6 mg, 0.285

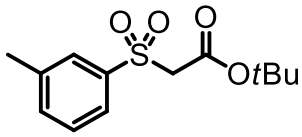
mmol) in 10 mL was added to the resulted mixture. Then the reaction mixture was allowed to stir at 50 ^{o}C for 24 hours under a nitrogen atmosphere. The reaction mixture was loaded on a plug of silica gel and eluted with DCM. A small amount of silica gel was added and the solvent was removed in vacuo. The product was loaded directly on a silica gel column and purified by flash chromatography to give a yellow solid. Yield: 526 mg, 60%. ^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.70 (d, *J* = 8.4 Hz, 2H), 7.58 (t, *J* = 8.0 Hz, 2H), 7.41 (d, *J* = 7.6 Hz, 4H), 7.36-7.33 (m, 3H), 7.17-7.13 (m, 5H), 6.86 (d, *J* = 7.2 Hz, 2H), 5.17-5.09 (m, 1H), 4.39 (d, *J* = 12.8 Hz, 1H), 3.27-3.22 (m, 6H), 1.34 (d, *J* = 6.4 Hz, 12H), 0.97 (d, *J* = 6.4 Hz, 12H); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm) *δ* = 192.27, 146.34, 140.33, 138.07, 134.72, 130.17, 129.81, 129.60, 128.12, 128.02, 127.89, 127.75, 127.49, 127.27, 126.60, 126.27, 124.26, 121.56, 109.05, 90.06, 47.63, 28.69, 28.55, 25.68, 25.43, 23.73, 23.58.
HR-MS (ESI): m/z 735.2931 (Calcd. $[M-Cl]^+$), 735.2915 (Found. $[M-Cl]^+$).

Palladium dimer complex 30: The yellow solid (10 mg) was dissolved in DCM (1 mL) in 10 mL test tube and the open vial was placed in a 50 mL Schlenk tube containing diethyl ether. The Schlenk tube was closed and single crystals were allowed to grow via the process of vapor diffusion in refrigerator during 3 days. The crystals were collected and examined by ^{1}H NMR and X-ray diffraction analysis. These data can be obtained from The Cambridge Crystallographic Data Centre. ^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.68-7.63 (m, 14H), 7.43-7.39 (m, 7H), 7.35-7.32 (m, 14H), 7.11-7.08 (m, 1H), 6.91-6.89 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm) *δ* = 134.97, 134.91, 134.85, 130.32, 128.07, 128.02, 127.73, 123.97; ^{31}P NMR (161 MHz, $CDCl_3$): δ = 26.69.

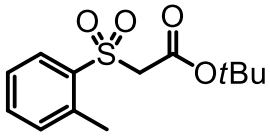
3. Data for the amination products

3a: 

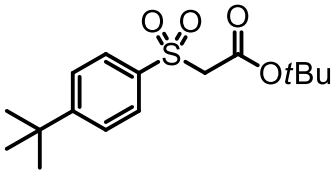
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.82 (d, *J* = 8.4 Hz, 2H), 7.37 (d, *J* = 8.4 Hz, 2H), 4.01 (s, 2H), 2.46 (s, 3H), 1.38 (s, 9H); ^{13}C NMR (100 MHz, CDCl3, 298 K, ppm) δ 161.20, 145.02, 135.80, 129.55, 128.32, 83.26, 61.97, 27.46, 21.45.

3b: 

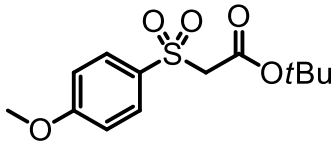
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.75-7.74 (m, 2H), 7.48-7.46 (m, 2H), 4.02 (s, 2H), 2.45 (s, 3H), 1.37 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm) δ 161.15, 139.29, 138.65, 134.78, 128.92, 128.63, 125.51, 83.38, 62.03, 27.53, 21.19.

3c: 

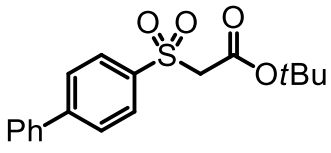
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 8.04-8.01 (m, 1H), 7.56-7.52 (m, 1H), 7.41-7.35 (m, 2H), 4.08 (s, 2H), 2.72 (s, 3H), 1.30 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm) δ 160.98, 138.06, 136.92, 133.99, 132.58, 130.55, 126.34, 83.38, 61.42, 27.43, 20.17.

4a: 

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.86 (d, J = 8.4 Hz, 2H), 7.58 (d, J = 8.8 Hz, 2H), 4.02 (s, 2H), 1.35 (s, 18H); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm) δ 161.29, 158.04, 135.90, 128.33, 126.07, 83.38, 62.16, 35.24, 30.97, 27.58.

4b: 

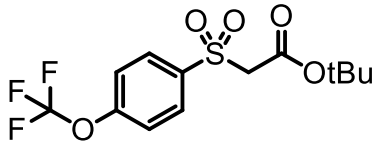
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.87 (d, J = 8.8 Hz, 2H), 7.02 (d, J = 8.8 Hz, 2H), 4.00 (s, 2H), 3.89 (s, 3H), 1.39 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm) δ 164.03, 161.50, 130.72, 130.36, 114.24, 83.39, 62.28, 55.69, 27.65.

4c: 

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 8.01 (d, J = 8.4 Hz, 2H), 7.78 (d, J = 8.4 Hz, 2H),

7.63-7.61 (m, 2H), 7.52-7.44 (m, 3H), 4.08 (s, 2H), 1.39 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 161.25, 146.99, 138.96, 137.36, 129.01, 128.67, 127.64, 127.32, 83.56, 62.12, 27.62.

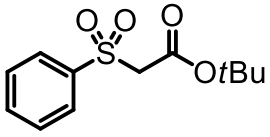
HR-MS (ESI): m/z 355.0980 (Calcd. $[M+Na]^+$), 355.0975 (Found. $[M+Na]^+$).

4d: 

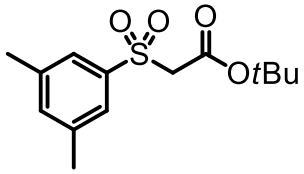
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 8.01 (d, *J* = 9.2 Hz, 2H), 7.40 (d, J = 8.4 Hz, 2H), 4.05 (s, 2H), 1.38 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 161.08, 153.21, 137.07, 130.95, 120.79, 83.83, 61.92, 27.56.

^{19}F NMR ($CDCl_3$, 376 MHz, 298 K): δ = -57.67.

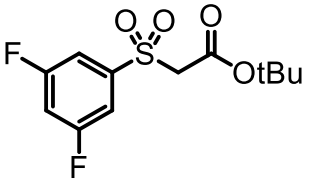
HR-MS (ESI): m/z 363.0490 (Calcd. $[M+Na]^+$), 363.0484 (Found. $[M+Na]^+$).

5a: 

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.96-7.94 (m, 2H), 7.70-7.67 (m, 2H), 7.60-7.56 (m, 2H), 4.04 (s, 2H), 1.36 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm) δ 161.03, 138.67, 133.95, 128.26, 83.34, 61.83, 27.42.

5b: 

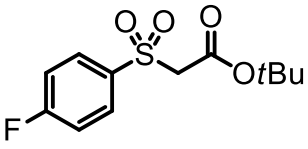
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.54 (s, 2H), 7.28 (s, 1H), 4.01 (s, 2H), 2.40 (s, 6H), 1.37 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm) δ 161.13, 139.04, 138.47, 135.57, 125.75, 83.20, 62.03, 27.45, 21.00.

5c: 

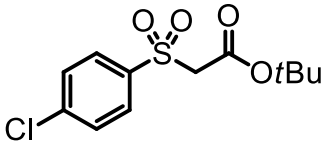
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.51-7.49 (m, 2H), 7.16-7.11 (m, 1H), 4.05 (s, 2H), 1.34 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 163.97, 163.86, 162.09, 161.43, 161.32, 160.70, 142.05, 141.97, 141.89, 112.36, 112.27, 112.16, 112.07, 109.98, 109.48, 84.14,

61.66, 27.60.

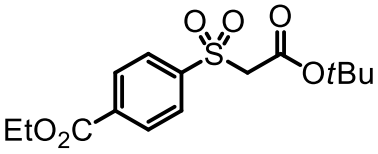
^{19}F NMR ($CDCl_3$, 376 MHz, 298 K): δ = -104.92.

6a: 

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.99-7.96 (m, 2H), 7.28-7.24 (m, 2H), 4.04 (s, 2H), 1.39 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 167.15, 164.59, 161.11, 131.45, 131.36, 116.41, 116.18, 83.58, 61.89, 27.51; ^{19}F NMR ($CDCl_3$, 376 MHz, 298 K): δ = -102.60.

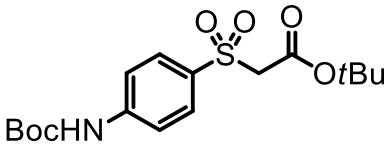
6b: 

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.88 (d, J = 8.4 Hz, 2H), 7.56 (d, J = 8.8 Hz, 2H), 4.03 (s, 2H), 1.39 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 161.03, 140.74, 137.24, 129.97, 129.30, 83.68, 61.81, 27.54.

7a: 

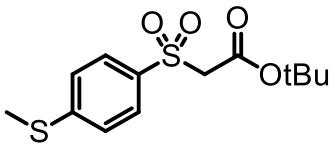
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 8.23 (s, 2H), 8.03 (s, 2H), 4.43 (q, J = 7.2 Hz, 2H), 4.06 (s, 2H), 1.42 (t, J = 7.2 Hz, 3H), 1.38 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 164.75, 160.89, 142.36, 135.32, 130.04, 128.48, 83.74, 61.71, 61.67, 27.52, 14.08.

HR-MS (ESI): m/z 346.1324 (Calcd. $[M+NH_4]^+$), 346.1319 (Found. $[M+NH_4]^+$).

7b: 

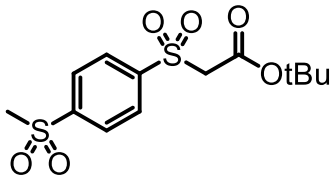
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.84 (d, J = 8.8 Hz, 2H), 7.56 (d, J = 8.4 Hz, 2H), 6.80 (s, 1H), 4.00 (s, 2H), 1.53 (s, 9H), 1.39 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 161.44, 152.04, 144.17, 131.51, 129.72, 117.59, 83.52, 81.42, 62.13, 28.08, 27.57.

HR-MS (ESI): m/z 389.1746 (Calcd. $[M+NH_4]^+$), 389.1741 (Found. $[M+NH_4]^+$).

8: 

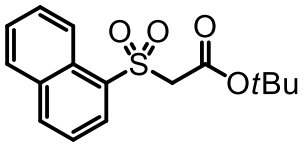
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.80 (d, J = 8.4 Hz, 2H), 7.35 (d, J = 8.4 Hz, 2H), 4.01 (s, 2H), 2.53 (s, 3H), 1.40 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm) δ = 161.28, 147.81, 134.31, 128.68, 125.03, 83.49, 62.08, 27.60, 14.59.

HR-MS (ESI): m/z 325.0544 (Calcd. $[M+Na]^+$), 325.0539 (Found. $[M+Na]^+$).

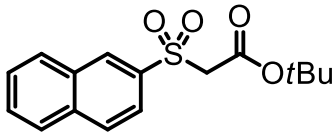
9: 

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 8.17 (s, 4H), 4.09 (s, 2H), 3.11 (s, 3H), 1.40 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm) δ 160.82, 145.64, 143.87, 129.81, 128.27, 84.29, 61.65, 44.25, 27.68.

HR-MS (ESI): m/z 357.0442 (Calcd. $[M+Na]^+$), 357.0437 (Found. $[M+Na]^+$).

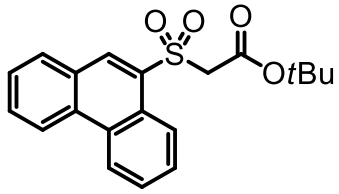
10a: 

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 8.69 (dd, J = 8.4 Hz, J = 0.4 Hz, 1H), 8.4 (dd, J = 7.6 Hz, J = 1.2 Hz, 1H), 8.17 (d, J = 8.0 Hz, 1H), 8.00 (d, J = 8.0 Hz, 1H), 7.76-7.72 (m, 1H), 7.67-7.60 (m, 2H), 4.24 (s, 2H), 1.25 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 161.00, 135.61, 134.10, 133.82, 131.31, 129.36, 128.94, 128.71, 127.04, 124.19, 123.67, 83.38, 61.80, 27.49.

10b: 

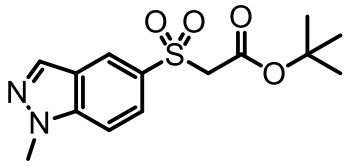
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 8.52 (d, J = 0.8 Hz, 1H), 8.01 (t, J = 8.8 Hz, 2H), 7.95-7.89 (m, 2H), 7.71-7.62 (m, 2H), 4.11 (s, 2H), 1.32 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 161.20, 135.73, 135.37, 131.89, 130.43, 129.42, 129.38, 129.36, 127.94, 127.71, 122.86, 83.52, 62.10, 27.55.

HR-MS (ESI): m/z 329.0823 (Calcd. $[M+Na]^+$), 329.0818 (Found. $[M+Na]^+$).

10c: 

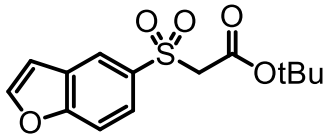
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 8.82-8.80 (m, 1H), 8.77-8.69 (m, 3H), 8.06 (d, *J* = 7.6 Hz, 1H), 7.87-7.83 (m, 1H), 7.81-7.76 (m, 2H), 7.74-7.70 (m, 1H), 4.29 (s, 2H), 1.18 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 160.97, 134.46, 132.73, 132.42, 131.17, 130.73, 130.36, 128.98, 128.17, 127.78, 127.71, 126.07, 124.61, 123.66, 122.78, 83.37, 61.61, 27.43.

HR-MS (ESI): m/z 379.0980 (Calcd. $[M+Na]^+$), 379.0975 (Found. $[M+Na]^+$).

11: 

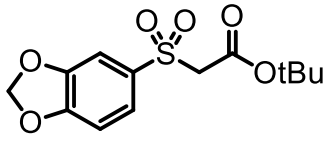
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 8.41 (s, 1H), 8.16 (s, 1H), 7.90 (d, *J* = 8.8 Hz, 1H), 7.54 (d, *J* = 8.8 Hz, 1H), 4.14 (s, 3H), 4.07 (s, 2H), 1.36 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 161.45, 141.28, 134.69, 130.99, 125.10, 124.41, 123.12, 109.60, 83.49, 62.38, 35.88, 27.63.

HR-MS (ESI): m/z 311.1066 (Calcd. $[M+H]^+$), 311.1060 (Found. $[M+H]^+$).

12: 

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 8.25 (d, *J* = 2.0 Hz, 1H), 7.90-7.88 (m, 1H), 7.79 (d, *J* = 2.0 Hz, 1H), 7.67 (d, *J* = 8.4 Hz, 1H), 6.91 (d, *J* = 1.2 Hz, 1H), 4.07 (s, 2H), 1.36 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 161.34, 157.35, 147.47, 133.55, 127.81, 124.48, 123.08, 112.07, 107.08, 83.44, 62.41, 27.57.

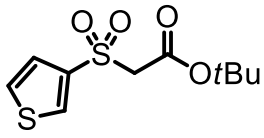
HR-MS (ESI): m/z 319.0616 (Calcd. $[M+Na]^+$), 319.0611 (Found. $[M+Na]^+$).

13: 

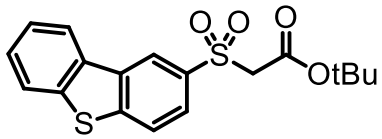
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.50 (dd, *J* = 8.4 Hz, *J* = 2.0 Hz, 1H), 7.33 (d, *J* = 2.0 Hz,

1H), 6.93 (d, *J* = 8.4 Hz, 1H), 6.11 (s, 2H), 3.99 (s, 2H), 1.41 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 161.28, 152.45, 148.06, 131.97, 124.56, 108.37, 108.23, 102.47, 83.42, 62.13, 27.58.

HR-MS (ESI): m/z 323.0565 (Calcd. $[M+Na]^+$), 323.0560 (Found. $[M+Na]^+$).

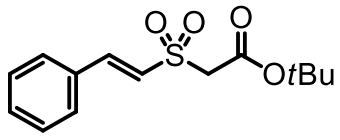
14: 

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 8.14-8.13 (m, 1H), 7.48-7.43 (m, 2H), 4.05 (s, 2H), 1.41 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 161.14, 139.11, 133.52, 127.95, 126.24, 83.46, 62.10, 27.54.

15: 

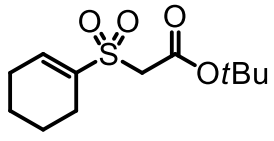
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 8.72 (d, *J* = 1.2 Hz, 1H), 8.25-8.23 (m, 1H), 8.04 (d, *J* = 8.4 Hz, 1H), 8.00-7.97 (m, 1H), 7.92-7.90 (m, 1H), 7.58-7.55 (m, 2H), 4.13 (s, 2H), 1.35 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 161.28, 145.51, 139.82, 135.65, 134.99, 134.32, 127.99, 125.48, 125.22, 123.33, 122.93, 122.19, 122.07, 83.61, 62.41, 27.63.

HR-MS (ESI): m/z 385.0544 (Calcd. $[M+Na]^+$), 385.0539 (Found. $[M+Na]^+$).

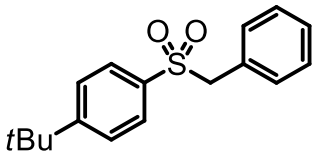
16: 

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.63 (d, *J* = 15.2 Hz, 1H), 7.54-7.52 (m, 2H), 7.46-7.41 (m, 3H), 7.07 (d, J = 15.6 Hz, 1H), 4.00 (s, 2H), 1.48 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 161.79, 145.07, 132.00, 131.47, 129.12, 128.63, 124.92, 83.79, 61.20, 27.79.

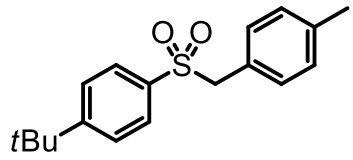
HR-MS (ESI): m/z 305.0823 (Calcd. $[M+Na]^+$), 305.0818 (Found. $[M+Na]^+$).

17: 

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 6.97-6.95 (m, 1H), 3.85 (s, 2H), 2.42-2.38 (m, 2H), 2.31-2.28 (m, 2H), 1.81-1.75 (m, 2H), 1.69-1.63 (m, 2H), 1.46 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 161.48, 141.22, 137.90, 83.18, 58.23, 27.64, 25.42, 23.32, 21.67, 20.58.

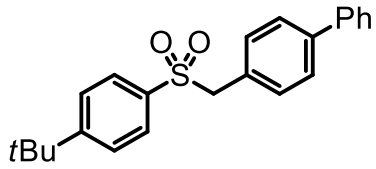
18: 

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.56 (d, J = 8.4 Hz, 2H), 7.45 (d, J = 8.8 Hz, 2H), 7.34-7.24 (m, 3H), 7.11-7.09 (m, 2H), 4.29 (s, 2H), 1.33 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 157.62, 134.95, 130.80, 128.62, 128.43, 128.38, 128.18, 125.80, 62.86, 30.98, 27.35.

19a: 

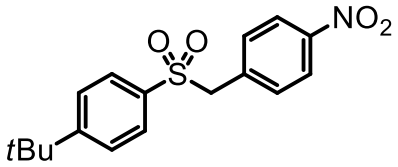
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.58 (d, J = 8.4 Hz, 2H), 7.46 (d, J = 8.4 Hz, 2H), 7.07 (d, J = 7.6 Hz, 2H), 6.99 (d, J = 8.0 Hz, 2H), 4.25 (s, 2H), 2.33 (s, 3H), 1.33 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 157.50, 138.51, 135.09, 130.65, 129.15, 128.36, 125.78, 124.99, 62.52, 35.14, 30.98, 21.14.

HR-MS (ESI): m/z 325.1238 (Calcd. $[M+Na]^+$), 325.1233 (Found. $[M+Na]^+$).

19b: 

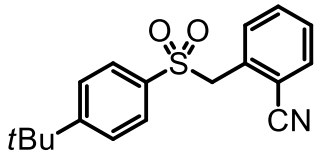
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.62 (d, J = 8.8 Hz, 2H), 7.57 (d, J = 7.2 Hz, 2H), 7.52-7.43 (m, 6H), 7.38-7.34 (m, 1H), 7.19 (d, J = 8.4 Hz, 2H), 4.34 (s, 2H), 1.34 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 157.67, 141.43, 140.17, 135.04, 131.21, 128.76, 128.38, 127.56, 127.10, 127.02, 126.97, 125.86, 62.51, 35.16, 30.97.

HR-MS (ESI): m/z 387.1395 (Calcd. $[M+Na]^+$), 387.1389 (Found. $[M+Na]^+$).

19c: 

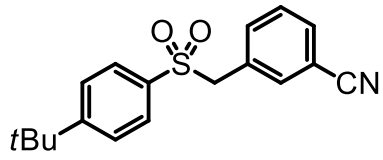
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 8.14 (d, J = 8.8 Hz, 4H), 7.60 (d, J = 8.8 Hz, 1H), 7.50 (d, J = 8.4 Hz, 2H), 7.32 (d, J = 8.8 Hz, 2H), 4.38 (s, 2H), 1.34 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 158.39, 148.05, 135.40, 134.60, 131.78, 128.30, 126.20, 123.56, 62.14, 30.96, 27.35.

HR-MS (ESI): m/z 356.0932 (Calcd. $[M+H]^+$), 356.0927 (Found. $[M+H]^+$).

20a: 

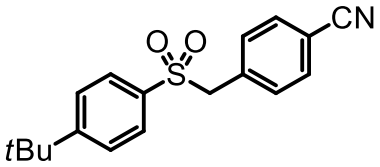
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.65-7.62 (m, 4H), 7.57 (d, J = 7.6 Hz, 1H), 7.52-7.44 (m, 3H), 4.54 (s, 2H), 1.34 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 158.29, 134.48, 132.76, 132.65, 132.09, 131.57, 129.21, 128.36, 126.16, 116.50, 114.33, 60.46, 35.17, 30.88.

HR-MS (ESI): m/z 314.1215 (Calcd. $[M+H]^+$), 314.1219 (Found. $[M+H]^+$).

20b: 

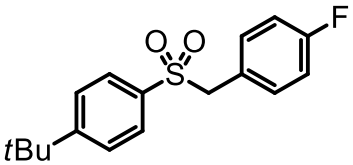
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.63-7.56 (m, 3H), 7.52-7.41 (m, 4H), 7.26 (s, 1H), 4.30 (s, 2H), 1.34 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 158.41, 135.19, 134.32, 134.12, 132.24, 129.96, 129.42, 128.35, 126.16, 117.88, 112.71, 62.01, 35.29, 30.96.

HR-MS (ESI): m/z 314.1215 (Calcd. $[M+H]^+$), 314.1209 (Found. $[M+H]^+$).

20c: 

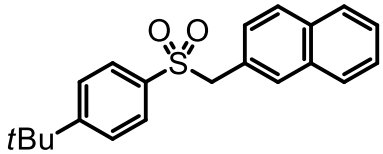
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.61-7.59 (m, 4H), 7.51 (d, J = 8.8 Hz, 1H), 7.28-7.26 (m, 2H), 4.35 (s, 2H), 1.36 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 158.31, 134.61, 133.48, 132.15, 131.54, 128.30, 126.15, 118.21, 112.70, 62.46, 30.97, 27.36.

HR-MS (ESI): m/z 314.1215 (Calcd. $[M+H]^+$), 314.1213 (Found. $[M+H]^+$).

21: 

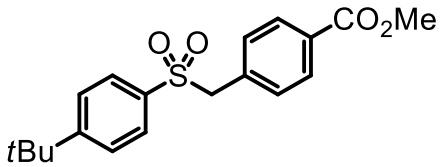
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.56 (d, J = 8.8 Hz, 2H), 7.47 (d, J = 8.8 Hz, 2H), 7.10-7.07 (m, 2H), 6.98-6.94 (m, 2H), 4.25 (s, 2H), 1.33 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 164.25, 157.86, 132.61, 132.52, 128.40, 125.95, 115.66, 115.45, 61.99, 31.01, 27.38; ^{19}F NMR ($CDCl_3$, 376 MHz, 298 K): δ = -112.61.

HR-MS (ESI): m/z 307.1168 (Calcd. $[M+H]^+$), 307.1163 (Found. $[M+H]^+$).

22: 

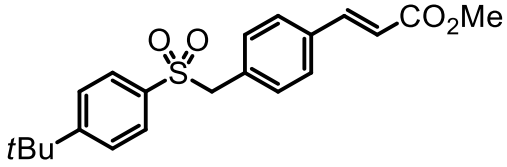
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.82 (d, J = 7.6 Hz, 2H), 7.75 (d, J = 8.4 Hz, 2H), 7.68 (d, J = 7.6 Hz, 2H), 7.55 (d, J = 8.4 Hz, 2H), 7.50-7.46 (m, 3H), 7.41 (d, J = 8.8 Hz, 2H), 7.24 (dd, J = 8.4 Hz, J = 1.6 Hz, 1H), 4.45 (s, 2H), 1.31 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 157.50, 134.68, 132.85, 132.82, 130.39, 128.01, 127.76, 127.70, 127.48, 126.43, 126.16, 125.70, 125.56, 62.88, 30.82, 27.21.

HR-MS (ESI): m/z 339.1419 (Calcd. $[M+H]^+$), 339.1413 (Found. $[M+H]^+$).

23: 

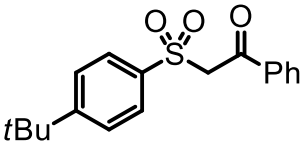
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.93 (d, J = 8.0 Hz, 2H), 7.56 (d, J = 8.8 Hz, 1H), 7.46 (d, J = 8.8 Hz, 2H), 7.18 (d, J = 8.4 Hz, 2H), 4.34 (s, 2H), 3.92 (s, 3H), 1.33 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 166.46, 157.92, 134.69, 133.17, 130.80, 130.29, 129.57, 128.32, 125.94, 62.53, 52.16, 35.18, 30.93.

HR-MS (ESI): m/z 347.1317 (Calcd. $[M+H]^+$), 347.1315 (Found. $[M+H]^+$).

24: 

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.67-7.57 (m, 3H), 7.48-7.41 (m, 4H), 7.14 (d, J = 8.4 Hz, 2H), 6.43 (d, J = 16 Hz, 1H), 4.30 (s, 2H), 3.81 (s, 3H), 1.33 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 167.11, 157.85, 143.81, 134.88, 134.66, 131.31, 130.21, 128.32, 128.01, 125.92, 118.59, 62.54, 51.70, 35.18, 30.95.

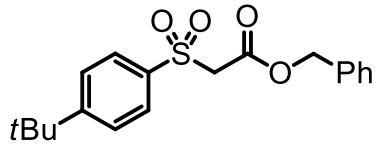
HR-MS (ESI): m/z 395.1293 (Calcd. $[M+Na]^+$), 395.1288 (Found. $[M+Na]^+$).

25a: 

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.92 (d, J = 7.2 Hz, 2H), 7.80 (d, J = 8.4 Hz, 2H),

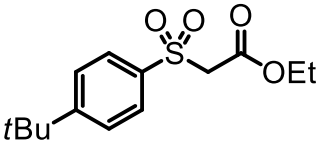
7.62-7.59 (m, 1H), 7.53 (d, *J* = 8.4 Hz, 2H), 7.46 (t, *J* = 8.0 Hz, 2H), 4.72 (s, 2H), 1.34 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 188.06, 158.15, 135.77, 134.18, 129.19, 128.74, 128.37, 126.16, 63.43, 30.96, 27.34.

HR-MS (ESI): m/z 317.1211 (Calcd. $[M+H]^+$), 317.1219 (Found. $[M+H]^+$).

25b: 

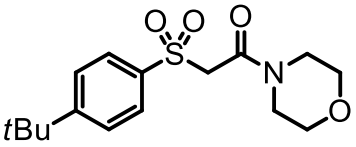
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.78 (d, *J* = 8.4 Hz, 2H), 7.49 (d, *J* = 8.8 Hz, 2H), 7.38-7.34 (m, 3H), 7.30-7,27 (m, 2H), 5.12 (s, 2H), 4.14 (s, 2H), 1.33 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 162.26, 158.13, 135.53, 134.41, 128.51, 128.28, 127.96, 127.12, 126.11, 67.89, 60.89, 35.18, 30.91.

HR-MS (ESI): m/z 369.1136 (Calcd. $[M+Na]^+$), 369.1131 (Found. $[M+Na]^+$).

26: 

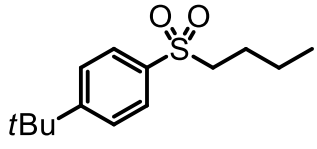
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.86 (d, *J* = 8.4 Hz, 2H), 7.58 (d, *J* = 8.8 Hz, 2H), 4.14 (q, *J* = 14.4 Hz, *J* = 7.2 Hz, 2H), 4.10 (s, 2H), 1.35 (s, 9H), 1.18 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 162.33, 158.14, 135.62, 128.27, 126.08, 62.13, 60.95, 35.17, 30.88, 13.69.

HR-MS (ESI): m/z 307.0980 (Calcd. $[M+Na]^+$), 307.0975 (Found. $[M+Na]^+$).

27: 

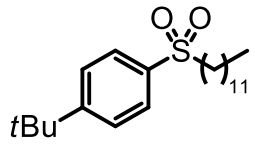
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.84 (d, *J* = 8.4 Hz, 2H), 7.58 (d, *J* = 8.8 Hz, 2H), 4.22 (s, 2H), 3.77 (t, *J* = 4.4 Hz, 2H), 3.70 (t, *J* = 4.0 Hz, 2H), 3.65-3.61 (m, 4H), 1.35 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 159.78, 158.11, 135.72, 128.06, 126.16, 66.45, 66.38, 59.36, 47.36, 42.50, 35.17, 30.87.

HR-MS (ESI): m/z 348.1245 (Calcd. $[M+Na]^+$), 348.1240 (Found. $[M+Na]^+$).

28a: 

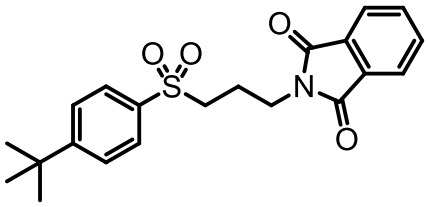
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.82 (d, J = 8.8 Hz, 2H), 7.57 (d, J = 8.8 Hz, 2H), 3.07 (t, J = 8.0 Hz, 2H), 1.74-1.67 (m, 2H), 1.44-1.37 (m, 2H), 1.36 (s, 9H), 0.90 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 157.39, 136.12, 127.80, 126.15, 56.03, 35.15, 30.98, 24.51, 21.46, 13.43.

HR-MS (ESI): m/z 277.1238 (Calcd. $[M+Na]^+$), 277.1233 (Found. $[M+Na]^+$).

28b: 

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.82 (d, J = 8.4 Hz, 2H), 7.56 (d, J = 8.8 Hz, 2H), 3.06 (t, J = 8.0 Hz, 2H), 1.75-1.68 (m, 2H), 1.35 (s, 9H), 1.32-1.22 (m, 18H), 0.87 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 157.46, 136.24, 127.88, 126.21, 56.37, 35.23, 31.88, 31.06, 29.55, 29.45, 29.30, 29.24, 28.99, 28.28, 22.66, 22.60, 14.10.

HR-MS (ESI): m/z 389.2490 (Calcd. $[M+Na]^+$), 389.2485 (Found. $[M+Na]^+$).

29: 

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.85-7.80 (m, 4H), 7.75-7.72 (m, 2H), 7.56 (d, J = 8.4 Hz, 2H), 3.78 (t, J = 6.8 Hz, 2H), 3.15 (t, J = 8.0 Hz, 2H), 2.15-2.08 (m, 2H), 1.34 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 168.09, 157.72, 135.63, 134.13, 131.79, 127.93, 126.33, 123.34, 53.96, 36.26, 35.21, 30.99, 22.39.

HR-MS (ESI): m/z 386.1426 (Calcd. $[M+H]^+$), 386.1421 (Found. $[M+H]^+$).

4. Crystal structure information for dimeric palladium intermediate 30.

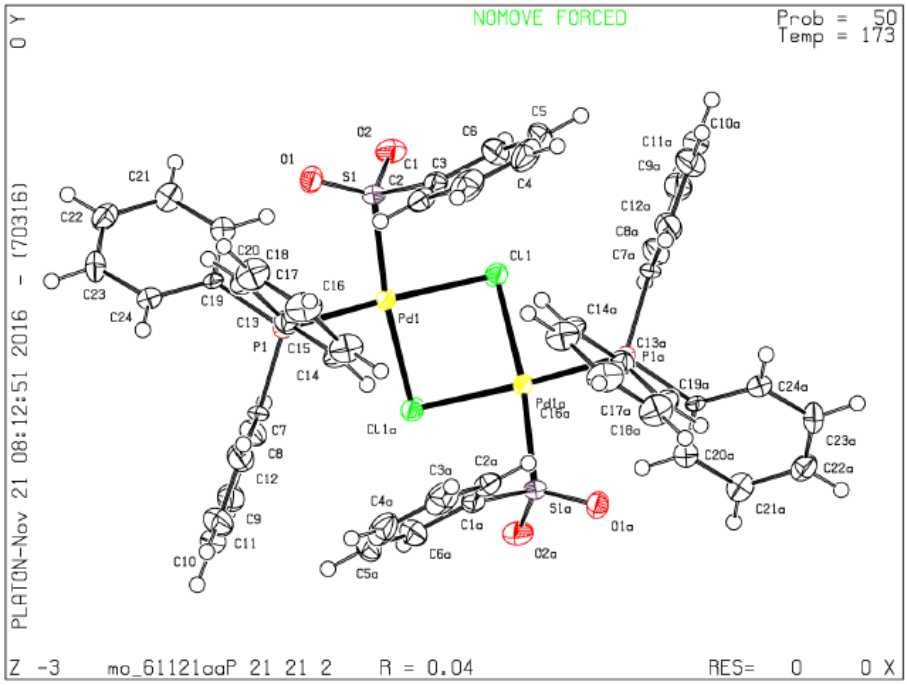


Figure S1 Molecular structure of palladium sulfinate dimer intermediate **30.**

Table 1. Crystal data and structure refinement for **30**.

Identification code	mo_61121aa	
Empirical formula	C48 H40 Cl2 O4 P2 Pd2 S2	
Formula weight	1090.56	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12$	
Unit cell dimensions	a = 15.3761(15) Å	= 90°.
	b = 17.6529(17) Å	= 90°.
	c = 8.9032(9) Å	= 90°.
Volume	2416.6(4) $Å^3$	
Z	2	
Density (calculated)	1.499 Mg/m^3	
Absorption coefficient	1.048 mm^{-1}	
F(000)	1096	
Crystal size	0.490 x 0.410 x 0.340 mm^3	
Theta range for data collection	1.756 to 27.538°.	
Index ranges	-11<=h<=19, -22<=k<=19, -11<=l<=6	

Reflections collected	12143
Independent reflections	5521 [R(int) = 0.0238]
Completeness to theta = 25.242°	98.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.647 and 0.585
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5521 / 0 / 271
Goodness-of-fit on F^2	1.101
Final R indices [I>2sigma(I)]	R1 = 0.0368, wR2 = 0.1282
R indices (all data)	R1 = 0.0397, wR2 = 0.1375
Absolute structure parameter	-0.004(15)
Extinction coefficient	n/a
Largest diff. peak and hole	0.768 and -1.257 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)

for **30**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Pd(1)	9129(1)	524(1)	6848(1)	17(1)
P(1)	7775(1)	259(1)	7717(2)	16(1)
S(1)	9044(1)	1728(1)	7671(2)	22(1)
Cl(1)	10591(1)	739(1)	5988(2)	25(1)
O(1)	8237(3)	1938(3)	8442(6)	27(1)
O(2)	9298(4)	2213(3)	6409(7)	36(1)
C(1)	9863(5)	1828(4)	9087(9)	24(1)
C(2)	9664(5)	1641(4)	10544(9)	28(2)
C(3)	10270(7)	1771(6)	11703(12)	47(2)
C(4)	11062(7)	2087(6)	11334(13)	49(3)
C(5)	11262(6)	2281(6)	9869(15)	52(3)
C(6)	10664(6)	2148(5)	8719(11)	35(2)
C(7)	7387(4)	-698(3)	7340(8)	20(1)
C(8)	7302(5)	-914(4)	5882(9)	27(2)
C(9)	6968(5)	-1641(5)	5498(11)	35(2)
C(10)	6705(5)	-2126(5)	6669(13)	40(2)
C(11)	6788(6)	-1907(5)	8107(13)	41(2)
C(12)	7123(5)	-1188(5)	8443(10)	33(2)
C(13)	7816(5)	325(4)	9696(8)	25(2)
C(14)	8464(5)	-87(4)	10500(8)	26(2)
C(15)	8579(6)	-4(5)	12033(9)	35(2)
C(16)	8052(6)	489(5)	12816(8)	36(2)
C(17)	7408(6)	893(5)	12095(9)	39(2)
C(18)	7285(5)	813(4)	10556(8)	28(2)
C(19)	6887(4)	843(4)	6961(10)	23(1)
C(20)	7046(5)	1417(4)	5960(8)	23(1)
C(21)	6358(5)	1818(5)	5336(8)	27(2)
C(22)	5510(5)	1635(5)	5739(9)	30(2)

C(23)	5353(5)	1073(5)	6715(12)	38(2)
C(24)	6038(5)	680(4)	7445(9)	27(2)

Table 3. Bond lengths [Å] and angles [°] for **30**.

Pd(1)-S(1)	2.2516(17)
Pd(1)-P(1)	2.2699(18)
Pd(1)-Cl(1)#1	2.3963(17)
Pd(1)-Cl(1)	2.4047(17)
Pd(1)-Pd(1)#1	3.2551(9)
P(1)-C(13)	1.767(7)
P(1)-C(7)	1.822(6)
P(1)-C(19)	1.838(7)
S(1)-O(1)	1.465(5)
S(1)-O(2)	1.466(6)
S(1)-C(1)	1.791(8)
Cl(1)-Pd(1)#1	2.3963(17)
C(1)-C(2)	1.372(11)
C(1)-C(6)	1.394(11)
C(2)-C(3)	1.410(12)
C(3)-C(4)	1.379(16)
C(4)-C(5)	1.383(18)
C(5)-C(6)	1.396(14)
C(7)-C(8)	1.360(10)
C(7)-C(12)	1.371(11)
C(8)-C(9)	1.423(11)
C(9)-C(10)	1.409(14)
C(10)-C(11)	1.344(16)
C(11)-C(12)	1.402(12)
C(13)-C(18)	1.413(11)
C(13)-C(14)	1.427(10)
C(14)-C(15)	1.384(11)
C(15)-C(16)	1.378(12)
C(16)-C(17)	1.379(13)
C(17)-C(18)	1.390(11)
C(19)-C(20)	1.371(11)
C(19)-C(24)	1.405(10)
C(20)-C(21)	1.388(10)
C(21)-C(22)	1.391(12)
C(22)-C(23)	1.341(13)
C(23)-C(24)	1.419(11)

S(1)-Pd(1)-P(1)	91.73(6)
S(1)-Pd(1)-Cl(1)#1	172.99(7)
P(1)-Pd(1)-Cl(1)#1	94.70(6)
S(1)-Pd(1)-Cl(1)	90.53(6)
P(1)-Pd(1)-Cl(1)	176.80(6)
Cl(1)#1-Pd(1)-Cl(1)	82.93(7)
S(1)-Pd(1)-Pd(1)#1	125.76(5)
P(1)-Pd(1)-Pd(1)#1	129.62(5)
Cl(1)#1-Pd(1)-Pd(1)#1	47.42(4)
Cl(1)-Pd(1)-Pd(1)#1	47.20(4)
C(13)-P(1)-C(7)	104.9(3)
C(13)-P(1)-C(19)	110.7(4)
C(7)-P(1)-C(19)	102.1(3)
C(13)-P(1)-Pd(1)	107.1(3)
C(7)-P(1)-Pd(1)	115.4(2)
C(19)-P(1)-Pd(1)	116.1(2)
O(1)-S(1)-O(2)	115.9(3)
O(1)-S(1)-C(1)	103.9(3)
O(2)-S(1)-C(1)	107.1(4)
O(1)-S(1)-Pd(1)	116.2(2)
O(2)-S(1)-Pd(1)	106.6(3)
C(1)-S(1)-Pd(1)	106.3(2)
Pd(1)#1-Cl(1)-Pd(1)	85.37(6)
C(2)-C(1)-C(6)	121.1(8)
C(2)-C(1)-S(1)	119.0(6)
C(6)-C(1)-S(1)	119.7(6)
C(1)-C(2)-C(3)	120.3(9)
C(4)-C(3)-C(2)	118.4(10)
C(3)-C(4)-C(5)	121.4(9)
C(4)-C(5)-C(6)	120.3(9)
C(1)-C(6)-C(5)	118.5(9)
C(8)-C(7)-C(12)	118.6(7)
C(8)-C(7)-P(1)	117.9(5)
C(12)-C(7)-P(1)	123.4(6)
C(7)-C(8)-C(9)	121.1(8)
C(10)-C(9)-C(8)	118.3(8)
C(11)-C(10)-C(9)	120.2(8)

C(10)-C(11)-C(12)	119.9(9)
C(7)-C(12)-C(11)	121.9(9)
C(18)-C(13)-C(14)	116.3(6)
C(18)-C(13)-P(1)	124.1(6)
C(14)-C(13)-P(1)	119.4(6)
C(15)-C(14)-C(13)	122.0(7)
C(16)-C(15)-C(14)	119.4(8)
C(15)-C(16)-C(17)	120.8(7)
C(16)-C(17)-C(18)	120.3(8)
C(17)-C(18)-C(13)	121.1(8)
C(20)-C(19)-C(24)	121.1(7)
C(20)-C(19)-P(1)	121.4(5)
C(24)-C(19)-P(1)	117.5(6)
C(19)-C(20)-C(21)	120.0(7)
C(20)-C(21)-C(22)	119.5(7)
C(23)-C(22)-C(21)	120.5(7)
C(22)-C(23)-C(24)	121.7(7)
C(19)-C(24)-C(23)	116.7(7)

Symmetry transformations used to generate equivalent atoms:

#1 -x+2,-y,z

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **30**. The anisotropic displacement factor exponent takes the form: $-2\ ^2[\ h^2\ a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pd(1)	16(1)	17(1)	18(1)	1(1)	0(1)	2(1)
P(1)	19(1)	17(1)	13(1)	1(1)	1(1)	1(1)
S(1)	23(1)	15(1)	27(1)	2(1)	0(1)	1(1)
Cl(1)	23(1)	24(1)	29(1)	8(1)	5(1)	3(1)
O(1)	21(2)	30(3)	29(3)	-6(2)	-1(2)	3(2)
O(2)	40(3)	23(3)	46(3)	7(2)	2(3)	1(2)
C(1)	24(3)	19(3)	29(4)	-9(3)	-3(3)	2(3)
C(2)	32(4)	23(3)	29(4)	-2(3)	-5(3)	7(3)
C(3)	50(5)	45(5)	47(5)	-20(5)	-8(5)	16(4)
C(4)	38(5)	44(5)	65(6)	-27(5)	-24(5)	16(4)
C(5)	26(4)	36(5)	94(9)	-26(5)	-9(5)	-6(4)
C(6)	31(4)	29(4)	44(4)	-4(3)	1(4)	-5(3)
C(7)	18(3)	9(3)	32(3)	1(2)	-3(3)	-2(2)
C(8)	29(4)	29(4)	24(3)	-5(3)	3(3)	-6(3)
C(9)	31(4)	32(4)	43(5)	-13(4)	-1(4)	-2(3)
C(10)	27(4)	25(4)	67(6)	-4(4)	-1(4)	-1(3)
C(11)	36(4)	34(4)	54(5)	9(4)	1(5)	-8(4)
C(12)	30(4)	33(4)	36(4)	3(3)	-3(3)	-4(3)
C(13)	23(3)	23(3)	28(4)	7(3)	-1(3)	-3(3)
C(14)	37(4)	22(3)	21(3)	-2(3)	-2(3)	6(3)
C(15)	48(5)	34(4)	21(3)	3(3)	-5(3)	3(4)
C(16)	57(5)	36(4)	16(3)	3(3)	2(3)	2(4)
C(17)	48(5)	42(5)	26(4)	-9(3)	6(4)	4(4)
C(18)	32(4)	27(3)	24(3)	0(3)	2(3)	6(3)
C(19)	17(3)	10(3)	42(4)	-7(3)	-5(3)	2(2)
C(20)	25(3)	27(4)	16(3)	-4(3)	0(3)	0(3)
C(21)	32(4)	34(4)	16(3)	2(3)	-2(3)	4(3)

C(22)	30(4)	29(4)	32(4)	-6(3)	-8(3)	8(3)
C(23)	17(3)	41(4)	56(5)	3(4)	-3(4)	1(3)
C(24)	26(4)	19(3)	35(4)	0(3)	-1(3)	2(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **30**.

	x	y	z	U(eq)
H(2)	9115	1422	10772	34
H(3)	10137	1644	12714	56
H(4)	11480	2172	12103	59
H(5)	11808	2506	9645	62
H(6)	10800	2273	7708	42
H(8)	7469	-575	5106	33
H(9)	6923	-1794	4478	42
H(10)	6469	-2610	6440	48
H(11)	6619	-2239	8895	50
H(12)	7169	-1037	9464	40
H(14)	8828	-429	9968	32
H(15)	9016	-284	12541	42
H(16)	8134	552	13866	44
H(17)	7047	1228	12652	46
H(18)	6835	1092	10075	33
H(20)	7627	1541	5693	27
H(21)	6466	2214	4639	33
H(22)	5038	1910	5320	36
H(23)	4768	934	6924	46
H(24)	5929	324	8223	32

Table 6. Torsion angles [°] for **30**.

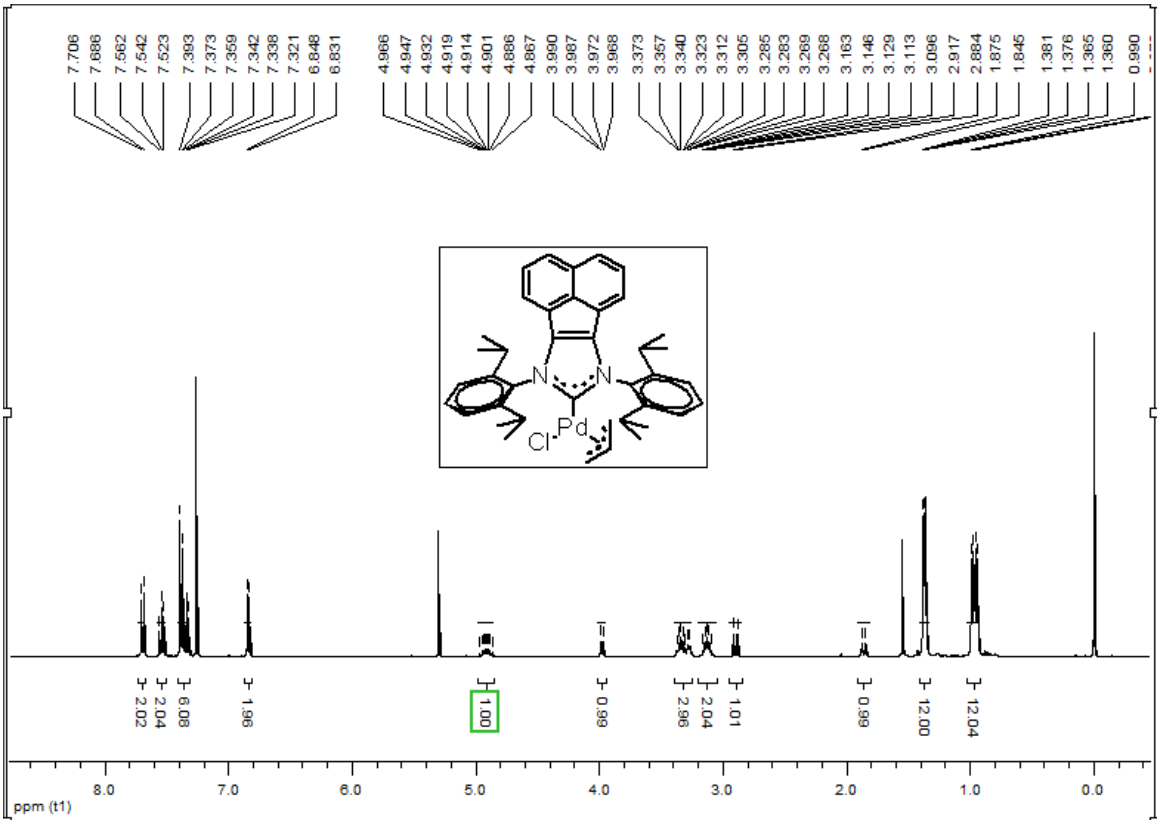
O(1)-S(1)-C(1)-C(2)	36.0(7)
O(2)-S(1)-C(1)-C(2)	159.2(6)
Pd(1)-S(1)-C(1)-C(2)	-87.1(6)
O(1)-S(1)-C(1)-C(6)	-138.9(6)
O(2)-S(1)-C(1)-C(6)	-15.8(7)
Pd(1)-S(1)-C(1)-C(6)	97.9(6)
C(6)-C(1)-C(2)-C(3)	0.1(12)
S(1)-C(1)-C(2)-C(3)	-174.8(6)
C(1)-C(2)-C(3)-C(4)	-0.2(12)
C(2)-C(3)-C(4)-C(5)	0.7(14)
C(3)-C(4)-C(5)-C(6)	-1.2(15)
C(2)-C(1)-C(6)-C(5)	-0.5(12)
S(1)-C(1)-C(6)-C(5)	174.4(7)
C(4)-C(5)-C(6)-C(1)	1.0(13)
C(13)-P(1)-C(7)-C(8)	-179.1(6)
C(19)-P(1)-C(7)-C(8)	65.3(7)
Pd(1)-P(1)-C(7)-C(8)	-61.6(6)
C(13)-P(1)-C(7)-C(12)	5.4(7)
C(19)-P(1)-C(7)-C(12)	-110.2(7)
Pd(1)-P(1)-C(7)-C(12)	122.9(6)
C(12)-C(7)-C(8)-C(9)	-1.6(12)
P(1)-C(7)-C(8)-C(9)	-177.3(6)
C(7)-C(8)-C(9)-C(10)	1.6(12)
C(8)-C(9)-C(10)-C(11)	-1.2(13)
C(9)-C(10)-C(11)-C(12)	1.0(14)
C(8)-C(7)-C(12)-C(11)	1.3(12)
P(1)-C(7)-C(12)-C(11)	176.8(7)
C(10)-C(11)-C(12)-C(7)	-1.1(14)
C(7)-P(1)-C(13)-C(18)	-114.5(7)
C(19)-P(1)-C(13)-C(18)	-5.1(8)
Pd(1)-P(1)-C(13)-C(18)	122.4(6)
C(7)-P(1)-C(13)-C(14)	70.7(6)
C(19)-P(1)-C(13)-C(14)	-179.8(6)
Pd(1)-P(1)-C(13)-C(14)	-52.3(6)
C(18)-C(13)-C(14)-C(15)	-1.2(12)
P(1)-C(13)-C(14)-C(15)	174.0(7)

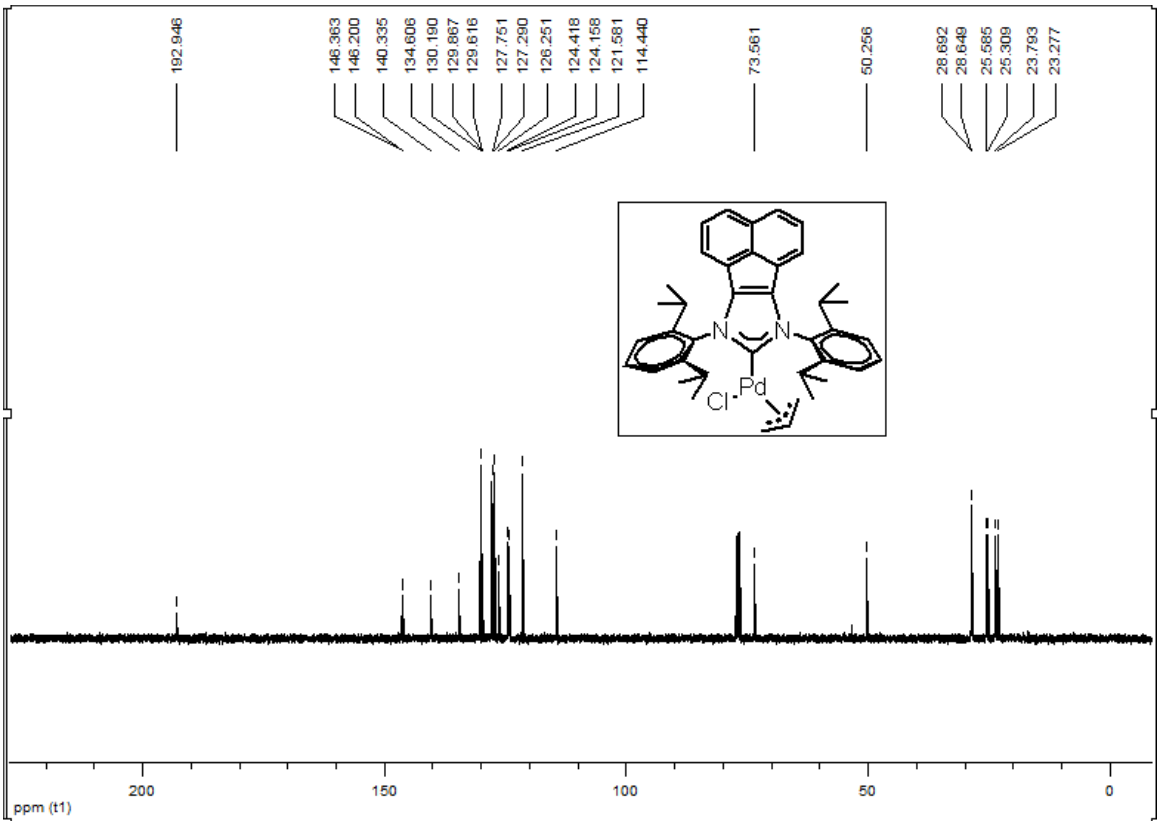
C(13)-C(14)-C(15)-C(16)	0.1(14)
C(14)-C(15)-C(16)-C(17)	0.8(14)
C(15)-C(16)-C(17)-C(18)	-0.6(15)
C(16)-C(17)-C(18)-C(13)	-0.5(14)
C(14)-C(13)-C(18)-C(17)	1.4(12)
P(1)-C(13)-C(18)-C(17)	-173.6(7)
C(13)-P(1)-C(19)-C(20)	120.8(6)
C(7)-P(1)-C(19)-C(20)	-128.0(6)
Pd(1)-P(1)-C(19)-C(20)	-1.6(7)
C(13)-P(1)-C(19)-C(24)	-59.3(7)
C(7)-P(1)-C(19)-C(24)	51.9(7)
Pd(1)-P(1)-C(19)-C(24)	178.3(5)
C(24)-C(19)-C(20)-C(21)	-4.0(11)
P(1)-C(19)-C(20)-C(21)	175.9(6)
C(19)-C(20)-C(21)-C(22)	0.3(11)
C(20)-C(21)-C(22)-C(23)	-0.5(12)
C(21)-C(22)-C(23)-C(24)	4.3(13)
C(20)-C(19)-C(24)-C(23)	7.4(11)
P(1)-C(19)-C(24)-C(23)	-172.5(6)
C(22)-C(23)-C(24)-C(19)	-7.6(13)

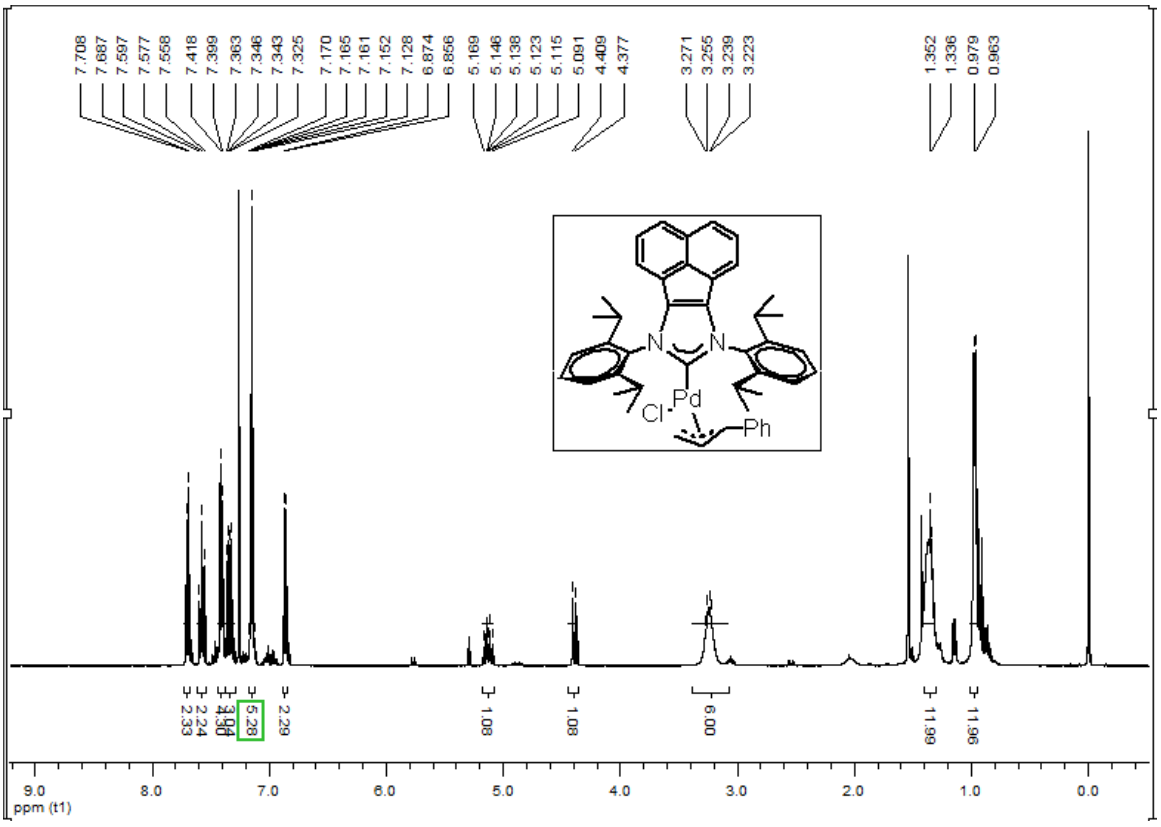
Symmetry transformations used to generate equivalent atoms:

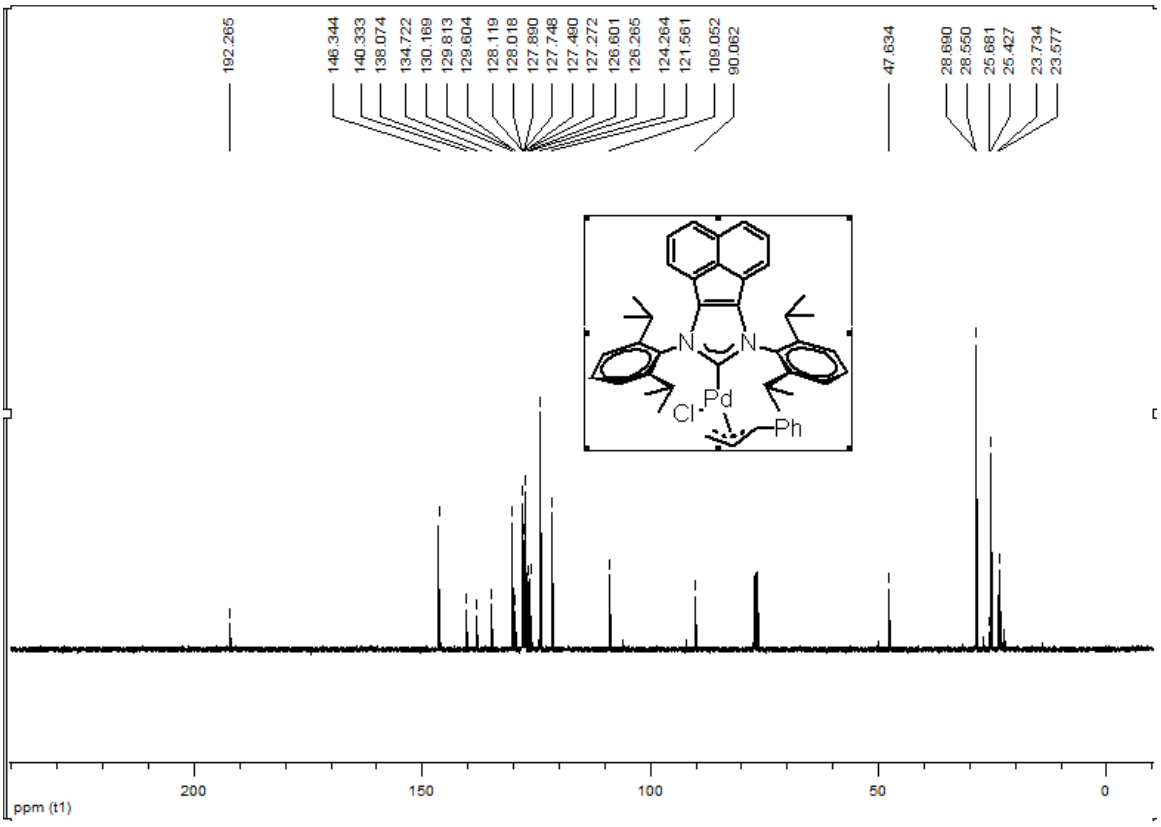
#1 -x+2,-y,z

5. ^{1}H NMR, ^{13}C NMR and MS spectra for important compounds.






7.708
7.687
7.597
7.577
7.558
7.418
7.399
7.363
7.346
7.343
7.325
7.170
7.165
7.161
7.152
7.128
6.874
6.856
5.169
5.146
5.138
5.123
5.115
5.091
4.409
4.377
3.271
3.255
3.239
3.223
1.352
1.336
0.979
0.963
Cl
Pd
Ph
2.33
2.24
1.30
5.28
2.29
1.08
1.08
6.00
11.99
11.96
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


192.265
146.344
140.333
138.074
134.722
130.169
129.813
129.604
128.119
128.018
127.890
127.748
127.490
127.272
126.601
126.265
124.264
121.561
109.052
90.062
47.634
28.690
28.550
25.681
25.427
23.734
23.577
Cl
Pd
Ph
200
150
100
50
0
ppm (t1)

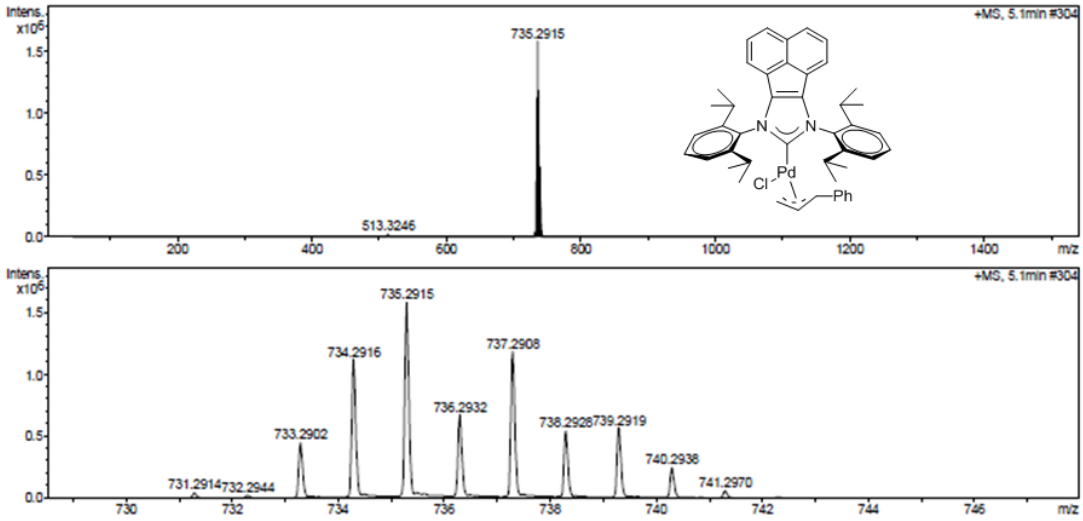
Display Report

Analysis Info

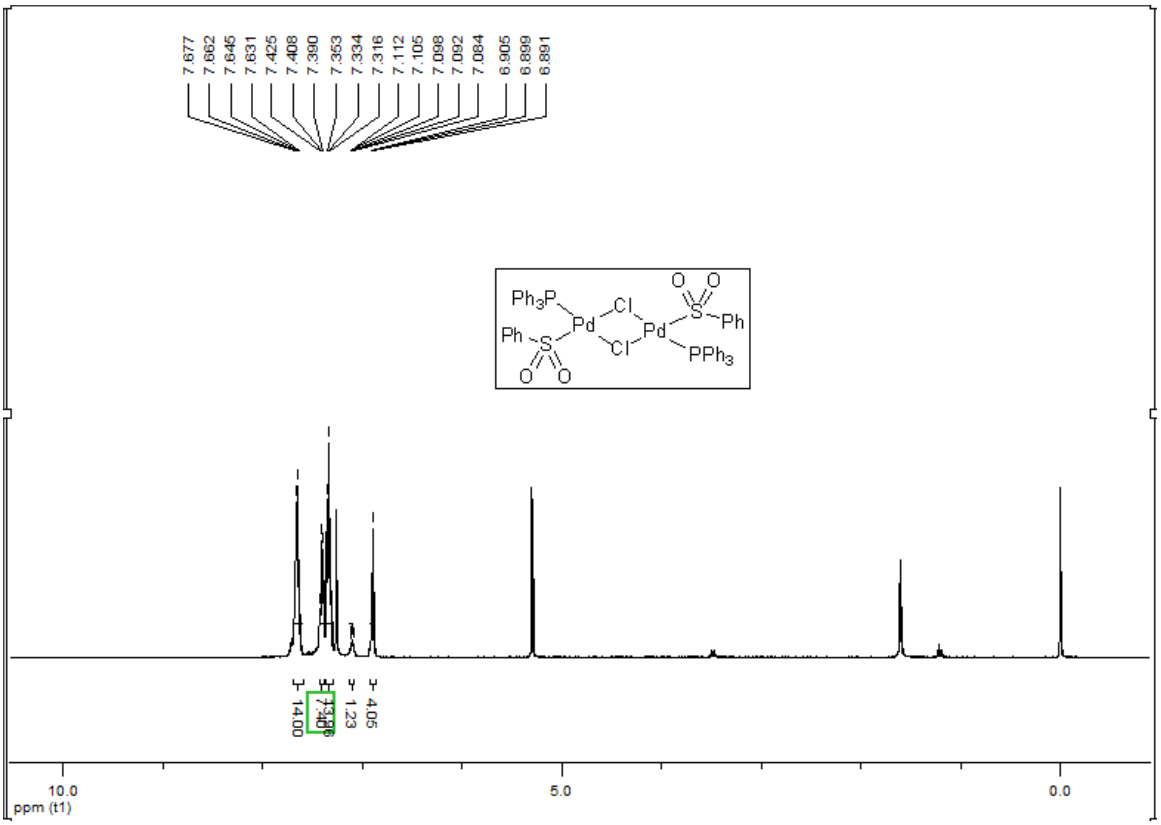
Analysis Name	D:\Data\2017MS\TT\0612\dq-33_BC5_01_538.d	Acquisition Date	6/14/2017 10:53:25 AM
Method	tune_200-800_hcoona-pos-8min.m	Operator	gftang
Sample Name	dq-33	Instrument / Ser#	micrOTOF II 10257
Comment			

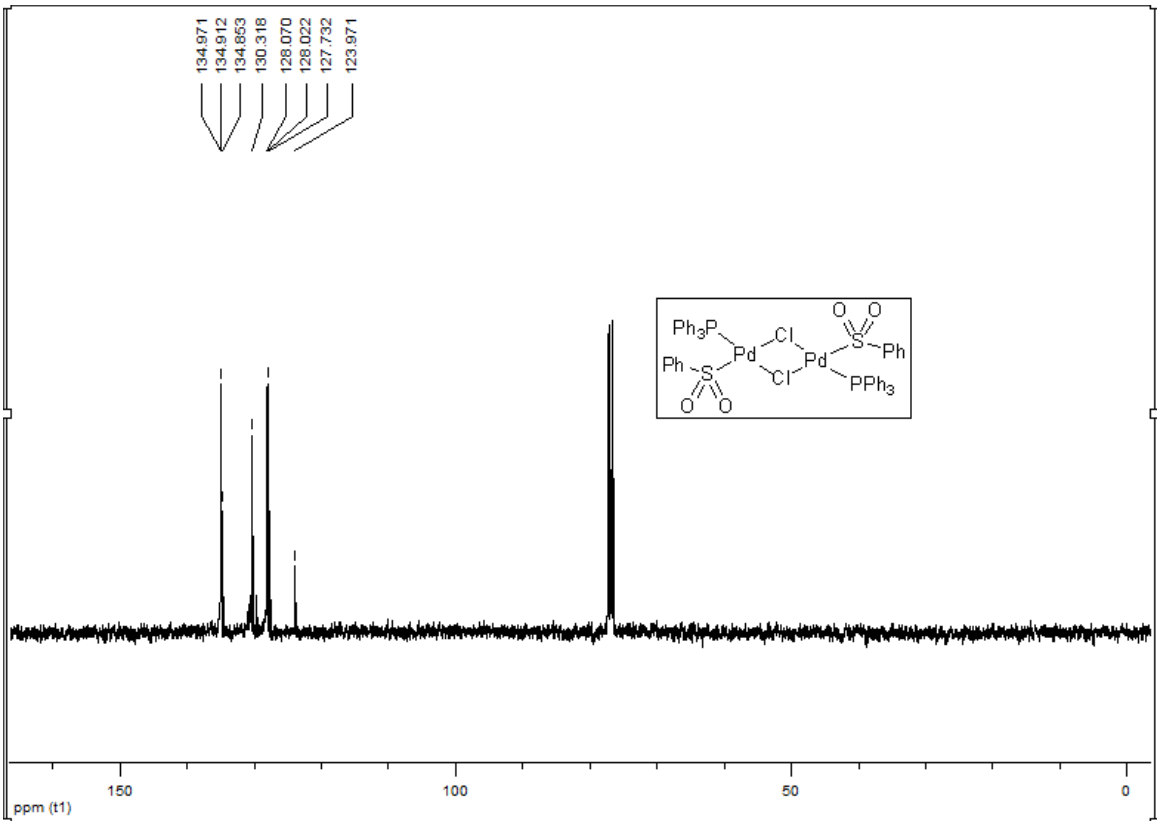
Acquisition Parameter

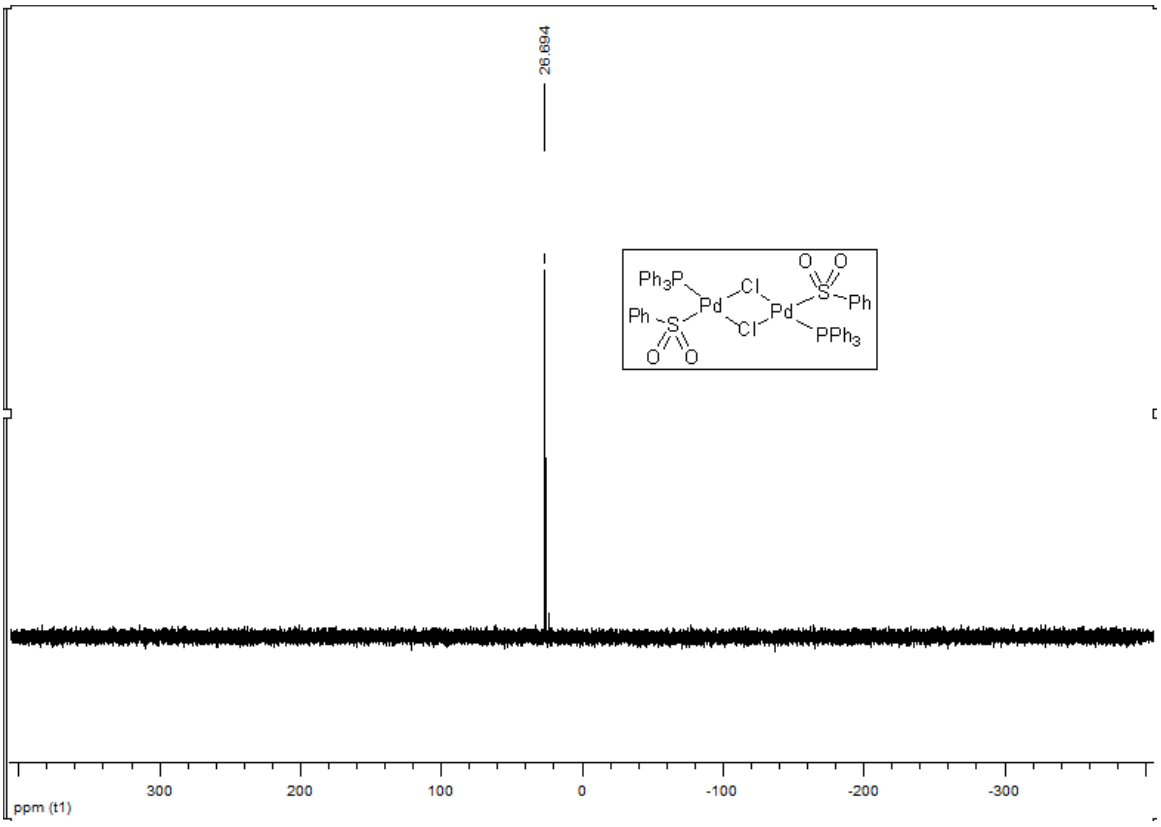
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	2500 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

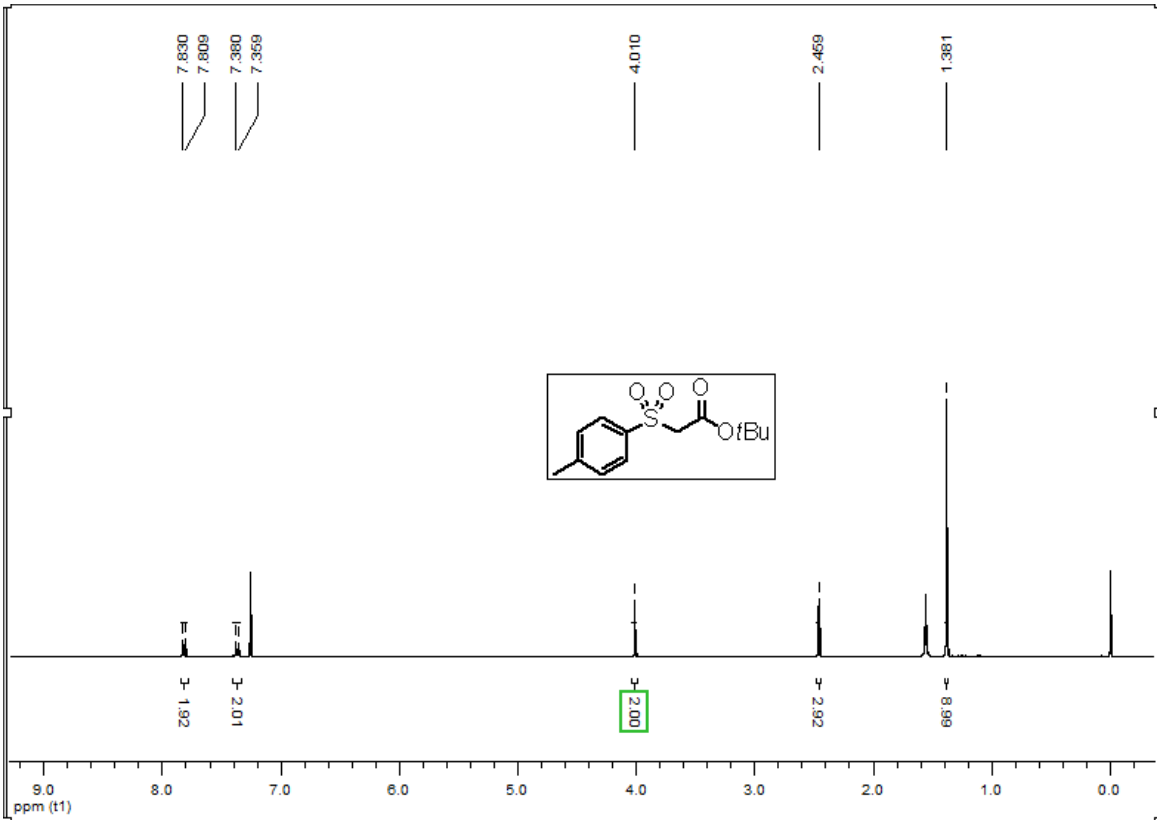


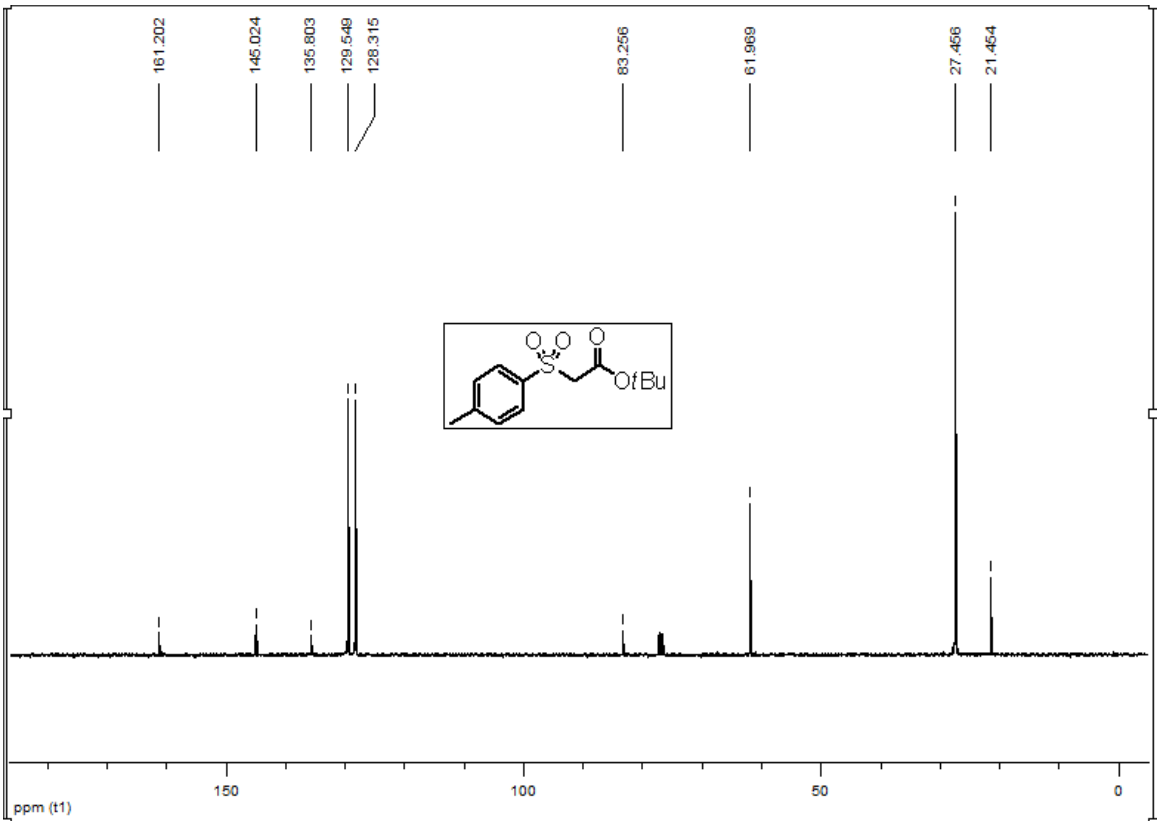
Bruker Compass DataAnalysis 4.0 printed: 6/14/2017 11:05:29 AM Page 1 of 1

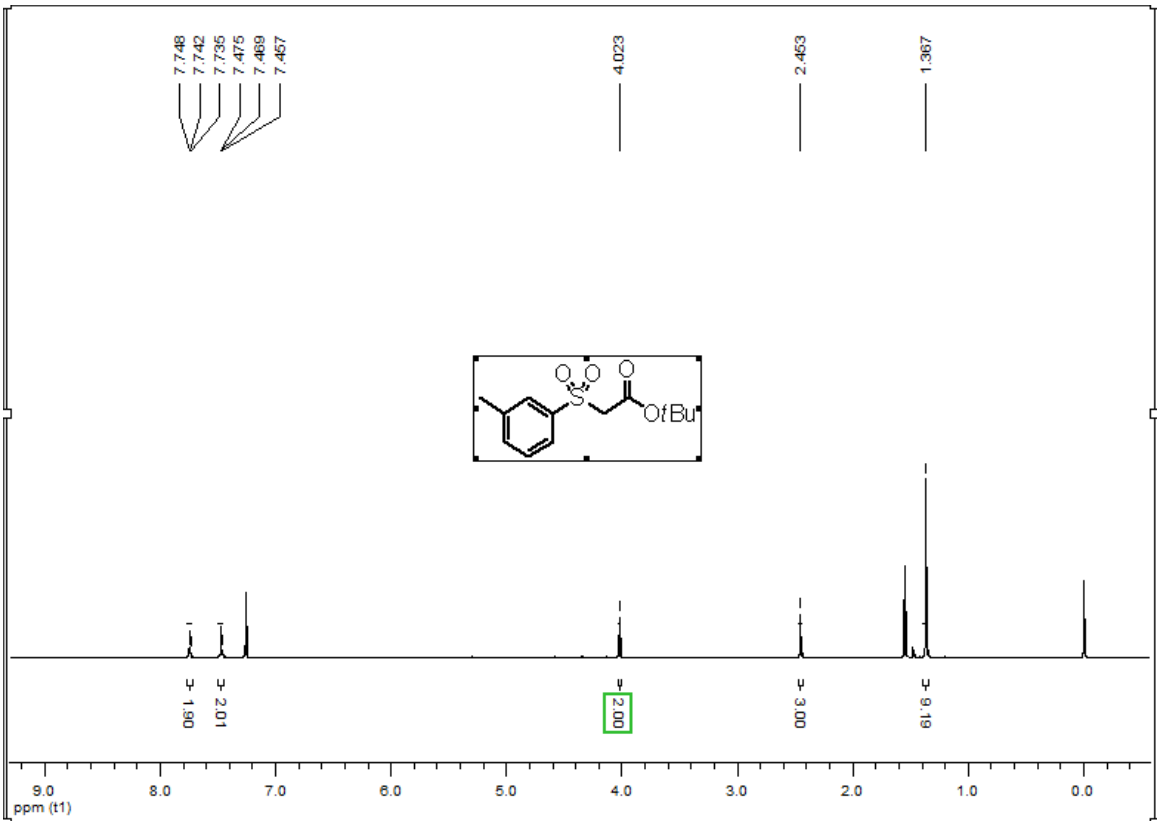


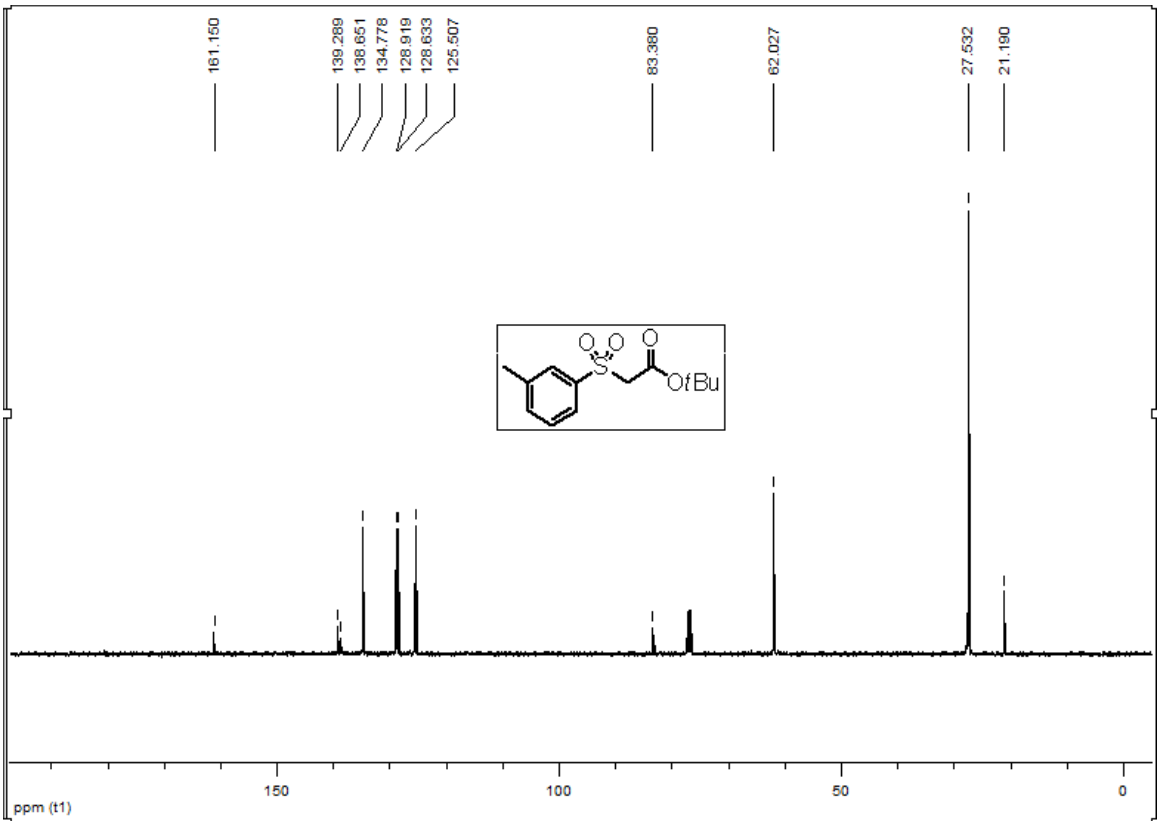

134.971
134.912
134.853
130.318
128.070
128.022
127.732
123.971
Ph3P
Pd
Cl
Pd
S
Ph
Ph
S
Cl
PPh3
O
O
O
O
150
100
50
0
ppm (t1)

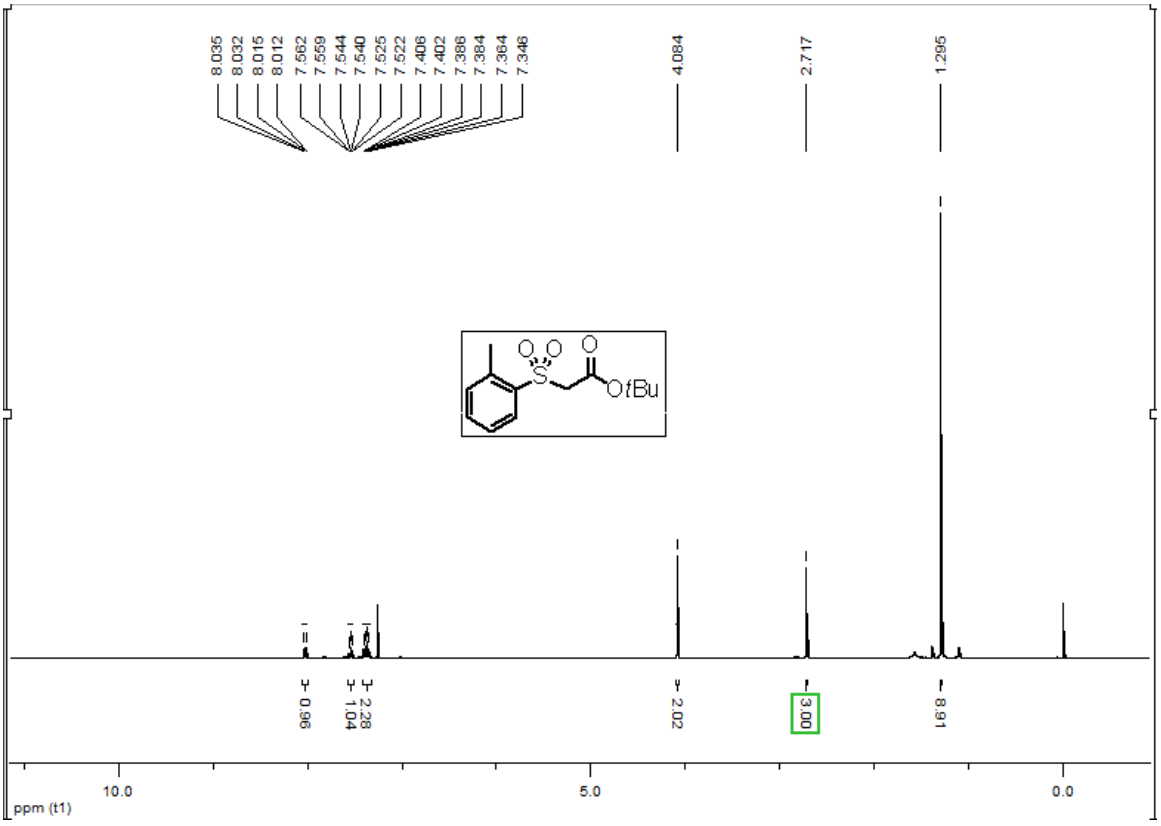

26.894
Ph3P
Pd
Cl
Pd
S
Ph
Ph
S
Cl
PPh3
O
O
O
O
300
200
100
0
-100
-200
-300
ppm (t1)

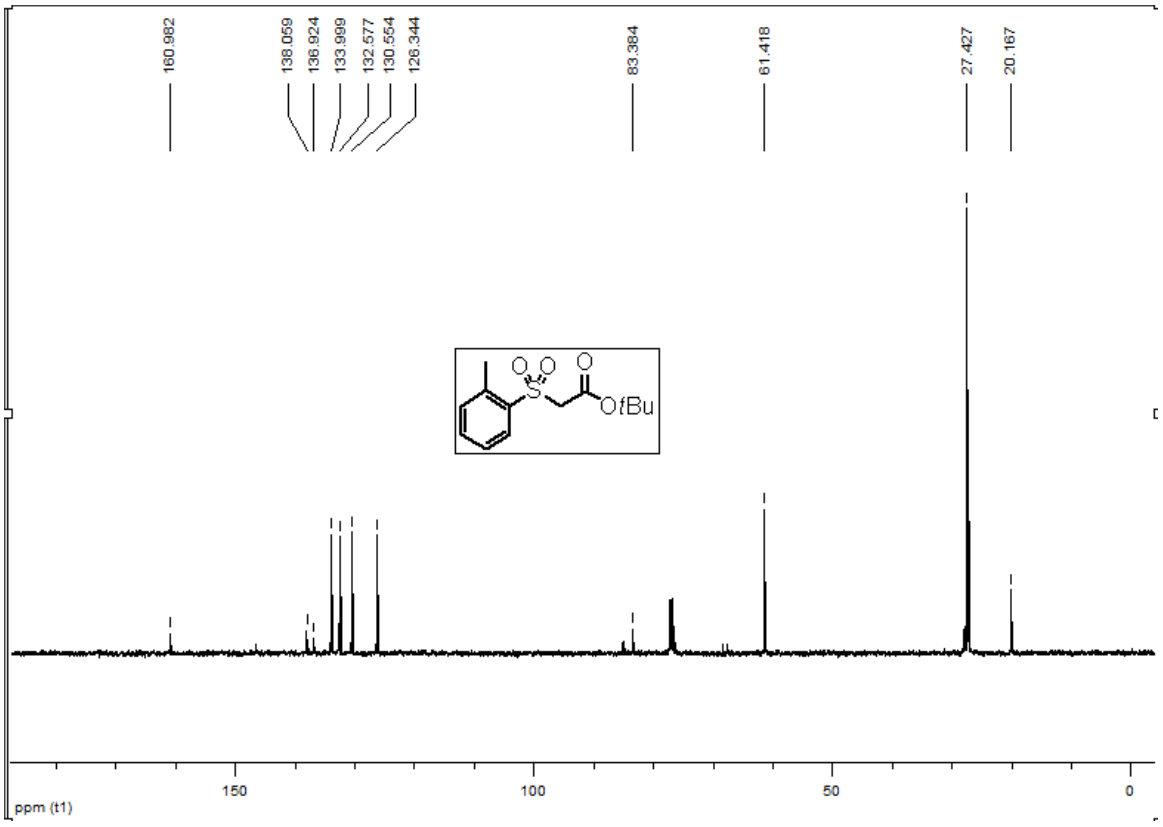

7.830
7.809
7.380
7.359
4.010
2.459
1.381
OtBu
1.92
2.01
2.00
2.92
8.99
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

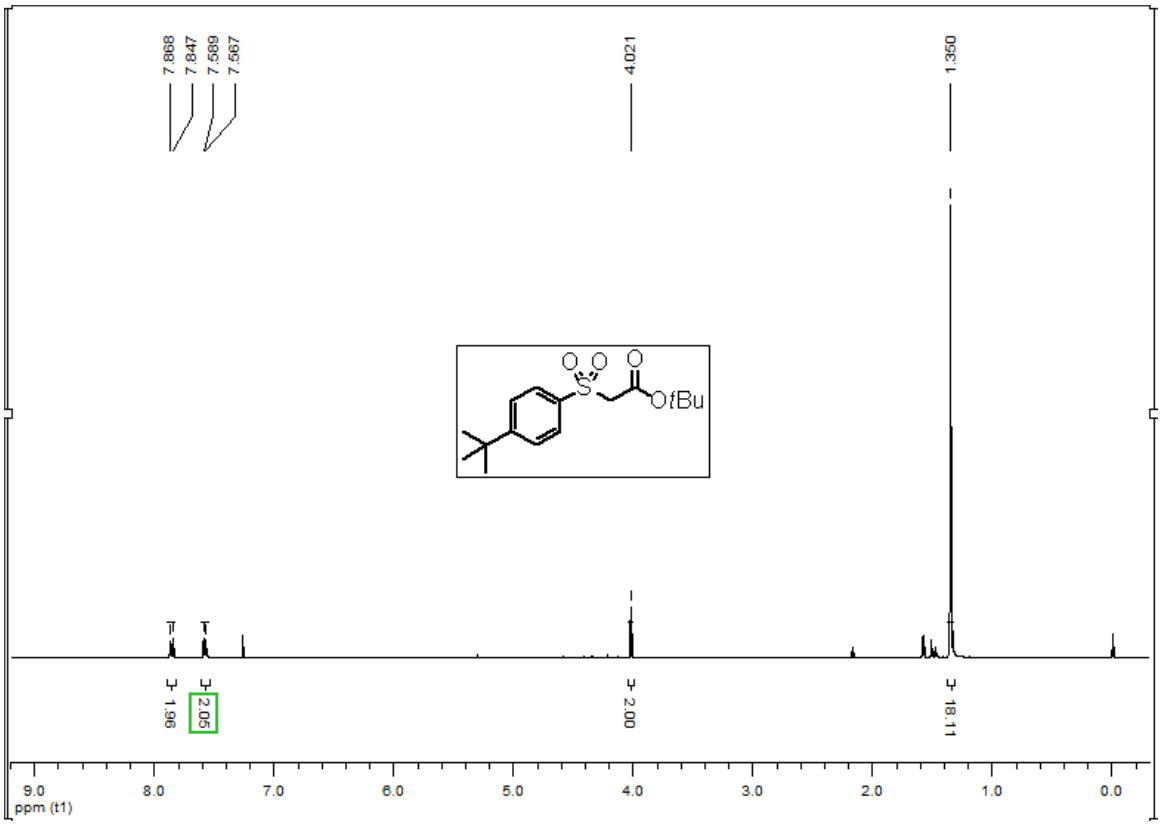

161.202
145.024
135.803
129.549
128.315
83.256
61.969
27.456
21.454
OtBu
150
100
50
0
ppm (t1)

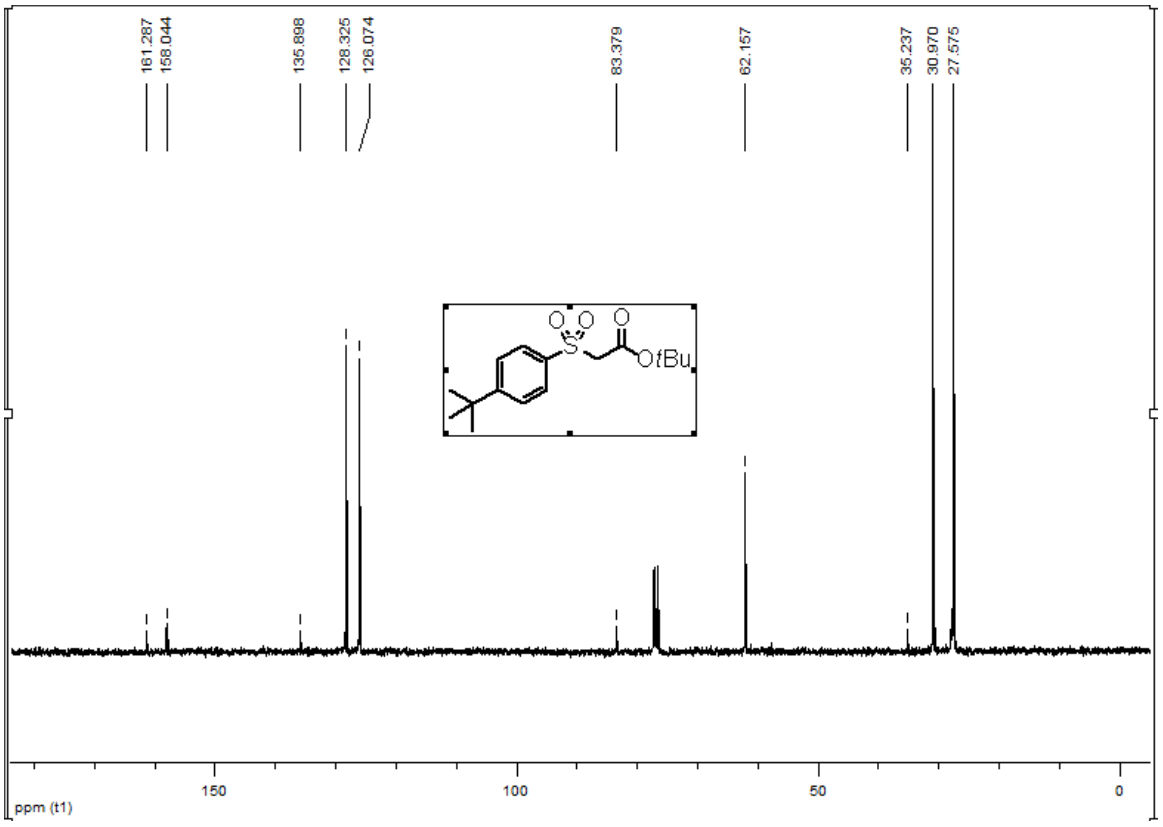

7.748
7.742
7.735
7.475
7.469
7.457
4.023
2.453
1.367
OtBu
1.00
2.01
2.00
3.00
9.19
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

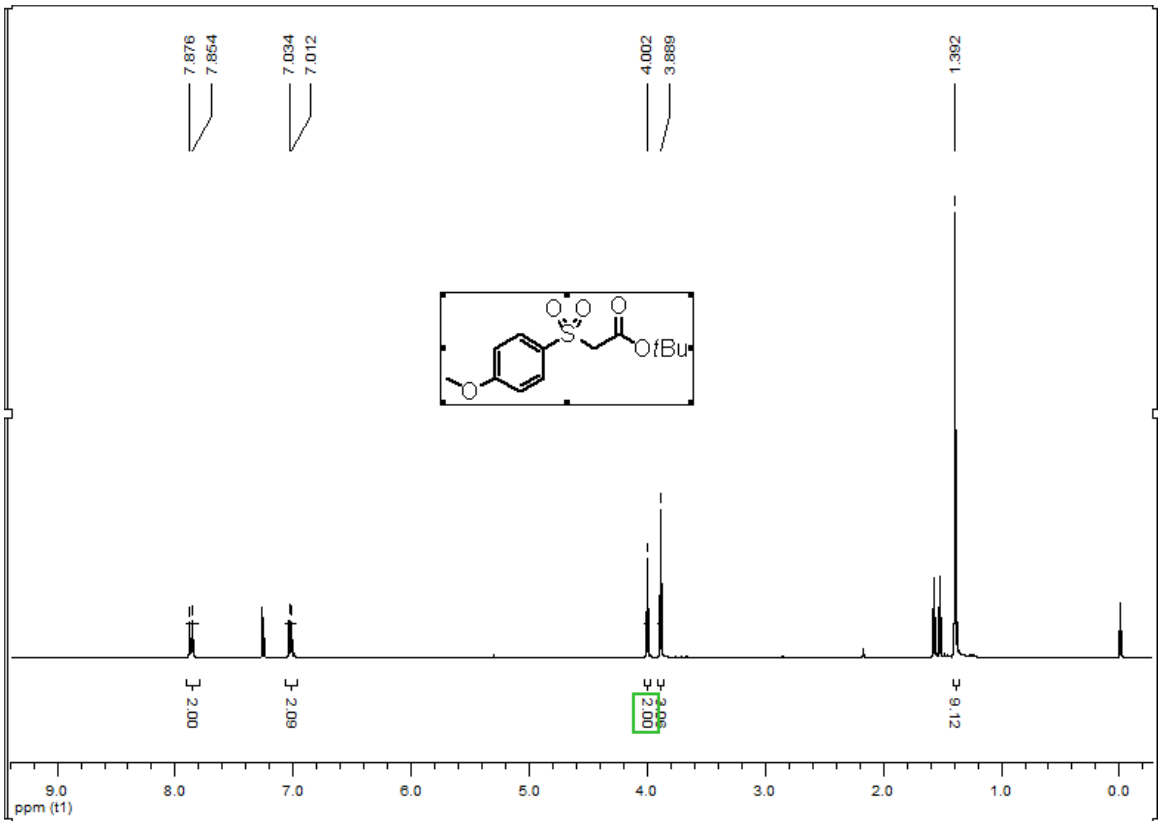

161.150
139.289
138.651
134.778
128.919
128.633
125.507
83.380
62.027
27.532
21.190
OtBu
150
100
50
0
ppm (t1)

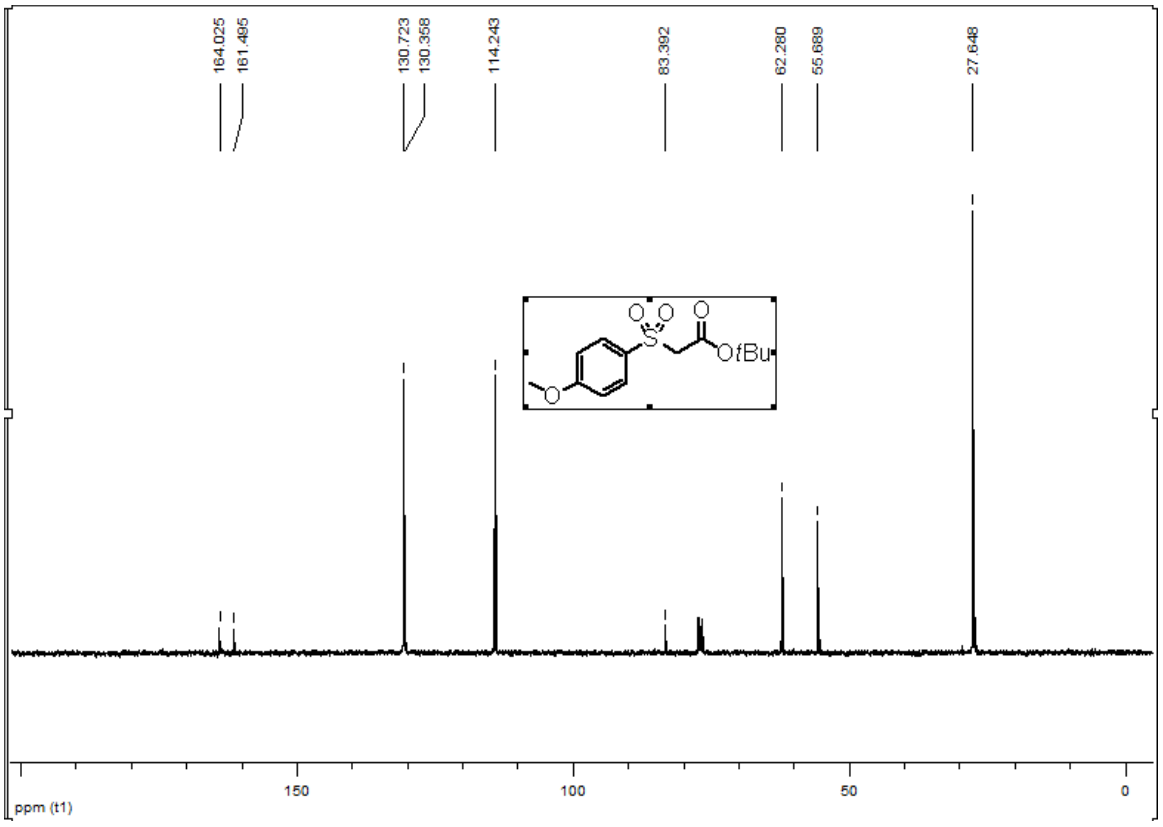

8.035
8.032
8.015
8.012
7.562
7.559
7.544
7.540
7.525
7.522
7.406
7.402
7.386
7.384
7.364
7.346
4.084
2.717
1.295
OtBu
0.96
2.28
1.04
2.02
3.00
8.91
10.0
5.0
0.0
ppm (t1)

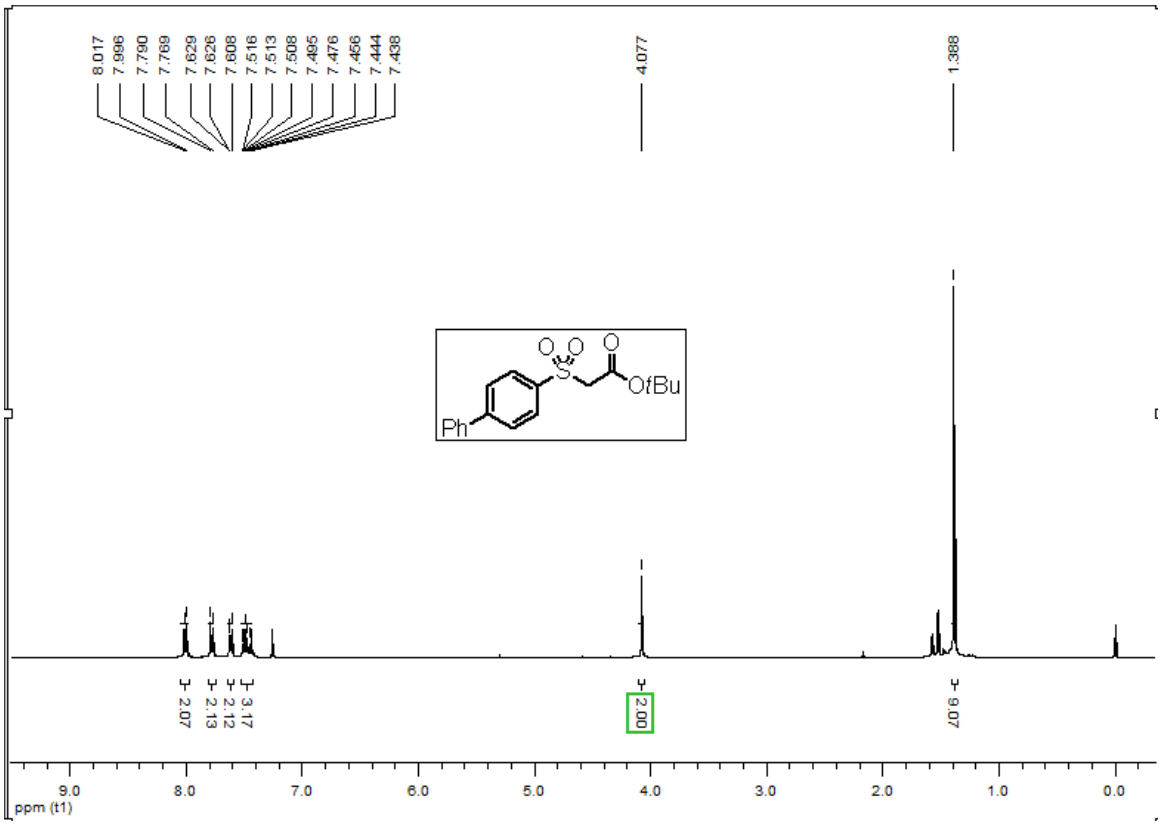

160.982
138.059
136.924
133.999
132.577
130.554
126.344
83.384
61.418
27.427
20.167
OtBu
150
100
50
0
ppm (t1)

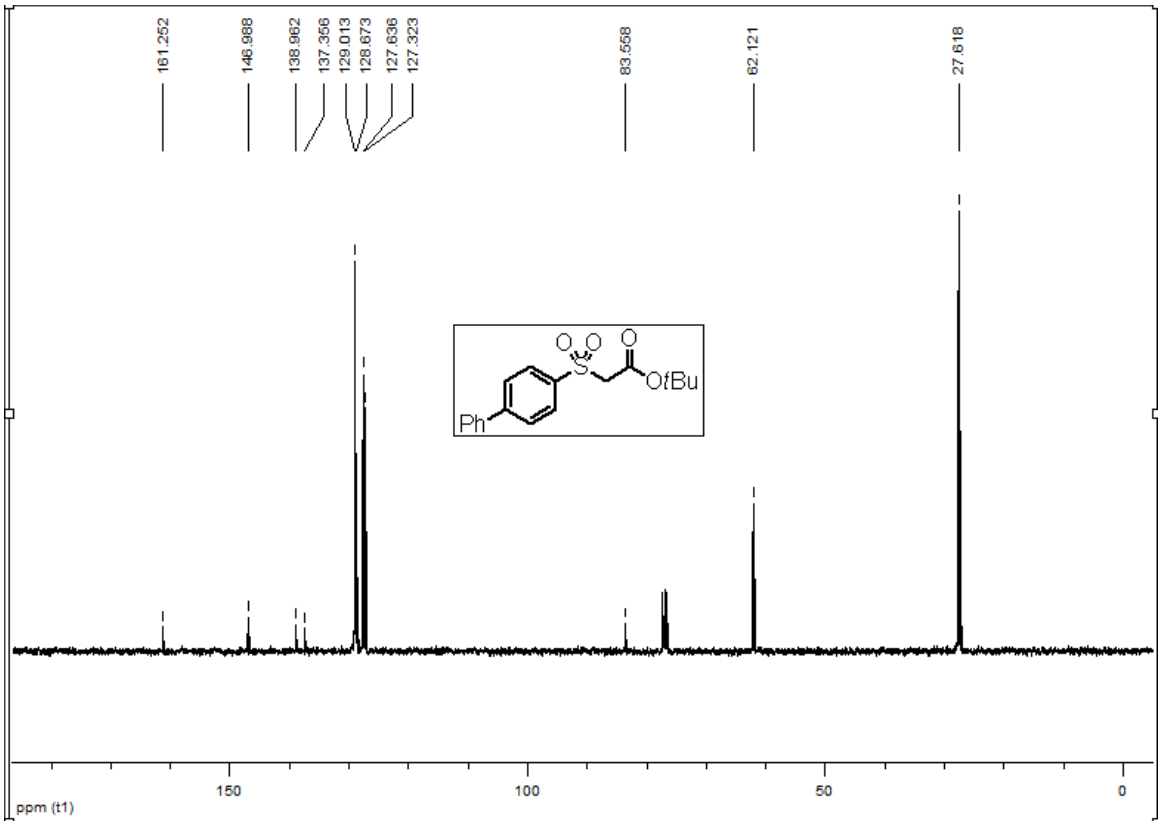

7.868
7.847
7.589
7.567
4.021
1.350
1.96
2.05
2.00
18.11
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)
OtBu


161.287
158.044
135.898
128.325
126.074
83.379
62.157
35.237
30.970
27.575
150
100
50
0
ppm (t1)
OtBu


7.876
7.854
7.034
7.012
4.002
3.889
1.392
OtBu
2.00
2.09
2.00
2.06
9.12
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


164.025
161.495
130.723
130.358
114.243
83.392
62.280
55.689
27.648
OtBu
150
100
50
0
ppm (t1)


8.017
7.996
7.790
7.769
7.629
7.626
7.608
7.516
7.513
7.508
7.495
7.476
7.456
7.444
7.438
4.077
1.388
O
O
O
OtBu
Ph
2.07
2.13
2.12
3.17
2.00
9.07
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


161.252
146.988
138.962
137.356
129.013
128.673
127.636
127.323
83.558
62.121
27.618
O
O
O
OtBu
Ph
150
100
50
0
ppm (t1)

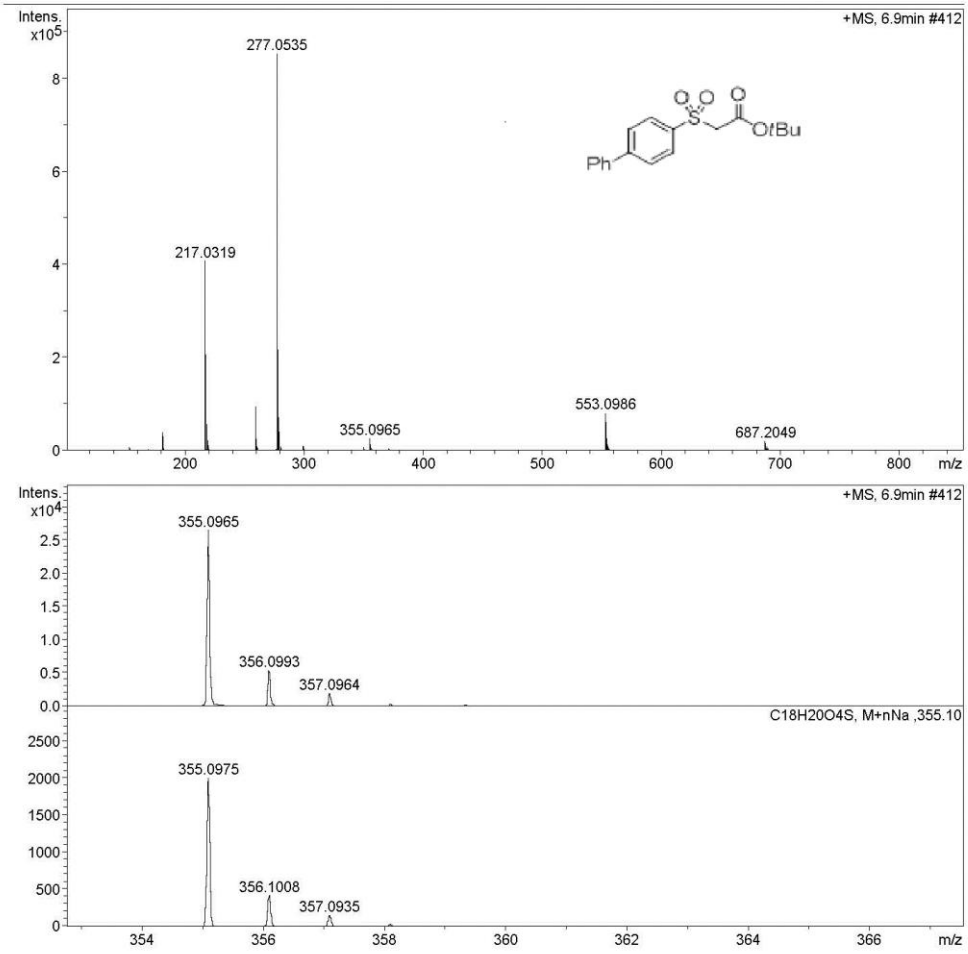
Display Report

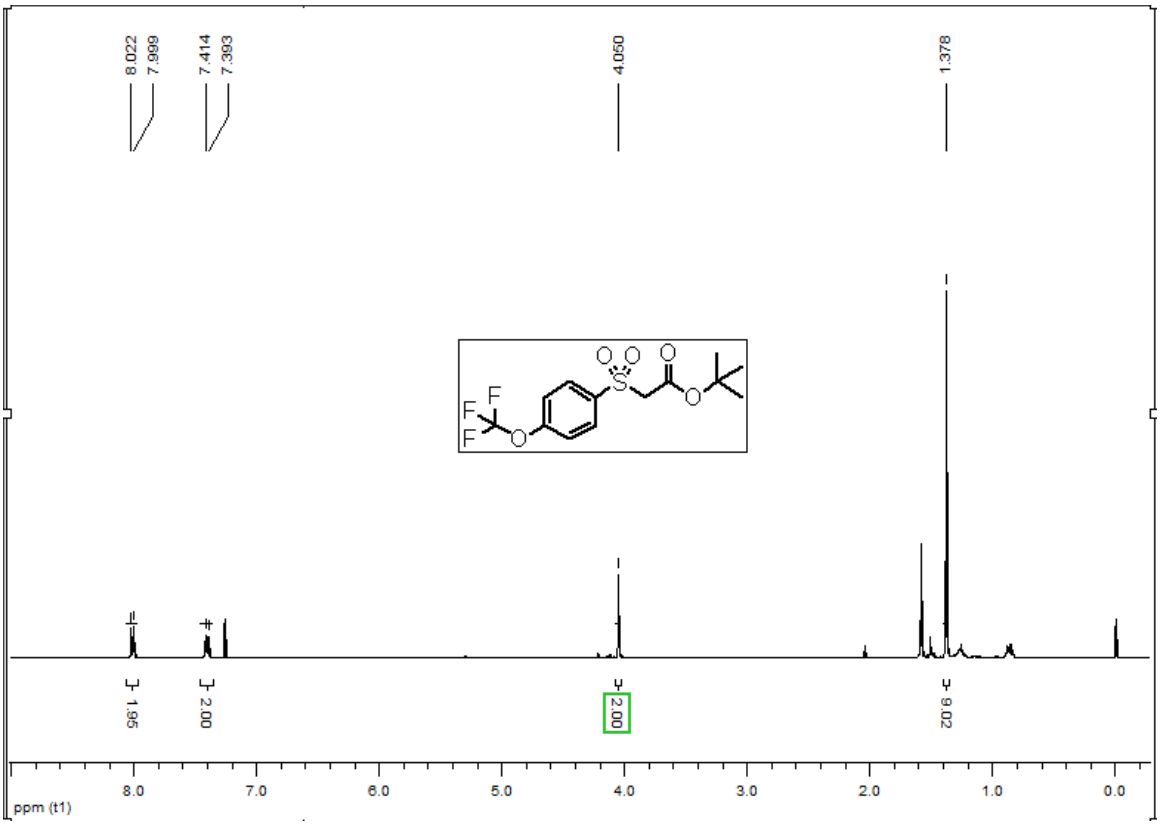
Analysis Info

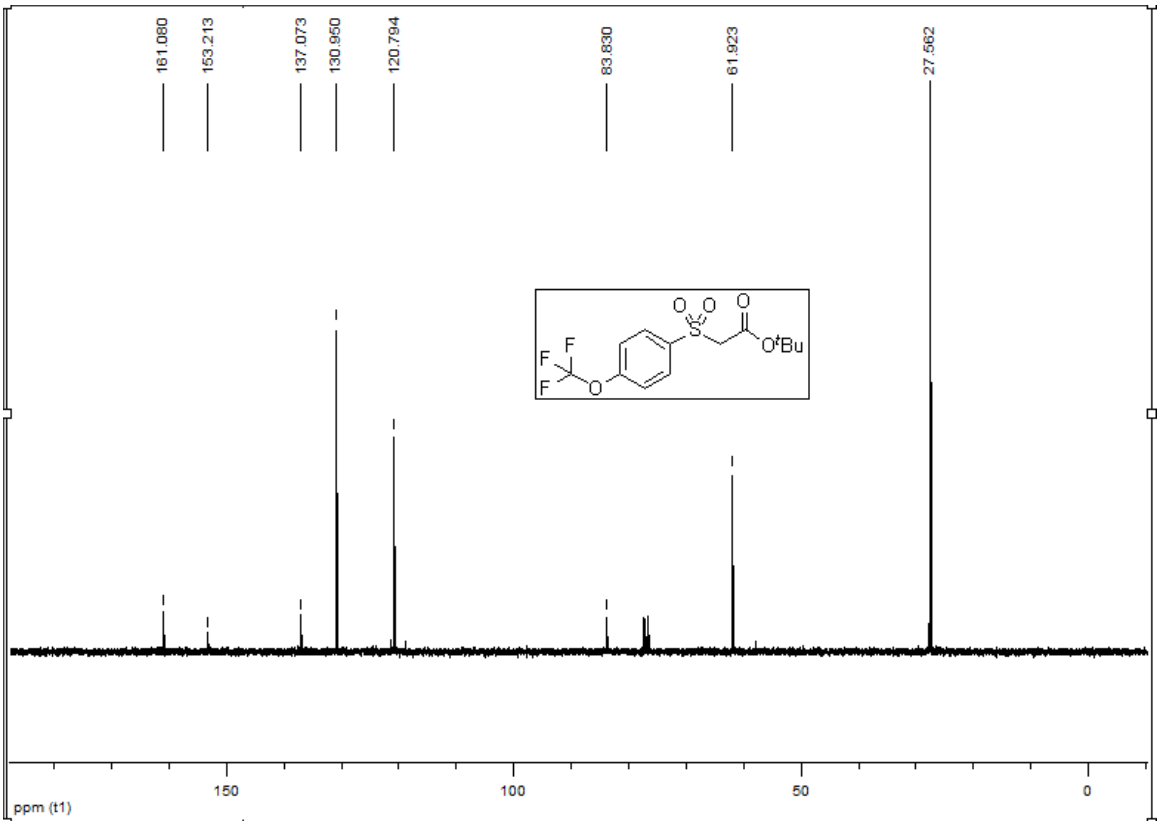
Analysis Name	D:\Data\2016MS\TT\0606\H14_RE1_01_9835.d	Acquisition Date	6/7/2016 1:14:56 AM
Method	tune_200-800_hcoona-pos-10min.m	Operator	gftang
Sample Name	H14	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	100 m/z	Set Capillary	4000 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste




8.022
7.999
7.414
7.393
4.050
1.378
1.95
2.00
2.00
9.02
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


161.080
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137.073
130.950
120.794
83.830
61.923
27.562
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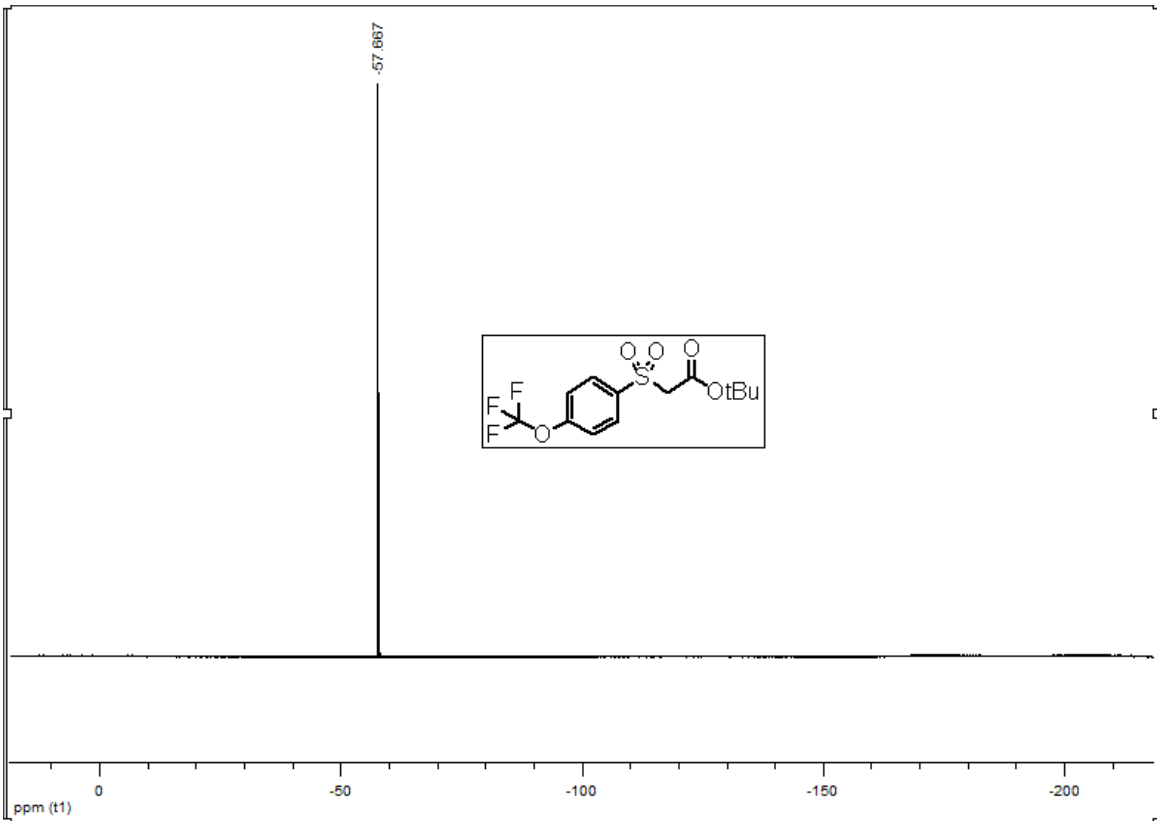
100
50
0
ppm (t1)


-57.667
F
F
F
O
O
O
O
S
OtBu
0
-50
-100
-150
-200
ppm (t1)

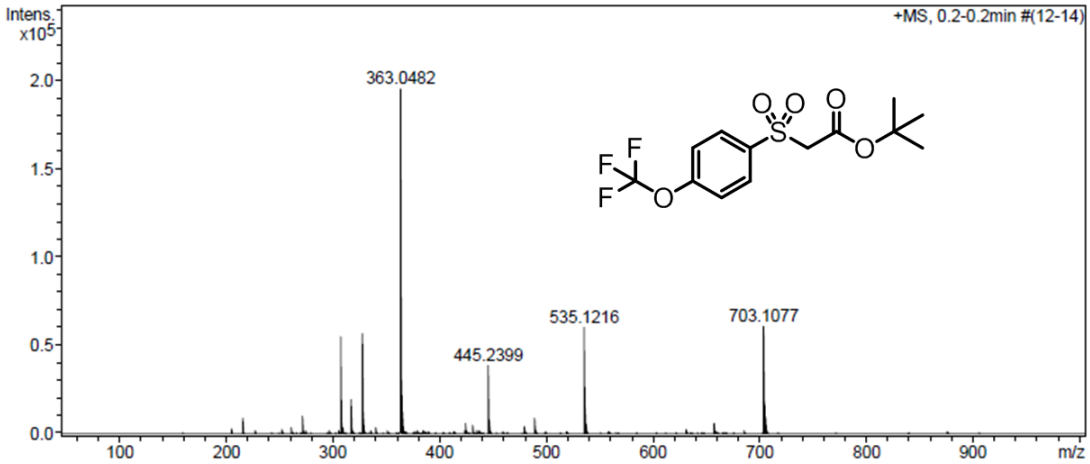
Display Report

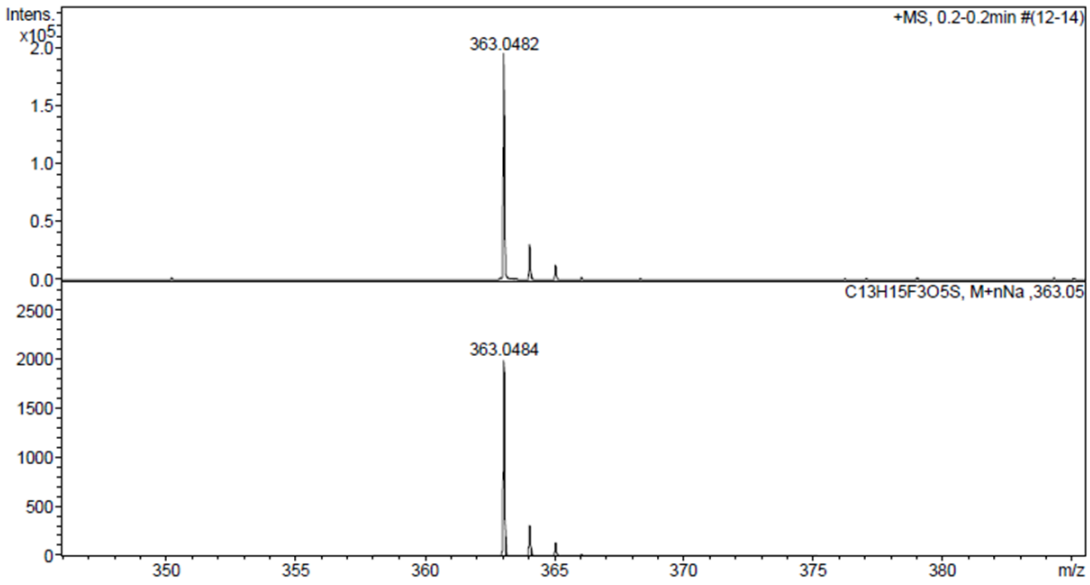
Analysis Info

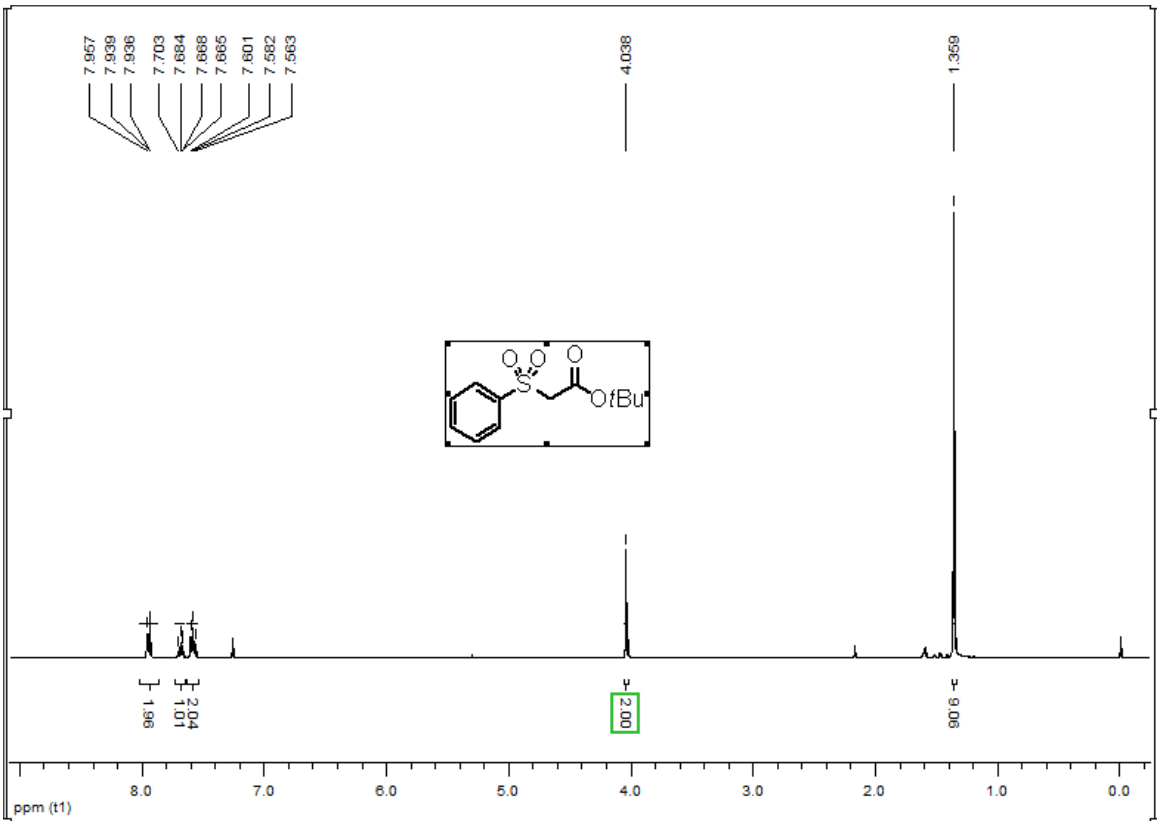
Analysis Name	D:\Data\2016MS\TT\0908\H-12_RE7_01_11005.d	Acquisition Date	9/8/2016 2:16:38 PM
Method	tune_200-800_hcoona-pos-2.5min.m	Operator	gftang
Sample Name	H-12	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.6 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	2500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste






7.957
7.939
7.936
7.703
7.684
7.668
7.665
7.601
7.582
7.563
4.038
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OtBu
1.96
2.01
2.04
2.00
9.06
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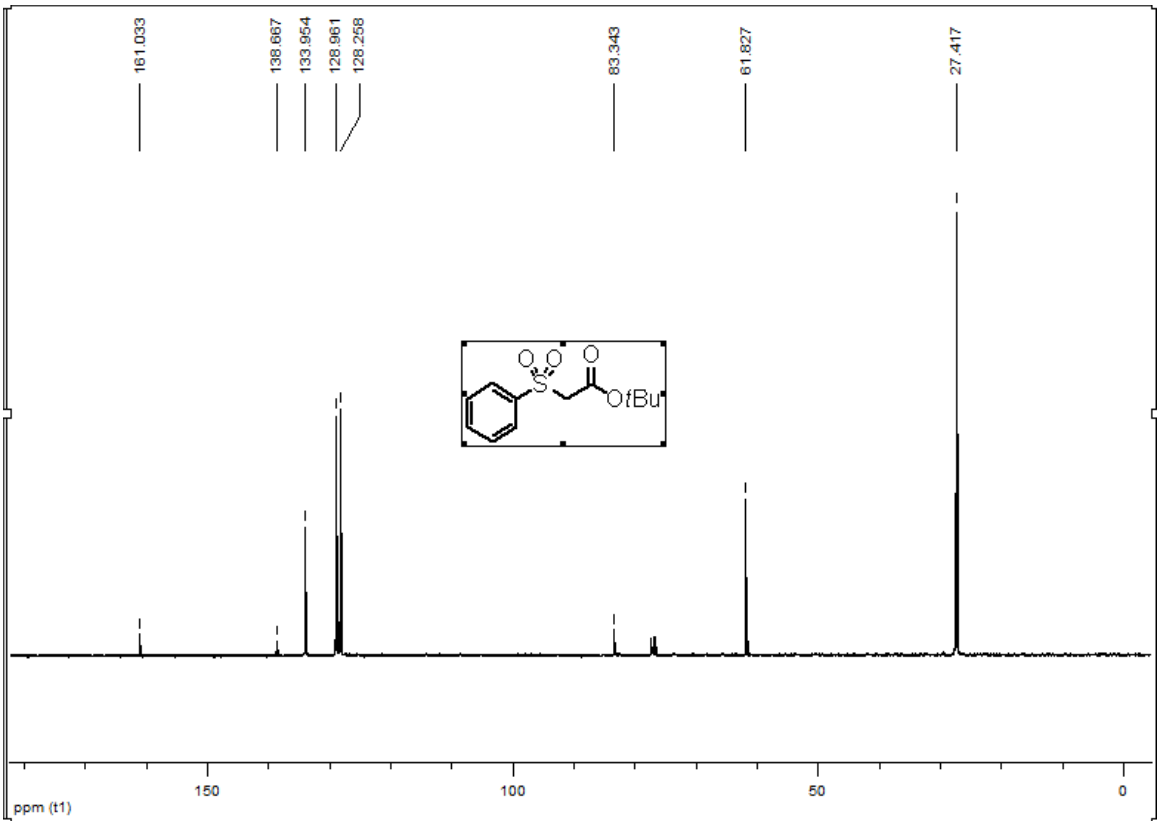
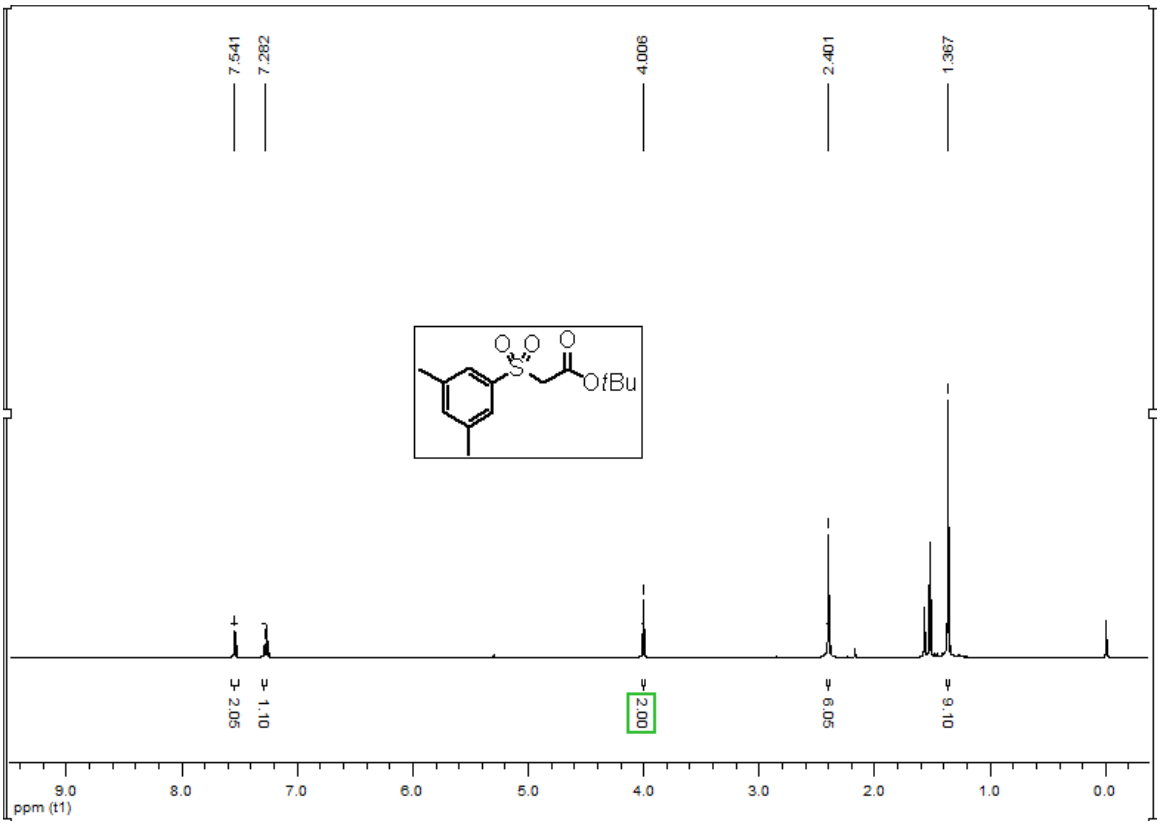
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


161.033
138.667
133.954
128.961
128.258
83.343
61.827
27.417
OtBu
150
100
50
0
ppm (t1)


7.541
7.282
4.006
2.401
1.367
OtBu
2.05
1.10
2.00
6.05
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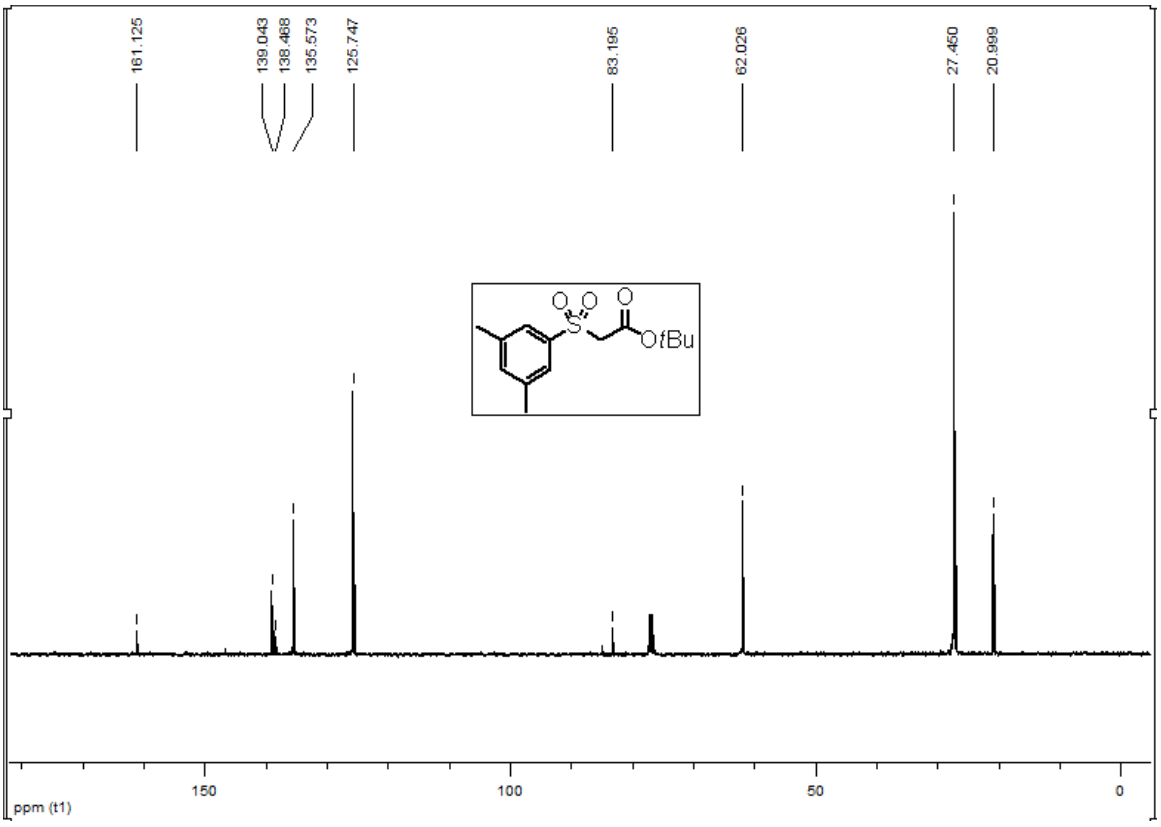
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


161.125
139.043
138.468
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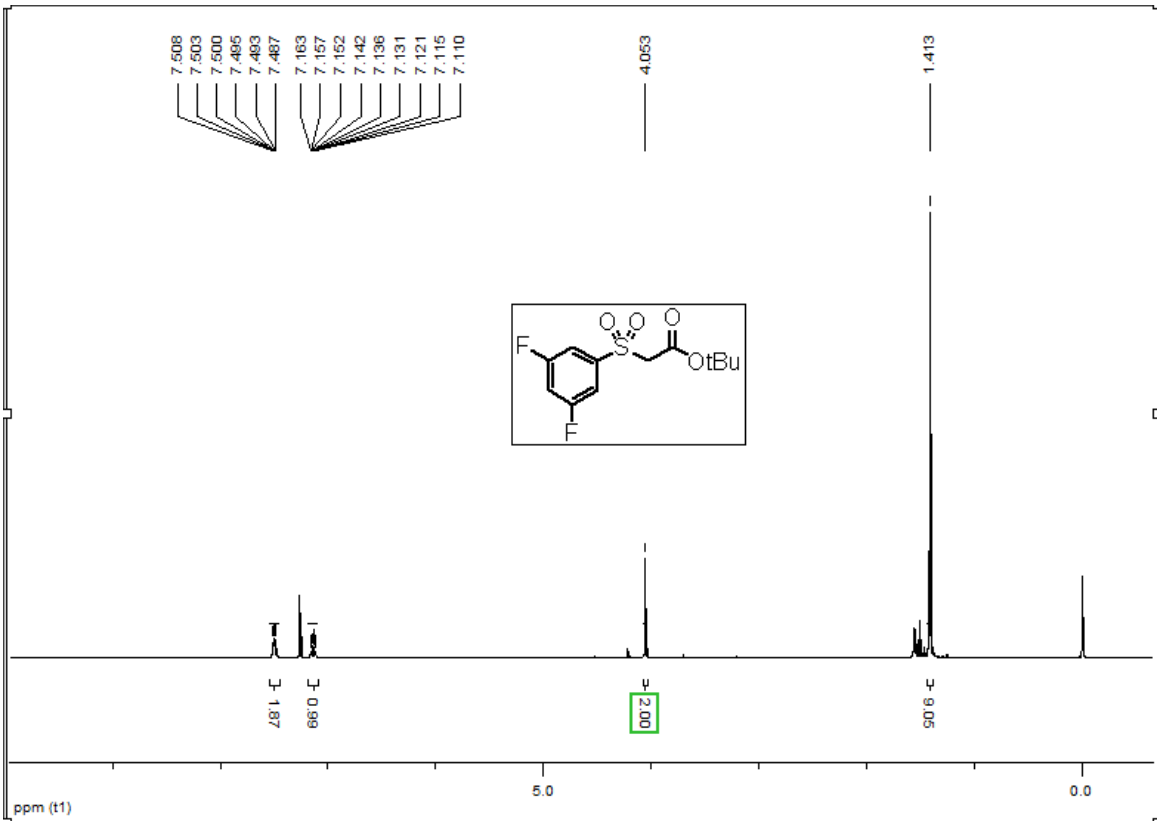
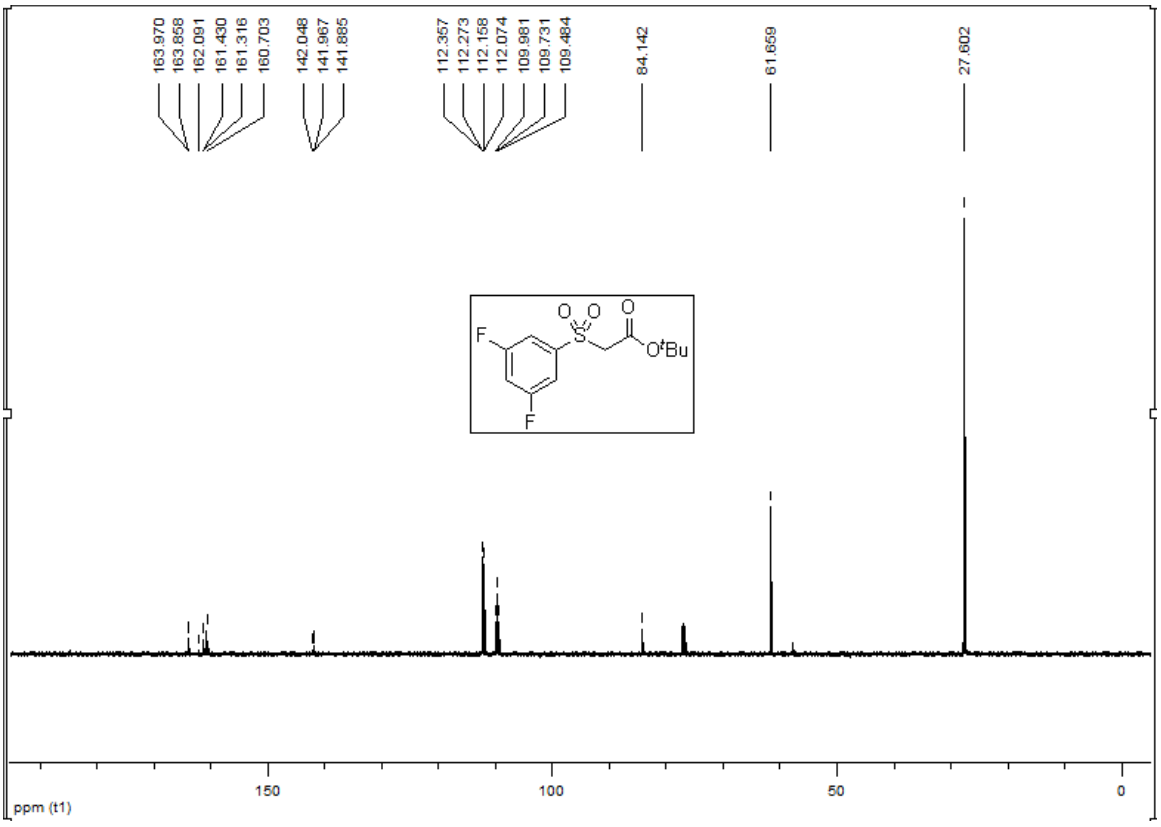
125.747
83.195
62.026
27.450
20.999
OtBu
150
100
50
0
ppm (t1)


7.508
7.503
7.500
7.495
7.493
7.487
7.163
7.157
7.152
7.142
7.136
7.131
7.121
7.115
7.110
4.053
1.413
F
F
O
O
O
S
OtBu
1.87
0.99
2.00
9.06
5.0
0.0
ppm (t1)


163.970
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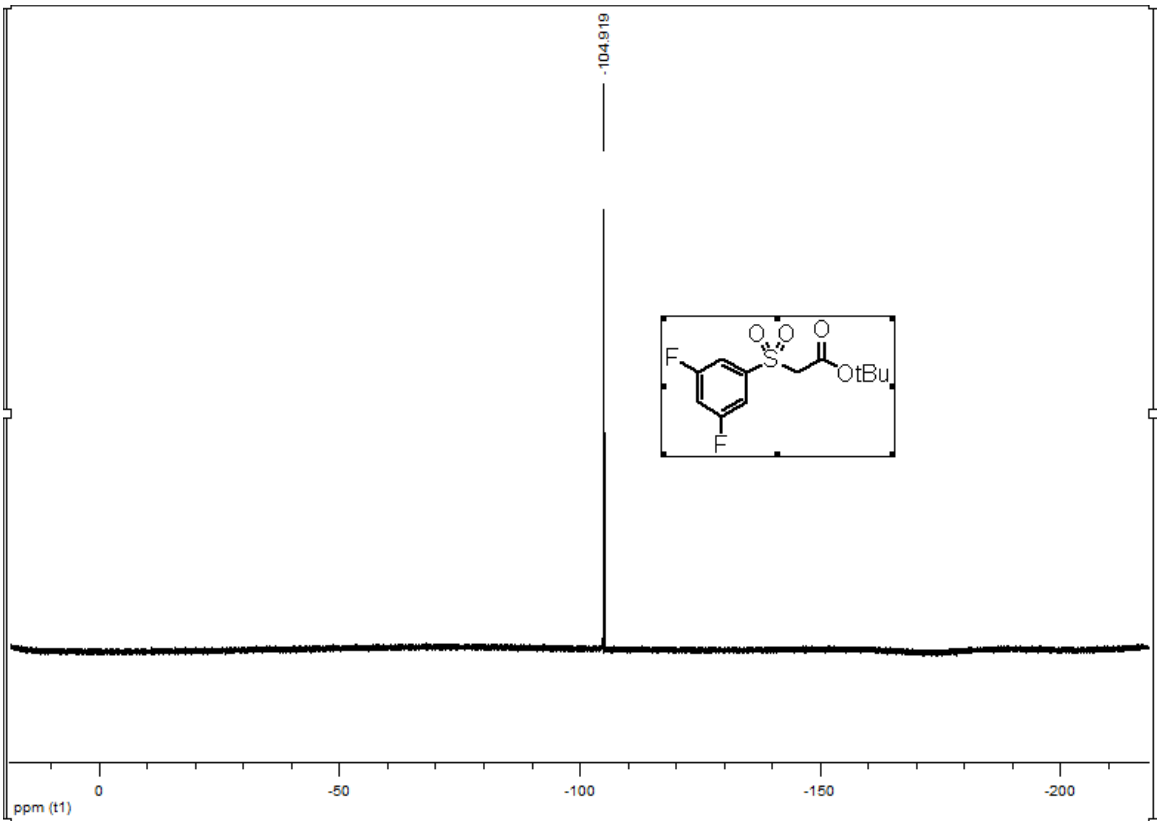
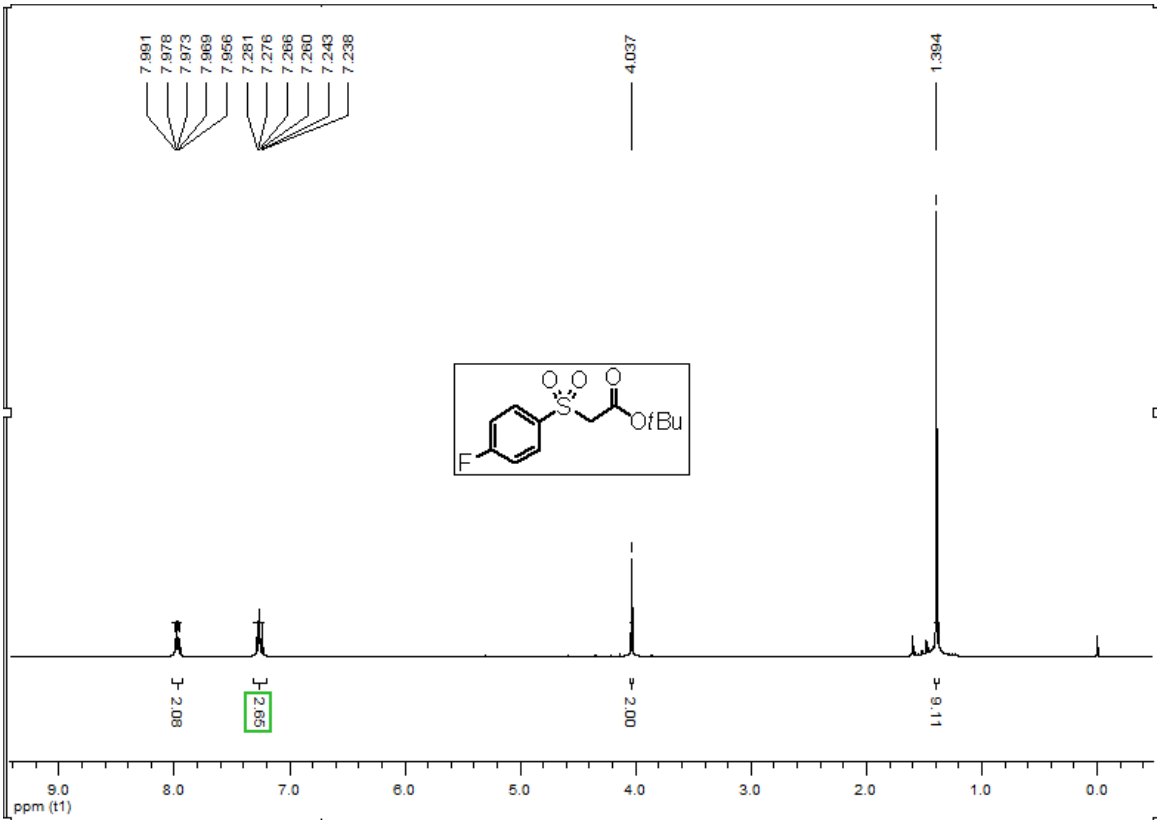
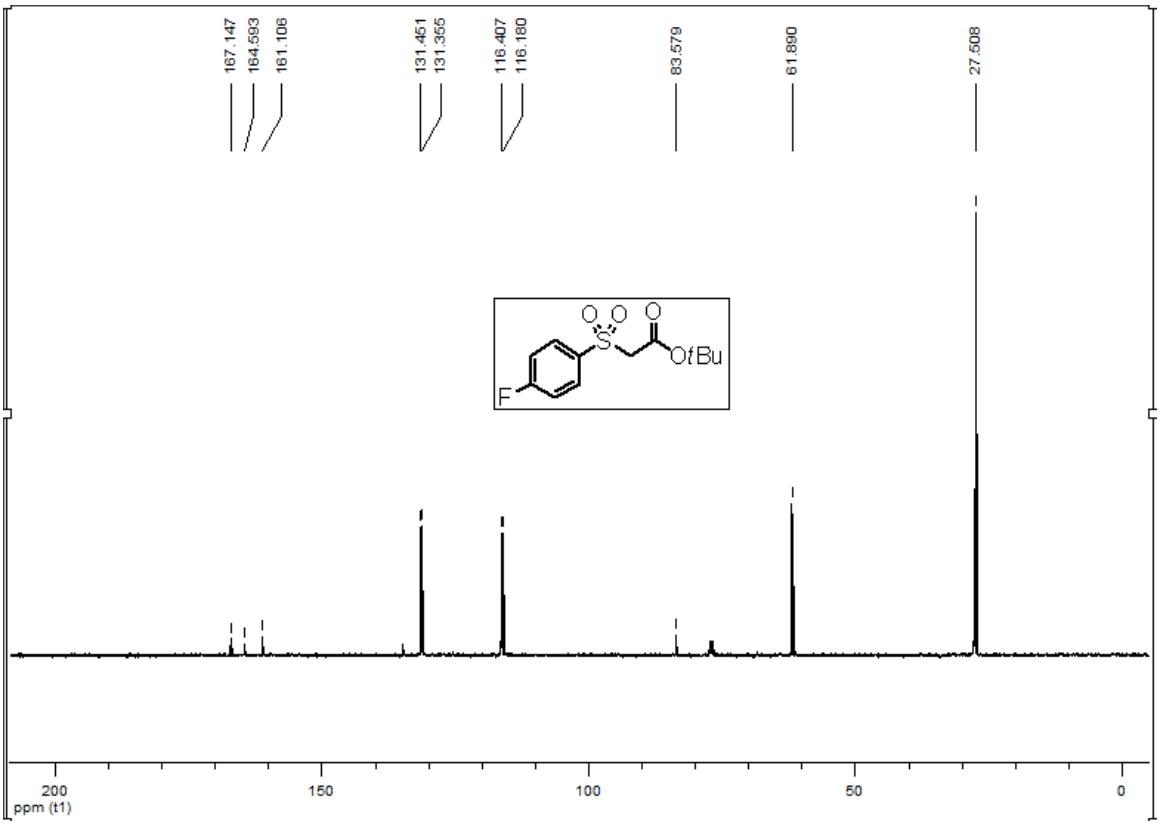
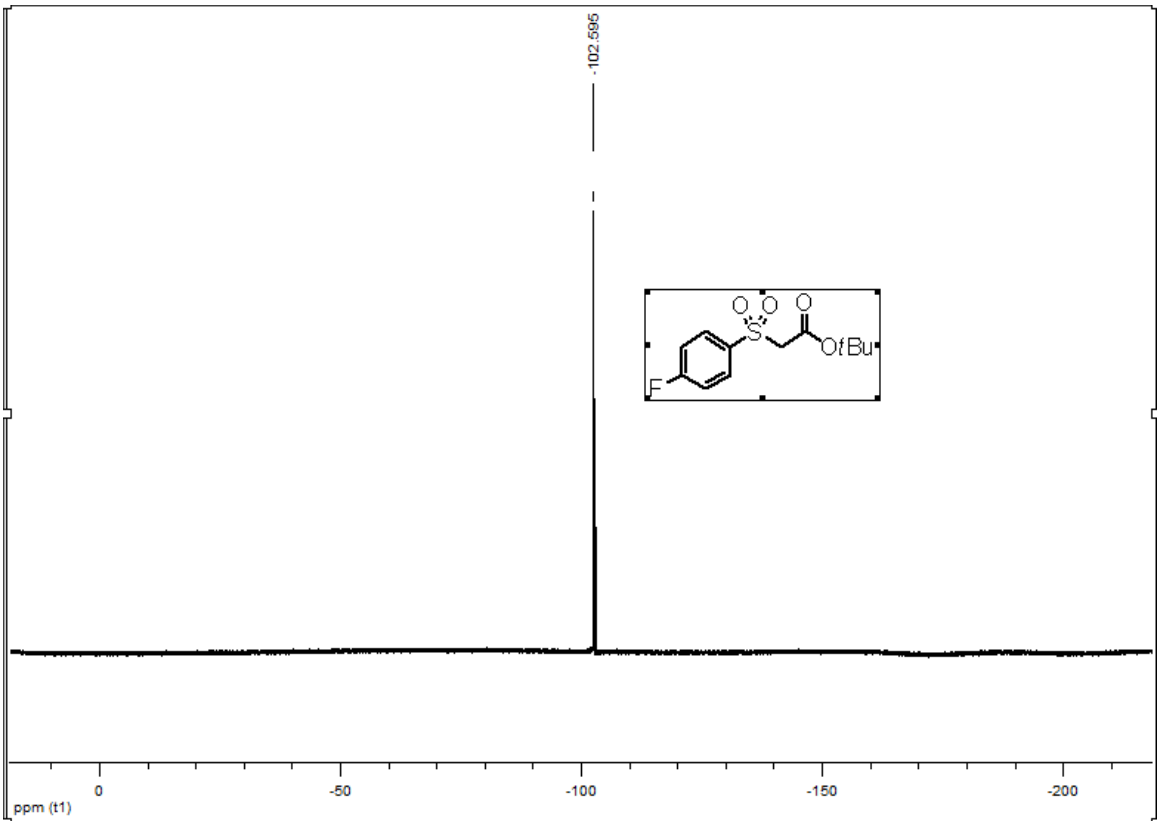
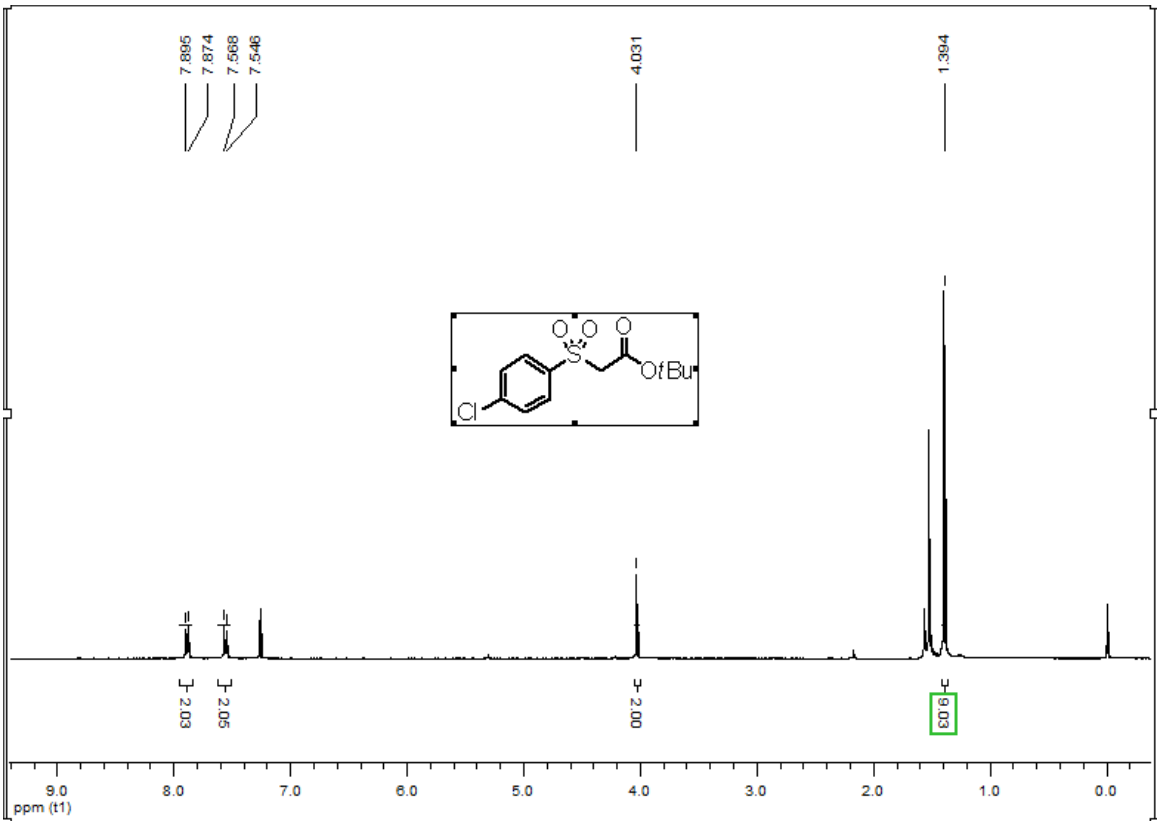
162.091
161.430
161.316
160.703
142.048
141.967
141.885
112.357
112.273
112.158
112.074
109.981
109.731
109.484
84.142
61.659
27.602
F
F
O
O
O
S
O'Bu
150
100
50
0
ppm (t1)

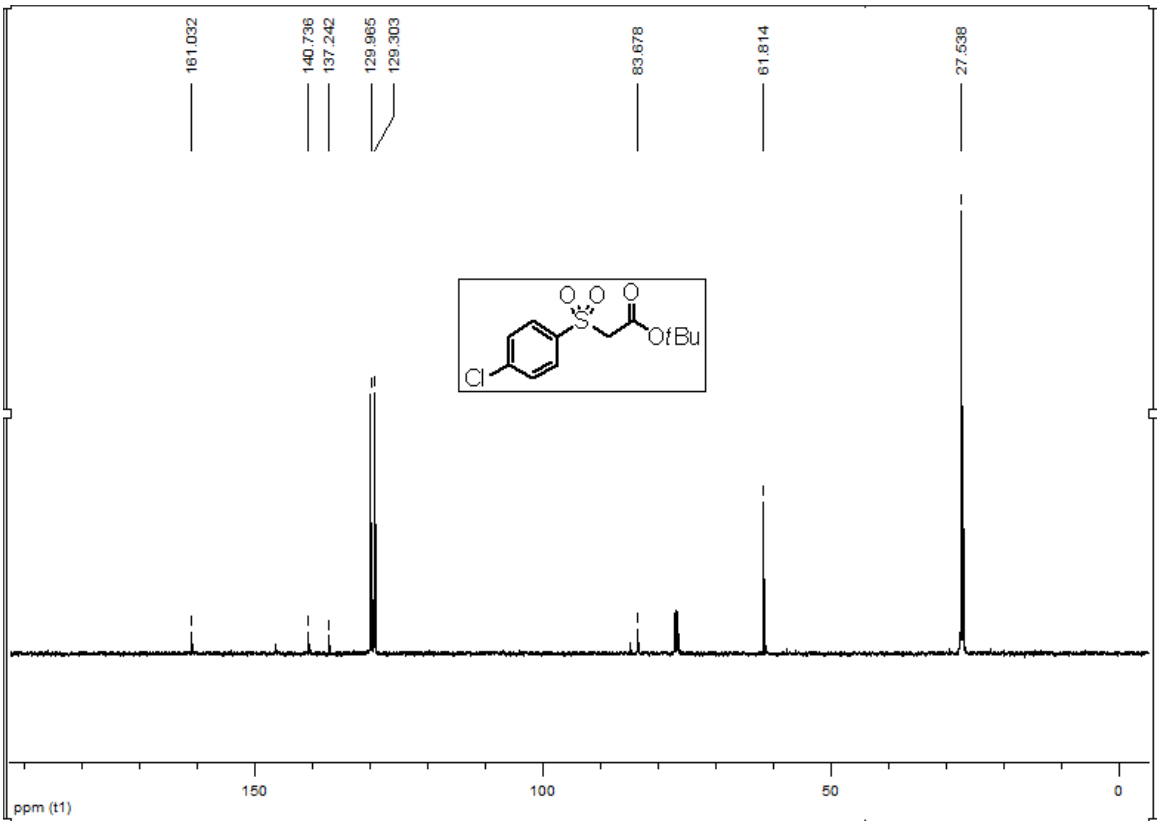

-104.919
F
F
OtBu
0
-50
-100
-150
-200
ppm (t1)

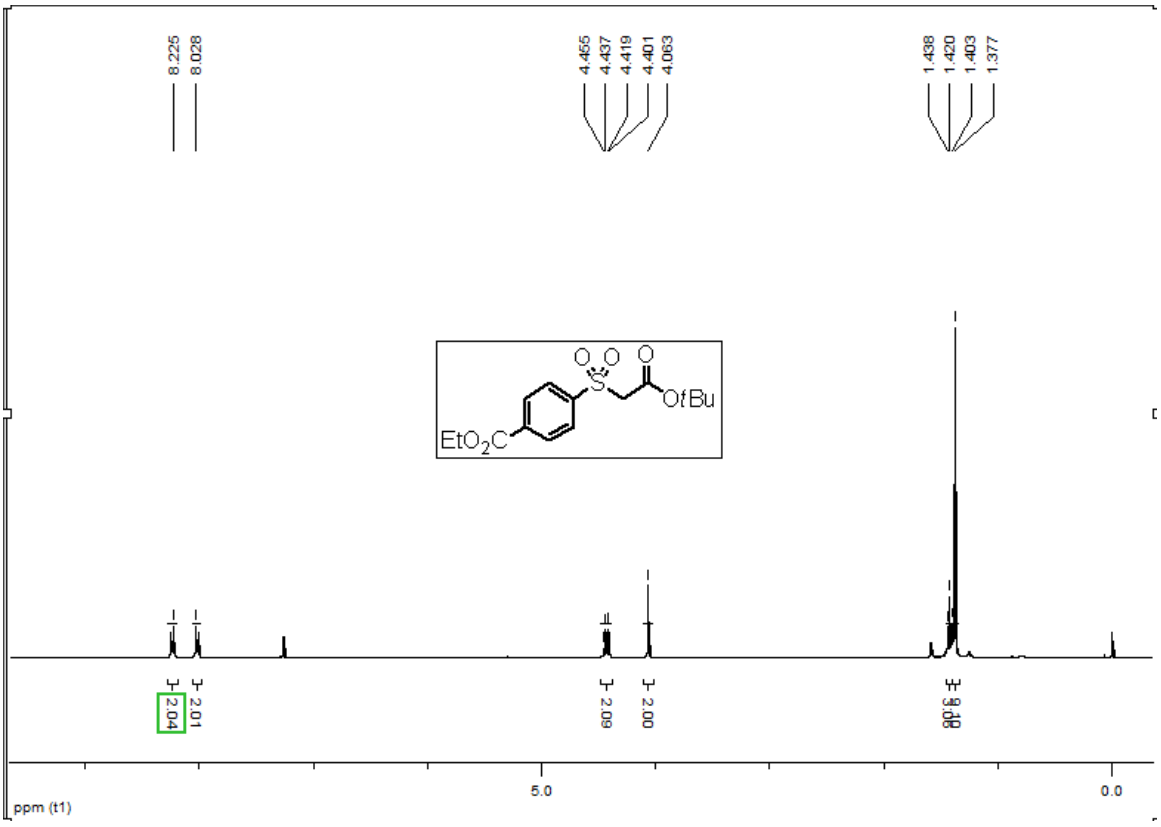

7.991
7.978
7.973
7.969
7.956
7.281
7.276
7.266
7.260
7.243
7.238
4.037
1.394
F
OtBu
2.08
2.65
2.00
9.11
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

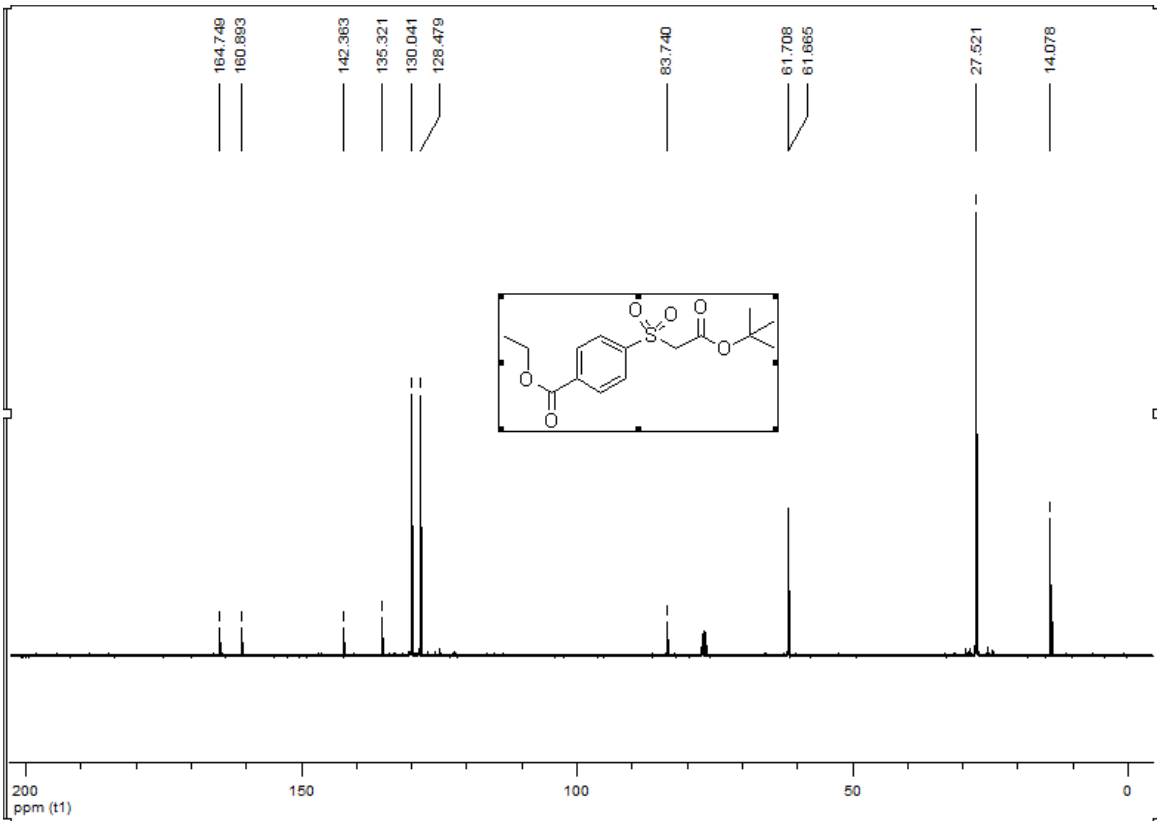

167.147
164.593
161.106
131.451
131.355
116.407
116.180
83.579
61.890
27.508
O
O
S
O
OtBu
F
200
150
100
50
0
ppm (t1)


-102.595
O
O
S
O
OtBu
F
0
-50
-100
-150
-200
ppm (t1)


7.895
7.874
7.568
7.546
4.031
1.394
O
O
S
O
O
tBu
Cl
2.03
2.06
2.00
9.03
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


161.032
140.736
137.242
129.965
129.303
83.678
61.814
27.538
O
O
S
O
O
tBu
Cl
150
100
50
0
ppm (t1)


8.225
8.028
4.455
4.437
4.419
4.401
4.063
1.438
1.420
1.403
1.377
OtBu
EtO2C
2.04
2.01
2.09
2.00
9.00
5.0
0.0
ppm (t1)


164.749
160.893
142.363
135.321
130.041
128.479
83.740
61.708
61.665
27.521
14.078
200
150
100
50
0
ppm (t1)

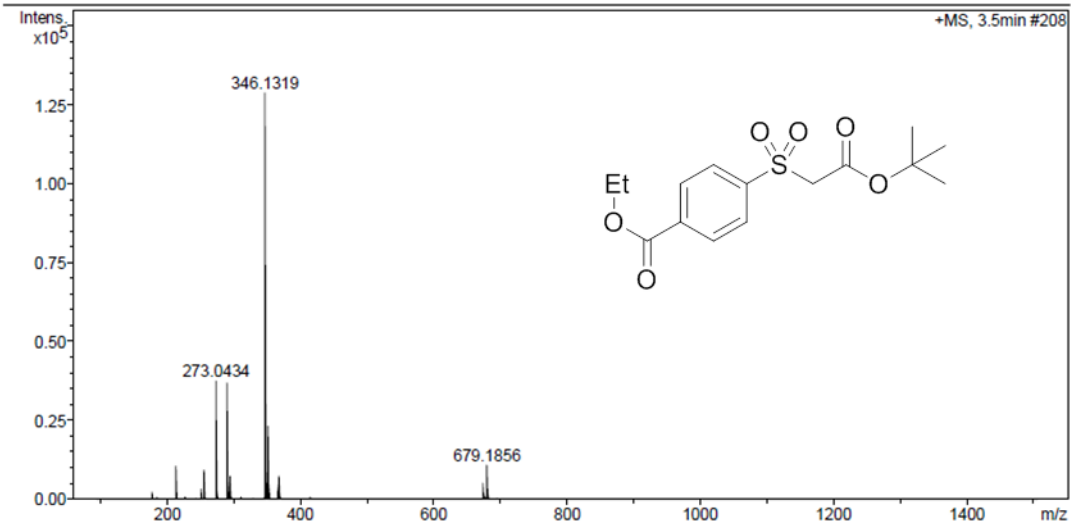
Display Report

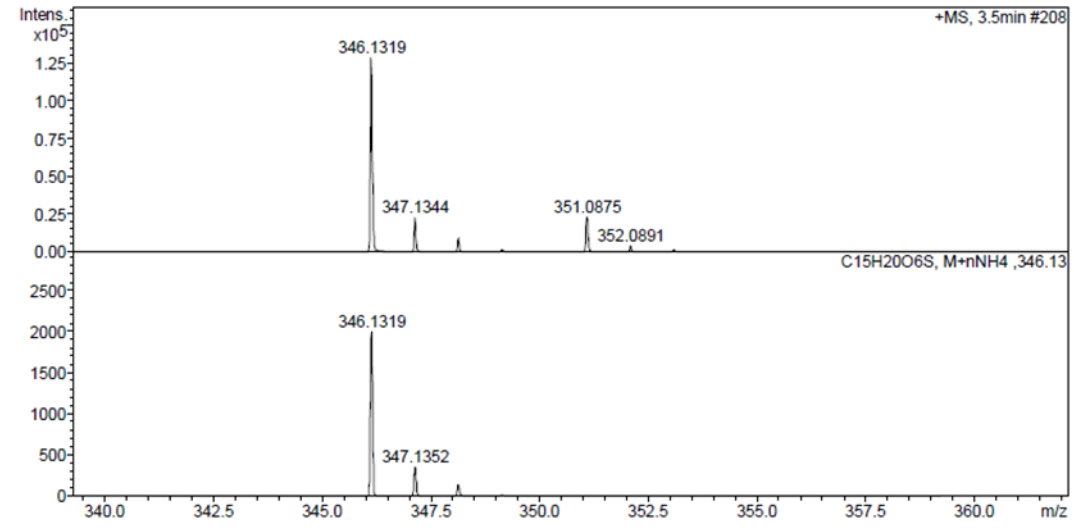
Analysis Info

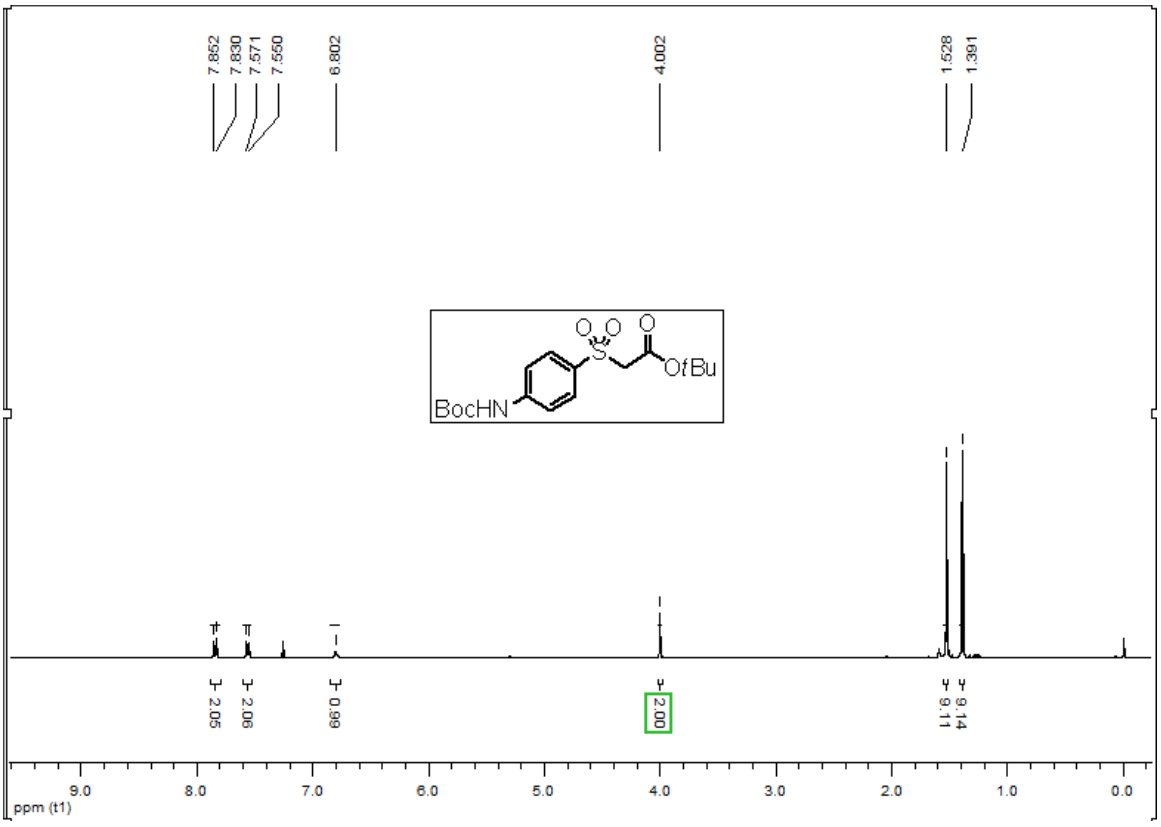
Analysis Name	D:\Data\2016MS\TT\1202\H5_RB5_01_12553.d	Acquisition Date	12/2/2016 10:23:57 PM
Method	tune_200-800_hcoona-pos-8min.m	Operator	gftang
Sample Name	H5	Instrument / Ser#	micrOTOF II 10257
Comment			

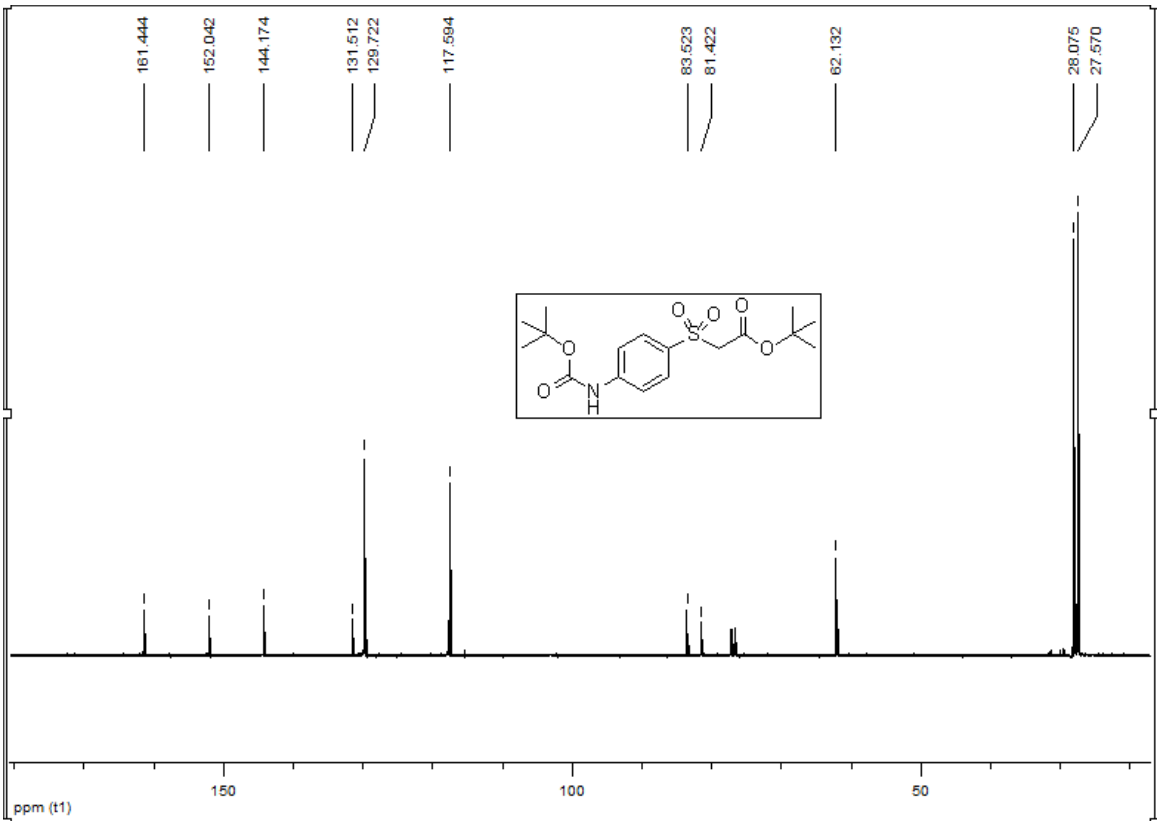
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 ℃
Scan Begin	100 m/z	Set Capillary	4000 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste






7.852
7.830
7.571
7.550
6.802
4.002
1.528
1.391
BocHN
OtBu
2.05
2.06
0.99
2.00
9.11
9.14
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


161.444
152.042
144.174
131.512
129.722
117.594
83.523
81.422
62.132
28.075
27.570
150
100
50
ppm (t1)

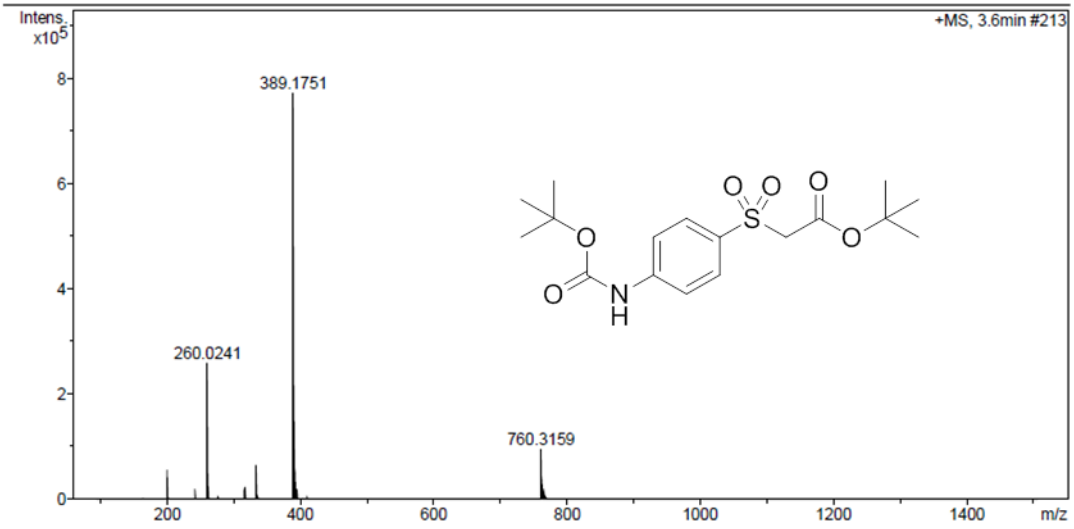
Display Report

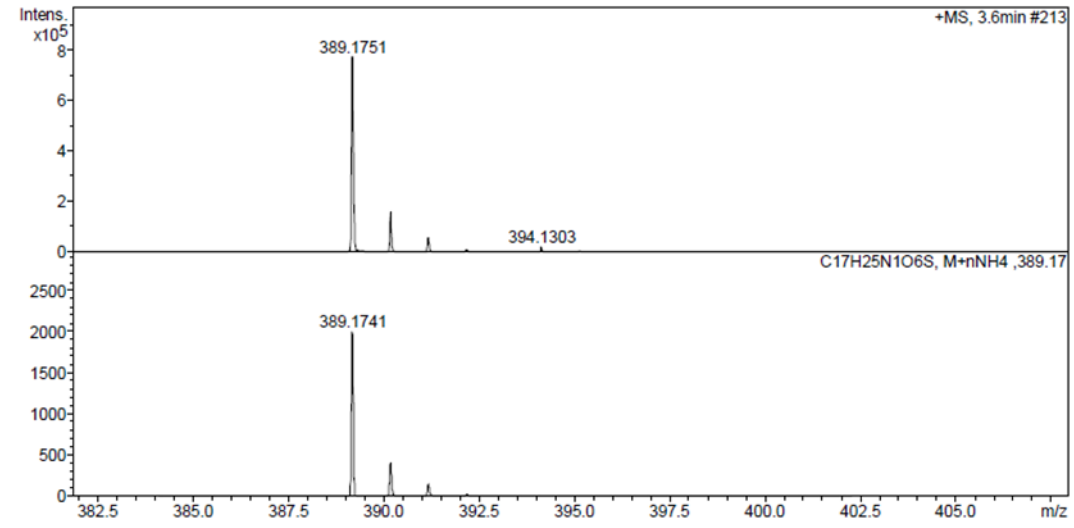
Analysis Info

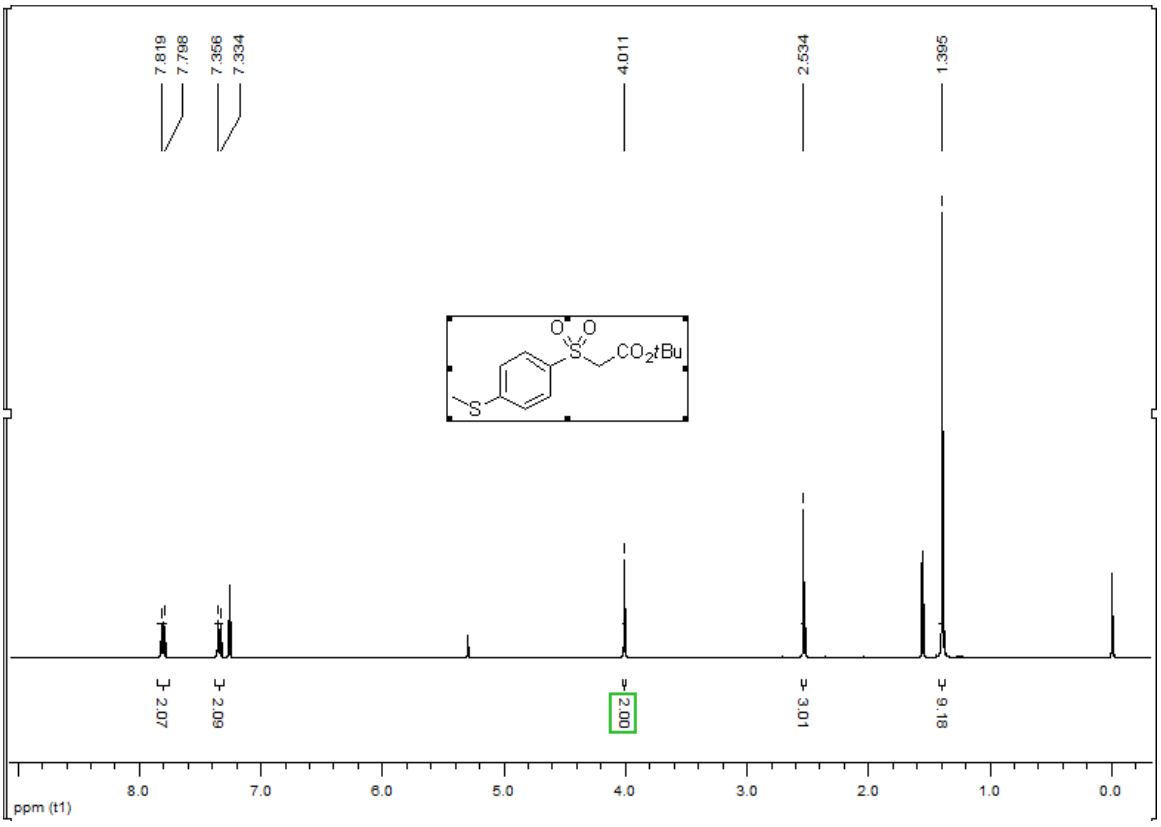
Analysis Name	D:\Data\2016MS\TT\1202\H4_RB4_01_12552.d	Acquisition Date	12/2/2016 10:14:24 PM
Method	tune_200-800_hcoona-pos-8min.m	Operator	gftang
Sample Name	H4	Instrument / Ser#	micrOTOF II 10257
Comment			

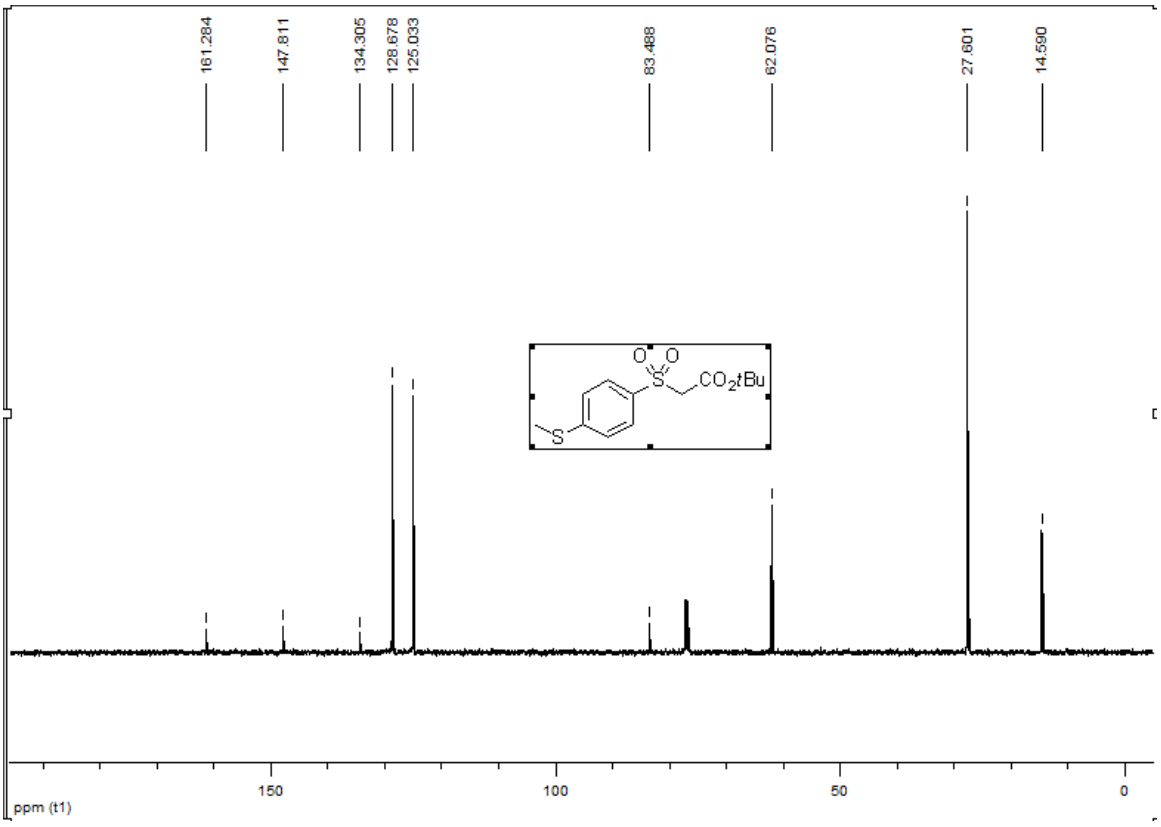
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 ℃
Scan Begin	100 m/z	Set Capillary	4000 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste






7.819
7.798
7.356
7.334
4.011
2.534
1.395
O
O
S
CO2tBu
S
2.07
2.09
2.00
3.01
9.18
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


161.284
147.811
134.305
128.678
125.033
83.488
62.076
27.601
14.590
O
O
S
CO2tBu
S
150
100
50
0
ppm (t1)

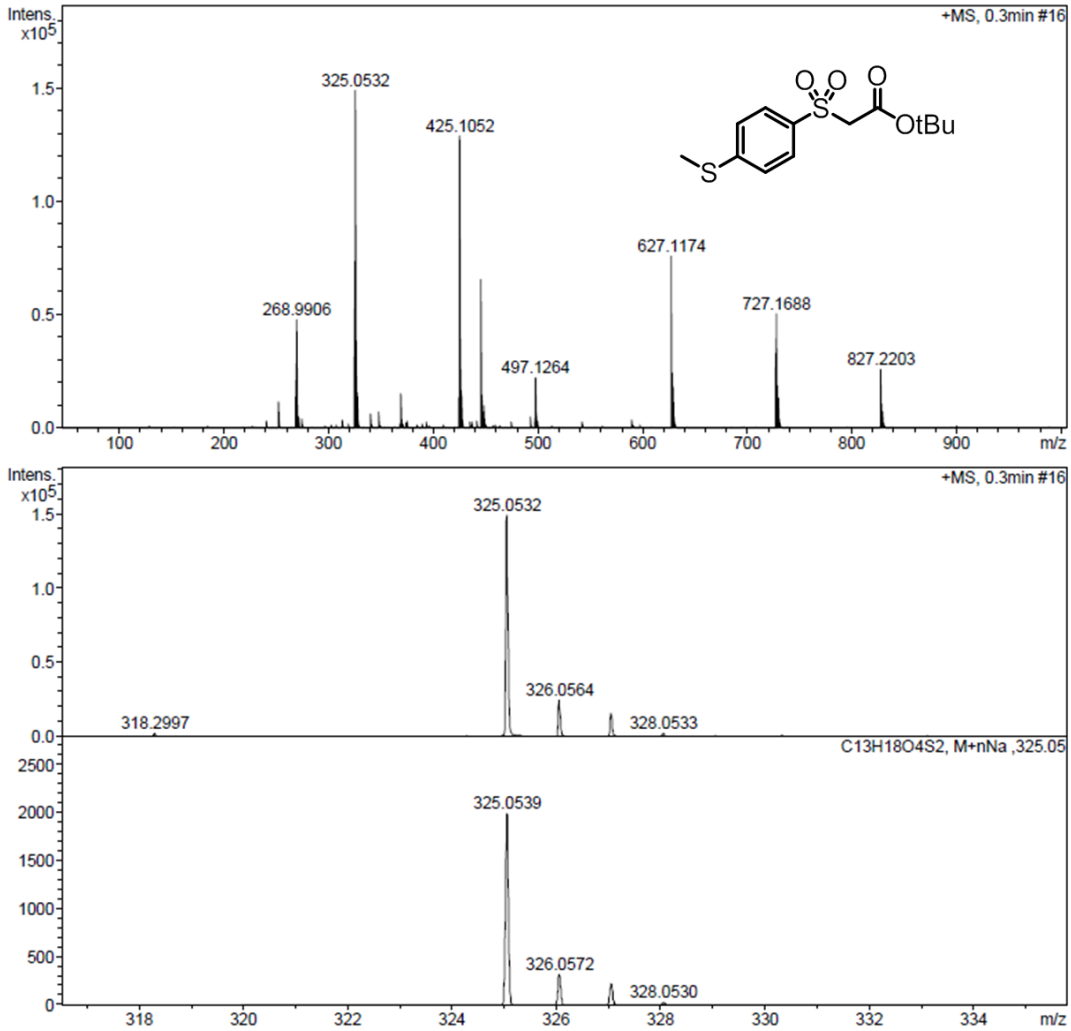
Display Report

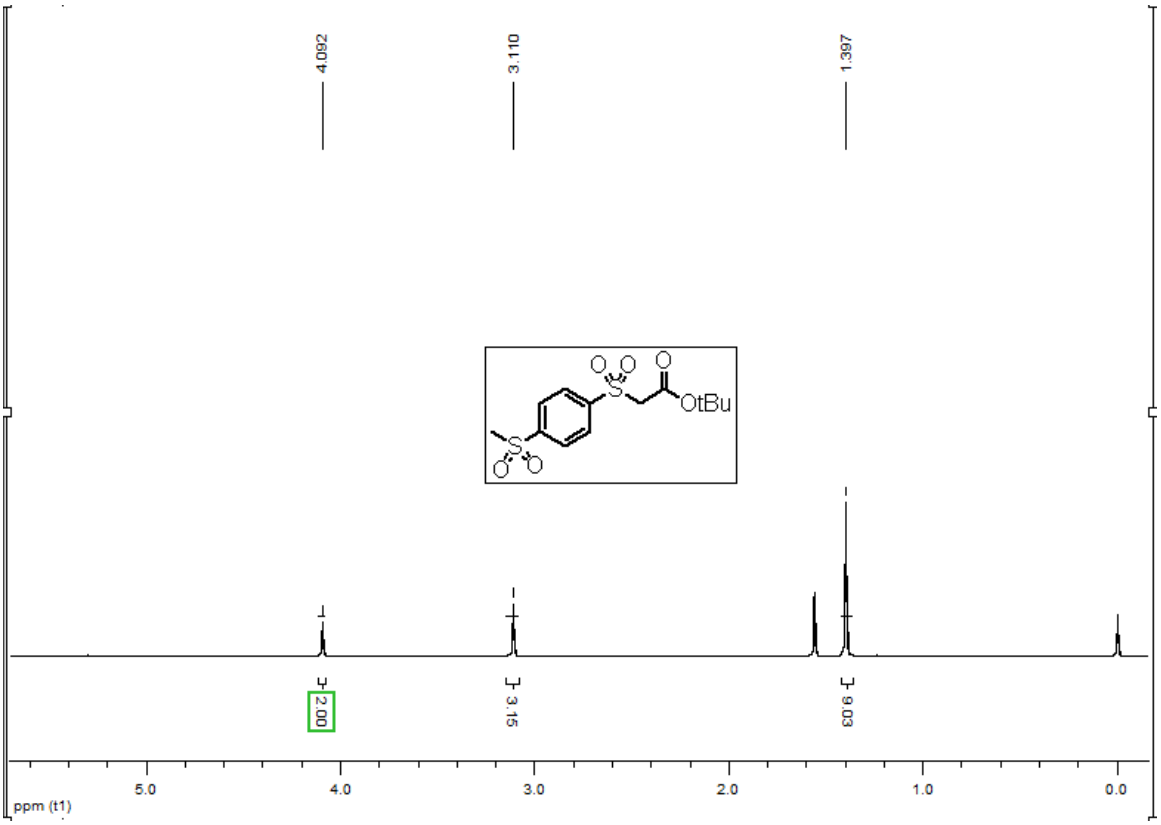
Analysis Info

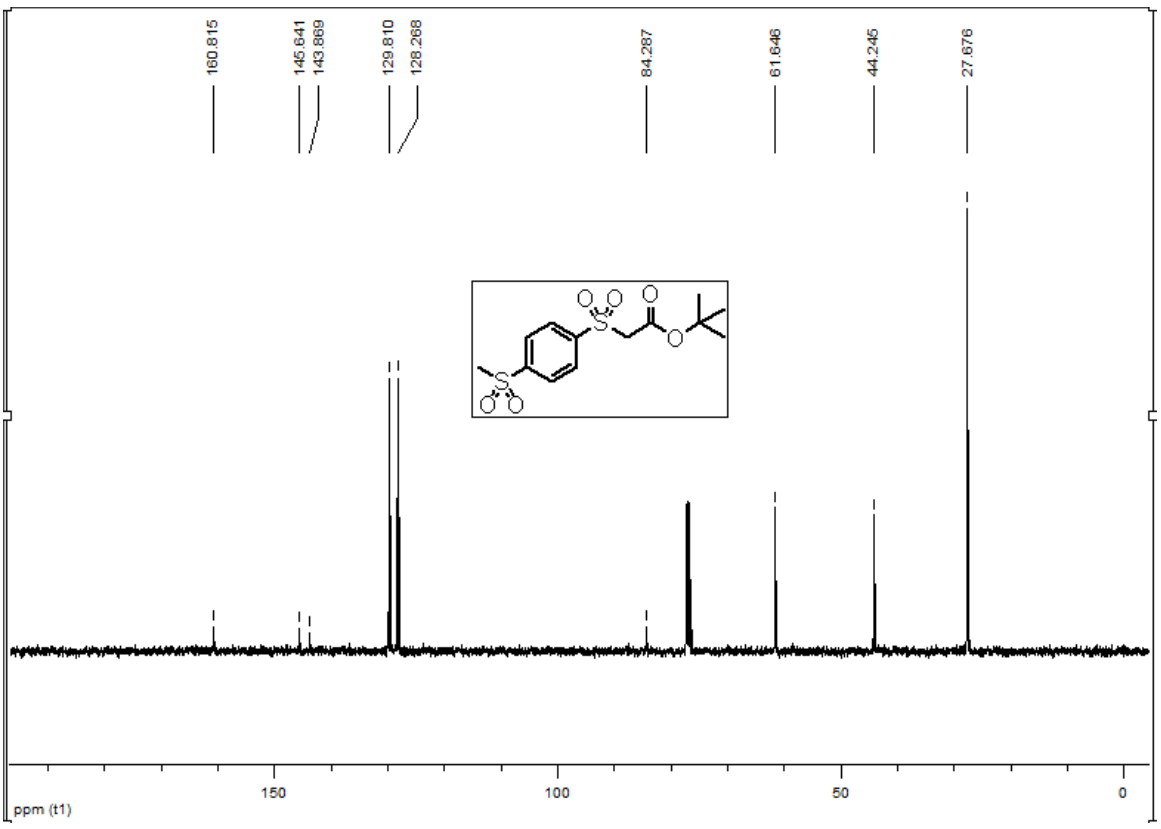
Analysis Name	D:\Data\2016MS\TT\0908\H-3_RD6_01_10996.d	Acquisition Date	9/8/2016 1:40:14 PM
Method	tune_200-800_hcoona-pos-2.5min.m	Operator	gftang
Sample Name	H-3	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.6 Bar
Focus	Not active			Set Dry Heater	180 ℃
Scan Begin	50 m/z	Set Capillary	2500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste




4.092
3.110
1.397
OtBu
2.00
3.15
9.03
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


160.815
145.641
143.869
129.810
128.268
84.287
61.646
44.245
27.676
150
100
50
0
ppm (t1)

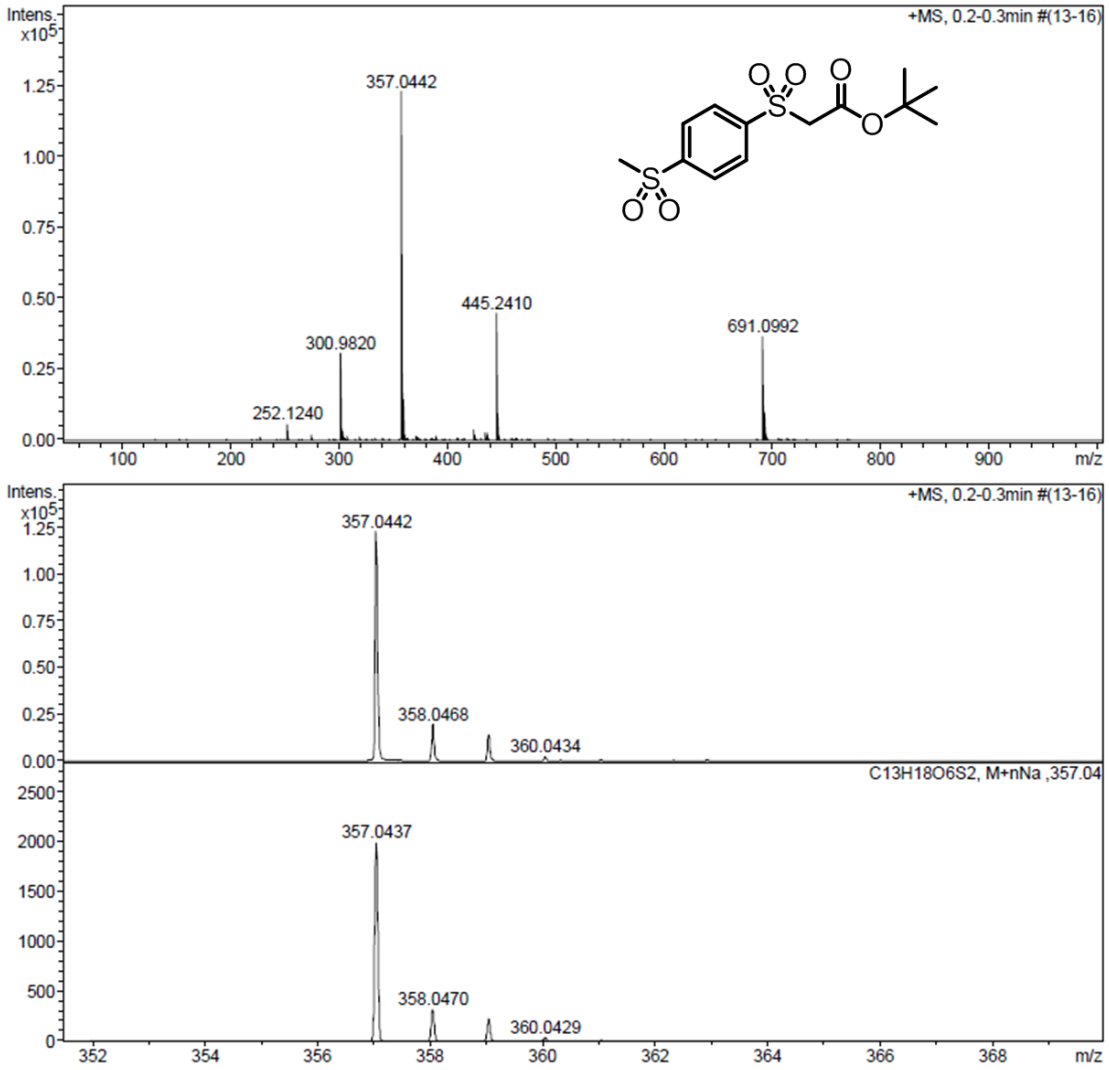
Display Report

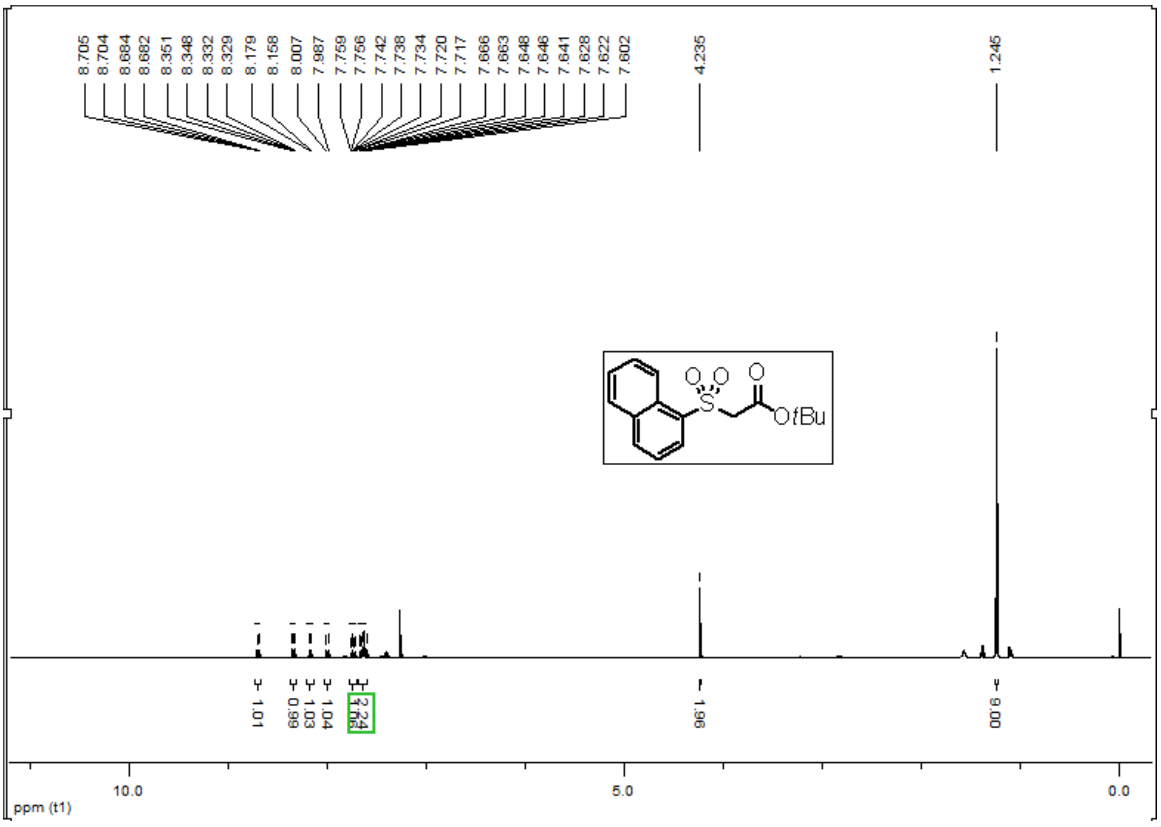
Analysis Info

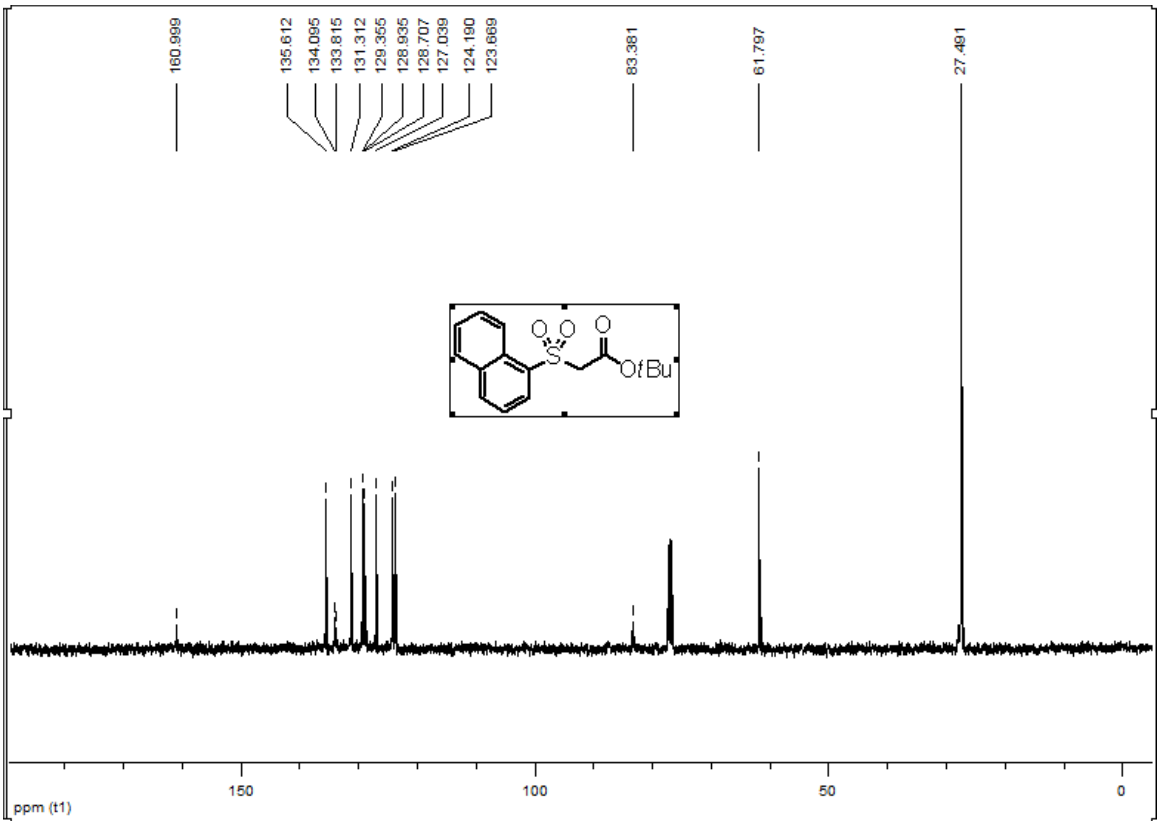
Analysis Name	D:\Data\2016MS\TT\0908\H-11_RE6_01_11004.d	Acquisition Date	9/8/2016 2:12:35 PM
Method	tune_200-800_hcoona-pos-2.5min.m	Operator	gftang
Sample Name	H-11	Instrument / Ser#	micrOTOF II 10257
Comment			

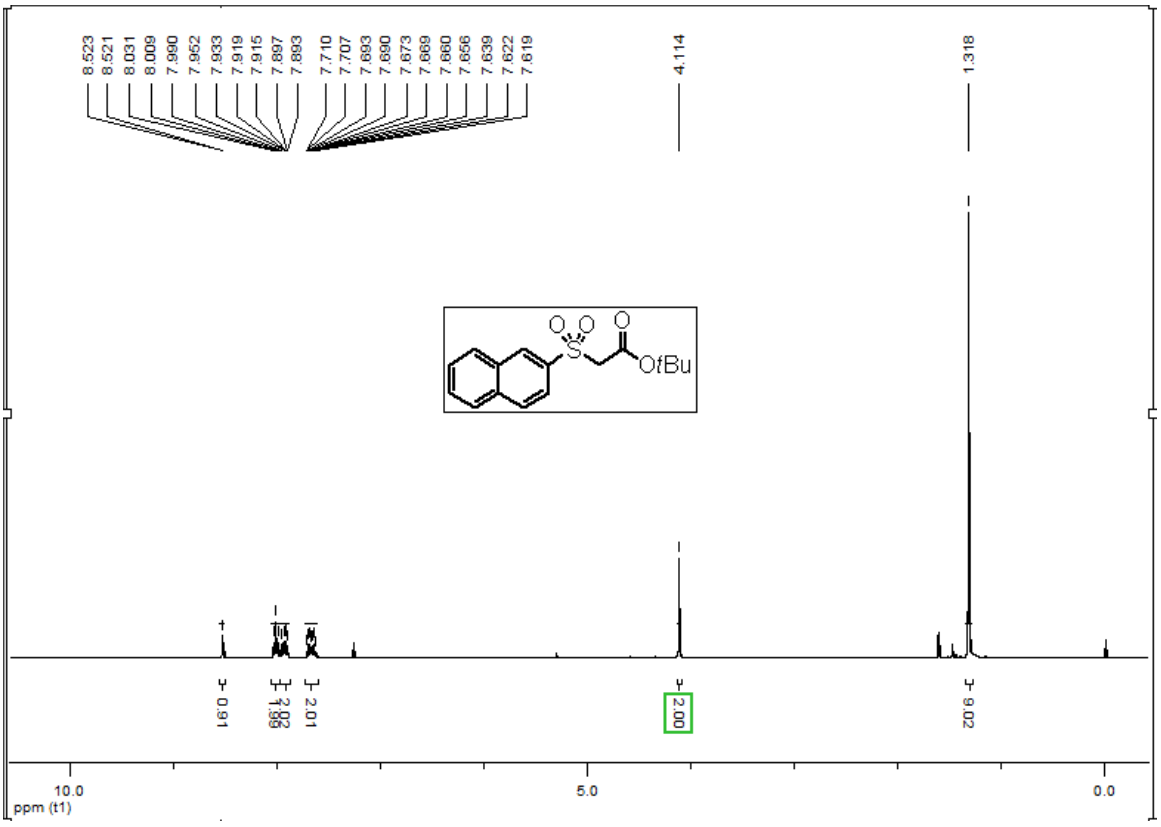
Acquisition Parameter

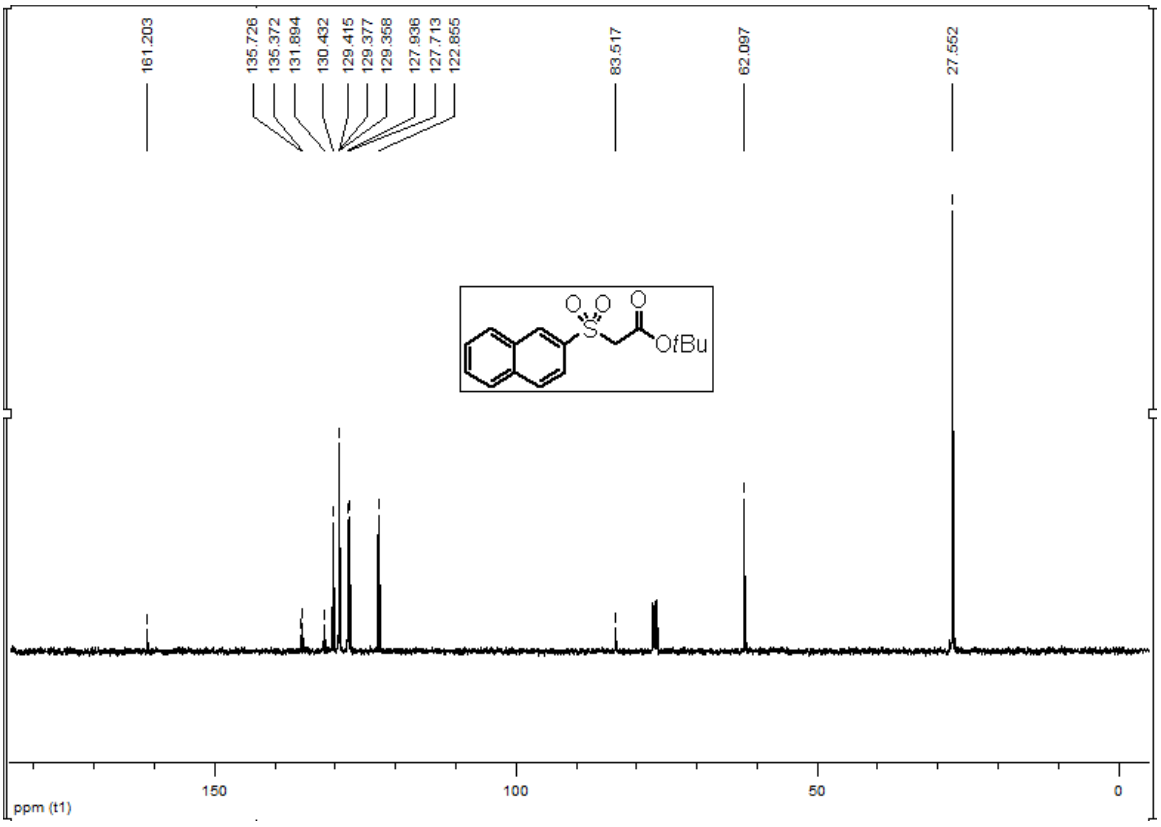
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.6 Bar
Focus	Not active			Set Dry Heater	180 ℃
Scan Begin	50 m/z	Set Capillary	2500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste




8.705
8.704
8.684
8.682
8.351
8.348
8.332
8.329
8.179
8.158
8.007
7.987
7.759
7.756
7.742
7.738
7.734
7.720
7.717
7.666
7.663
7.648
7.646
7.641
7.628
7.622
7.602
4.235
1.245
OtBu
1.01
0.99
1.03
1.04
2.23
1.96
9.00
10.0
5.0
0.0
ppm (t1)


160.999
135.612
134.095
133.815
131.312
129.355
128.935
128.707
127.039
124.190
123.669
83.381
61.797
27.491
OtBu
150
100
50
0
ppm (t1)


8.523
8.521
8.031
8.009
7.990
7.952
7.933
7.919
7.915
7.897
7.893
7.710
7.707
7.693
7.690
7.673
7.669
7.660
7.656
7.639
7.622
7.619
4.114
1.318
OtBu
0.91
1.99
2.01
2.00
9.02
10.0
5.0
0.0
ppm (t1)


161.203
135.726
135.372
131.894
130.432
129.415
129.377
129.358
127.936
127.713
122.855
83.517
62.097
27.552
OtBu
150
100
50
0
ppm (t1)

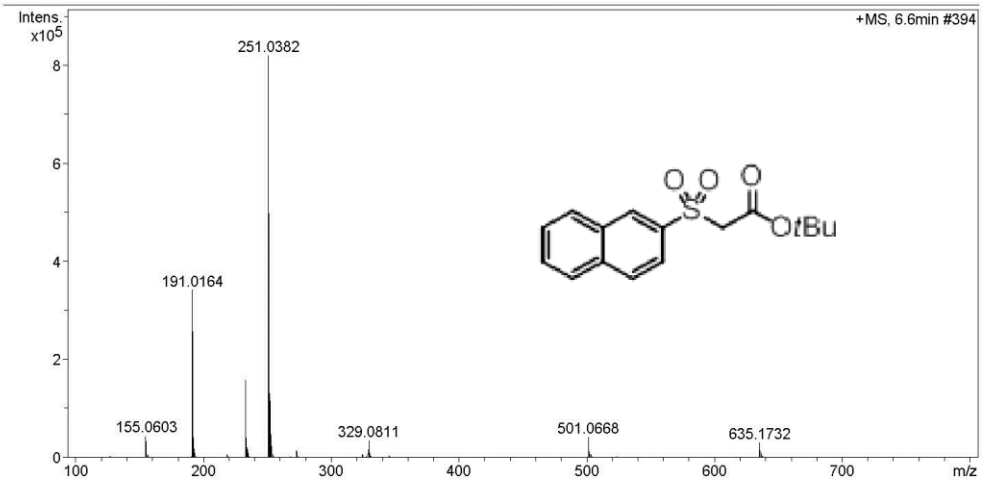
Display Report

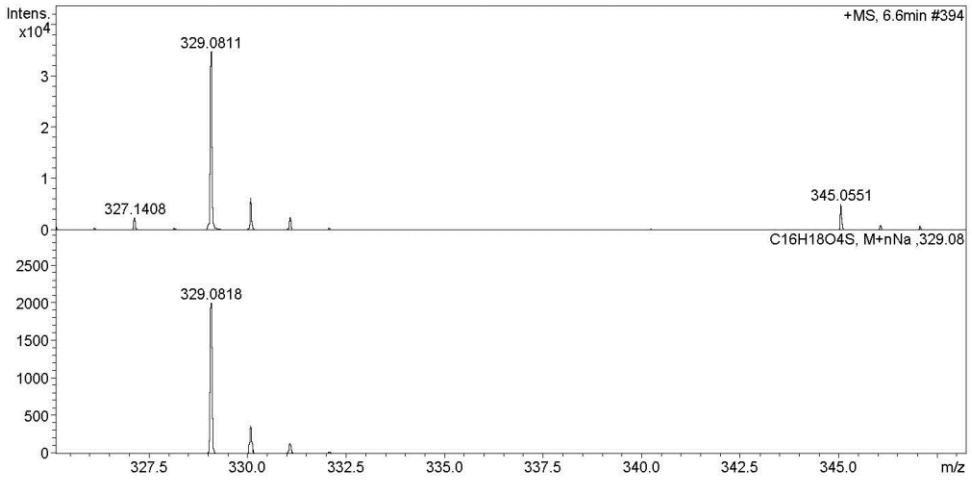
Analysis Info

Analysis Name	D:\Data\2016MS\TT\0606\H13_RD8_01_9834.d	Acquisition Date	6/7/2016 12:59:04 AM
Method	tune_200-800_hcoona-pos-10min.m	Operator	gftang
Sample Name	H13	Instrument / Ser#	micrOTOF II 10257
Comment			

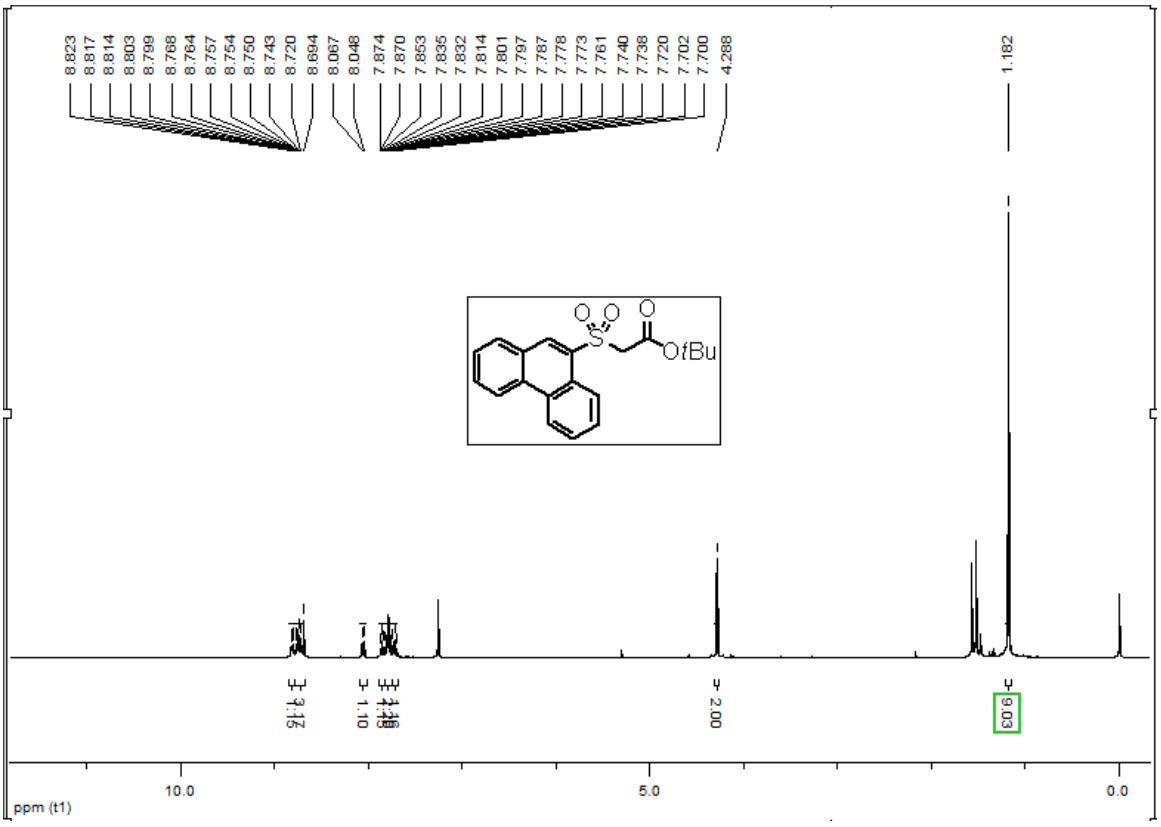
Acquisition Parameter

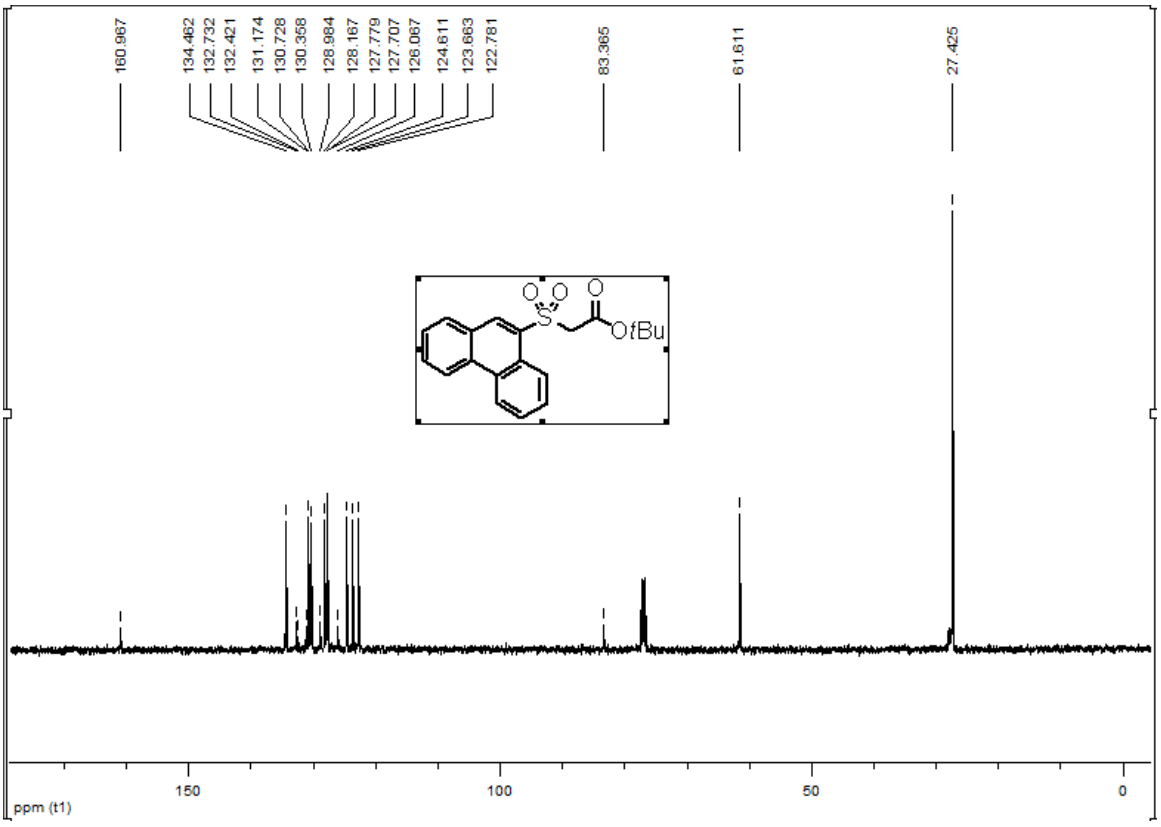
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	100 m/z	Set Capillary	4000 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste





Bruker Compass DataAnalysis 4.0 printed: 6/7/2016 2:14:15 PM Page 1 of 1


8.823
8.817
8.814
8.803
8.799
8.768
8.764
8.757
8.754
8.750
8.743
8.720
8.694
8.067
8.048
7.874
7.870
7.853
7.835
7.832
7.814
7.801
7.797
7.787
7.778
7.773
7.761
7.740
7.738
7.720
7.702
7.700
4.288
1.182
OtBu
3.15
1.10
1.49
2.00
9.03
10.0
5.0
0.0
ppm (t1)


160.967
134.462
132.732
132.421
131.174
130.728
130.358
128.984
128.167
127.779
127.707
126.067
124.611
123.663
122.781
83.365
61.611
27.425
OtBu
150
100
50
0
ppm (t1)

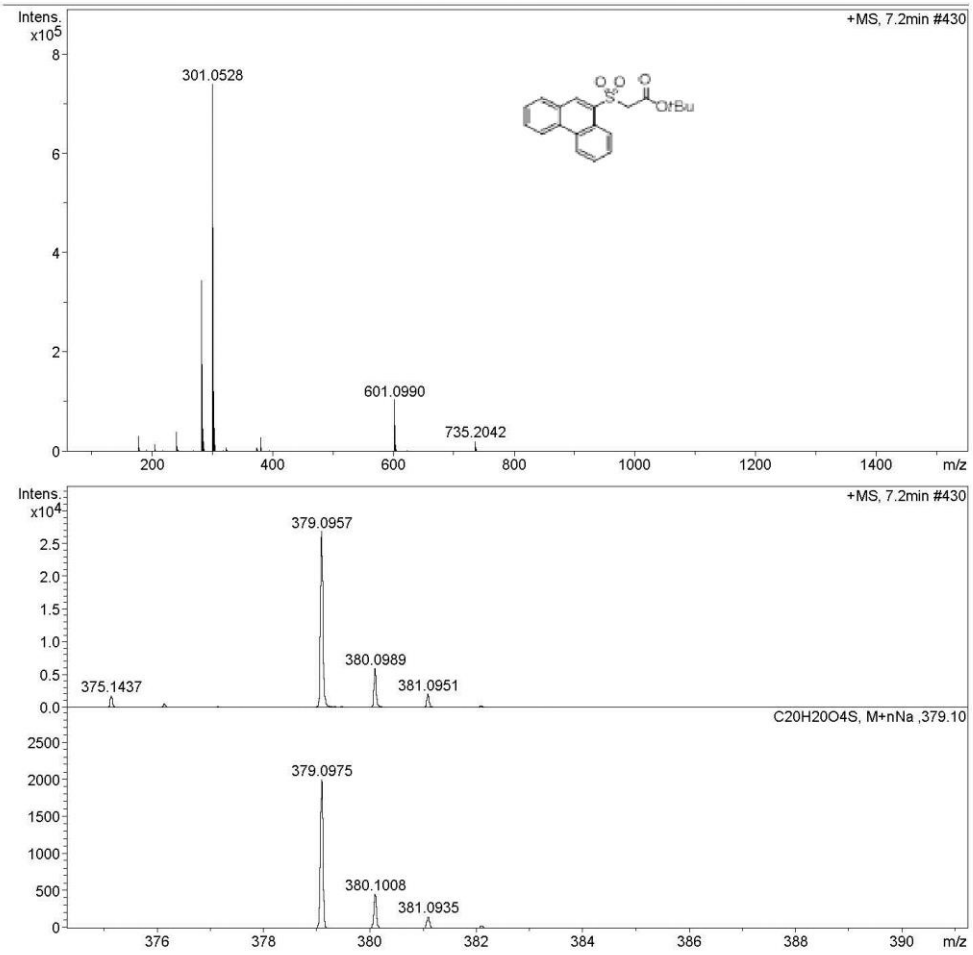
Display Report

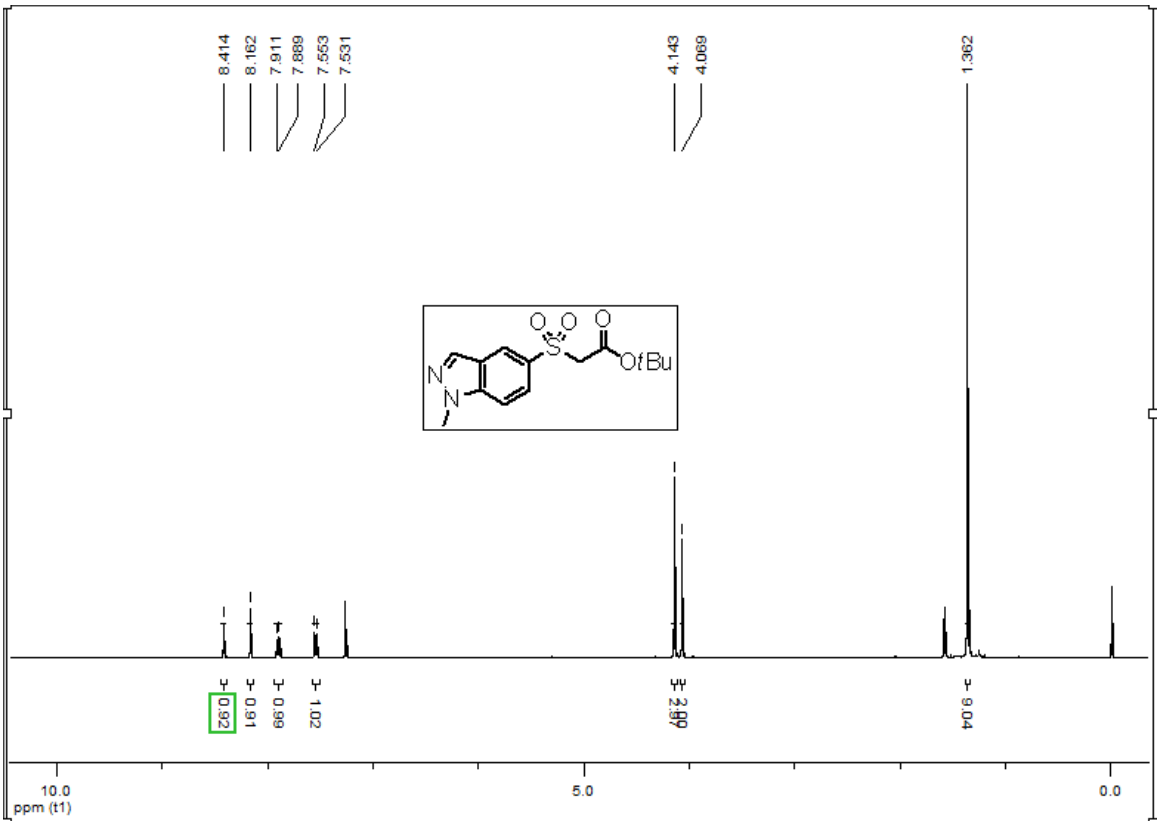
Analysis Info

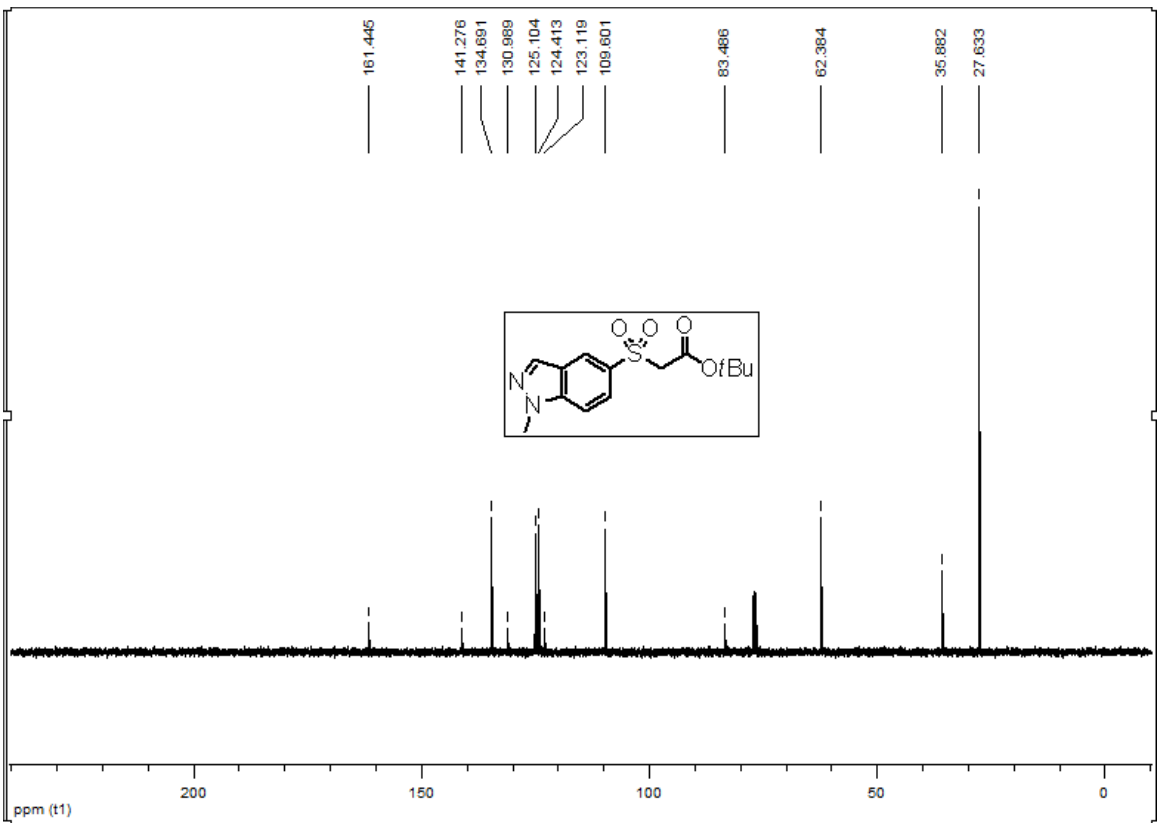
Analysis Name	D:\Data\2016MS\TT\0606\H12_RD7_01_9833.d	Acquisition Date	6/7/2016 12:43:12 AM
Method	tune_200-800_hcoona-pos-10min.m	Operator	gftang
Sample Name	H12	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	100 m/z	Set Capillary	4000 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste




8.414
8.162
7.911
7.889
7.553
7.531
4.143
4.069
1.362
OtBu
0.92
0.91
0.99
1.02
2.89
9.04
10.0
5.0
0.0
ppm (t1)


161.445
141.276
134.691
130.989
125.104
124.413
123.119
109.601
83.486
62.384
35.882
27.633
OtBu
200
150
100
50
0
ppm (t1)

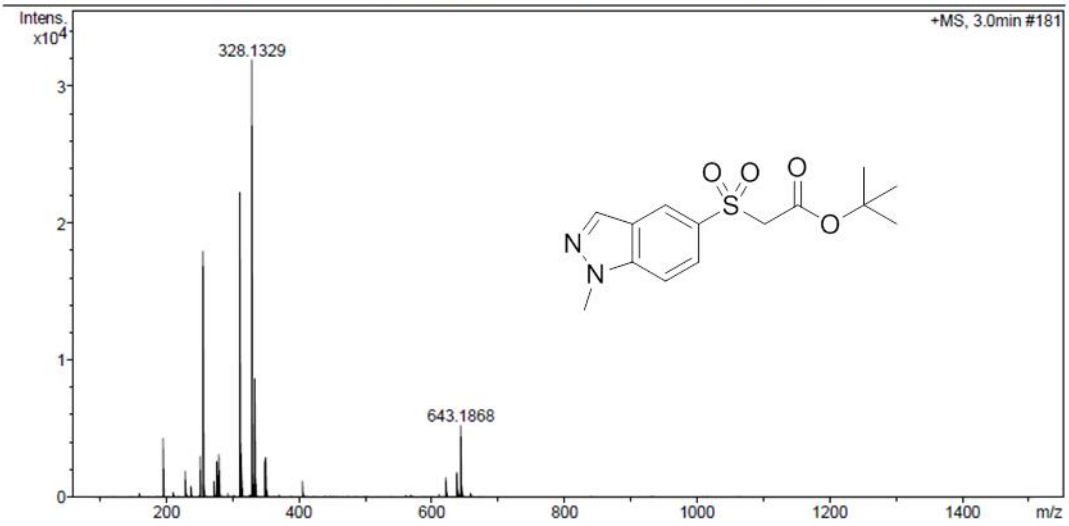
Display Report

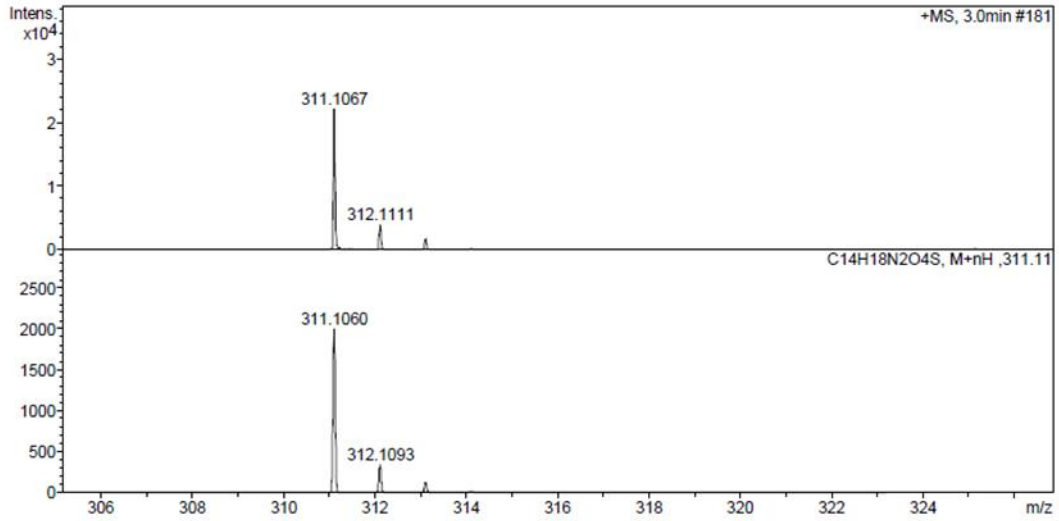
Analysis Info

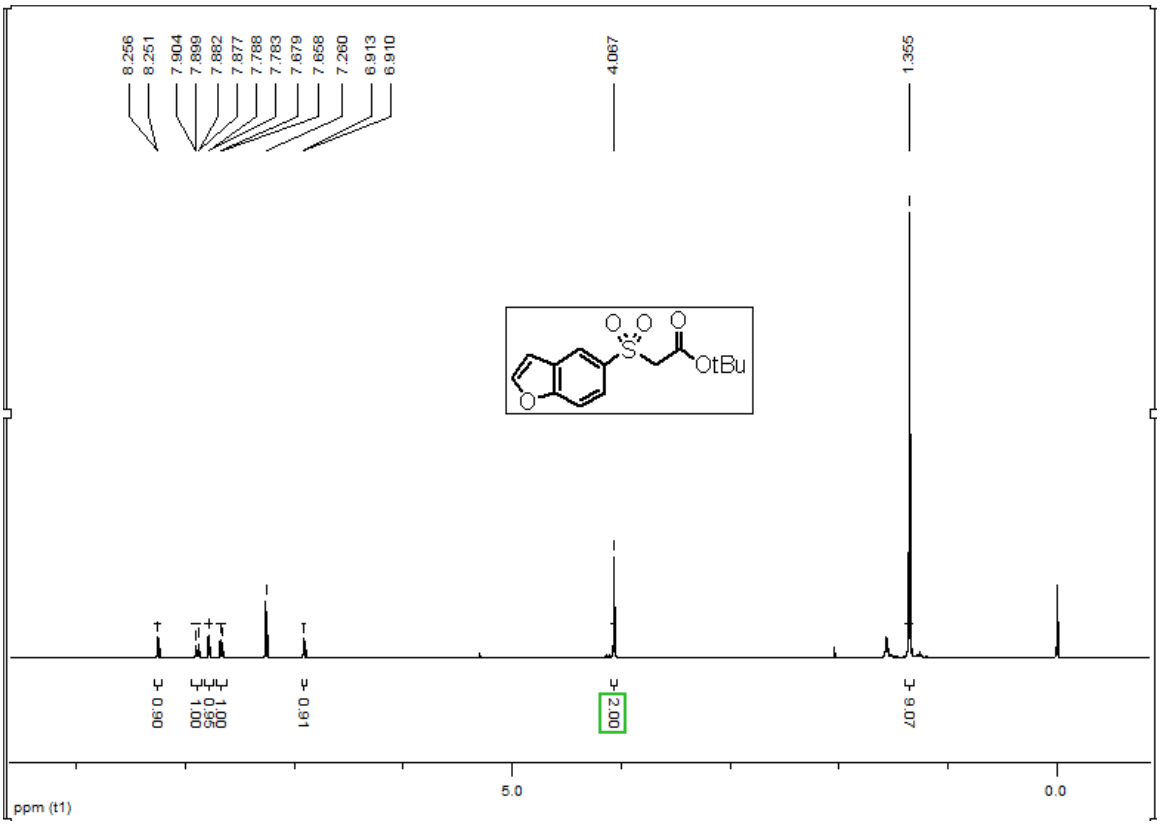
Analysis Name	D:\Data\2016MS\TT\1202\H3_RB3_01_12551.d	Acquisition Date	12/2/2016 10:04:52 PM
Method	tune_200-800_hcoona-pos-8min.m	Operator	gftang
Sample Name	H3	Instrument / Ser#	micrOTOF II 10257
Comment			

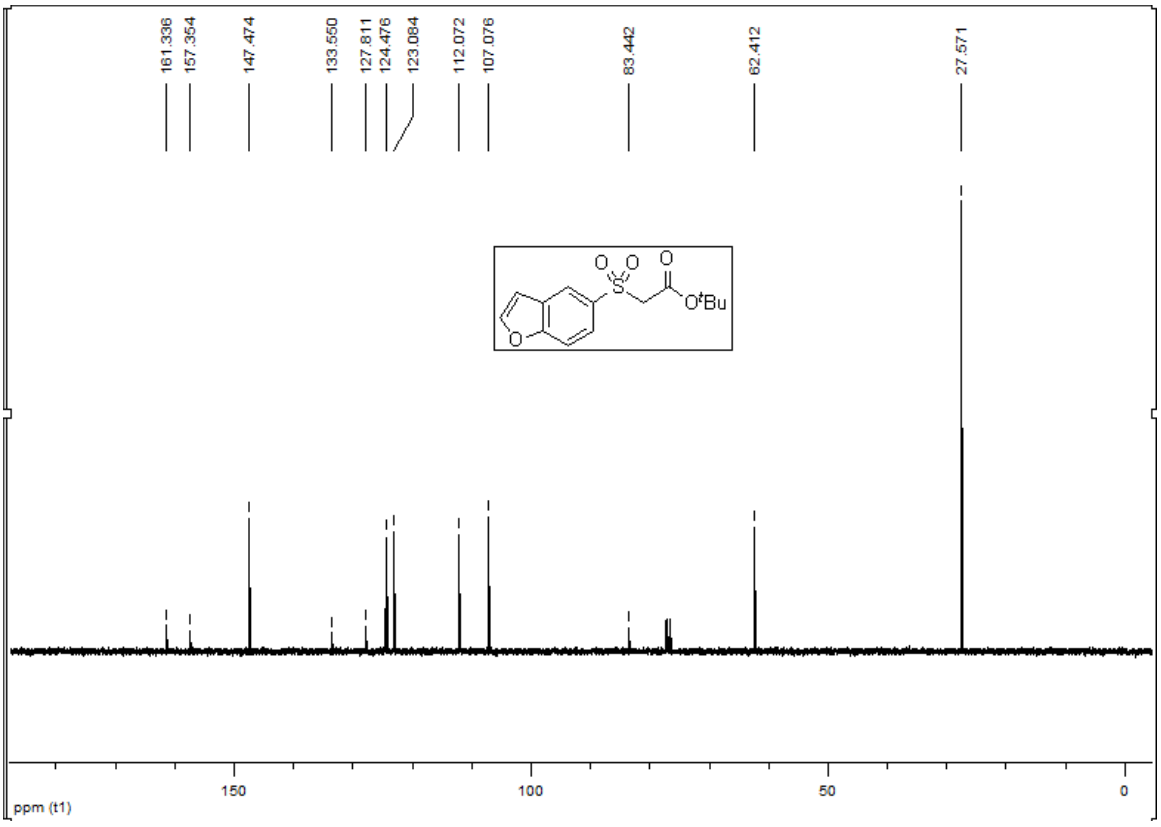
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	100 m/z	Set Capillary	4000 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste






8.256
8.251
7.904
7.899
7.882
7.877
7.788
7.783
7.679
7.658
7.260
6.913
6.910
4.067
1.355
OtBu
0.90
1.00
0.96
1.00
0.91
2.00
9.07
5.0
0.0
ppm (t1)


161.336
157.354
147.474
133.550
127.811
124.476
123.084
112.072
107.076
83.442
62.412
27.571
OtBu
150
100
50
0
ppm (t1)

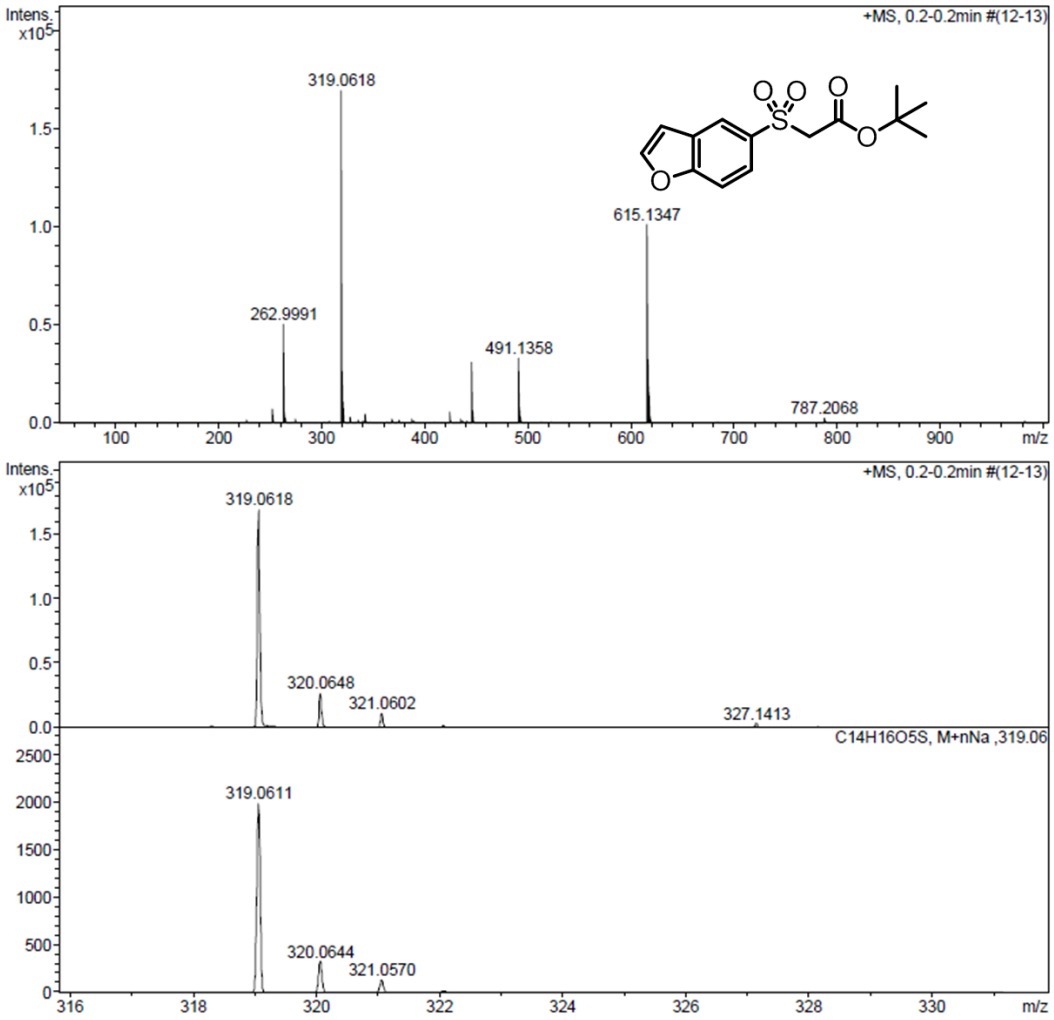
Display Report

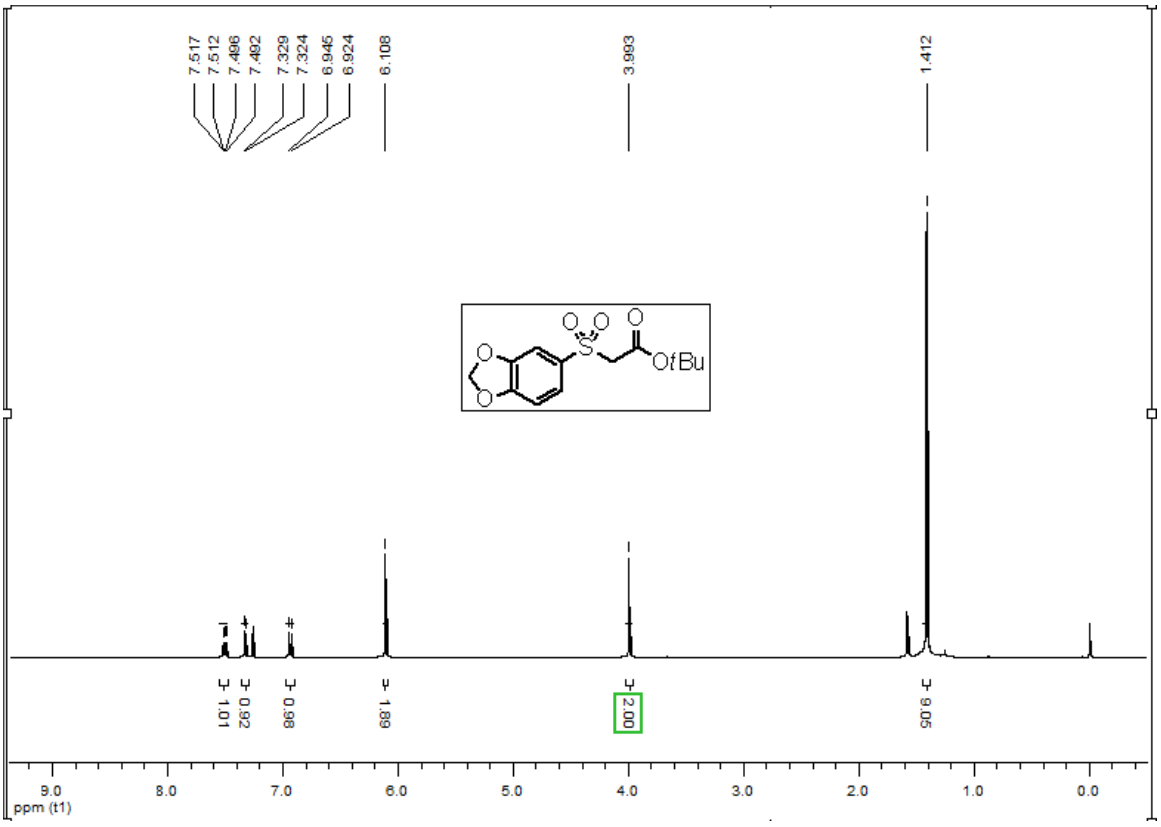
Analysis Info

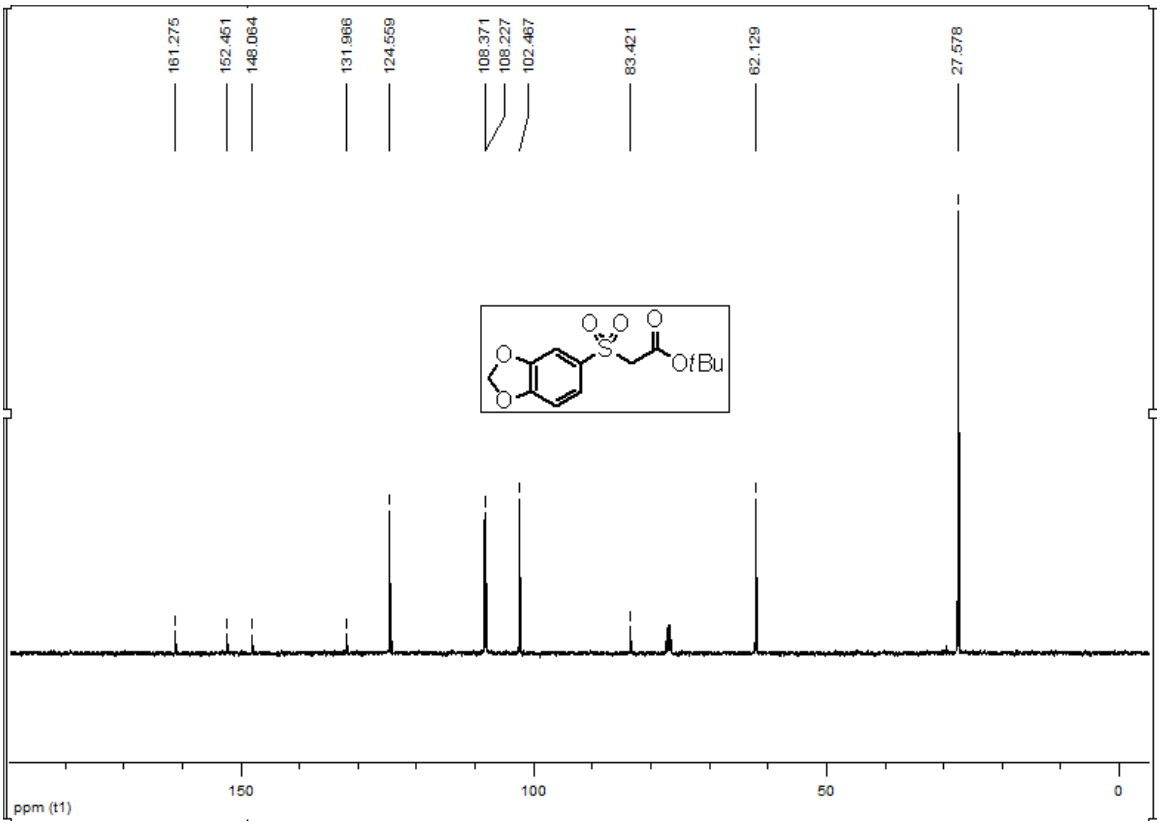
Analysis Name	D:\Data\2016MS\TT\0908\H-9_RE4_01_11002.d	Acquisition Date	9/8/2016 2:04:30 PM
Method	tune_200-800_hcoona-pos-2.5min.m	Operator	gftang
Sample Name	H-9	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.6 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	2500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste




7.517
7.512
7.496
7.492
7.329
7.324
6.945
6.924
6.108
3.993
1.412
1.01
0.92
0.98
1.89
2.00
9.06
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)
OtBu


161.275
152.451
148.064
131.966
124.559
108.371
108.227
102.467
83.421
62.129
27.578
150
100
50
0
ppm (t1)
OtBu

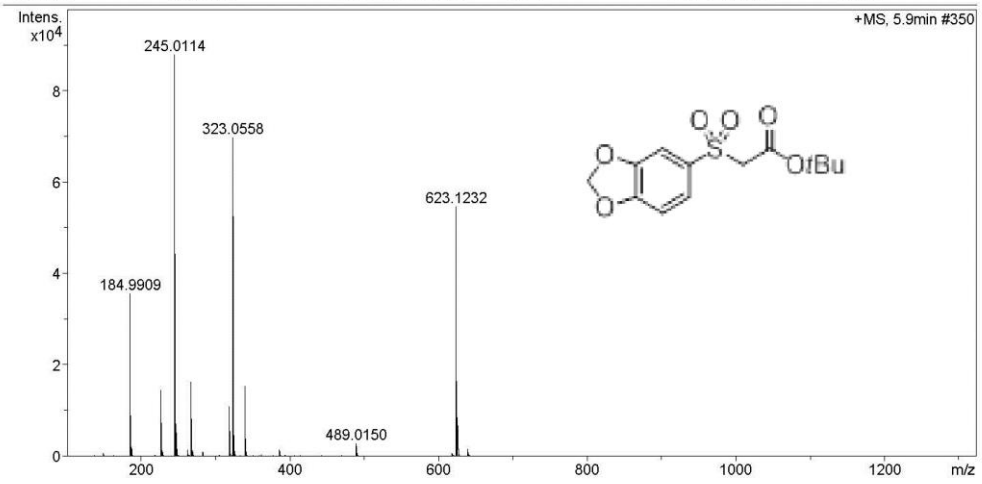
Display Report

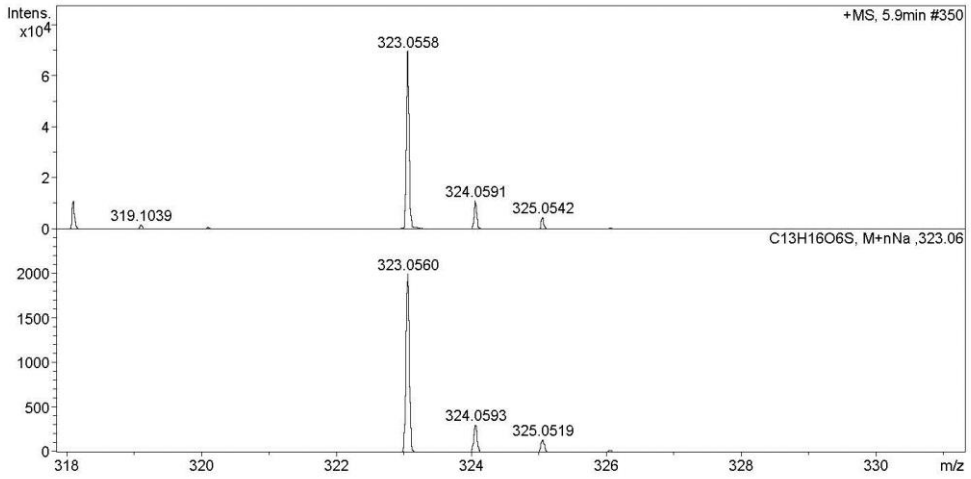
Analysis Info

Analysis Name	D:\Data\2016MS\TT\0606\H11_RD6_01_9832.d	Acquisition Date	6/7/2016 12:27:20 AM
Method	tune_200-800_hcoona-pos-10min.m	Operator	gftang
Sample Name	H11	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	100 m/z	Set Capillary	4000 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste





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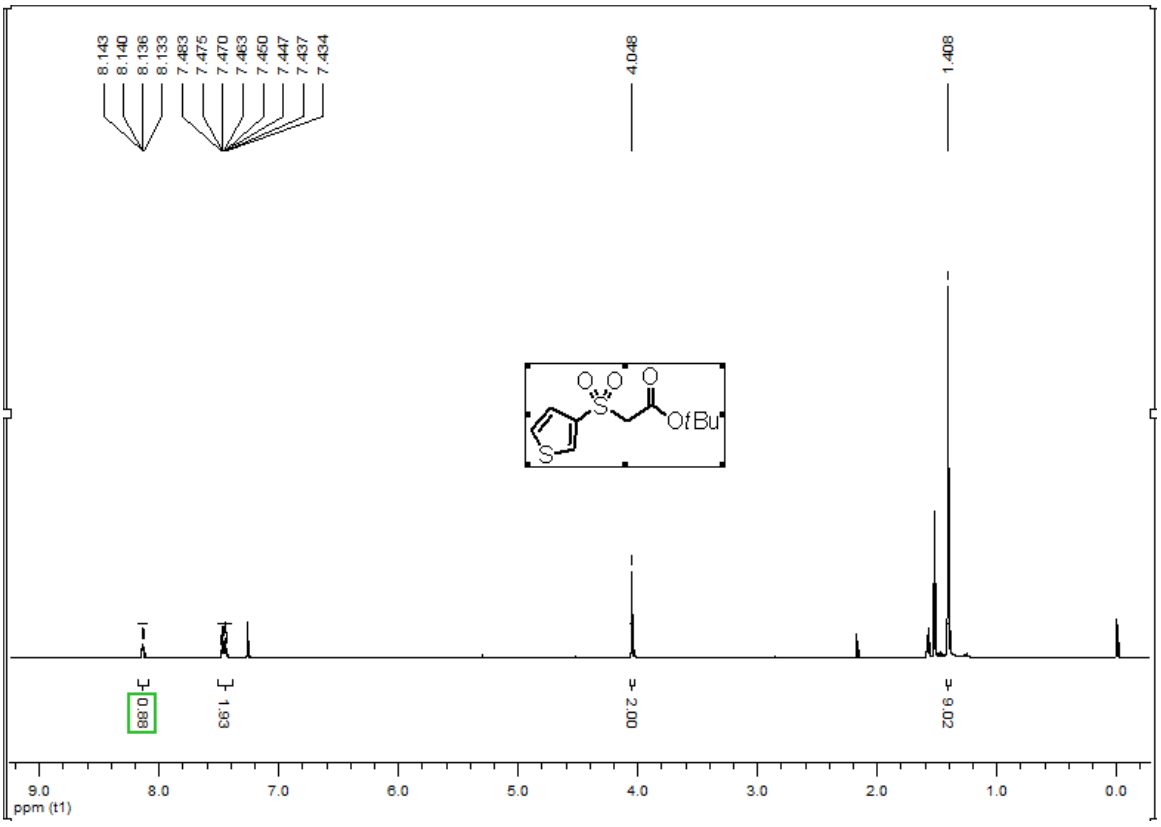
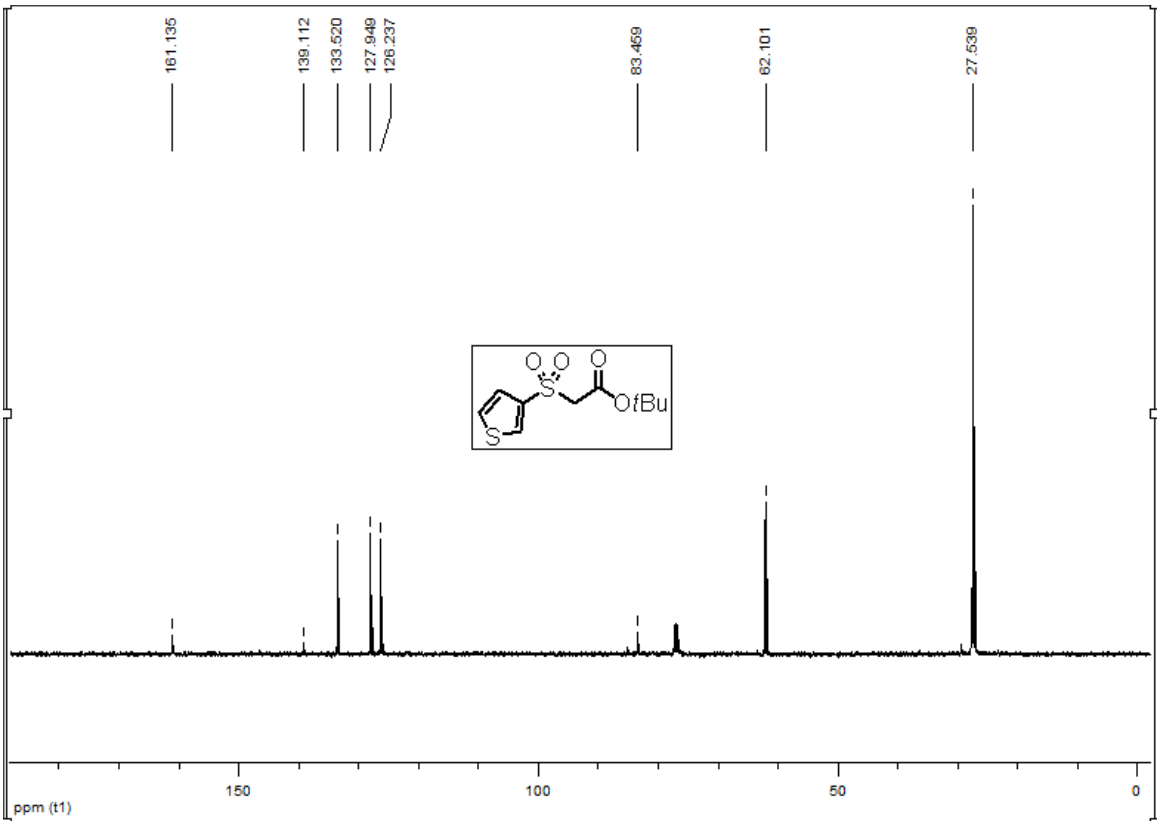
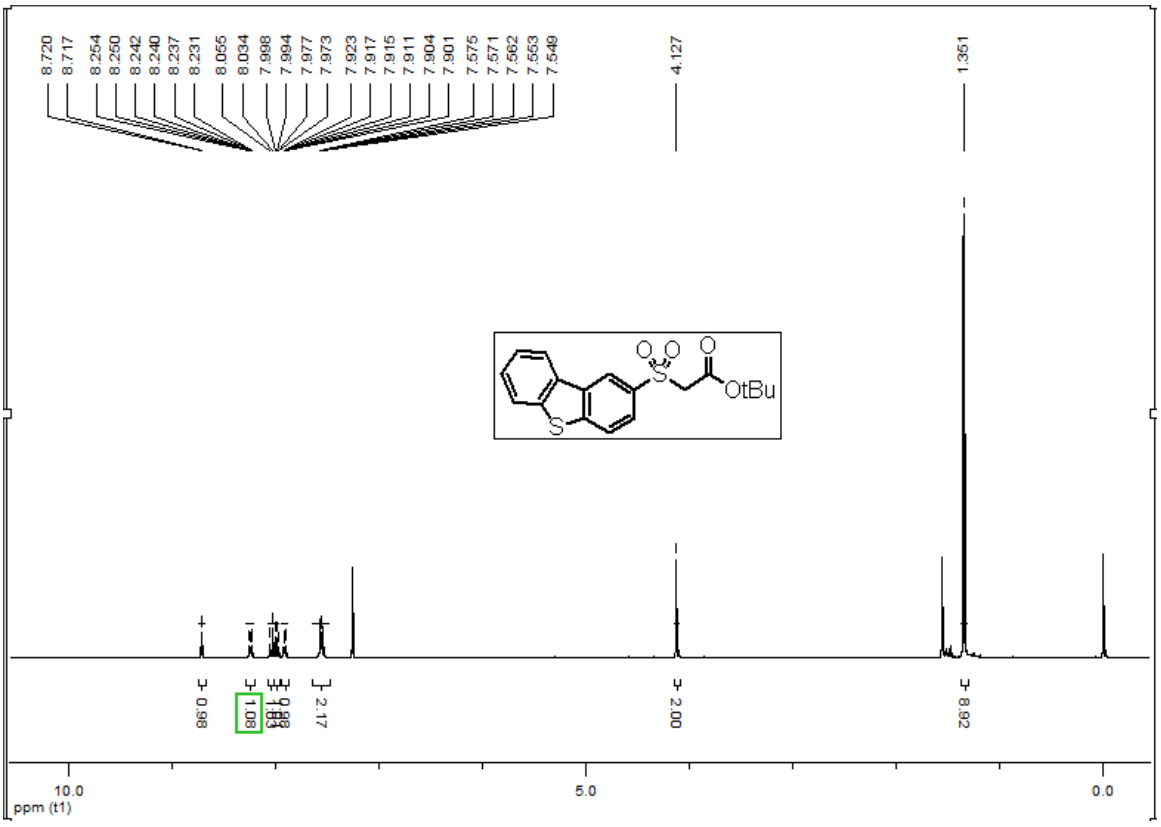
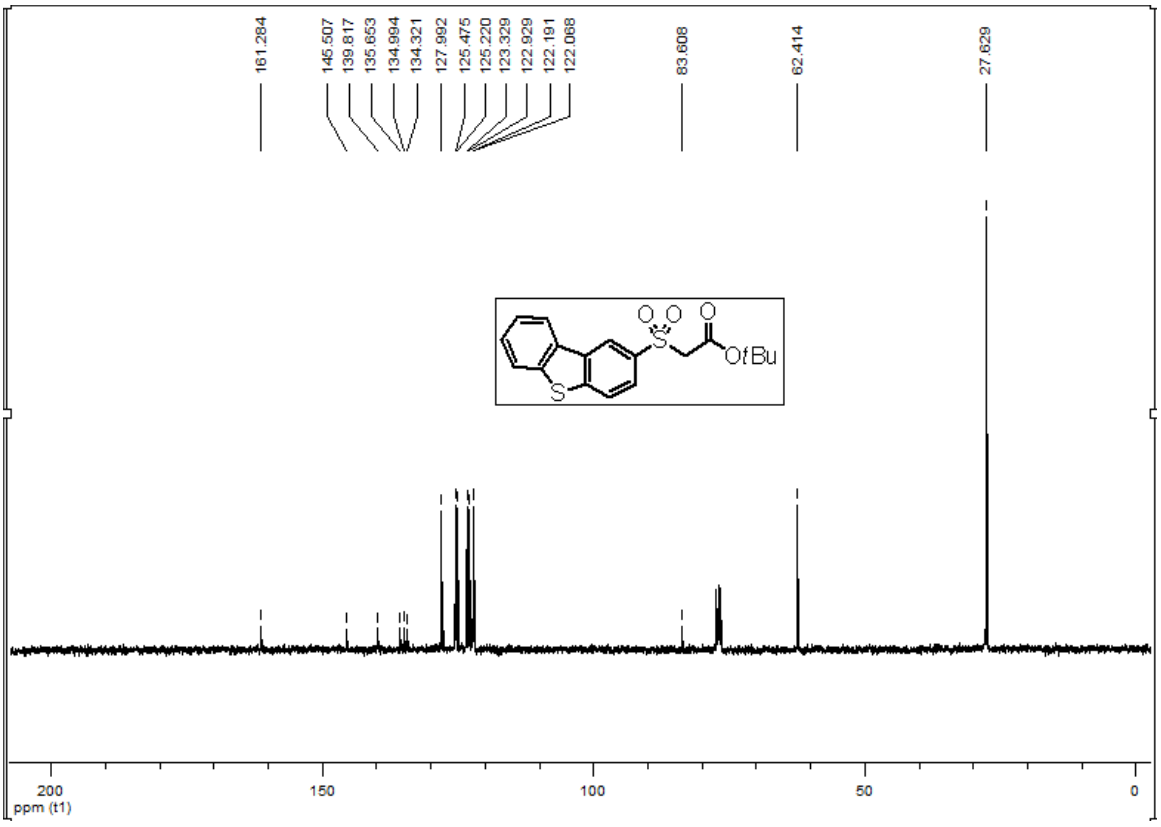

8.143
8.140
8.136
8.133
7.483
7.475
7.470
7.463
7.450
7.447
7.437
7.434
4.048
1.408
OtBu
0.88
1.93
2.00
9.02
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


161.135
139.112
133.520
127.949
126.237
83.459
62.101
27.539
OtBu
150
100
50
0
ppm (t1)


8.720
8.717
8.254
8.250
8.242
8.240
8.237
8.231
8.055
8.034
7.998
7.994
7.977
7.973
7.923
7.917
7.915
7.911
7.904
7.901
7.575
7.571
7.562
7.553
7.549
4.127
1.351
OtBu
0.98
1.08
2.17
2.00
8.92
10.0
5.0
0.0
ppm (t1)


161.284
145.507
139.817
135.653
134.994
134.321
127.992
125.475
125.220
123.329
122.929
122.191
122.068
83.608
62.414
27.629
OtBu
200
150
100
50
0
ppm (t1)

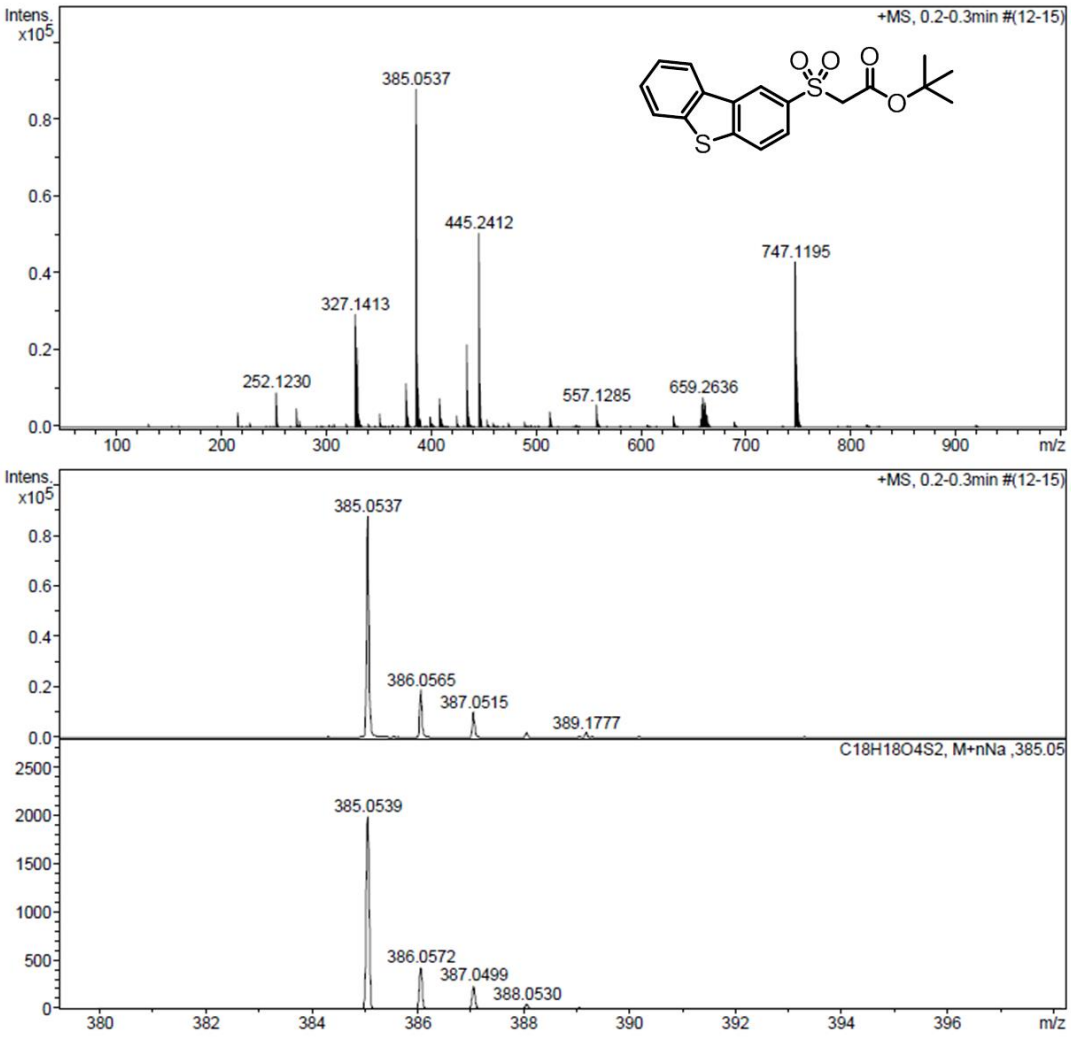
Display Report

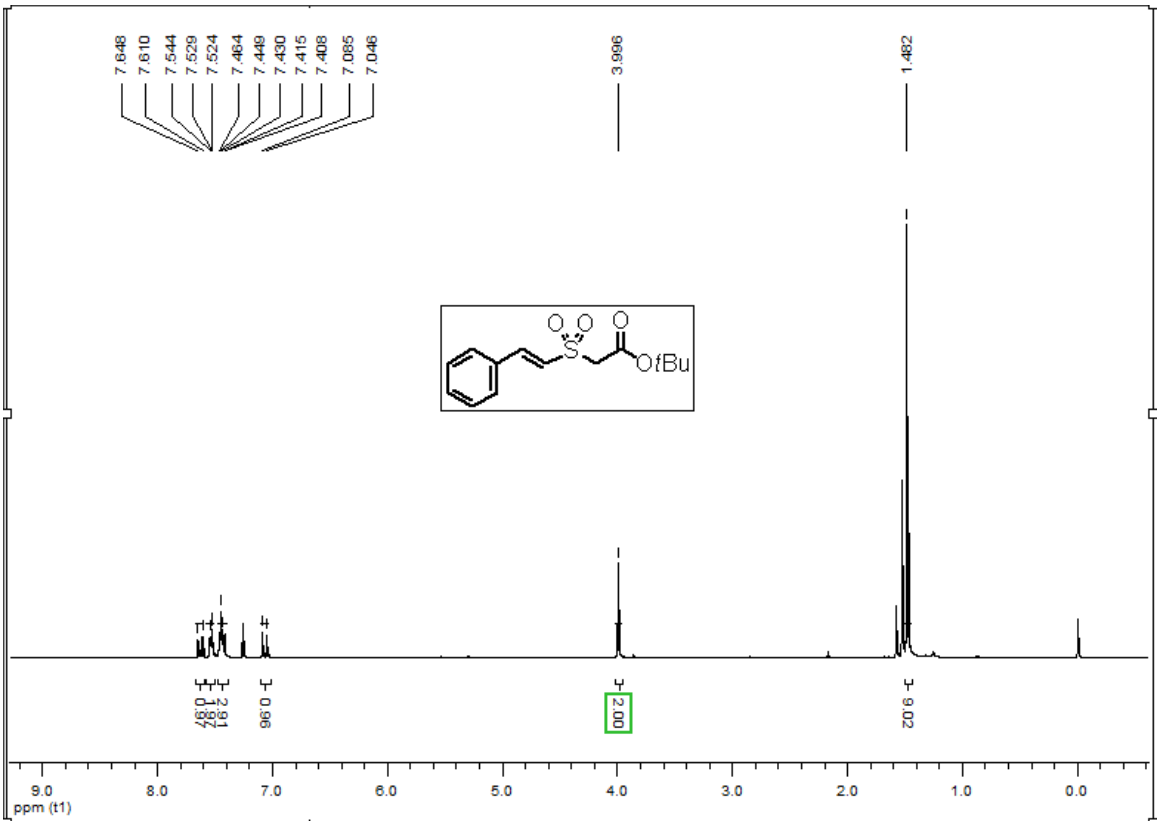
Analysis Info

Analysis Name	D:\Data\2016MS\TT\0908\H-8_RE3_01_11001.d	Acquisition Date	9/8/2016 2:00:27 PM
Method	tune_200-800_hcoona-pos-2.5min.m	Operator	gftang
Sample Name	H-8	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.6 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	2500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

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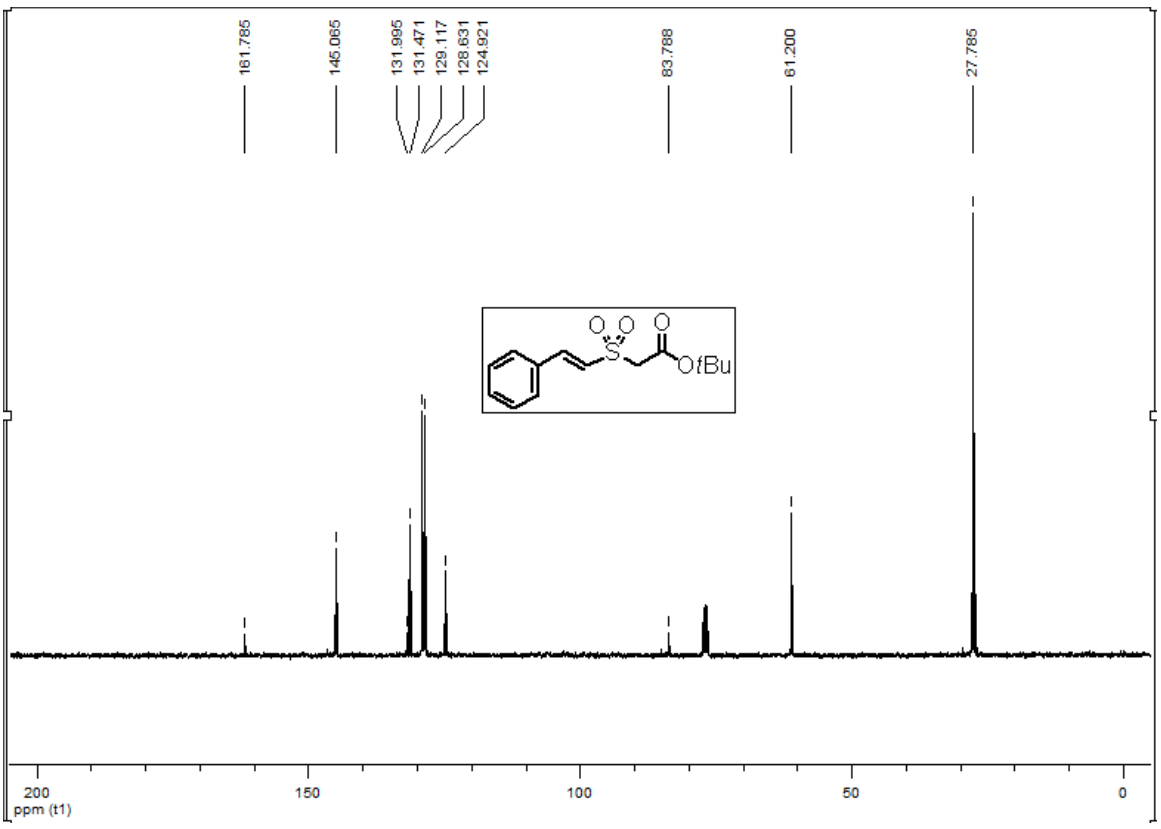
7.610
7.544
7.529
7.524
7.464
7.449
7.430
7.415
7.408
7.085
7.046
3.996
1.482
OtBu
2.00
9.02
ppm (t1)


161.785
145.065
131.995
131.471
129.117
128.631
124.921
83.788
61.200
27.785
OtBu
ppm (t1)

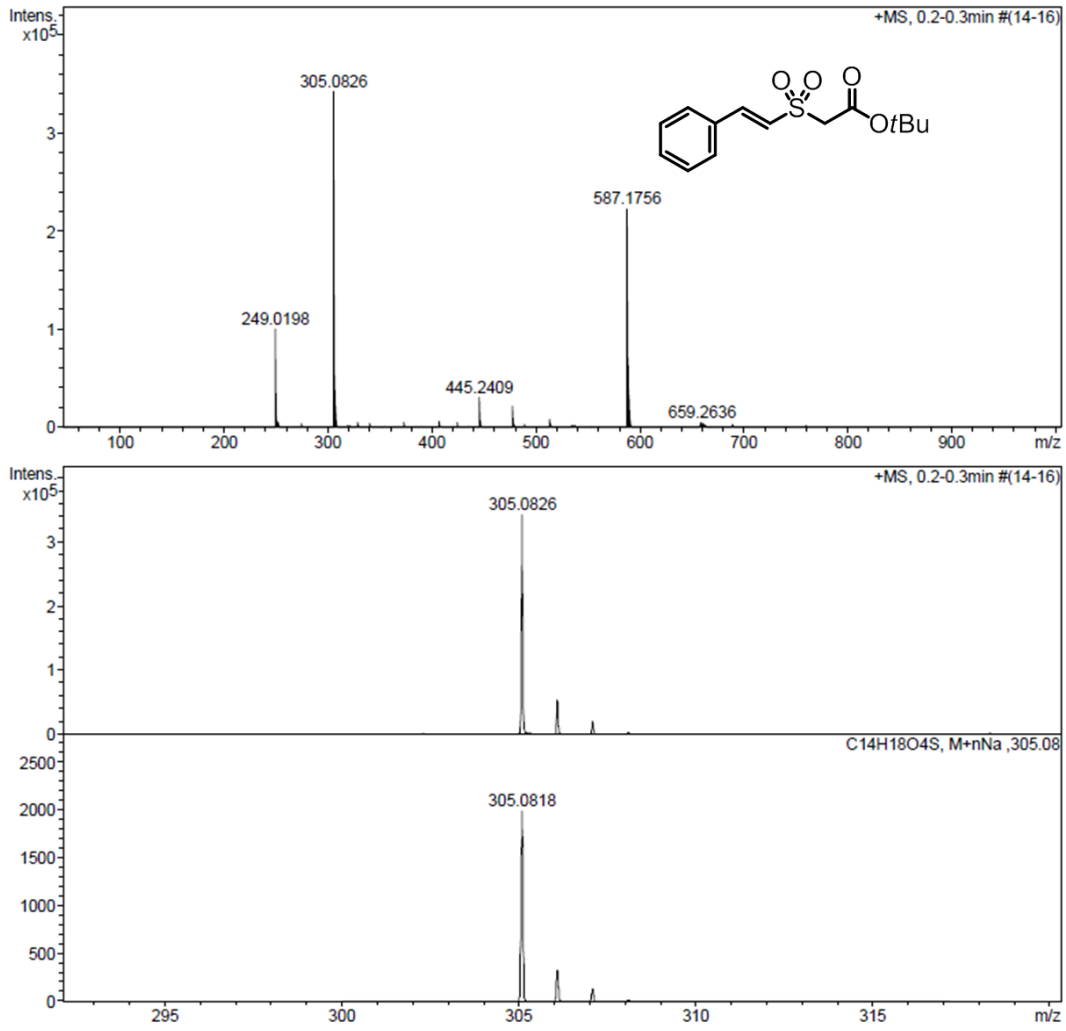
Display Report

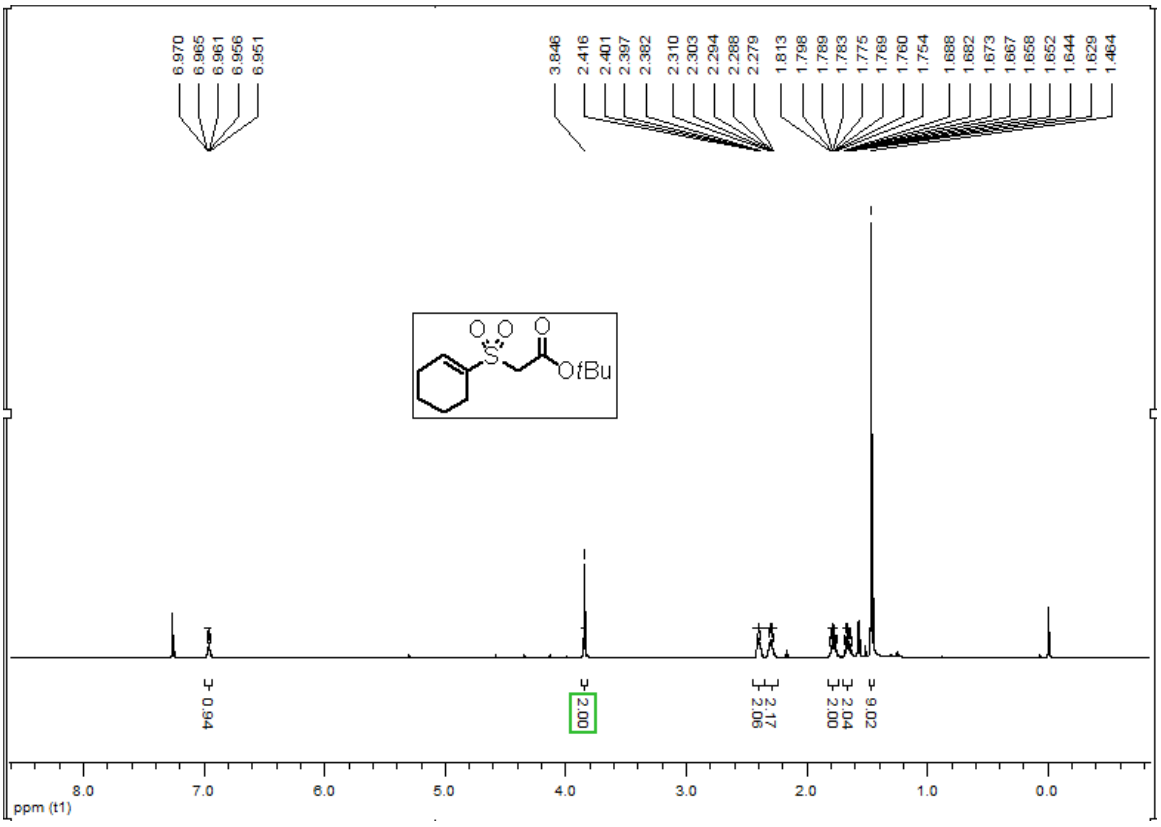
Analysis Info

Analysis Name	D:\Data\2016MS\TT\0908\H-5_RD8_01_10998.d	Acquisition Date	9/8/2016 1:48:19 PM
Method	tune_200-800_hcoona-pos-2.5min.m	Operator	gftang
Sample Name	H-5	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.6 Bar
Focus	Not active			Set Dry Heater	180 ℃
Scan Begin	50 m/z	Set Capillary	2500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

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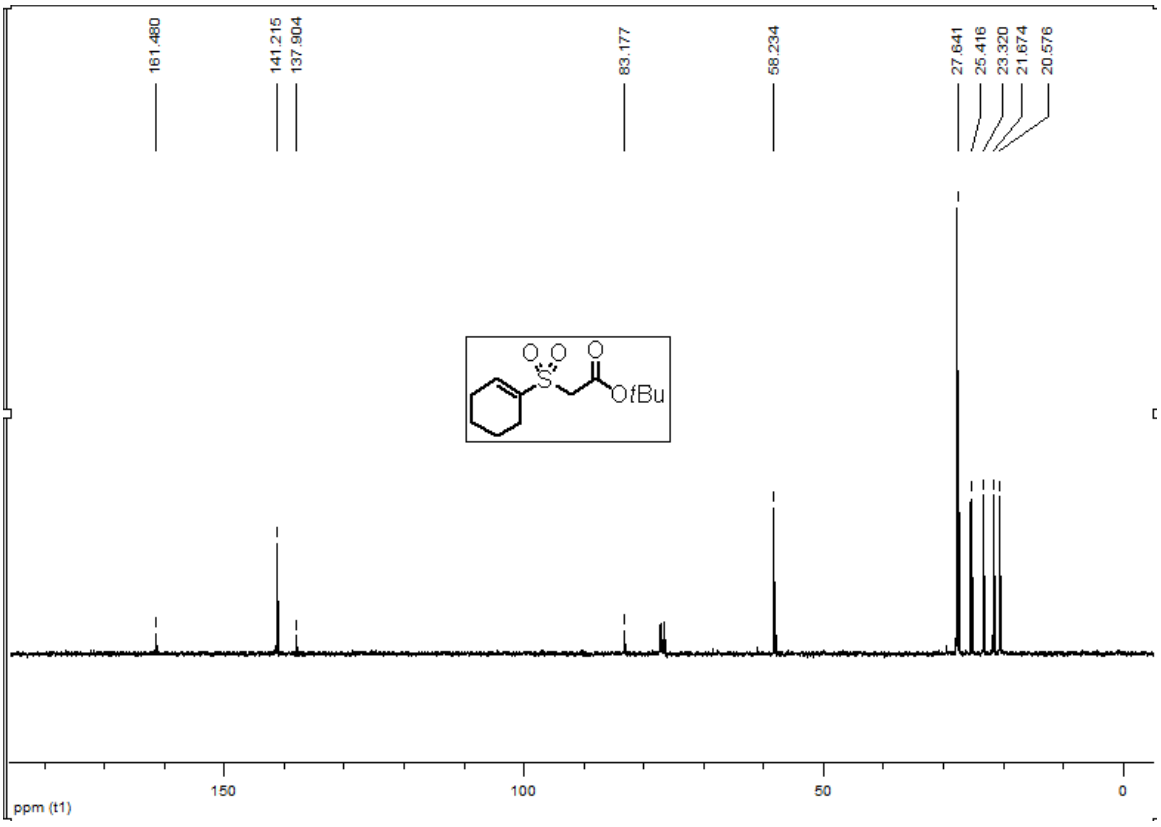
6.961
6.956
6.951
3.846
2.416
2.401
2.397
2.382
2.310
2.303
2.294
2.288
2.279
1.813
1.798
1.789
1.783
1.775
1.769
1.760
1.754
1.688
1.682
1.673
1.667
1.658
1.652
1.644
1.629
1.464
OtBu
0.94
2.00
2.17
2.06
2.04
2.00
9.02
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


161.480
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137.904
83.177
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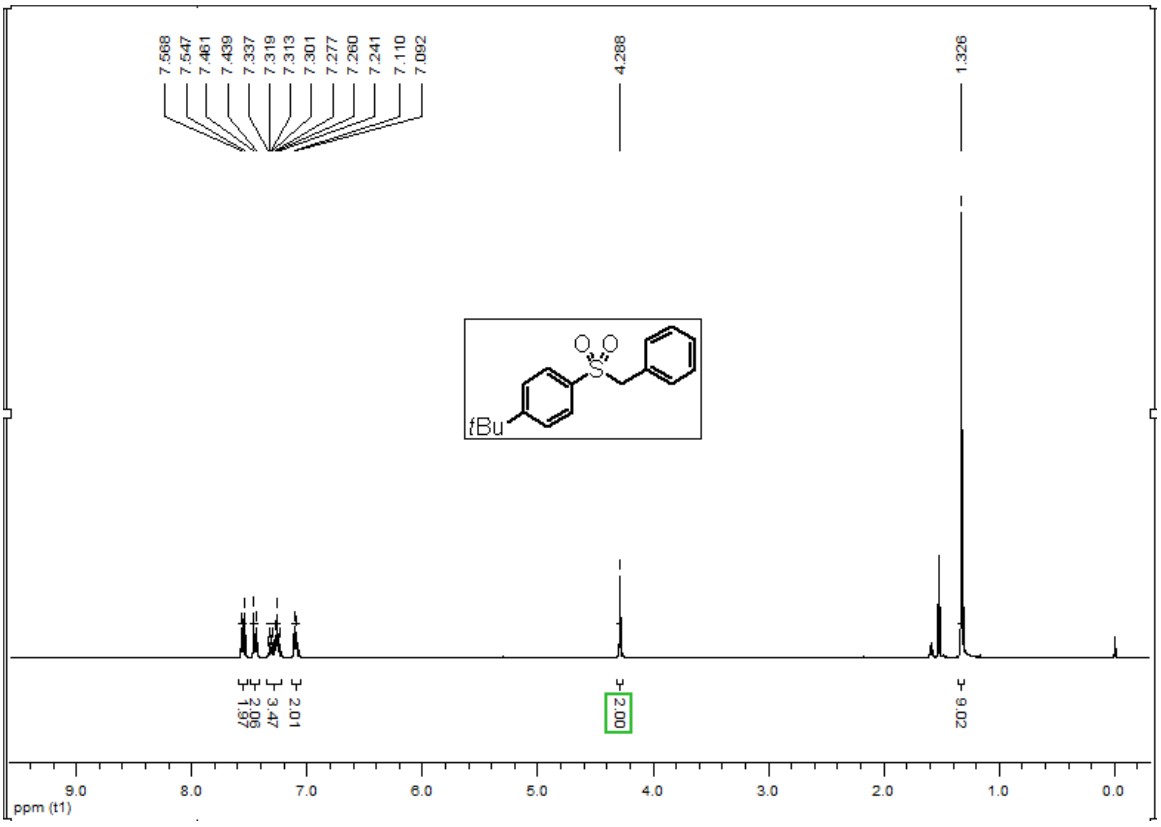
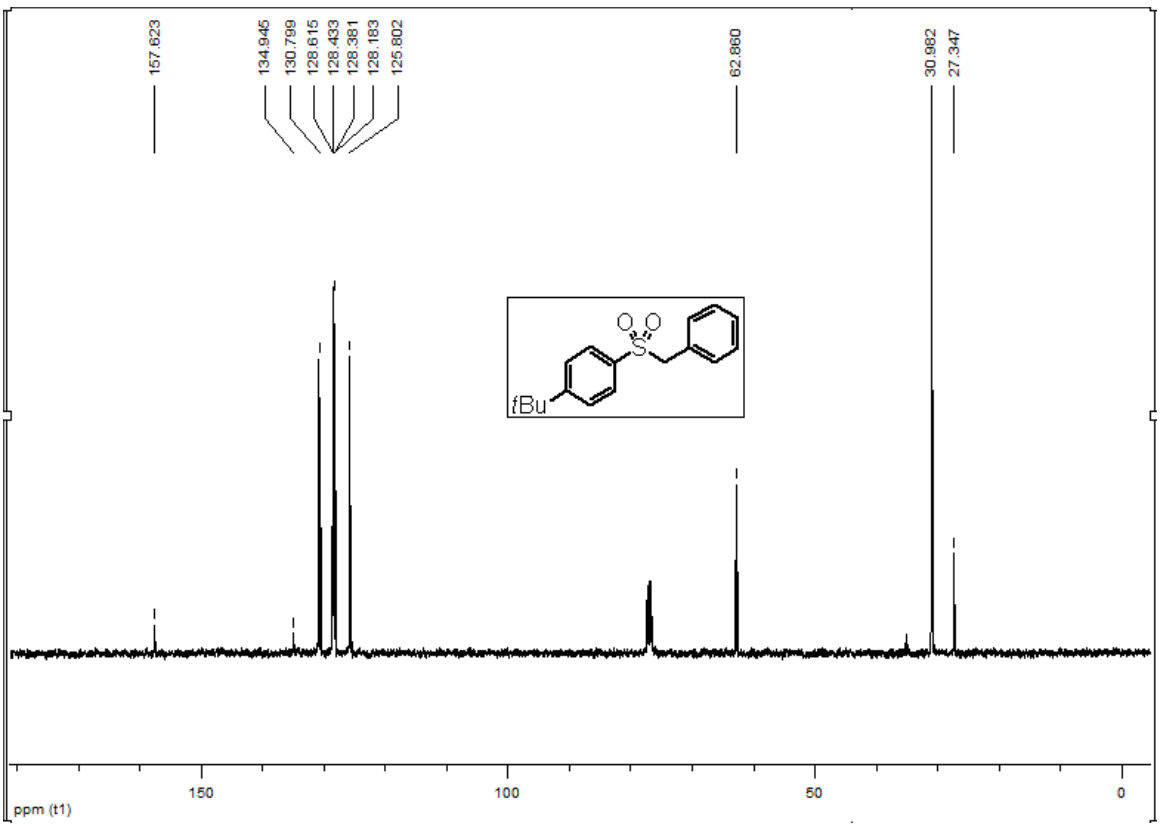
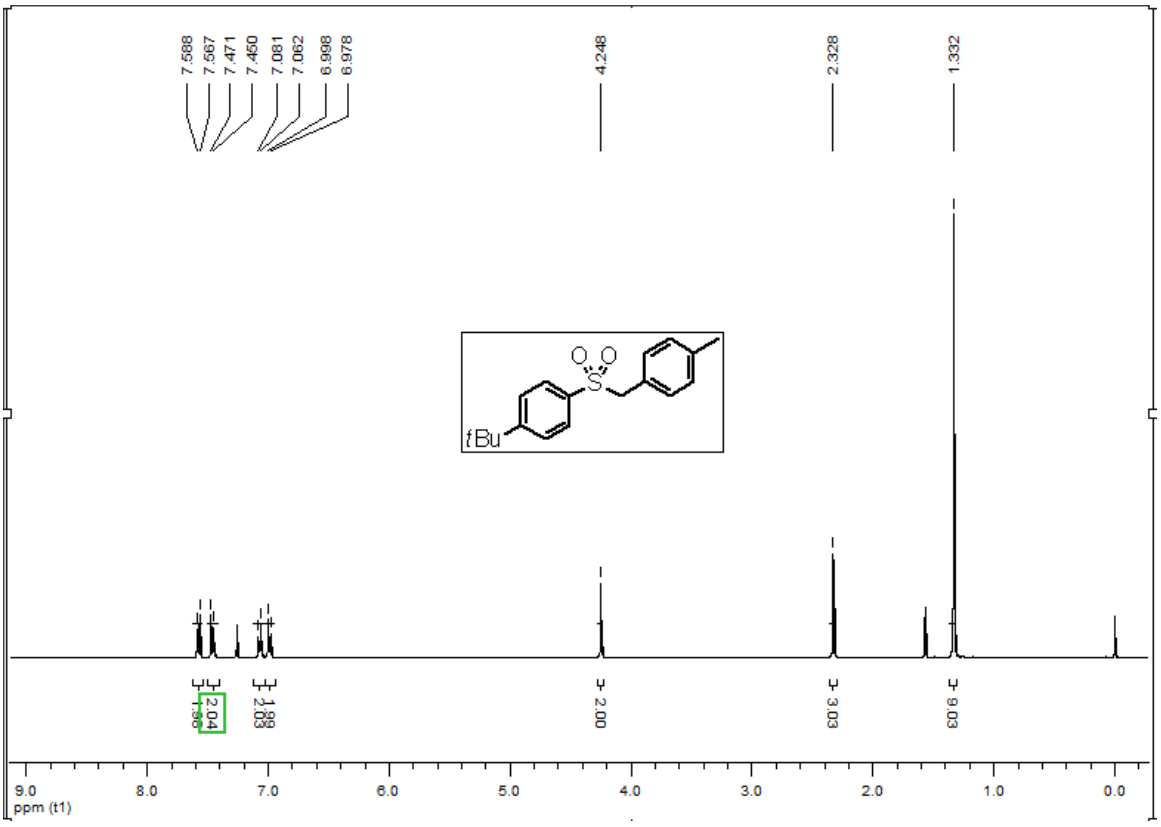
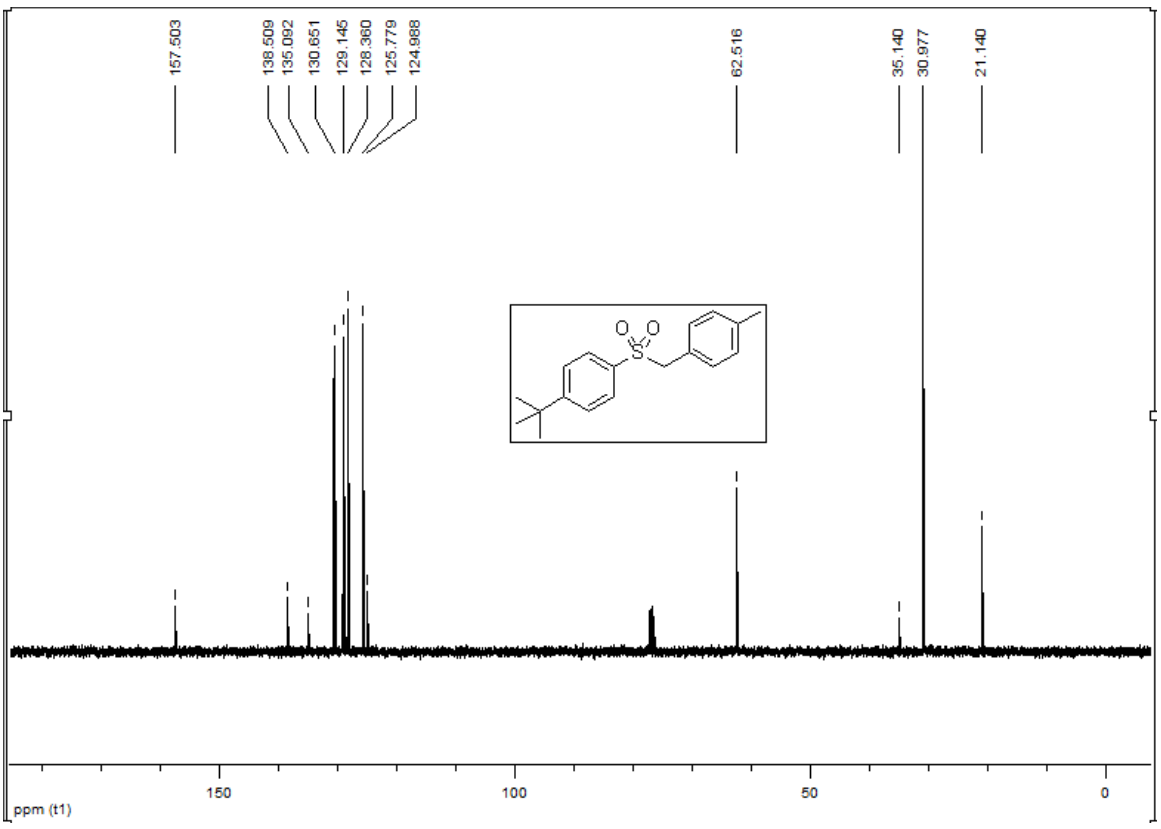
27.641
25.416
23.320
21.674
20.576
OtBu
150
100
50
0
ppm (t1)


7.568
7.547
7.461
7.439
7.337
7.319
7.313
7.301
7.277
7.260
7.241
7.110
7.092
4.288
1.326
tBu
2.09
1.97
3.47
2.01
2.00
9.02
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


157.623
134.945
130.799
128.615
128.433
128.381
128.183
125.802
62.860
30.982
27.347
tBu
150
100
50
0
ppm (t1)


7.588
7.567
7.471
7.450
7.081
7.062
6.998
6.978
4.248
2.328
1.332
tBu
1.96
2.04
3.88
2.00
3.03
9.03
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


157.503
138.509
135.092
130.651
129.145
128.360
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124.988
62.516
35.140
30.977
21.140
150
100
50
0
ppm (t1)

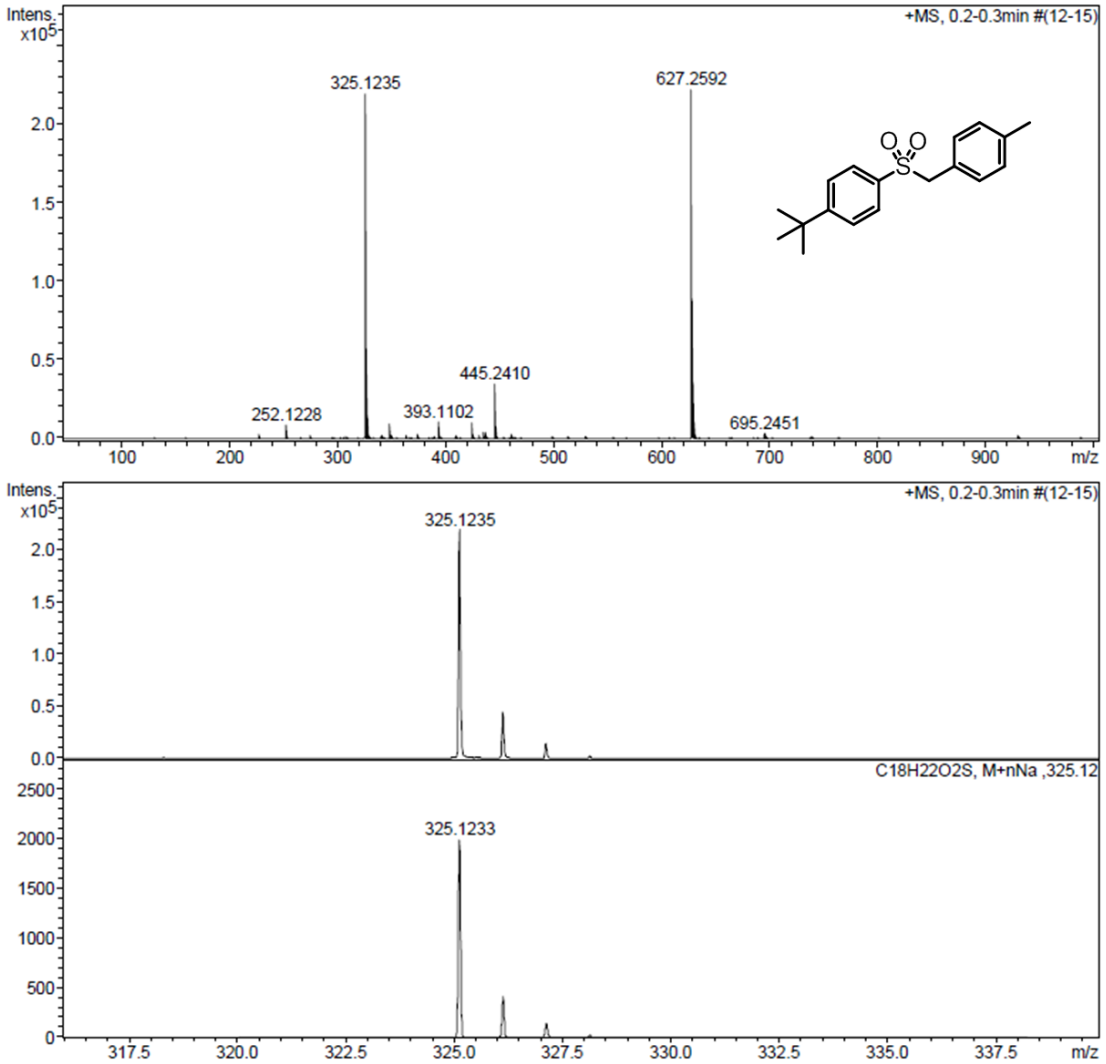
Display Report

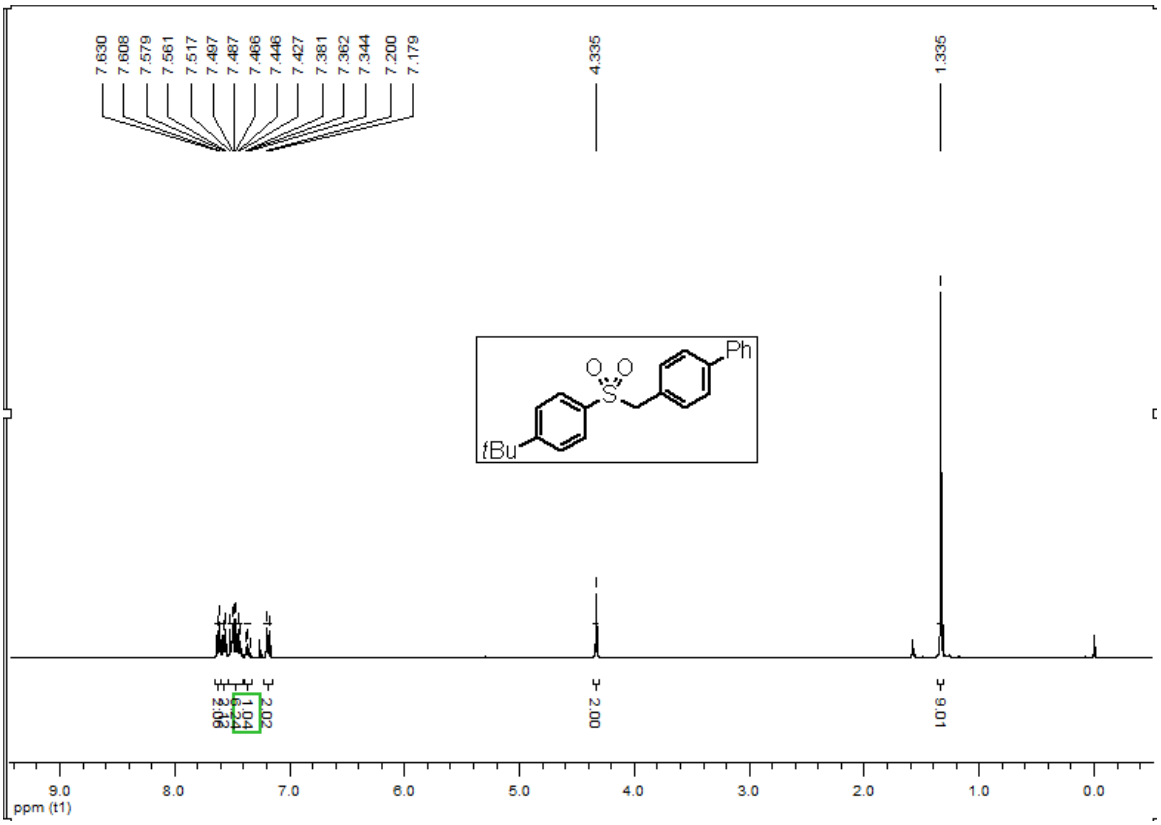
Analysis Info

Analysis Name	D:\Data\2016MS\TT\0908\H-7_RE2_01_11000.d	Acquisition Date	9/8/2016 1:56:24 PM
Method	tune_200-800_hcoona-pos-2.5min.m	Operator	gftang
Sample Name	H-7	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.6 Bar
Focus	Not active			Set Dry Heater	180 ℃
Scan Begin	50 m/z	Set Capillary	2500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

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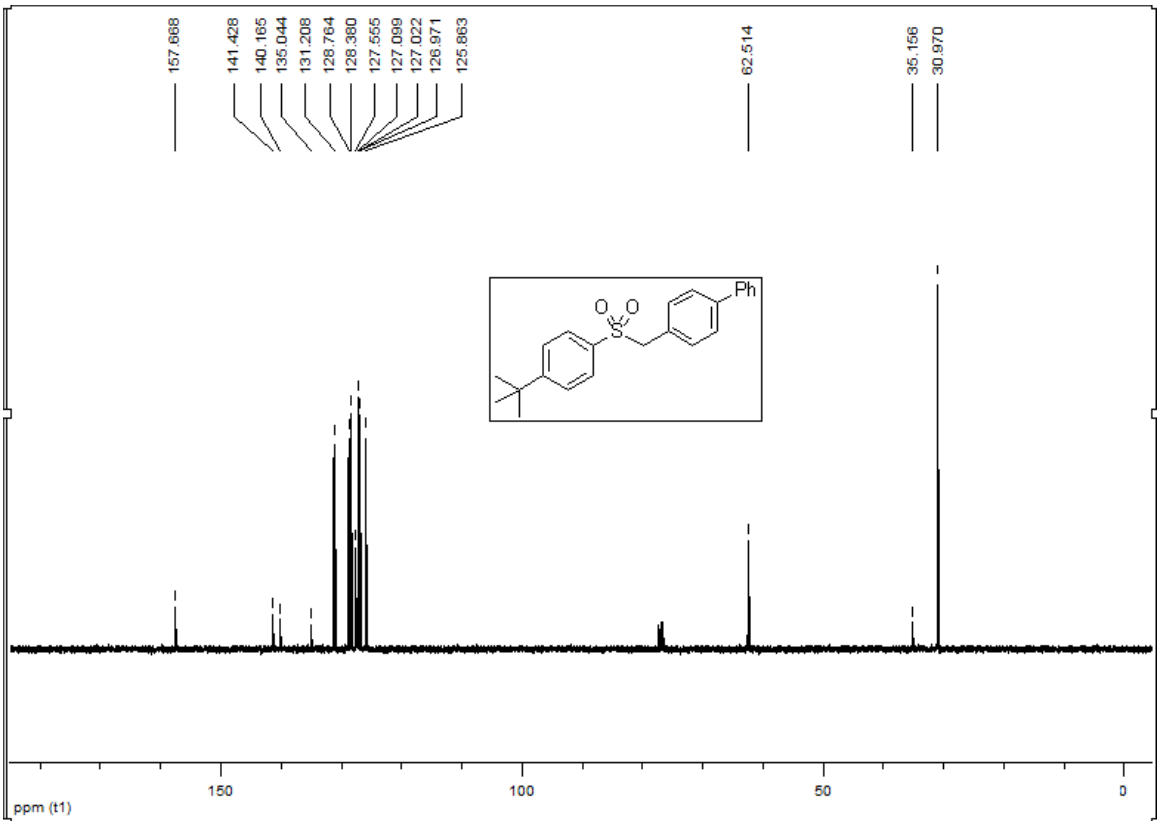
7.608
7.579
7.561
7.517
7.497
7.487
7.466
7.446
7.427
7.381
7.362
7.344
7.200
7.179
4.335
1.335
Ph
O
O
S
tBu
2.00
2.00
9.01
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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141.428
140.165
135.044
131.208
128.764
128.380
127.555
127.099
127.022
126.971
125.863
62.514
35.156
30.970
Ph
O
O
S
150
100
50
0
ppm (t1)

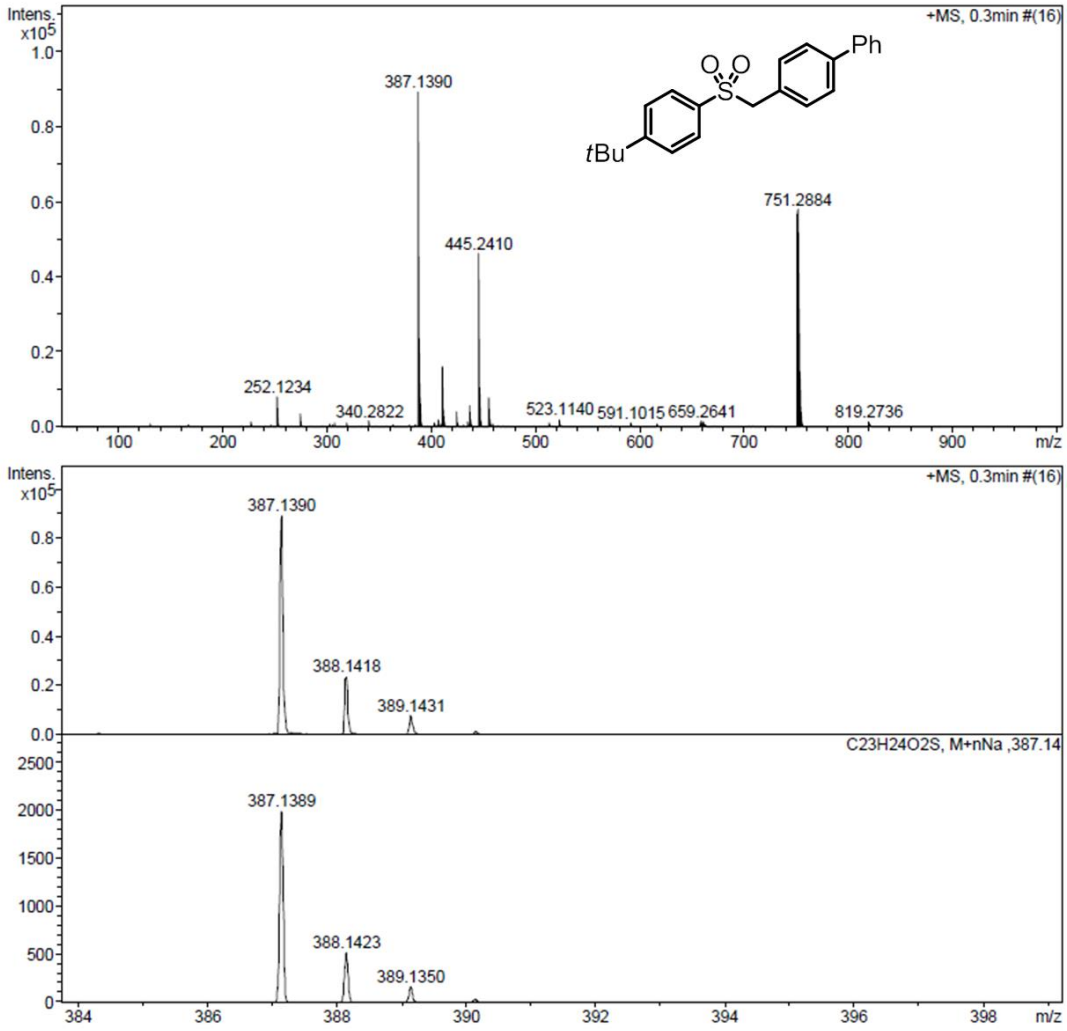
Display Report

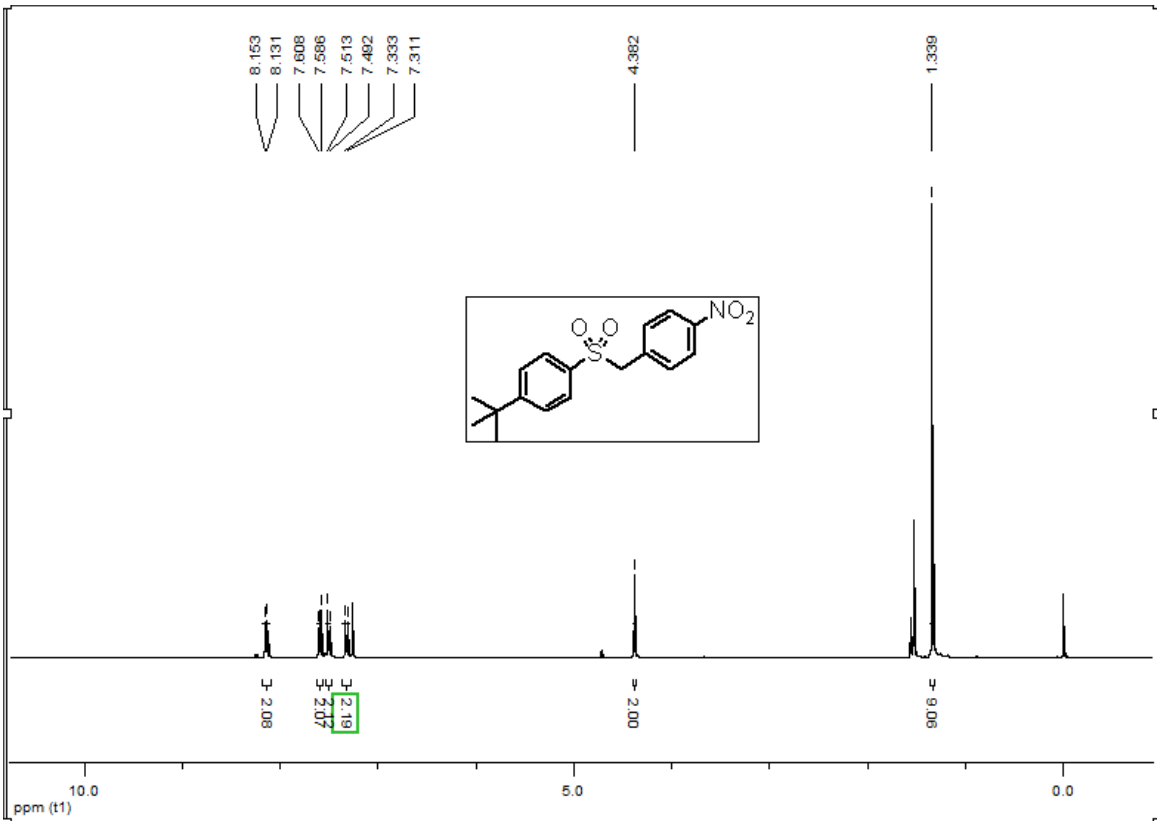
Analysis Info

Analysis Name	D:\Data\2016MS\TT\0908\H-6_RE1_01_10999.d	Acquisition Date	9/8/2016 1:52:21 PM
Method	tune_200-800_hcoona-pos-2.5min.m	Operator	gftang
Sample Name	H-6	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.6 Bar
Focus	Not active			Set Dry Heater	180 ℃
Scan Begin	50 m/z	Set Capillary	2500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

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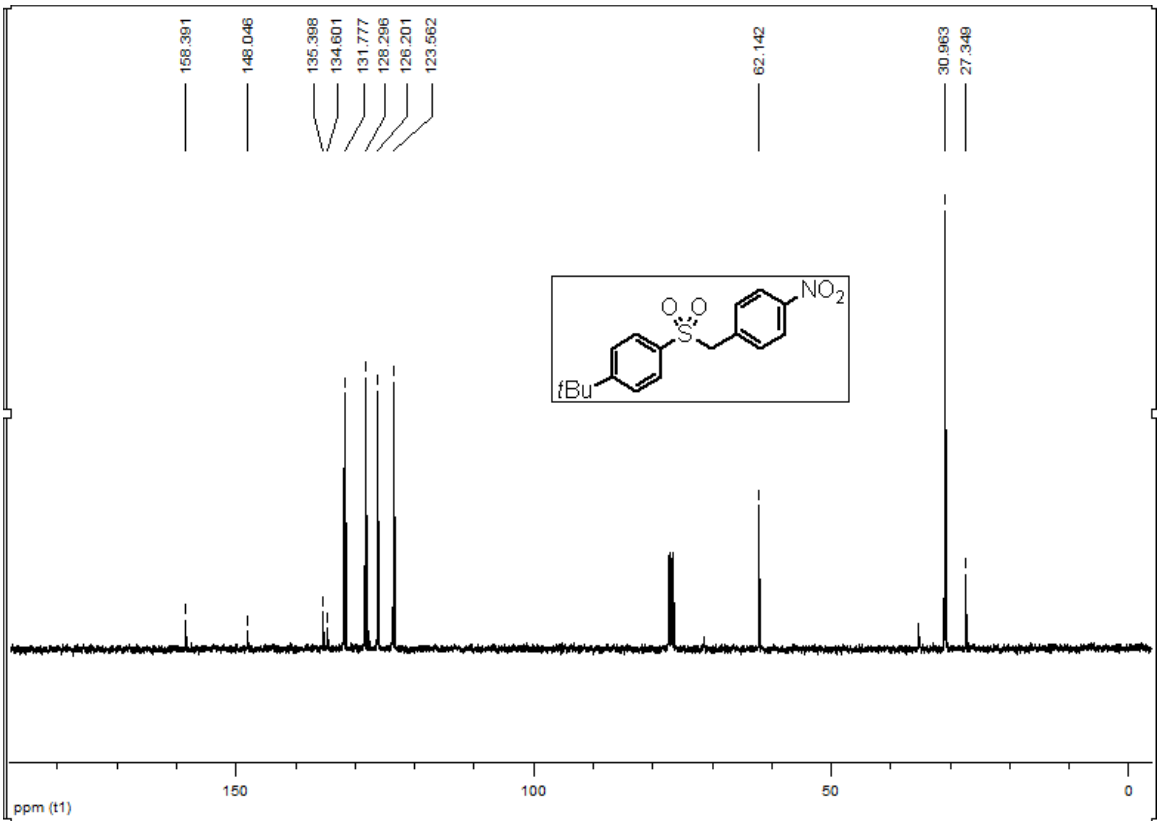
8.131
7.608
7.586
7.513
7.492
7.333
7.311
4.382
1.339
2.08
2.07
2.19
2.00
9.06
10.0
5.0
0.0
ppm (t1)
NO2
O
O
S


158.391
148.046
135.398
134.601
131.777
128.296
126.201
123.562
62.142
30.963
27.349
150
100
50
0
ppm (t1)
NO2
O
O
S
tBu

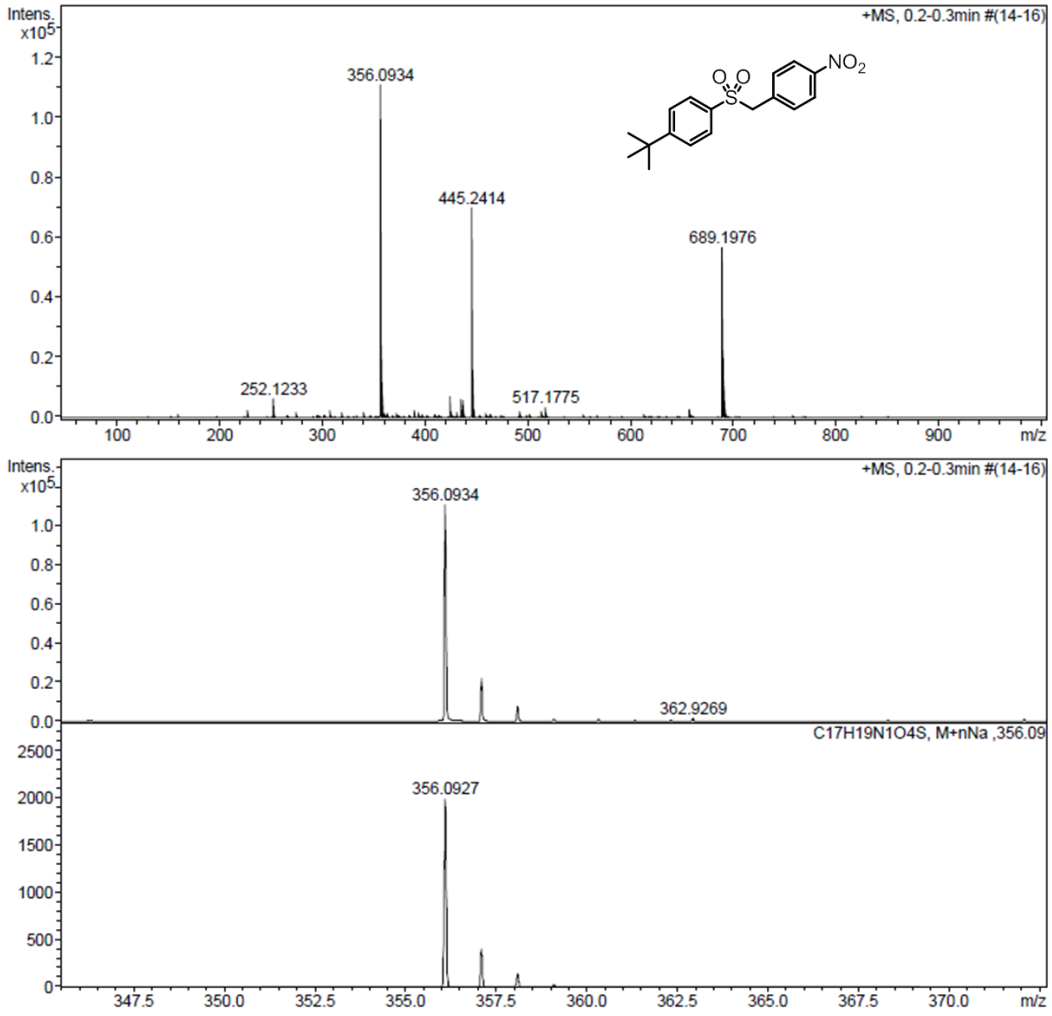
Display Report

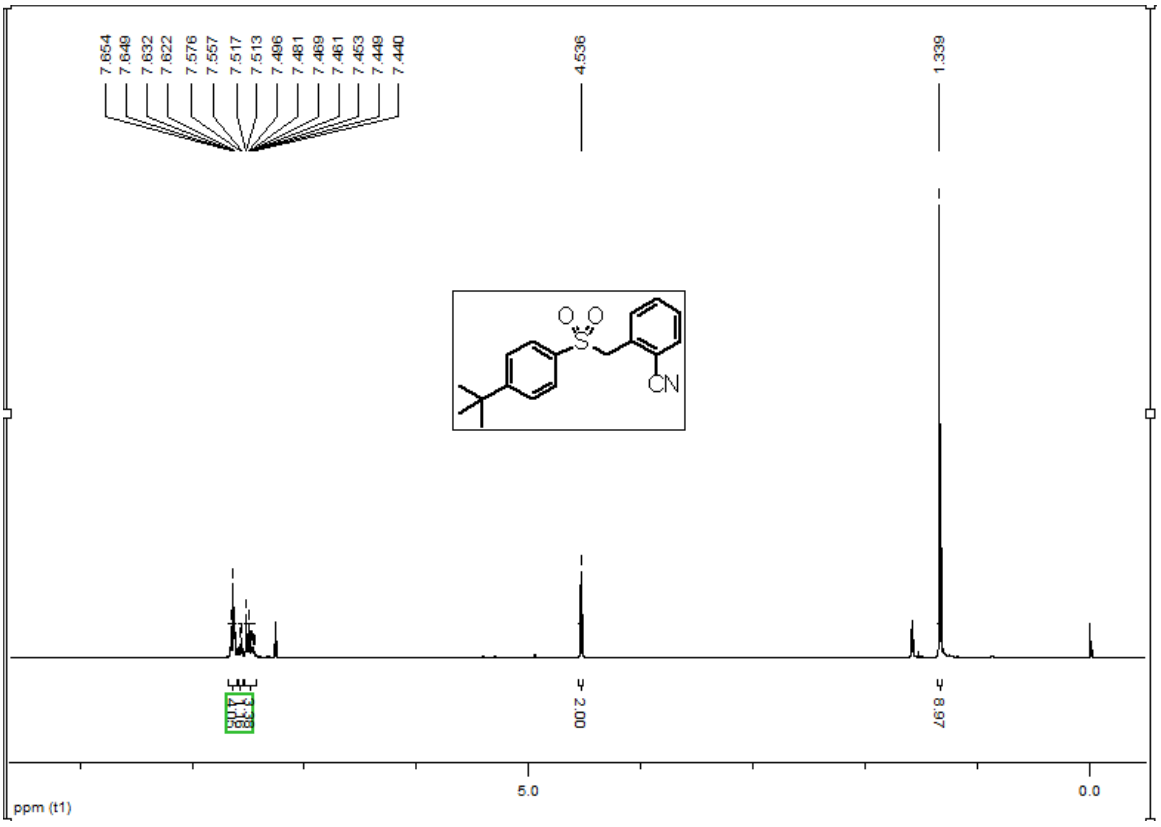
Analysis Info

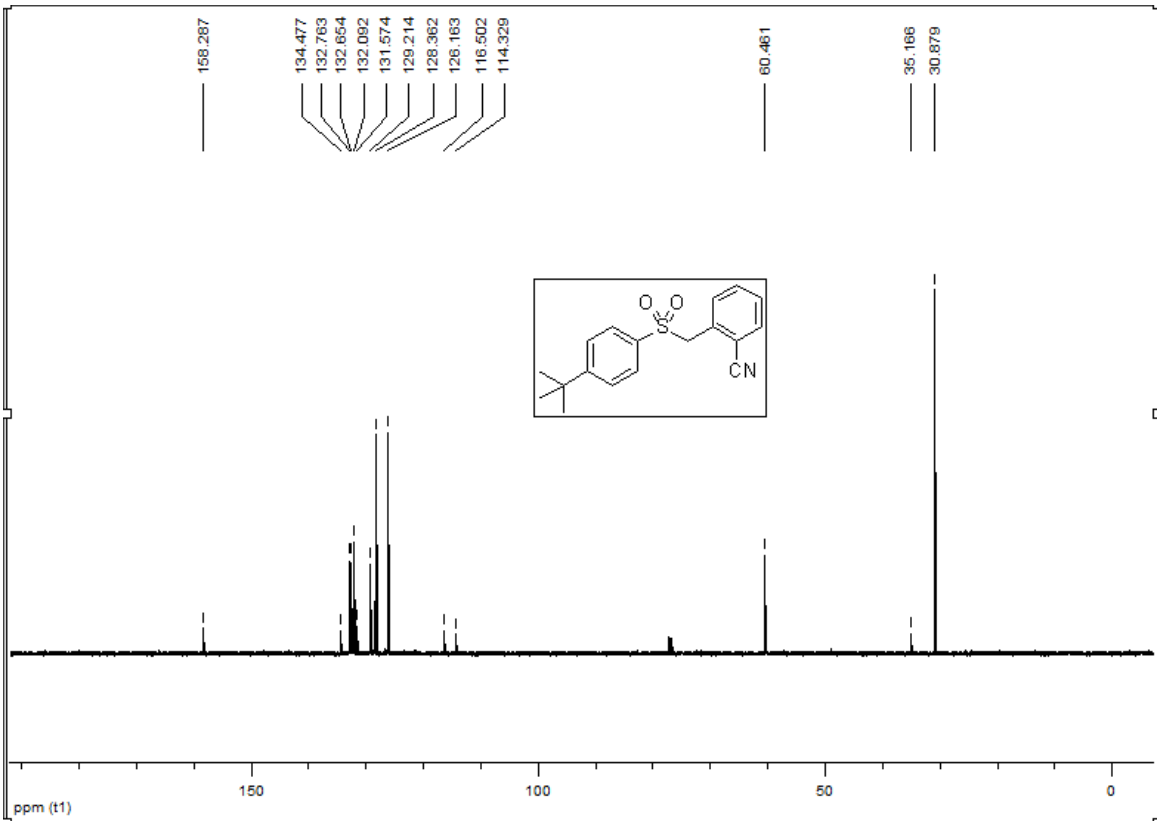
Analysis Name	D:\Data\2016MS\TT\0908\H-4_RD7_01_10997.d	Acquisition Date	9/8/2016 1:44:16 PM
Method	tune_200-800_hcoona-pos-2.5min.m	Operator	gftang
Sample Name	H-4	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.6 Bar
Focus	Not active			Set Dry Heater	180 ℃
Scan Begin	50 m/z	Set Capillary	2500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste




7.654
7.649
7.632
7.622
7.576
7.557
7.517
7.513
7.496
7.481
7.469
7.461
7.453
7.449
7.440
4.536
1.339
O
O
S
CN
4.00
2.00
8.97
5.0
0.0
ppm (t1)


158.287
134.477
132.763
132.654
132.092
131.574
129.214
128.362
126.163
116.502
114.329
60.461
35.166
30.879
O
O
S
CN
150
100
50
0
ppm (t1)

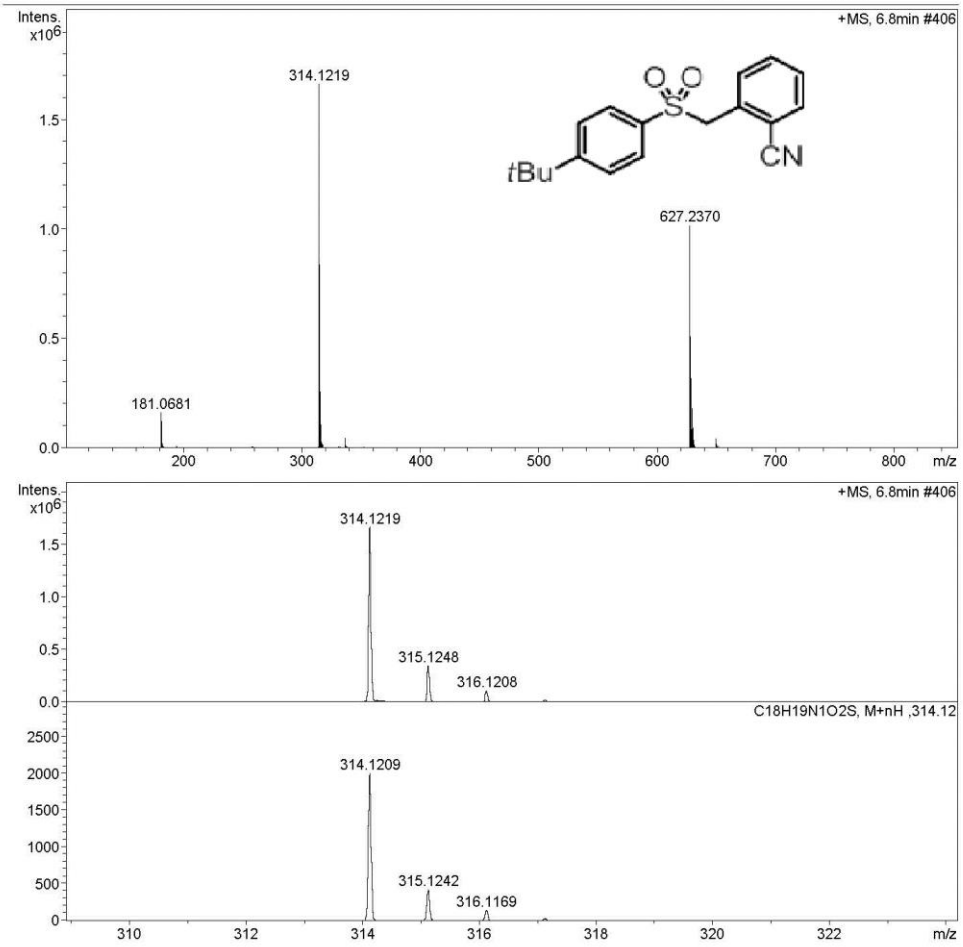
Display Report

Analysis Info

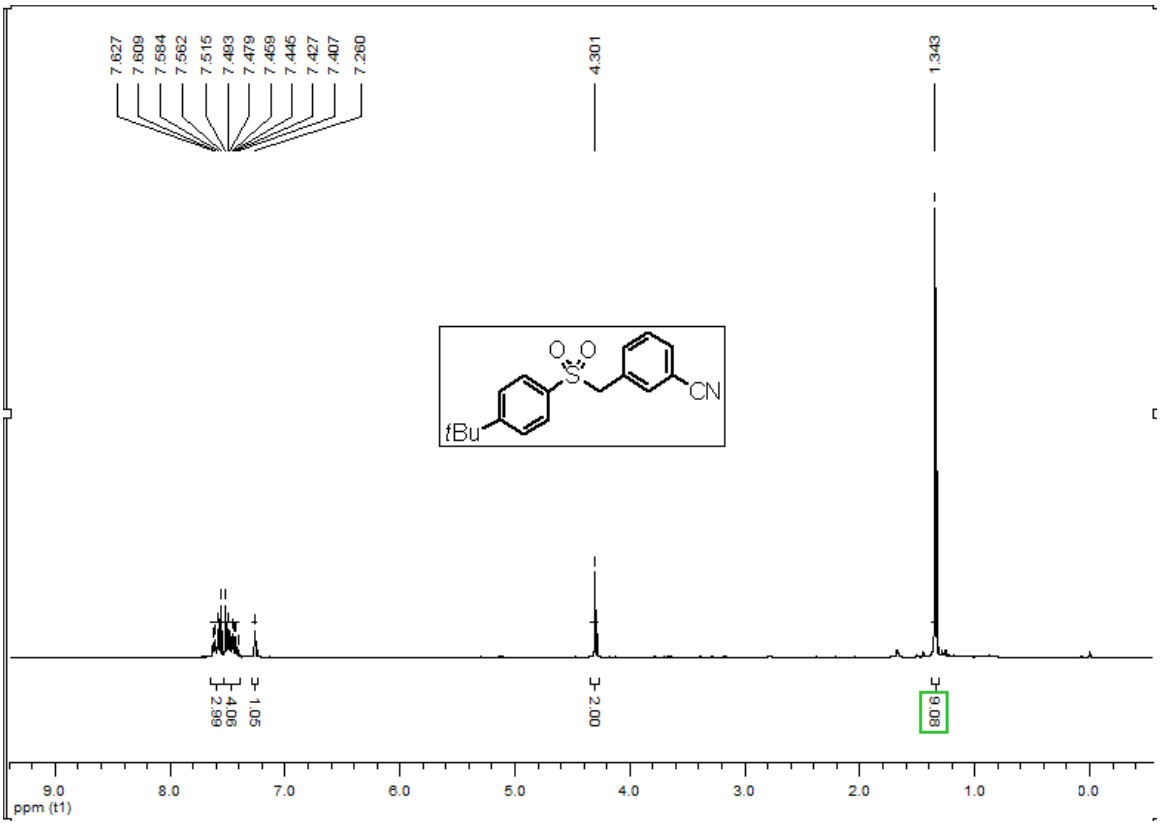
Analysis Name	D:\Data\2016MS\TT\0606\H15_RE2_01_9836.d	Acquisition Date	6/7/2016 1:30:48 AM
Method	tune_200-800_hcoona-pos-10min.m	Operator	gftang
Sample Name	H15	Instrument / Ser#	micrOTOF II 10257
Comment			

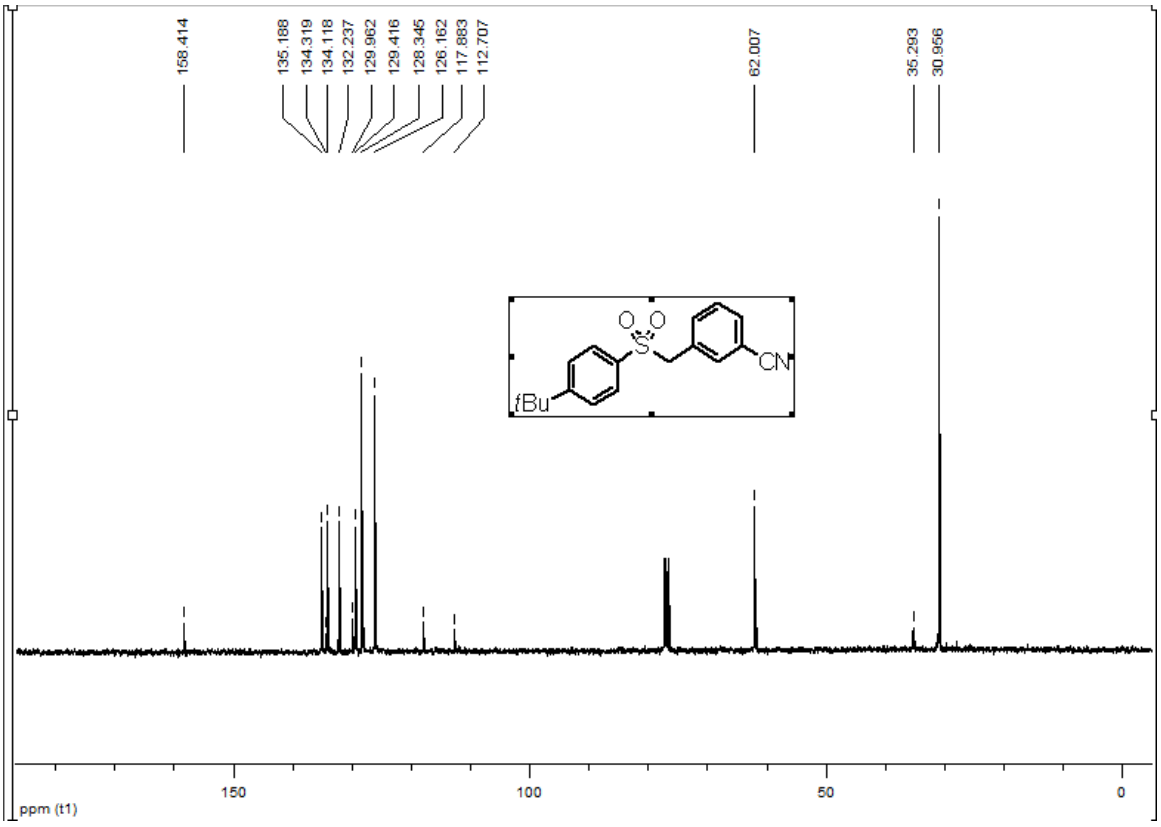
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	100 m/z	Set Capillary	4000 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Bruker Compass DataAnalysis 4.0 printed: 6/7/2016 2:16:08 PM Page 1 of 1


7.627
7.609
7.584
7.562
7.515
7.493
7.479
7.459
7.445
7.427
7.407
7.260
4.301
1.343
O
O
S
CN
tBu
2.99
4.06
1.05
2.00
9.08
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


168.414
135.188
134.319
134.118
132.237
129.962
129.416
128.345
126.162
117.883
112.707
62.007
36.293
30.956
O
O
S
CN
tBu
150
100
50
0
ppm (t1)

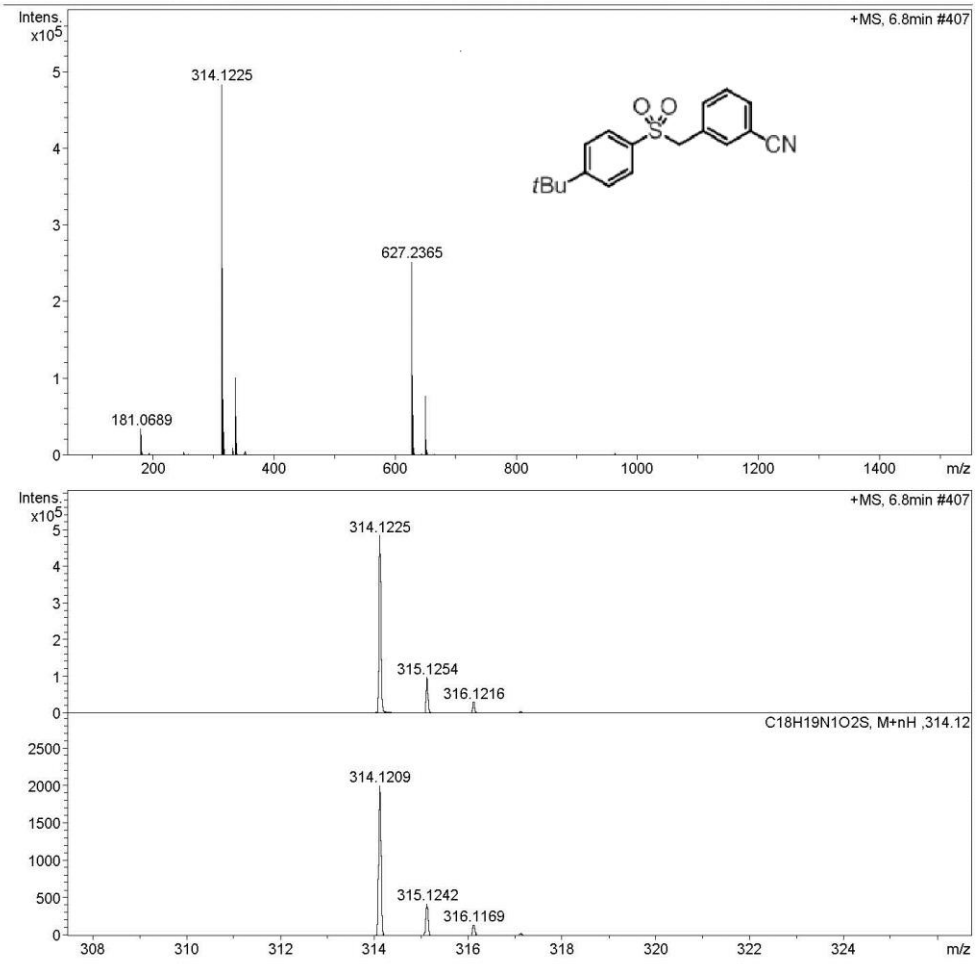
Display Report

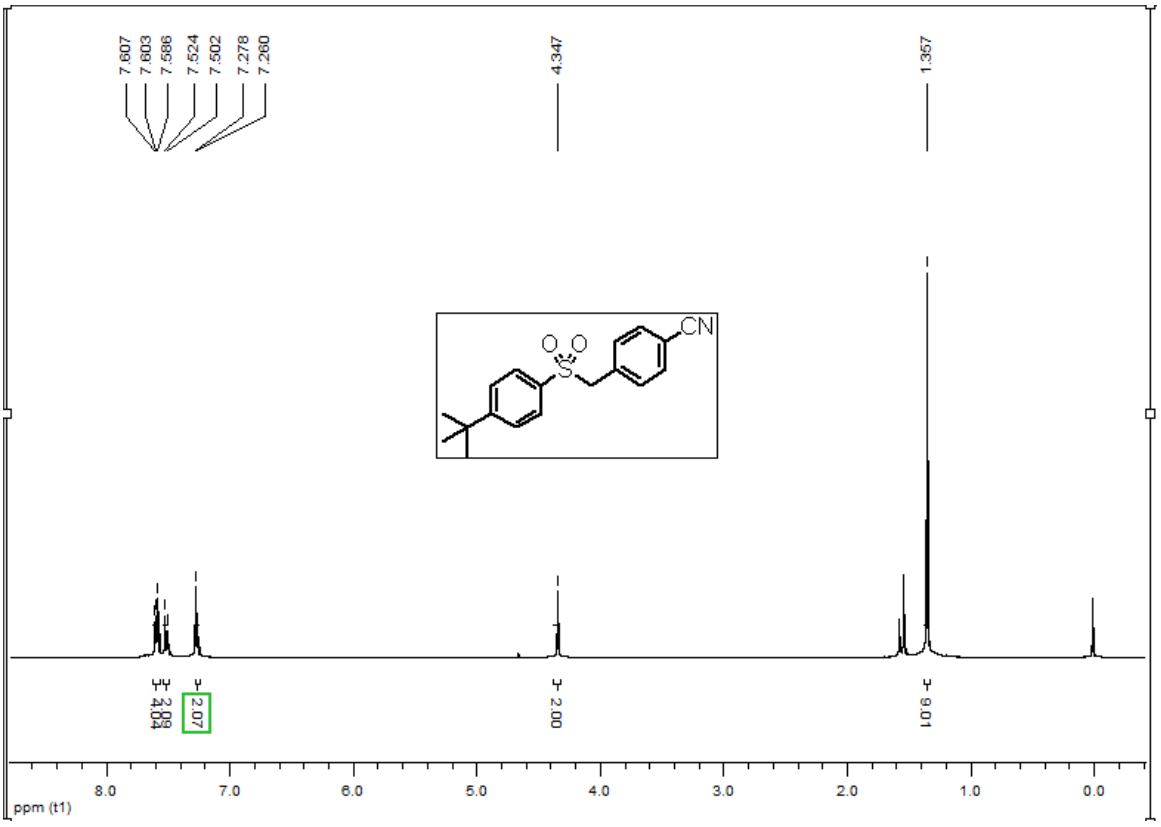
Analysis Info

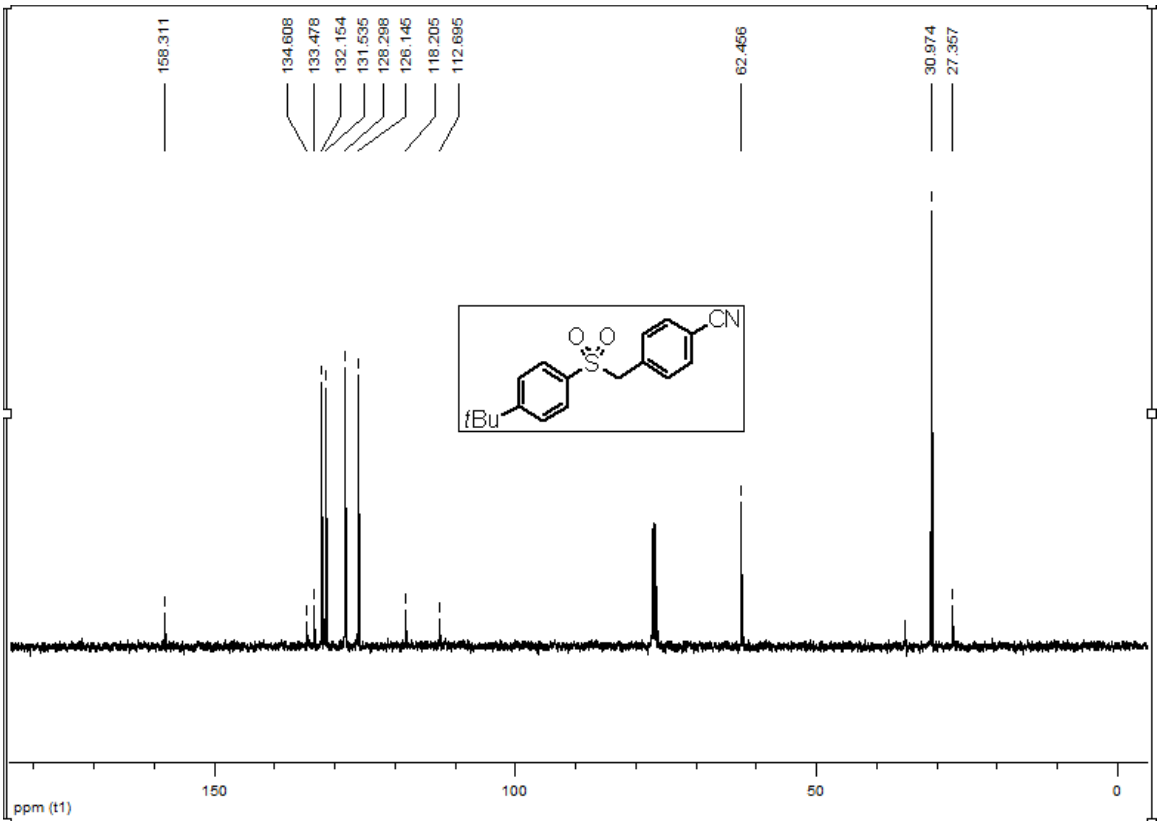
Analysis Name	D:\Data\2016MS\TT\0606\H16_RE3_01_9837.d	Acquisition Date	6/7/2016 1:46:39 AM
Method	tune_200-800_hcoona-pos-10min.m	Operator	gftang
Sample Name	H16	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	100 m/z	Set Capillary	4000 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste




7.607
7.603
7.586
7.524
7.502
7.278
7.260
4.347
1.357
CN
O
O
S
2.04
2.07
2.00
9.01
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


158.311
134.608
133.478
132.154
131.535
128.298
126.145
118.205
112.695
62.456
30.974
27.357
CN
O
O
S
tBu
150
100
50
0
ppm (t1)

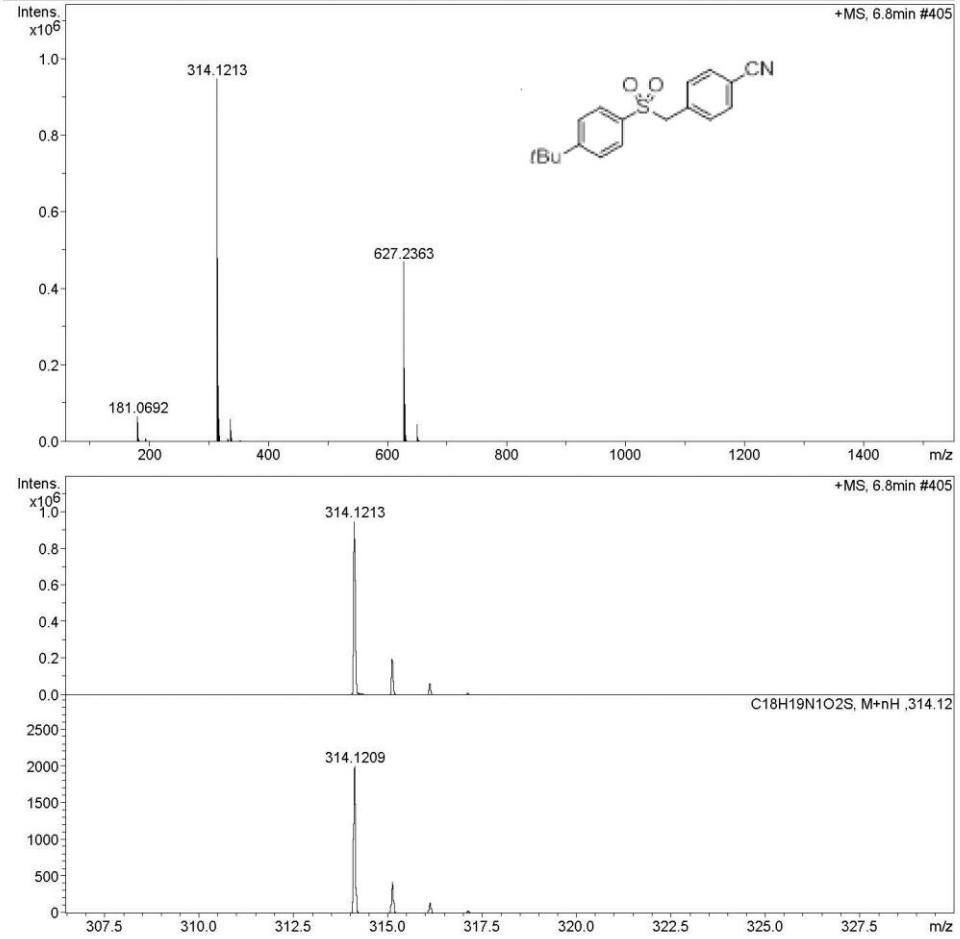
Display Report

Analysis Info

Analysis Name	D:\Data\2016MS\TT\0606\H18_RE5_01_9839.d	Acquisition Date	6/7/2016 2:18:25 AM
Method	tune_200-800_hcoona-pos-10min.m	Operator	gftang
Sample Name	H18	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	100 m/z	Set Capillary	4000 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



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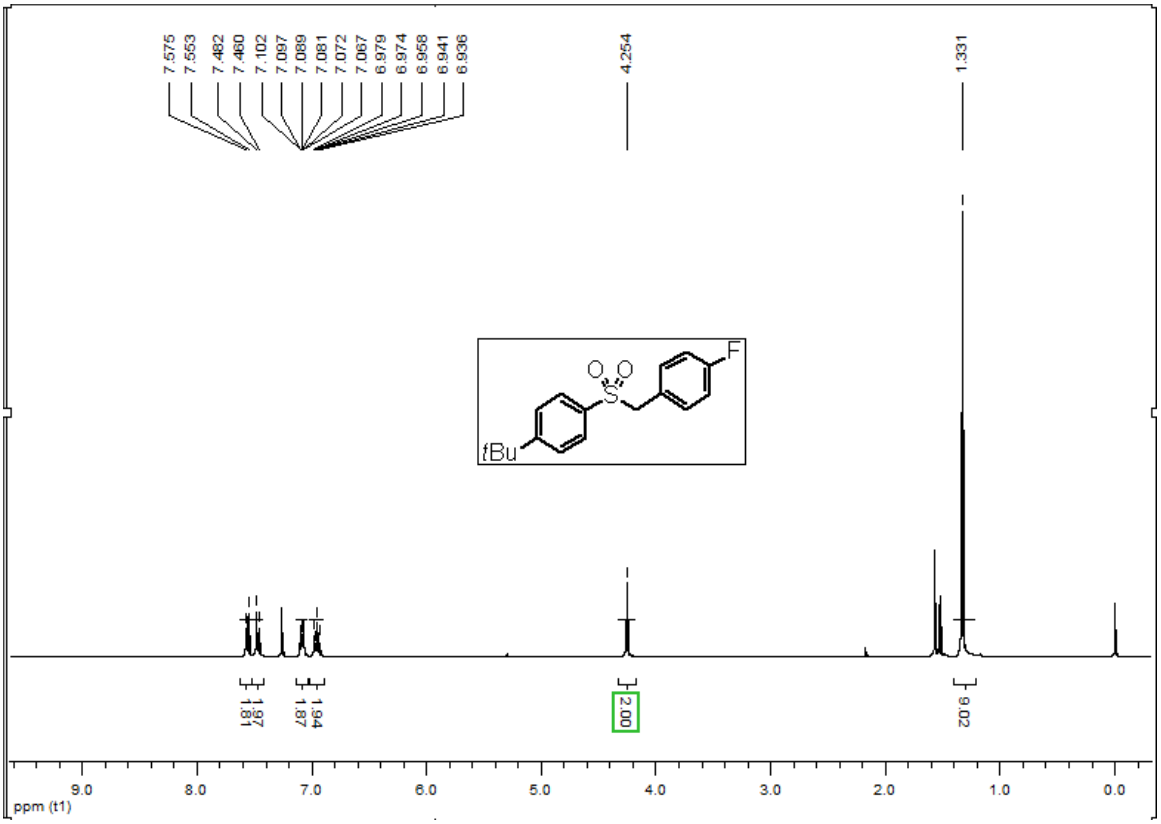
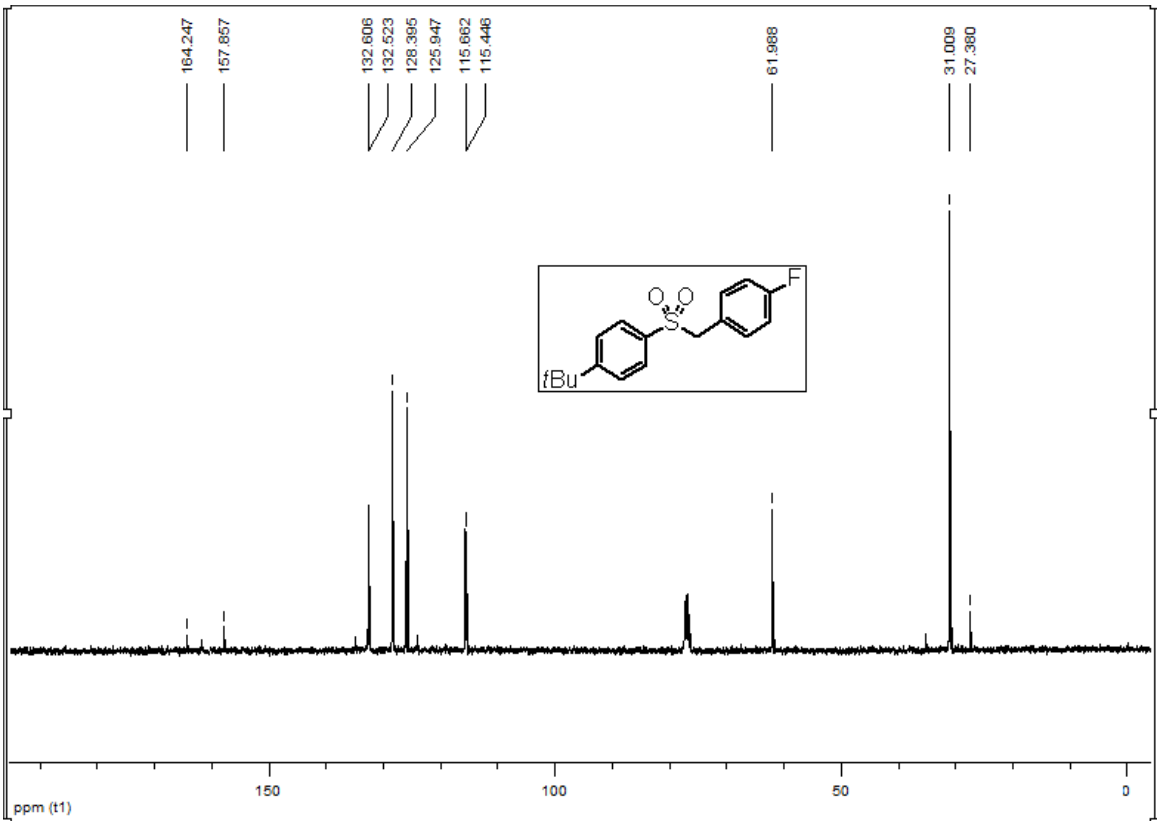
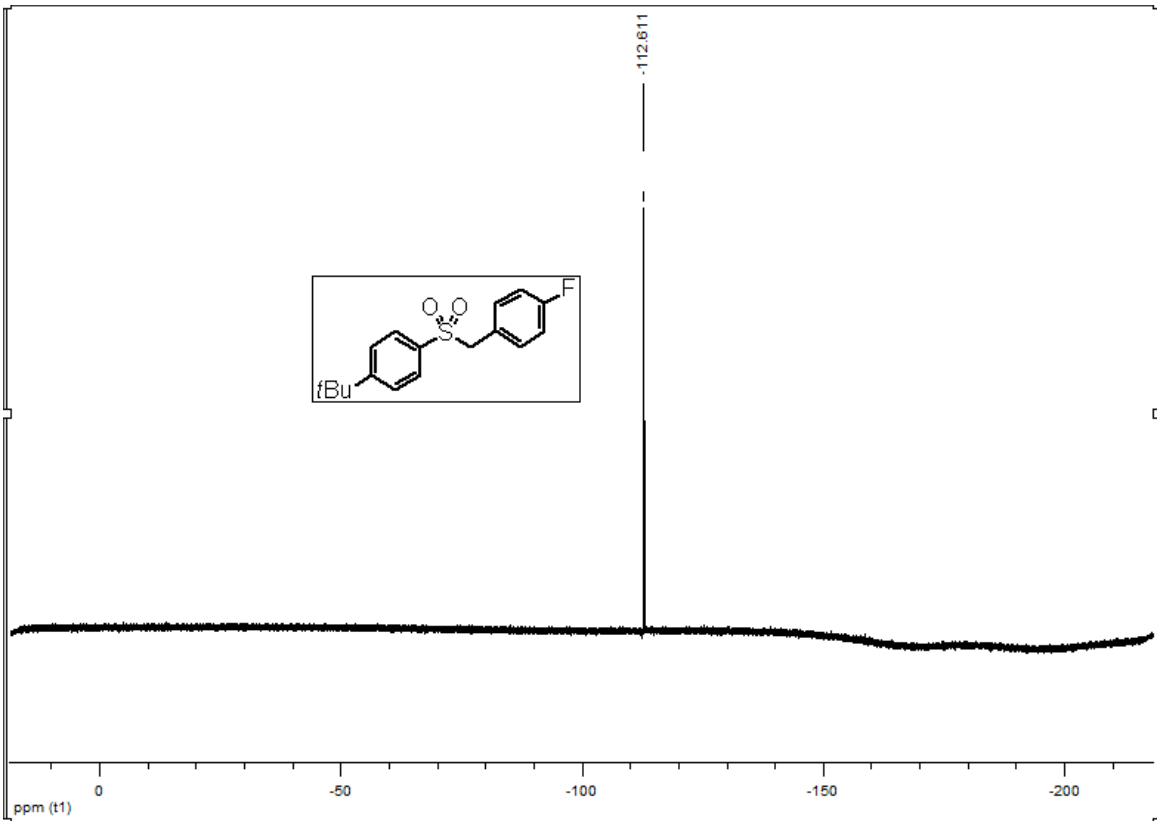

7.575
7.553
7.482
7.460
7.102
7.097
7.089
7.081
7.072
7.067
6.979
6.974
6.958
6.941
6.936
4.254
1.331
F
O
O
S
tBu
1.97
1.81
1.94
1.87
2.00
9.02
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


164.247
157.857
132.606
132.523
128.395
125.947
115.662
115.446
61.988
31.009
27.360
F
O
O
S
tBu
150
100
50
0
ppm (t1)


-112.611
F
O
O
S
tBu
0
-50
-100
-150
-200
ppm (t1)

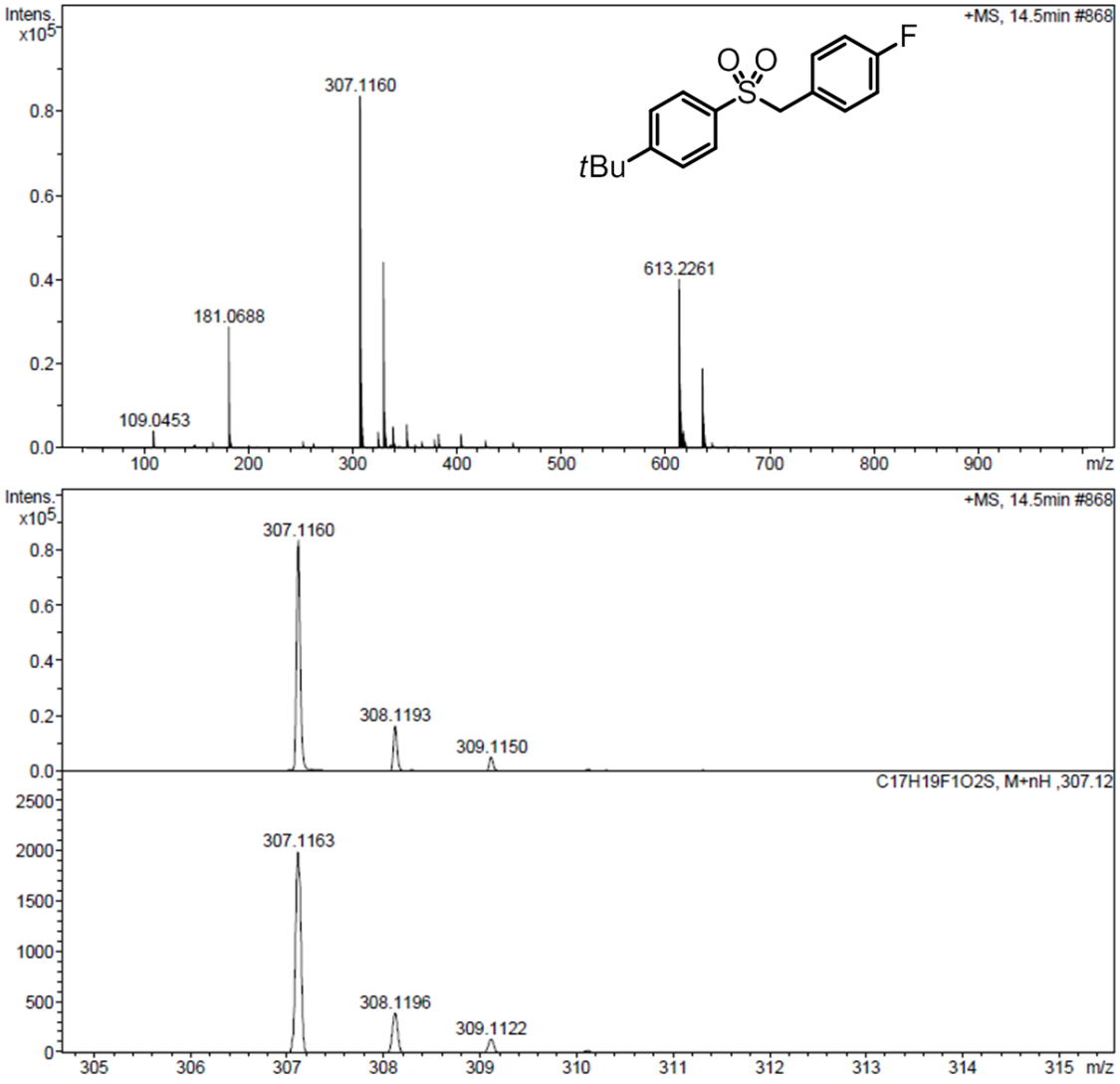
Display Report

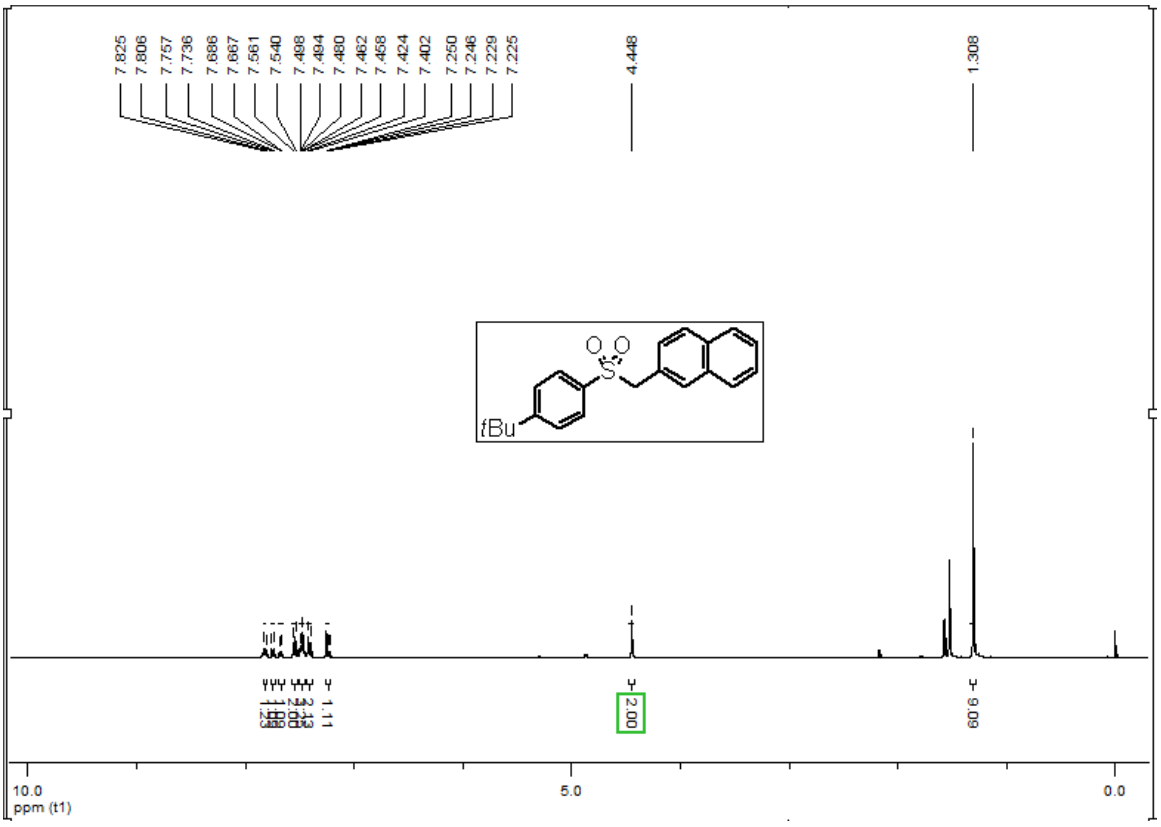
Analysis Info

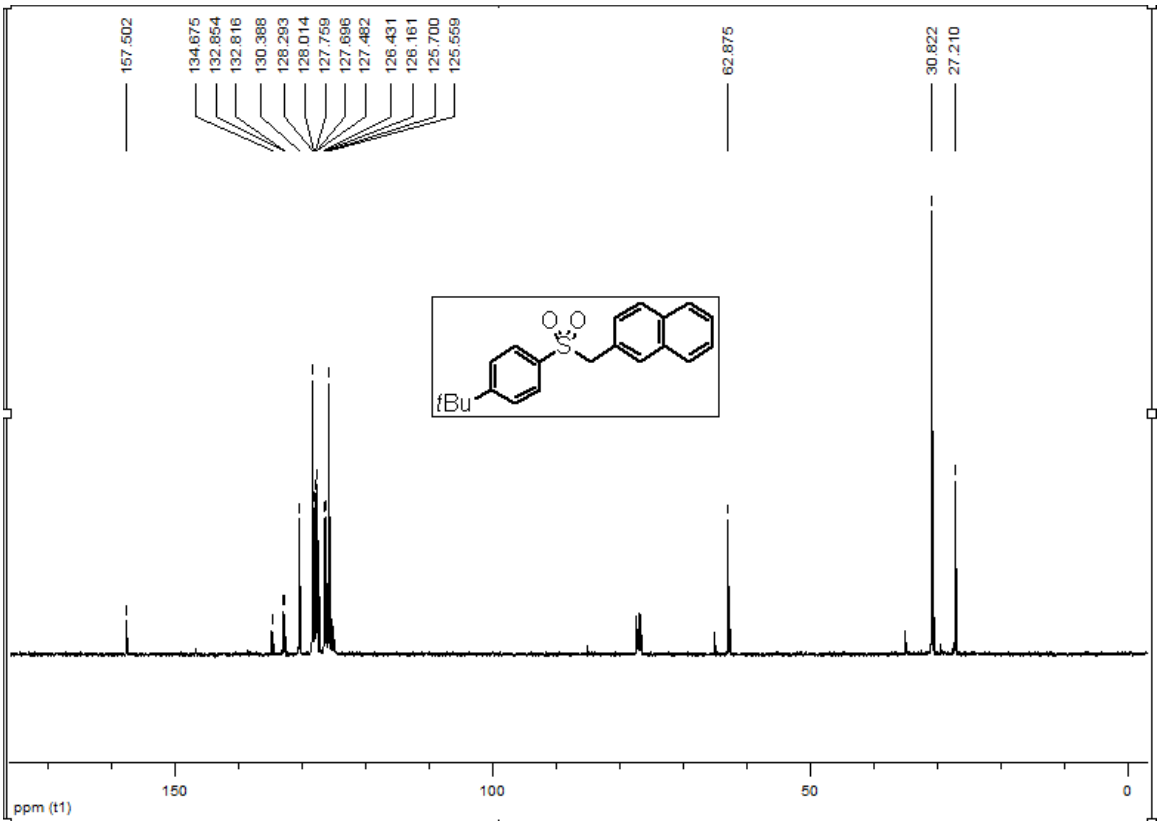
Analysis Name	D:\Data\2016MS\TT\0908\15_BD2_01_11054.d	Acquisition Date	9/13/2016 9:01:47 AM
Method	tune_200-800_hcoona-pos-2.5min.m	Operator	gftang
Sample Name	15	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	180 ℃
Scan Begin	50 m/z	Set Capillary	2500 V	Set Dry Gas	6.0 l/min
Scan End	1000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste




7.825
7.806
7.757
7.736
7.686
7.667
7.561
7.540
7.498
7.494
7.480
7.462
7.458
7.424
7.402
7.250
7.246
7.229
7.225
4.448
1.308
tBu
2.00
9.09
10.0
ppm (t1)
5.0
0.0


157.502
134.675
132.854
132.816
130.388
128.293
128.014
127.759
127.696
127.482
126.431
126.161
125.700
125.559
62.875
30.822
27.210
tBu
150
100
50
0
ppm (t1)

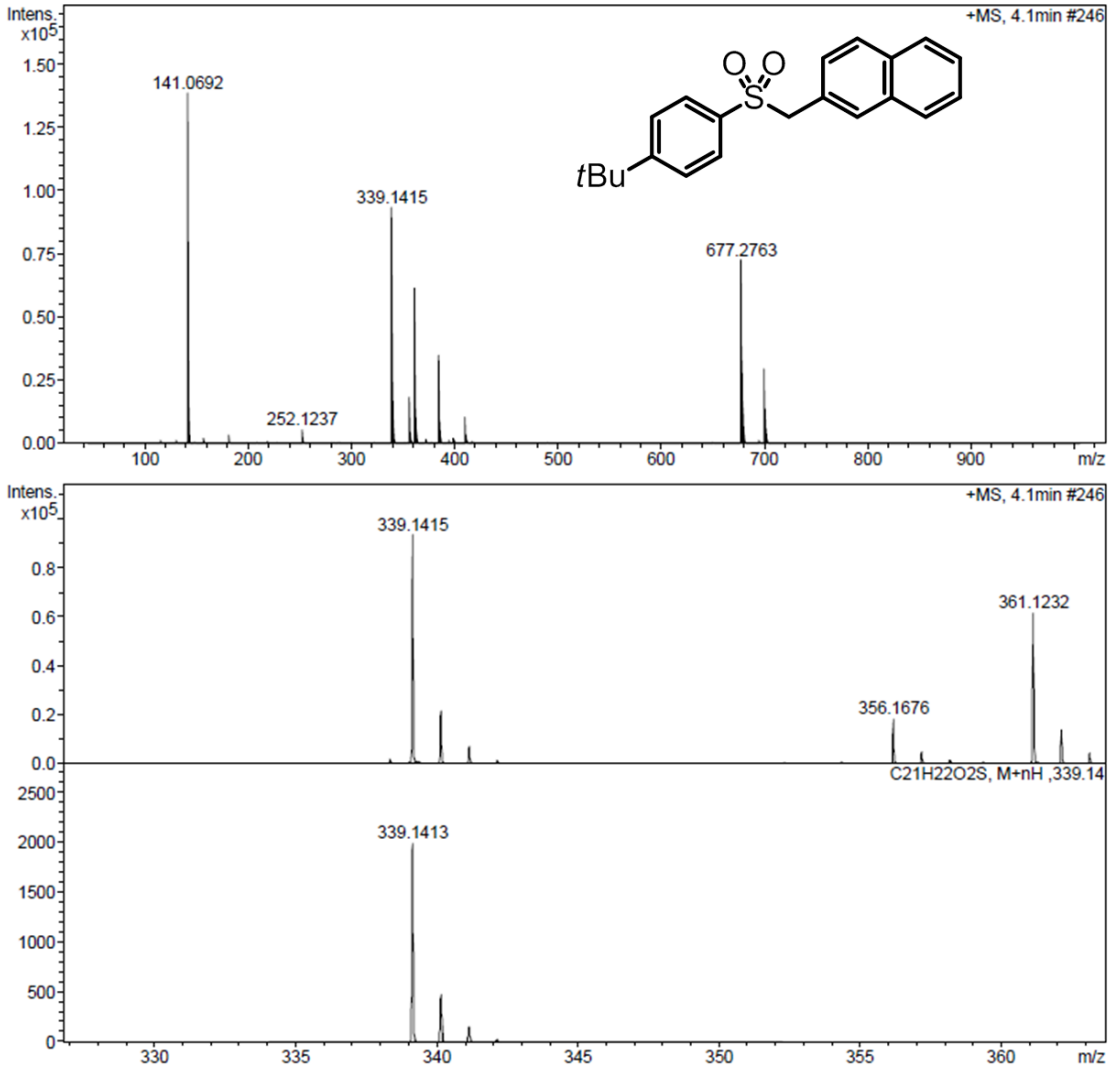
Display Report

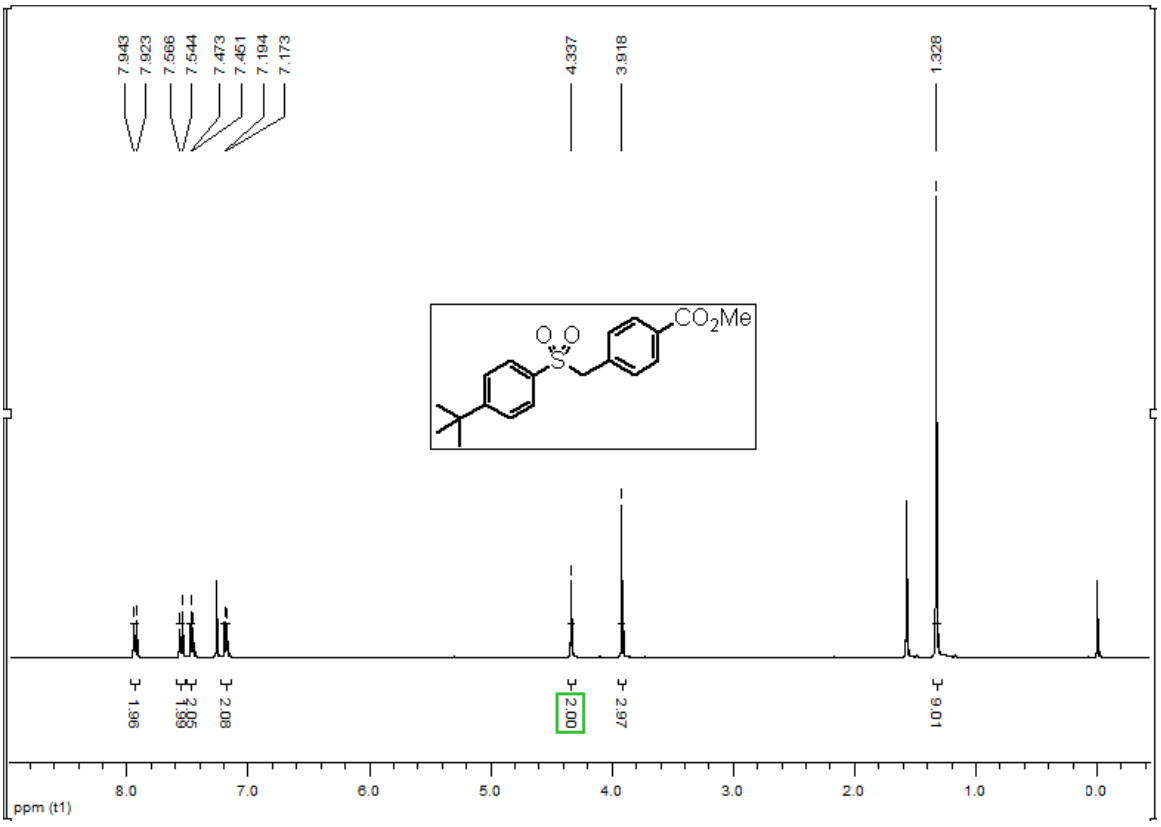
Analysis Info

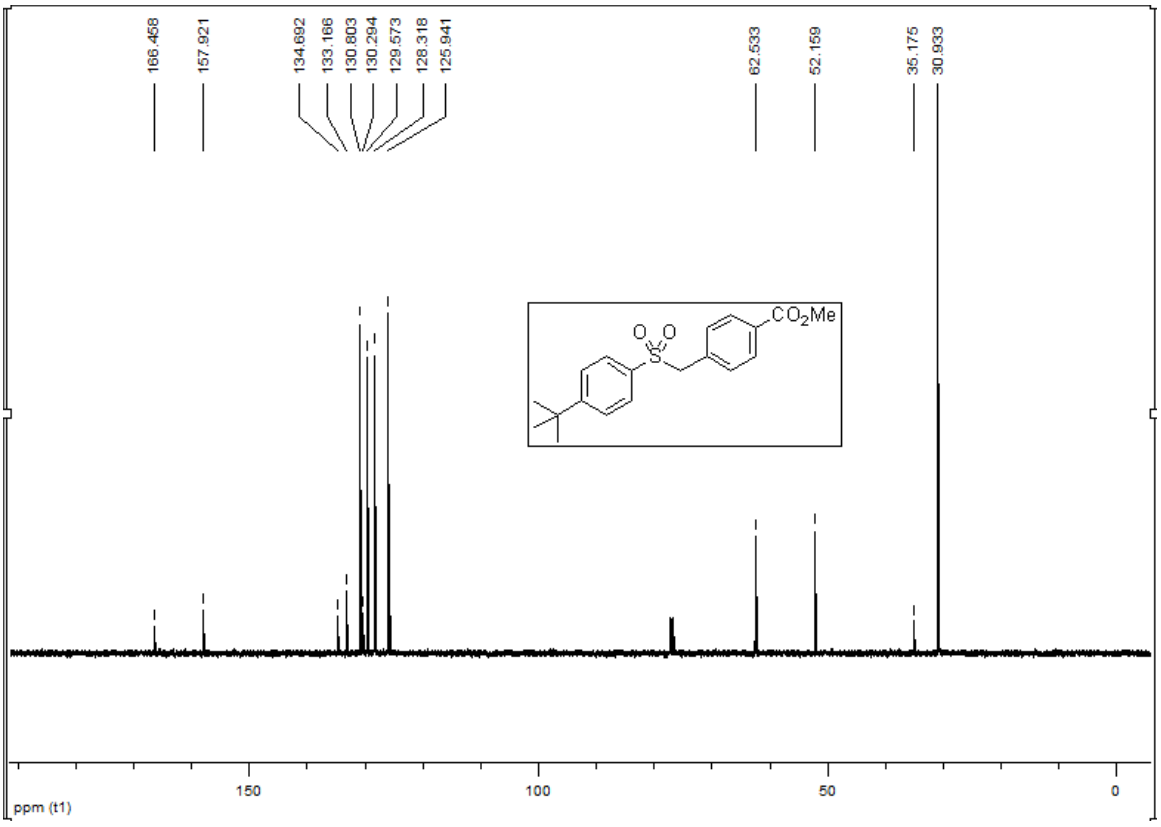
Analysis Name	D:\Data\2016MS\TT\0908\16_BD3_01_11080.d	Acquisition Date	9/13/2016 2:51:33 PM
Method	tune_200-800_hcoona-pos-6min.m	Operator	gftang
Sample Name	16	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	180 ℃
Scan Begin	50 m/z	Set Capillary	2500 V	Set Dry Gas	6.0 l/min
Scan End	1000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste




7.943
7.923
7.566
7.544
7.473
7.451
7.194
7.173
4.337
3.918
1.328
CO2Me
O
O
S
1.96
2.05
1.99
2.08
2.00
2.97
9.01
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


166.458
157.921
134.692
133.166
130.803
130.294
129.573
128.318
125.941
62.533
52.159
35.175
30.933
CO2Me
O
O
S
150
100
50
0
ppm (t1)

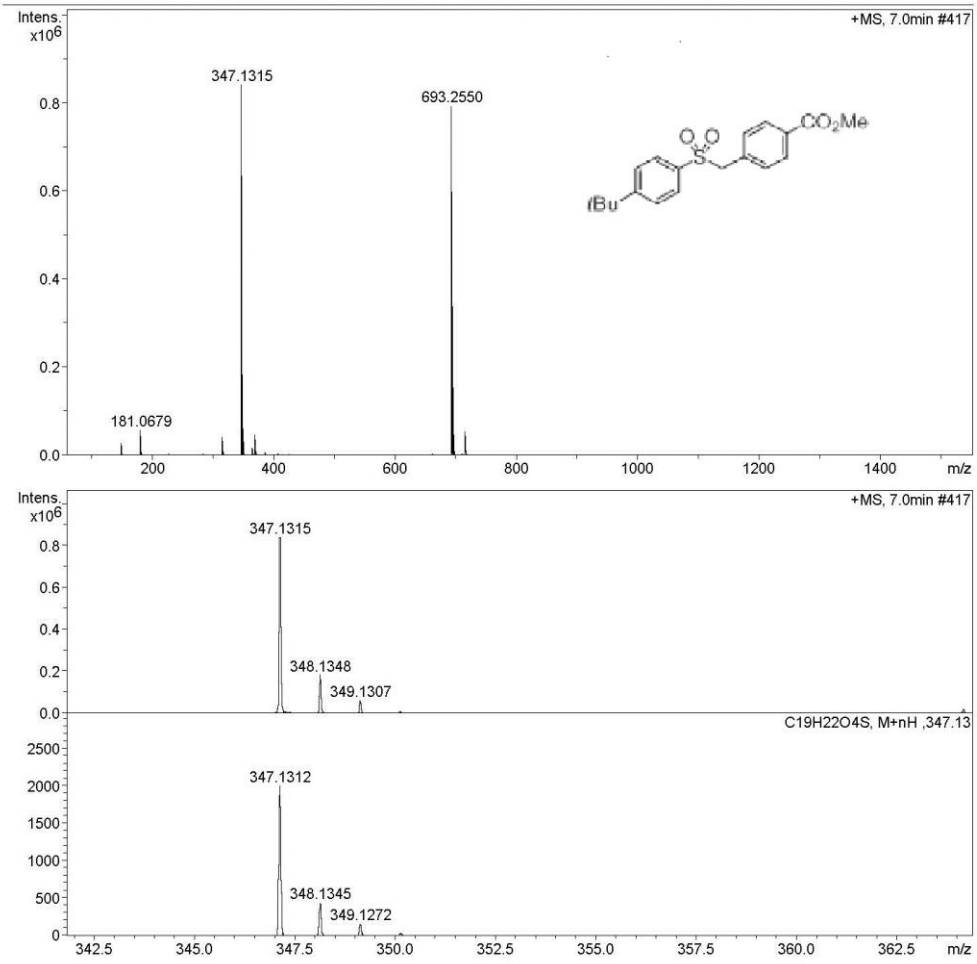
Display Report

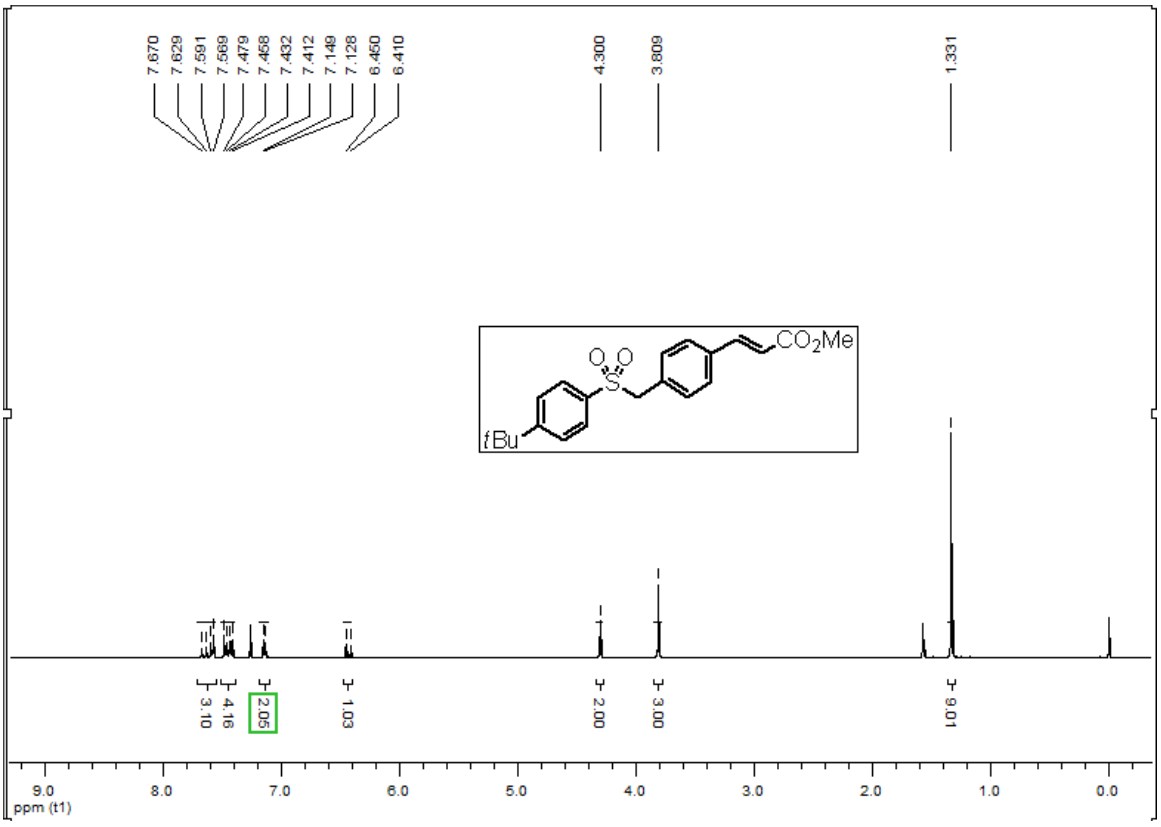
Analysis Info

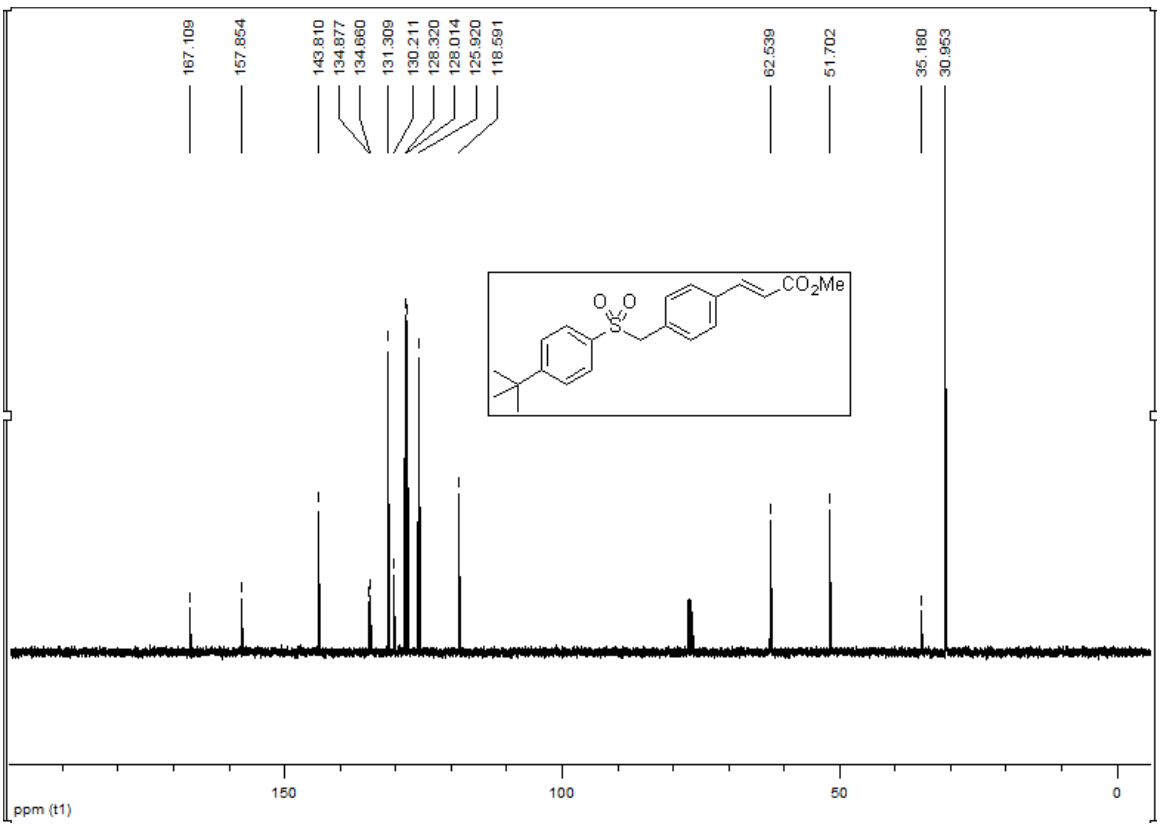
Analysis Name	D:\Data\2016MS\TT\0606\H17_RE4_01_9838.d	Acquisition Date	6/7/2016 2:02:32 AM
Method	tune_200-800_hcoona-pos-10min.m	Operator	gftang
Sample Name	H17	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	100 m/z	Set Capillary	4000 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste




7.670
7.629
7.591
7.569
7.479
7.458
7.432
7.412
7.149
7.128
6.450
6.410
4.300
3.809
1.331
CO2Me
tBu
3.10
4.16
2.05
1.03
2.00
3.00
9.01
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


167.109
157.854
143.810
134.877
134.660
131.309
130.211
128.320
128.014
125.920
118.591
62.539
51.702
35.180
30.953
CO2Me
150
100
50
0
ppm (t1)

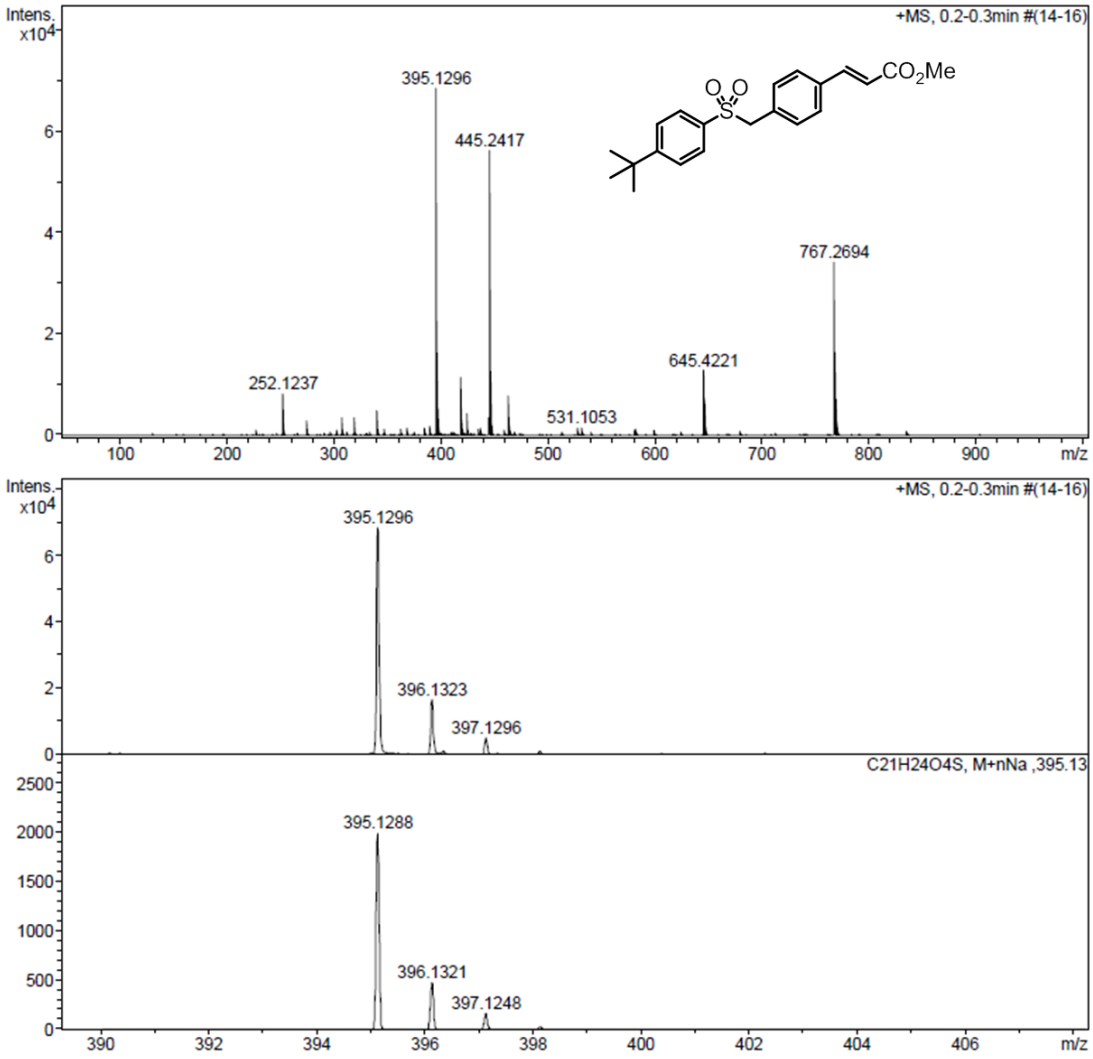
Display Report

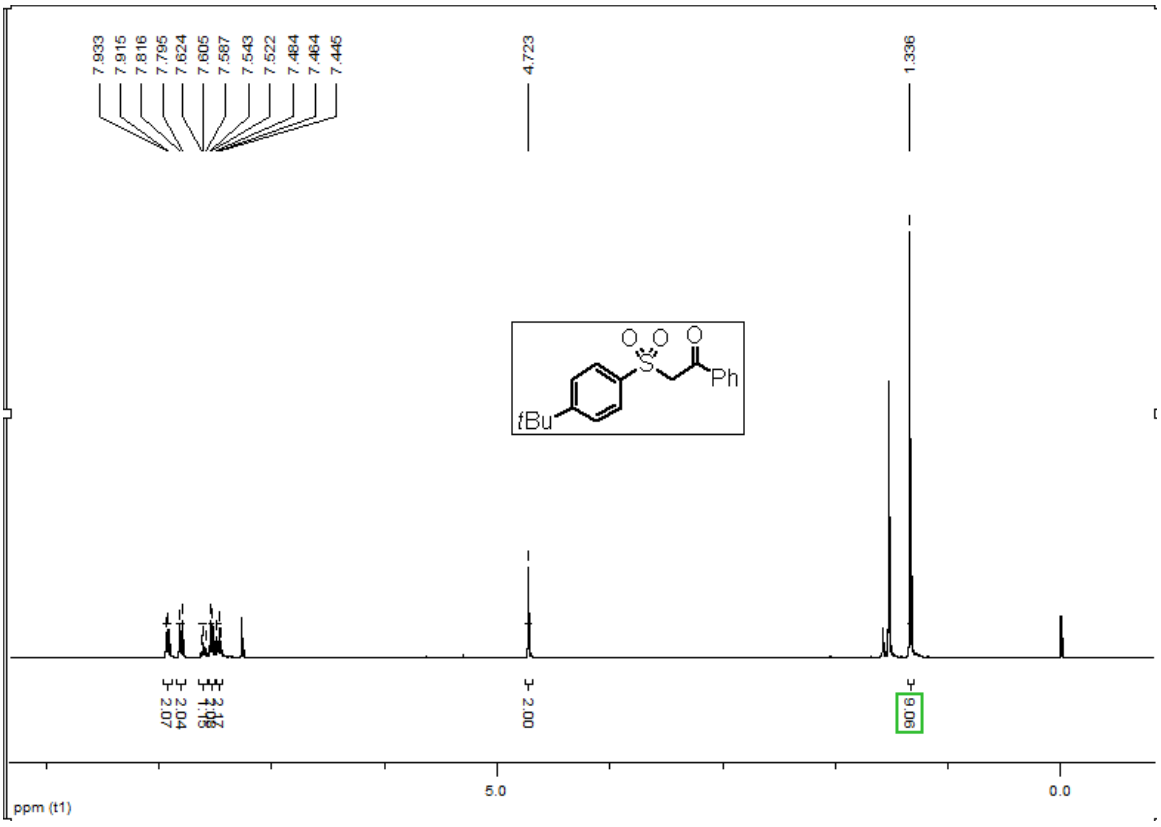
Analysis Info

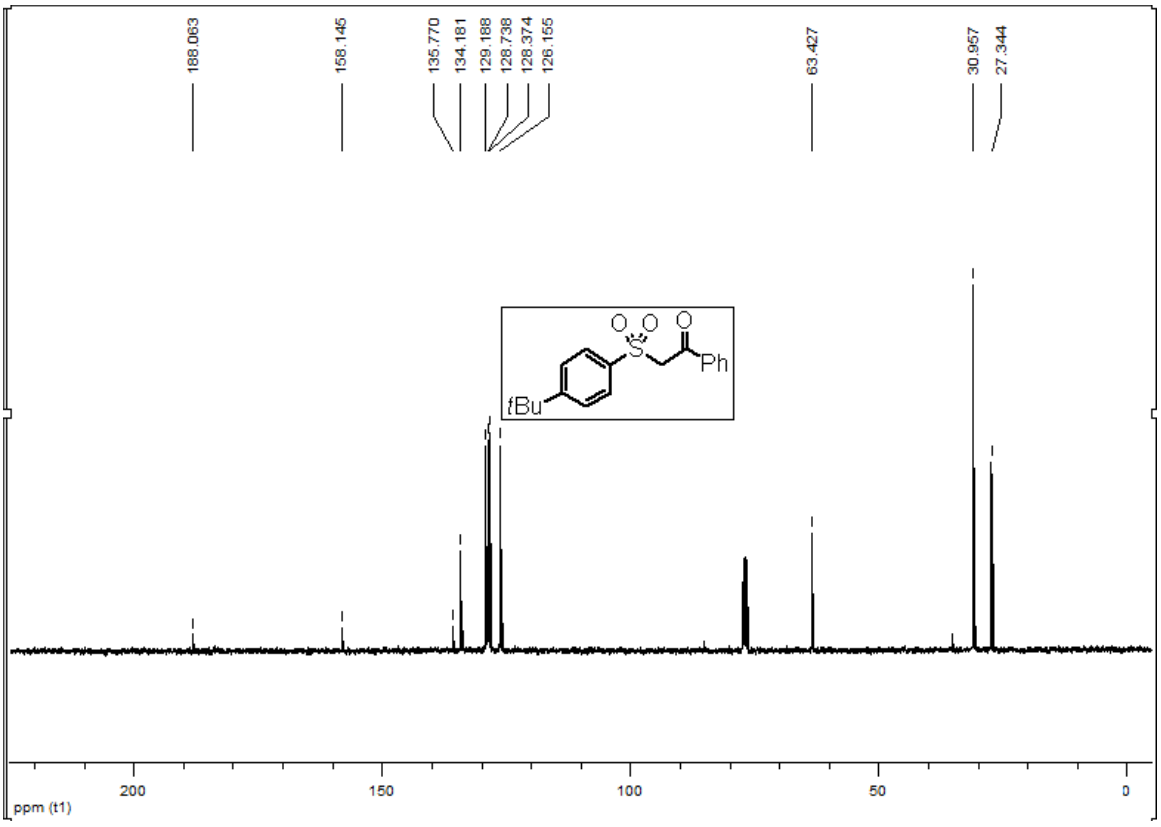
Analysis Name	D:\Data\2016MS\TT\0908\H-2_RD5_01_10995.d	Acquisition Date	9/8/2016 1:36:11 PM
Method	tune_200-800_hcoona-pos-2.5min.m	Operator	gftang
Sample Name	H-2	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.6 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	2500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste




7.933
7.915
7.816
7.795
7.624
7.605
7.587
7.543
7.522
7.484
7.464
7.445
4.723
1.336
tBu
Ph
2.07
2.04
2.14
2.00
9.06
5.0
0.0
ppm (t1)


188.063
158.145
135.770
134.181
129.188
128.738
128.374
126.155
63.427
30.957
27.344
tBu
Ph
200
150
100
50
0
ppm (t1)

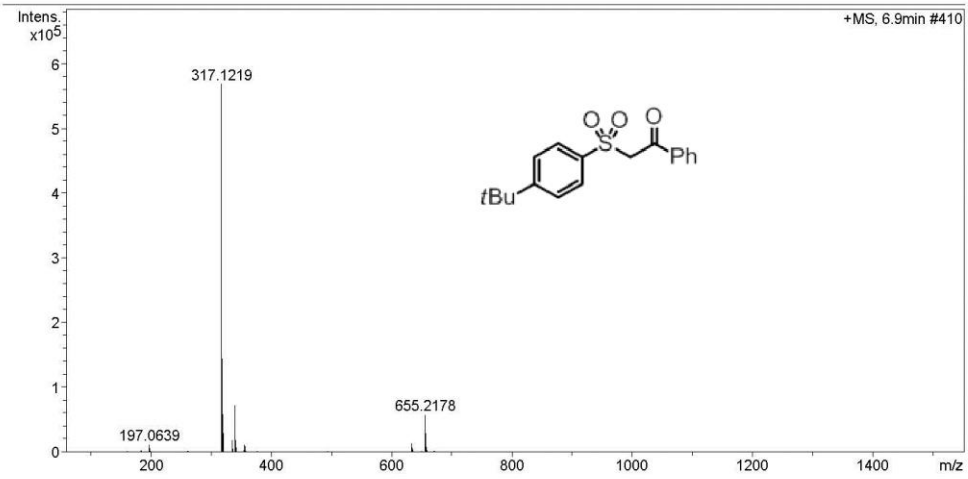
Display Report

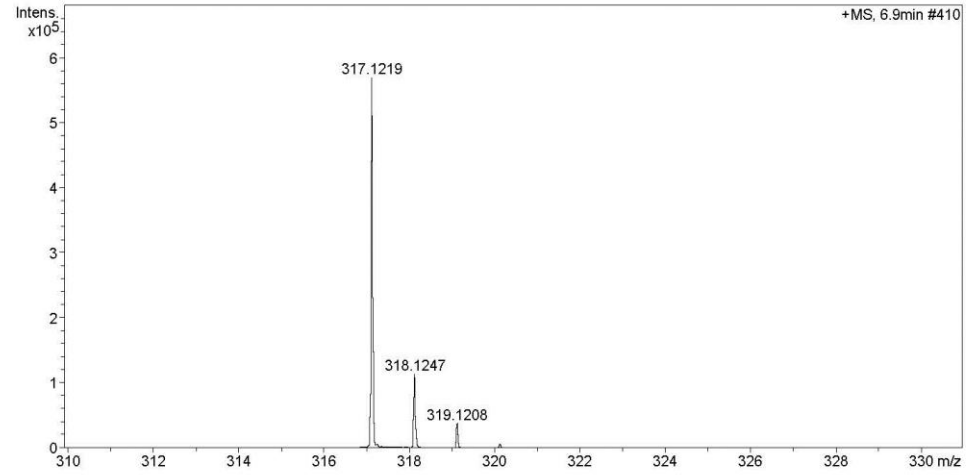
Analysis Info

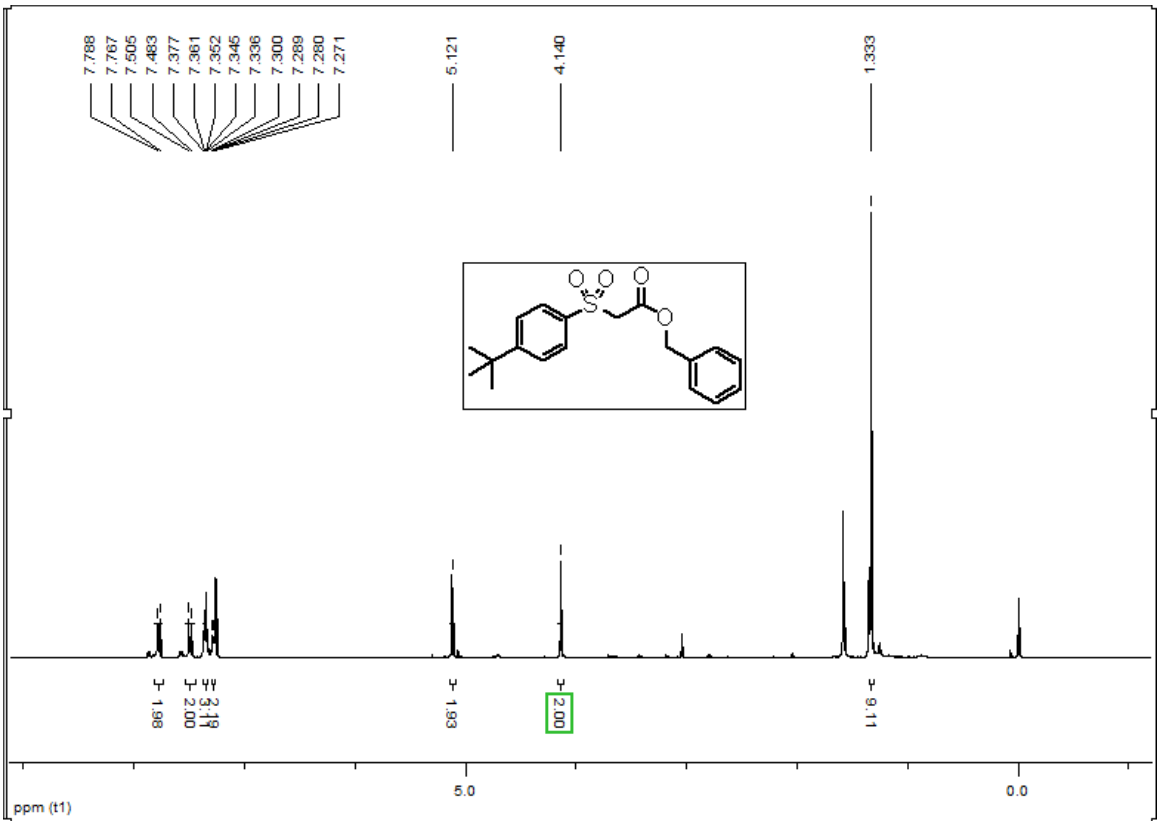
Analysis Name	D:\Data\2016MS\TT\0606\H19_RE6_01_9840.d	Acquisition Date	6/7/2016 2:34:17 AM
Method	tune_200-800_hcoona-pos-10min.m	Operator	gftang
Sample Name	H19	Instrument / Ser#	micrOTOF II 10257
Comment			

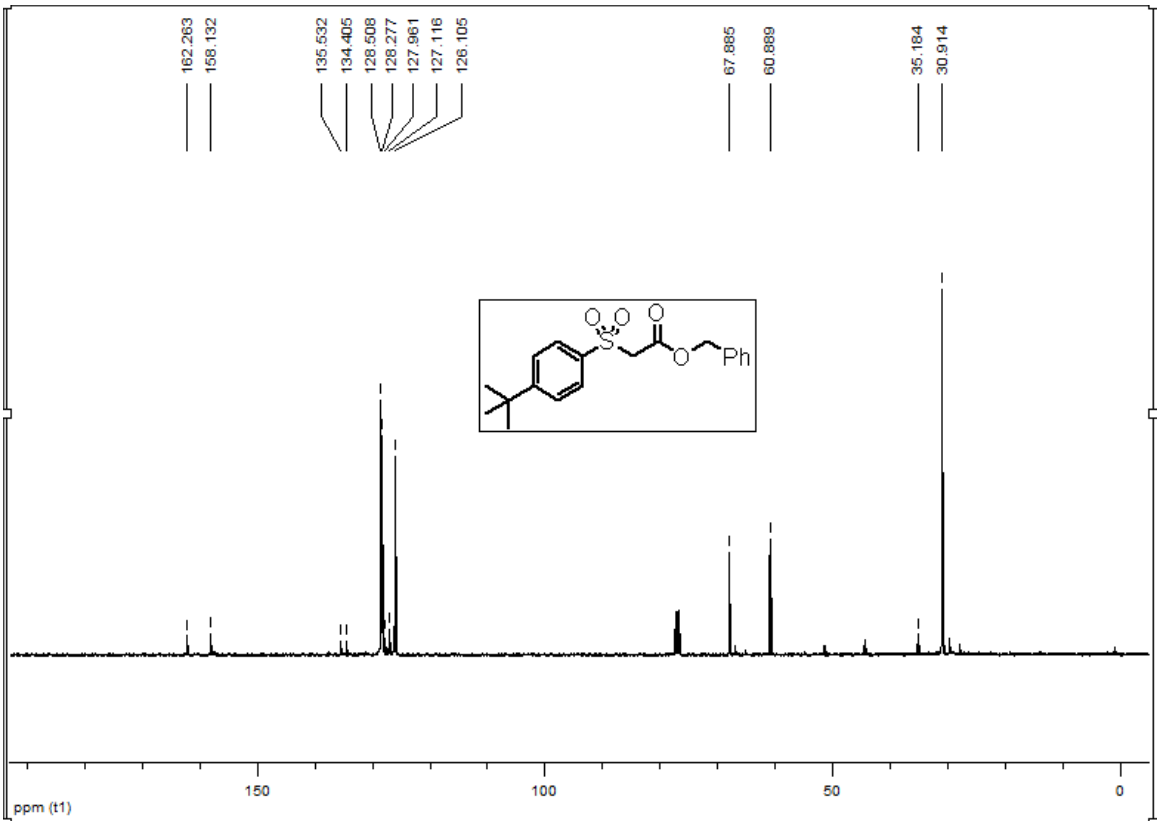
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	100 m/z	Set Capillary	4000 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste






7.788
7.767
7.505
7.483
7.377
7.361
7.352
7.345
7.336
7.300
7.289
7.280
7.271
5.121
4.140
1.333
1.98
2.00
3.14
1.93
2.00
9.11
5.0
0.0
ppm (t1)


162.263
158.132
135.532
134.405
128.508
128.277
127.961
127.116
126.105
67.885
60.889
35.184
30.914
Ph
150
100
50
0
ppm (t1)

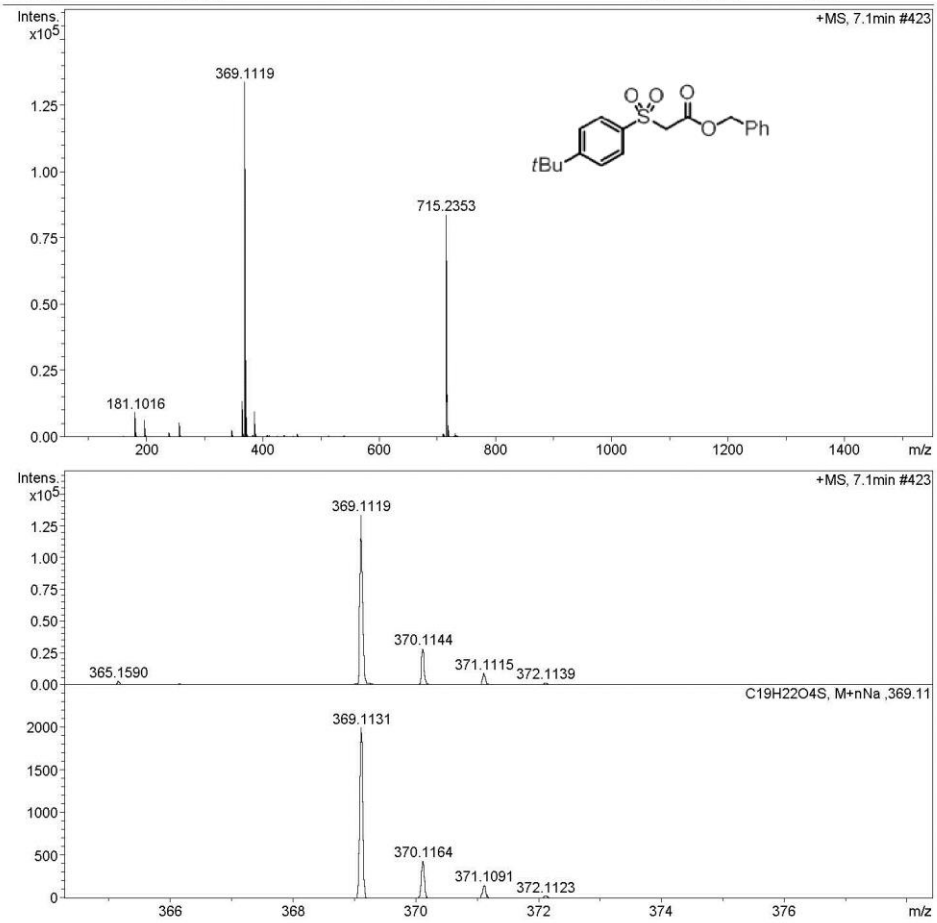
Display Report

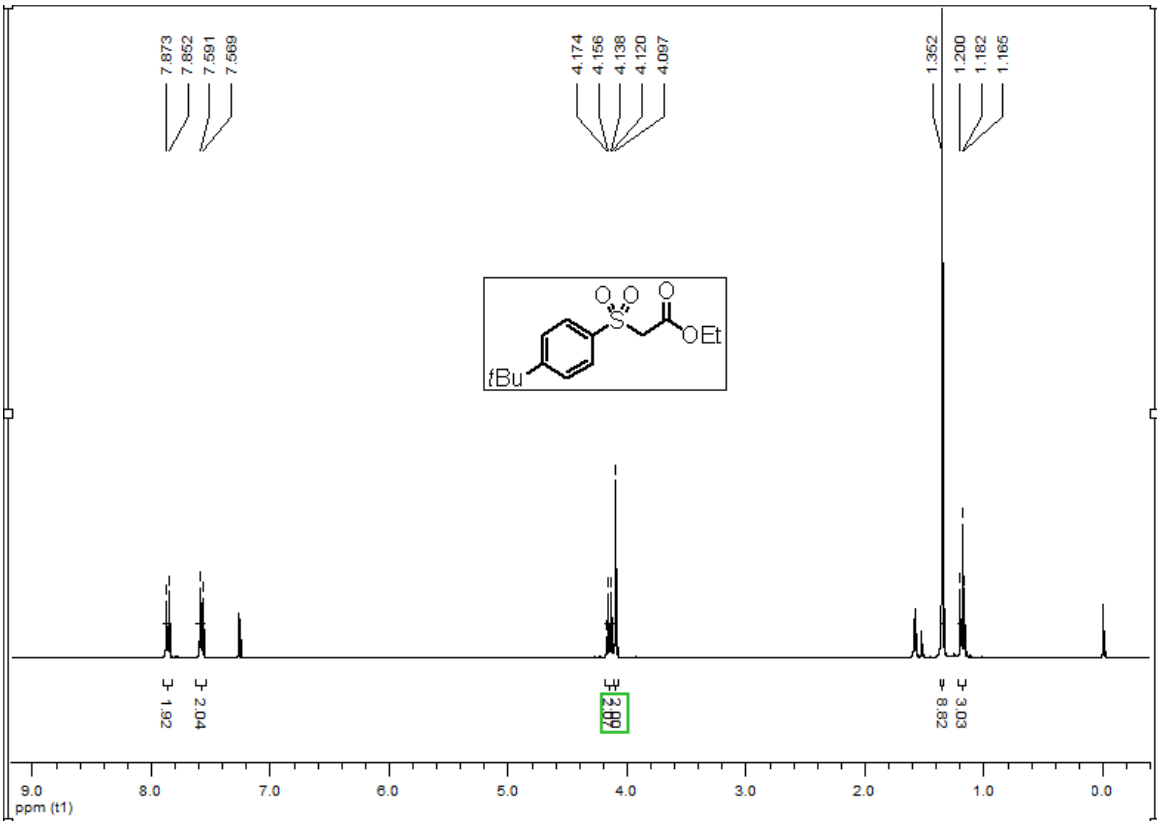
Analysis Info

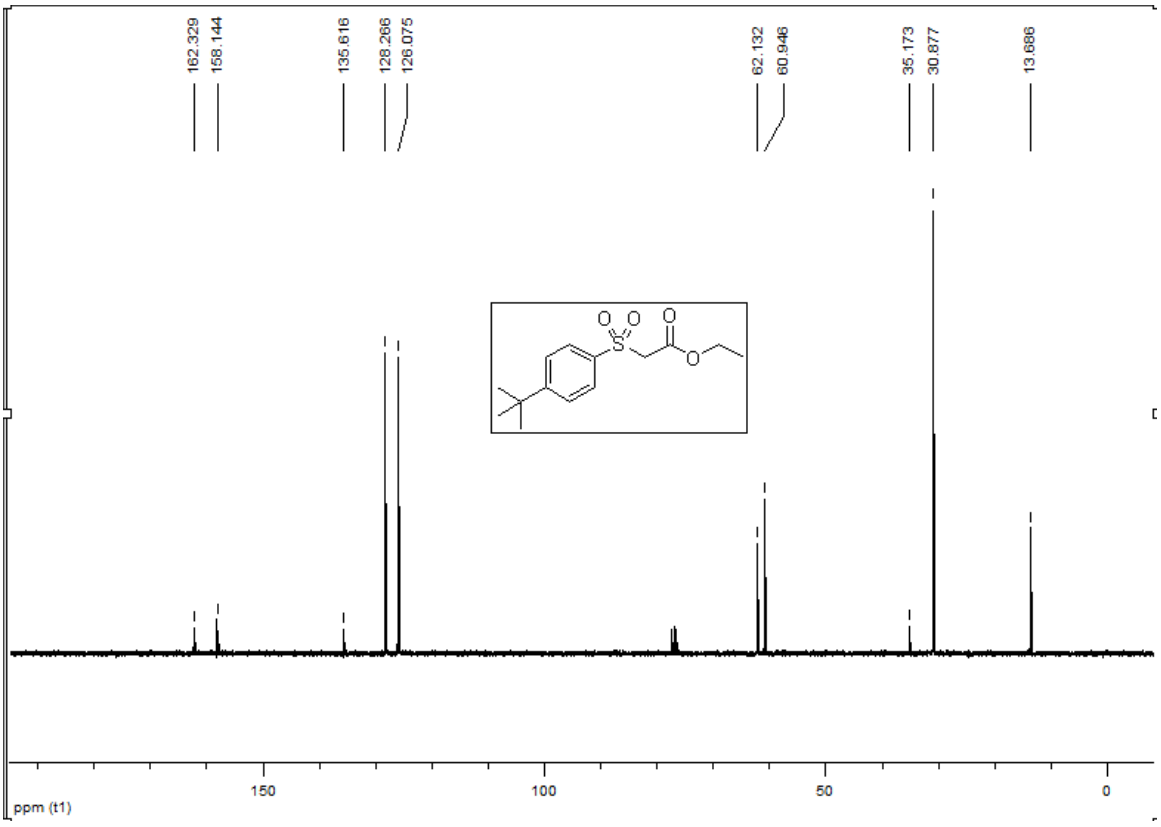
Analysis Name	D:\Data\2016MS\TT\0606\H20_RE7_01_9841.d	Acquisition Date	6/7/2016 2:50:10 AM
Method	tune_200-800_hcoona-pos-10min.m	Operator	gftang
Sample Name	H20	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	100 m/z	Set Capillary	4000 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste




7.873
7.852
7.591
7.569
4.174
4.156
4.138
4.120
4.097
1.352
1.200
1.182
1.165
tBu
OEt
1.92
2.04
2.00
8.62
3.03
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


162.329
158.144
135.616
128.266
126.075
62.132
60.946
35.173
30.877
13.686
150
100
50
0
ppm (t1)

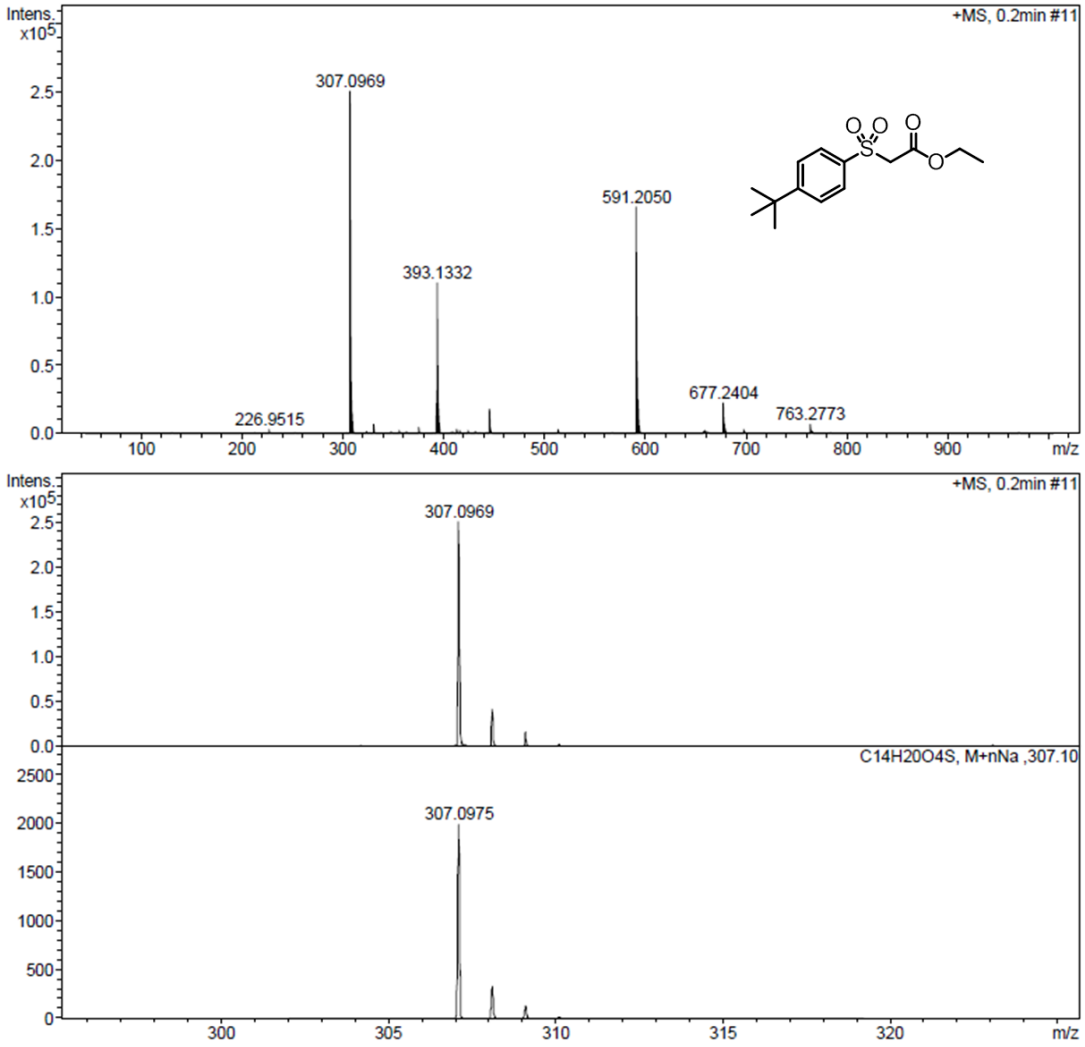
Display Report

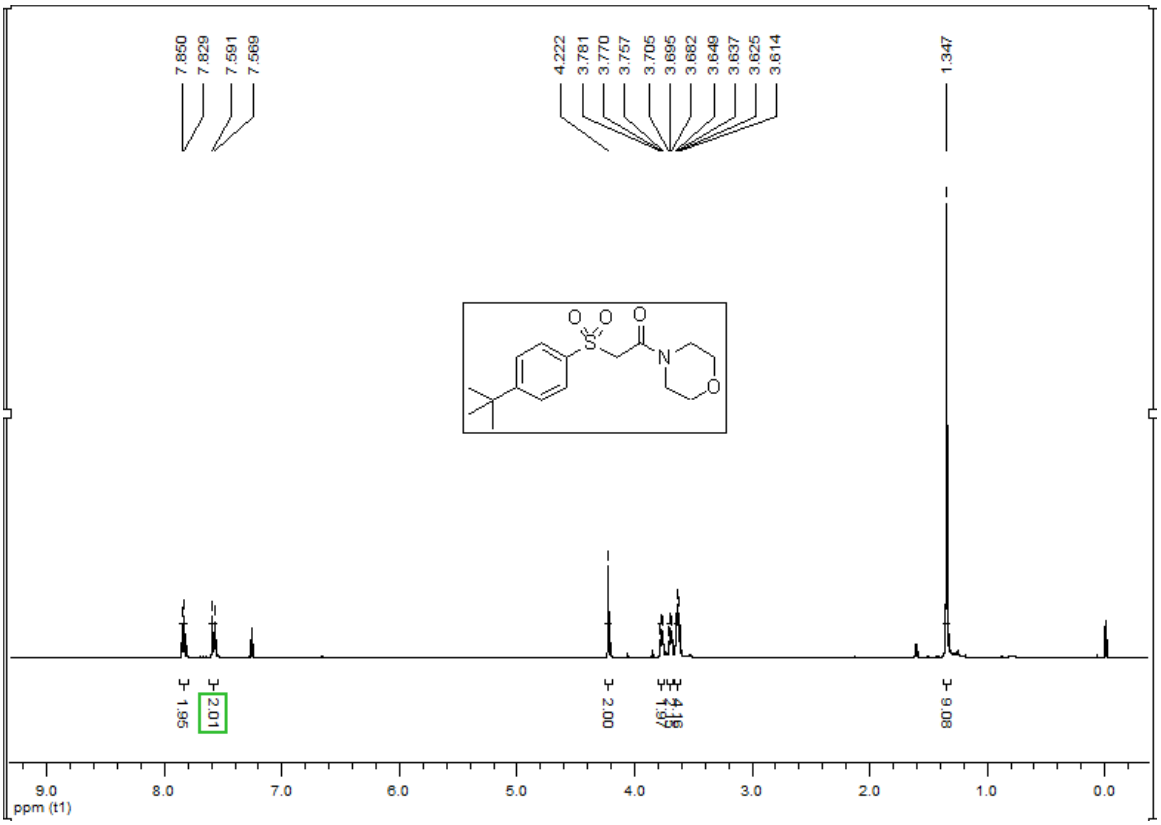
Analysis Info

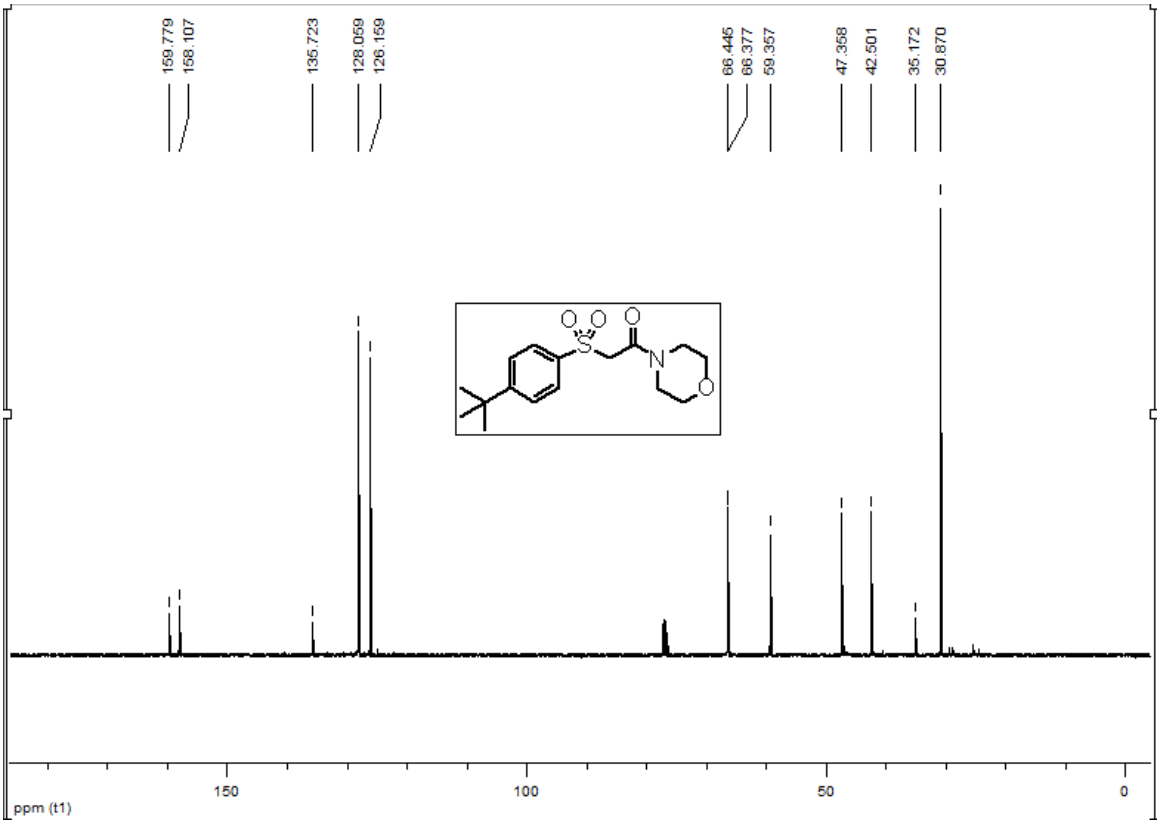
Analysis Name	D:\Data\2016MS\TT\0908\H-1_RD4_01_10994.d	Acquisition Date	9/8/2016 1:32:09 PM
Method	tune_200-800_hcoona-pos-2.5min.m	Operator	gftang
Sample Name	H-1	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.6 Bar
Focus	Not active			Set Dry Heater	180 ℃
Scan Begin	50 m/z	Set Capillary	2500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste




7.850
7.829
7.591
7.569
4.222
3.781
3.770
3.757
3.705
3.695
3.682
3.649
3.637
3.625
3.614
1.347
1.95
2.01
2.00
4.19
4.19
9.08
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


159.779
158.107
135.723
128.059
126.159
66.445
66.377
59.357
47.358
42.501
35.172
30.870
150
100
50
0
ppm (t1)

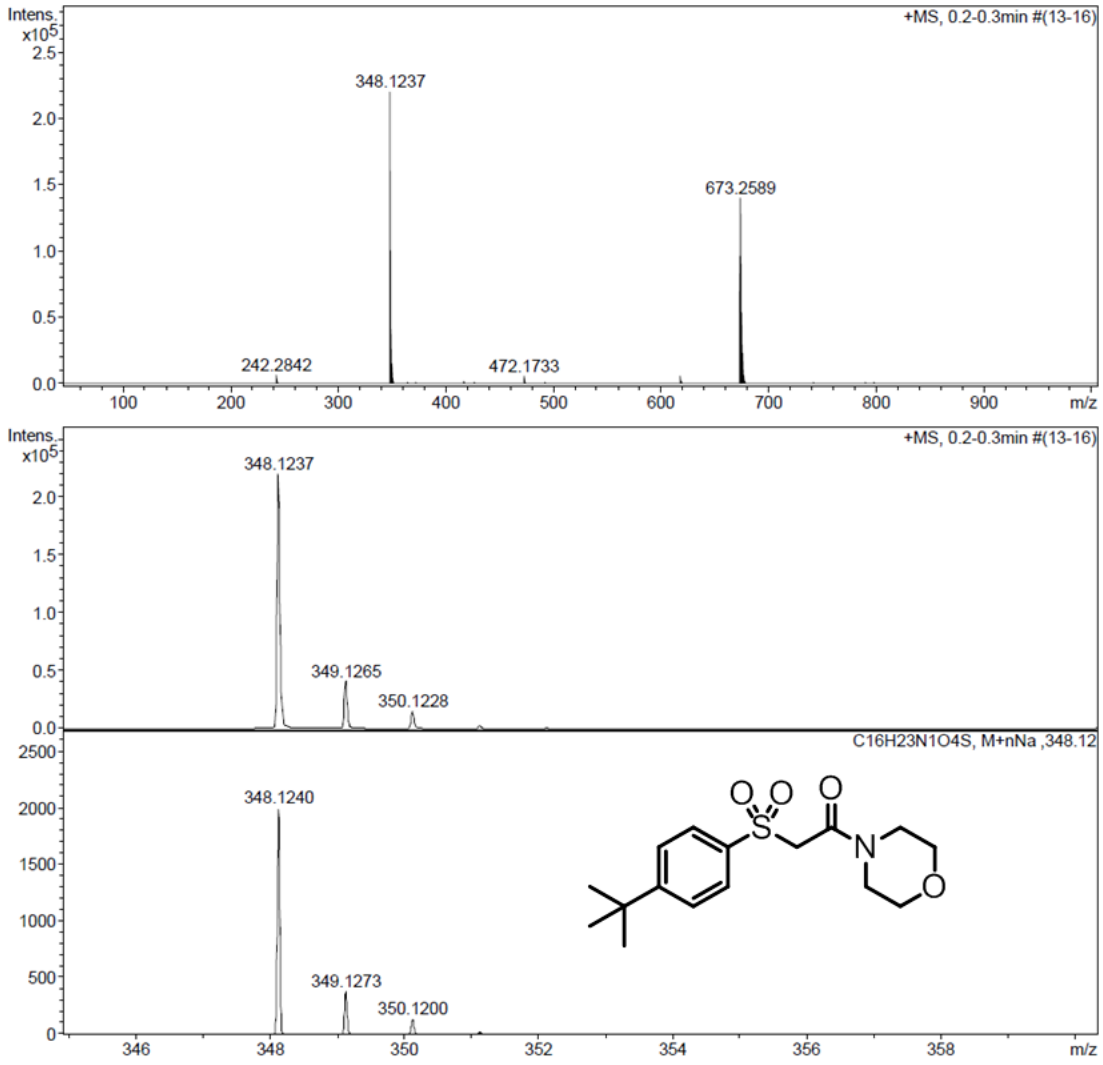
Display Report

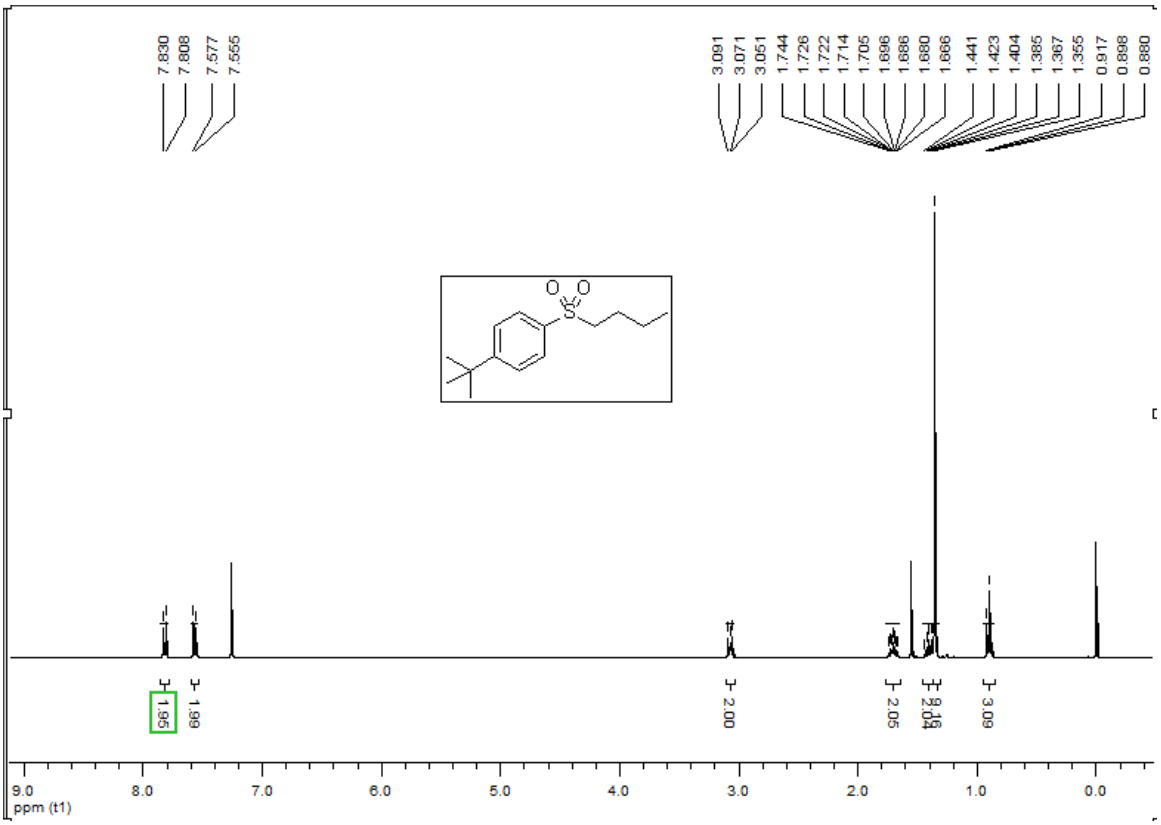
Analysis Info

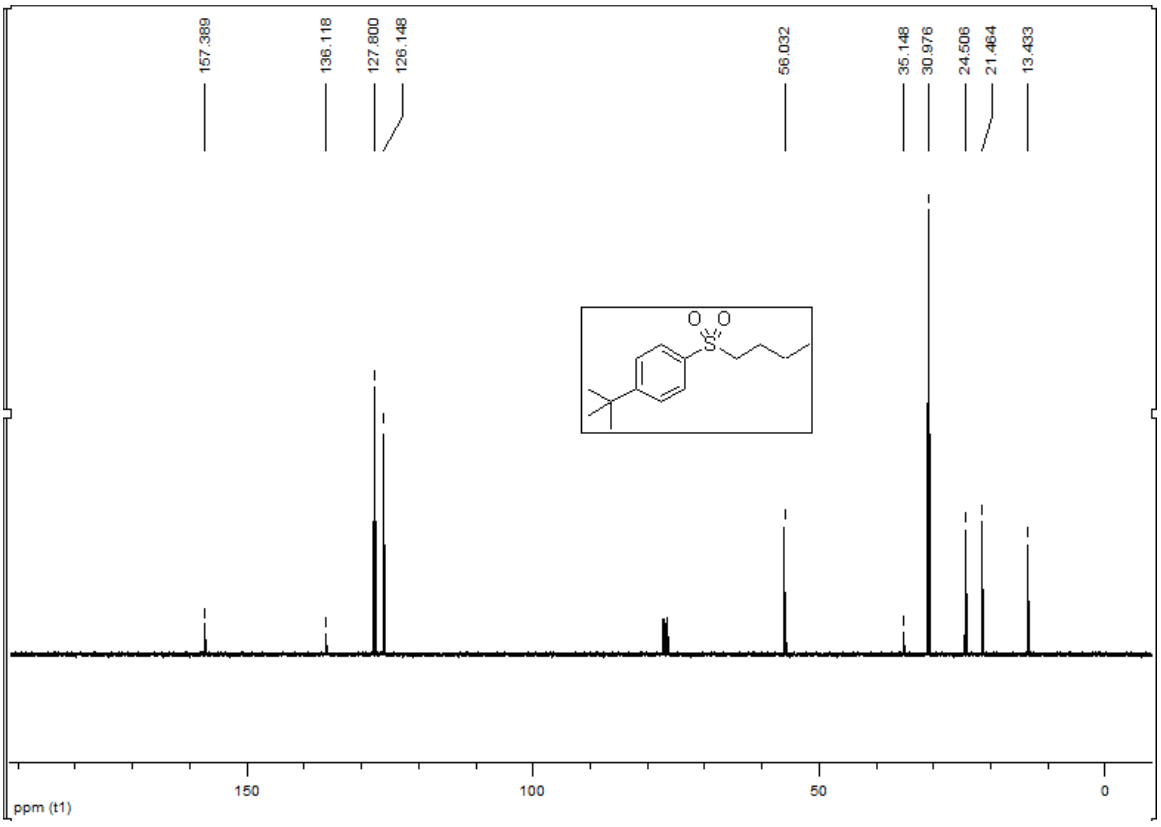
Analysis Name	D:\Data\2016MS\TT\1108\H2_BC4_01_12041.d	Acquisition Date	11/8/2016 4:08:46 PM
Method	tune_200-800_hcoona-pos-2.5min.m	Operator	gftang
Sample Name	H2	Instrument / Ser#	micrOTOF II 10257
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	180 ℃
Scan Begin	50 m/z	Set Capillary	2500 V	Set Dry Gas	6.0 l/min
Scan End	1000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste




7.830
7.808
7.577
7.555
3.091
3.071
3.051
1.744
1.726
1.722
1.714
1.705
1.696
1.686
1.680
1.666
1.441
1.423
1.404
1.385
1.367
1.355
0.917
0.898
0.880
1.86
1.99
2.00
2.05
9.18
3.09
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


157.389
136.118
127.800
126.148
56.032
35.148
30.976
24.506
21.464
13.433
150
100
50
0
ppm (t1)

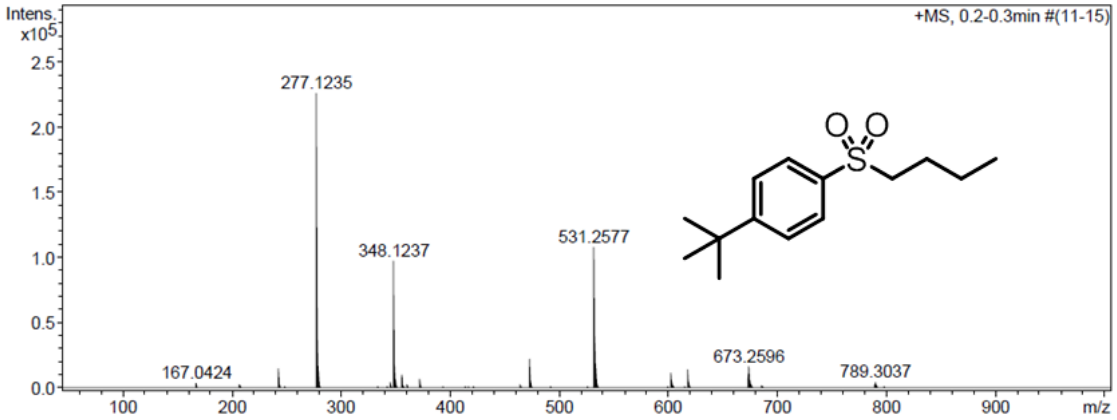
Display Report

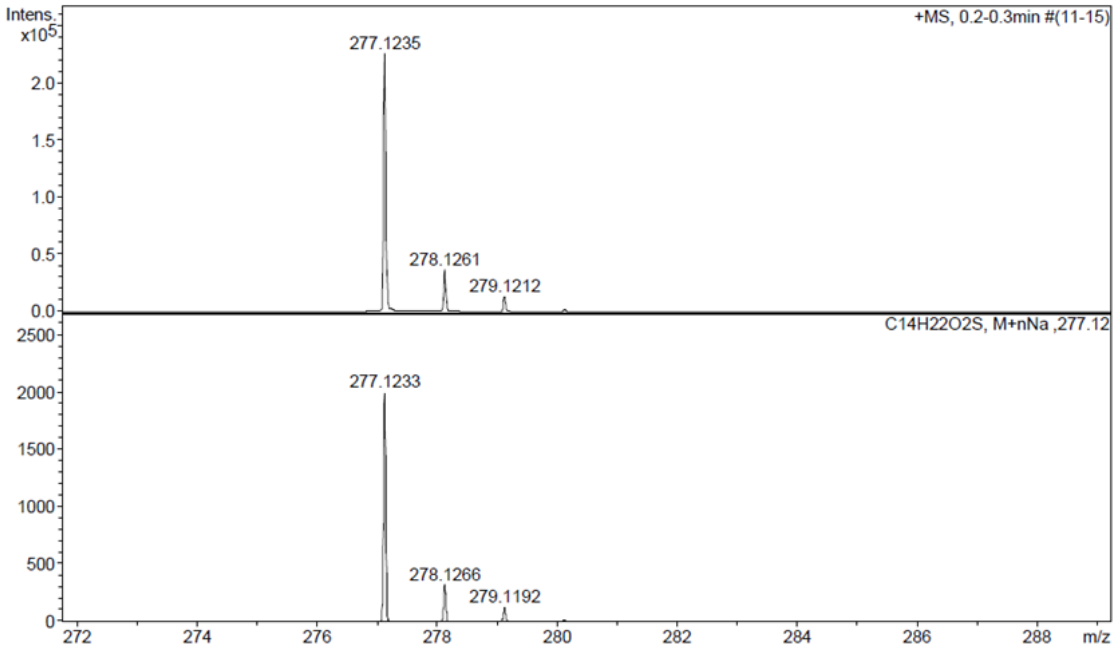
Analysis Info

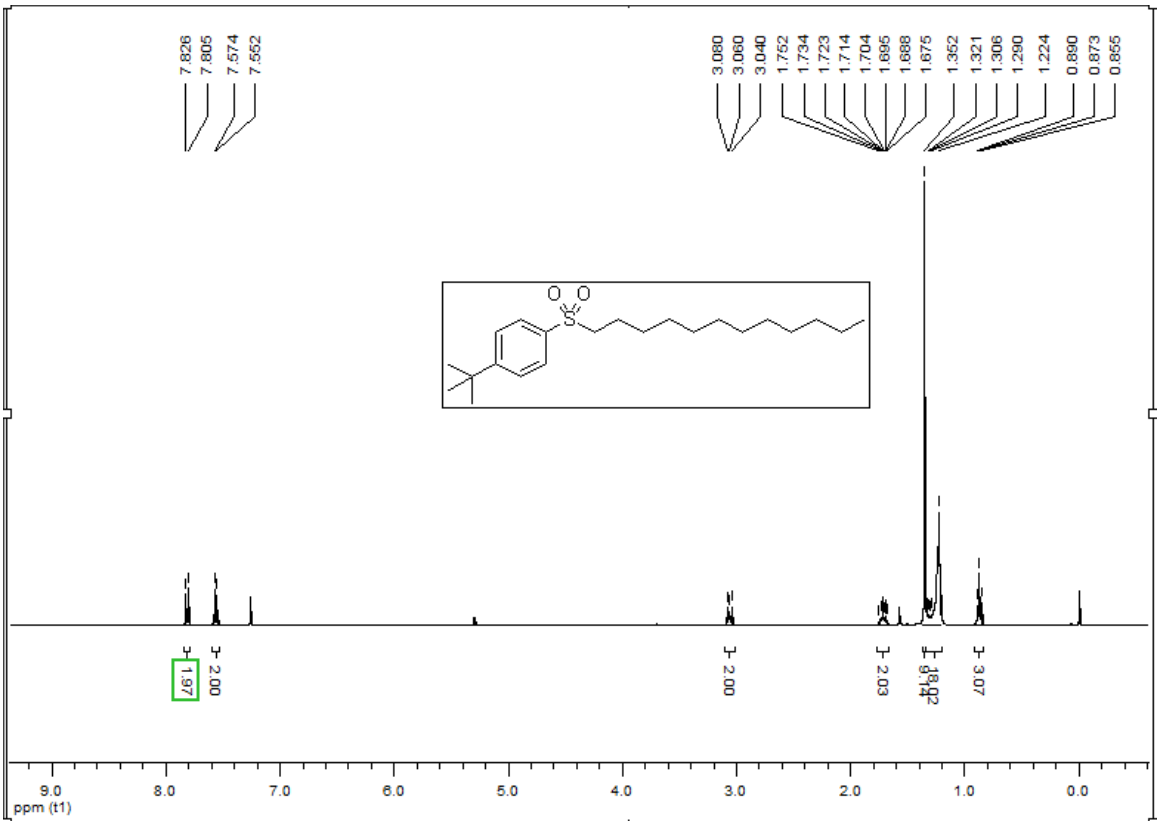
Analysis Name	D:\Data\2016MS\TT\1108\H3_BC5_01_12042.d	Acquisition Date	11/8/2016 4:12:47 PM
Method	tune_200-800_hcoona-pos-2.5min.m	Operator	gftang
Sample Name	H3	Instrument / Ser#	micrOTOF II 10257
Comment			

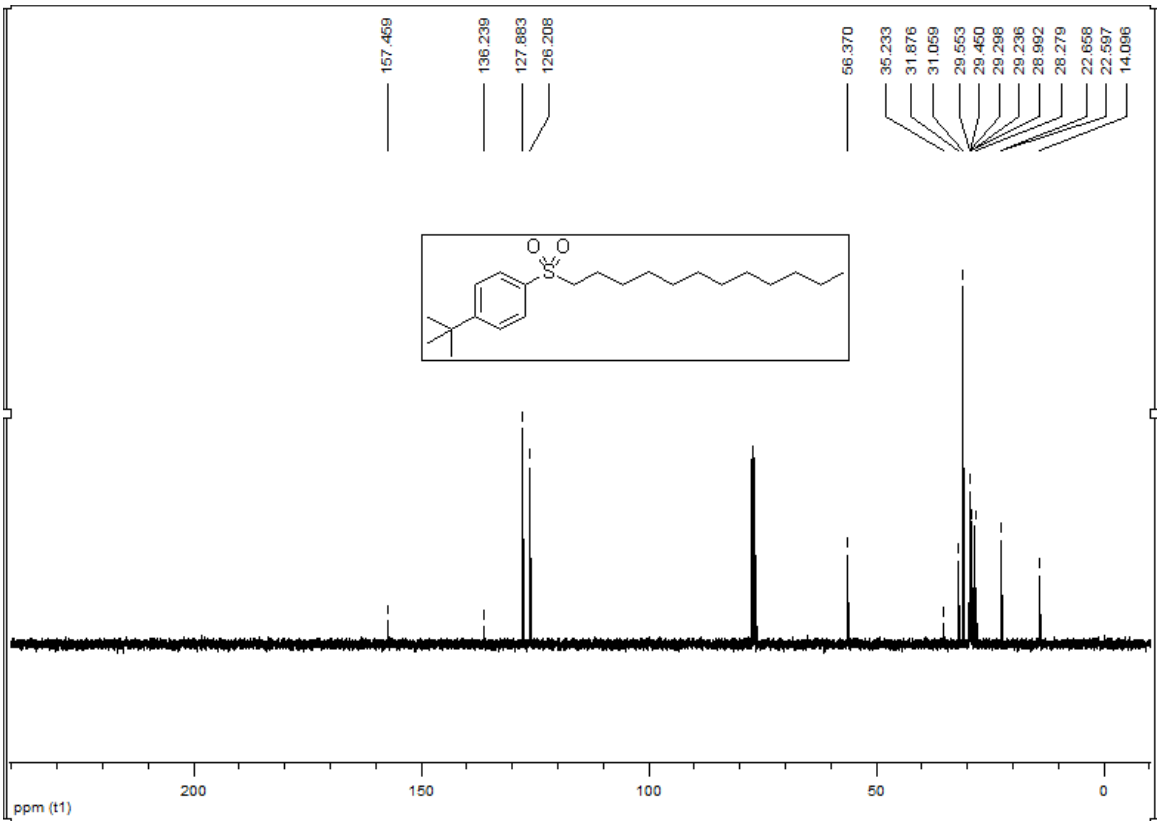
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	180 ℃
Scan Begin	50 m/z	Set Capillary	2500 V	Set Dry Gas	6.0 l/min
Scan End	1000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste






7.826
7.805
7.574
7.552
3.080
3.060
3.040
1.752
1.734
1.723
1.714
1.704
1.695
1.688
1.675
1.352
1.321
1.306
1.290
1.224
0.890
0.873
0.855
1.97
2.00
2.00
2.03
18.02
9.14
3.07
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


157.459
136.239
127.883
126.208
58.370
35.233
31.876
31.059
29.553
29.460
29.298
29.236
28.992
28.279
22.658
22.597
14.096
200
150
100
50
0
ppm (t1)

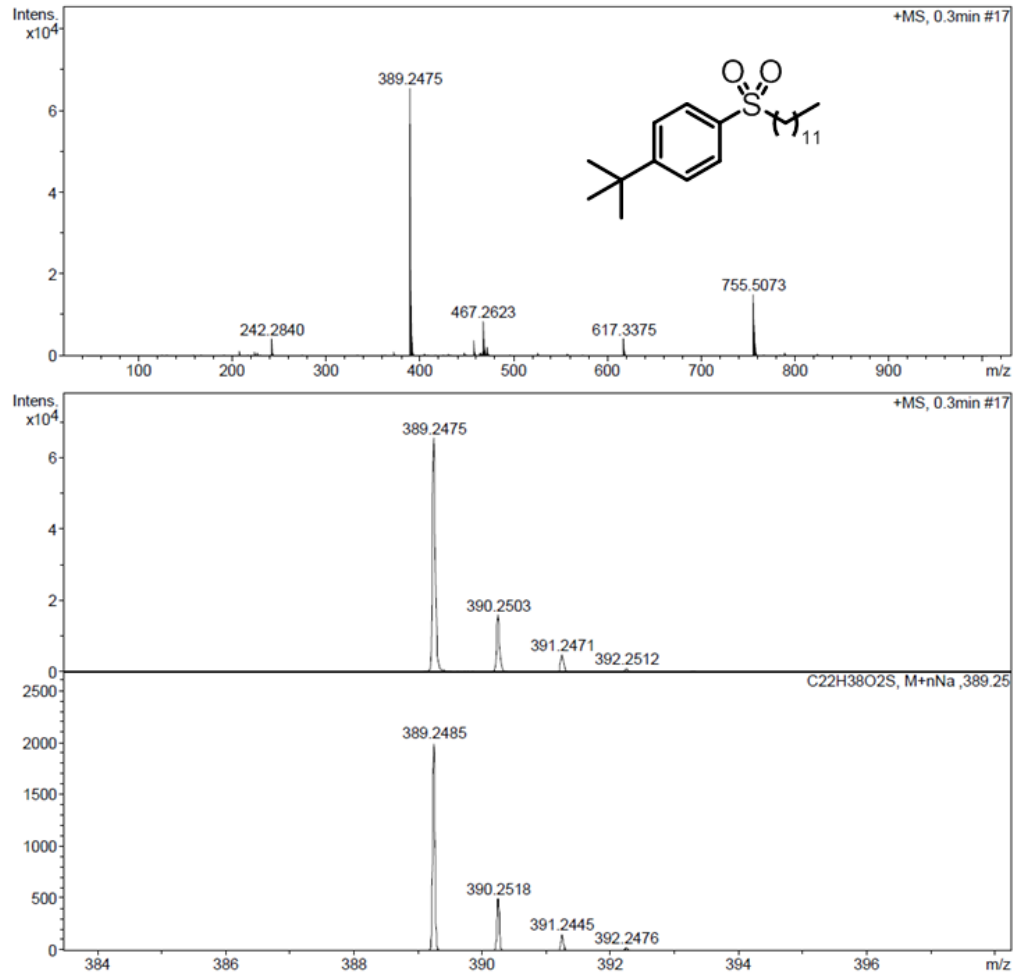
Display Report

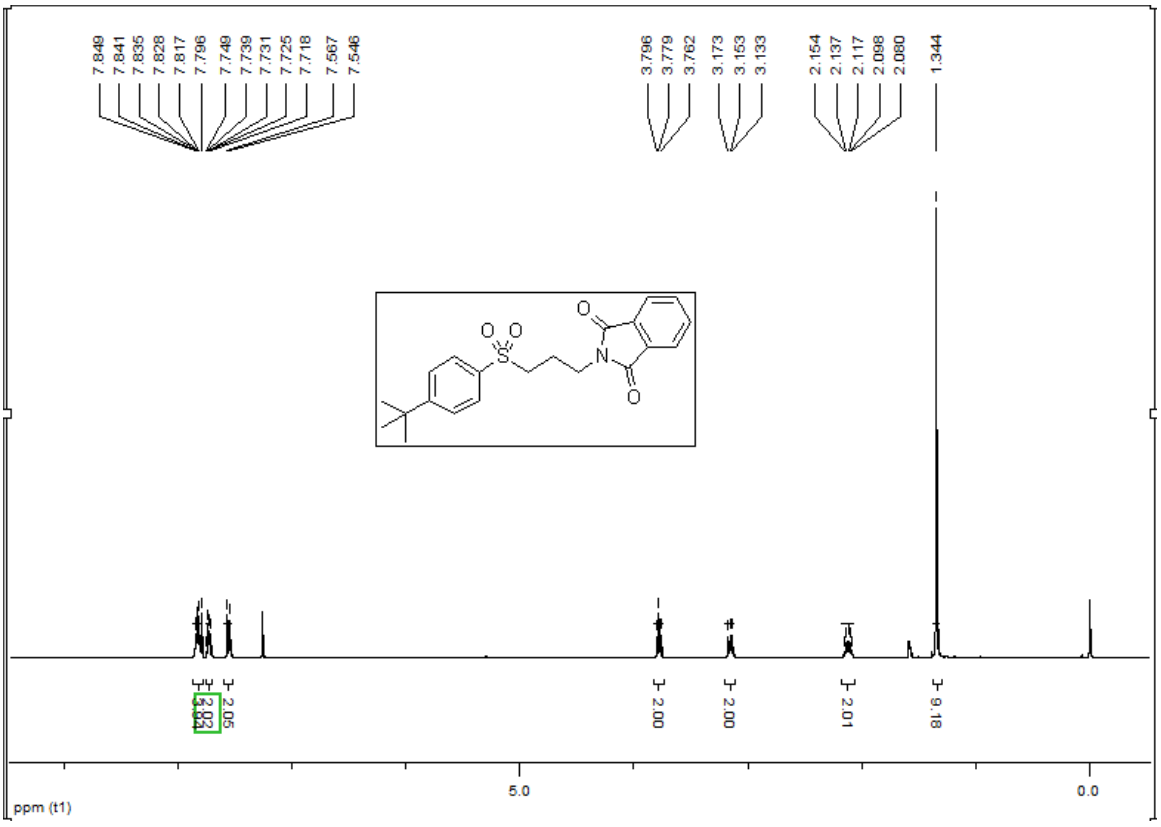
Analysis Info

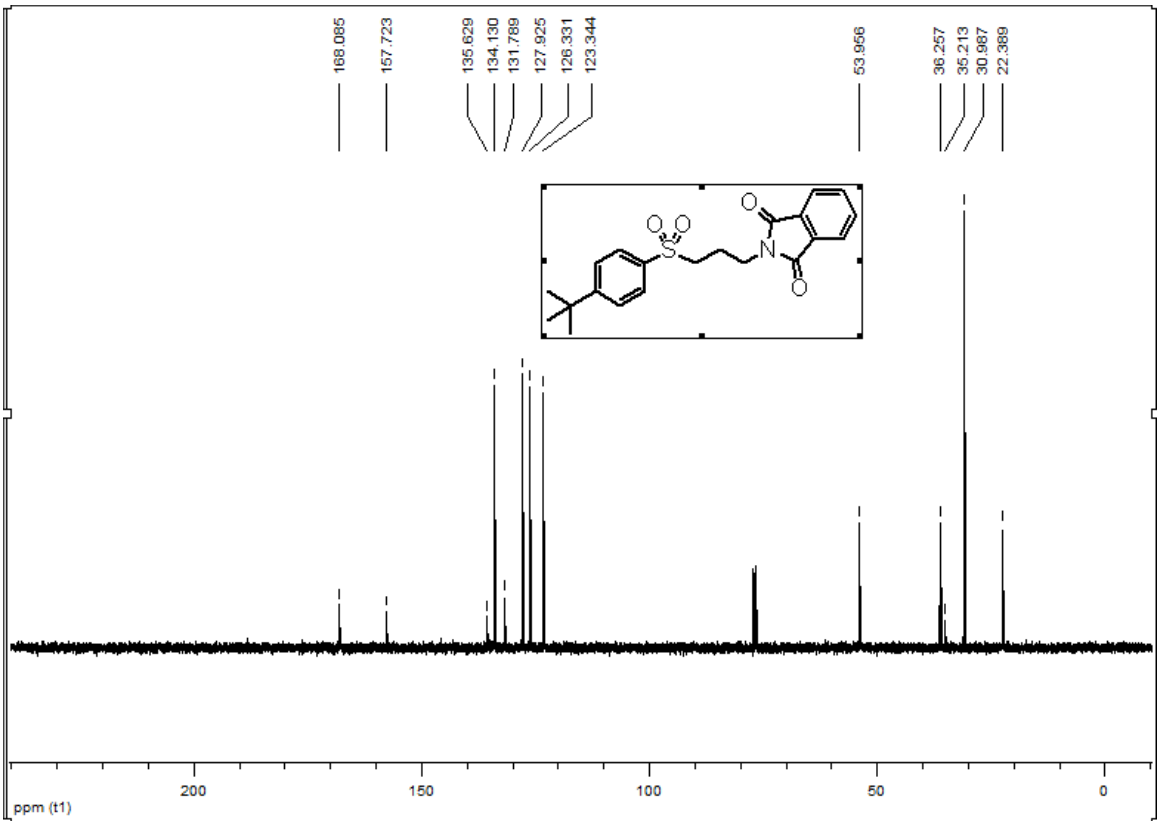
Analysis Name	D:\Data\2016MS\TT\1108\H1_BC3_01_12040.d	Acquisition Date	11/8/2016 4:04:43 PM
Method	tune_200-800_hcoona-pos-2.5min.m	Operator	gftang
Sample Name	H1	Instrument / Ser#	micrOTOF II 10257
Comment			

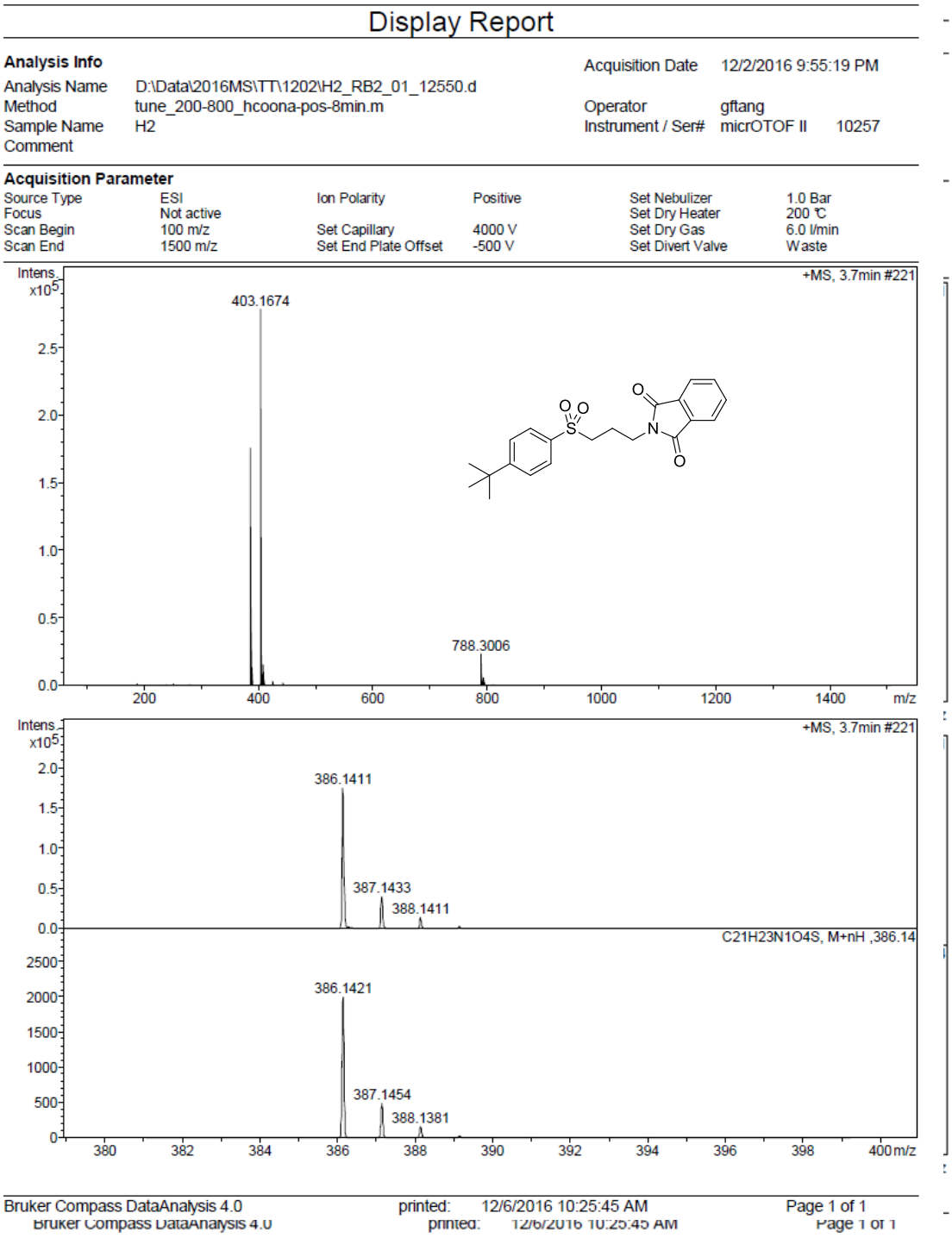
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	2500 V	Set Dry Gas	6.0 l/min
Scan End	1000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste




7.849
7.841
7.835
7.828
7.817
7.796
7.749
7.739
7.731
7.725
7.718
7.567
7.546
3.796
3.779
3.762
3.173
3.153
3.133
2.154
2.137
2.117
2.098
2.080
1.344
2.06
2.00
2.00
2.01
9.18
5.0
0.0
ppm (t1)


168.085
157.723
135.629
134.130
131.789
127.925
126.331
123.344
53.956
36.257
35.213
30.987
22.389
200
150
100
50
0
ppm (t1)



6. References.

S1 W. Fang, Q. Deng, M. Xu and T. Tu, *Org. Lett.*, 2013, *15*, 3678-3681.

S2 G. Jung, Y. T. Lee, S. Y. Choi, D. S. Choi, Y. K. Kang and Y. K. Chung, *J. Organomet. Chem.*, 2009, *694*, 297-303.