Supporting Materials for:

Suzuki-Miyaura Coupling of Phosphinoyl-*α*-Allenic Alcohols with Arylboronic Acids Catalyzed by Palladium Complex "On Water": An Efficient Method to Generate Phosphinoyl 1,3-Buta-dienes and Derivatives

Teng Liu [a], Jie Dong [a,§], Shu-Jun Cao [a,§], Li-Cheng Guo [a,§] AND Lei Wu [a,b]*

[a] *Jiangsu Key Laboratory of Pesticide Science and Department of Chemistry, College of Sciences, Nanjing Agricultural University, Nanjing 210095, P. R. China.*

Tel/Fax: +86-25-84396716; E-mail: rickywu@njau.edu.cn

[b] *CAS Key Laboratory of Molecular Recognition and Function, Institute of Chemistry, Chinese Academy of Sciences, Beijing 100190, P. R. China. E-mail: rickywu@iccas.ac.cn*

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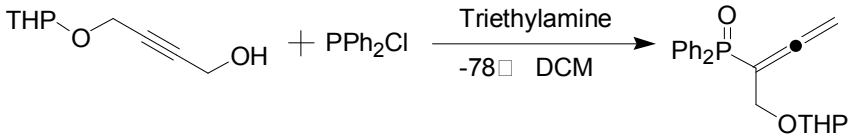
1. General Information:

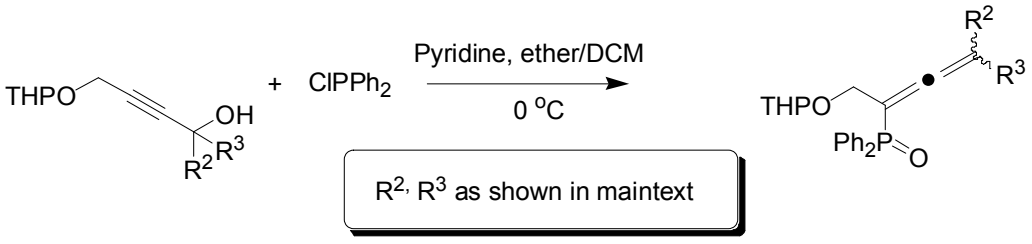
Solvents and reagents were reagent grade and used without purification unless otherwise noted. Anhydrous solvents were obtained as follow: THF and dioxane by distillation from sodium and benzophenone; All reactions were carried out under nitrogen or argon unless otherwise specified. All 1H-NMR (400 MHz) spectra were recorded on a Bruker-DMX 400 using $CDCl_3$ solution in the presence of tetramethylsilane (TMS) as an internal standard and are reported in ppm (δ). Coupling constants are reported in Hertz (Hz). Spectral splitting patterns are designated as s, singlet; d, doublet; t, triplet; q, quartet; p, pentet; m, multiplet; and br, broad. High and Low resolution fast atom bombardment (FAB) measurements were made with a JEOL JMS-AX505HA mass spectrometer.

2. General Procedures for Substrates Preparation:

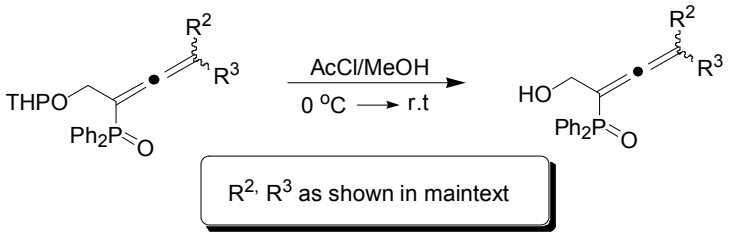


To a stirred and cooled (-78 °C) solution of the acetylenic alcohol (40 mmol) in CH_2Cl_2 (50 mL) under nitrogen was added sequencely triethylamine (11.17 ml, 80 mmol) and diphenylphosphine chloride (40 mmol) in CH_2Cl_2 (50 mL). The stirring was maintained 1 h at -78 °C and then at room temperature for overnight. The solution was quenched with ice water and extracted with CH_2Cl_2, and the organic layer was dried with Na_2SO_4. Concentration *in vacuo* gave the crude product that was subjected to flash chromatography on silica gel eluting with ethyl acetate/petroleum ether (1:3).

[When R_2,R_3≠H]:



To a stirred and cooled (0 °C) solution of the acetylenic alcohol (40 mmol) in anhydrous ether (50 mL) and pyridine (3.88 mL, 48 mmol) under nitrogen was added dropwisely diphenylphosphine chloride (40 mmol) in CH_2Cl_2 (50 mL). The stirring was maintained 1 h at 0 °C and then at room temperature overnight. The solution was quenched with ice water and extracted with CH_2Cl_2, and the organic layer was dried with Na_2SO_4. Concentration *in vacuo* gave the crude product that was subjected to flash chromatography on silica gel eluting with ether.



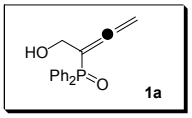
The cooled solution of THP-protected phosphinoyl-allenic alcohol (32 mmol) in methanol (150 mL) was added acetyl chloride (0.32 mmol) dropwisely. The stirring was maintained 5 min at 0 °C and at room temperature for 1h. The reaction was quenched with ice water and extracted with ethyl acetate, and the organic layer was collected and dried with Na_2SO_4. Concentration *in vacuo* gave the crude product that was subjected to flash chromatography on silica gel eluting with ethyl acetate/petroleum ether (3:1).

3. General Procedures for the Palladium-Catalyzed Couplings of Phosphinoyl-α-Allenic Alcohols with Arylboronic Acids

To a 10 mL flask equipped with condenser under nitrogen was added phosphinoyl-*α*-allenic alcohol (1 mmol), arylboronic acid (2 mmol), $Pd(PPh_3)_2Cl_2$ (5 mol%), and 10 mL degassed water. The reaction mixture was then heated to reflux for several hours until the complete consuming of starting materials monitored by TLC. After being cooling down, the reaction was extracted with ethyl acetate (5 mL×2). The crude product was purified on flash chromatography, with ethyl acetate/ petroleum ether (1:1) as eluents to afford product.

4. Characterizations of Substrates and Coupling Products (^{1}H-NMR, ^{13}C-NMR, ^{31}P-NMR, HR-MS)

4.1 Substrates:

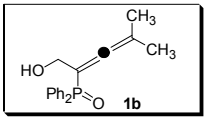


White solids, m.p.: 113.5-115.1°C.

^{1}H-NMR: δ 3.482 (br. s, 1H), 4.468-4.507 (m, 2H), 4.837-4.874 (m, 2H), 7.484-7.528(m, 4H), 7.568-7.608 (m, 2H), 7.744-7.795 (m, 4H).

^{13}C-NMR: δ 61.47, 61.53, 96.64, 97.66, 128.44, 128.56, 130.93, 131.66, 131.76, 131.98, 132.27, 132.29, 210.72, 210.80.

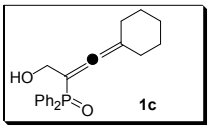
^{31}P-NMR: δ 30.724



White solids, m.p.: 125.7-130.8 °C.

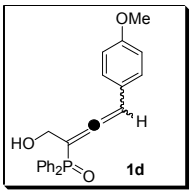
^{1}H-NMR: δ 1.491 (d, *J* = 4.0 Hz, 6H), 2.365 (br. s, 1H), 4.405 (d, *J* = 12.0 Hz, 2H), 7.438-7.482 (m, 4H), 7.516-7.566 (m, 2H), 7.678-7.729 (m, 4H).

^{13}C-NMR: δ 19.15, 19.20, 61.73, 61.81, 95.42, 96.45, 99.28, 99.41, 128.33, 128.45, 131.48, 131.58, 131.69, 131.96, 131.98, 132.73, 206.73, 206.80.
^{31}P-NMR: δ 32.889.



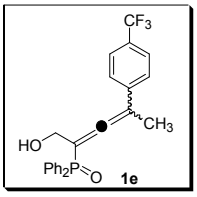
Pale yellow solids, m.p.: 144.4-147.4 °C.

^{1}H-NMR: δ 0.925-1.024 (m, 2H), 1.247-1.513 (m, 4H), 1.862-2.207 (m, 4H), 4.395 (d, *J* = 11.6 Hz, 2H), 7.426-7.546 (m, 6H), 7.673-7.724 (m, 4H).
^{13}C-NMR: δ 25.42, 26.36, 26.39, 29.98, 30.03, 61.95, 62.03, 95.28, 96.31, 105.42, 105.55, 128.35, 128.47, 131.63, 131.72, 131.94, 131.96, 132.74, 203.49, 203.56.
^{31}P-NMR: δ 33.263.



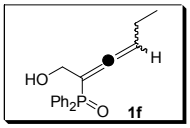
White solids, m.p.: 135.5-138.1 °C.

^{1}H-NMR: δ 3.812 (s, 3H), 4.573-4.607 (m, 2H), 6.228-6.264 (m, 1H), 6.799 (d, *J* = 8.8 Hz, 2H), 7.009 (d, *J* = 8.8 Hz, 2H), 7.353-7.398 (m, 2H), 7.451-7.503 (m, 3H), 7.543-7.588 (m, 1H), 7.719-7.815(m, 4H).
^{13}C-NMR: δ 55.37, 62.13, 62.19, 96.98, 97.12, 101.54, 102.55, 114.26, 123.60, 123.66, 128.33, 128.35, 128.38, 128.47, 128.50, 128.60, 131.39, 131.47, 131.57, 131.62, 131.72, 132.09, 132.18, 132.20, 132.28, 132.31, 132.38, 132.43, 159.48, 209.06, 209.13.
^{31}P-NMR: δ 30.499.



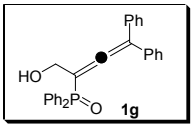
White solids, **m.p.**: 146.0-146.4 °C.

^{1}H-NMR: δ 1.907 (d, *J* = 5.6 Hz, 3H),3.015 (br. s, 1H), 4.530-4.623 (m, 2H), 7.198 (d, *J* = 8.4 Hz, 2H), 7.288-7.333 (m, 2H), 7.411-7.568 (m, 6H), 7.627-7.745 (m, 4H).
^{13}C-NMR: δ 15.85, 15.91, 61.17, 61.24, 100.55, 101.54, 103.56, 103.70, 122.67, 125.22, 125.26, 125.29, 125.32, 125.38, 125.94, 125.96, 127.70, 127.83, 128.28, 128.40, 128.45, 128.57, 129.21, 129.54, 130.79, 130.91, 131.30, 131.35, 131.40, 131.42, 131.52, 131.84, 131.96, 132.16, 132.19, 132.23, 132.26, 138.63, 138.69, 209.05, 209.11.
^{31}P-NMR: δ 31.659.



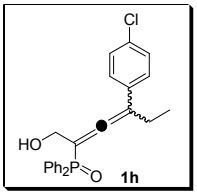
Pale yellow liquid, **m.p.**: 72.0-74.5 °C.

^{1}H-NMR: δ 0.727 (t, *J* = 7.6 Hz, 3H), 1.807-1.895 (m, 2H), 3.617 (br. s, 1H), 4.377-4.479 (m, 2H), 5.221-5.281 (m, 1H), 7.417-7.559 (m, 6H), 7.670-7.764 (m, 4H).
^{13}C-NMR: δ 13.15 ,13.18, 21.06, 21.11, 61.41, 61.45, 61.49, 61.52, 95.95, 96.08, 98.13, 99.14, 128.26, 128.34, 128.38, 128.47, 131.21, 131.28, 131.51, 131.56, 131.62, 131.72, 131.97, 132.00, 132.02, 132.04, 132.07, 132.26, 132.32, 132.56, 132.61, 207.71, 207.78.
^{31}P-NMR: δ 31.683.



White solids, **m.p.**: 165.1-165.7 °C.

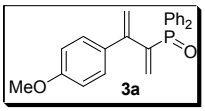
^{1}H-NMR: δ 3.462 (br. s, 1H), 4.640 (d, *J* = 11.2 Hz, 2H), 6.959-6.990 (m, 4H), 7.276-7.371 (m, 10H), 7.460-7.519 (m, 2H), 7.619-7.670 (m, 4H).
^{13}C-NMR (d^{6}-DMSO): δ 58.53, 58.65, 103.03, 103.98, 112.99, 113.12, 127.95, 127.97, 128.10, 128.49, 128.61, 128.67, 130.96, 131.06, 131.79, 132.09, 132.12, 132.82, 134.69, 134.75, 208.83, 208.87.
^{31}P-NMR: δ 31.180.



White solids, **m.p.**: 122.0-122.4 °C.

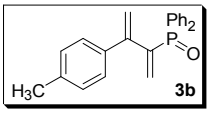
^{1}H-NMR: δ 0.870 (t, *J* = 7.2 Hz, 3H), 2.064-2.286 (m, 2H), 4.531-4.625 (m, 2H), 7.071 (d, *J* = 8.8 Hz, 2H), 7.251 (d, *J* = 8.8 Hz, 2H), 7.310-7.355 (m, 1H), 7.366-7.485 (m, 3H), 7.641-7.745 (m, 6H).
^{13}C-NMR: δ 12.31, 23.04, 23.09, 61.57, 61.63, 102.67, 110.55, 110.69, 127.42, 127.44, 127.94, 128.06, 128.35, 128.48, 128.72, 130.86, 130.92, 130.95, 131.01, 131.34, 131.44, 131.53, 131.57, 131.67, 131.92, 131.94, 132.01, 132.19, 133.08, 133.15, 133.42, 208.71, 208.81.
^{31}P-NMR: δ 31.793.

4.2 Coupling products:



Viscous liquid.

^{1}H-NMR: δ 3.74 (s, 3H), 5.31 (s, 1H), 5.55 (s, 1H), 5.87 (d, *J* = 19.6 Hz, 1H), 6.03 (d, *J* = 40 Hz, 1H), 6.76 (d, *J* = 8.8 Hz, 2H), 7.16 (d, *J* = 8.8 Hz, 2H), 7.39-7.42 (m, 4H), 7.46-7.49 (m, 2H), 7.73-7.78 (m, 4H).
^{13}C-NMR: δ 55.28, 113.54, 118.09, 118.14, 128.37, 128.50, 129.10, 131.20, 131.80, 131.86, 131.90, 132.23, 132.69, 132.75, 133.91, 134.01, 143.86, 144.77, 145.30, 145.40, 159.25.
^{31}P-NMR: δ 29.200
HR-MS (ESI): $C_{23}H_{21}NaO_2P$ (calcd.: 383.1177), found: 383.1171 ($[M+Na]^+$)



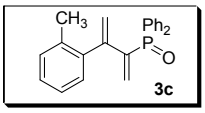
Viscous pale yellow liquid

^{1}H-NMR: 2.314 (s, 3H), 5.349 (s, 1H), 5.627 (d, *J* = 1.6 Hz, 1H), 5.837 (d, *J* = 20.0 Hz, 1H), 6.001 (d, *J* = 40.4 Hz, 1H), 7.054 (d, *J* = 8.0 Hz, 2H), 7.125 (d, J = 8.0 Hz, 2H), 7.414-7.529 (m, 6H), 7.736-7.786 (m, 4H).

^{13}C-NMR: 21.12, 118.95, 119.00, 127.84, 128.33, 128.45, 128.82, 131.22, 131.77, 131.81, 131.86, 132.25, 133.88, 133.98, 137.44, 137.47, 137.53, 143.52, 144.43, 145.51, 145.60.

^{31}P-NMR: δ 30.171

HR-MS (ESI): $C_{23}H_{21}NaOP$ (calcd.: 367.1228), found:367.1222 ($[M+Na]^+$)



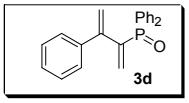
Viscous pale yellow liquid

^{1}H-NMR: δ 2.227 (s, 3H), 5.256 (s, 1H), 5.461-5.621 (m, 2H), 6.076 (s, 1H), 7.098-7.239 (m, 5H), 7.488-7.588 (m, 6H), 7.802-7.849 (m, 4H).

^{13}C-NMR: δ 19.73, 122.44, 122.48, 125.74, 127.64, 128.54, 128.66, 129.53, 130.04, 131.55, 131.73, 131.82, 131.97, 132.00, 132.58, 133.04, 133.15, 135.87, 140.42, 140.50, 142.17, 143.08, 144.61, 144.72.

^{31}P-NMR: δ 33.554

HR-MS (ESI): $C_{23}H_{21}NaOP$ (calcd.: 367.1228), found:367.1222 ($[M+Na]^+$)

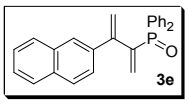


Viscous pale yellow liquid

^{1}H-NMR: δ 5.378 (s, 1H), 5.681 (d, *J* = 1.6Hz, 1H), 5.852 (d, *J* = 19.6 Hz, 1H), 5.994 (d, *J* = 40.0 Hz, 1H), 7.236-7.261 (m, 4H), 7.419-7.534 (m, 6H), 7.739-7.790 (m, 4H).

^{31}P-NMR: δ 30.101

HR-MS (ESI): $C_{22}H_{19}NaOP$ (calcd.: 353.1071), found: 353.1066 ($[M+Na]^+$)



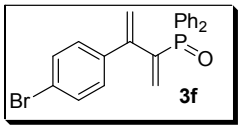
Viscous pale yellow liquid.

^{1}H-NMR: δ 5.404 (d, *J* = 16.8 Hz, 2H), 5.521 (d, *J* = 41.6 Hz, 1H), 6.263 (s, 1H), 7.300-7.320 (m, 1H), 7.397-7.455 (m, 3H), 7.469-7.581 (m, 6H), 7.773-7.870 (m, 7H).

^{13}C-NMR: δ 123.74, 123.78, 123.80, 123.81, 125.29, 125.31, 125.89, 126.01, 126.11, 126.80, 127.97, 128.17, 128.54, 128.56, 128.66, 128.68, 131.82, 131.91, 132.02, 133.57, 133.61, 133.71, 133.79, 138.72, 138.77, 138.79, 143.60, 143.68.

^{31}P-NMR: δ 31.662.

HR-MS (ESI): **HR-MS (ESI)**: $C_{26}H_{21}NaOP$ (calcd.: 403.1228), found:403.1222 ($[M+Na]^+$)

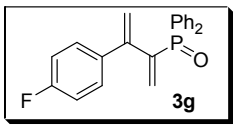


Viscous pale yellow liquid

^{1}H-NMR: δ 5.414 (s, 1H), 5.690 (d, *J* = 1.6 Hz, 1H), 5.901 (d, *J* = 19.6 Hz, 1H), 6.030 (d, *J* = 40 Hz, 1H), 7.127 (d, *J* = 8.4 Hz, 2H), 7.401 (d, *J* = 8.4 Hz, 2H), 7.464-7.589 (m, 6H), 7.756-7.807 (m, 4H).

^{31}P-NMR: δ 29.518

HR-MS (ESI): $C_{22}H_{18}BrNaOP$ (calcd.: 431.0176), found:431.0171 ($[M+Na]^+$)



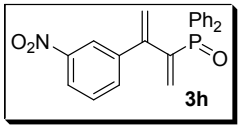
Viscous pale yellow liquid

^{1}H-NMR: δ 5.341 (s, 1H), 5.616 (d, *J* = 2.0 Hz, 1H), 5.861 (d, *J* = 19.6 Hz, 1H), 5.997 (d, *J* = 39.6 Hz, 1H), 6.916 (t, *J* = 8.8Hz, 2H), 6.894-7.191 (m, 2H), 7.418-7.538 (m, 6H), 7.715-7.766 (m, 4H).

^{13}C-NMR: δ 114.96, 115.17, 119.42, 119.47, 128.46, 128.53, 128.58, 128.65, 129.57, 129.65, 130.90, 131.80, 131.89, 131.93, 132.01, 132.04, 132.09, 132.15, 134.06, 134.15, 136.20, 136.24, 136.25, 136.29, 143.70, 144.62, 144.93, 145.03, 161.17, 163.62.

^{31}P-NMR: δ 29.627

HR-MS (ESI): $C_{22}H_{18}FNaOP$ (calcd.: 371.0977), found:371.0972 ($[M+Na]^+$)

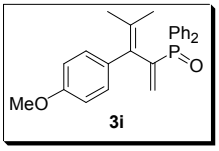


Viscous pale yellow liquid

^{1}H-NMR: δ 5.489 (s, 1H), 5.743 (d, *J* = 2.0 Hz), 5.909 (d, *J* = 21.6 Hz, 1H), 6.029 (d, J = 39.2 Hz, 1H), 7.422-7.524 (m, 8H), 7.716-7.767 (m, 4H), 7.971-8.085 (m, 2H).

^{31}P-NMR: δ 28.681

HR-MS (ESI): $C_{22}H_{18}NNaO_3P$ (calcd.: 398.0922), found:398.0917($[M+Na]^+$)



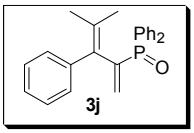
Viscous pale yellow liquid

^{1}H-NMR: δ 1.305 (s, 3H), 1.321 (s, 3H), 3.494 (d, *J* = 8.4 Hz, 2H), 3.762 (s, 3H), 6.733 (d, *J* = 8.8 Hz, 2H), 7.036 (d, *J* = 8.8 Hz, 2H), 7.397-7.489 (m, 6H), 7.651-7.702 (m, 4H).

^{13}C-NMR: δ 18.84, 18.90, 33.20, 33.29, 55.12, 96.53, 97.55, 99.12, 99.26, 113.43, 128.05, 128.17, 128.42, 128.54, 129.96, 130.95, 131.01, 131.42, 131.51, 131.77, 131.94, 132.04, 132.80, 157.93, 208.55, 208.62.

^{31}P-NMR: δ 31.546

HR-MS (ESI): $C_{25}H_{25}NaO_2P$ (calcd.: 411.1490), found:411.1484 ($[M+Na]^+$)



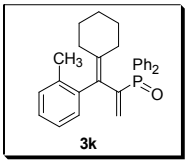
Pale yellow solids**, m.p.**: 85.6-86.8 °C.

^{1}H-NMR: δ 1.291 (s, 3H), 1.307 (s, 3H), 3.548 (d, *J* = 8.4 Hz, 2H), 7.113-7.217 (m, 5H), 7.395-7.510 (m, 6H), 7.662-7.712 (m, 4H).

^{13}C-NMR: δ 18.85, 18.90, 34.09, 34.18, 96.48, 97.51, 99.25, 99.39, 126.16, 128.09, 128.13, 128.25, 129.12, 131.57, 131.59, 131.66, 131.92, 132.95, 139.06, 139.12, 208.73, 208.80.

^{31}P-NMR: δ 31.317

HR-MS (ESI): $C_{24}H_{23}NaOP$ (calcd.: 381.1384), found:381.1379 ($[M+Na]^+$)



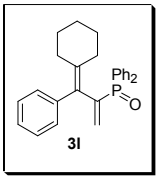
Pale yellow solids, **m.p.**: 117.7-120.0 °C.

^{1}H-NMR: δ 0.940-1.261 (m, 6H), 1.535-1.614 (m, 2H), 1.688-1.757 (m, 2H), 2.239 (s, 3H), 3.538 (d, *J* = 6.0 Hz, 2H), 7.066 (s, 4H), 7.420-7.528 (m, 6H), 7.712-7.762 (m, 4H).

^{13}C-NMR: δ 19.26, 25.41, 26.34, 26.37, 29.77, 29.82, 31.97, 32.06, 95.85, 96.89, 105.70, 105.81, 125.80, 126.47, 128.19, 128.31, 129.93, 130.05, 131.60, 131.62, 131.72, 132.01, 133.04, 136.57, 137.35, 137.43, 204.88, 204.96 .

^{31}P-NMR: δ 30.230.

HR-MS (ESI): $C_{28}H_{29}NaOP$ (calcd.: 435.1854), found:435.1848 ($[M+Na]^+$)



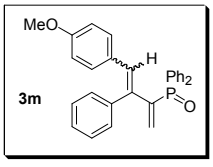
Viscous yellow liquid

^{1}H-NMR: δ 0.979-1.262 (m, 6H), 1.612-1.686 (m, 2H), 1.746-1.818 (m, 2H), 3.555 (d, *J* = 7.6 Hz, 2H), 7.118-7.213 (m, 5H), 7.383-7.487 (m, 6H), 7.691-7.741 (m, 4H).

^{13}C-NMR: δ 25.35, 26.24, 26.27, 29.71, 29.76, 34.16, 34.25, 96.61, 97.65, 105.60, 105.74, 126.10, 128.04, 128.14, 128.26, 129.17, 131.54, 131.58, 131.67, 131.85, 132.88, 139.01, 139.09, 205.25, 205.33.

^{31}P-NMR: δ 30.829

HR-MS (ESI): $C_{27}H_{27}NaOP$ (calcd.: 421.1697), found:421.1692($[M+Na]^+$)

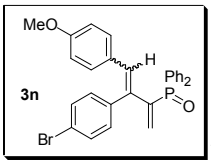


Viscous pale yellow liquid

^{1}H-NMR: δ 3.698 (s, 3H), 3.810 (s, 1H), 5.505 (d, *J* = 20 Hz, 1H), 5.702 (d, *J* = 41.6 Hz, 1H), 6.575 (d, *J* = 8.8 Hz, 2H), 6.694 (d, *J* = 8.8 Hz, 2H), 7.160-7.218 (m, 4H), 7.337-7.566 (m, 7H), 7.828-7.874 (m, 4H).

^{31}P-NMR: δ 32.243, 29.661

HR-MS (ESI): $C_{29}H_{25}NaO_2P$ (calcd.: 459.1490), found:459.1484 ($[M+Na]^+$)



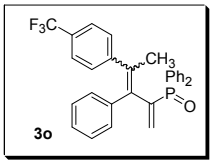
Viscous pale yellow liquid

^{1}H-NMR: δ 3.592-3.744 (m, 2H), 3.779 (s, 3H), 5.989-6.030 (m, 1H), 6.753 (d, *J* = 8.4 Hz, 2H), 6.855 (d, *J* = 8.4 Hz, 2H), 7.041 (d, *J* = 8.4 Hz, 2H), 7.262-7.325 (m, 4H), 7.371-7.412 (m, 2H), 7.469-7.518 (m, 2H), 7.639-7.736 (m, 4H).

^{13}C-NMR: δ 34.08, 34.16, 55.34, 97.65, 97.78, 102.23, 103.22, 114.18, 120.42, 123.98, 124.05, 126.78, 127.97, 127.99, 128.24, 128.33, 128.36, 128.46, 128.60, 130.99, 131.35, 131.48, 131.52, 131.58, 131.62, 131.67, 131.76, 131.82, 131.85, 131.87, 131.92, 131.94, 132.02, 132.05, 132.30, 137.52, 137.58, 159.33, 210.55, 210.61.

^{31}P-NMR: δ 29.5176, 29.698.

HR-MS (ESI): $C_{29}H_{24}BrNaO_2P$ (calcd.: 537.0595), found:537.0590 ([M+Na]$^{+}$)



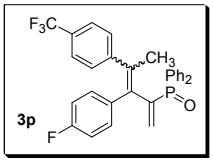
Viscous yellow liquid

^{1}H-NMR: δ 1.701 (d, *J* = 6.0 Hz, 3H), 3.714 (d, *J* = 8.8 Hz, 2H), 6.944 (d, *J* = 8.4 Hz, 2H), 7.118-7.211 (m, 5H), 7.290-7.335 (m, 2H), 7.378-7.521 (m, 6H), 7.619-7.708 (m, 4H).

^{13}C-NMR: δ 14.67, 14.73, 33.15, 33.23, 100.67, 101.65, 103.05, 103.19, 124.04, 124.07, 124.57, 124.59, 125.53, 127.15, 127.27, 127.30, 127.40, 128.00, 129.89, 129.99, 130.27, 130.37, 130.40, 130.49, 130.87, 130.90, 130.93, 131.03, 137.30, 137.37, 138.16, 138.23, 209.57, 209.64.

^{31}P-NMR: δ 30.482

HR-MS (ESI): $C_{30}H_{24}F_3NaOP$ (calcd.: 511.1415), found:511.1409([M+Na]$^{+}$)



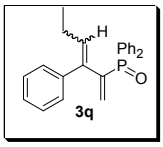
Viscous pale yellow liquid

^{1}H-NMR: δ 1.767 (d, *J* = 6.0 Hz, 3H), 3.736 (d, *J* = 8.4 Hz, 2H), 6.913 (t, J= 8.8 Hz, 2H), 7.028 (d, J = 8.4 Hz, 2H), 7.145-7.180 (m, 2H), 7.336-7.381 (m, 2H), 7.428-7.572 (m, 6H), 7.657-7.747 (m, 4H).

^{13}C-NMR: δ 15.84, 15.90, 33.67, 33.74, 101.86, 102.84, 104.30, 104.44, 115.12, 115.33, 122.85, 125.28, 125.31, 125.56, 125.72, 125.74, 128.34, 128.46, 128.49, 128.61, 129.17, 129.50, 130.64, 130.72, 131.01, 131.09, 131.40, 131.50, 131.53, 131.62, 132.06, 132.09, 132.13, 132.16, 134.21, 134.24, 134.27, 134.31, 139.15, 139.22, 160.53, 162.96, 210.58, 210.64.

^{31}P-NMR: δ 30.310

HR-MS (ESI): $C_{30}H_{23}F_4NaOP$ (calcd.: 529.1320), found:529.1315([M+Na]$^{+}$)



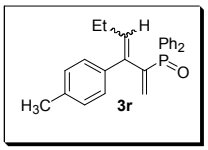
Viscous pale yellow liquid

^{1}H-NMR: δ 0.656 (t, *J* = 7.6 Hz, 3H), 1.739-1.813 (m, 2H), 5.380 (d, *J* = 20.4 Hz, 1H), 5.523 (d, *J* = 42.0 Hz, 1H), 6.502 (t, *J* = 7.6 Hz, 1H), 7.096 (d, *J* = 6.8 Hz, 2H), 7.215-7.330 (m, 3H), 7.417-7.500 (m, 6H), 7.773-7.819 (m, 4H).

^{13}C-NMR: δ 13.55, 23.25, 127.05, 128.28, 128.35, 128.47, 129.39, 131.70, 131.73, 131.76, 131.79, 131.86, 132.79, 137.30, 137.40, 138.42, 138.49, 138.58, 138.63, 143.98, 144.89.

^{31}P-NMR: δ 32.172, 36.036

HR-MS (ESI): $C_{24}H_{23}NaOP$ (calcd.: 381.1384), found:381.1379 ($[M+Na]^+$)



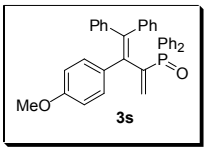
Viscous pale yellow liquid

^{1}H-NMR: δ 0.653 (t, *J* = 7.6 Hz, 3H), 1.739-1.814 (m, 2H), 2.337 (s, 3H), 5.362 (d, *J* = 20.4 Hz, 3H), 5.537 (d, *J* = 42.4 Hz, 3H), 6.376-6.416 (m, 1H), 6.955 (d, *J* = 8.0 Hz, 2H), 7.112 (d, *J* = 8.0 Hz, 2H), 7.433-7.536 (m, 6H),7.731-7.781(m, 4H).

^{13}C-NMR: δ 13.60, 21.26, 23.32, 29.71, 29.75, 30.25, 31.50, 128.39, 128.51, 128.56, 129.00, 129.38, 131.73, 131.76, 131.81, 131.90, 132.01, 132.80, 135.45, 135.53, 136.65, 137.21, 137.31, 138.60, 138.64, 144.05, 144.97.

^{31}P-NMR: δ 32.112, 29.468.

HR-MS (ESI): $C_{25}H_{26}OP$ (calcd.: 373.1721), found:373.1716 ($[M+H]^+$)



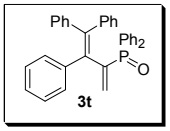
Viscous pale yellow liquid

^{1}H-NMR: δ 3.697 (d, *J* = 7.6 Hz, 2H), 3.718 (s, 3H), 6.681-6.730 (m, 6H), 7.040 (d, *J* = 8.8 Hz, 2H), 7.176-7.334 (m, 6H), 7.409-7.449 (m, 6H), 7.613-7.661 (m, 4H).

^{13}C-NMR: δ 33.88, 33.96, 55.45, 102.26, 103.24, 113.88, 127.89, 128.17, 128.20, 128.44, 128.56, 130.38, 130.46, 130.56, 131.31, 131.70, 131.80, 132.05, 132.08, 132.35, 135.16, 135.22, 158.52, 210.10, 210.16.

^{31}P-NMR: δ 29.838

HR-MS (ESI): $C_{35}H_{29}NaO_2P$ (calcd.: 535.1803), found:535.1800 ($[M+Na]^+$)



Pale yellow solids, **m.p.**: 95.2-97.6 °C.

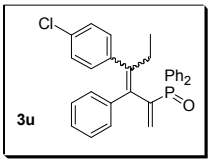
^{1}H-NMR: δ 3.737 (d, *J* = 7.6 Hz, 2H), 6.639-6.660 (m, 4H), 7.107-7.227 (m, 10H), 7.303-7.348 (m, 4H), 7.428-7.469 (m, 3H), 7.614-7.664 (m, 4H).

^{13}C-NMR: δ 34.52, 34.60, 101.80, 102.79, 113.65, 113.79, 126.68, 127.78, 128.03, 128.05,

128.13, 128.31, 128.35, 128.38, 128.47, 128.54, 129.46, 131.07, 131.60, 131.70, 131.99, 132.01, 132.11, 134.96, 135.02, 138.18, 138.25, 210.03, 210.09.

^{31}P-NMR: δ 29.803

HR-MS (ESI): $C_{34}H_{27}NaOP$ (calcd.: 505.1697), found:505.1692 ($[M+Na]^+$)



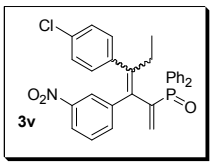
Viscous pale yellow liquid

^{1}H-NMR: δ 0.689 (t, *J* = 7.6 Hz, 3H), 1.836-2.008 (m, 2H), 3.690 (d, *J* = 7.6 Hz, 2H), 6.767 (d, *J* = 8.0 Hz, 2H), 7.145-7.220 (m, 6H), 7.310-7.515 (m, 7H), 7.613-7.685 (m, 4H).

^{13}C-NMR: δ 12.12, 12.14, 23.09, 23.15, 34.30, 34.38, 102.79, 103.78, 111.14, 111.28, 126.51, 127.07, 127.09, 128.09, 128.19, 128.23, 128.27, 128.32, 128.35, 128.41, 129.27, 130.98, 131.11, 131.36, 131.46, 131.53, 131.63, 131.82, 131.85, 131.88, 132.02, 132.14, 132.96, 132.97, 133.75, 133.81, 138.40, 138.47, 209.82, 209.89.

^{31}P-NMR: δ 32.796.

HR-MS (ESI): $C_{30}H_{27}ClOP$ (calcd.: 469.1488), found:469.1483 ($[M+H]^+$)



Viscous pale yellow liquid

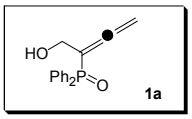
^{1}H-NMR: δ 0.713 (t, *J* = 7.2 Hz, 3H), 1.099-2.094 (m, 2H), 3.806 (d, *J* = 8.0 Hz, 2H), 6.830 (d, *J* = 7.6 Hz, 2H), 7.200 (d, *J* = 7.2 Hz, 2H), 7.404-7.477 (m, 5H), 7.614-7.697 (m, 7H), 8.004 (d, *J* = 13.2 Hz, 2H).

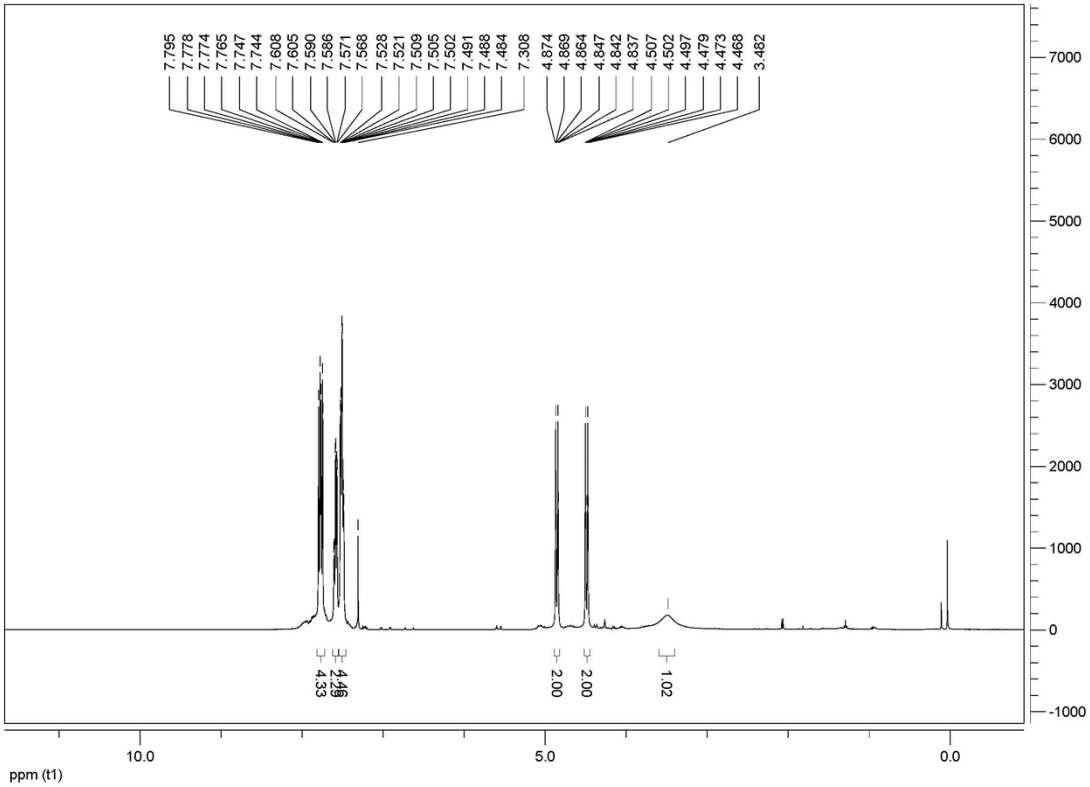
^{13}C-NMR: δ 12.27, 23.19, 23.24, 29.75, 34.24, 34.32, 101.99, 102.99, 111.77, 111.90, 121.75, 124.15, 127.04, 127.06, 128.43, 128.52, 128.55, 128.64, 128.73, 129.28, 131.35, 131.45, 131.53, 131.62, 132.03, 132.06, 132.09, 132.19, 133.52, 135.63, 140.51, 140.57, 148.10, 209.58, 209.643.

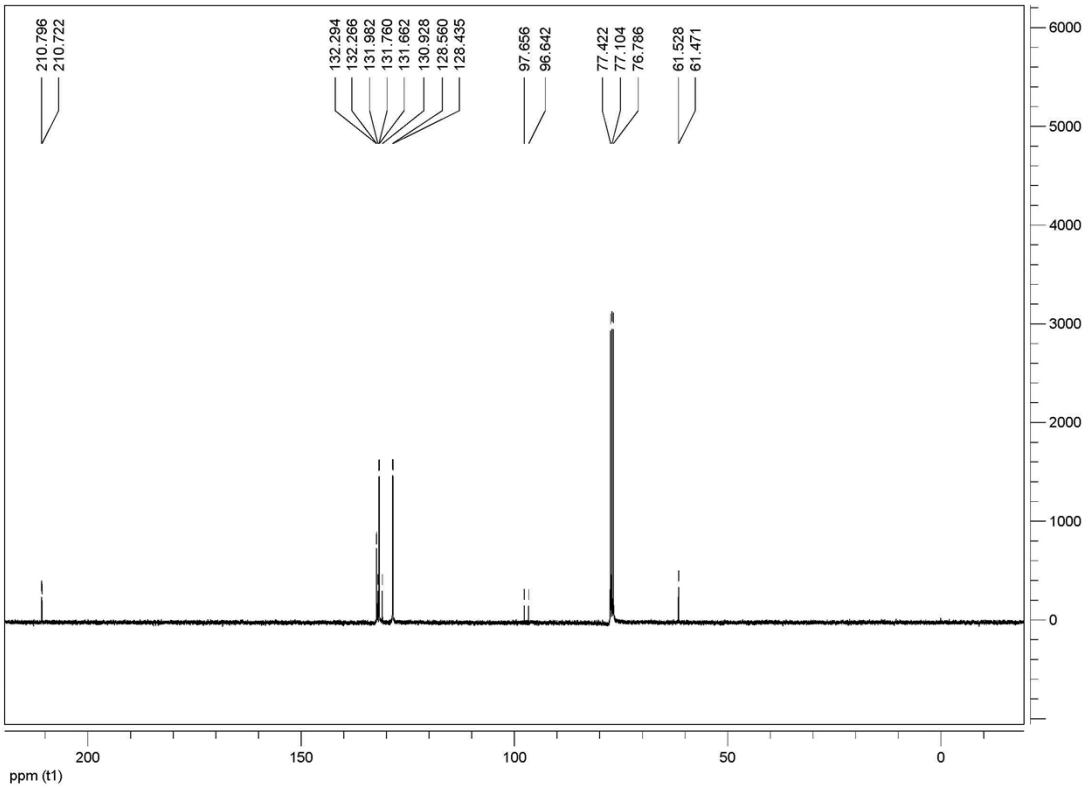
^{31}P-NMR: 30.030, 29.596.

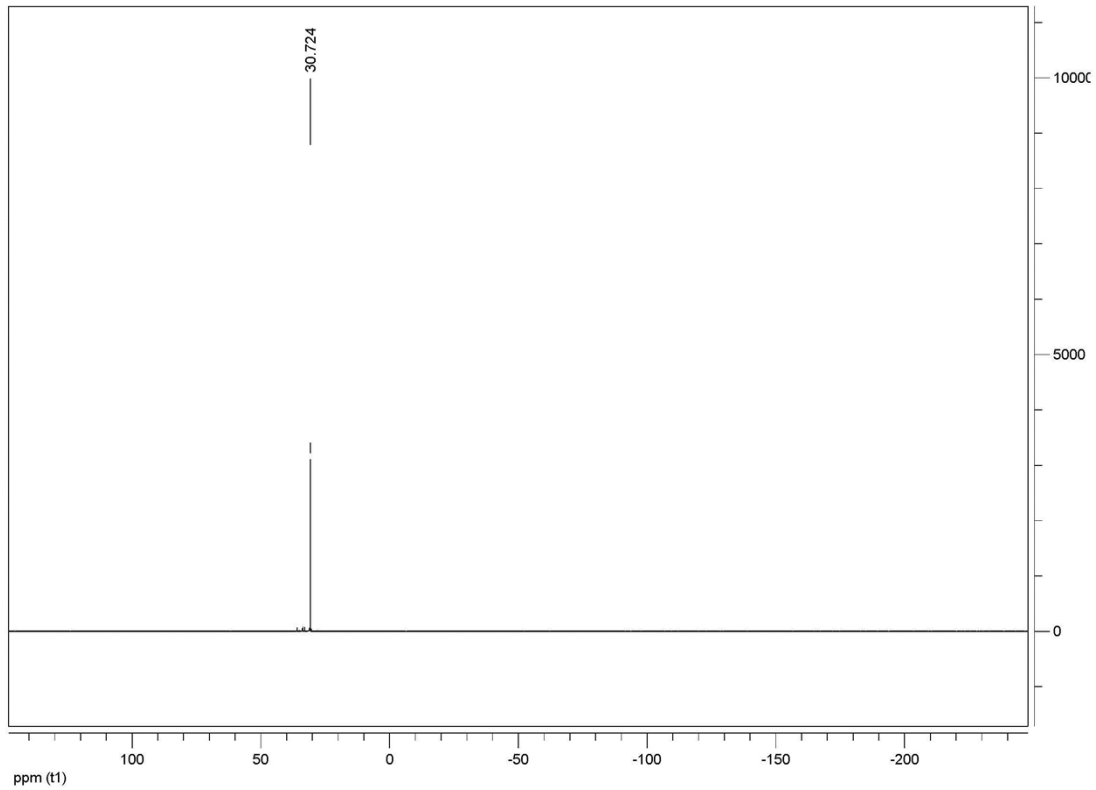
HR-MS (ESI): $C_{30}H_{26}ClNO_3P$ (calcd.: 514.1339), found:514.1333 ($[M+H]^+$)

5. Selected Spectra of Substrates and Products





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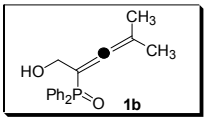
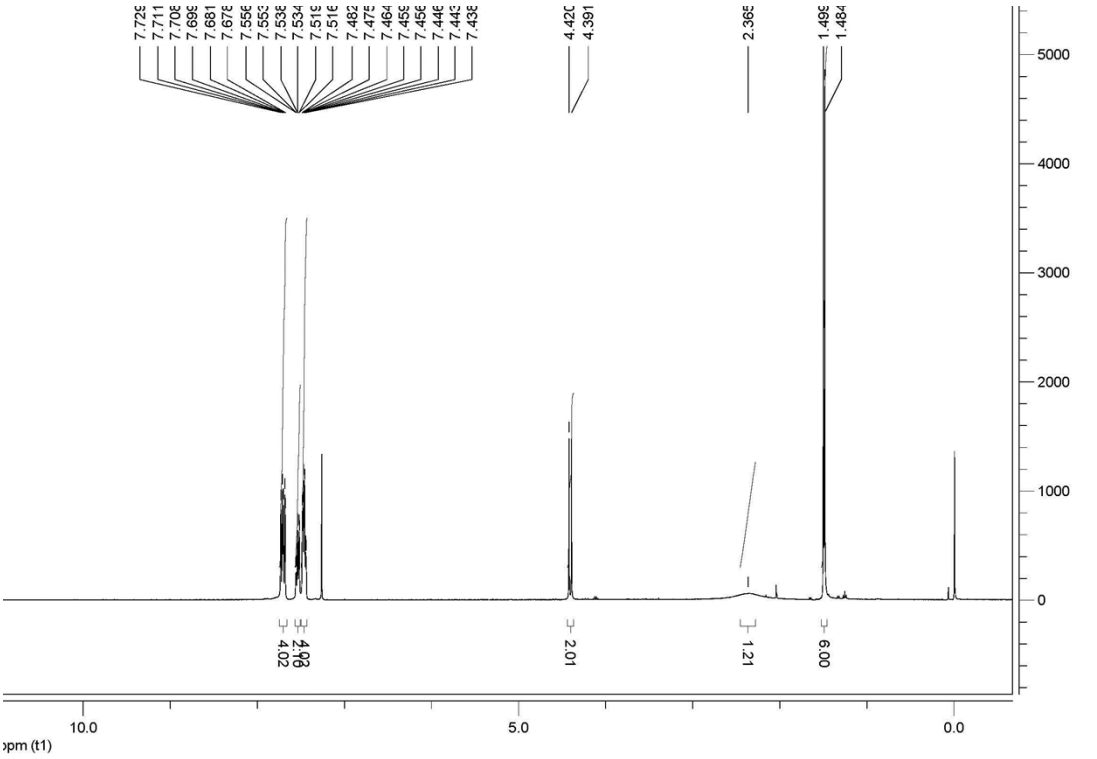
100
50
0
-50
-100
-150
-200
ppm (t1)


CH3
CH3
HO
Ph2P=O
1b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7.708
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7.678
7.556
7.553
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7.534
7.516
7.482
7.475
7.464
7.458
7.456
7.446
7.443
7.438
4.420
4.391
2.365
1.498
1.484
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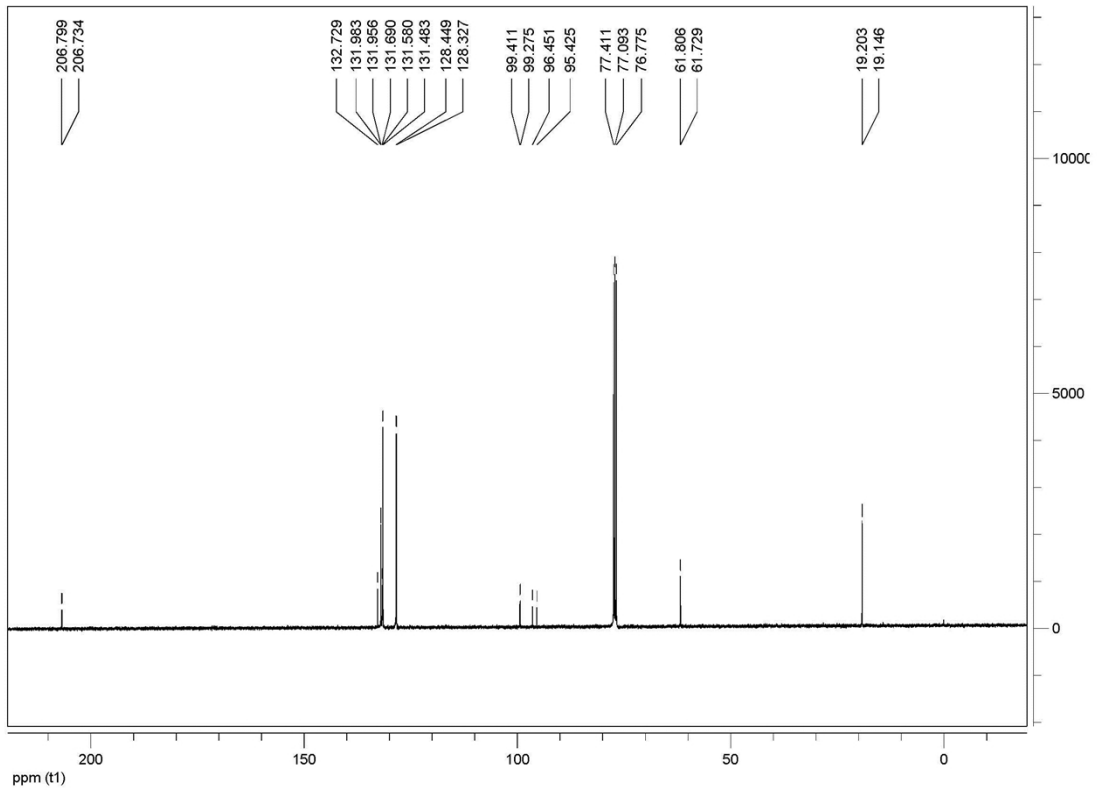
4000
3000
2000
1000
0
4.02
2.01
1.21
6.00
10.0
5.0
0.0
ppm (t1)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206.734
132.729
131.983
131.956
131.690
131.580
131.483
128.449
128.327
99.411
99.275
96.451
95.425
77.411
77.093
76.775
61.806
61.729
19.203
19.146
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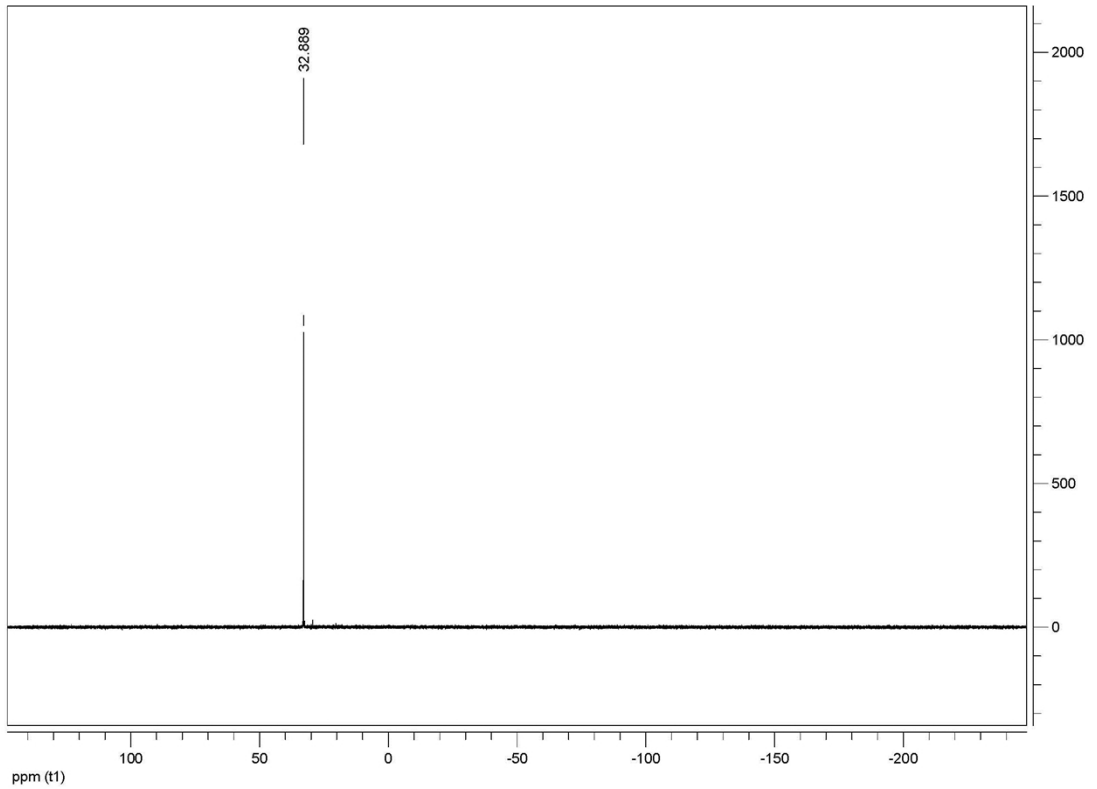
150
100
50
0
ppm (t1)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1500
1000
500
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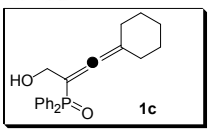
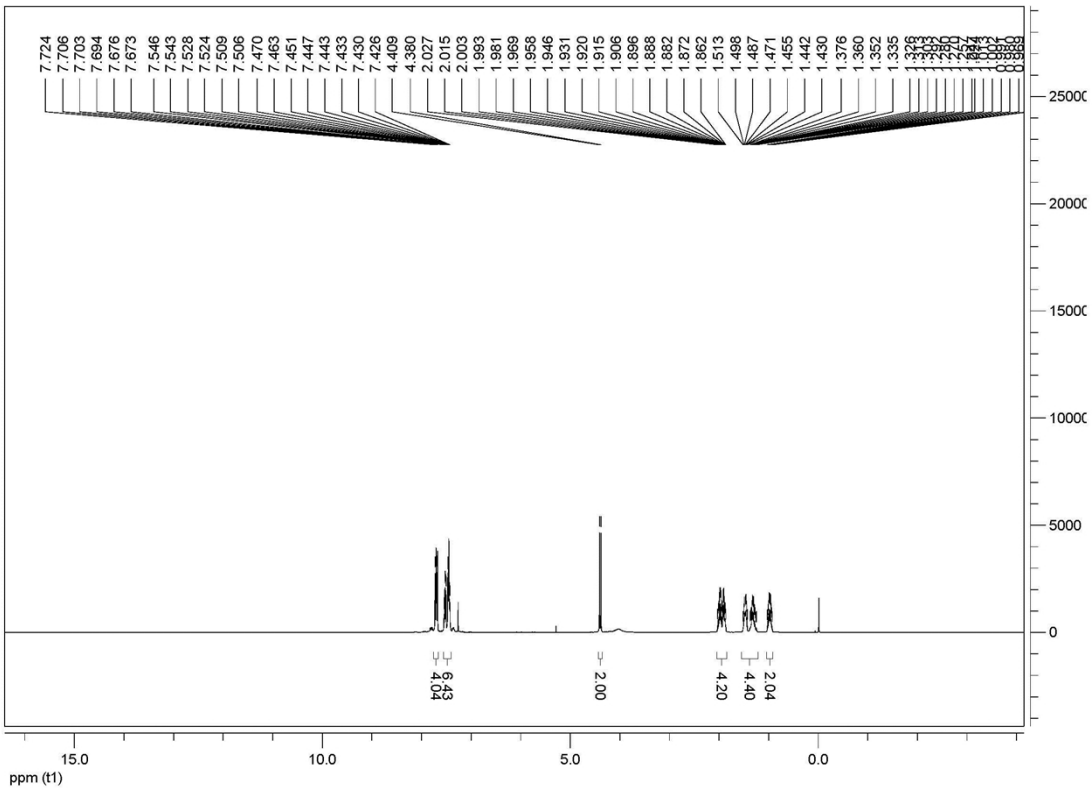
100
50
0
-50
-100
-150
-200
ppm (t1)


HO
Ph2P
O
1c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7.543
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7.470
7.463
7.451
7.447
7.443
7.433
7.430
7.426
4.409
4.380
2.027
2.015
2.003
1.993
1.981
1.969
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1.920
1.915
1.906
1.896
1.888
1.882
1.872
1.862
1.513
1.498
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1.430
1.376
1.360
1.352
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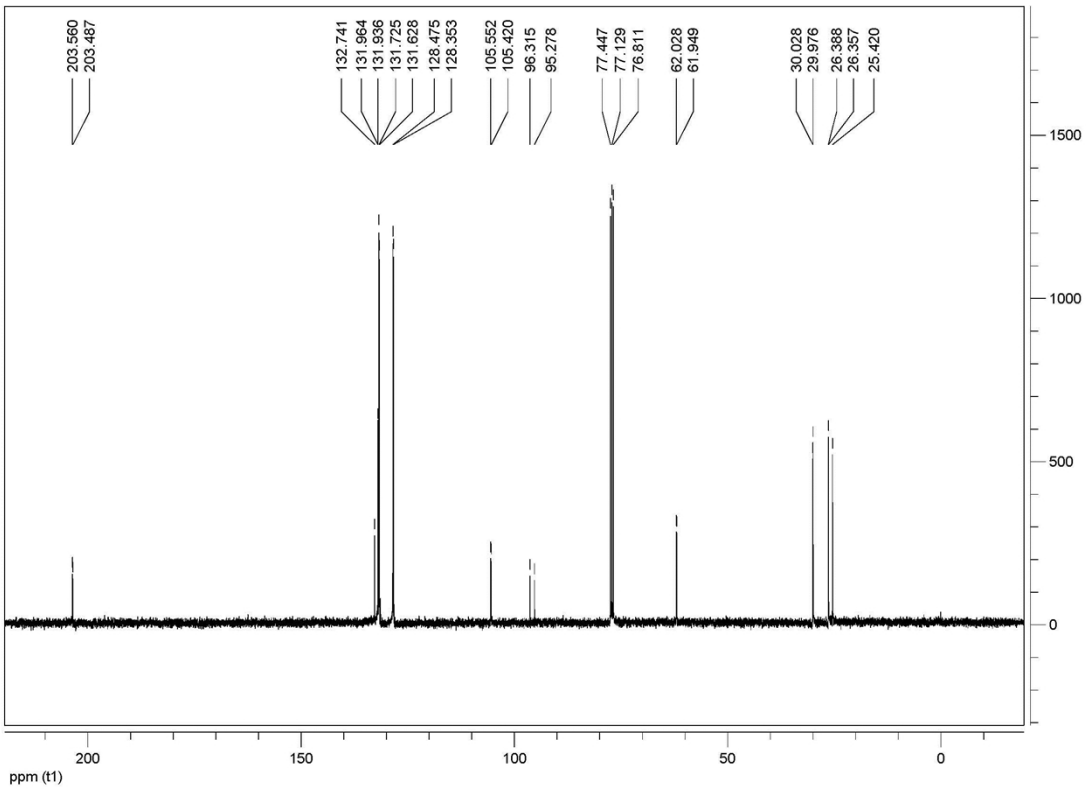
ppm (t1)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105.552
105.420
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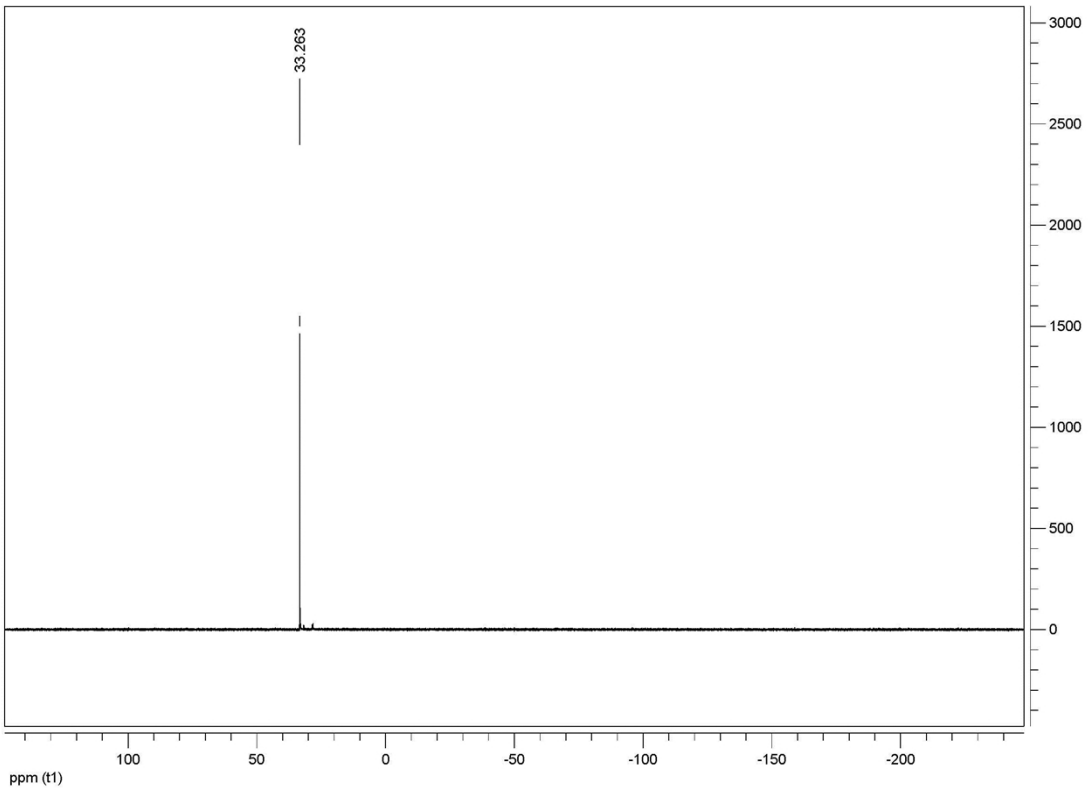
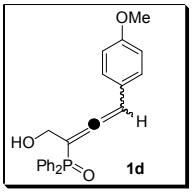
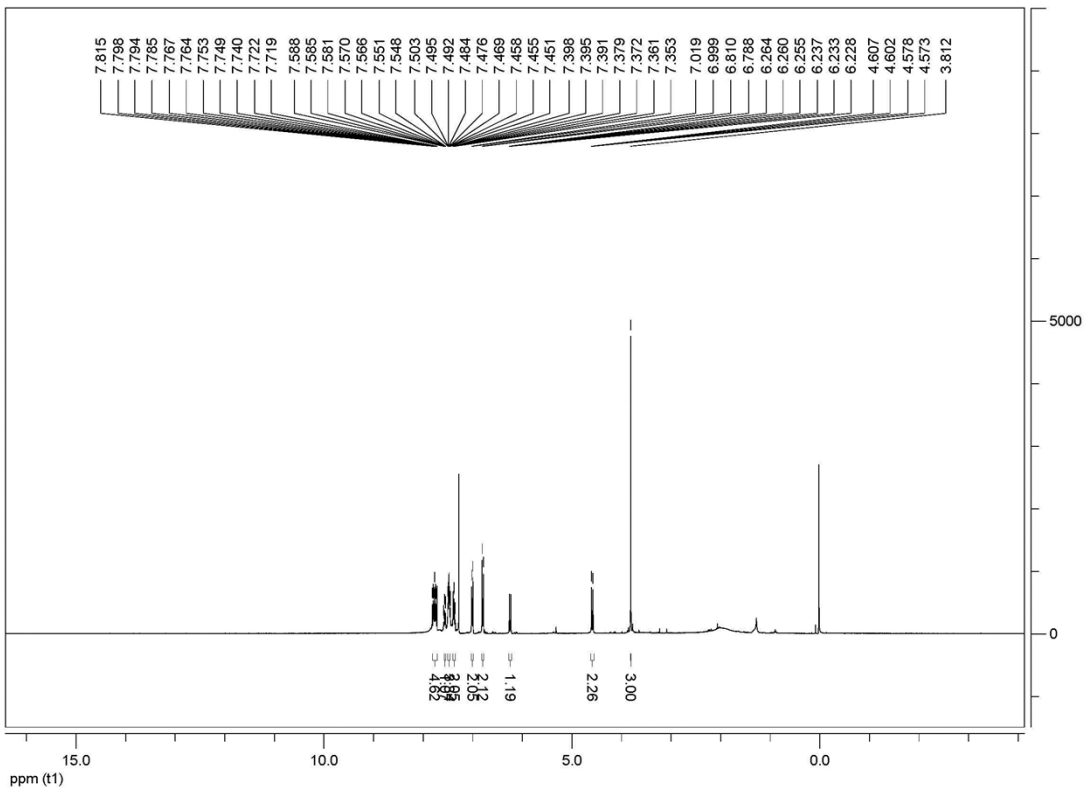
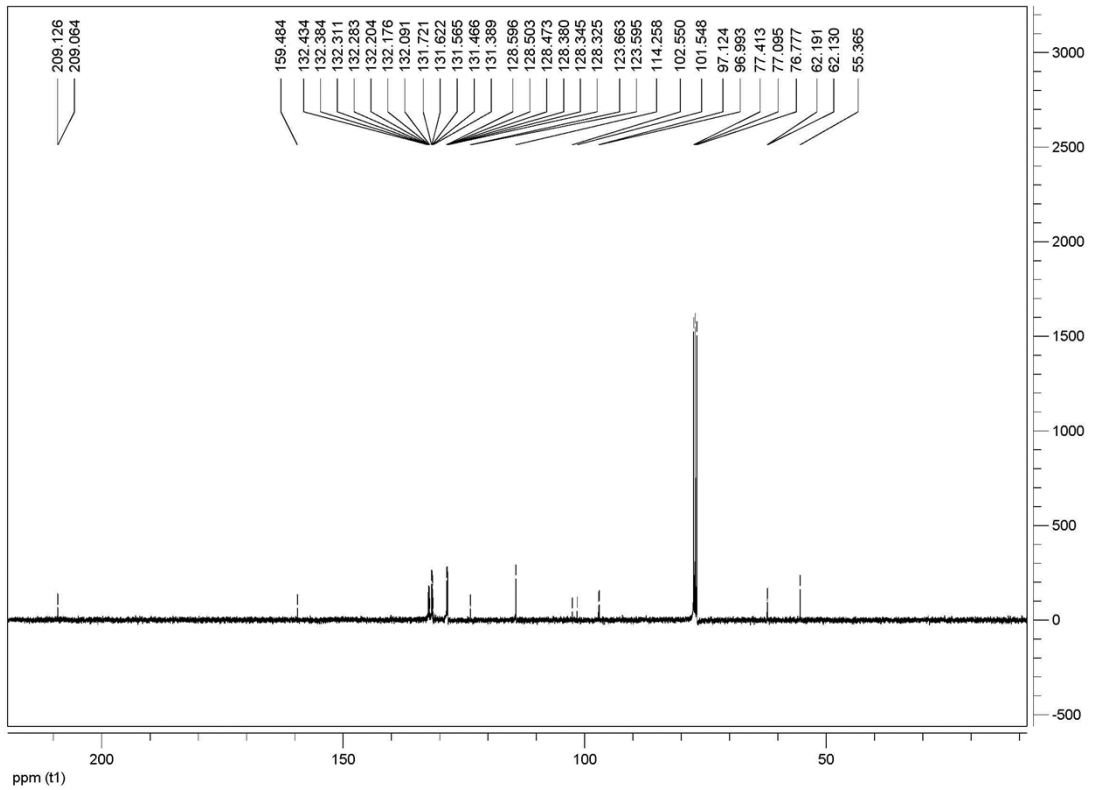
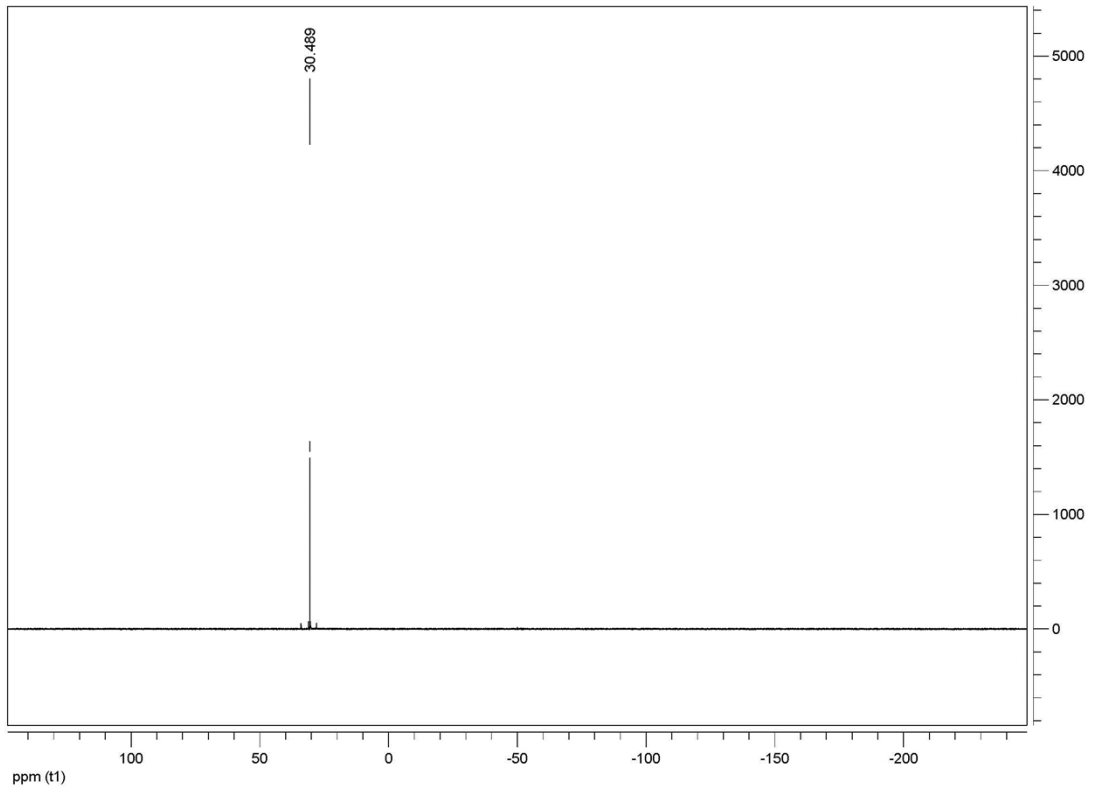
77.447
77.129
76.811
62.028
61.949
30.028
29.976
26.388
26.357
25.420
200
150
100
50
0
ppm (t1)

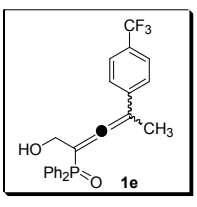

33.263
3000
2500
2000
1500
1000
500
0
100
50
0
-50
-100
-150
-200
ppm (t1)

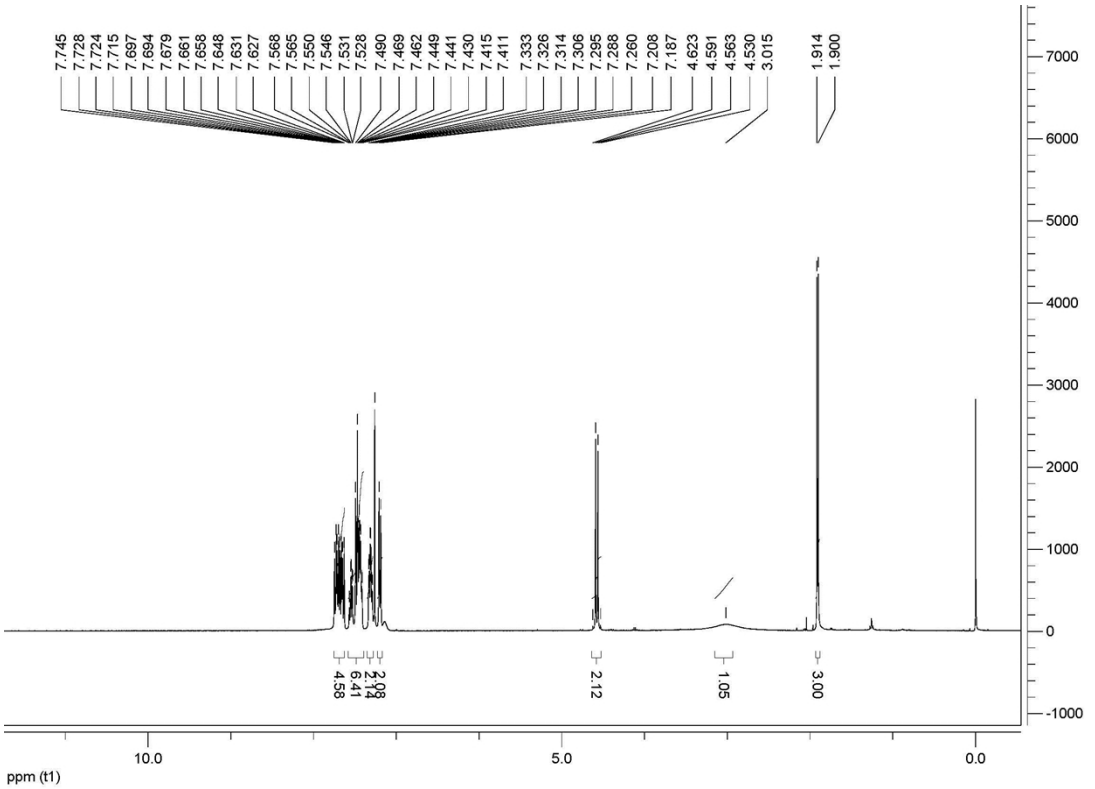

OMe
H
HO
Ph2P
O
1d

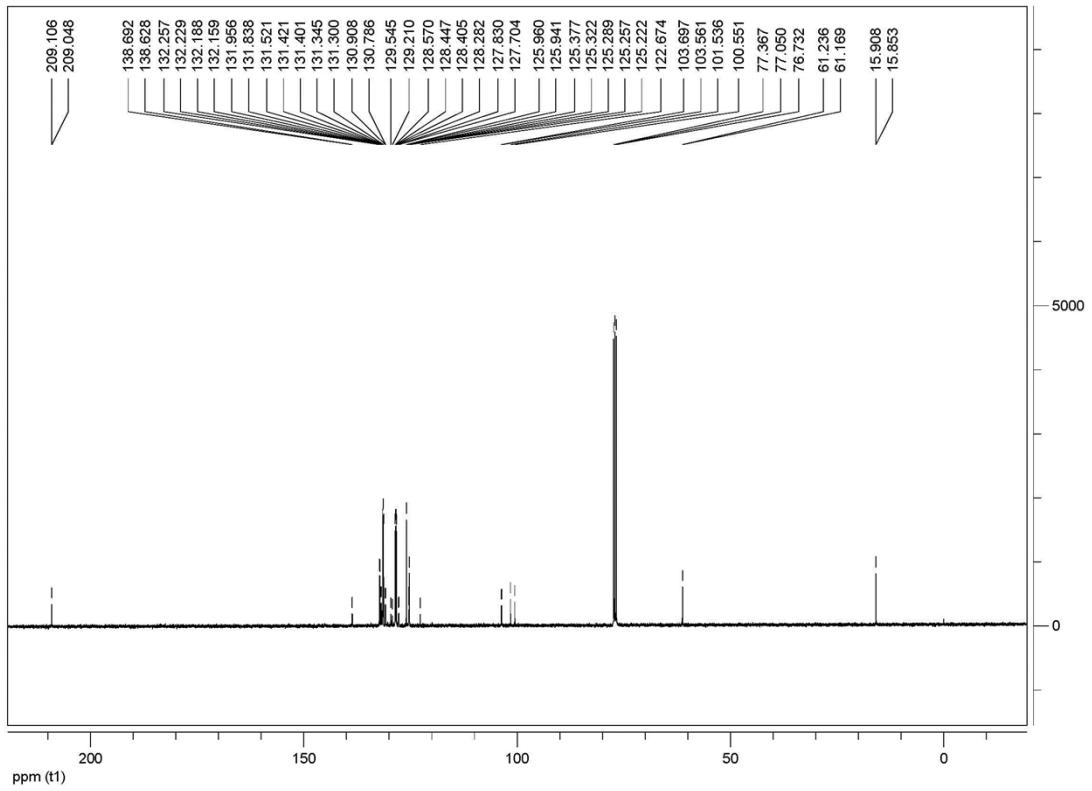

7.815
7.798
7.794
7.785
7.767
7.764
7.753
7.749
7.740
7.722
7.719
7.588
7.585
7.581
7.570
7.566
7.551
7.548
7.503
7.495
7.492
7.484
7.476
7.469
7.458
7.455
7.451
7.398
7.395
7.391
7.379
7.372
7.361
7.353
7.019
6.999
6.810
6.788
6.264
6.260
6.255
6.237
6.233
6.228
4.607
4.602
4.578
4.573
3.812
5000
0
1.19
2.26
3.00
15.0
10.0
5.0
0.0
ppm (t1)

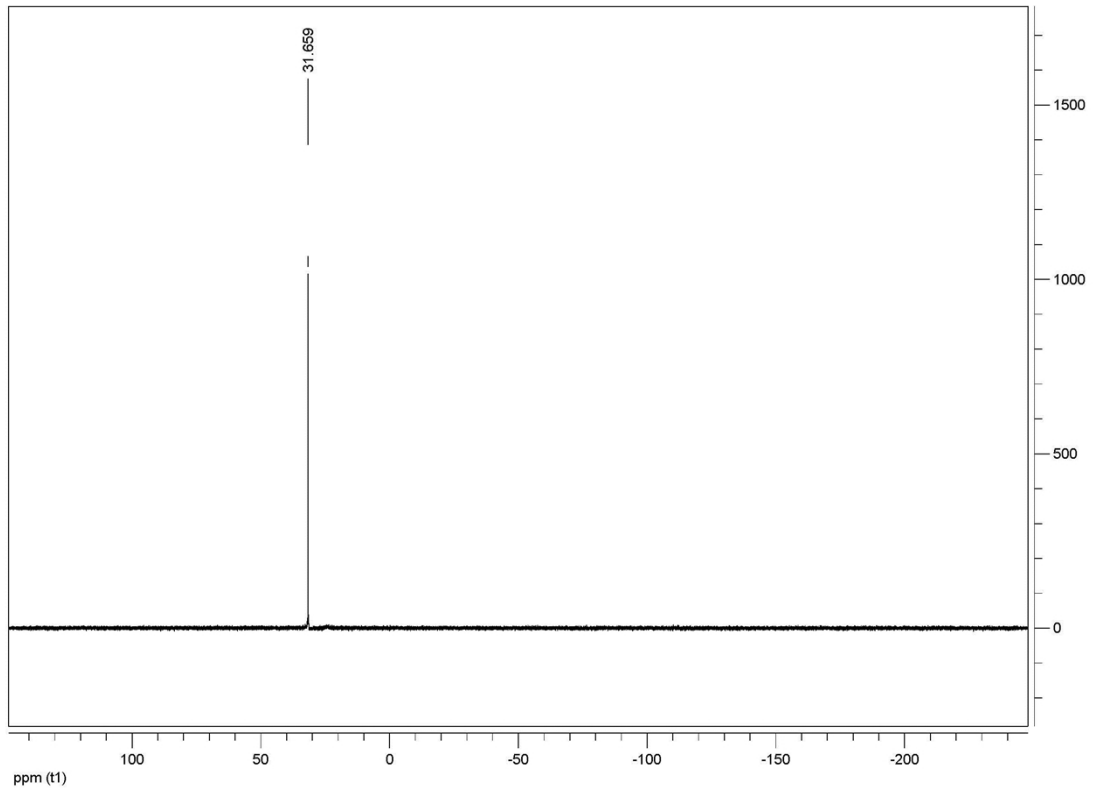

209.126
209.064
159.484
132.434
132.384
132.311
132.283
132.204
132.176
132.091
131.721
131.622
131.565
131.466
131.389
128.596
128.503
128.473
128.380
128.345
128.325
123.663
123.595
114.258
102.550
101.548
97.124
96.993
77.413
77.095
76.777
62.191
62.130
55.365
3000
2500
2000
1500
1000
500
0
-500
200
150
100
50
ppm (t1)

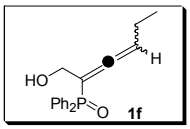

30.489
5000
4000
3000
2000
1000
0
100
50
0
-50
-100
-150
-200
ppm (t1)

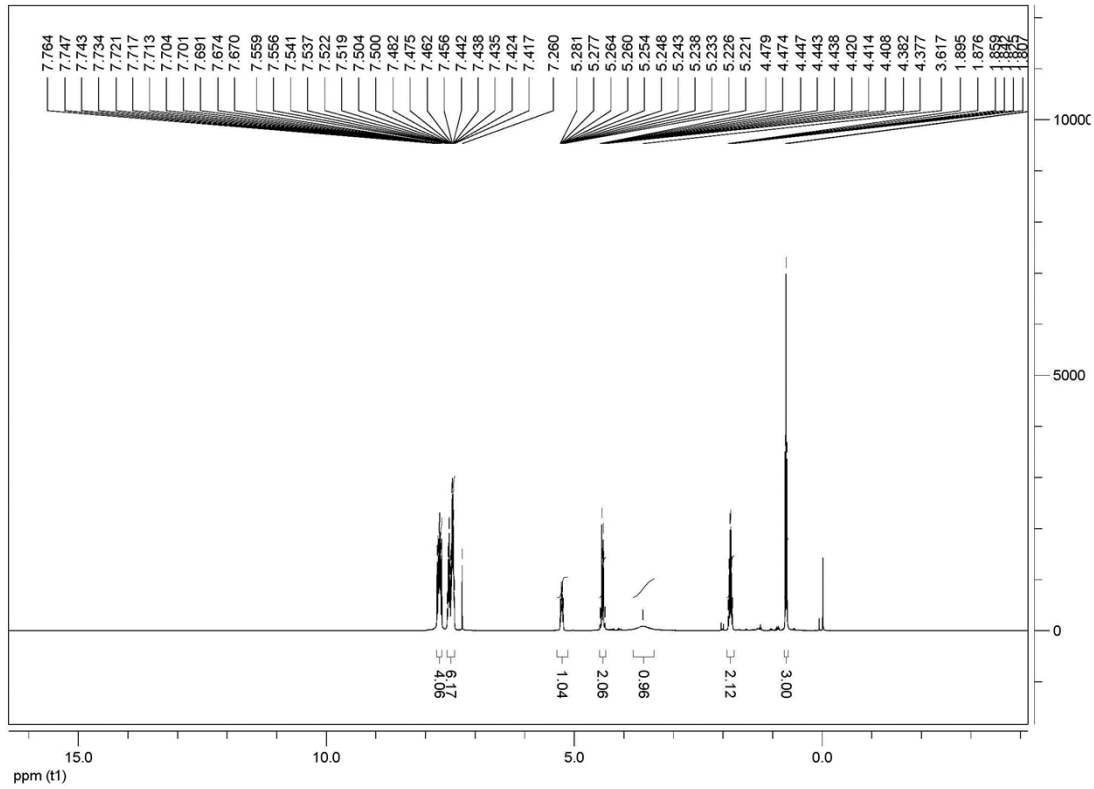

CF3
CH3
HO
Ph2P
O
1e

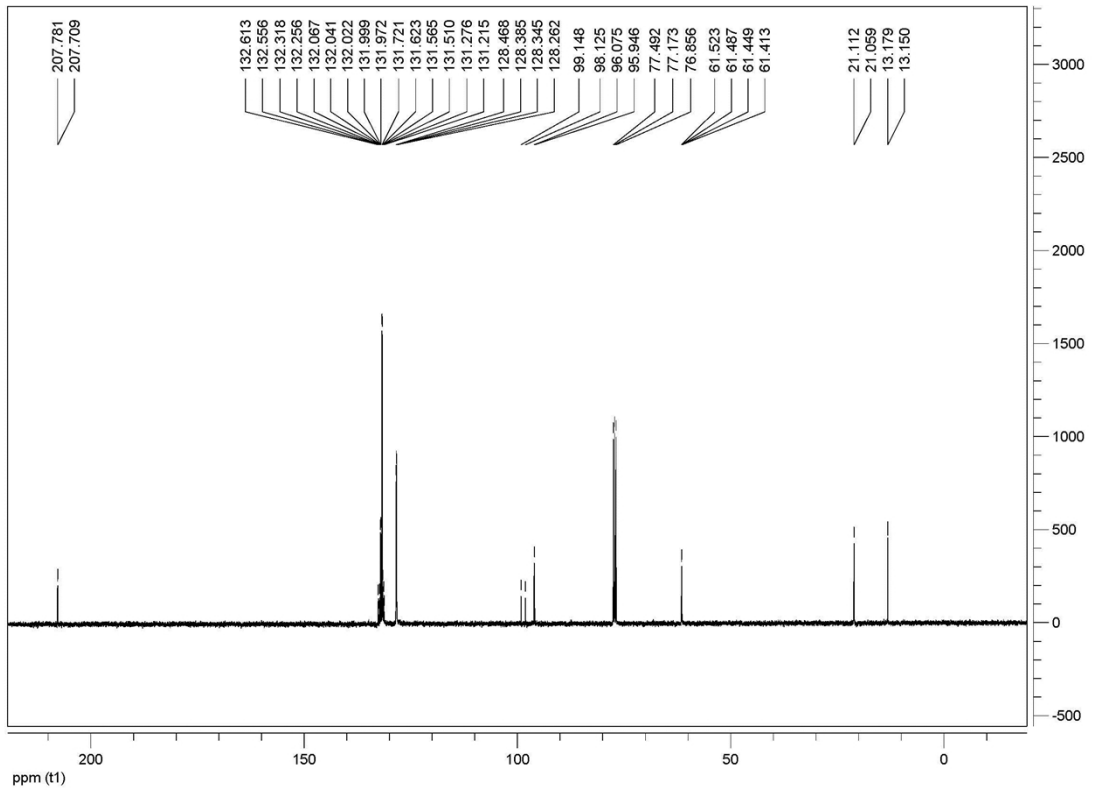

7.745
7.728
7.724
7.715
7.697
7.694
7.679
7.661
7.658
7.648
7.631
7.627
7.568
7.565
7.550
7.546
7.531
7.528
7.490
7.469
7.462
7.449
7.441
7.430
7.415
7.411
7.333
7.326
7.314
7.306
7.295
7.288
7.260
7.208
7.187
4.623
4.591
4.563
4.530
3.015
1.914
1.900
4.58
6.41
2.12
1.05
3.00
ppm (t1)

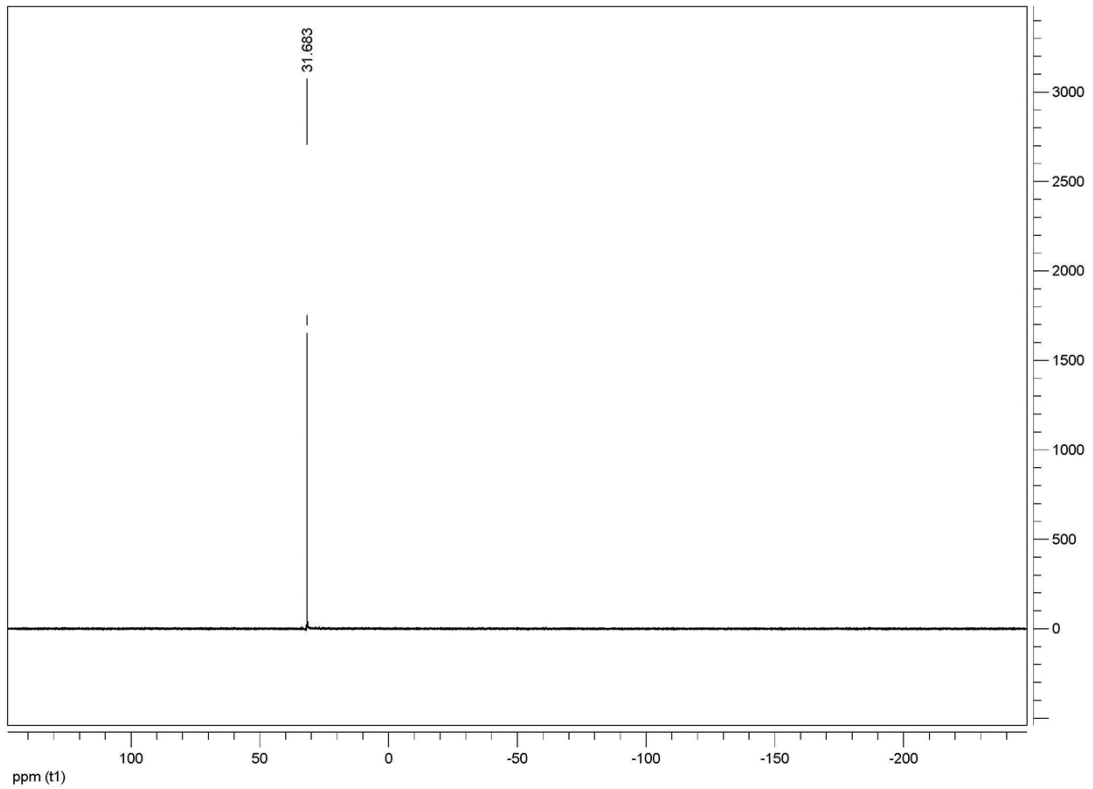

209.106
209.048
138.692
138.628
132.257
132.229
132.188
132.159
131.956
131.838
131.521
131.421
131.401
131.345
131.300
130.908
130.786
129.545
129.210
128.570
128.447
128.405
128.282
127.830
127.704
125.960
125.941
125.377
125.322
125.289
125.257
125.222
122.674
103.697
103.561
101.536
100.551
77.367
77.050
76.732
61.236
61.169
15.908
15.853
ppm (t1)

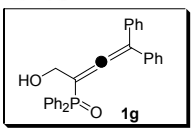

31.659
1500
1000
500
0
100
50
0
-50
-100
-150
-200
ppm (t1)

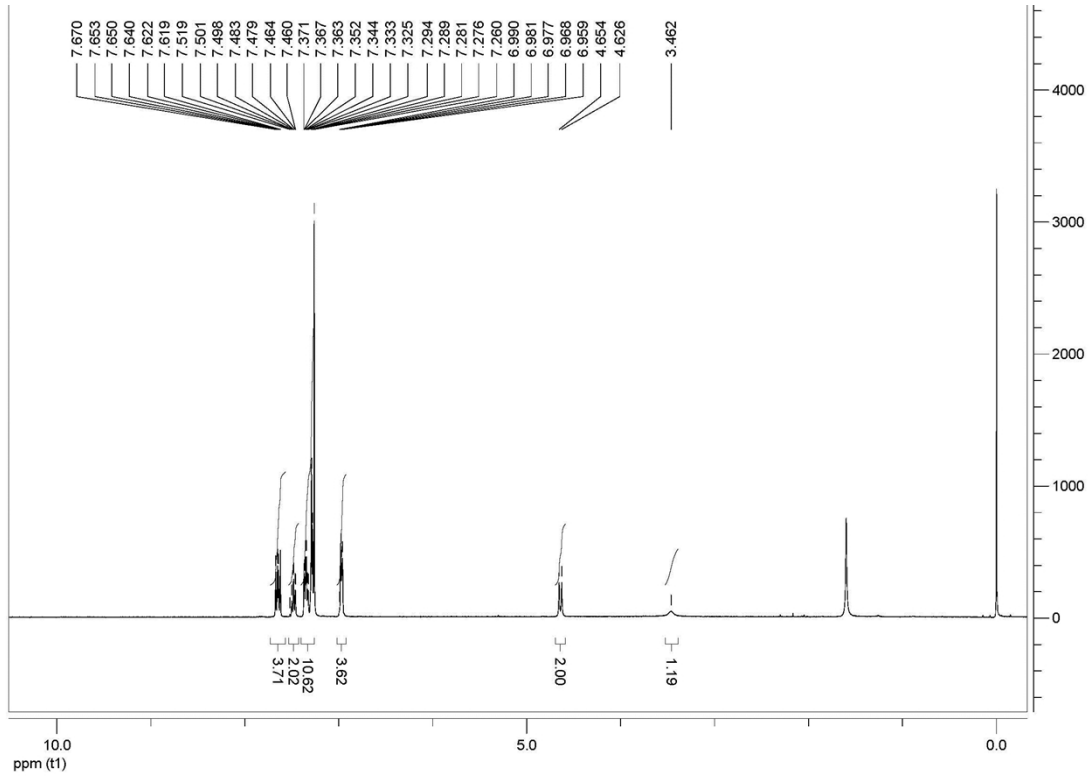

HO
H
Ph2P
O
1f

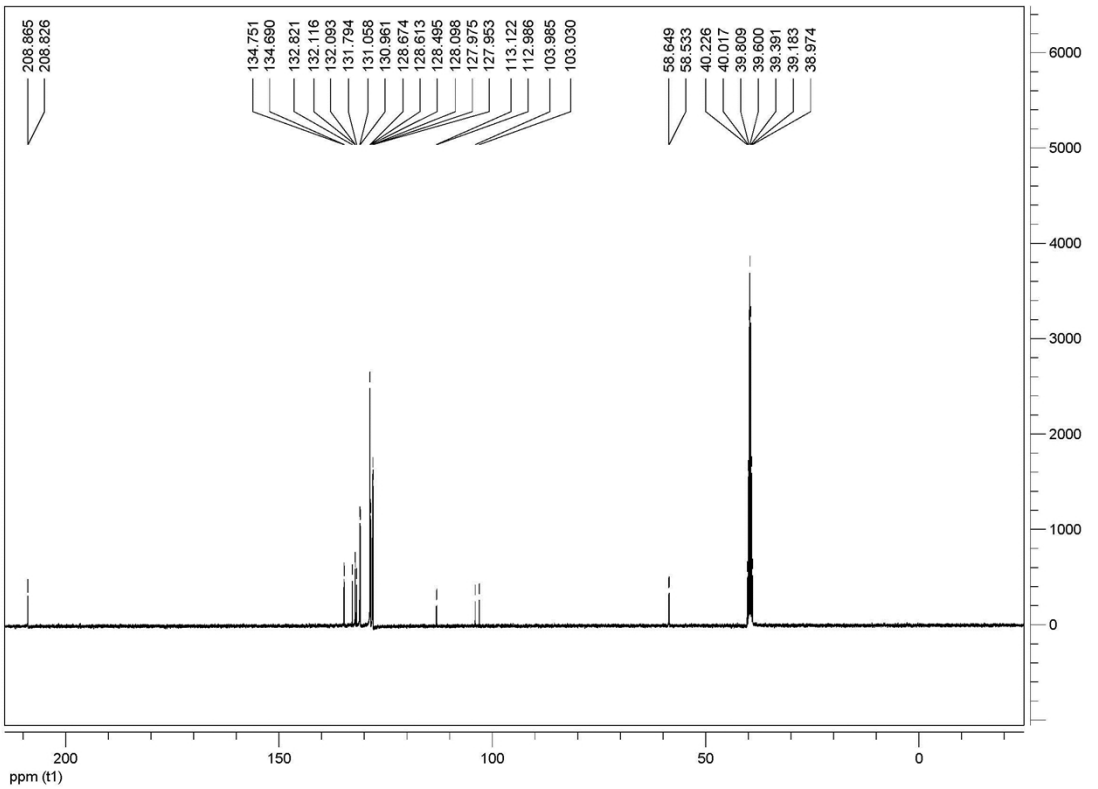

7.764
7.747
7.743
7.734
7.721
7.717
7.713
7.704
7.701
7.691
7.674
7.670
7.559
7.556
7.541
7.537
7.522
7.519
7.504
7.500
7.482
7.475
7.462
7.456
7.442
7.438
7.435
7.424
7.417
7.260
5.281
5.277
5.264
5.260
5.254
5.248
5.243
5.238
5.233
5.226
5.221
4.479
4.474
4.447
4.443
4.438
4.420
4.414
4.408
4.382
4.377
3.617
1.895
1.876
10000
5000
0
4.06
6.17
1.04
2.06
0.96
2.12
3.00
15.0
10.0
5.0
0.0
ppm (t1)

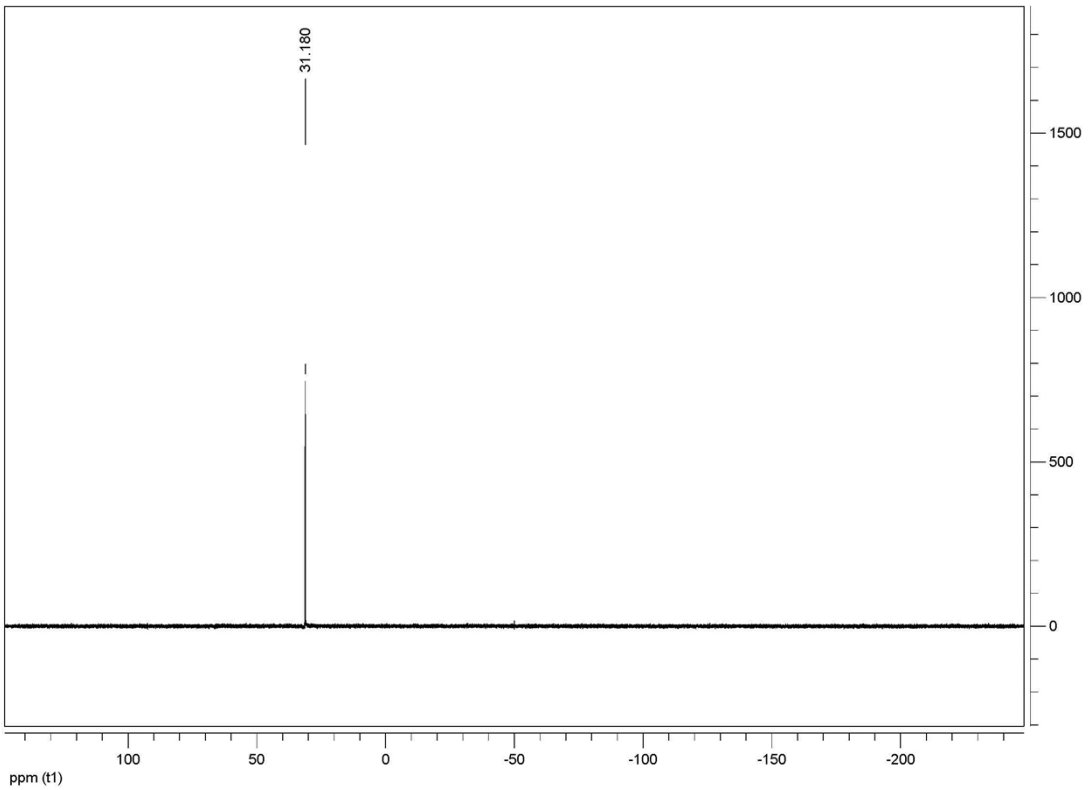

207.781
207.709
132.613
132.556
132.318
132.256
132.067
132.041
132.022
131.999
131.972
131.721
131.623
131.565
131.510
131.276
131.215
128.468
128.385
128.345
128.262
99.148
98.125
96.075
95.946
77.492
77.173
76.856
61.523
61.487
61.449
61.413
21.112
21.059
13.179
13.150
3000
2500
2000
1500
1000
500
0
-500
200
150
100
50
0
ppm (t1)

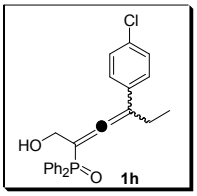

31.683
3000
2500
2000
1500
1000
500
0
100
50
0
-50
-100
-150
-200
ppm (t1)

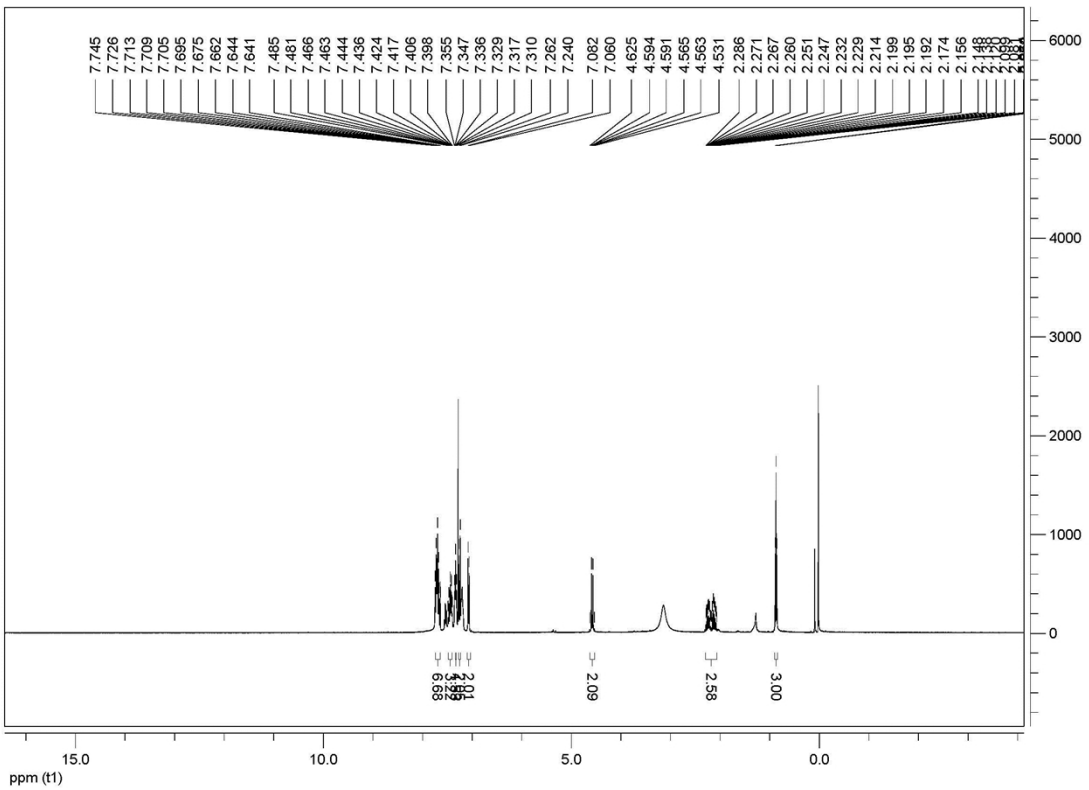

Ph
Ph
HO
Ph2P
O
1g

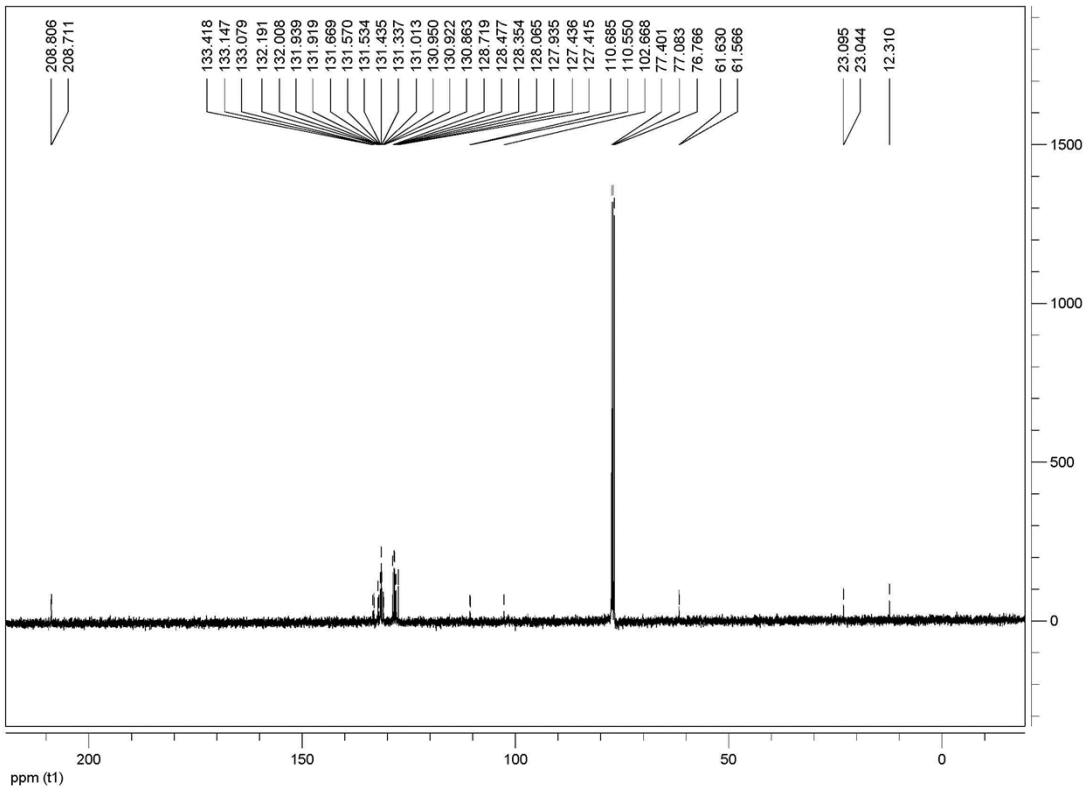

7.670
7.653
7.650
7.640
7.622
7.619
7.519
7.501
7.498
7.483
7.479
7.464
7.460
7.371
7.367
7.363
7.352
7.344
7.333
7.325
7.294
7.289
7.281
7.276
7.260
6.990
6.981
6.977
6.968
6.959
4.654
4.626
3.462
3.71
2.02
10.62
3.62
2.00
1.19
4000
3000
2000
1000
0
10.0
5.0
0.0
ppm (t1)

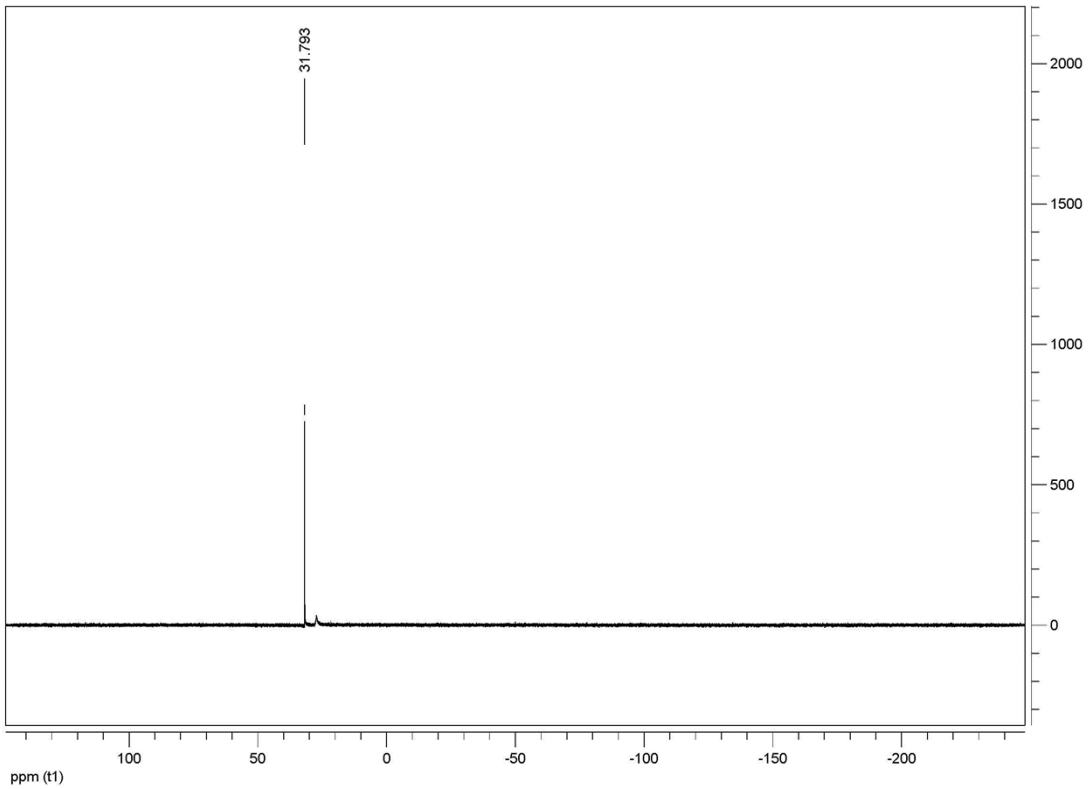

208.865
208.826
134.751
134.690
132.821
132.116
132.093
131.794
131.058
130.961
128.674
128.613
128.495
128.098
127.975
127.953
113.122
112.986
103.985
103.030
58.649
58.533
40.226
40.017
39.809
39.600
39.391
39.183
38.974
6000
5000
4000
3000
2000
1000
0
200
150
100
50
0
ppm (t1)

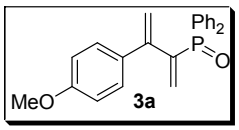

31.180
1500
1000
500
0
100
50
0
-50
-100
-150
-200
ppm (t1)

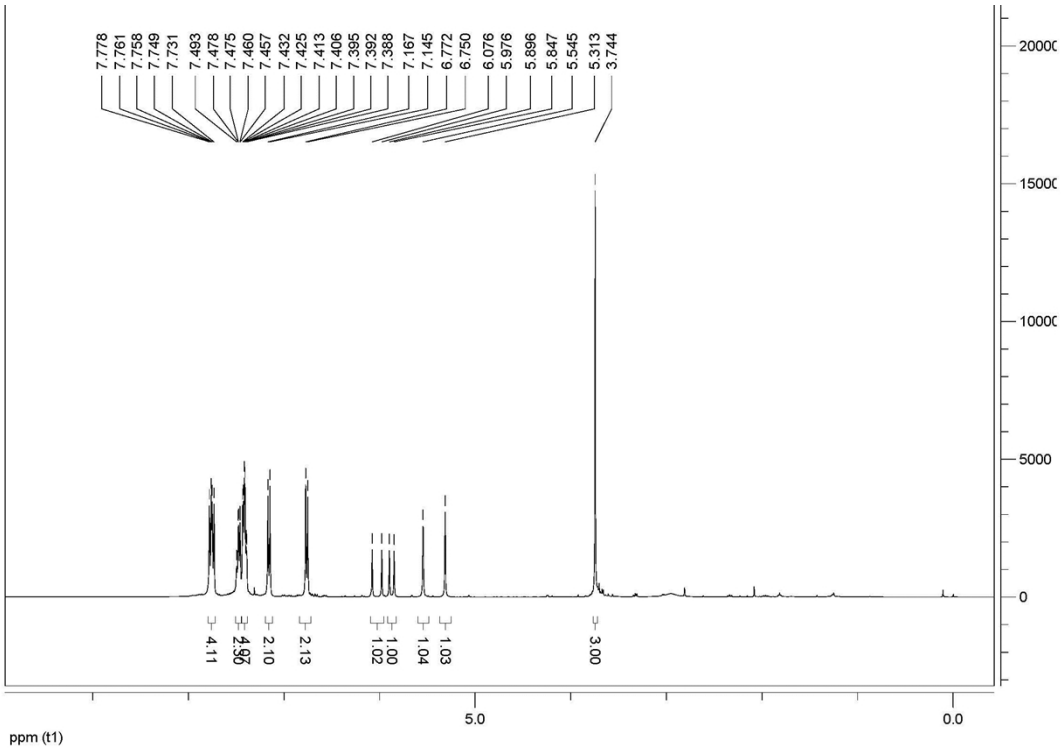

Cl
HO
Ph2P
O
1h

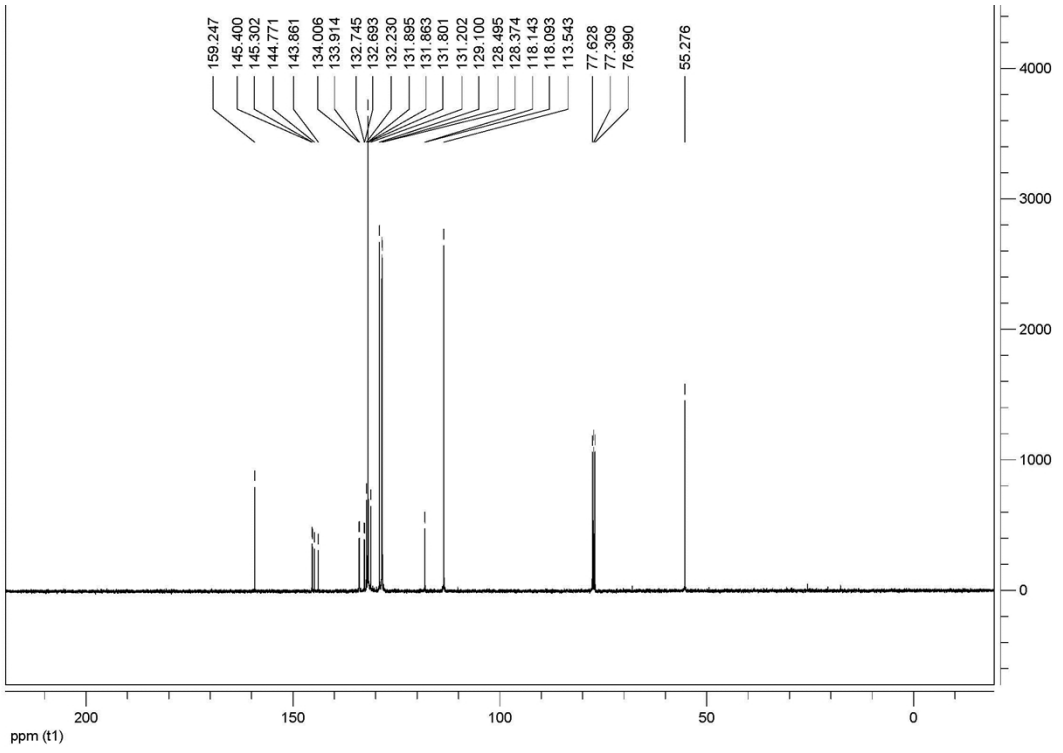

7.745
7.726
7.713
7.709
7.705
7.695
7.675
7.662
7.644
7.641
7.485
7.481
7.466
7.463
7.444
7.436
7.424
7.417
7.406
7.398
7.355
7.347
7.336
7.329
7.317
7.310
7.262
7.240
7.082
7.060
4.625
4.594
4.591
4.565
4.563
4.531
2.286
2.271
2.267
2.260
2.251
2.247
2.232
2.229
2.214
2.199
2.195
2.192
2.174
2.156
6000
5000
4000
3000
2000
1000
0
6.99
2.01
2.09
2.58
3.00
15.0
10.0
5.0
0.0
ppm (t1)

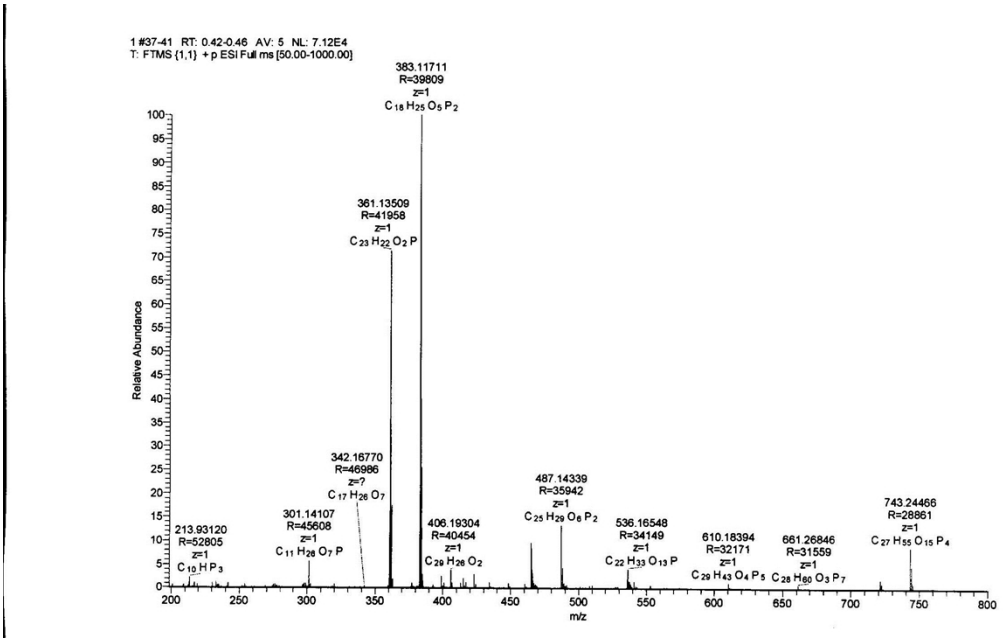

208.806
208.711
133.418
133.147
133.079
132.191
132.008
131.939
131.919
131.669
131.570
131.534
131.435
131.337
131.013
130.950
130.922
130.863
128.719
128.477
128.354
128.065
127.935
127.436
127.415
110.685
110.550
102.668
77.401
77.083
76.766
61.630
61.566
23.095
23.044
12.310
1500
1000
500
0
200
150
100
50
0
ppm (t1)

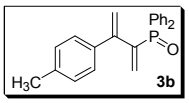

31.793
2000
1500
1000
500
0
100
50
0
-50
-100
-150
-200
ppm (t1)

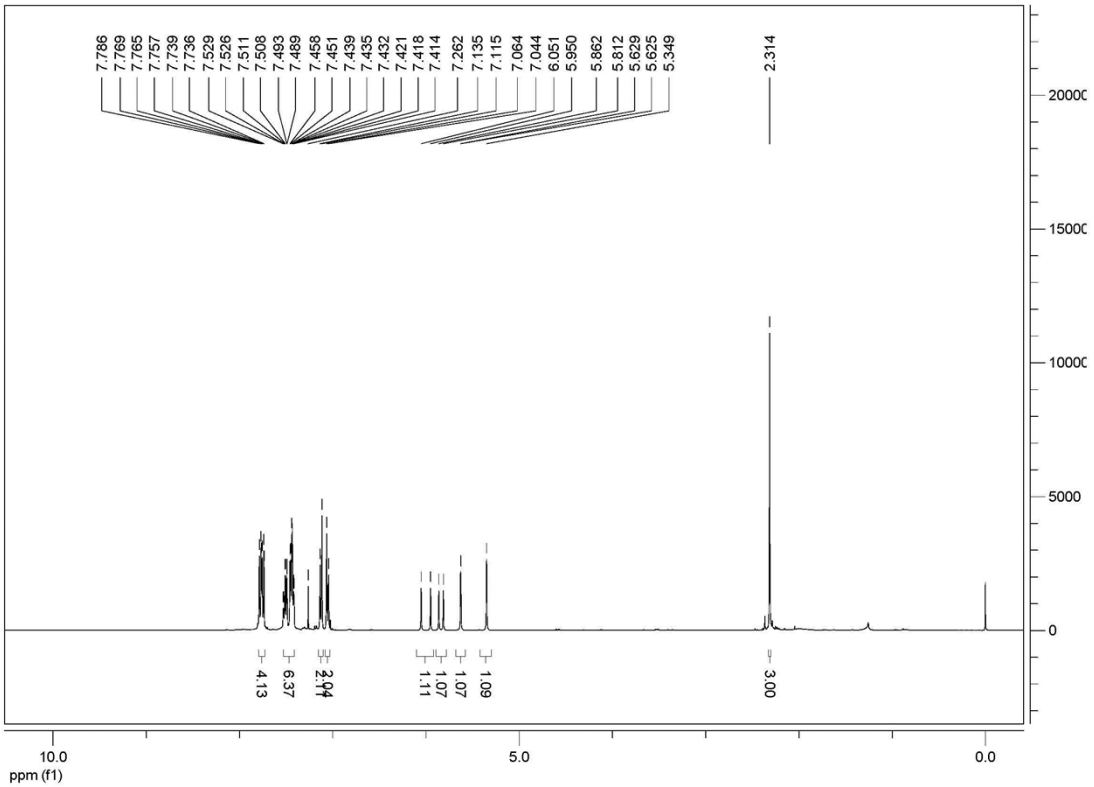

Ph2
P
O
MeO
3a

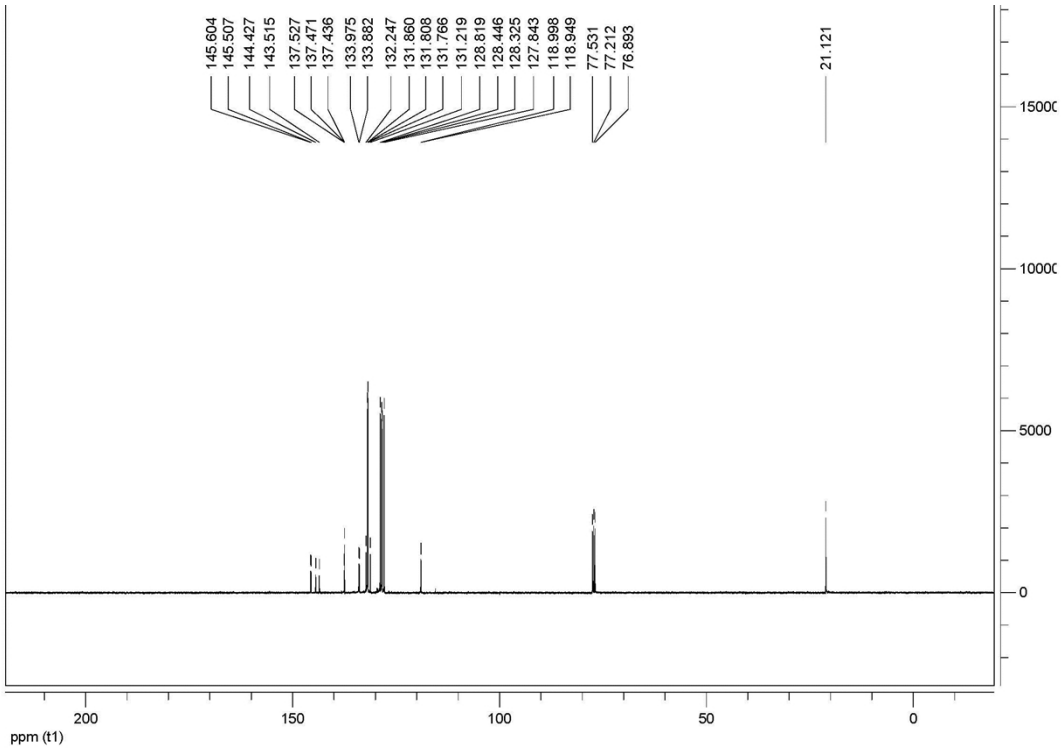

7.778
7.761
7.758
7.749
7.731
7.493
7.478
7.475
7.460
7.457
7.432
7.425
7.413
7.406
7.395
7.392
7.388
7.167
7.145
6.772
6.750
6.076
5.976
5.896
5.847
5.545
5.313
3.744
4.11
2.10
2.13
1.02
1.00
1.04
1.03
3.00
5.0
0.0
ppm (t1)


159.247
145.400
145.302
144.771
143.861
134.006
133.914
132.745
132.693
132.230
131.895
131.863
131.801
131.202
129.100
128.495
128.374
118.143
118.093
113.543
77.628
77.309
76.990
55.276
200
150
100
50
0
ppm (t1)

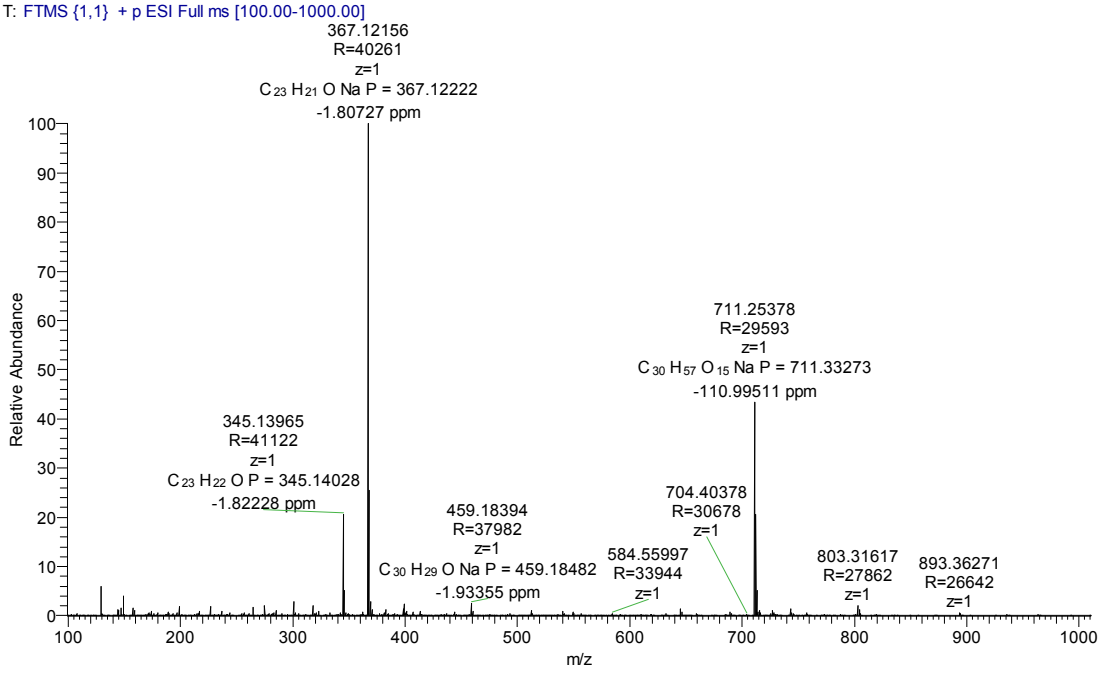

1 #37-41 RT: 0.42-0.46 AV: 5 NL: 7.12E4
T: FTMS {1,1} + p ESI Full ms [50.00-1000.00]
383.11711
R=39809
z=1
C 18 H 25 O 5 P 2
361.13509
R=41958
z=1
C 23 H 22 O 2 P
342.16770
R=46986
z=7
C 17 H 26 O 7
301.14107
R=45608
z=1
C 11 H 28 O 7 P
213.93120
R=52805
z=1
C 10 H P 3
406.19304
R=40454
z=1
C 29 H 26 O 2
487.14339
R=35942
z=1
C 25 H 29 O 6 P 2
536.16548
R=34149
z=1
C 22 H 33 O 13 P
610.18394
R=32171
z=1
C 29 H 43 O 4 P 5
661.26846
R=31559
z=1
C 28 H 60 O 3 P 7
743.24466
R=28861
z=1
C 27 H 55 O 15 P 4
Relative Abundance
m/z

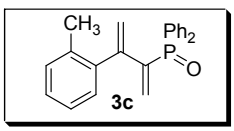

Ph2
P
O
H3C
3b

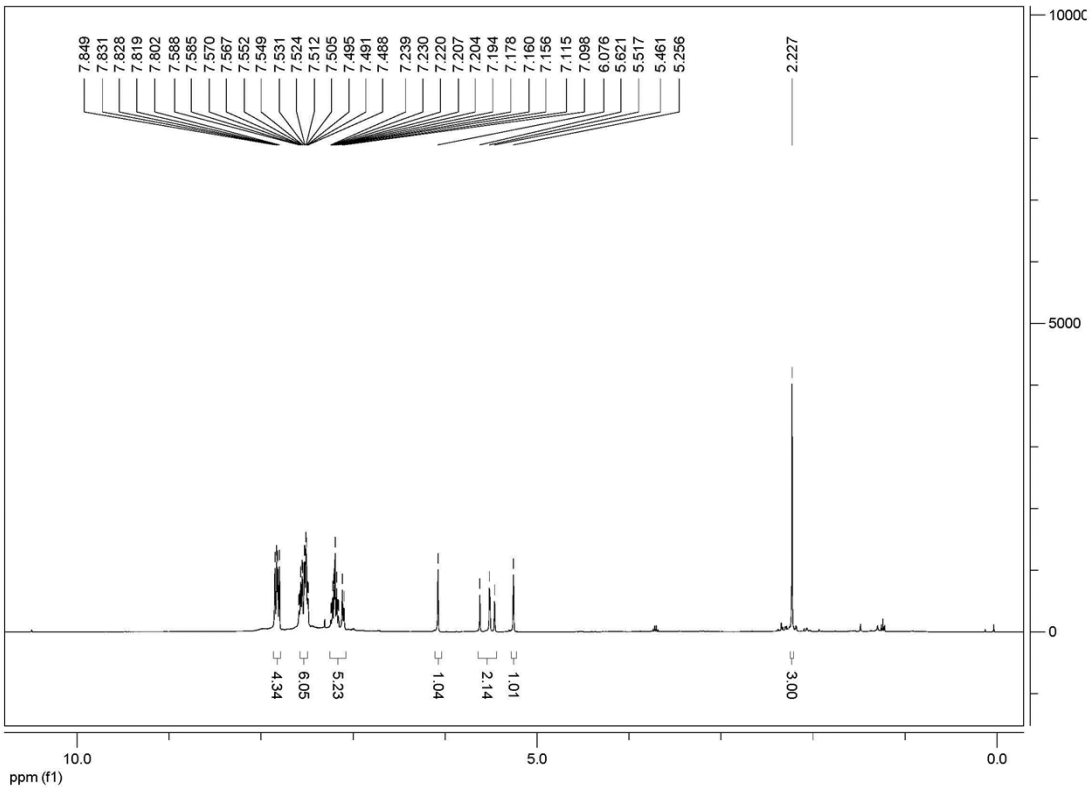

7.786
7.769
7.765
7.757
7.739
7.736
7.529
7.526
7.511
7.508
7.493
7.489
7.458
7.451
7.439
7.435
7.432
7.421
7.418
7.414
7.262
7.135
7.115
7.064
7.044
6.051
5.950
5.862
5.812
5.629
5.625
5.349
2.314
4.13
6.37
1.11
1.07
1.07
1.09
3.00
ppm (f1)

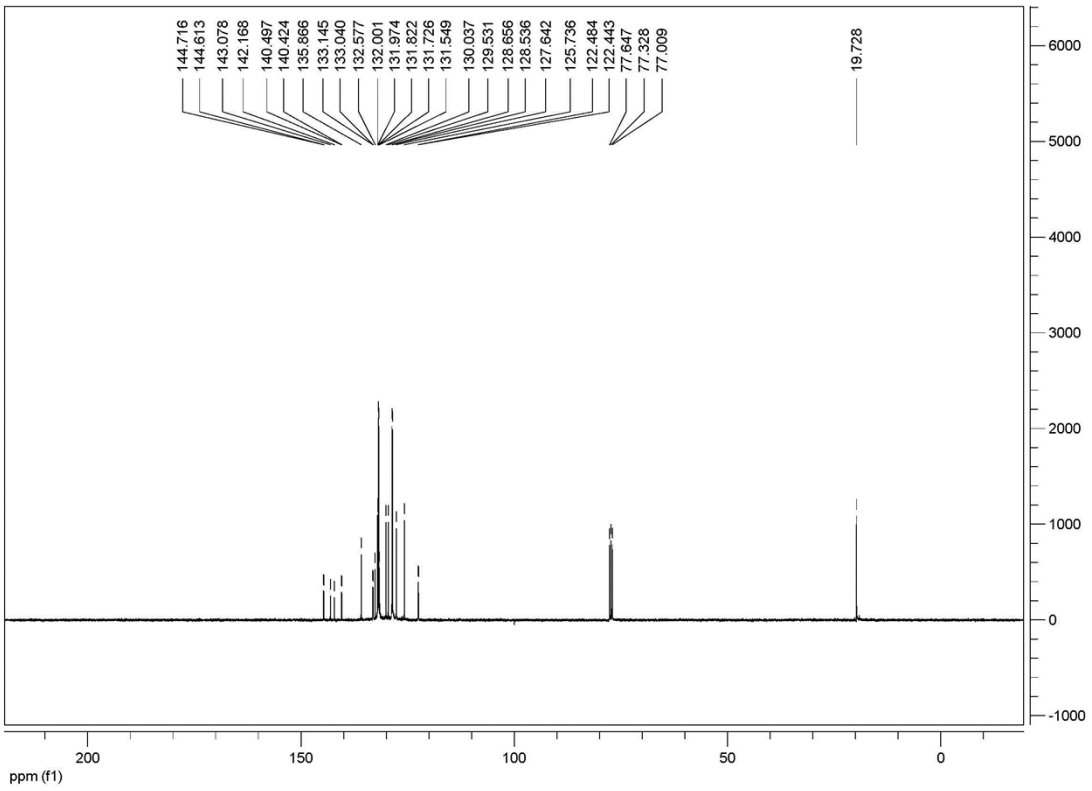


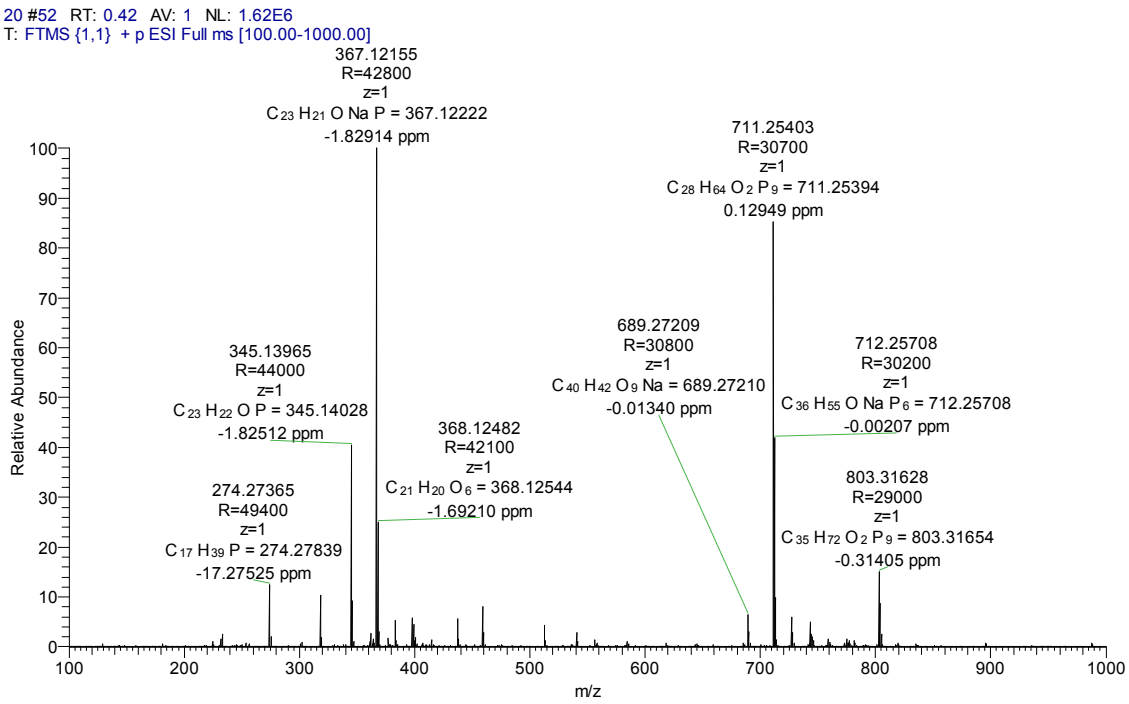
15 #40-41 RT: 0.35-0.36 AV: 2 NL: 5.88E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

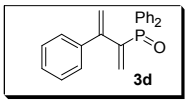


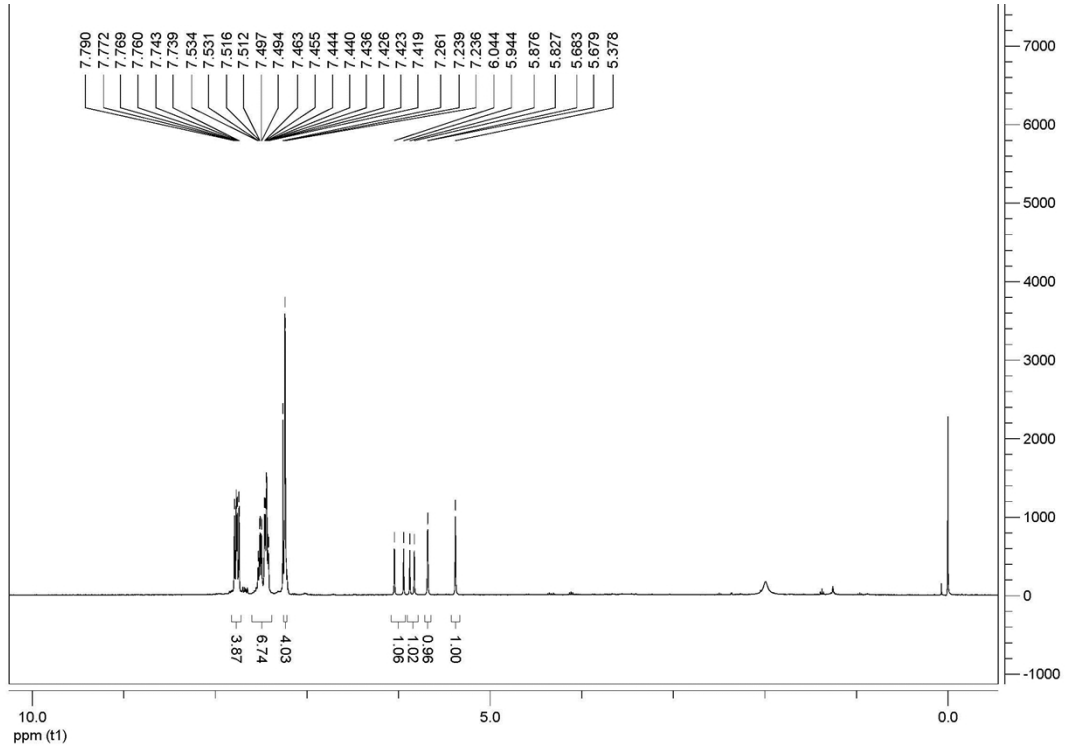


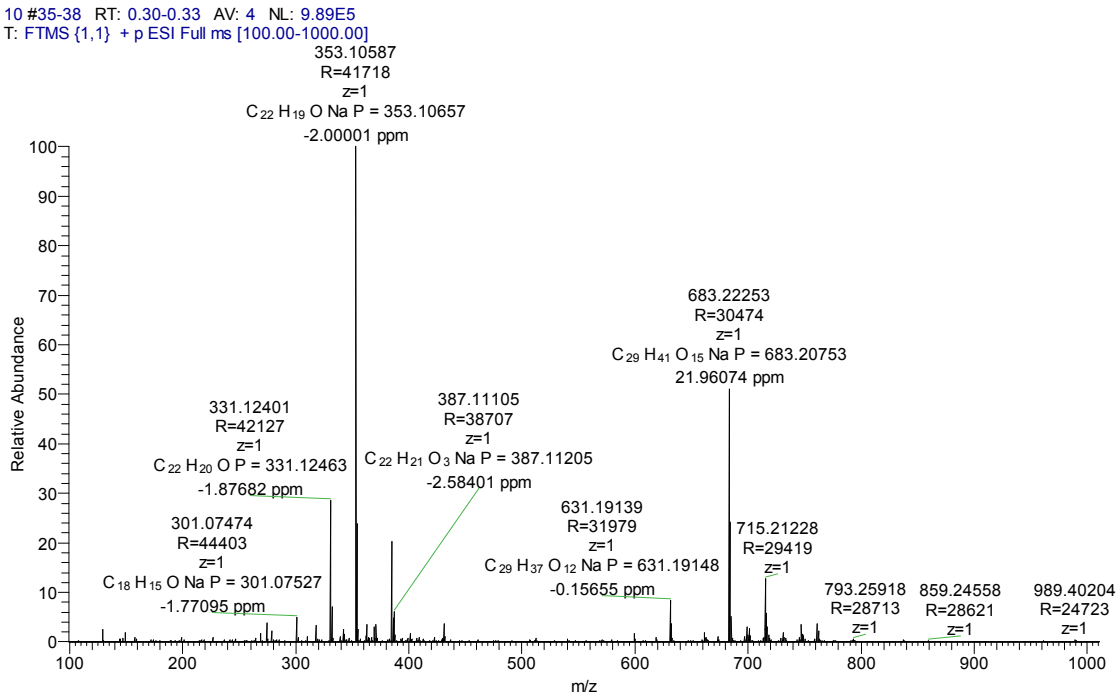

7.849
7.831
7.828
7.819
7.802
7.588
7.585
7.570
7.567
7.552
7.549
7.531
7.524
7.512
7.505
7.495
7.491
7.488
7.239
7.230
7.220
7.207
7.204
7.194
7.178
7.160
7.156
7.115
7.098
6.076
5.621
5.517
5.461
5.256
2.227
4.34
6.05
5.23
1.04
2.14
1.01
3.00
10000
5000
0
10.0
5.0
0.0
ppm (f1)

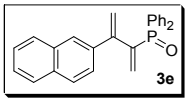

144.716
144.613
143.078
142.168
140.497
140.424
135.866
133.145
133.040
132.577
132.001
131.974
131.822
131.726
131.549
130.037
129.531
128.656
128.536
127.642
125.736
122.484
122.443
77.647
77.328
77.009
19.728
6000
5000
4000
3000
2000
1000
0
-1000
200
150
100
50
0
ppm (f1)

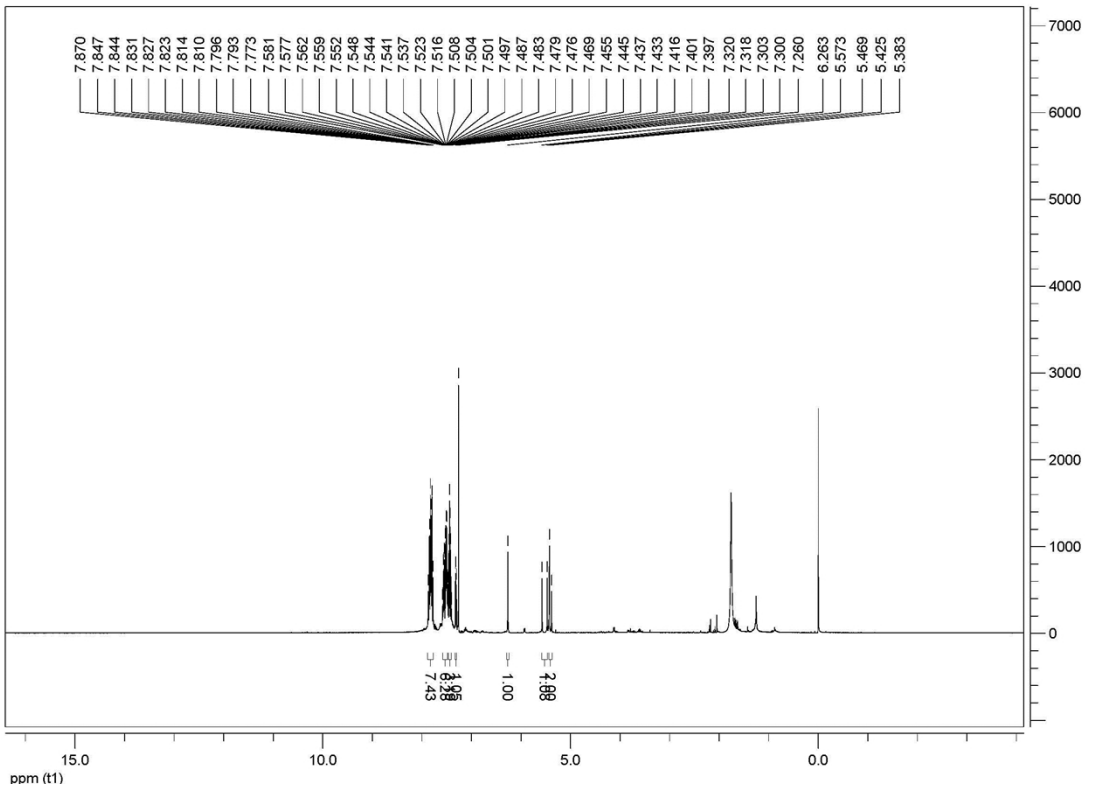

20 #52 RT: 0.42 AV: 1 NL: 1.62E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]
367.12155
R=42800
z=1
C23 H21 O Na P = 367.12222
-1.82914 ppm
711.25403
R=30700
z=1
C28 H64 O2 P9 = 711.25394
0.12949 ppm
689.27209
R=30800
z=1
C40 H42 O9 Na = 689.27210
-0.01340 ppm
712.25708
R=30200
z=1
C36 H55 O Na P6 = 712.25708
-0.00207 ppm
345.13965
R=44000
z=1
C23 H22 O P = 345.14028
-1.82512 ppm
368.12482
R=42100
z=1
C21 H20 O6 = 368.12544
-1.69210 ppm
803.31628
R=29000
z=1
C35 H72 O2 P9 = 803.31654
-0.31405 ppm
274.27365
R=49400
z=1
C17 H39 P = 274.27839
-17.27525 ppm
Relative Abundance
m/z

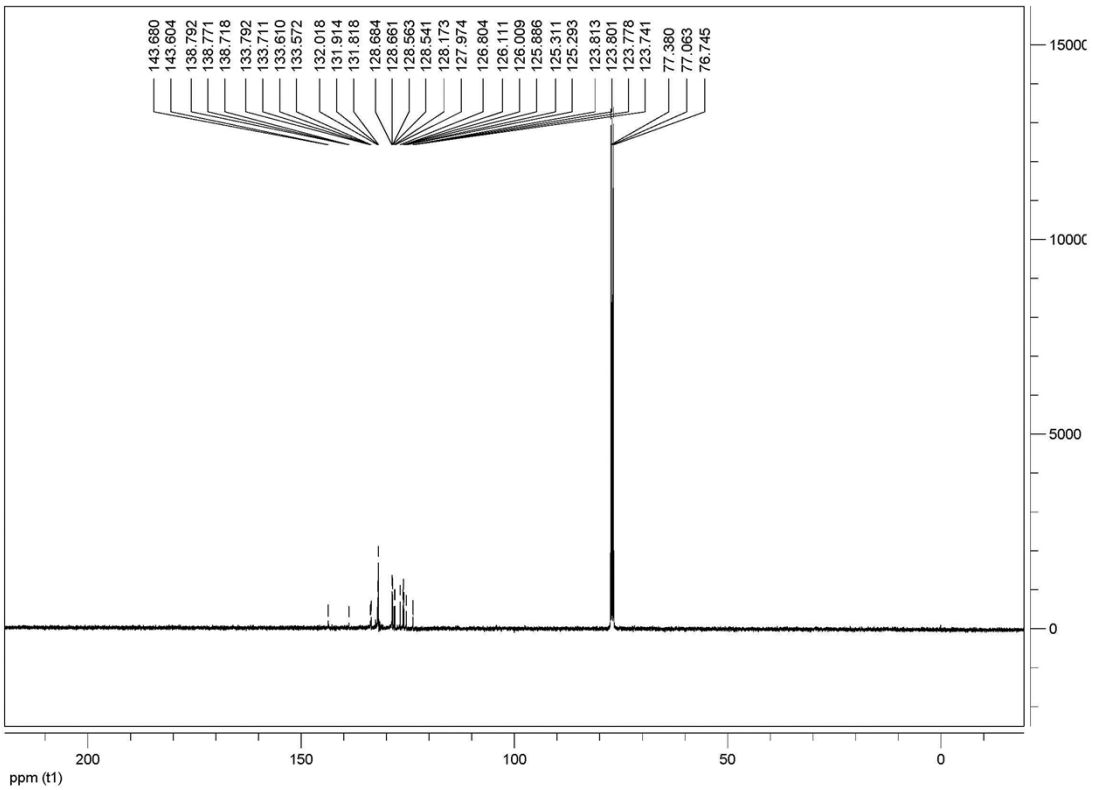

Ph2
P
O
3d

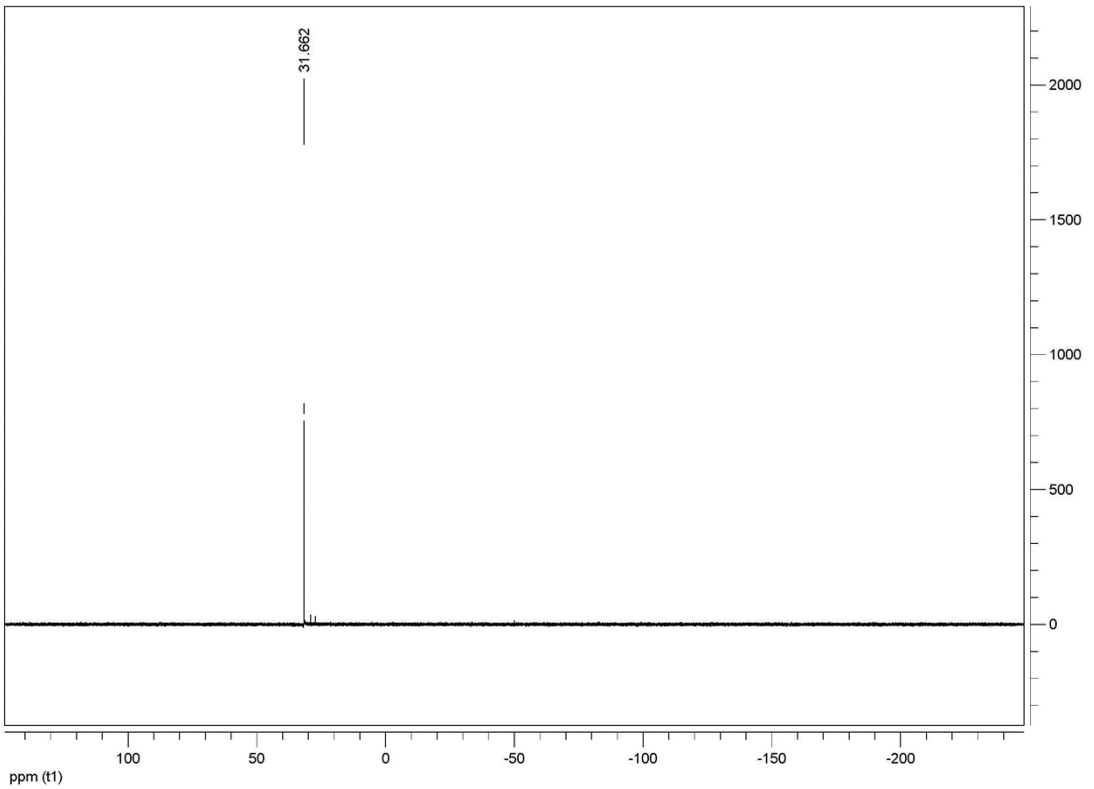

7.790
7.772
7.769
7.760
7.743
7.739
7.534
7.531
7.516
7.512
7.497
7.494
7.463
7.455
7.444
7.440
7.436
7.426
7.423
7.419
7.261
7.239
7.236
6.044
5.944
5.876
5.827
5.683
5.679
5.378
3.87
6.74
4.03
1.06
1.02
0.96
1.00
10.0
5.0
0.0
ppm (t1)

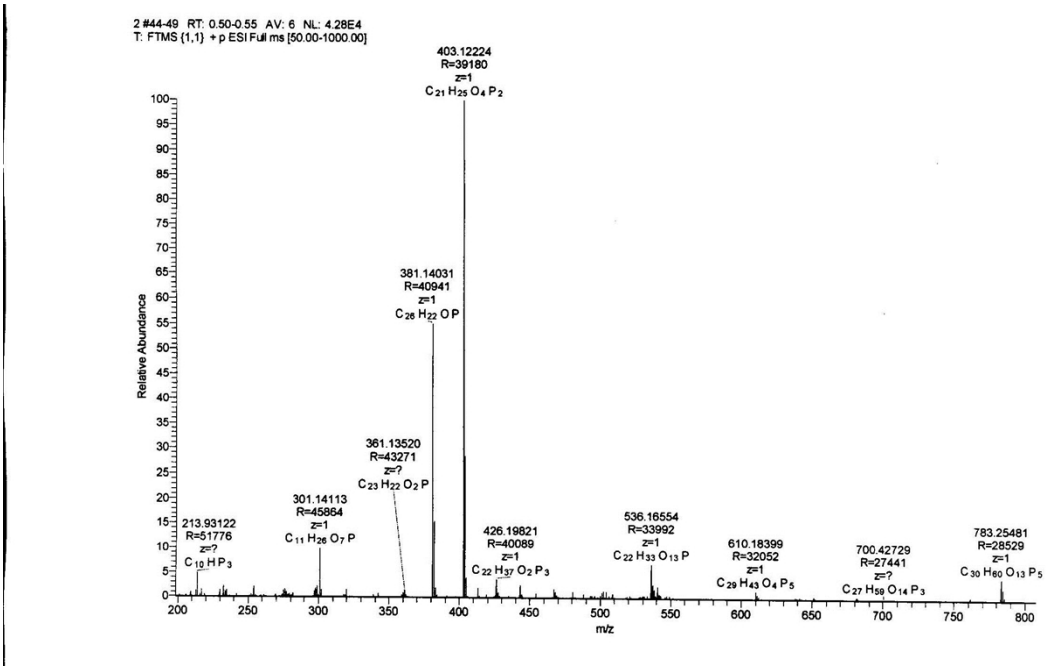

10 #35-38 RT: 0.30-0.33 AV: 4 NL: 9.89E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]
353.10587
R=41718
z=1
C22 H19 O Na P = 353.10657
-2.00001 ppm
683.22253
R=30474
z=1
C29 H41 O15 Na P = 683.20753
21.96074 ppm
331.12401
R=42127
z=1
C22 H20 O P = 331.12463
-1.87682 ppm
387.11105
R=38707
z=1
C22 H21 O3 Na P = 387.11205
-2.58401 ppm
631.19139
R=31979
z=1
C29 H37 O12 Na P = 631.19148
-0.15655 ppm
301.07474
R=44403
z=1
C18 H15 O Na P = 301.07527
-1.77095 ppm
715.21228
R=29419
z=1
793.25918
R=28713
z=1
859.24558
R=28621
z=1
989.40204
R=24723
z=1
Relative Abundance
m/z

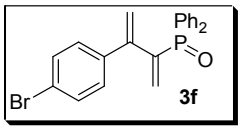

Ph2
P
O
3e

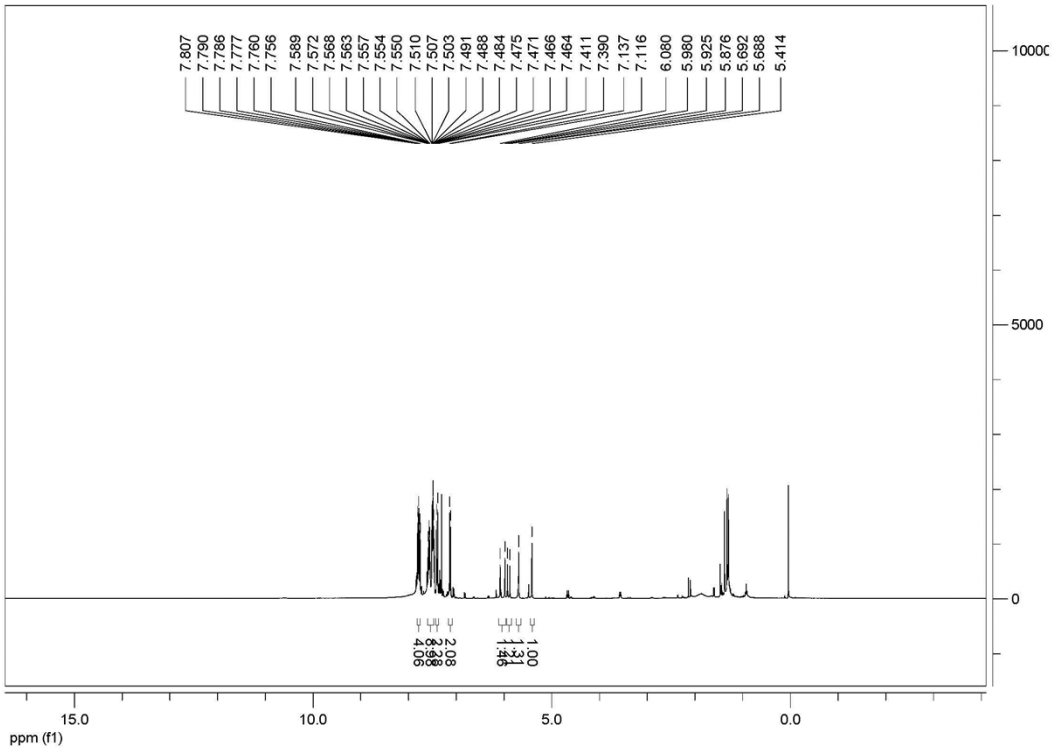

7.870
7.847
7.844
7.831
7.827
7.823
7.814
7.810
7.796
7.793
7.773
7.581
7.577
7.562
7.559
7.552
7.548
7.544
7.541
7.537
7.523
7.516
7.508
7.504
7.501
7.497
7.487
7.483
7.479
7.476
7.469
7.455
7.445
7.437
7.433
7.416
7.401
7.397
7.320
7.318
7.303
7.300
7.260
6.263
5.573
5.469
5.425
5.383
7.43
1.00
2.00
ppm (t1)

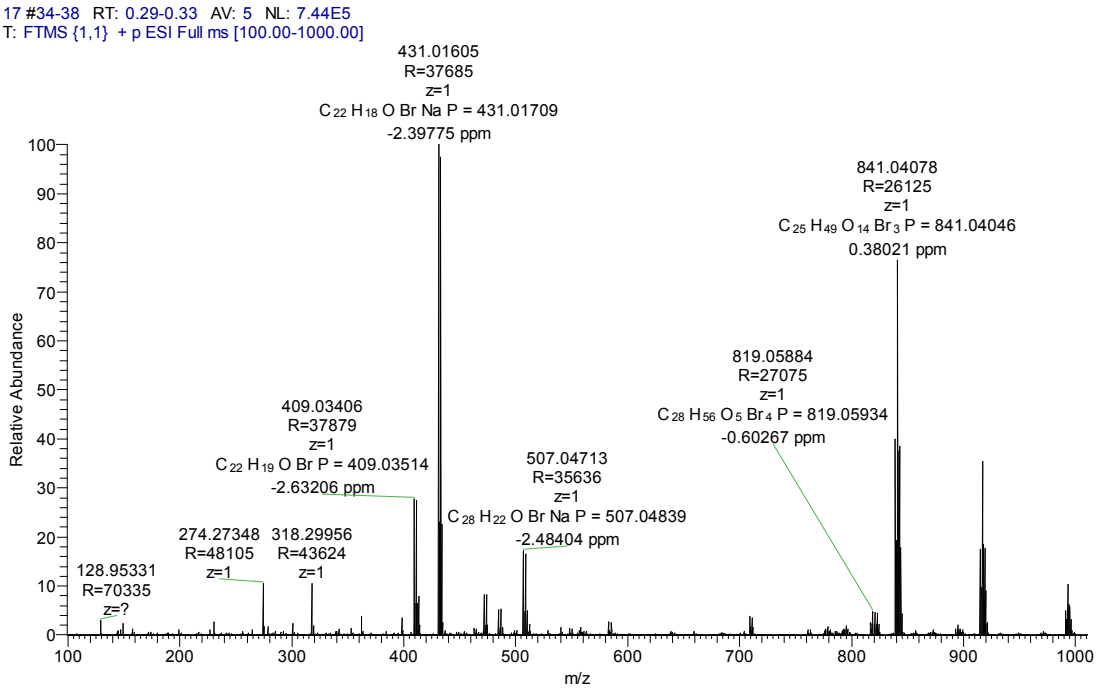

143.680
143.604
138.792
138.771
138.718
133.792
133.711
133.610
133.572
132.018
131.914
131.818
128.684
128.661
128.563
128.541
128.173
127.974
126.804
126.111
126.009
125.886
125.311
125.293
123.813
123.801
123.778
123.741
77.380
77.063
76.745
15000
10000
5000
0
200
150
100
50
0
ppm (t1)

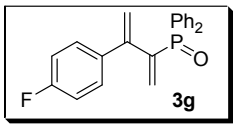

31.662
2000
1500
1000
500
0
100
50
0
-50
-100
-150
-200
ppm (t1)

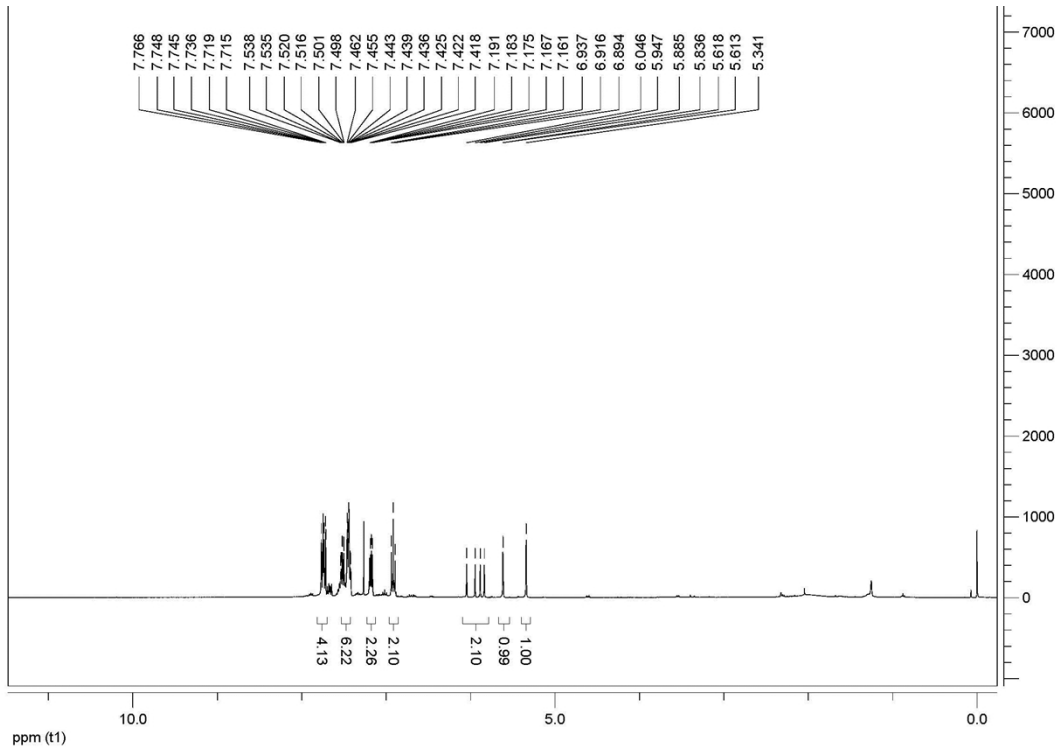

2 #44-49 RT: 0.50-0.55 AV: 6 NL: 4.28E4
T: FTMS {1,1} + p ESI Full ms [50.00-1000.00]
403.12224
R=39180
z=1
C21 H25 O4 P2
381.14031
R=40941
z=1
C26 H22 O P
361.13520
R=43271
z=?
C23 H22 O2 P
301.14113
R=45864
z=1
C11 H26 O7 P
213.93122
R=51776
z=?
C10 H P3
426.19821
R=40089
z=1
C22 H37 O2 P3
536.16554
R=33992
z=1
C22 H33 O13 P
610.18399
R=32052
z=1
C29 H43 O4 P5
700.42729
R=27441
z=?
C27 H59 O14 P3
783.25481
R=28529
z=1
C30 H60 O13 P5
Relative Abundance
m/z

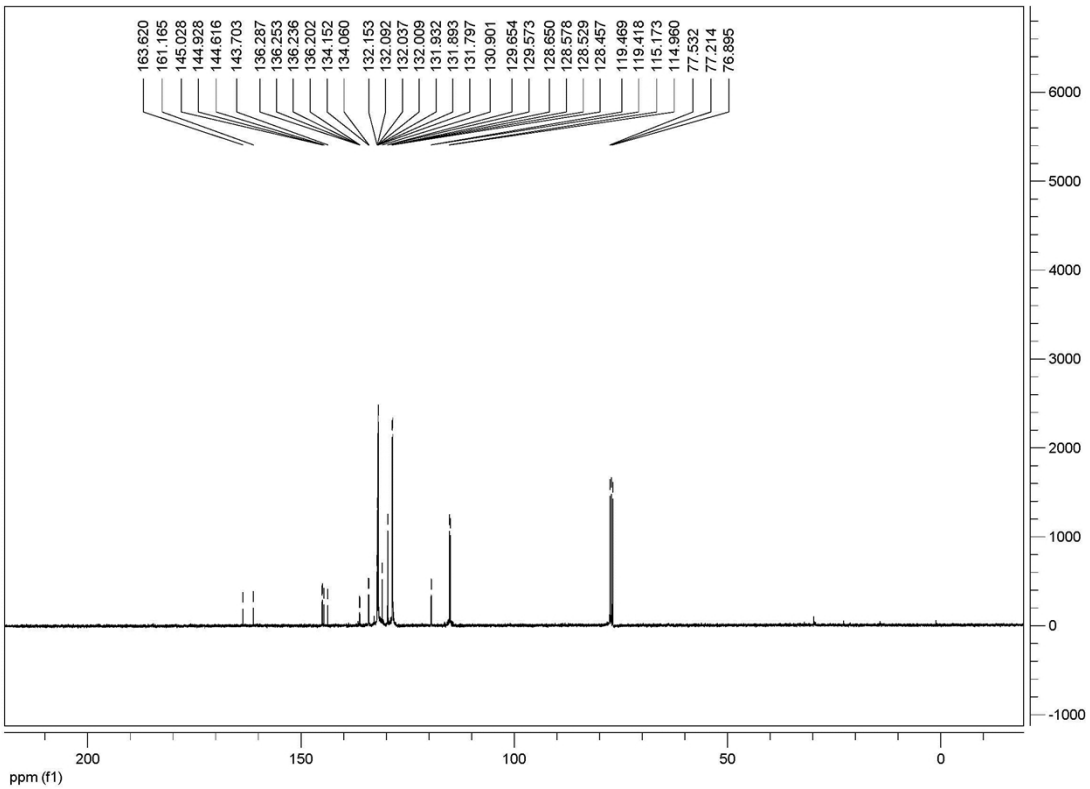

Ph2
P
O
Br
3f


7.807
7.790
7.786
7.777
7.760
7.756
7.589
7.572
7.568
7.563
7.557
7.554
7.550
7.510
7.507
7.503
7.491
7.488
7.484
7.475
7.471
7.466
7.464
7.411
7.390
7.137
7.116
6.080
5.980
5.925
5.876
5.692
5.688
5.414
4.06
2.96
2.88
2.80
1.46
1.21
1.00
10000
5000
0
15.0
10.0
5.0
0.0
ppm (f1)

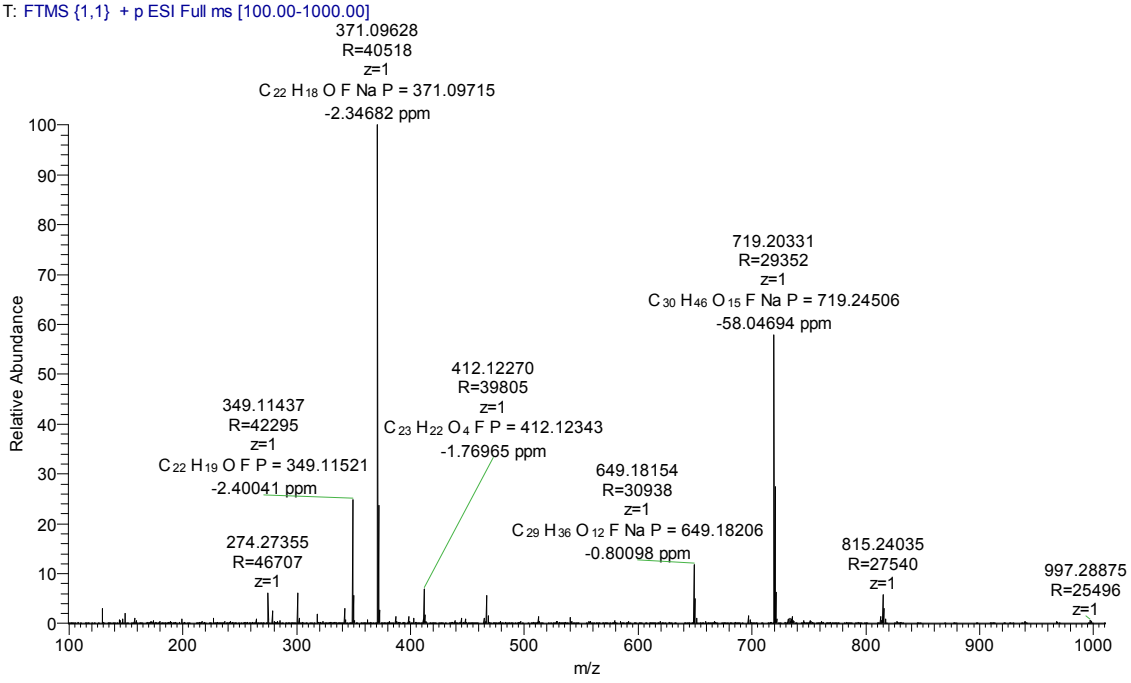

17 #34-38 RT: 0.29-0.33 AV: 5 NL: 7.44E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]
431.01605
R=37685
z=1
C22 H18 O Br Na P = 431.01709
-2.39775 ppm
841.04078
R=26125
z=1
C25 H49 O14 Br3 P = 841.04046
0.38021 ppm
819.05884
R=27075
z=1
C28 H56 O5 Br4 P = 819.05934
-0.60267 ppm
409.03406
R=37879
z=1
C22 H19 O Br P = 409.03514
-2.63206 ppm
507.04713
R=35636
z=1
C28 H22 O Br Na P = 507.04839
-2.48404 ppm
274.27348
R=48105
z=1
318.29956
R=43624
z=1
128.95331
R=70335
z=?
Relative Abundance
m/z

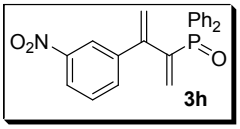

Ph2
P
O
F
3g

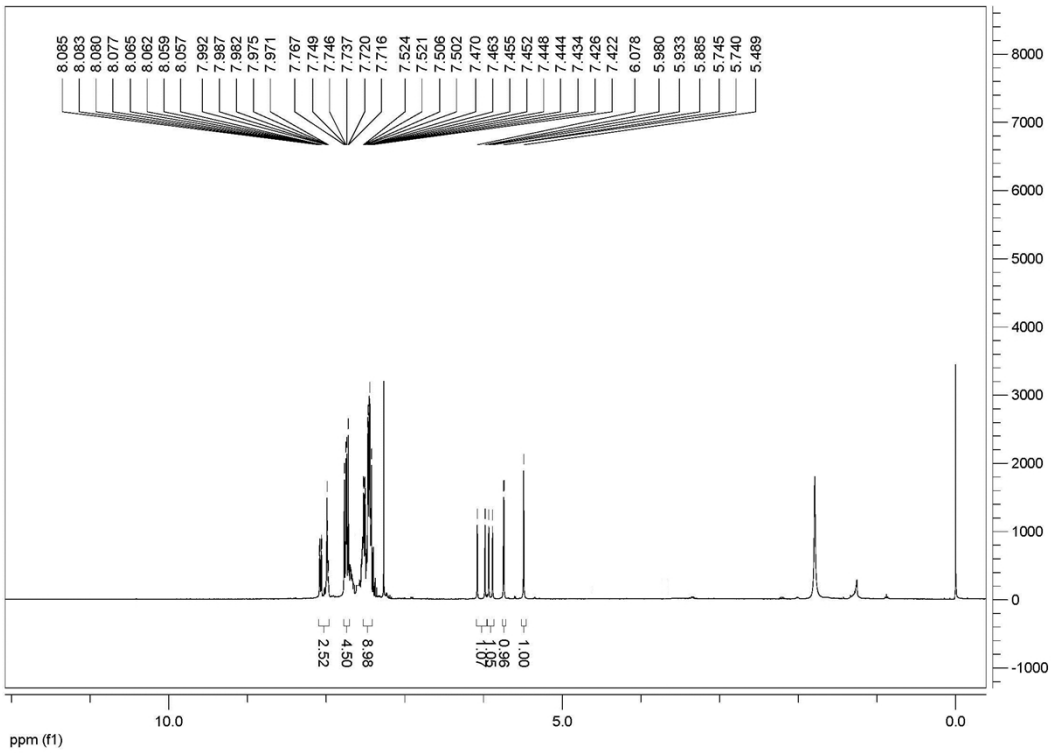

7.766
7.748
7.745
7.736
7.719
7.715
7.538
7.535
7.520
7.516
7.501
7.498
7.462
7.455
7.443
7.439
7.436
7.425
7.422
7.418
7.191
7.183
7.175
7.167
7.161
6.937
6.916
6.894
6.046
5.947
5.885
5.836
5.618
5.613
5.341
4.13
6.22
2.26
2.10
2.10
0.99
1.00
ppm (t1)



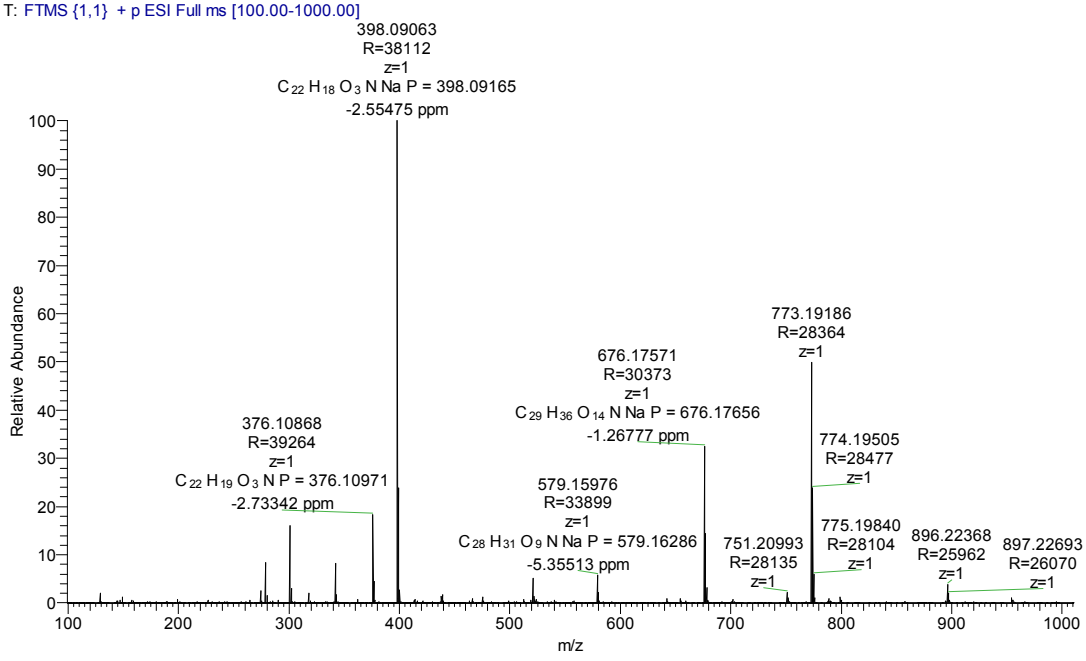
14 #47-48 RT: 0.39-0.40 AV: 2 NL: 1.15E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

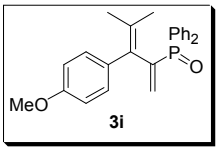




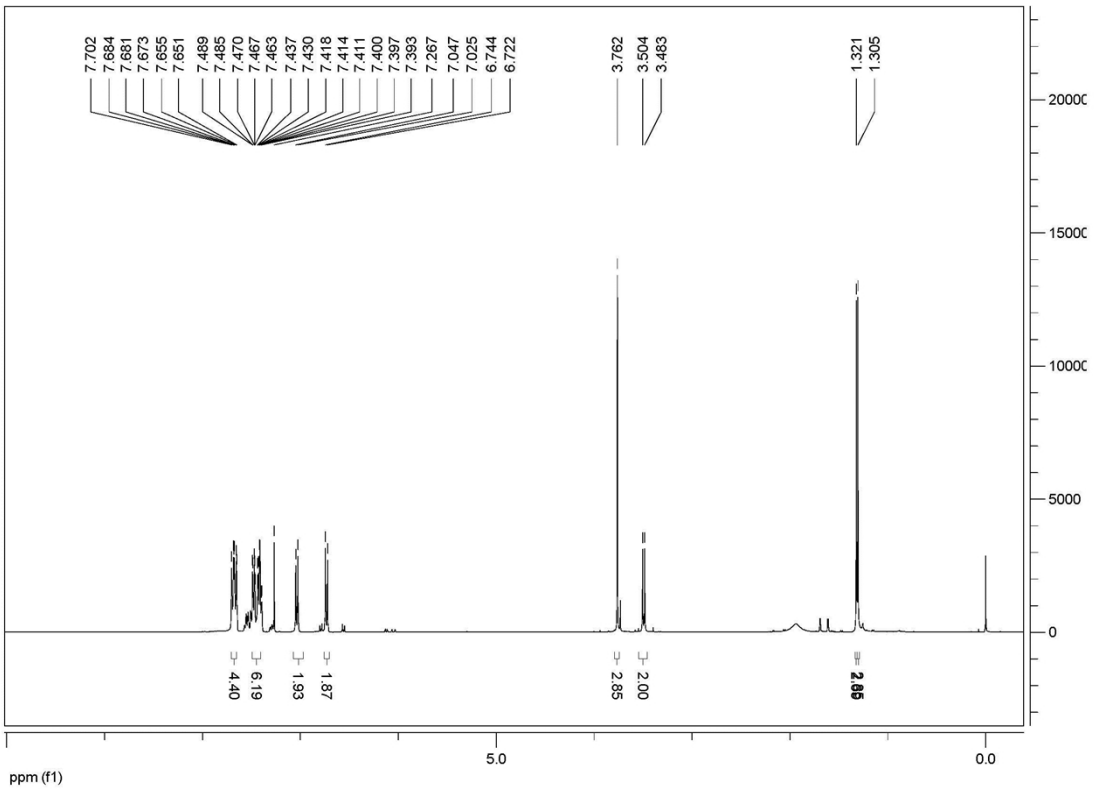


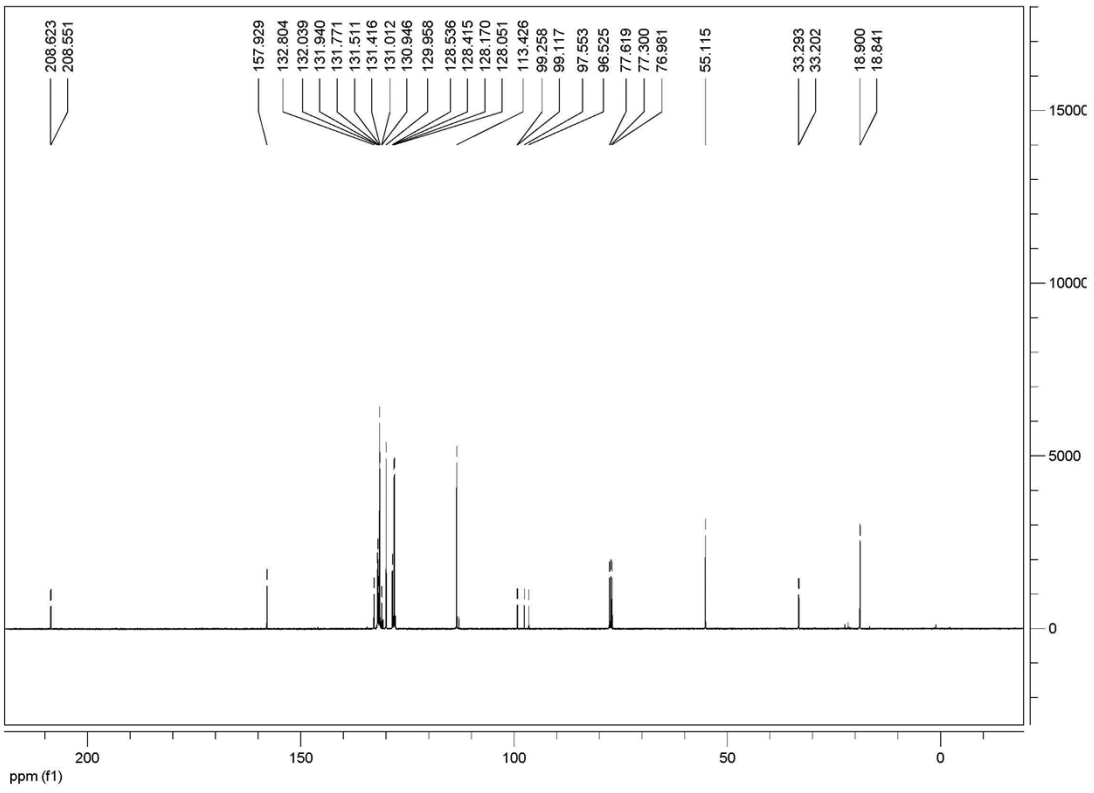
13 #40-44 RT: 0.33-0.37 AV: 5 NL: 1.65E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

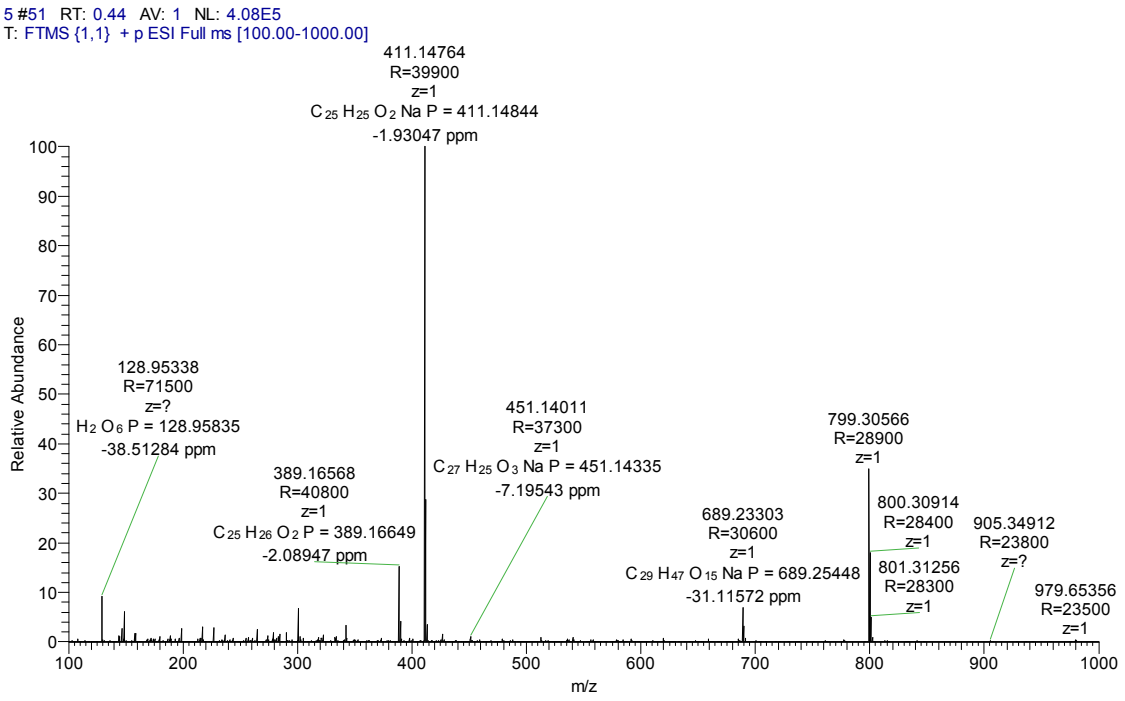


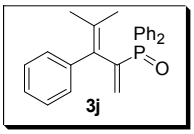


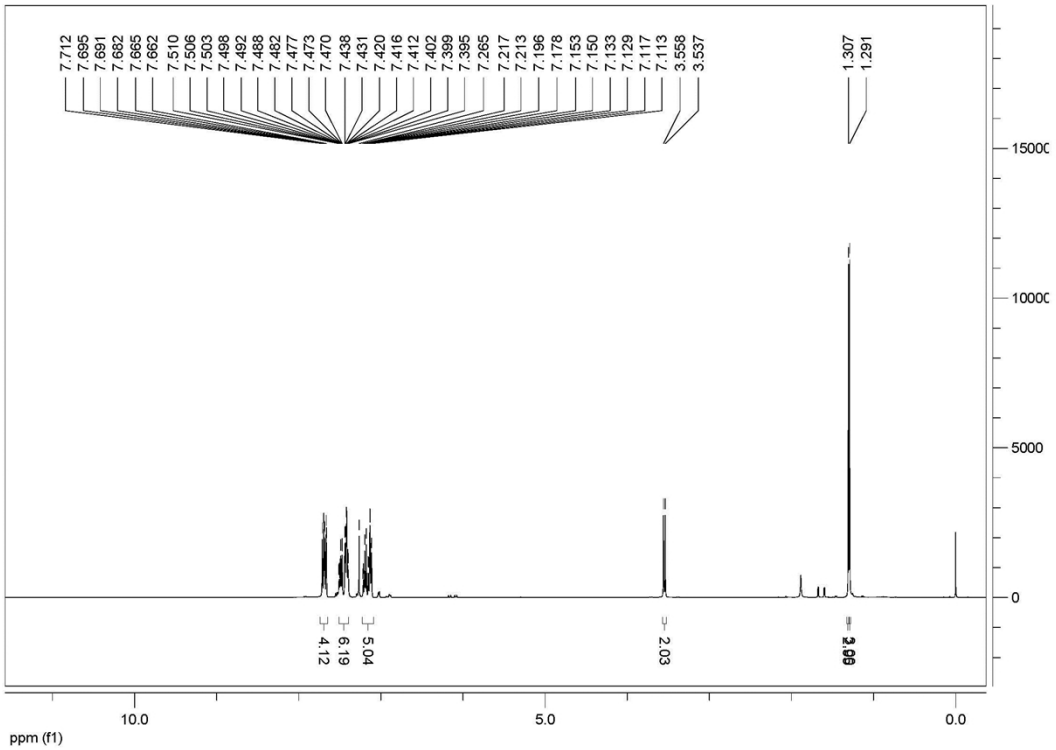
3i

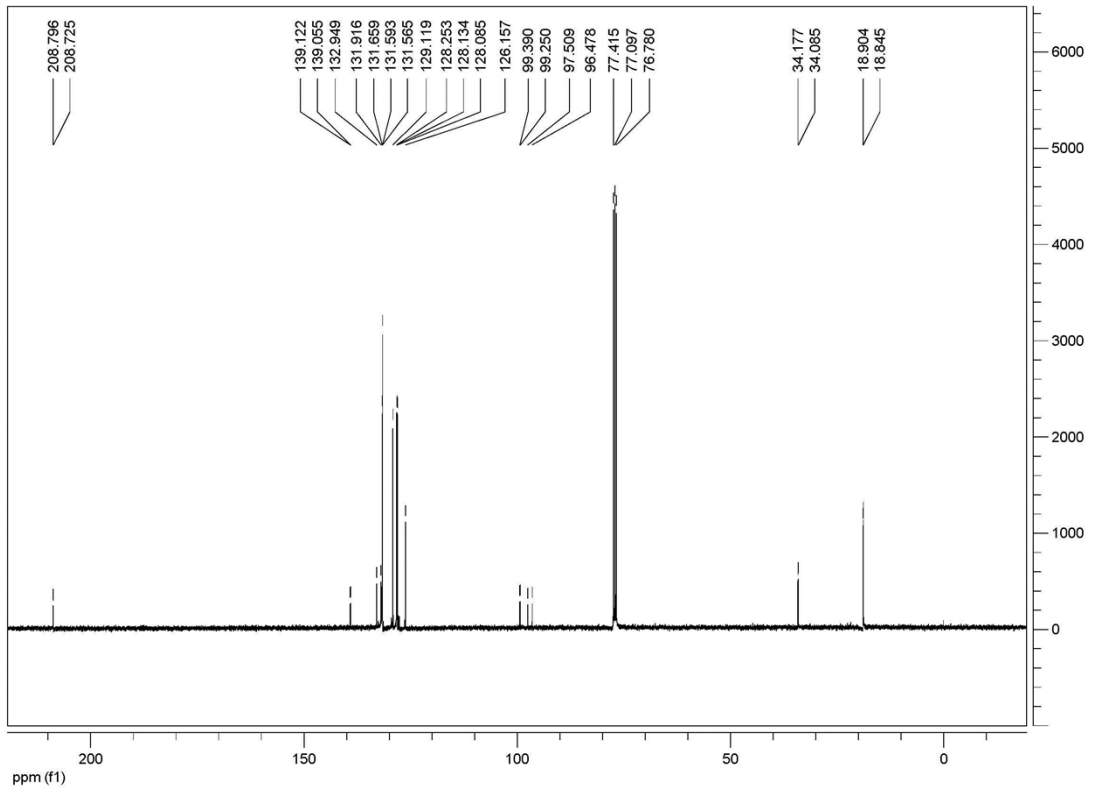

7.702
7.684
7.681
7.673
7.655
7.651
7.489
7.485
7.470
7.467
7.463
7.437
7.430
7.418
7.414
7.411
7.400
7.397
7.393
7.267
7.047
7.025
6.744
6.722
3.762
3.504
3.483
1.321
1.305
4.40
6.19
1.93
1.87
2.85
2.00
2.05
5.0
0.0
ppm (f1)


208.623
208.551
157.929
132.804
132.039
131.940
131.771
131.511
131.416
131.012
130.946
129.958
128.536
128.415
128.170
128.051
113.426
99.258
99.117
97.553
96.525
77.619
77.300
76.981
55.115
33.293
33.202
18.900
18.841
200
150
100
50
0
ppm (f1)

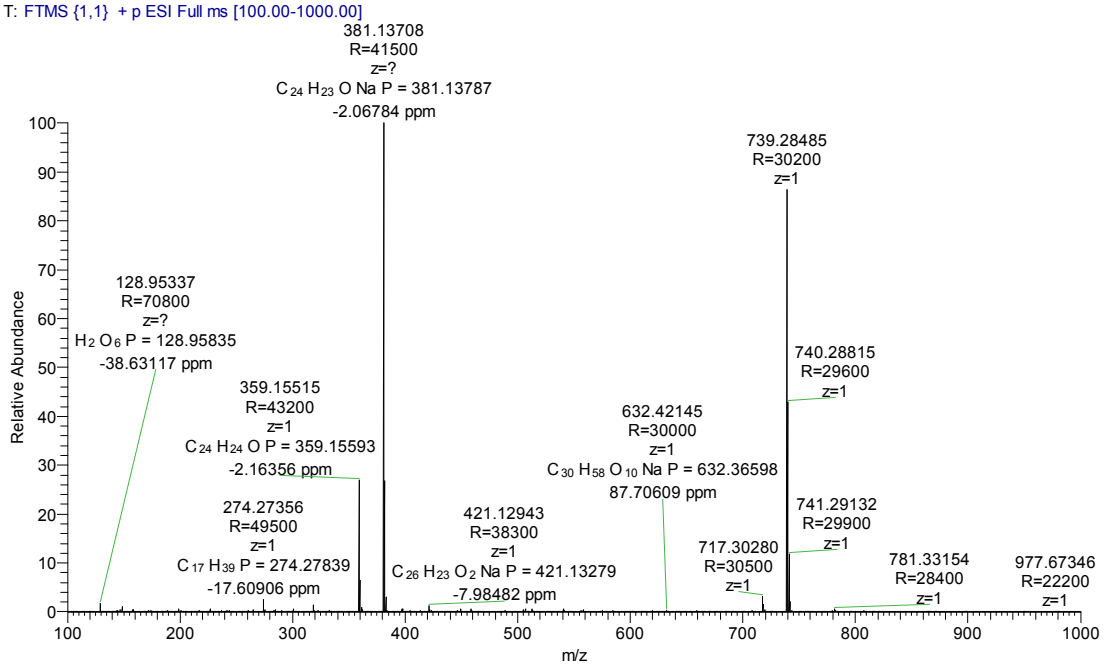

5 #51 RT: 0.44 AV: 1 NL: 4.08E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]
411.14764
R=39900
z=1
C25 H25 O2 Na P = 411.14844
-1.93047 ppm
128.95338
R=71500
z=?
H2 O6 P = 128.95835
-38.51284 ppm
389.16568
R=40800
z=1
C25 H26 O2 P = 389.16649
-2.08947 ppm
451.14011
R=37300
z=1
C27 H25 O3 Na P = 451.14335
-7.19543 ppm
689.23303
R=30600
z=1
C29 H47 O15 Na P = 689.25448
-31.11572 ppm
799.30566
R=28900
z=1
800.30914
R=28400
z=1
801.31256
R=28300
z=1
905.34912
R=23800
z=?
979.65356
R=23500
z=1
Relative Abundance
m/z

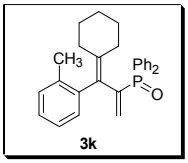

Ph2
P
O
3j


7.712
7.695
7.691
7.682
7.665
7.662
7.510
7.506
7.503
7.498
7.492
7.488
7.482
7.477
7.473
7.470
7.438
7.431
7.420
7.416
7.412
7.402
7.399
7.395
7.265
7.217
7.213
7.196
7.178
7.153
7.150
7.133
7.129
7.117
7.113
3.558
3.537
1.307
1.291
4.12
6.19
5.04
2.03
3.00
ppm (f1)

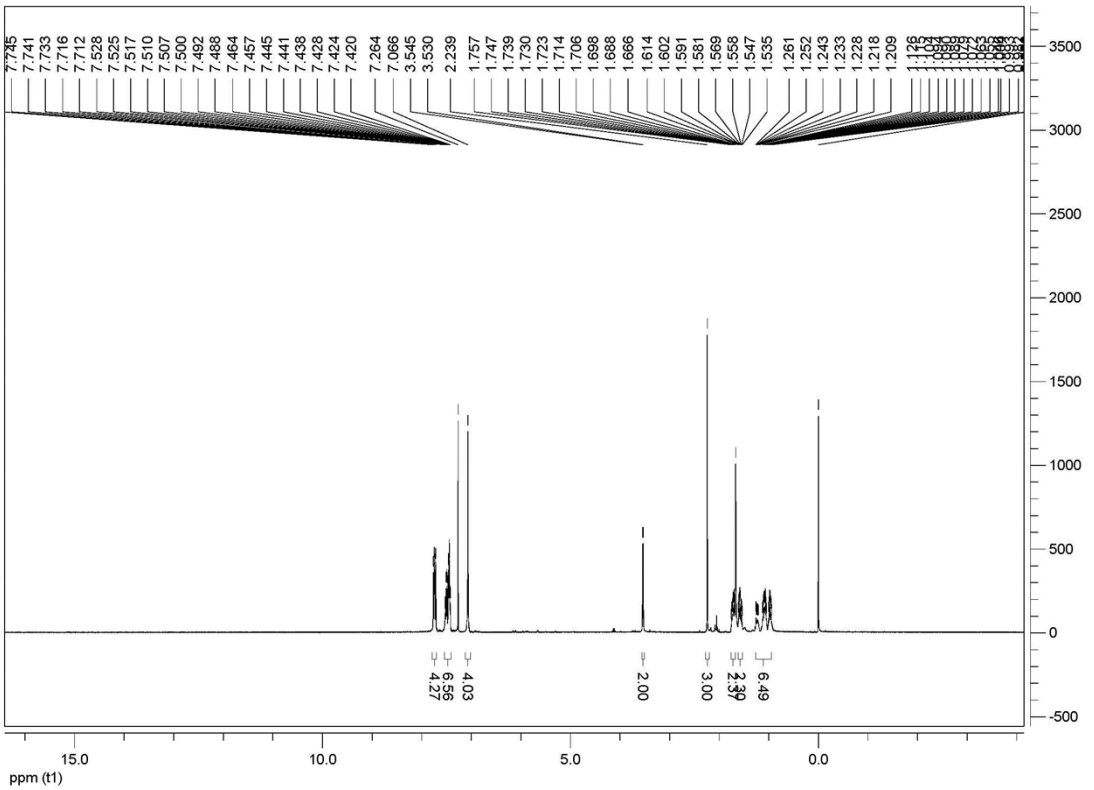


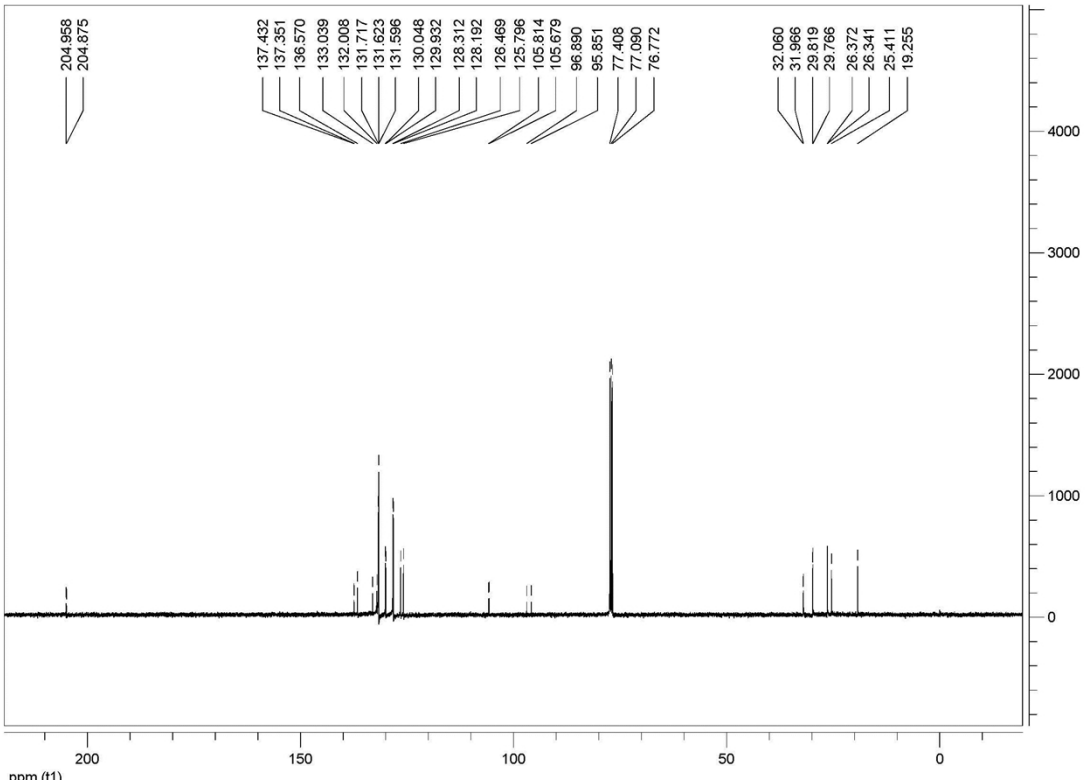
6 #46 RT: 0.38 AV: 1 NL: 1.98E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

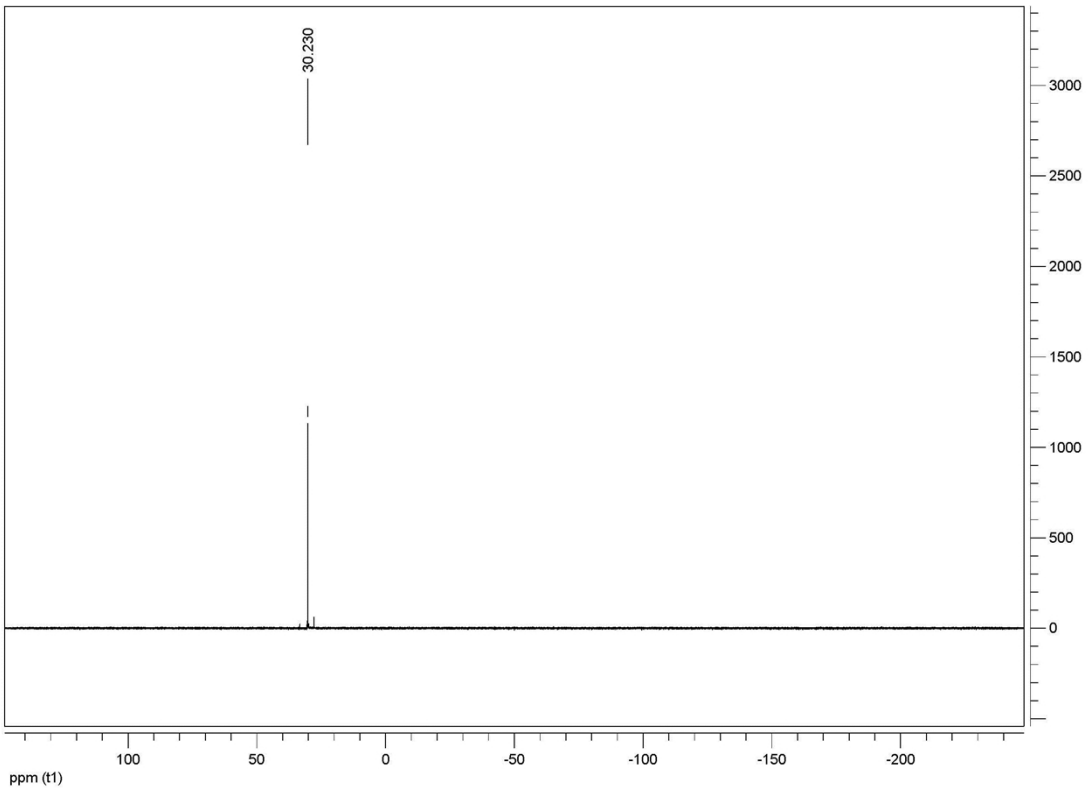




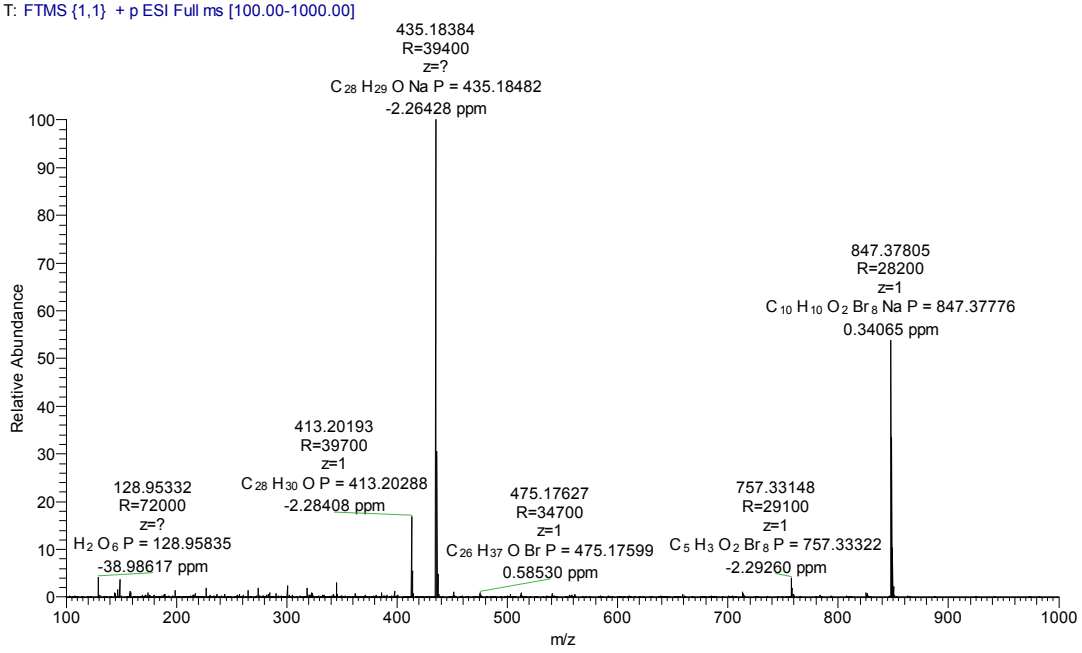
3k

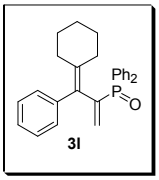

7.745
7.741
7.733
7.716
7.712
7.528
7.525
7.517
7.510
7.507
7.500
7.492
7.488
7.464
7.457
7.445
7.441
7.438
7.428
7.424
7.420
7.264
7.066
3.545
3.530
2.239
1.757
1.747
1.739
1.730
1.723
1.714
1.706
1.698
1.688
1.666
1.614
1.602
1.591
1.581
1.569
1.558
1.547
1.535
1.261
1.252
1.243
1.233
1.228
1.218
1.209
4.27
6.96
4.03
2.00
3.00
2.39
2.39
6.49
ppm (t1)

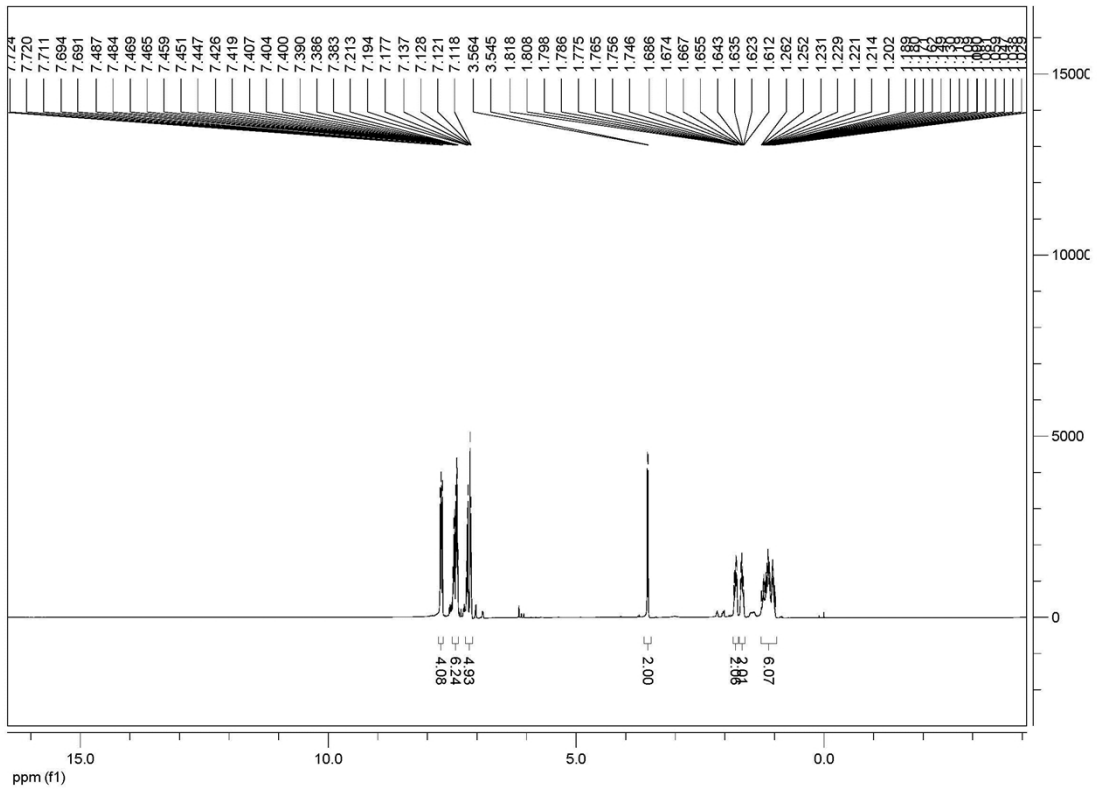

204.958
204.875
137.432
137.351
136.570
133.039
132.008
131.717
131.623
131.596
130.048
129.932
128.312
128.192
126.469
125.796
105.814
105.679
96.890
95.851
77.408
77.090
76.772
32.060
31.966
29.819
29.766
26.372
26.341
25.411
19.255
ppm (t1)

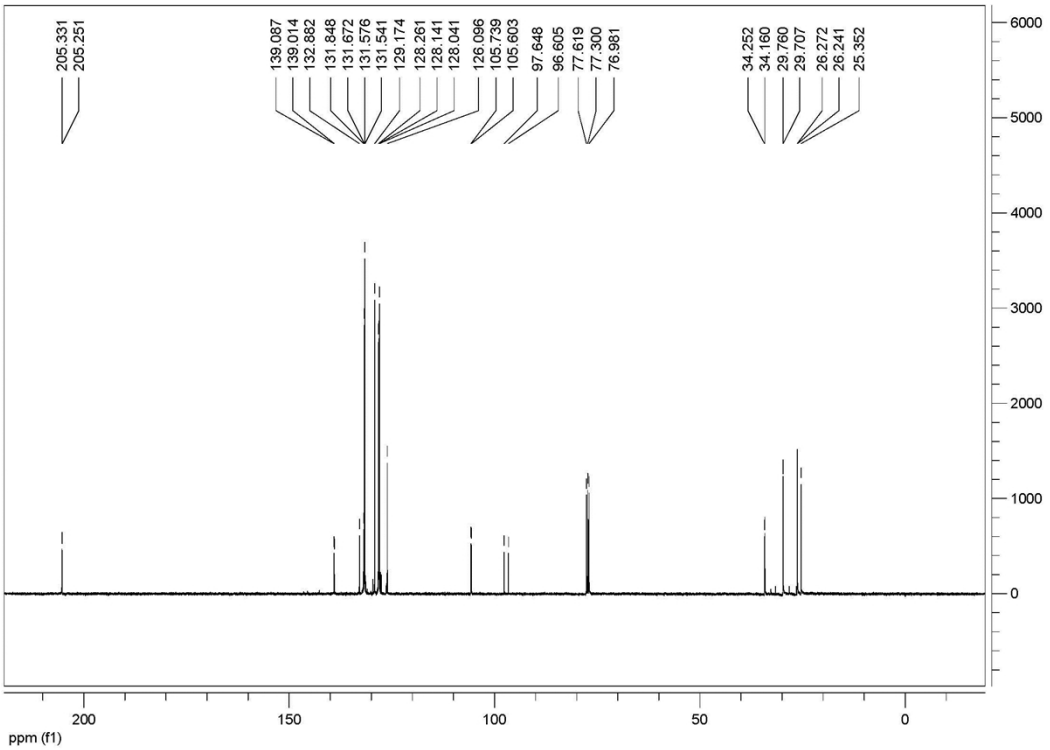


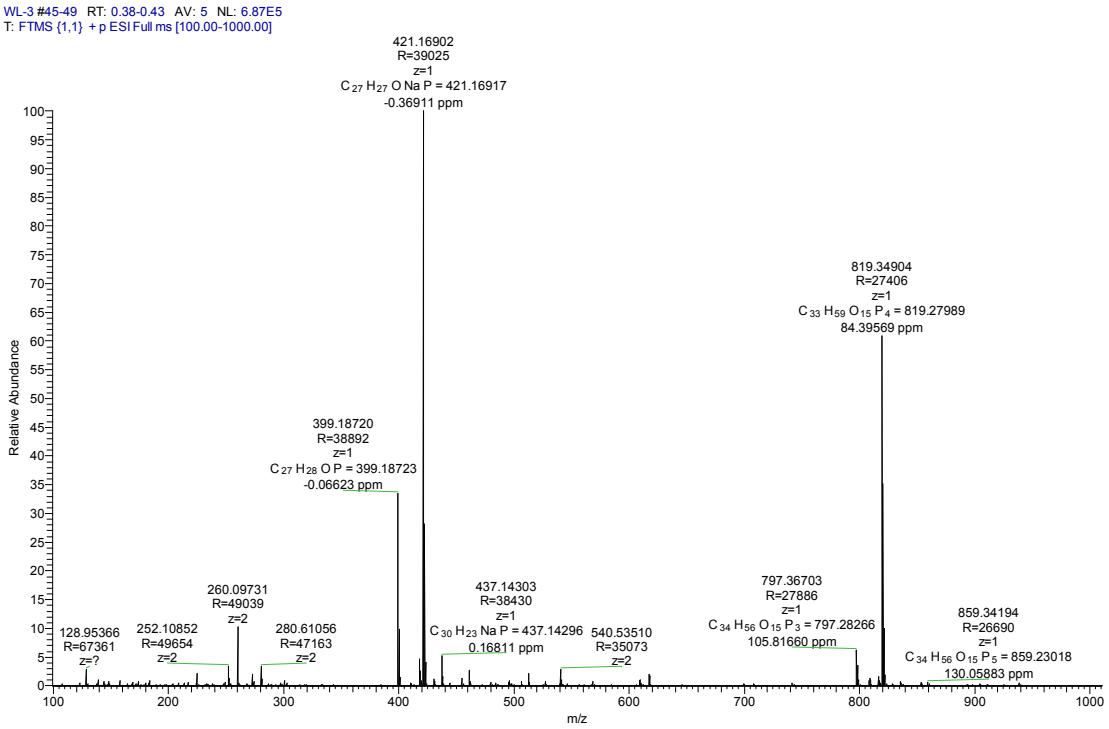
19 #41 RT: 0.36 AV: 1 NL: 6.89E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

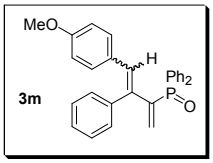


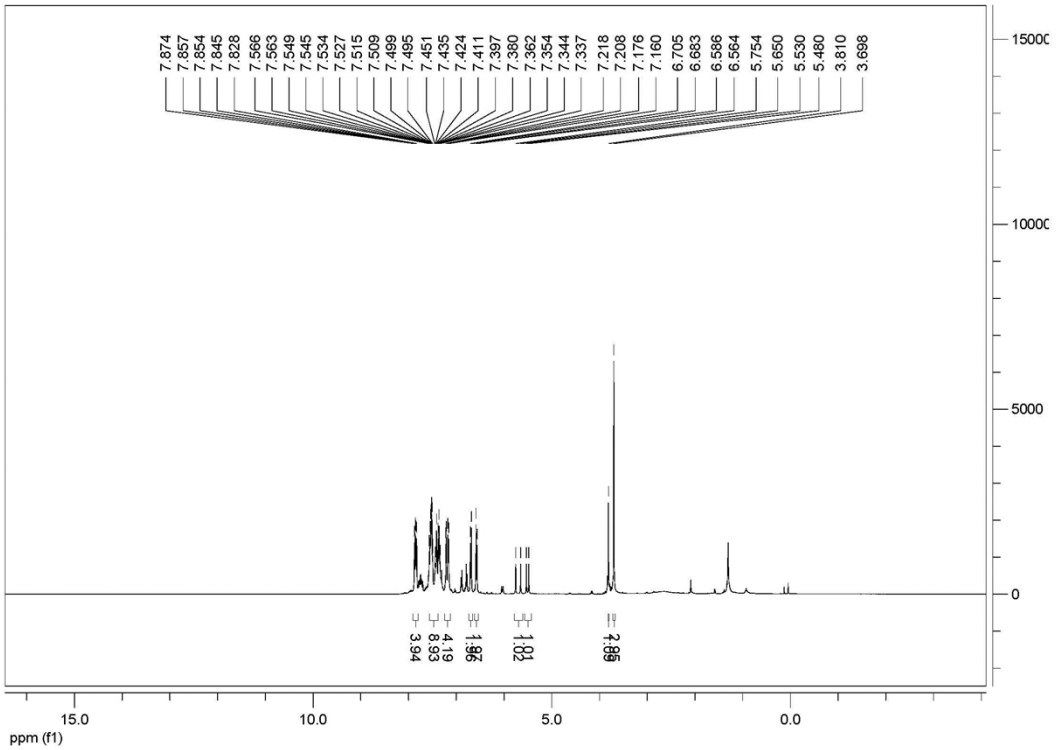


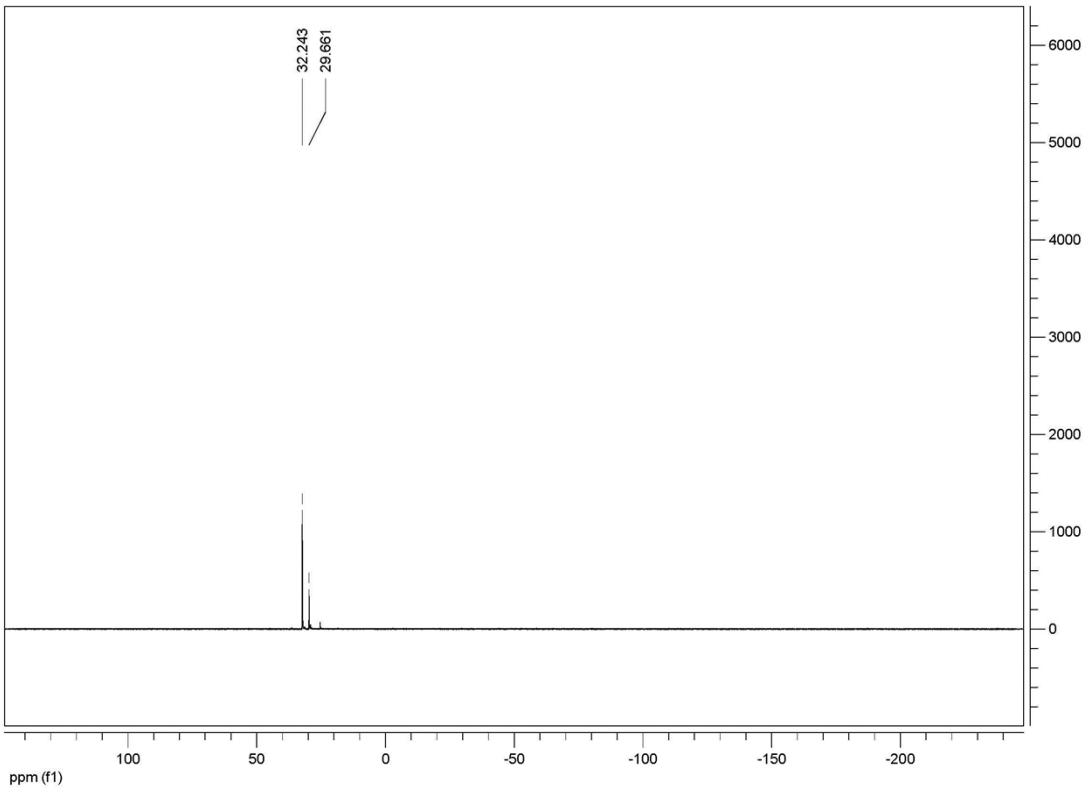

7.720
7.711
7.694
7.691
7.487
7.484
7.469
7.465
7.459
7.451
7.447
7.426
7.419
7.407
7.404
7.400
7.390
7.386
7.383
7.213
7.194
7.177
7.137
7.128
7.121
7.118
3.564
3.545
1.818
1.808
1.798
1.786
1.775
1.765
1.756
1.746
1.686
1.674
1.667
1.655
1.643
1.635
1.623
1.612
1.262
1.252
1.231
1.229
1.221
1.214
1.202
15000
10000
5000
0
4.08
6.24
4.63
2.00
2.01
6.07
15.0
10.0
5.0
0.0
ppm (f1)


205.331
205.251
139.087
139.014
132.882
131.848
131.672
131.576
131.541
129.174
128.261
128.141
128.041
126.096
105.739
105.603
97.648
96.605
77.619
77.300
76.981
34.252
34.160
29.760
29.707
26.272
26.241
25.352
6000
5000
4000
3000
2000
1000
0
200
150
100
50
0
ppm (f1)

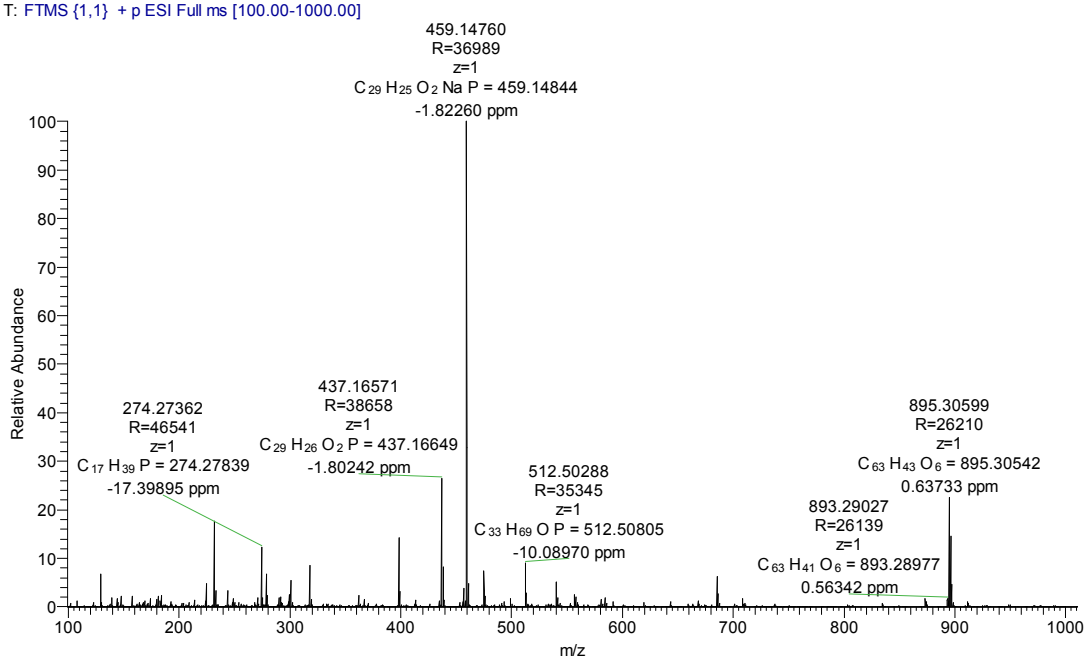

WL-3 #45-49 RT: 0.38-0.43 AV: 5 NL: 6.87E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]
421.16902
R=39025
z=1
C 27 H 27 O Na P = 421.16917
-0.36911 ppm
819.34904
R=27406
z=1
C 33 H 59 O 15 P 4 = 819.27989
84.39569 ppm
399.18720
R=38892
z=1
C 27 H 28 O P = 399.18723
-0.06623 ppm
260.09731
R=49039
z=2
437.14303
R=38430
z=1
C 30 H 23 Na P = 437.14296
0.16811 ppm
797.36703
R=27886
z=1
C 34 H 56 O 15 P 3 = 797.28266
105.81660 ppm
859.34194
R=26690
z=1
C 34 H 56 O 15 P 5 = 859.23018
130.05883 ppm
128.95366
R=67361
z=?
252.10852
R=49654
z=2
280.61056
R=47163
z=2
540.53510
R=35073
z=2
Relative Abundance
m/z

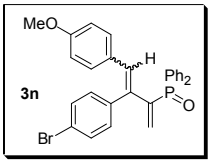

MeO
H
Ph2
P
O
3m

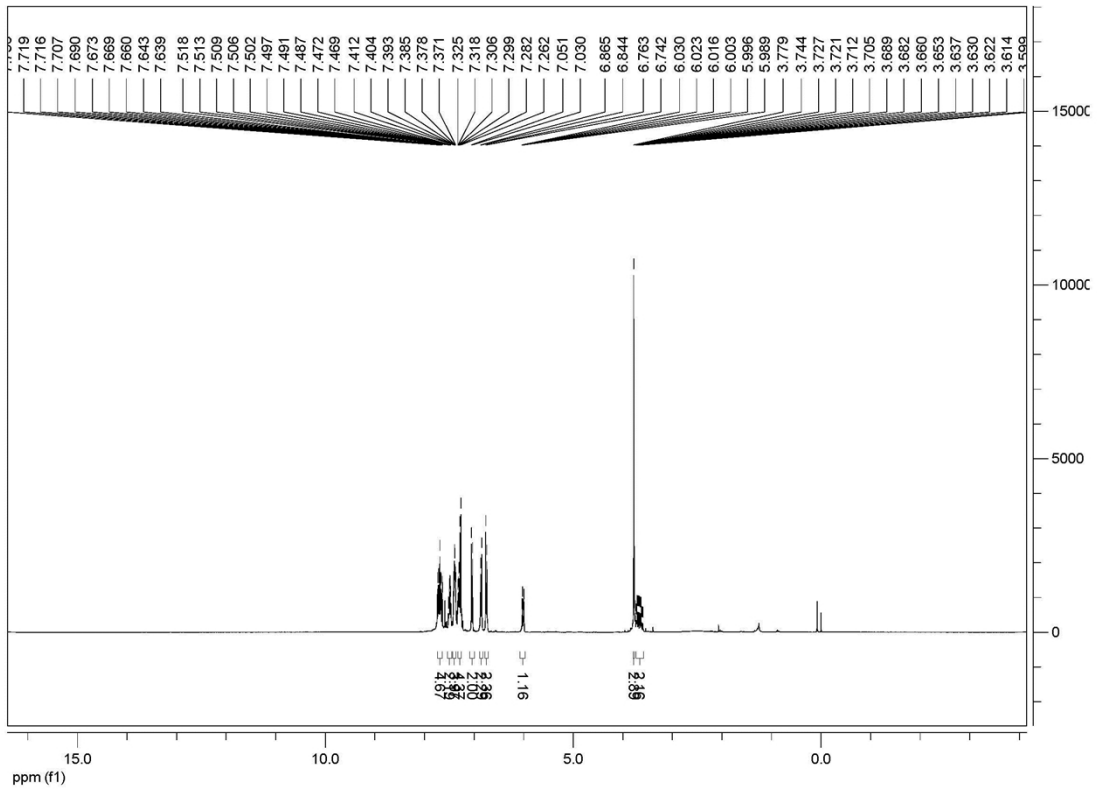

7.874
7.857
7.854
7.845
7.828
7.566
7.563
7.549
7.545
7.534
7.527
7.515
7.509
7.499
7.495
7.451
7.435
7.424
7.411
7.397
7.380
7.362
7.354
7.344
7.337
7.218
7.208
7.176
7.160
6.705
6.683
6.586
6.564
5.754
5.650
5.530
5.480
3.810
3.698
3.94
8.63
4.19
1.96
1.97
1.02
1.01
2.85
2.89
15.0
10.0
5.0
0.0
ppm (f1)

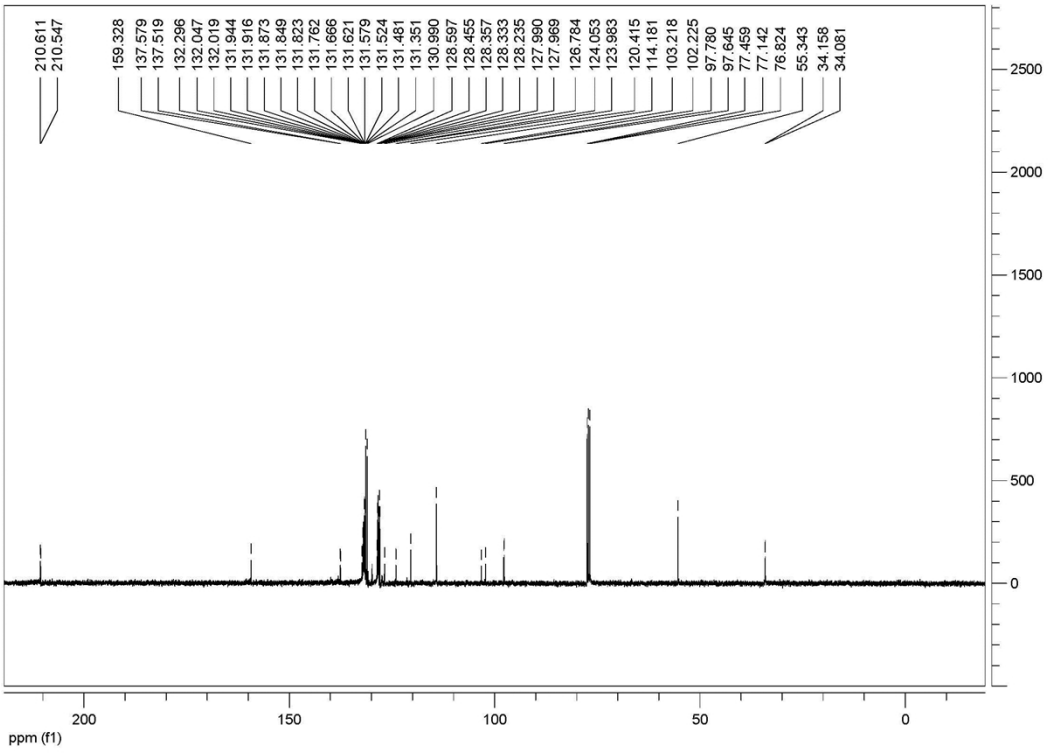


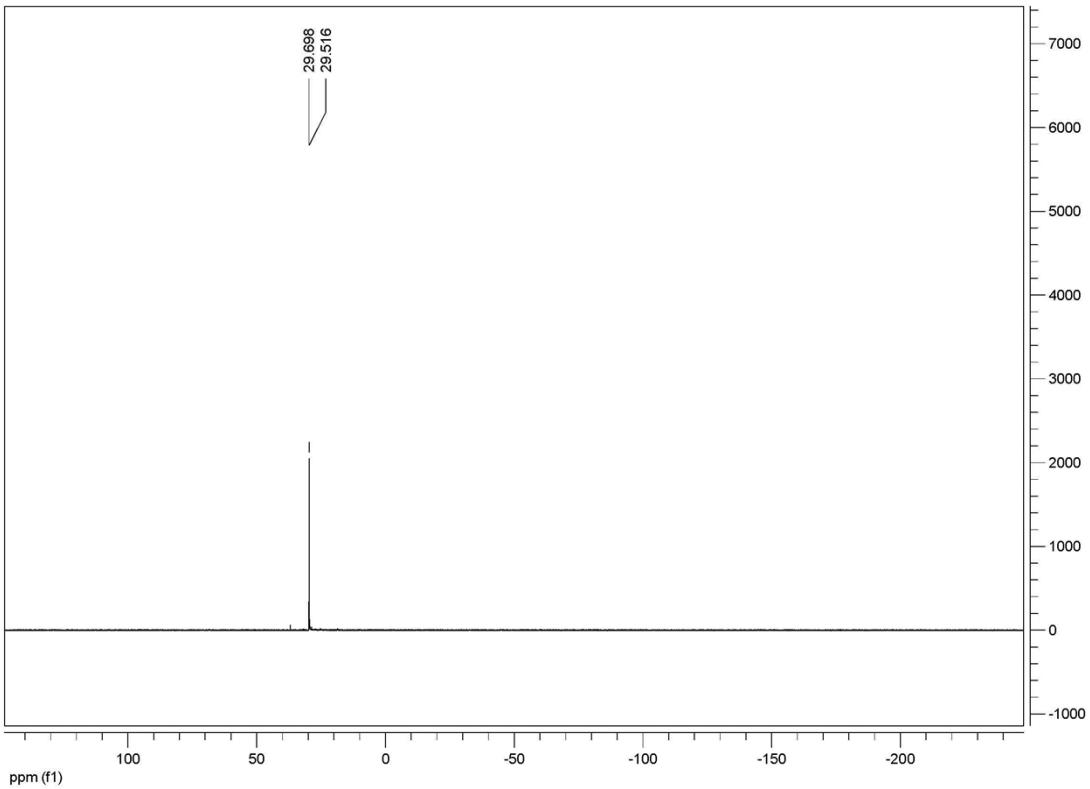
23 #45-47 RT: 0.39-0.42 AV: 3 NL: 2.68E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]



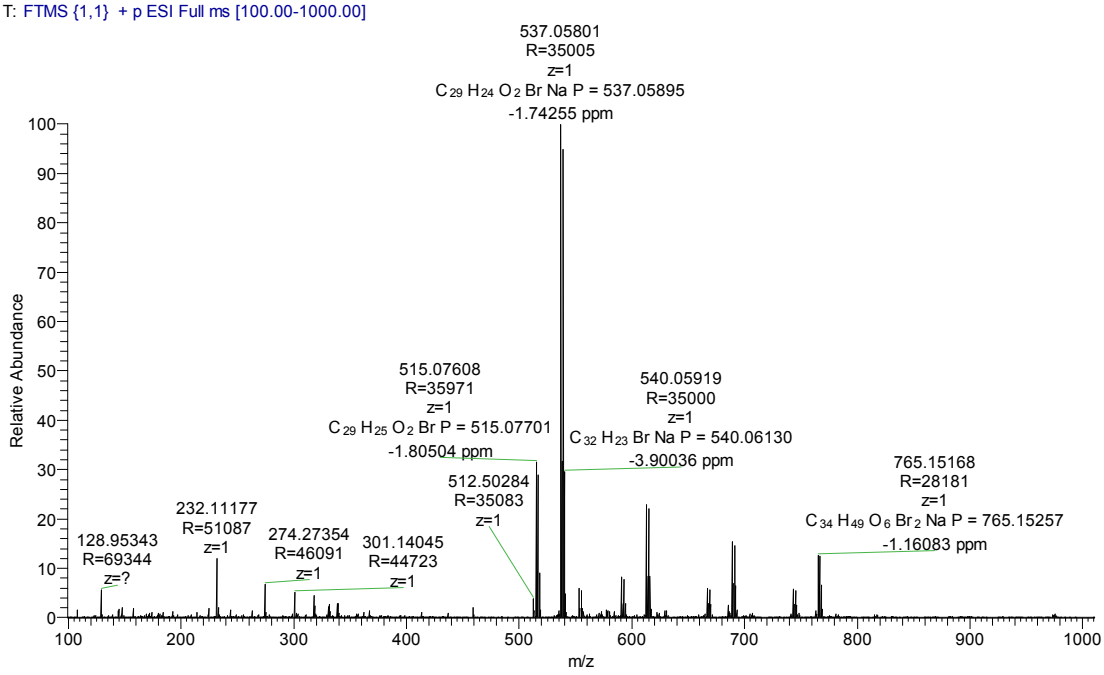


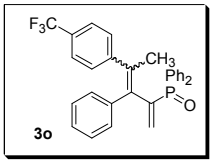

7.719
7.716
7.707
7.690
7.673
7.669
7.660
7.643
7.639
7.518
7.513
7.509
7.506
7.502
7.497
7.491
7.487
7.472
7.469
7.412
7.404
7.393
7.385
7.378
7.371
7.325
7.318
7.306
7.299
7.282
7.262
7.051
7.030
6.885
6.844
6.763
6.742
6.030
6.023
6.016
6.003
5.996
5.989
3.779
3.744
3.727
3.721
3.712
3.705
3.689
3.682
3.660
3.653
3.637
3.630
3.622
3.614
1.19
2.16
15000
10000
5000
0
15.0
10.0
5.0
0.0
ppm (f1)

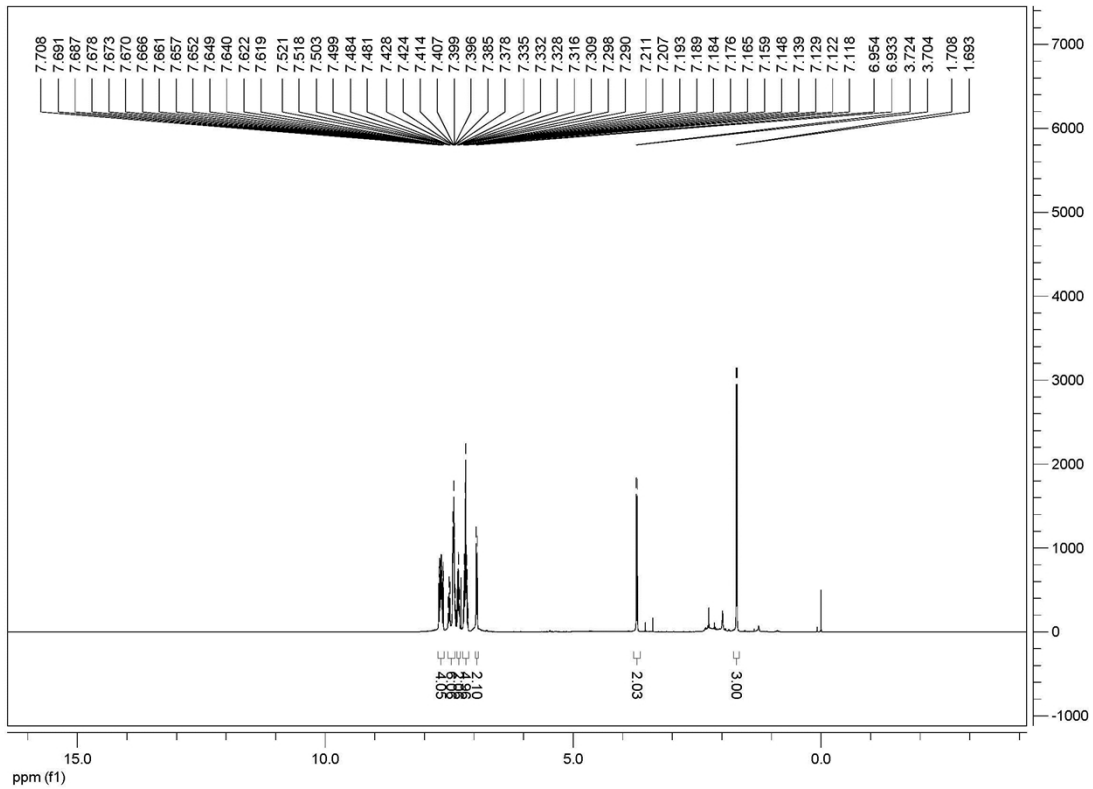

210.611
210.547
159.328
137.579
137.519
132.296
132.047
132.019
131.944
131.916
131.873
131.849
131.823
131.762
131.666
131.621
131.579
131.524
131.481
131.351
130.990
128.597
128.455
128.357
128.333
128.235
127.990
127.969
126.784
124.053
123.983
120.415
114.181
103.218
102.225
97.780
97.645
77.459
77.142
76.824
55.343
34.158
34.081
2500
2000
1500
1000
500
0
200
150
100
50
0
ppm (f1)

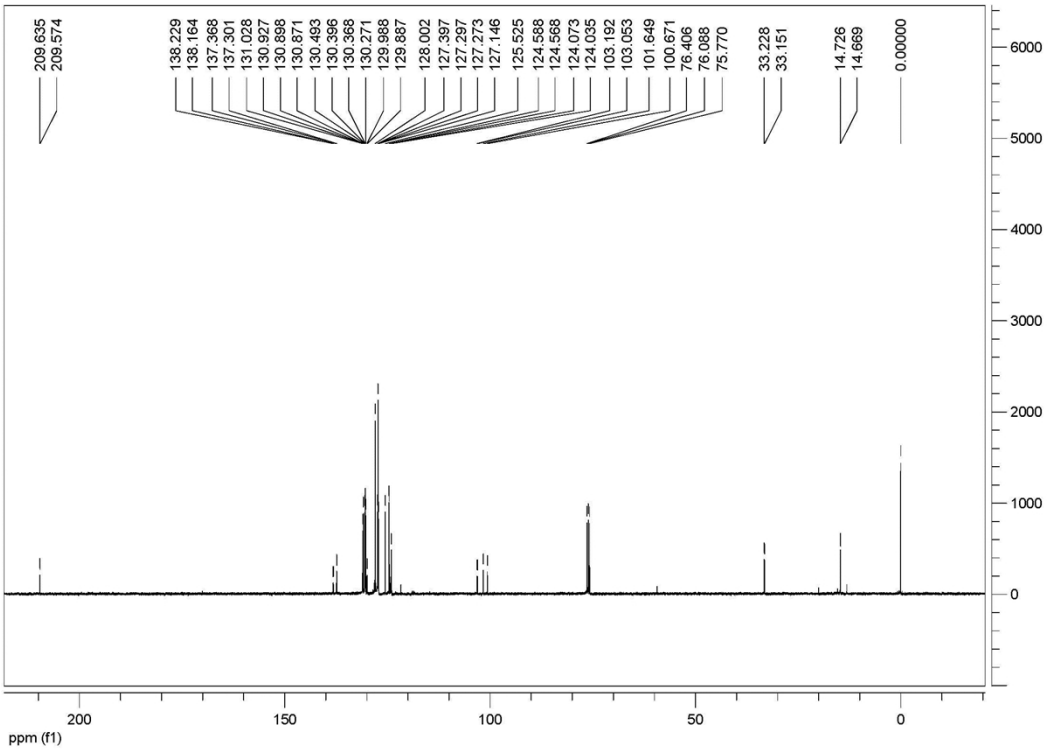


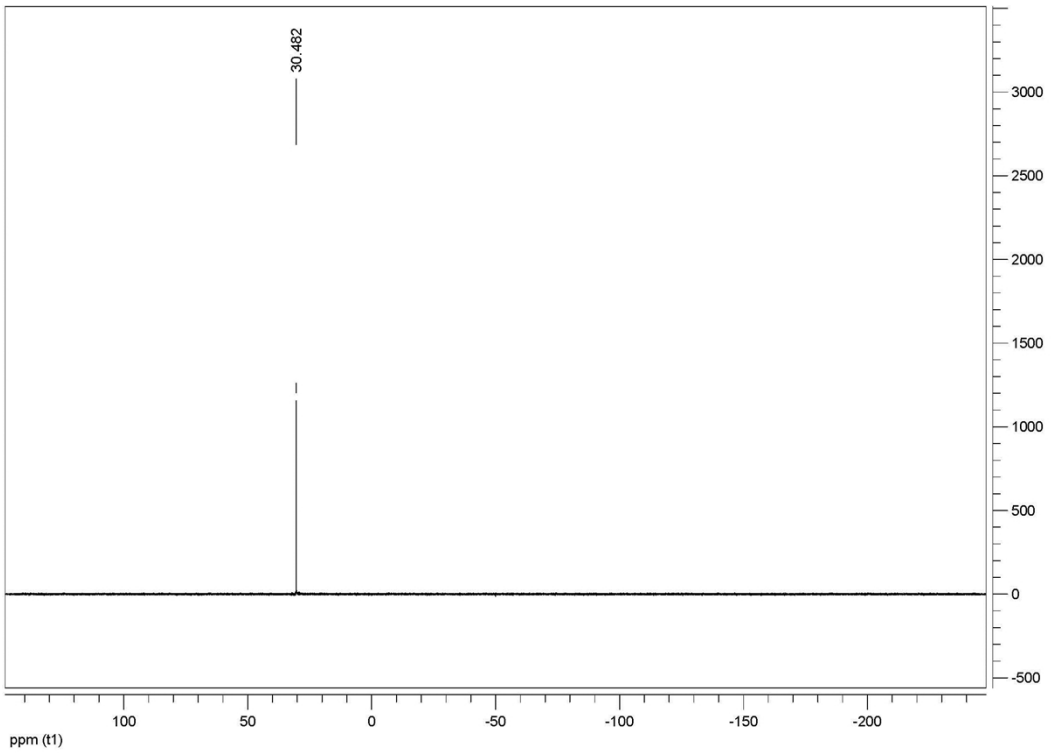
24 #53-56 RT: 0.45-0.48 AV: 4 NL: 2.52E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]



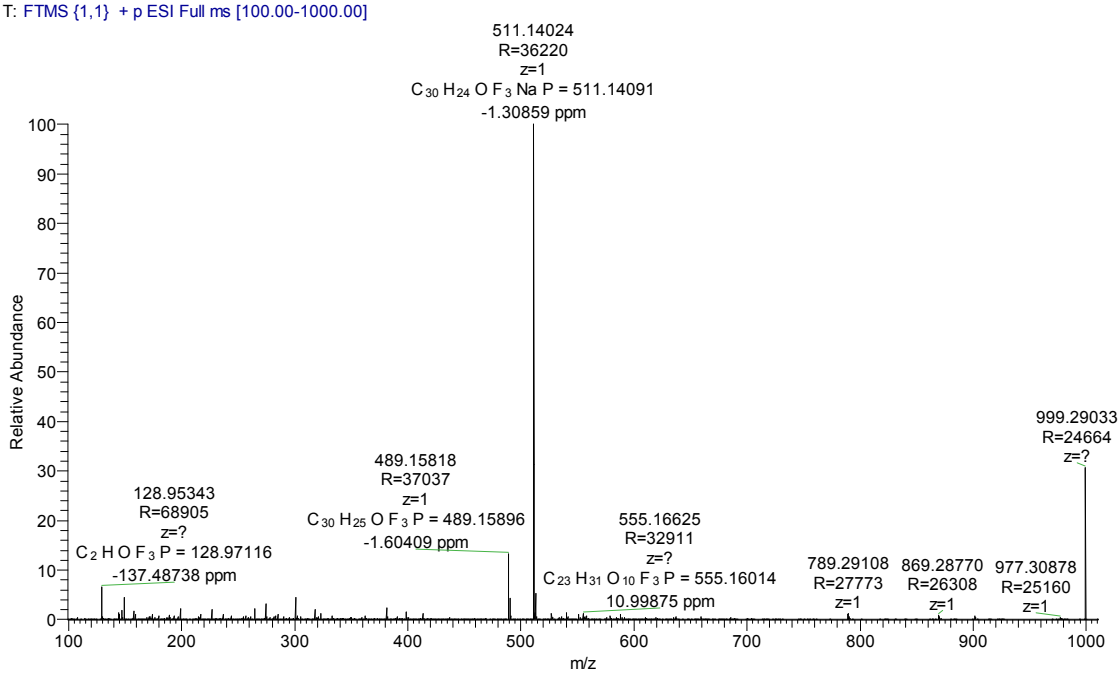


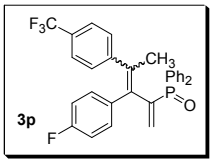

7.708
7.691
7.687
7.678
7.673
7.670
7.666
7.661
7.657
7.652
7.649
7.640
7.622
7.619
7.521
7.518
7.503
7.499
7.484
7.481
7.428
7.424
7.414
7.407
7.399
7.396
7.385
7.378
7.335
7.332
7.328
7.316
7.309
7.298
7.290
7.211
7.207
7.193
7.189
7.184
7.176
7.165
7.159
7.148
7.139
7.129
7.122
7.118
6.954
6.933
3.724
3.704
1.708
1.693
2.10
2.03
3.00
ppm (f1)

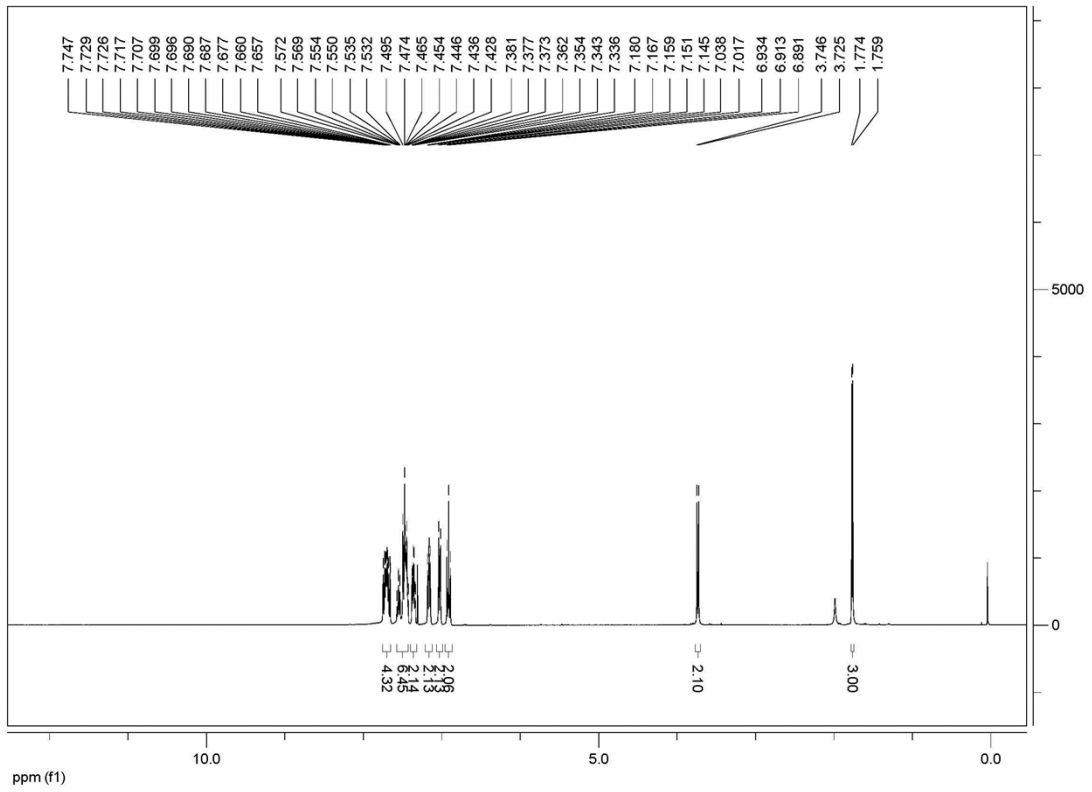

209.635
209.574
138.229
138.164
137.368
137.301
131.028
130.927
130.898
130.871
130.493
130.396
130.368
130.271
129.988
129.887
128.002
127.397
127.297
127.273
127.146
125.525
124.588
124.568
124.073
124.035
103.192
103.053
101.649
100.671
76.406
76.088
75.770
33.228
33.151
14.726
14.669
0.00000
ppm (f1)

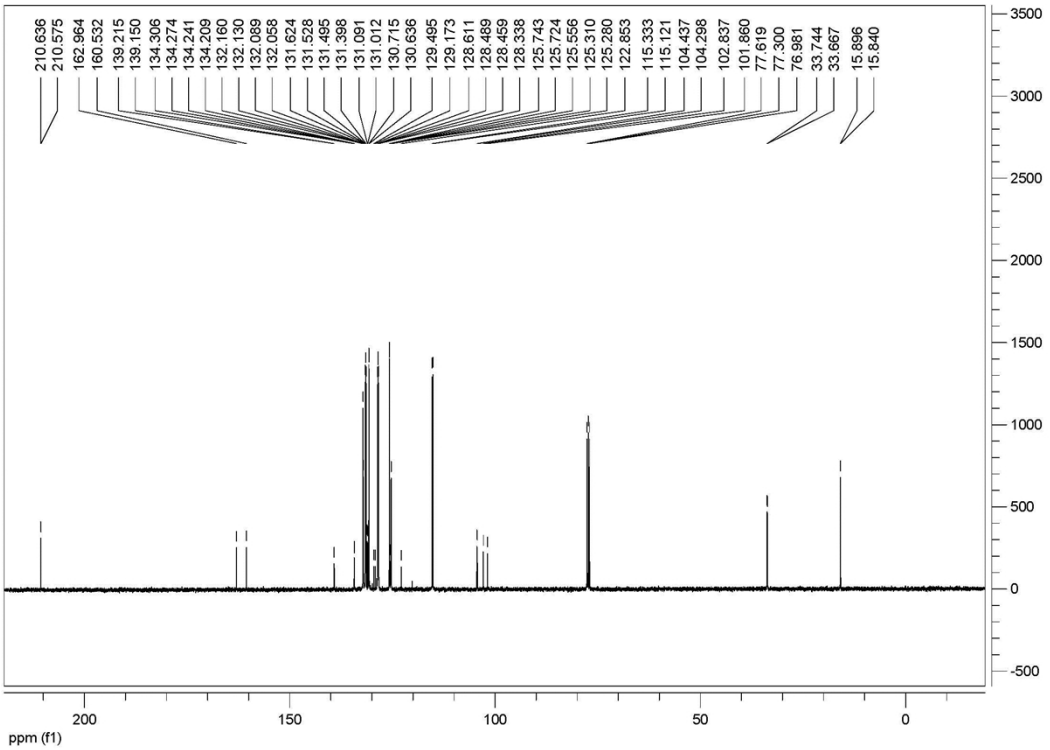


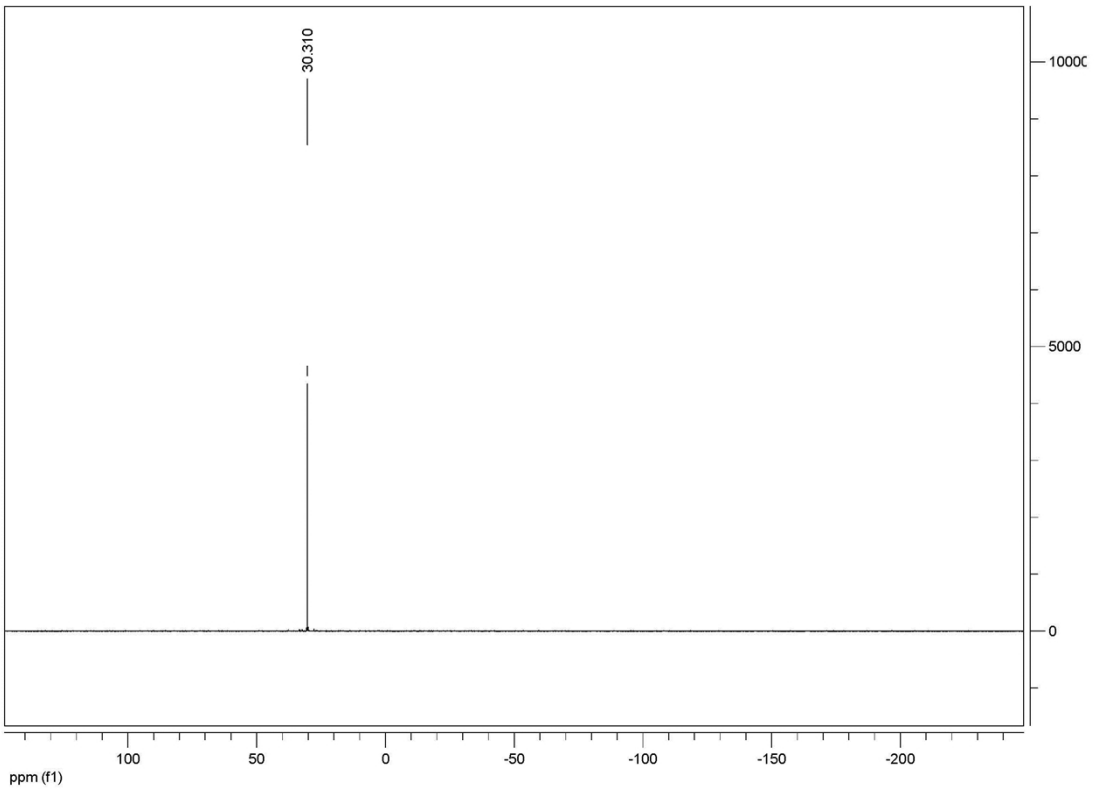
7 #42-43 RT: 0.37-0.39 AV: 2 NL: 6.00E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]



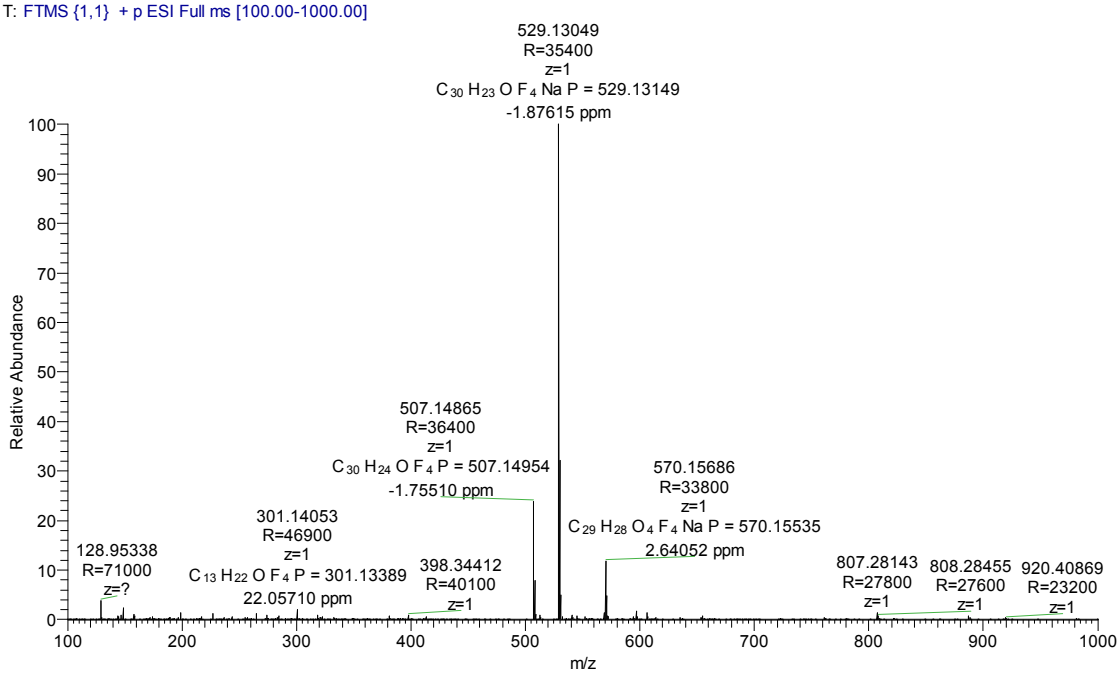


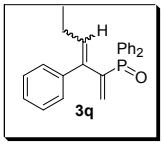

7.747
7.729
7.726
7.717
7.707
7.699
7.696
7.690
7.687
7.677
7.660
7.657
7.572
7.569
7.554
7.550
7.535
7.532
7.495
7.474
7.465
7.454
7.446
7.436
7.428
7.381
7.377
7.373
7.362
7.354
7.343
7.336
7.180
7.167
7.159
7.151
7.145
7.038
7.017
6.934
6.913
6.891
3.746
3.725
1.774
1.759
5000
0
4.32
6.45
2.14
2.13
2.06
2.03
2.10
3.00
10.0
5.0
0.0
ppm (f1)

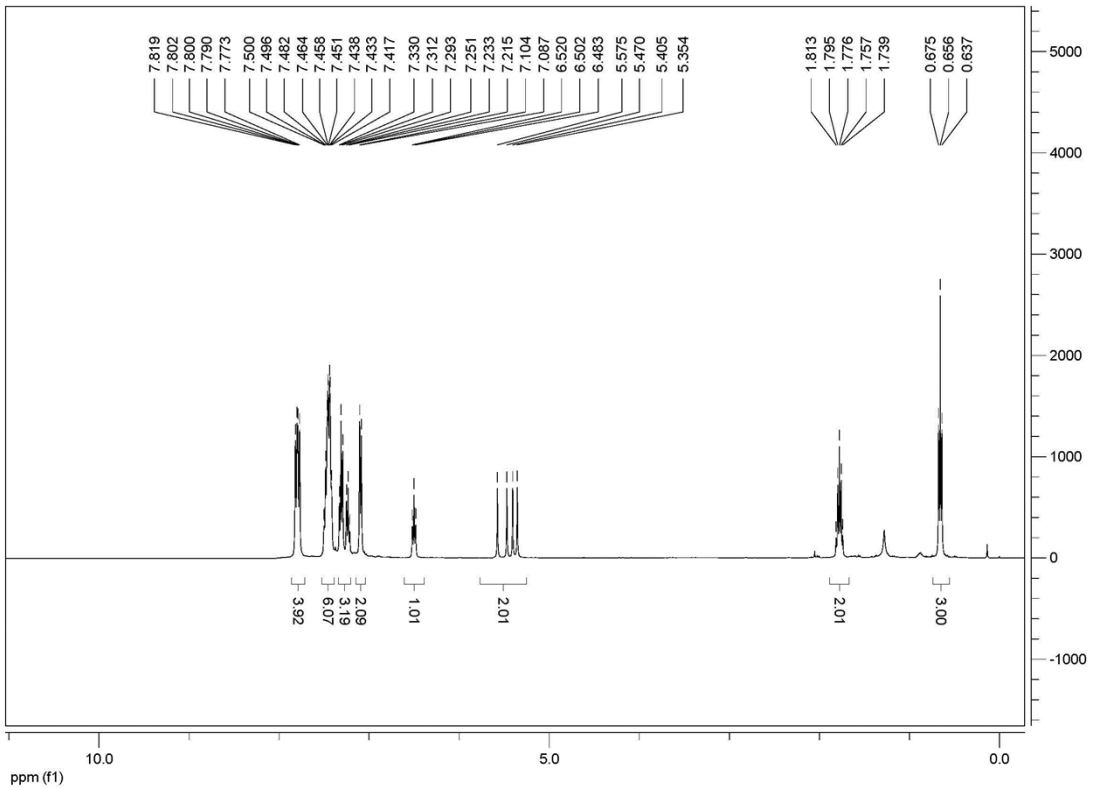

210.636
210.575
162.964
160.532
139.215
139.150
134.306
134.274
134.241
134.209
132.160
132.130
132.089
132.058
131.624
131.528
131.495
131.398
131.091
131.012
130.715
130.636
129.495
129.173
128.611
128.489
128.459
128.338
125.743
125.724
125.556
125.310
125.280
122.853
115.333
115.121
104.437
104.298
102.837
101.860
77.619
77.300
76.981
33.744
33.667
15.896
15.840
3500
3000
2500
2000
1500
1000
500
0
-500
200
150
100
50
0
ppm (f1)

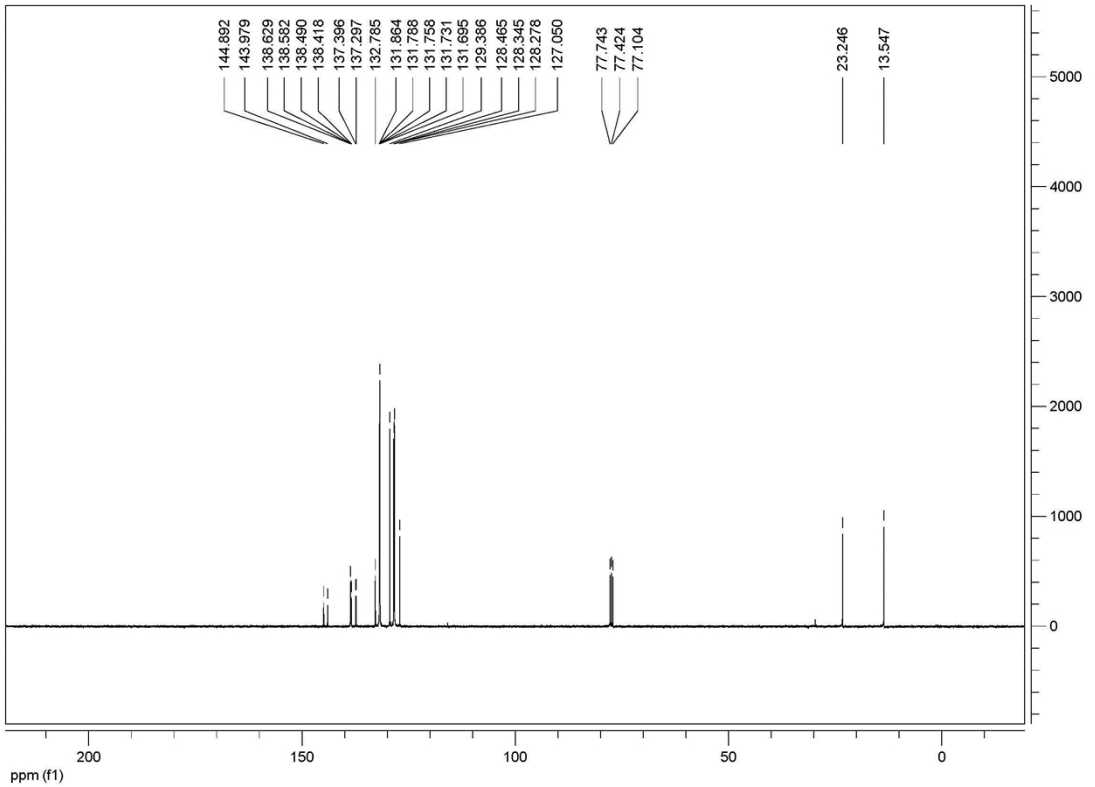


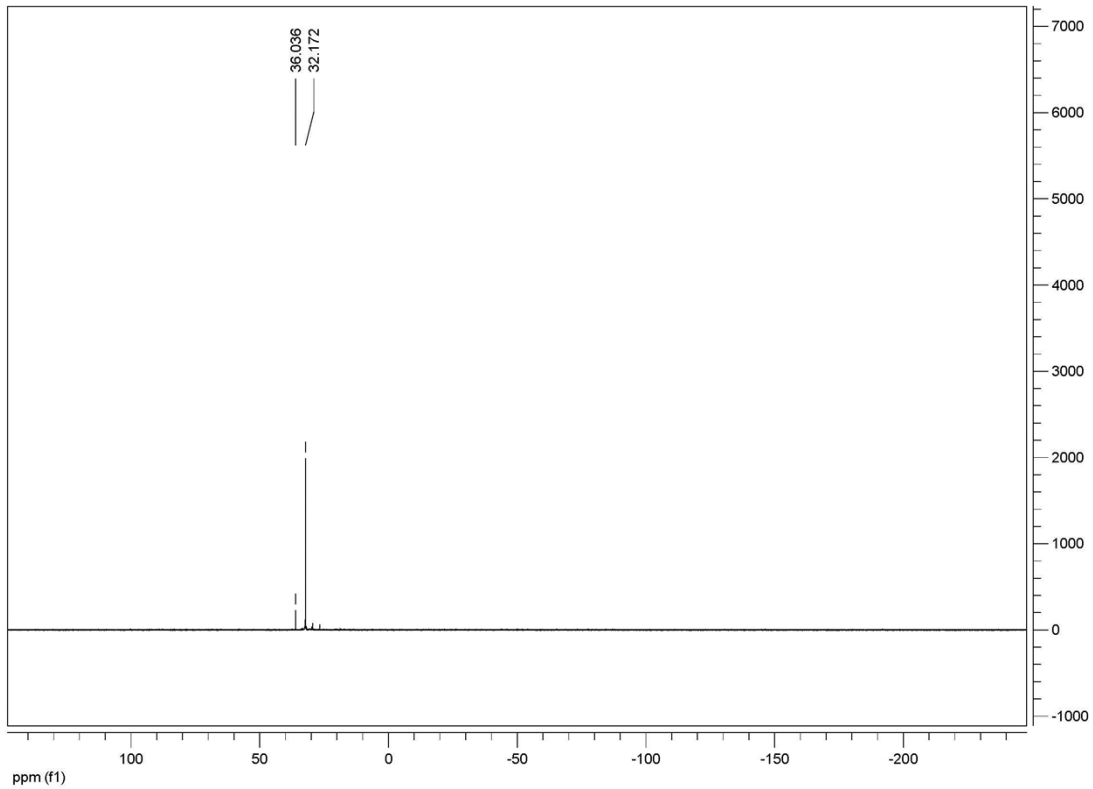
8 #49 RT: 0.41 AV: 1 NL: 7.83E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]



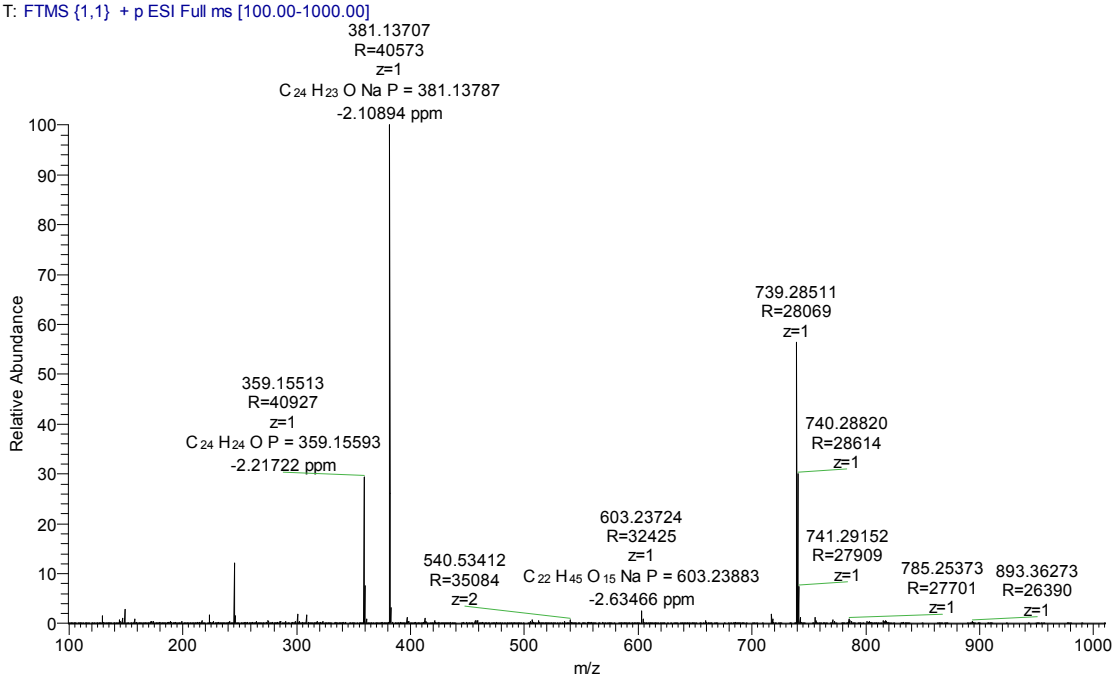


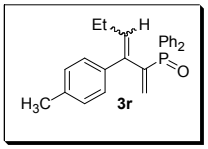

7.819
7.802
7.800
7.790
7.773
7.500
7.496
7.482
7.464
7.458
7.451
7.438
7.433
7.417
7.330
7.312
7.293
7.251
7.233
7.215
7.104
7.087
6.520
6.502
6.483
5.575
5.470
5.405
5.354
1.813
1.795
1.776
1.757
1.739
0.675
0.656
0.637
3.92
6.07
3.19
2.09
1.01
2.01
2.01
3.00
5000
4000
3000
2000
1000
0
-1000
10.0
5.0
0.0
ppm (f1)

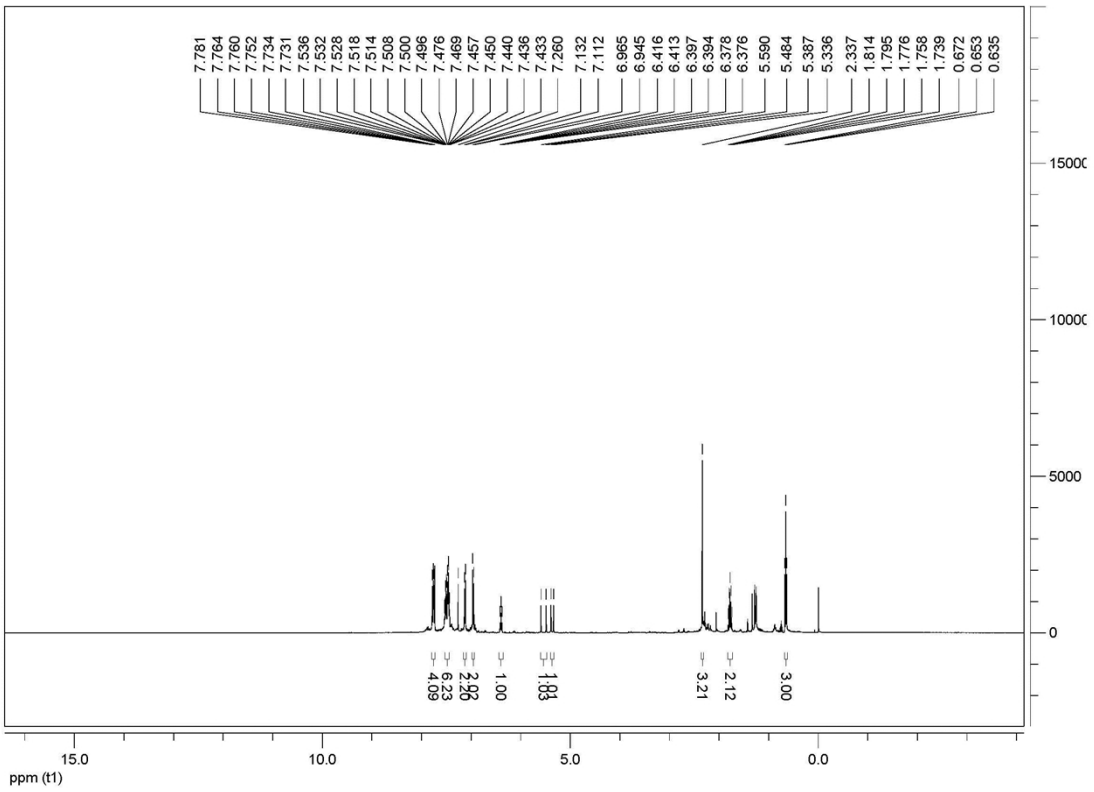

144.892
143.979
138.629
138.582
138.490
138.418
137.396
137.297
132.785
131.864
131.788
131.758
131.731
131.695
129.386
128.465
128.345
128.278
127.050
77.743
77.424
77.104
23.246
13.547
5000
4000
3000
2000
1000
0
200
150
100
50
0
ppm (f1)

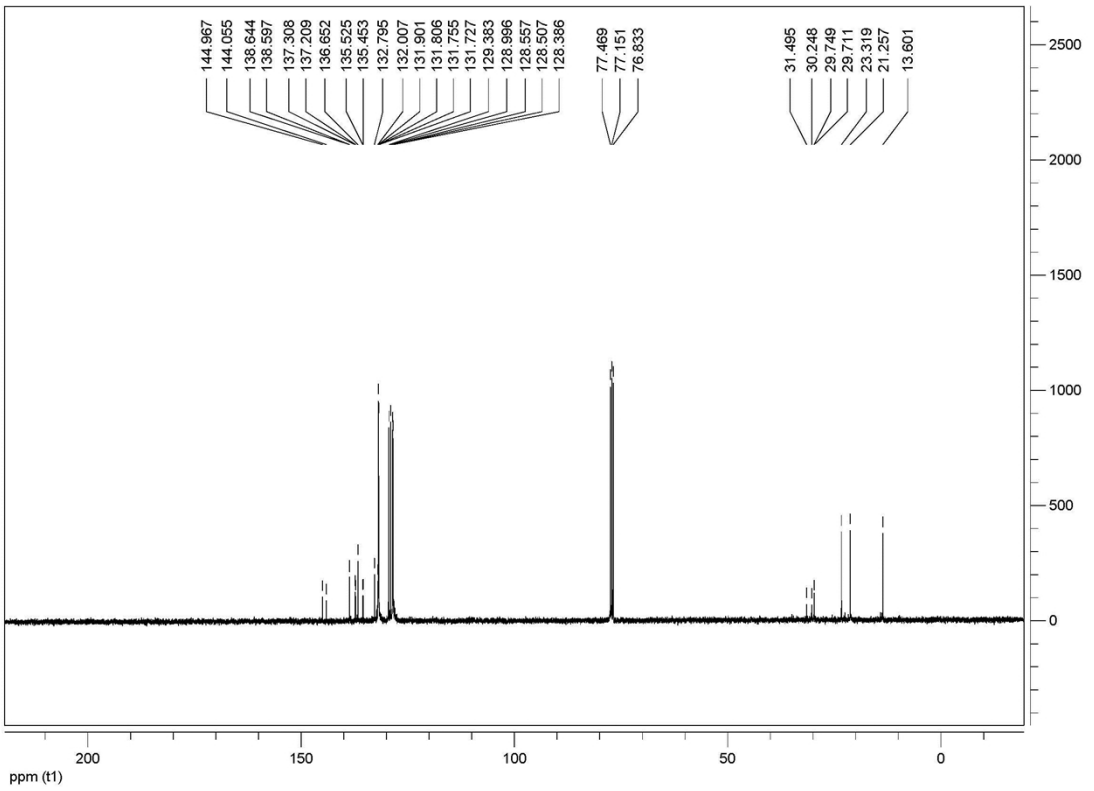


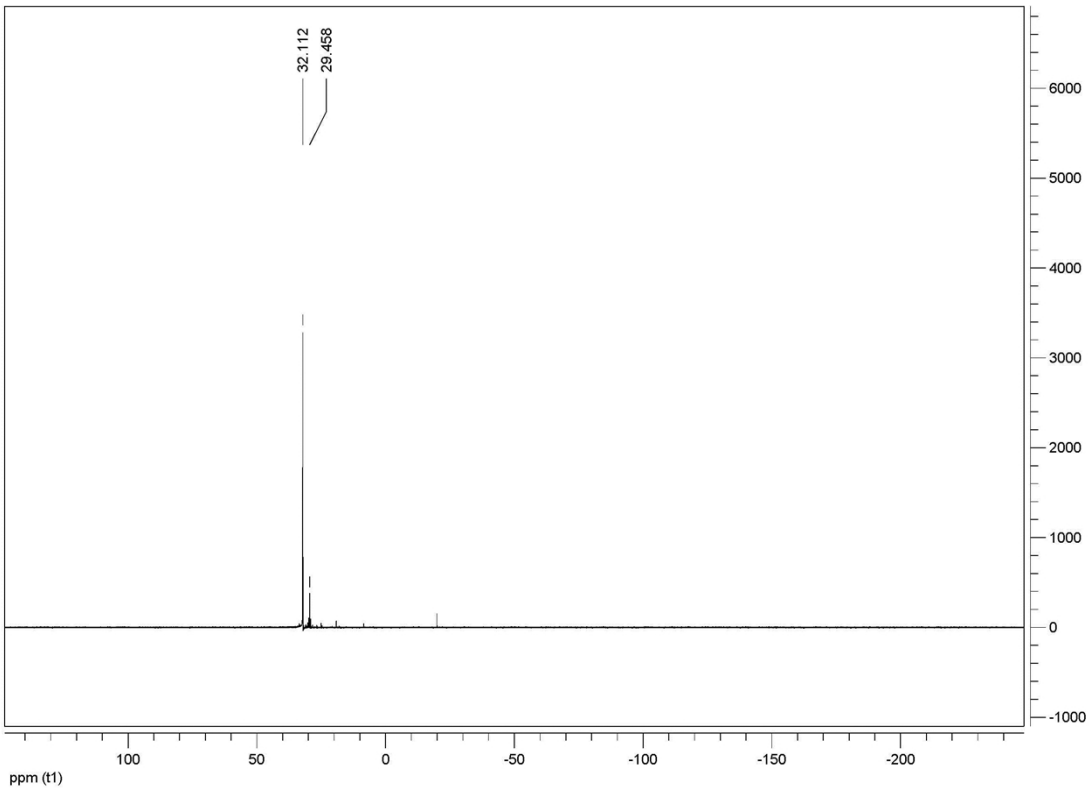
9 #48-50 RT: 0.40-0.42 AV: 3 NL: 1.40E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]



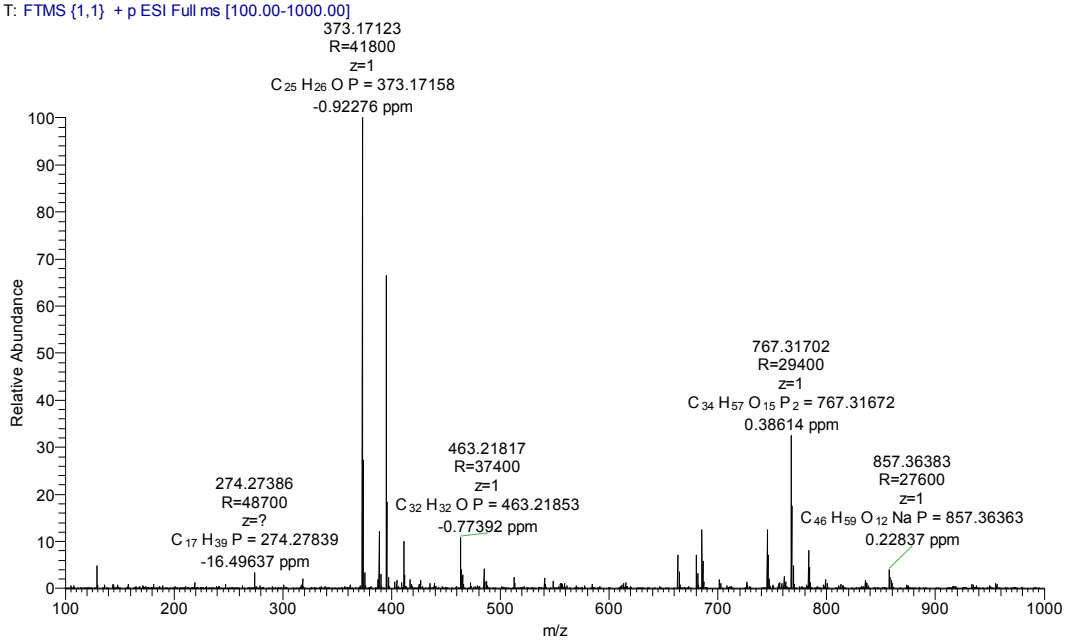


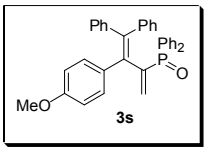

7.781
7.764
7.760
7.752
7.734
7.731
7.536
7.532
7.528
7.518
7.514
7.508
7.500
7.496
7.476
7.469
7.457
7.450
7.440
7.436
7.433
7.260
7.132
7.112
6.965
6.945
6.416
6.413
6.397
6.394
6.378
6.376
5.590
5.484
5.387
5.336
2.337
1.814
1.795
1.776
1.758
1.739
0.672
0.653
0.635
15000
10000
5000
0
4.09
6.23
2.00
2.26
1.00
1.03
1.01
3.21
2.12
3.00
15.0
10.0
5.0
0.0
ppm (t1)


144.967
144.055
138.644
138.597
137.308
137.209
136.652
135.525
135.453
132.795
132.007
131.901
131.806
131.755
131.727
129.383
128.996
128.557
128.507
128.386
77.469
77.151
76.833
31.495
30.248
29.749
29.711
23.319
21.257
13.601
2500
2000
1500
1000
500
0
200
150
100
50
0
ppm (t1)

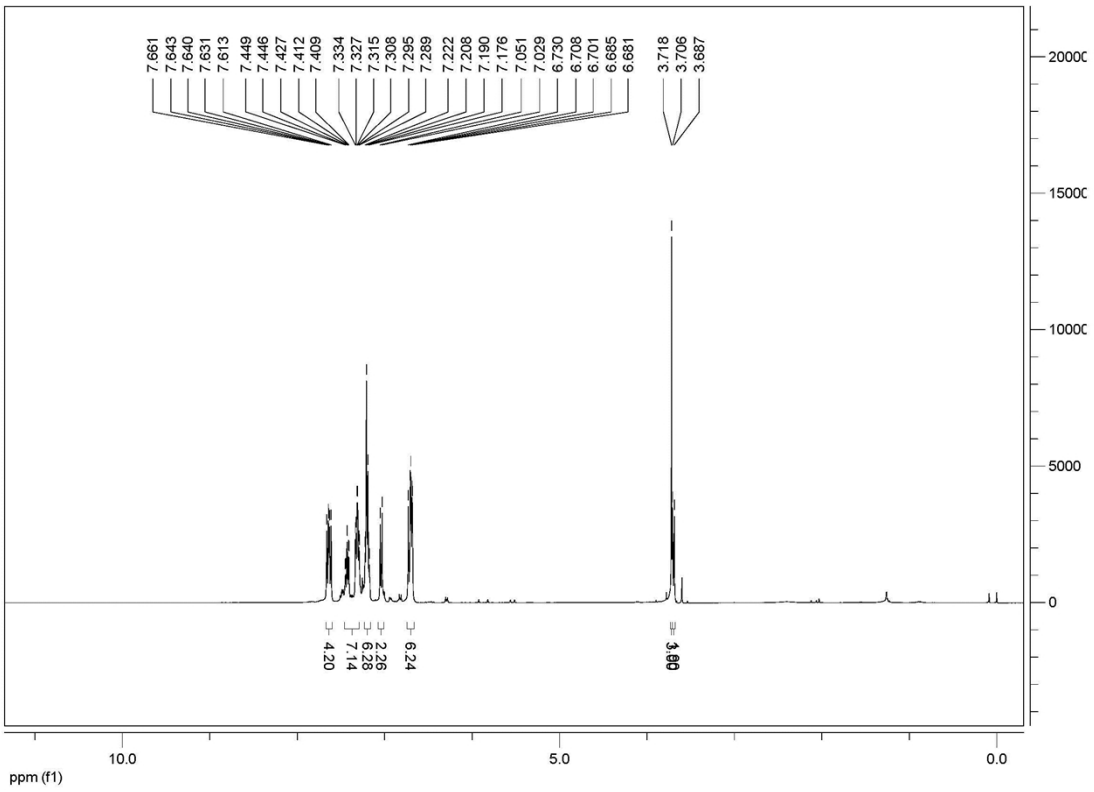


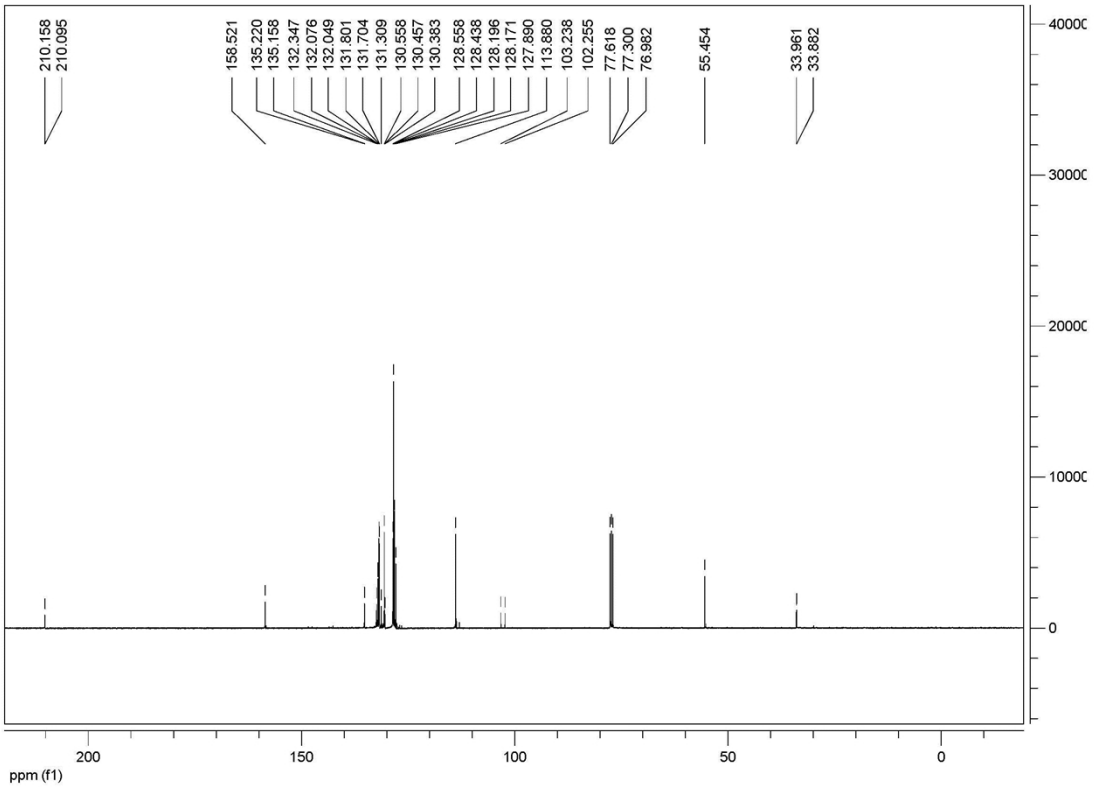
2 #55 RT: 0.51 AV: 1 NL: 2.28E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

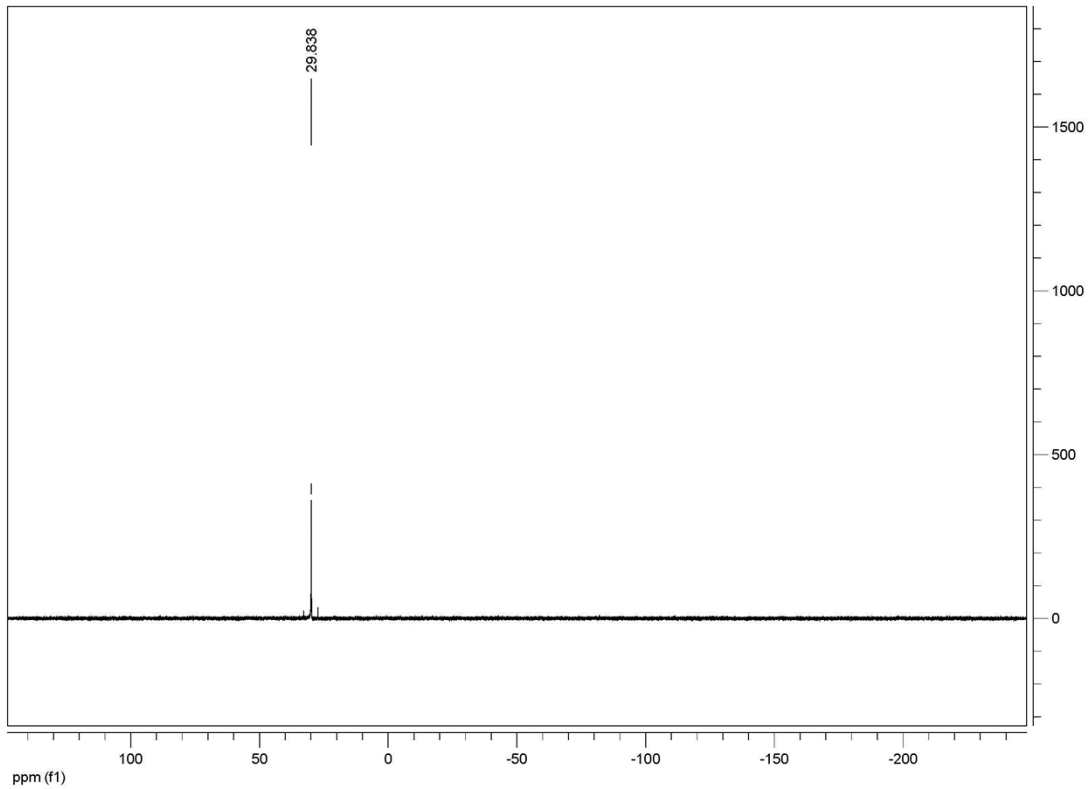




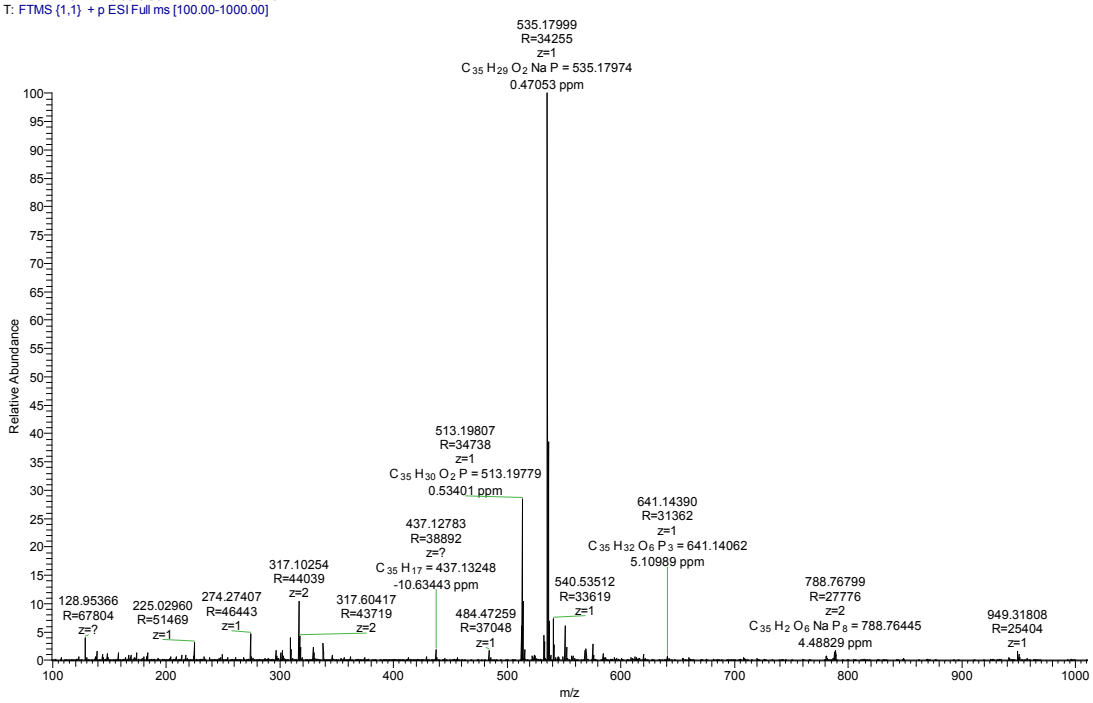
3s

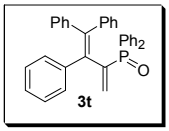

7.661
7.643
7.640
7.631
7.613
7.449
7.446
7.427
7.412
7.409
7.334
7.327
7.315
7.308
7.295
7.289
7.222
7.208
7.190
7.176
7.051
7.029
6.730
6.708
6.701
6.685
6.681
3.718
3.706
3.687
4.20
7.14
6.28
2.26
6.24
3.00
20000
15000
10000
5000
0
10.0
5.0
0.0
ppm (f1)


210.158
210.095
158.521
135.220
135.158
132.347
132.076
132.049
131.801
131.704
131.309
130.558
130.457
130.383
128.558
128.438
128.196
128.171
127.890
113.880
103.238
102.255
77.618
77.300
76.982
55.454
33.961
33.882
40000
30000
20000
10000
0
200
150
100
50
0
ppm (f1)

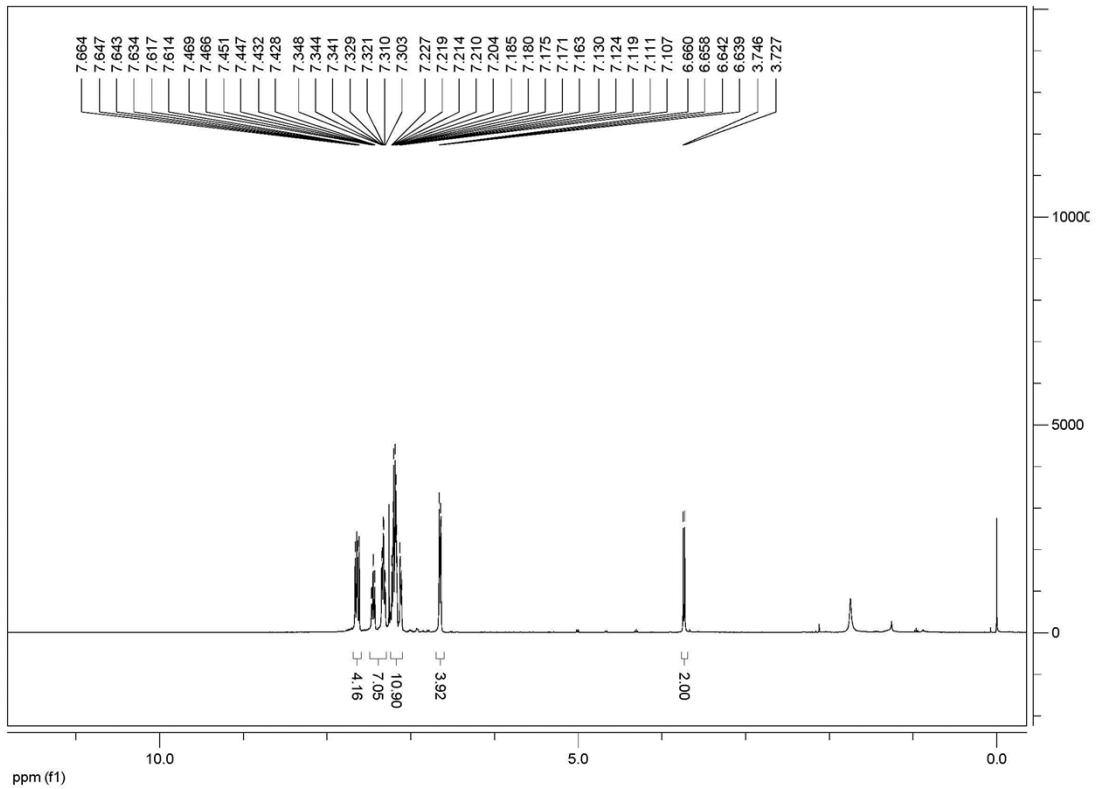


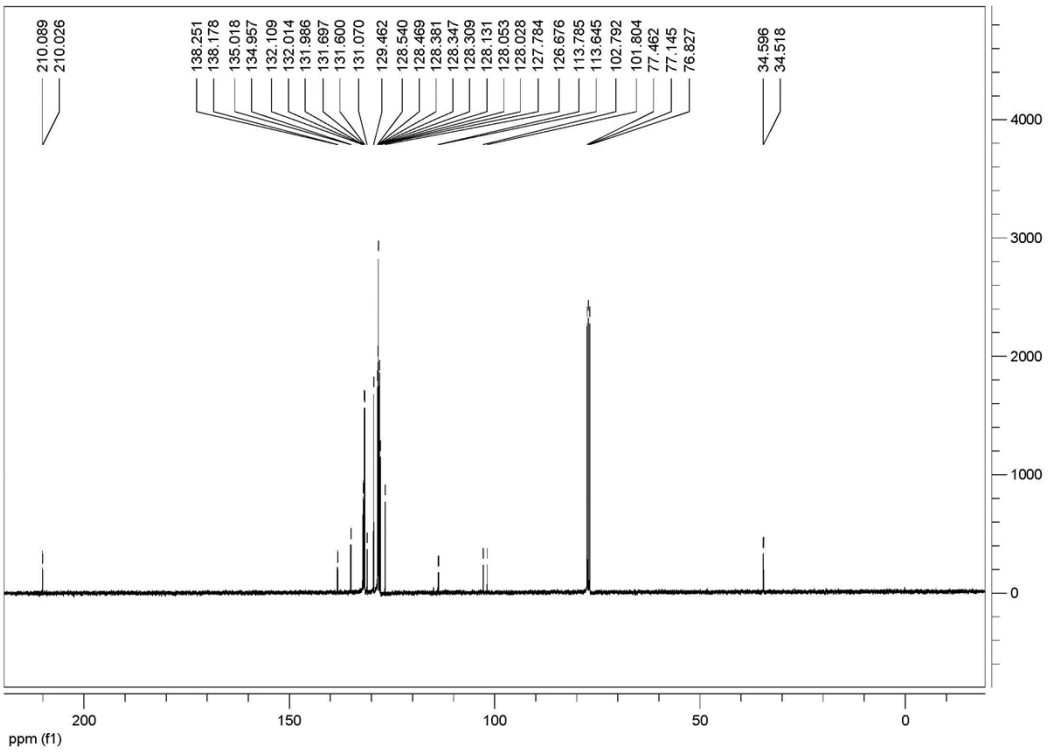
WL-4 #42-43 RT: 0.38-0.39 AV: 2 NL: 4.55E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

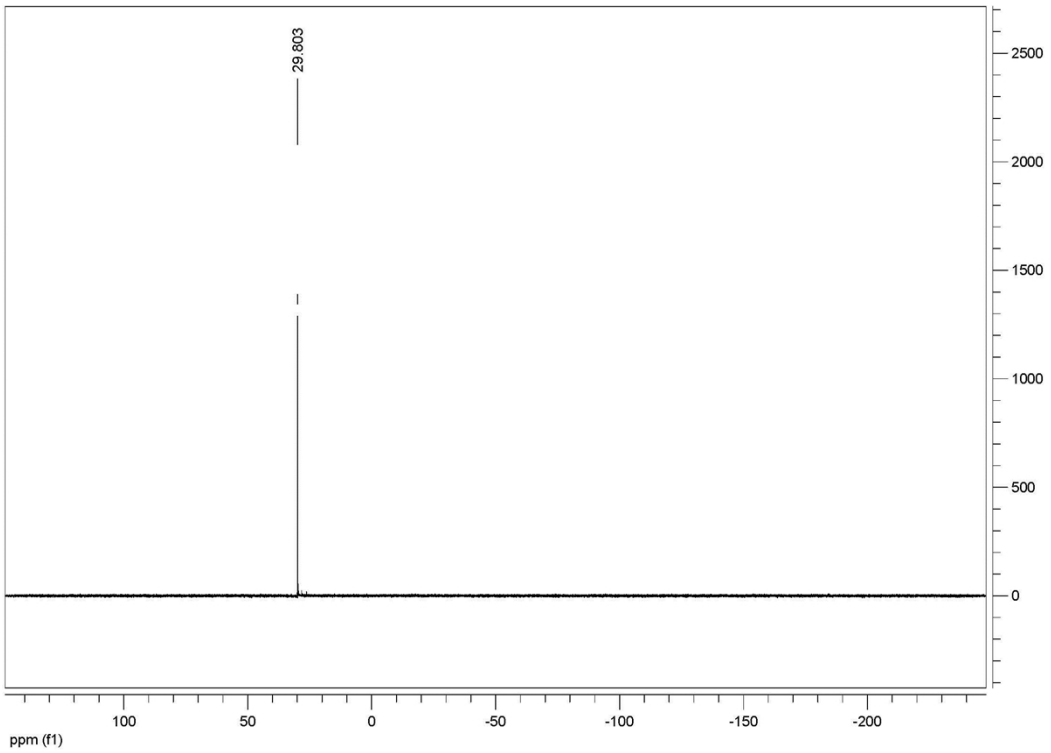


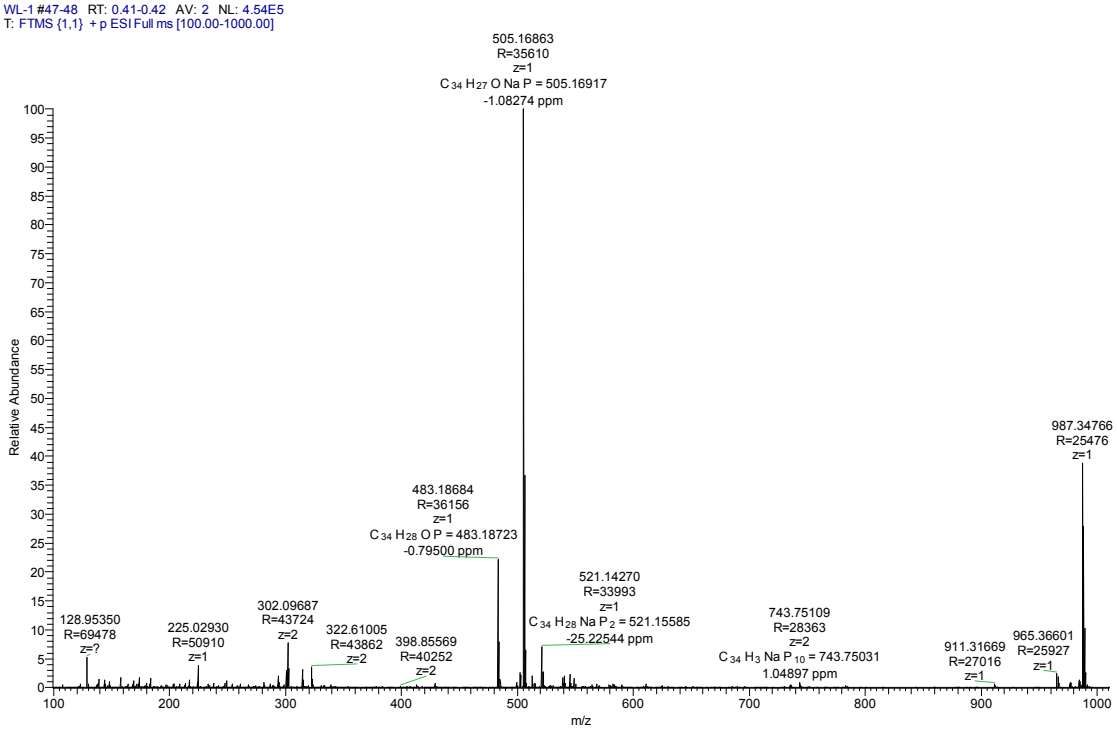


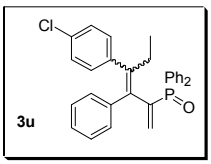
3t

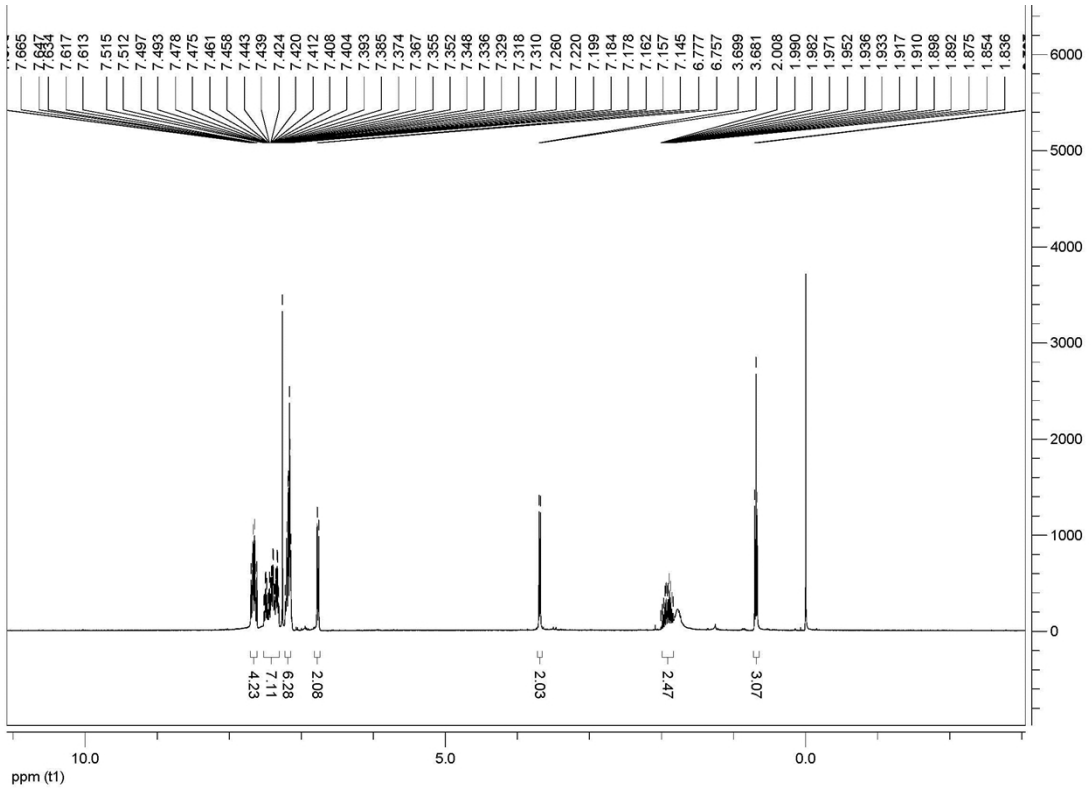

7.664
7.647
7.643
7.634
7.617
7.614
7.469
7.466
7.451
7.447
7.432
7.428
7.348
7.344
7.341
7.329
7.321
7.310
7.303
7.227
7.219
7.214
7.210
7.204
7.185
7.180
7.175
7.171
7.163
7.130
7.124
7.119
7.111
7.107
6.660
6.658
6.642
6.639
3.746
3.727
4.16
7.05
10.90
3.92
2.00
10000
5000
0
10.0
5.0
0.0
ppm (f1)

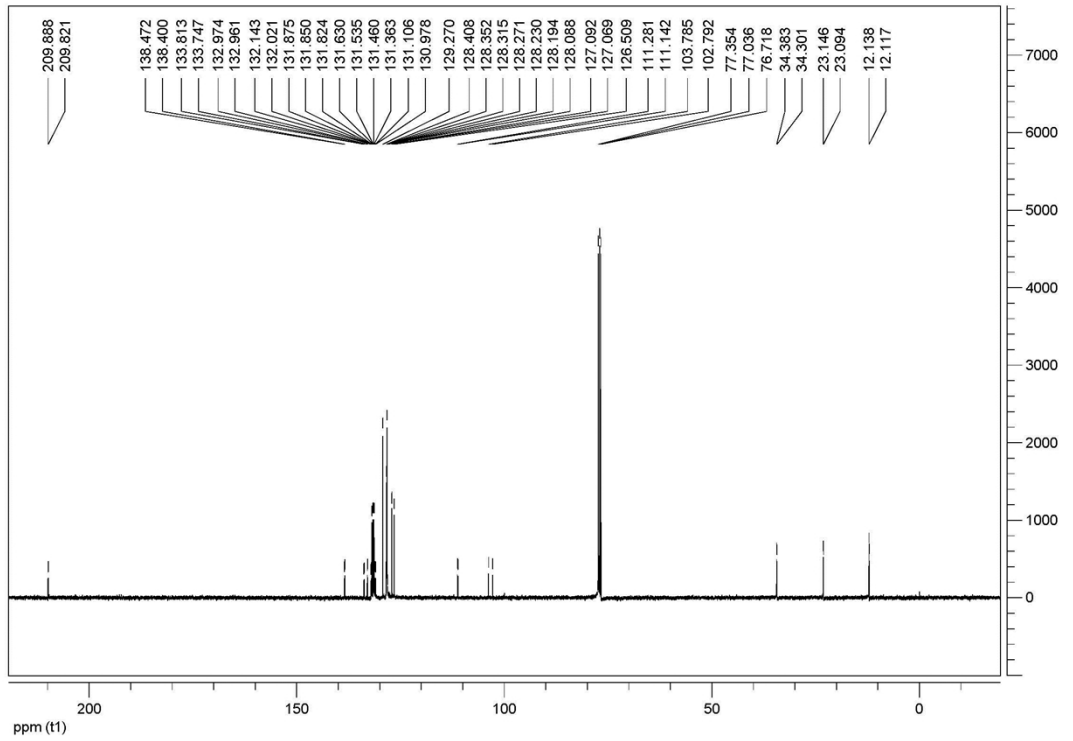

210.089
210.026
138.251
138.178
135.018
134.957
132.109
132.014
131.886
131.697
131.600
131.070
129.462
128.540
128.469
128.381
128.347
128.309
128.131
128.053
128.028
127.784
126.676
113.785
113.645
102.792
101.804
77.462
77.145
76.827
34.596
34.518
4000
3000
2000
1000
0
200
150
100
50
0
ppm (f1)

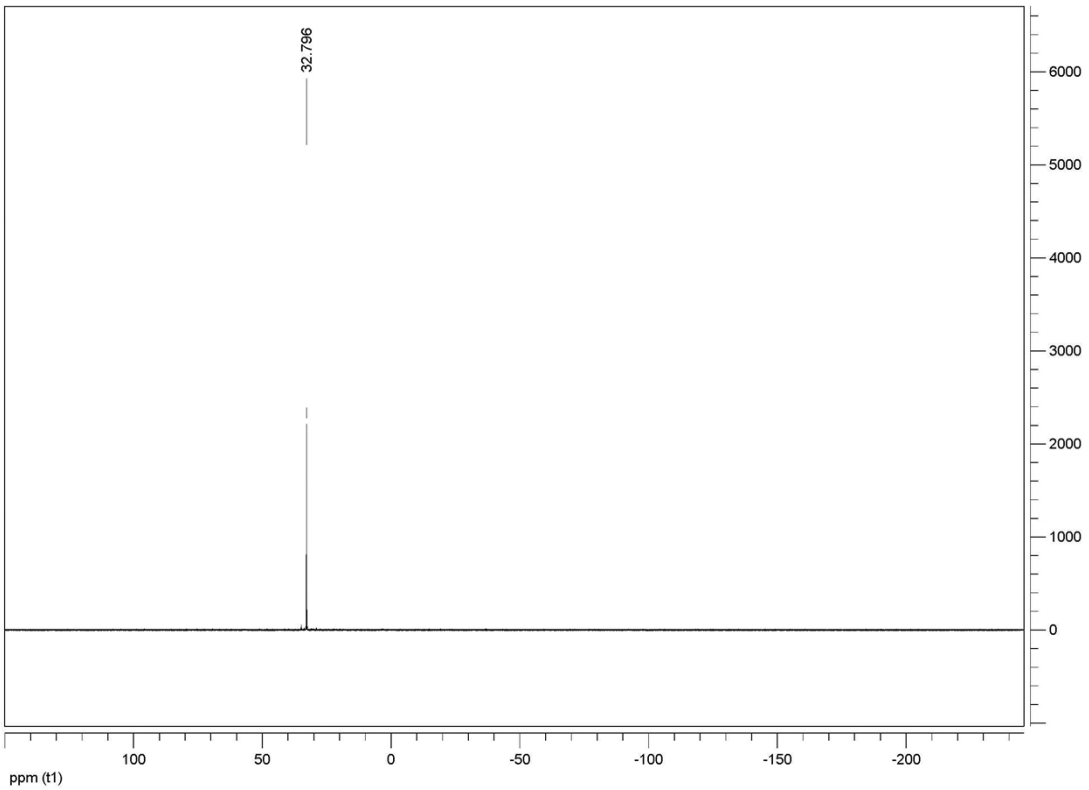

29.803
2500
2000
1500
1000
500
0
100
50
0
-50
-100
-150
-200
ppm (f1)


WL-1 #47-48 RT: 0.41-0.42 AV: 2 NL: 4.54E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]
505.16863
R=35610
z=1
C 34 H 27 O Na P = 505.16917
-1.08274 ppm
483.18684
R=36156
z=1
C 34 H 28 O P = 483.18723
-0.79500 ppm
521.14270
R=33993
z=1
C 34 H 28 Na P 2 = 521.15585
-25.22544 ppm
987.34766
R=25476
z=1
743.75109
R=28363
z=2
C 34 H 3 Na P 10 = 743.75031
1.04897 ppm
965.36601
R=25927
z=1
911.31669
R=27016
z=1
128.95350
R=69478
z=?
225.02930
R=50910
z=1
302.09687
R=43724
z=2
322.61005
R=43862
z=2
398.85569
R=40252
z=2
Relative Abundance
m/z

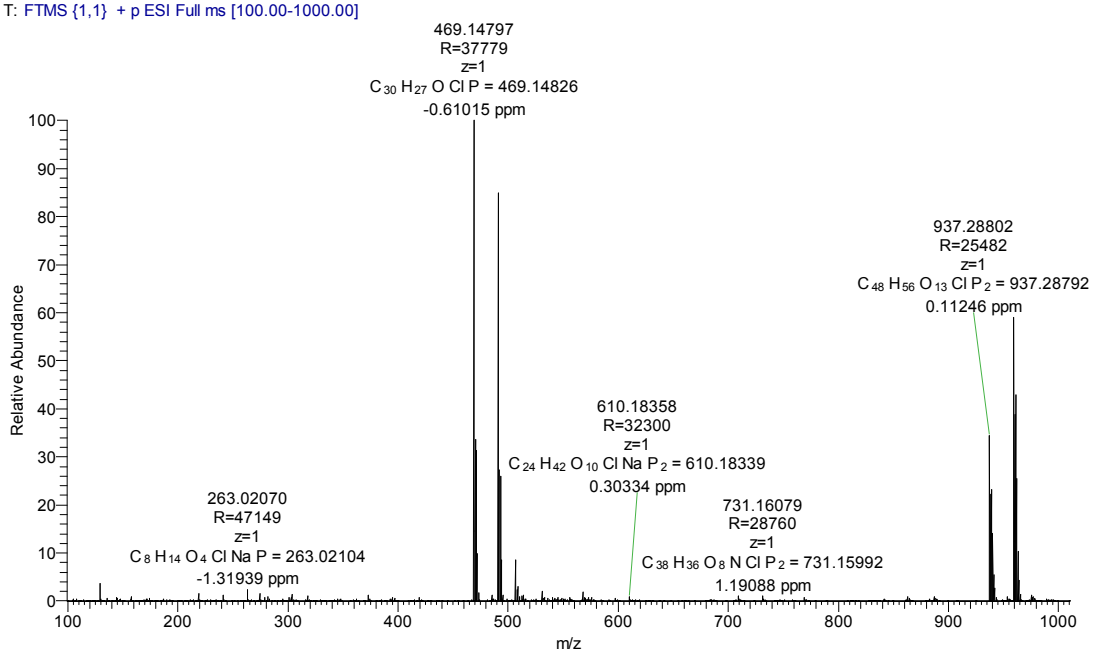

Cl
Ph2
P
O
3u

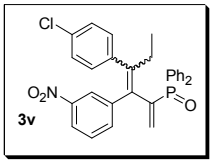

7.665
7.647
7.634
7.617
7.613
7.515
7.512
7.497
7.493
7.478
7.475
7.461
7.458
7.443
7.439
7.424
7.420
7.412
7.408
7.404
7.393
7.385
7.374
7.367
7.355
7.352
7.348
7.336
7.329
7.318
7.310
7.260
7.220
7.199
7.184
7.178
7.162
7.157
7.145
6.777
6.757
3.699
3.681
2.008
1.990
1.982
1.971
1.952
1.936
1.933
1.917
1.910
1.898
1.892
1.875
1.854
1.836
4.23
7.11
6.28
2.08
2.03
2.47
3.07
ppm (t1)

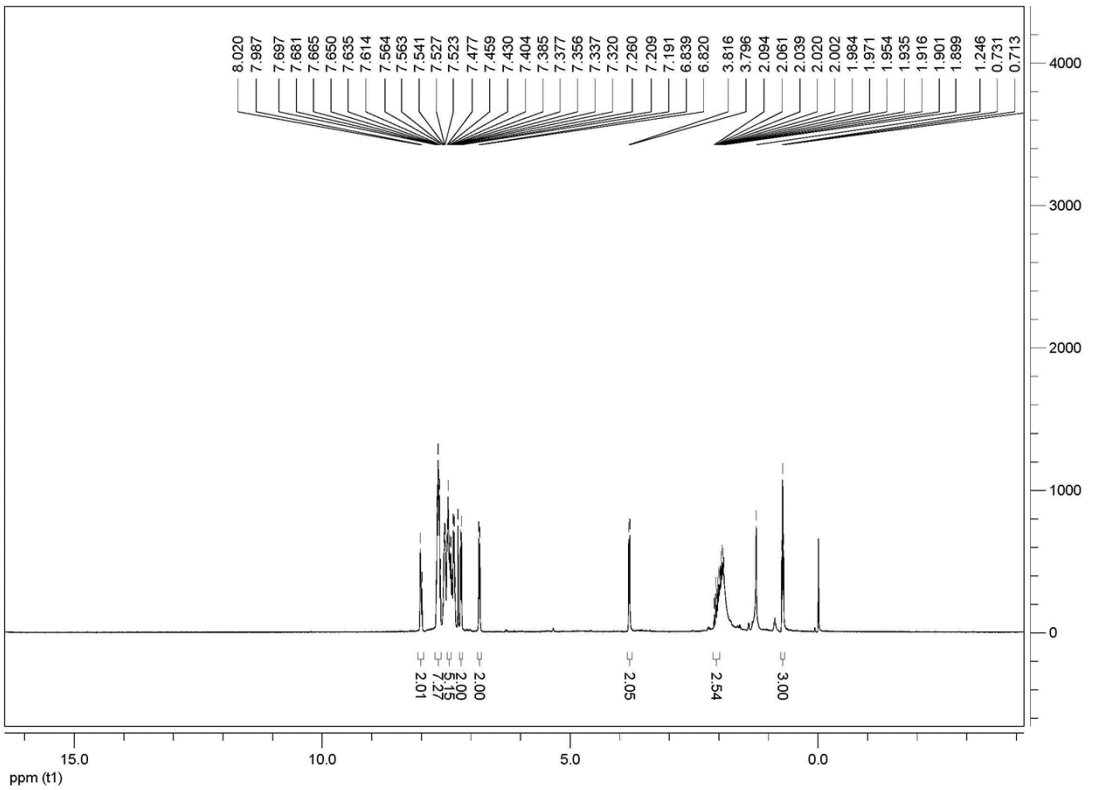

209.888
209.821
138.472
138.400
133.813
133.747
132.974
132.961
132.143
132.021
131.875
131.850
131.824
131.630
131.535
131.460
131.363
131.106
130.978
129.270
128.408
128.352
128.315
128.271
128.230
128.194
128.088
127.092
127.069
126.509
111.281
111.142
103.785
102.792
77.354
77.036
76.718
34.383
34.301
23.146
23.094
12.138
12.117
ppm (t1)

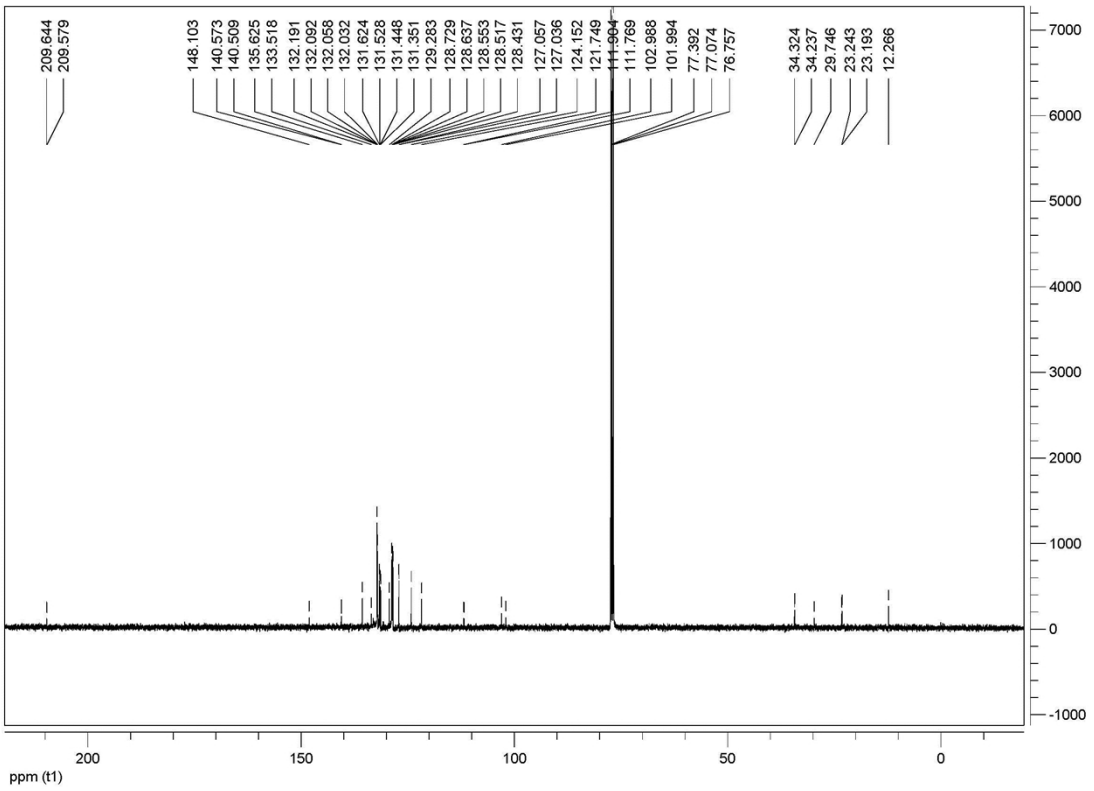


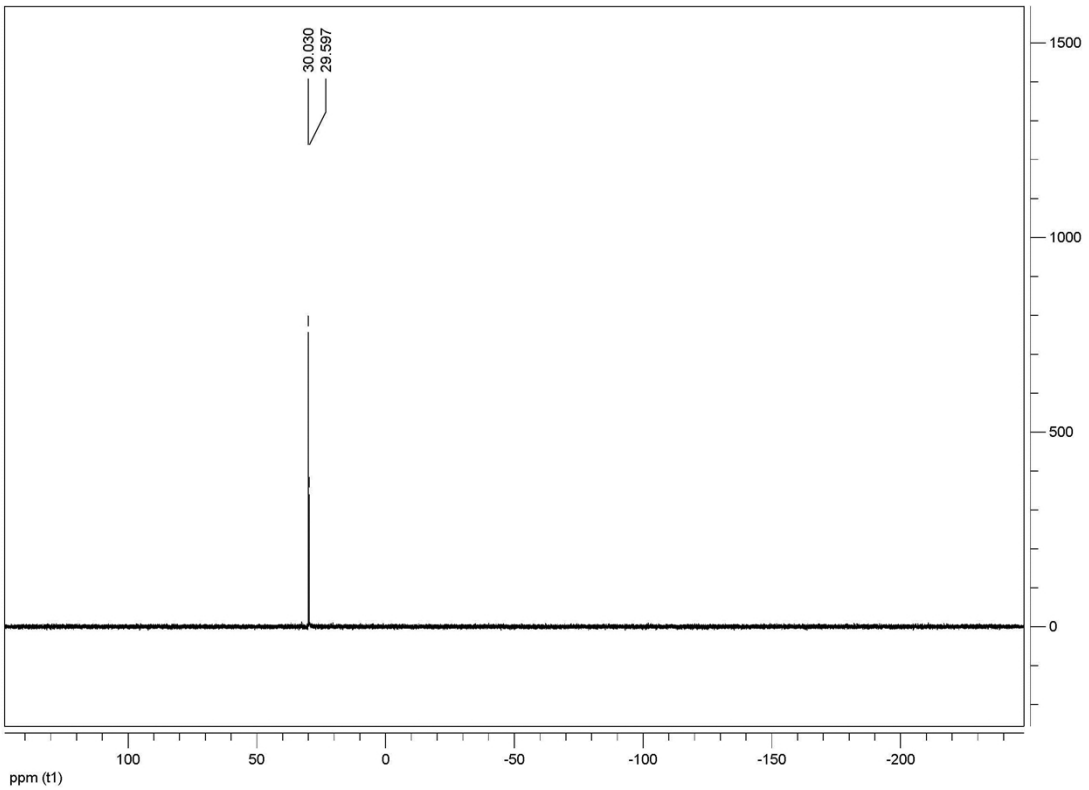
4 #49-50 RT: 0.45-0.47 AV: 2 NL: 2.41E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]






8.020
7.987
7.697
7.681
7.665
7.650
7.635
7.614
7.564
7.563
7.541
7.527
7.523
7.477
7.459
7.430
7.404
7.385
7.377
7.356
7.337
7.320
7.260
7.209
7.191
6.839
6.820
3.816
3.796
2.094
2.061
2.039
2.020
2.002
1.984
1.971
1.954
1.935
1.916
1.901
1.899
1.246
0.731
0.713
2.01
7.27
5.15
2.00
2.00
2.05
2.54
3.00
ppm (t1)


209.644
209.579
148.103
140.573
140.509
135.625
133.518
132.191
132.092
132.058
132.032
131.624
131.528
131.448
131.351
129.283
128.729
128.637
128.553
128.517
128.431
127.057
127.036
124.152
121.749
111.904
111.769
102.988
101.994
77.392
77.074
76.757
34.324
34.237
29.746
23.243
23.193
12.266
ppm (t1)



3 #43-44 RT: 0.42-0.43 AV: 2 NL: 1.35E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]