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Supporting Information

Palladium-Catalyzed Desulfitative C-P Coupling of Arylsulfinate Metal Salts and H-phosphonates

Junchen Li, Xiaojing Bi, Hongmei Wang, Junhua Xiao*

State Key Laboratory of NBC Protection for Civilian, Beijing 102205, P. R. China

*E-mail: xiao.junhua@pku.edu.cn

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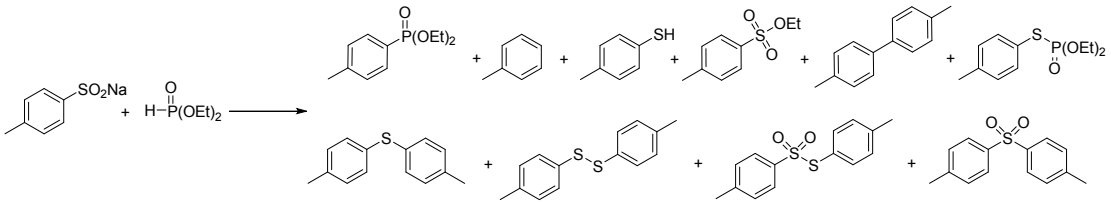
General procedures. All commercially obtained reagents for the desulfitative C-P coupling reaction were used as received; sodium arylsulfinates and potassium arylsulfinates were prepared according to literature[1]; silver arylsulfinates were prepared according to literature[2]. Dipropyl phosphonate and bis(2-methoxyethyl) phosphonate were prepared according to literature[3]. Dichloromethane (DCE), hexane, and ethyl acetate (EA) were distilled prior to use. All reactions were carried out in the air. Reactions were monitored by thin layer chromatography (TLC) carried out on 0.25 mm E. Merck silica gel plates (60F-254) using UV light as the visualizing agent and an acidic mixture (10%) of phosphomolybdic acid, ethanol, and heat as developing agents. Qingdao haiyang silica gel (200-300 mesh) was used for flash column chromatography. NMR spectra were recorded on Bruker Advance III-400 instruments and calibrated using residual undeuterated solvent as an internal reference (TMS @ 0.00 ppm ^{1}H NMR, $CHCl_3$ @ 77.16 ppm ^{13}C NMR, no reference for ^{31}P NMR). The following abbreviations (or combinations thereof) were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, b = broad. Mass spectra were recorded on Polaris Q by electron impact ionization method.

The reactions involving microwave irradiation were conducted under air in heavy-walled glass vessel purchased from CEM. The inner diameter of the vial was 1.3 cm, and its volume, 10 mL. The microwave heating was performed in a CEM DISCOVER SCLASS focused single-mode microwave cavity using a *dynamic method*. The reaction mixtures were stirred with a magnetic stir bar at high speed during the irradiation. The temperature, pressure, and irradiation power were monitored during the course of the reactions using the provided software. The irradiation power for solvent: toluene-160W, xylene-180W, EtOAc-120W, THF-50W, DCE-50W, DMF-40W, DMSO-40W, DMF/DMSO-40W.

General procedure for the preparation of the arylphosphonates. Arylsulfinate metal salt (0.36 mmol or 0.18 mmol), $PdCl_2$ (0.06 mmol), Ag_2CO_3 (0.6 mmol), DMF/DMSO (v/v = 19/1, 2 mL), and H-phosphonate (0.3 mmol) were added to the microwave tube. The tube was sealed and stirred for 20 minutes at ambient temperature. The tube was then heated at 40W for 10 minutes at 120 ºC. After cooling down, the resulting suspension were filtered through a pad of celite and washed with CH_2Cl_2. A small amount of silica gel was added into the filtrate, and then evaporated. The residue was purified by flash chromatography on silica with hexane/ethyl acetate to provide the desired product.

All possible products of the model reaction determined by GC-MS.

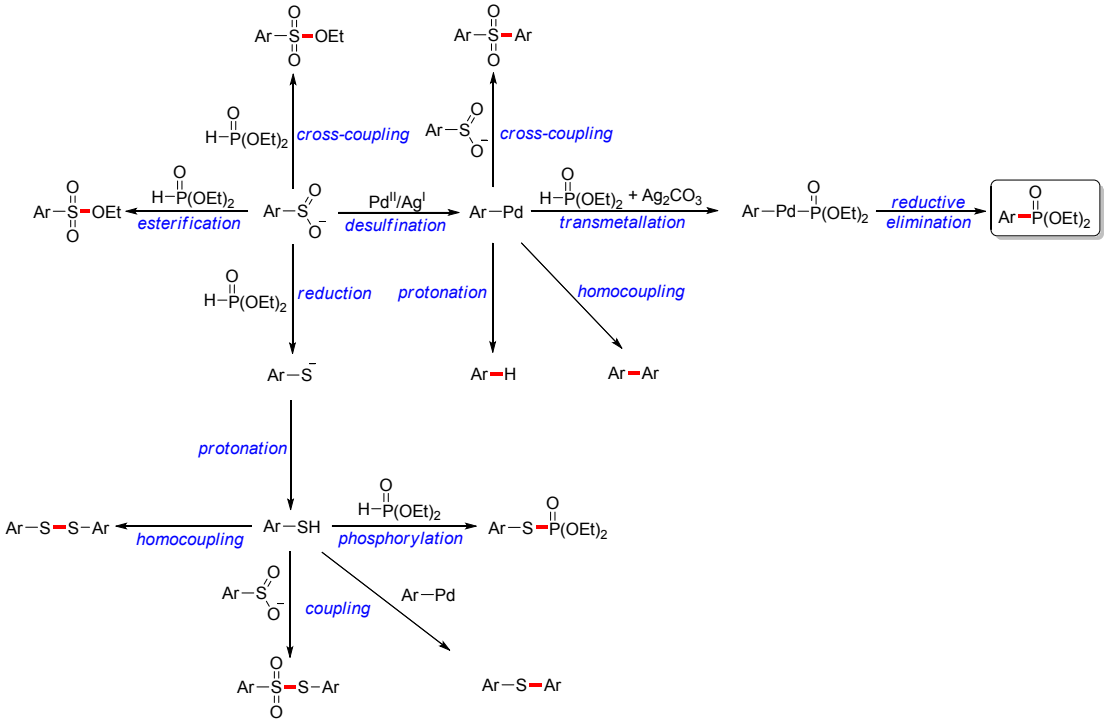


Diethyl pyrophosphate was also generated in several conditions. Due to the too many types of side products, we would like to solely employ inorganic Palladium catalysts and inorganic

[1] Crowell, T. A.; Halliday, B. D.; McDonald III, J. H.; Indelicato, J. M.; Pasini, C. E.; Wu, E. C. Y. *J. Med. Chem.* **1989**, *32*, 2436.
[2] Huang, W.-Y.; Hu, L.-Q. *J. Fluor. Chem.* **1989**, *44*, 25.
[3] Santschi, N.; Togni, A. *J. Org. Chem.* **2011**, *76*, 4189.

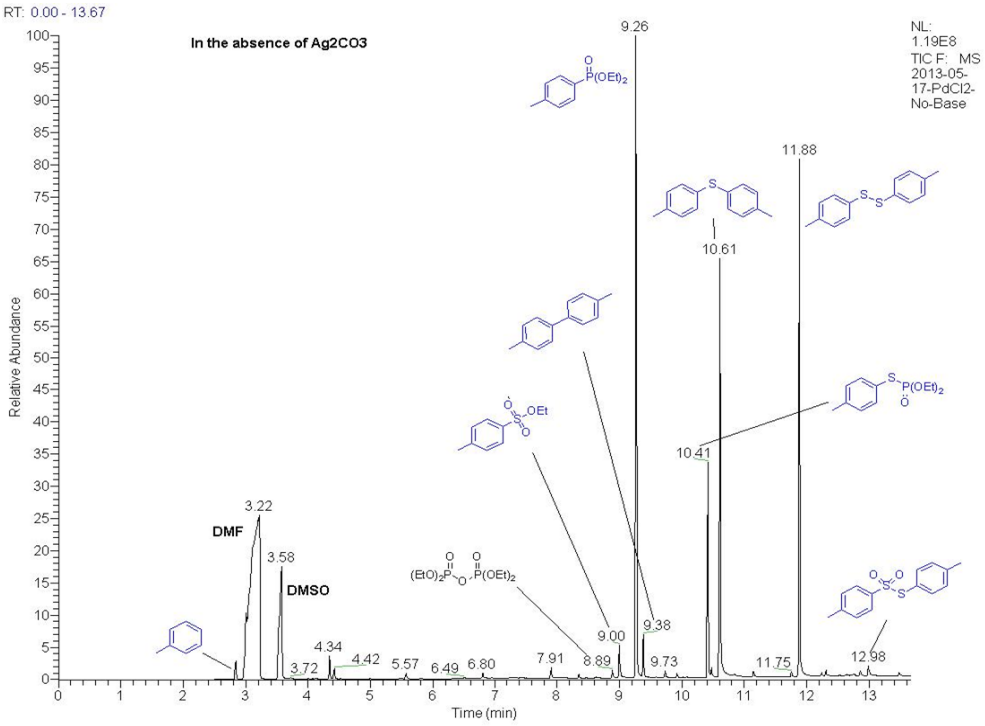
oxidants to avoid the introduction of other organic side-products. Similar product distributions were obtained with other arylsulfinate metal salts. We also ran the model reaction at 130 °C for 10h under conventional reflux condition. Relatively low conversion of diethyl phosphite and low yield were observed by GC-MS.

Plausible mechanism for the formation of the products

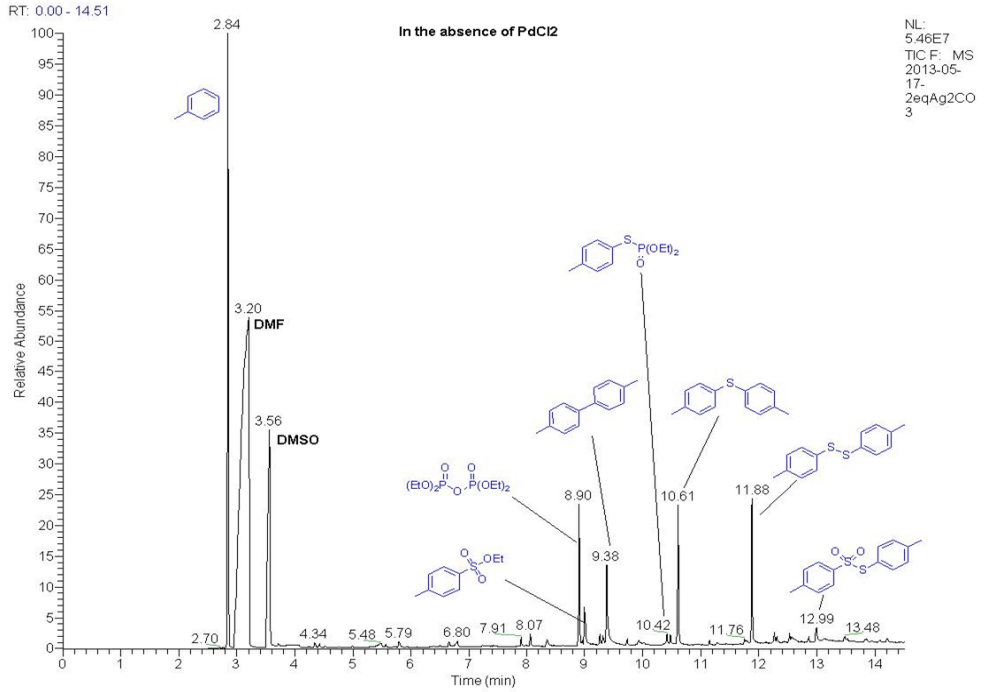


Selected GC-MS Results.

1) Reaction carried out in the absence of Ag_2CO_3 (Table 1, entry 14).

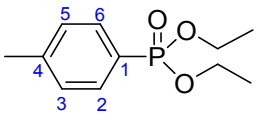


2) Reaction carried out in the absence of $PdCl_2$ (Table 1, entry 15).



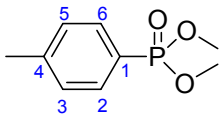
Characterization data for arylphosphonates

Diethyl *p*-tolylphosphonate (3a)



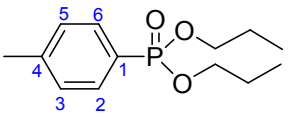
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (2H, dd, H2, H6, $J_{2/6\text{-}P}$ = 13.6 Hz, $J_{2/6\text{-}3/5}$ = 7.8 Hz), 7.28 (2H, m, H3, H5), 4.10 (4H, m, CH_2), 2.40 (3H, s, C4CH_3), 1.31 (6H, t, CH_2CH_3, $J_{CH3\text{-}CH2}$ = 7.1 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.03 (d, C4, J = 3.0 Hz), 131.92 (d, C2, C6, J = 10.3 Hz), 129.30 (d, C3, C5, J = 15.4 Hz), 125.09 (d, C1, J = 190.0 Hz), 62.06 (d, CH_2, J = 5.3 Hz), 21.74 (C4CH_3), 16.41 (d, CH_2CH_3, J = 6.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 19.56. MS (70 eV, EI) m/z (%): 228 (M^+, 11), 200 (10), 172 (100), 155 (38), 137 (6.6), 119 (33), 108 (30.3), 91 (65.7), 65 (17).

Dimethyl *p*-tolylphosphonate (3b)



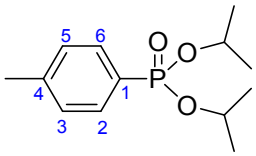
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (dd, 2H, H2, H6, $J_{2/6\text{-}P}$ = 13.1 Hz, $J_{2/6\text{-}3/5}$ = 8.1 Hz), 7.29 (dd, 2H, H3, H5, $J_{3/5\text{-}2/6}$ = 8.1 Hz, $J_{3/5\text{-}P}$ = 4.1 Hz), 3.76 (s, 3H, OCH_3), 3.73 (s, 3H, OCH_3), 2.41 (s, 3H, C4CH_3). ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.40 (d, C4, J = 3.2 Hz), 132.09 (d, C2, C6, J = 10.3 Hz), 129.44 (d, C3, C5, J = 15.5 Hz), 123.68 (d, C1, J = 191.0 Hz), 52.73 (d, OCH_3, J = 5.5 Hz), 21.80 (d, C4CH_3, J = 1.2 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 22.39. MS (70 eV, EI) m/z (%): 199 (M^+-1, 51), 169 (11), 155 (36), 105 (100).

Dipropyl *p*-tolylphosphonate (3c)



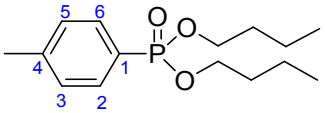
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (2H, dd, H2, H6, $J_{2/6\text{-}P}$ = 13.1 Hz, $J_{2/6\text{-}3/5}$ = 8.1 Hz), 7.27 (2H, dd, H3, H5, $J_{3/5\text{-}2/6}$ = 7.3 Hz, $J_{3/5\text{-}P}$ = 3.7 Hz), 3.98 (4H, m, OCH_2), 2.40 (3H, s, C4CH_3), 1.69 (4H, tq, CH_2CH$_3$, $J_{CH2\text{-}CH2}$ = 7.2 Hz, $J_{CH2\text{-}CH3}$ = 7.2 Hz), 0.93 (6H, t, CH_2CH_3, $J_{CH3\text{-}CH2}$ = 7.4 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.97 (d, C4, J = 3.1 Hz), 131.94 (d, C2, C6, J = 10.2 Hz), 129.29 (d, C3, C5, J = 15.4 Hz), 125.12 (d, C1, J = 190.6 Hz), 67.56 (d, OCH_2, J = 5.6 Hz), 23.90 (d, CH_2CH$_3$, J = 6.7 Hz), 21.75 (C4CH_3), 10.18 (CH_2CH_3). ^{31}P NMR (162 MHz, $CDCl_3$): δ MS (70 eV, EI) m/z (%): 257 (M^+-1, 3.6), 215 (54), 197 (7.2), 173 (100), 155 (40), 132 (34), 117 (11), 91 (36), 65 (9).

Diisopropyl *p*-tolylphosphonate (3d)



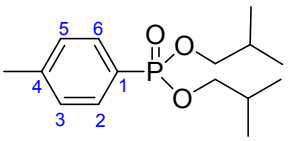
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (dd, 2H, H2, H6, $J_{2/6\text{-}P}$ = 13.1 Hz, $J_{2/6\text{-}3/5}$ = 8.1 Hz), 7.25 (dd, 2H, H3, H5, $J_{3/5\text{-}2/6}$ = 8.1 Hz, $J_{3/5\text{-}P}$ = 4.0 Hz), 4.67 (m, 2H, CH), 2.40 (s, 3H, C4CH_3), 1.36 (d, 6H, CHCH_3, $J_{CH3\text{-}CH}$ = 6.2 Hz), 1.22 (d, 6H, CHCH_3, $J_{CH3\text{-}CH}$ = 6.2 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.59 (d, C4, J = 3.2 Hz), 131.89 (d, C2, C6, J = 10.2 Hz), 129.13 (d, C3, C5, J = 15.4 Hz), 126.80 (d, C1, J = 190.7 Hz), 70.59 (d, *C*H, J = 5.4 Hz), 24.19 (d, C4*C*H$_3$, J = 3.9 Hz), 23.94 (d, CHCH_3, J = 4.9 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 17.33. MS (70 eV, EI) m/z (%): 257 (M^++1, 4), 215 (22), 182 (72), 173 (100), 167 (45), 155 (51).

Dibutyl *p*-tolylphosphonate (3e)



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (2H, dd, H2, H6, $J_{2/6\text{-}P}$ = 13.1 Hz, $J_{2/6\text{-}3/5}$ = 7.4 Hz), 7.27 (2H, m, H3, H5), 4.02 (4H, m, OCH_2), 2.40 (3H, s, C4CH_3), 1.64 (4H, m, OCH$_2$CH_2), 1.38 (4H, tq, $J_{CH2\text{-}CH2}$ = 7.3 Hz, $J_{CH2\text{-}CH3}$ = 7.3 Hz), 0.90 (6H, t, CH_2CH_3, $J_{CH3\text{-}CH2}$ = 7.4 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.95 (d, C4, J = 3.3 Hz), 131.94 (d, C2, C6, J = 10.2 Hz), 129.28 (d, C3, C5, J = 15.4 Hz), 125.11 (d, C1, J = 190.11 Hz), 65.77 (d, OCH_2, J = 5.6 Hz), 32.55 (d, OCH$_2$CH_3, J = 6.6 Hz), 21.76 (C4CH_3), 18.85 (CH_2CH$_3$), 13.70 (CH_2CH_3). ^{31}P NMR (162 MHz, $CDCl_3$): δ 19.54. MS (70 eV, EI) m/z (%): 284 (M^+, 4), 229 (29), 173 (100), 155 (24), 146 (8), 131 (3.7), 91 (11.4), 65 (3).

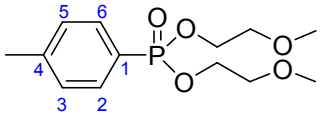
Diisobutyl *p*-tolylphosphonate (3f)



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (dd, 2H, H2, H6, $J_{C\text{-}P}$ = 13.1 Hz, $J_{2/6\text{-}5/5}$ = 8.0 Hz), 7.27 (dd,

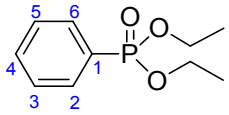
2H, H3, H5, $J_{3/5\text{-}2/6}$ = 8.0 Hz, $J_{3/5\text{-}P}$ = 4.0 Hz), 3.79 (m, 4H, OCH_2), 2.40 (s, 3H, C4CH_3), 1.94 (m, 2H, CH), 0.93 (dd, 12H, CH_3, $J_{CH3\text{-}P}$ = 6.7 Hz, $J_{CH3\text{-}CH}$ = 1.9 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.90 (d, C4, J = 3.2 Hz), 131.93 (d, C2, C6, J = 10.2 Hz), 129.26 (d, C3, C5, J = 15.4 Hz), 125.08 (d, C1, J = 191.1 Hz), 71.92 (d, OCH_2, J = 6.0 Hz), 29.26 (d, CH, J = 6.8 Hz), 21.75 (d, C4CH_3, J = 1.2 Hz), 18.84 (d, CHCH_3, J = 1.0 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 19.24. MS (70 eV, EI) m/z (%): 285 (M^+, 2), 229 (12), 173 (100).

Bis(2-methoxyethyl) *p*-tolylphosphonate (3h)



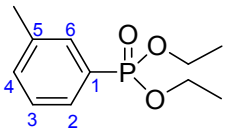
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73 (dd, 2H, H2, H6, $J_{2/6\text{-}P}$ = 13.3 Hz, $J_{2/6\text{-}3/5}$ = 8.1 Hz), 7.27 (dd, 2H, H3, H5, $J_{3/5\text{-}2/6}$ = 8.1 Hz, $J_{3/5\text{-}P}$ = 4.2 Hz), 4.19 (m, 4H, POCH_2), 3.59 (m, 4H, CH_2OCH_3), 3.35 (s, 6H, OCH_3), 2.40 (s, 3H, C4CH_3). ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.24 (d, C4, J = 3.2 Hz), 132.09 (d, C2, C6, J = 10.5 Hz), 129.30 (d, C3, C5, J = 15.7 Hz), 124.56 (d, C1, J = 192.6 Hz), 71.66 (d, CH$_2$OCH$_3$, J = 6.6 Hz), 64.91 (d, POCH_2, J = 5.6 Hz), 59.03 (OCH_3), 21.80 (d, C4CH_3, J = 1.2 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 20.52. MS (70 eV, EI) m/z (%): 289 (M^++1, 2), 231 (90), 201 (23), 173 (100).

Diethyl phenylphosphonate (3i)



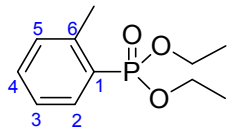
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (ddd, 2H, H2, H6, $J_{2/6\text{-}P}$ = 13.3 Hz, $J_{2/6\text{-}3/5}$ = 8.2 Hz, $J_{2/6\text{-}4}$ = 1.3 Hz), 7.55 (ddd, 1H, H4, $J_{4\text{-}3/5}$ = 7.5 Hz, $J_{4\text{-}P}$ = J4-6/2 = 1.4 Hz), 7.47 (ddd, 2H, H3, H5, $J_{3/5\text{-}2/6}$ = 8.2 Hz, $J_{3/5\text{-}4}$ = 7.5 Hz, $J_{3/5\text{-}P}$ = 4.2 Hz), 4.12 (m, 4H, CH_2), 1.32 (t, 6H, CH_3, $J_{CH3\text{-}CH2}$ = 7.1 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 132.42 (d, C4, J = 3.0 Hz), 131.82 (d, C3, C5, J = 9.9 Hz), 128.52 (d, C2, C6, J = 15.0 Hz), 128.47 (d, C1, J = 187.8 Hz), 62.15 (d, CH$_2$, J = 5.4 Hz), 16.40 (d, CH$_3$, J = 6.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 18.78. MS (70 eV, EI) m/z (%): 213 (M^+-1, 17), 186 (22), 158 (71), 141 (68), 105 (72), 94 (100).

Diethyl *m*-tolylphosphonate (3j)



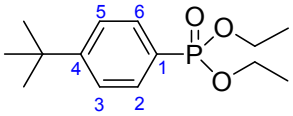
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64 (d, 1H, H6, J6-P = 13.4 Hz), 7.58 (m, 1H, H2), 7.35 (m, H3, H4, 2H), 4.11 (m, 4H, CH_2), 2.40 (s, 3H, C5CH_3), 1.33 (t, 6H, CH_2CH_3, $J_{CH3\text{-}CH2}$ = 7.1 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.34 (d, C5, J = 15.0 Hz), 133.23 (d, C4, J = 3.2 Hz), 132.35 (d, C6, J = 10.0 Hz), 128.84 (d, C2, J = 9.7 Hz), 128.44 (d, C3, J = 15.8 Hz), 128.19 (d, C1, J = 186.8 Hz), 62.09 (d, CH$_2$, J = 5.4 Hz), 21.37 (C5CH_3), 16.40 (d, CH_2CH_3, J = 6.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 19.28. MS (70 eV, EI) m/z (%): 228 (M^+, 19), 200 (18), 172 (100), 155 (31).

Diethyl *o*-tolylphosphonate (3k)



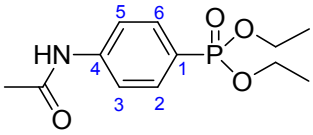
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91 (ddd, 1H, H2, $J_{2\text{-}P}$ = 14.4 Hz, $J_{2\text{-}3}$ = 7.9 Hz, $J_{2\text{-}4}$ = 1.4 Hz), 7.43 (m, 1H, H4), 7.27 (m, 2H, H3, H5), 4.12 (m, 4H, CH_2), 2.58 (d, 3H, C6CH_3, $J_{C6CH3\text{-}P}$ = 1.5 Hz), 1.33 (t, 6H, $J_{CH3\text{-}CH2}$ = 7.1 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.89 (d, C6, J = 10.2 Hz), 133.99 (d, C2, J = 10.3 Hz), 132.52 (d, C4, J = 3.0 Hz), 131.28 (d, C5, J = 14.9 Hz), 126.92 (d, C1, J = 183.9 Hz), 125.45 (d, C3, J = 14.9 Hz), 61.95 (d, CH_2, J = 5.5 Hz), 21.31 (d, C6CH_3, J = 3.6 Hz), 16.41 (d, CH_2CH_3, J = 6.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 19.44. MS (70 eV, EI) m/z (%): 228 (M^+, 46), 213 (90), 200 (31), 185 (47), 172 (97), 154 (100).

Diethyl (4-(*tert*-butyl)phenyl)phosphonate (3l)



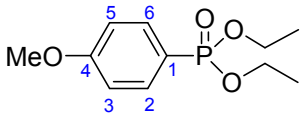
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 (dd, 2H, $J_{2/6\text{-}P}$ = 13.0 Hz, $J_{2/6\text{-}3/5}$ = 8.5 Hz), 7.48 (dd, 2H, H3, H5, $J_{3/5\text{-}2/6}$ = 8.5 Hz, $J_{3/5\text{-}P}$ = 3.9 Hz), 4.11 (m, 4H, CH_2), 1.33 (s, 9H, C(CH_3)$_3$), 1.32 (t, 6H, $J_{CH3\text{-}CH2}$ = 7.1 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.90 (d, C4, J = 3.2 Hz), 131.71 (d, C3, C5, J = 10.3 Hz), 125.51 (d, C2, C6, J = 15.2 Hz), 125.05 (d, C1, J = 190.1 Hz), 61.99 (d, CH_2, J = 5.4 Hz), 35.07 (d, $C(CH_3)_3$, J = 0.6 Hz), 31.13 (C(CH_3)$_3$), 16.39 (d, CH_2CH_3, J = 6.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 19.44. MS (70 eV, EI) m/z (%): 270 (M^+, 12), 255 (100), 242 (12), 227 (88), 214 (23), 199 (41), 181 (10).

Diethyl (4-acetamidophenyl)phosphonate (3m)



^{1}H NMR (400 MHz, $CDCl_3$): δ 9.44 (s, 1H, N*H*), 7.76 (dd, 2H, H3, H5, $J_{3/5\text{-}2/6}$ = 8.6 Hz, $J_{3/5\text{-}P}$ = 3.9 Hz), 7.70 (dd, 2H, H2, H6, $J_{2/6\text{-}P}$ = 12.6 Hz, $J_{2/6\text{-}3/5}$ = 8.6 Hz), 4.08 (m, 4H, CH_2), 2.20 (s, 3H, COCH_3), 1.32 (t, 3H, CH_2CH_3, $J_{CH3\text{-}CH2}$ = 7.1 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.70 (CO), 142.94 (C4), 132.71 (d, C2, C6, J = 10.8 Hz), 121.91 (d, C1, J = 192.5 Hz), 119.33 (d, C3, C5, J = 15.3 Hz), 62.32 (d, CH_2, J = 5.5 Hz), 24.52 (COCH_3), 16.34 (d, CH_2CH_3, J = 6.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 18.88. MS (70 eV, EI) m/z (%): 271 (M^+, 47), 243 (86), 229 (36), 201 (100), 173 (66), 155 (12).

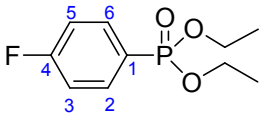
Diethyl (4-methoxyphenyl)phosphonate (3n)



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (dd, 2H, H2, H6, $J_{2/6\text{-}P}$ = 12.7 Hz, $J_{2/6\text{-}3/5}$ = 8.8 Hz), 6.97 (dd, 2H, H3, H5, $J_{3/5\text{-}2/6}$ = 8.8 Hz, $J_{3/5\text{-}P}$ = 3.1 Hz), 4.09 (m, 4H, CH_2), 3.85 (s, 3H, OCH_3), 1.32 (t, 6H, CH_2CH_3, $J_{CH3\text{-}CH2}$ = 7.1 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.89 (d, C4, J = 3.4 Hz), 133.82 (d,

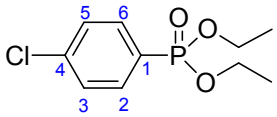
C2, C6, J = 11.3 Hz), 119.59 (d, C1, J = 194.58 Hz), 114.06 (d, C3, C5, J = 16.0 Hz), 61.95 (d, CH_2, J = 5.3 Hz), 55.38 (OCH_3), 16.39 (d, CH_2CH_3, J = 6.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 19.70. MS (70 eV, EI) m/z (%): 244 (M^+, 28), 216 (60), 188 (100), 171 (19).

Diethyl (4-fluorophenyl)phosphonate (3o)



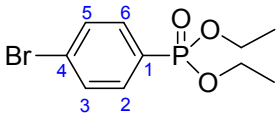
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (ddd, 2H, H2, H6, $J_{2/6\text{-}P}$ = 12.8 Hz, $J_{2/6\text{-}3/5}$ = 8.8 Hz, $J_{2/6\text{-}F}$ = 5.6 Hz), 7.16 (ddd, 2H, H5, H6, $J_{3/5\text{-}F}$ = $J_{3/5\text{-}2/6}$ = 8.8 Hz, $J_{3/5\text{-}P}$ = 3.2 Hz), 4.11 (m, 4H, CH_2), 1.33 (t, 6H, CH_3, $J_{CH3\text{-}CH2}$ = 7.1 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.50 (d, C4, $J_{C\text{-}F}$ = 253.5 Hz), 134.50 (dd, C2, C6, $J_{C\text{-}P}$ = 11.0 Hz, $J_{C\text{-}F}$ = 8.9 Hz), 124.68 (dd, C1, $J_{C\text{-}P}$ = 192.7 Hz, $J_{C\text{-}F}$ = 3.4 Hz), 115.96 (dd, C3, C5, $J_{C\text{-}F}$ = 21.4 Hz, $J_{C\text{-}P}$ = 16.3 Hz), 62.33 (d, CH_2, $J_{C\text{-}P}$ = 5.4 Hz), 16.47 (d, CH_3, $J_{C\text{-}P}$ = 6.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 17.81. MS (70 eV, EI) m/z (%): 232 (M^+, 16), 204 (21), 176 (100), 159 (39).

Diethyl (4-chlorophenyl)phosphonate (3p)



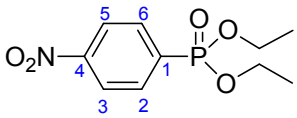
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (dd, 2H, H2, H6, $J_{2/6\text{-}P}$ = 12.9 Hz, $J_{2/6\text{-}3/5}$ = 8.5 Hz), 7.45 (dd, 2H, H3, H5, $J_{3/5\text{-}2/6}$ = 8.5 Hz, $J_{3/5\text{-}P}$ = 3.4 Hz), 4.11 (m, 4H, OCH_2), 1.32 (t, 6H, CH_3, $J_{CH3\text{-}CH2}$ = 7.1 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.05 (d, C4, J = 4.0 Hz), 133.31 (d, C2, C6, J = 10.7 Hz), 128.95 (d, C3, C5, J = 15.6 Hz), 127.13 (d, C1, J = 191.0 Hz), 62.38 (d, OCH_2, J = 5.5 Hz), 16.43 (d, CH_3, J = 6.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 17.60. MS (70 eV, EI) m/z (%): 248 (M^+, 19), 220 (17), 192 (100), 175 (30).

Diethyl (4-bromophenyl)phosphonate (3q)



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68 (dd, 2H, H2, H6, $J_{2/6\text{-}P}$ = 12.7 Hz, $J_{2/6\text{-}3/5}$ = 8.5 Hz), 7.62 (dd, H3, H5, $J_{3/5\text{-}2/6}$ = 8.5 Hz, $J_{3/5\text{-}P}$ = 3.7 Hz), 4.11 (m, 4H, OCH_2), 1.32 (t, 6H, CH_3, $J_{CH3\text{-}CH2}$ = 7.1 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 133.40 (d, C2, C6, J = 10.6 Hz), 131.89 (d, C3, C5, J = 15.5 Hz), 127.63 (d, C4, J = 4.1 Hz), 127.60 (d, C1, J = 190.4 Hz), 62.38 (d, OCH_2, J = 5.5 Hz), 16.41 (6.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 17.73. MS (70 eV, EI) m/z (%): 294 (M^++1, 14), 292 (M^+-1, 14), 265 (15), 236 (100), 221 (25), 213 (25), 185 (20), 172 (18).

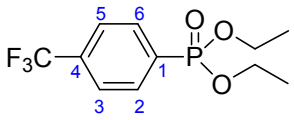
Diethyl (4-nitrophenyl)phosphonate (3r)



^{1}H NMR (400 MHz, $CDCl_3$): δ 8.32 (dd, 2H, H3, H5, $J_{3/5\text{-}2/6}$ = 8.4 Hz, $J_{3/5\text{-}P}$ = 2.8 Hz), 8.03 (dd, H2, H6, $J_{2/6\text{-}P}$ = 12.6 Hz, $J_{2/6\text{-}3/5}$ = 8.4 Hz), 4.18 (m, 4H, CH_2), 1.36 (t, 6H, CH_3, $J_{CH3\text{-}CH2}$ = 7.1 Hz).

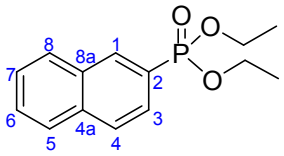
^{13}C NMR (100 MHz, $CDCl_3$): δ 150.24 (d, C4, J = 3.7 Hz), 135.87 (d, C1, J = 187.2 Hz), 132.97 (d, C2, C6, J = 10.5 Hz), 123.35 (d, C3, C5, J = 15.3 Hz), 62.77 (d, CH_2, J = 5.7 Hz), 16.34 (d, CH_3, J = 6.2 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 14.86. MS (70 eV, EI) m/z (%): 258 (M^+-1, 10), 242 (45), 232 (33), 214 (26), 204 (100), 186 (39).

Diethyl (4-(trifluoromethyl)phenyl)phosphonate (3s)



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95 (dd, 2H, H2, H6, $J_{2/6\text{-}P}$ = 13.0 Hz, $J_{2/6\text{-}3/5}$ = 7.9 Hz), 7.73 (dd, 2H, H3, H5, $J_{3/5\text{-}2/6}$ = 7.9 Hz, $J_{3/5\text{-}P}$ = 3.6 Hz), 4.15 (m, 4H, OCH_2), 1.34 (t, 6H, $J_{CH3\text{-}CH2}$ = 7.1 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 134.19 (dd, $J_{C4\text{-}F}$ = 32.7 Hz, $J_{C4\text{-}P}$ = 3.3 Hz), 132.99 (d, C1, $J_{C1\text{-}P}$ = 187.9 Hz), 132.36 (d, C2, C6, $J_{C2/C6\text{-}P}$ = 10.1 Hz), 125.45 (dq, C3, C5, $J_{C3/C5\text{-}P}$ = 15.1 Hz, $J_{C3/C5\text{-}F}$ = 3.7 Hz), 123.69 (d, CF_3, $J_{CF3\text{-}F}$ = 272.8 Hz), 62.63 (d, CH_2, $J_{CH2\text{-}P}$ = 5.5 Hz), 16.46 (d, CH_3, $J_{CH3\text{-}P}$ = 6.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 16.26. MS (70 eV, EI) m/z (%): 281 (M^+-1, 11), 263 (11), 255 (18), 227 (100), 209 (43), 189 (7), 172 (23), 162 (30).

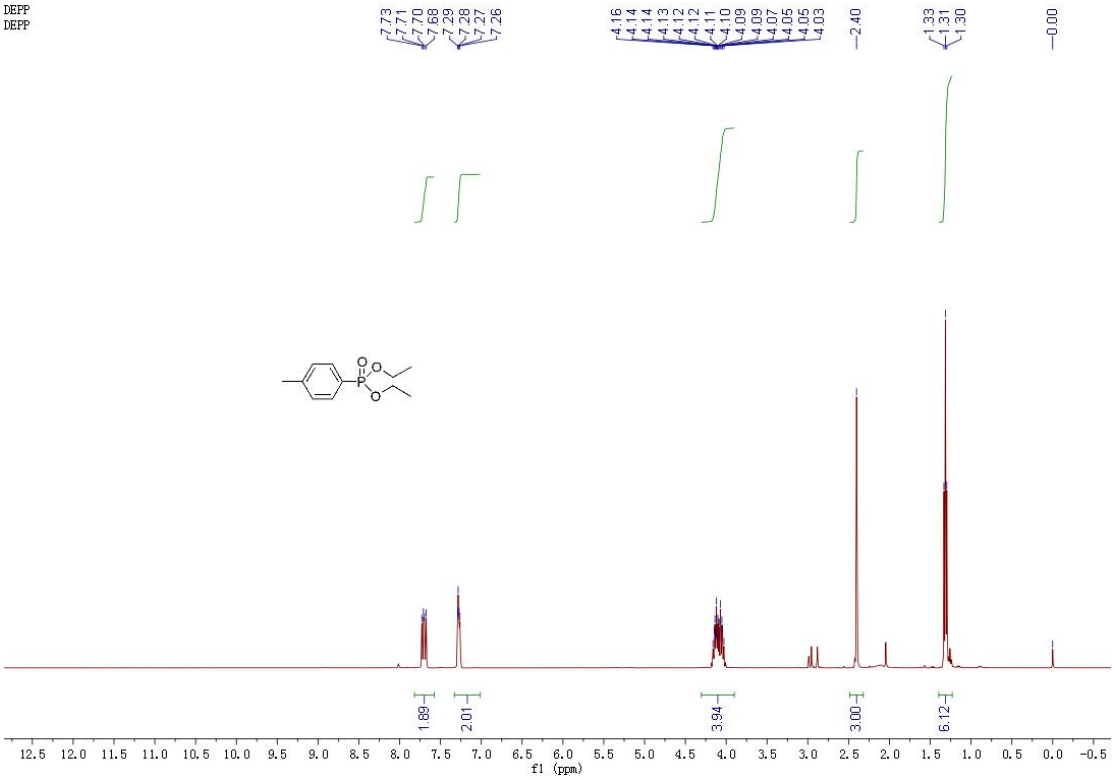
Diethyl naphthalen-2-ylphosphonate (3t)

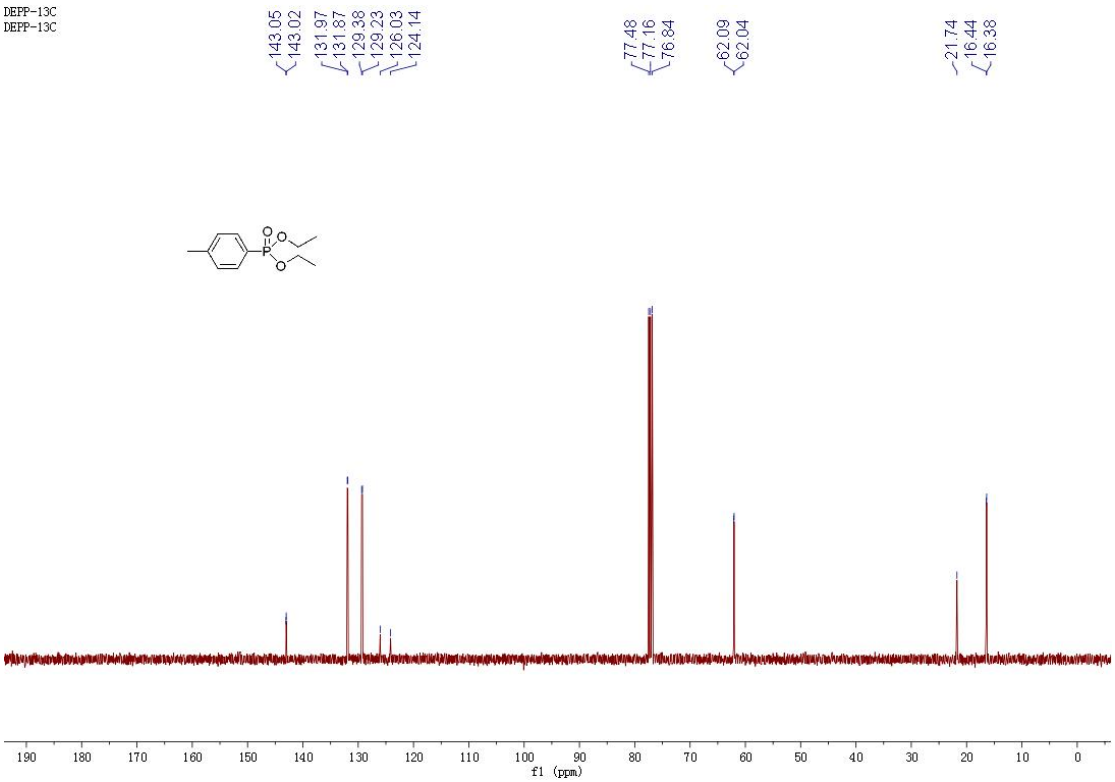


^{1}H NMR (400 MHz, $CDCl_3$): δ 8.44 (d, 1H, H1, $J_{1\text{-}P}$ = 15.1 Hz), .7.92 (m, 2H, H8, H4), 7.88 (d, 2H, H5, J = 7.9 Hz), 7.77 (ddd, 1H, H3, $J_{3\text{-}P}$ = 10.9 Hz, $J_{3\text{-}4}$ = 8.4 Hz, $J_{3\text{-}1}$ = 1.4 Hz), 7.58 (m, 2H, H6, H7), 4.16 (m, 4H, CH_2), 1.34 (t, 6H, CH_3, $J_{CH3\text{-}CH2}$ = 7.1 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 135.09 (d, C4a, J = 2.7 Hz), 134.14 (d, C1, J = 10.2 Hz), 132.44 (d, C8a, J = 16.6 Hz), 129.02 (C8), 128.46 (d, C4, J = 14.3 Hz), 128.1 (C5), 127.90 (d, C6, J = 0.7 Hz), 126.97 (d, C7, J = 1.1 Hz), 126.54 (d, C3, J = 9.8 Hz), 125.52 (d, C2, J = 187.9 Hz), 62.46 (d, CH_2, J = 5.3 Hz), 16.46 (d, CH_3, J = 6.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 19.08. MS (70 eV, EI) m/z (%): 264 (M^+, 84), 236 (100), 208 (92), 190 (26), 155 (35).

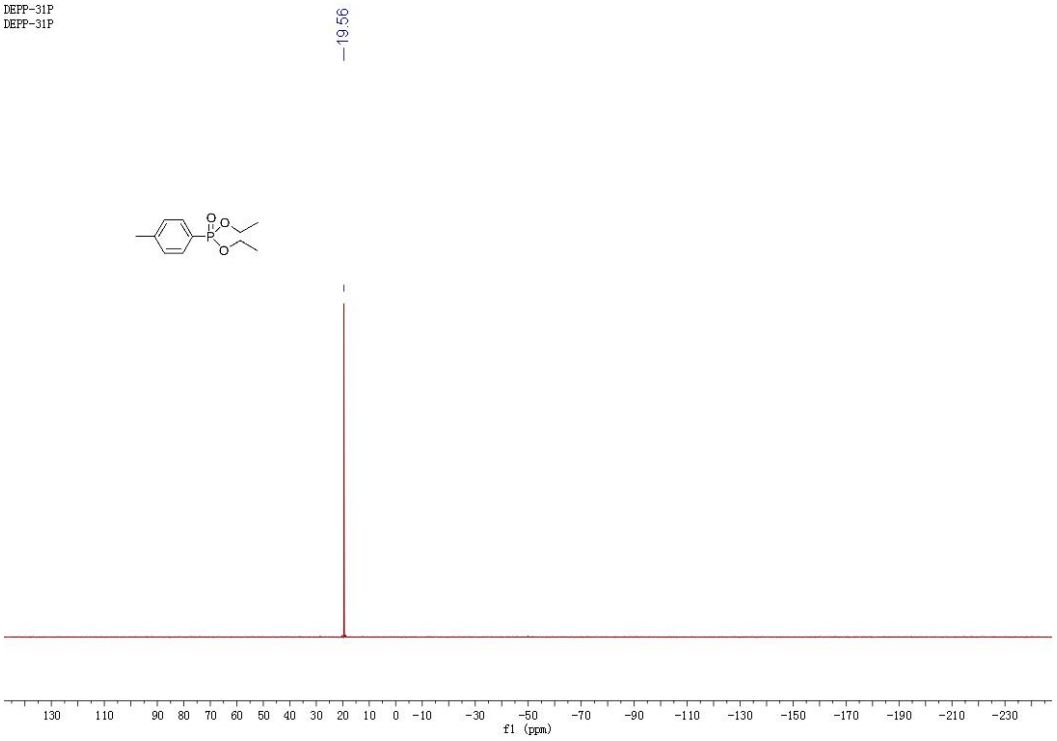
^{1}H, ^{13}C, ^{31}P NMR, and MS spectra of arylphosphonates.

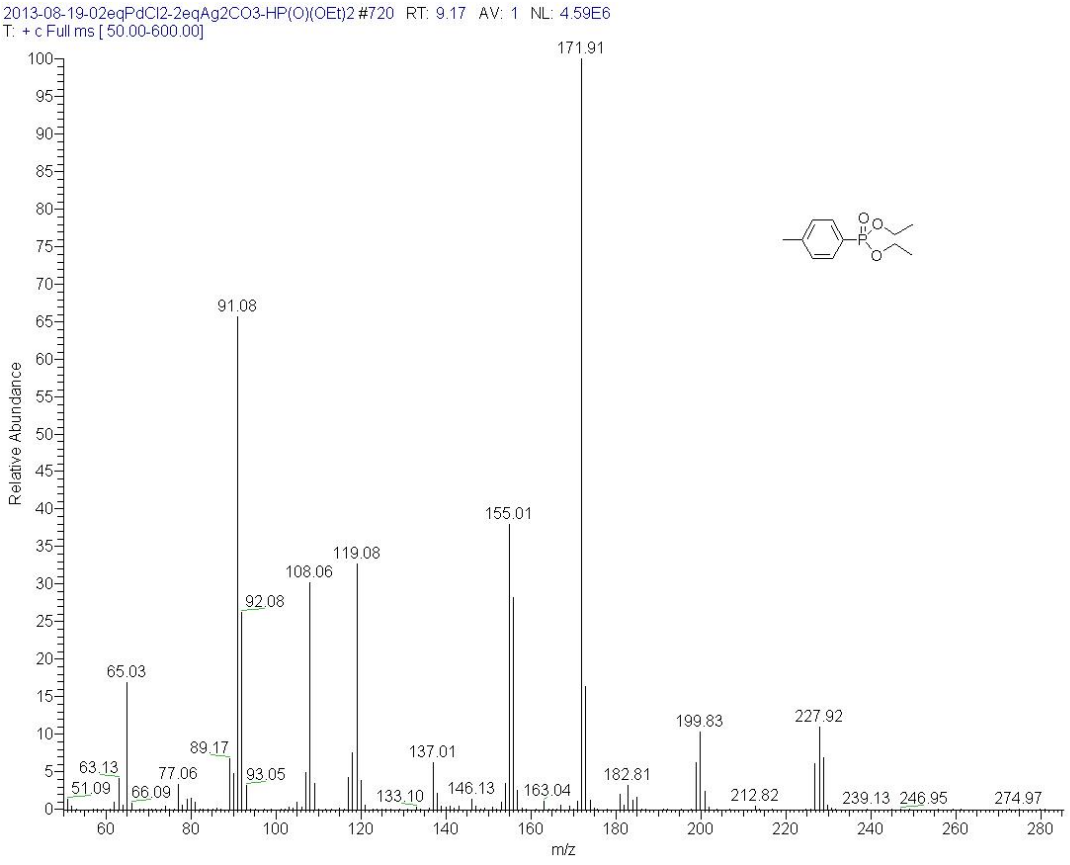
3a



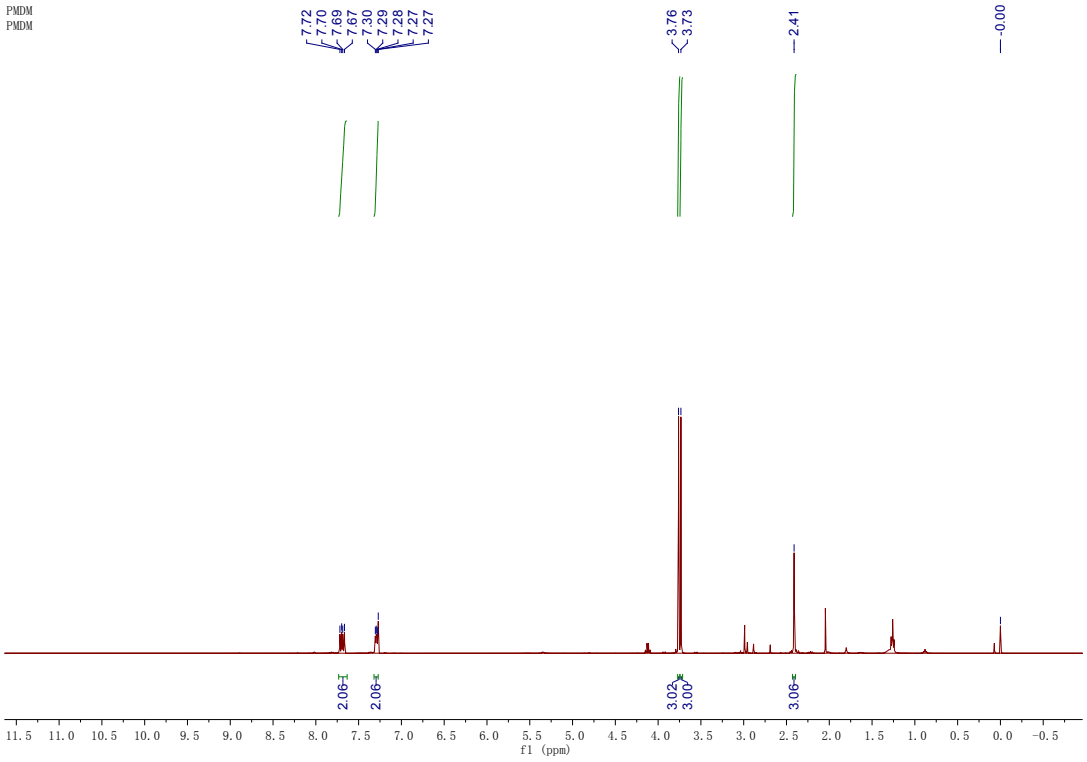


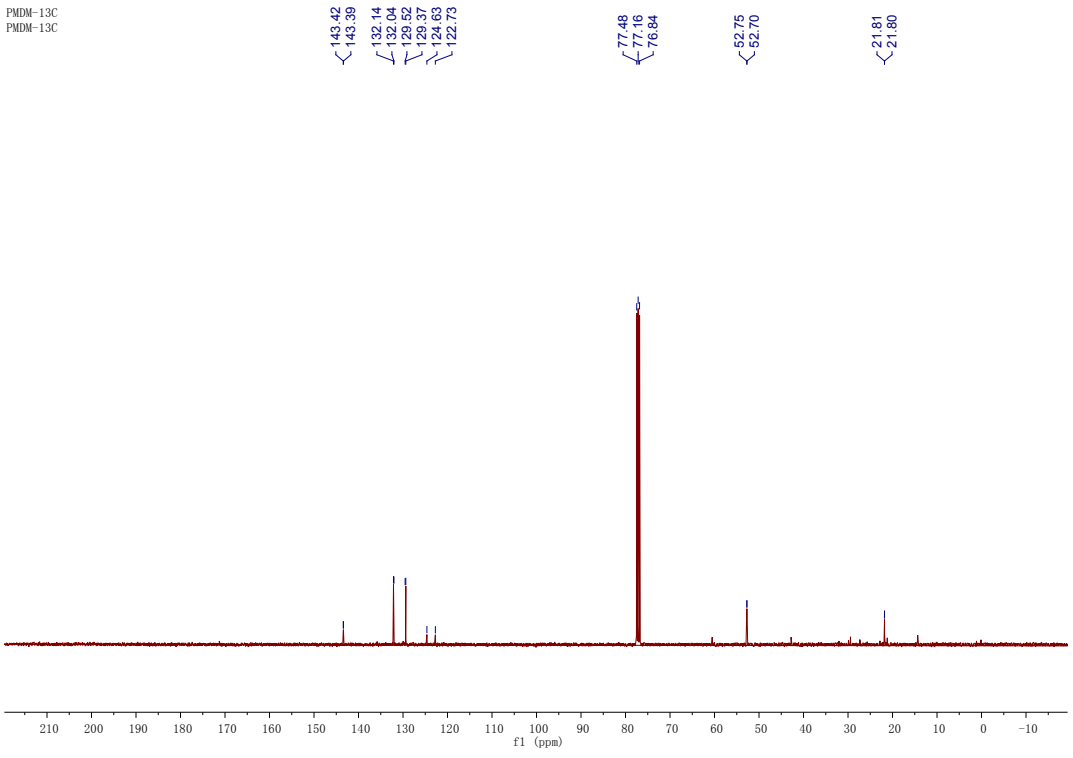
3a


DEPP-31P
DEPP-31P
19.56
f1 (ppm)

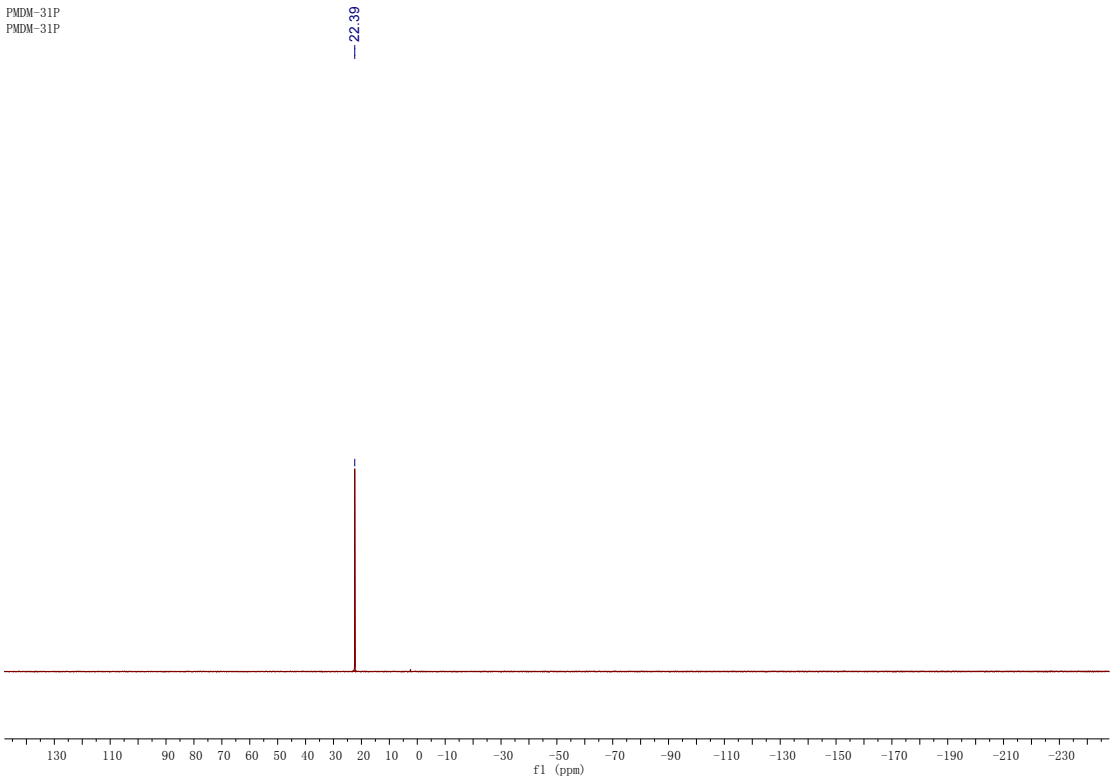

2013-08-19-02eqPdCl2-2eqAg2CO3-HP(O)(OEt)2 #720 RT: 9.17 AV: 1 NL: 4.59E6
T: + c Full ms [50.00-600.00]
Relative Abundance
m/z
171.91
91.08
155.01
119.08
108.06
92.08
65.03
89.17
137.01
199.83
227.92
63.13
77.06
93.05
182.81
51.09
66.09
133.10
146.13
163.04
212.82
239.13
246.95
274.97

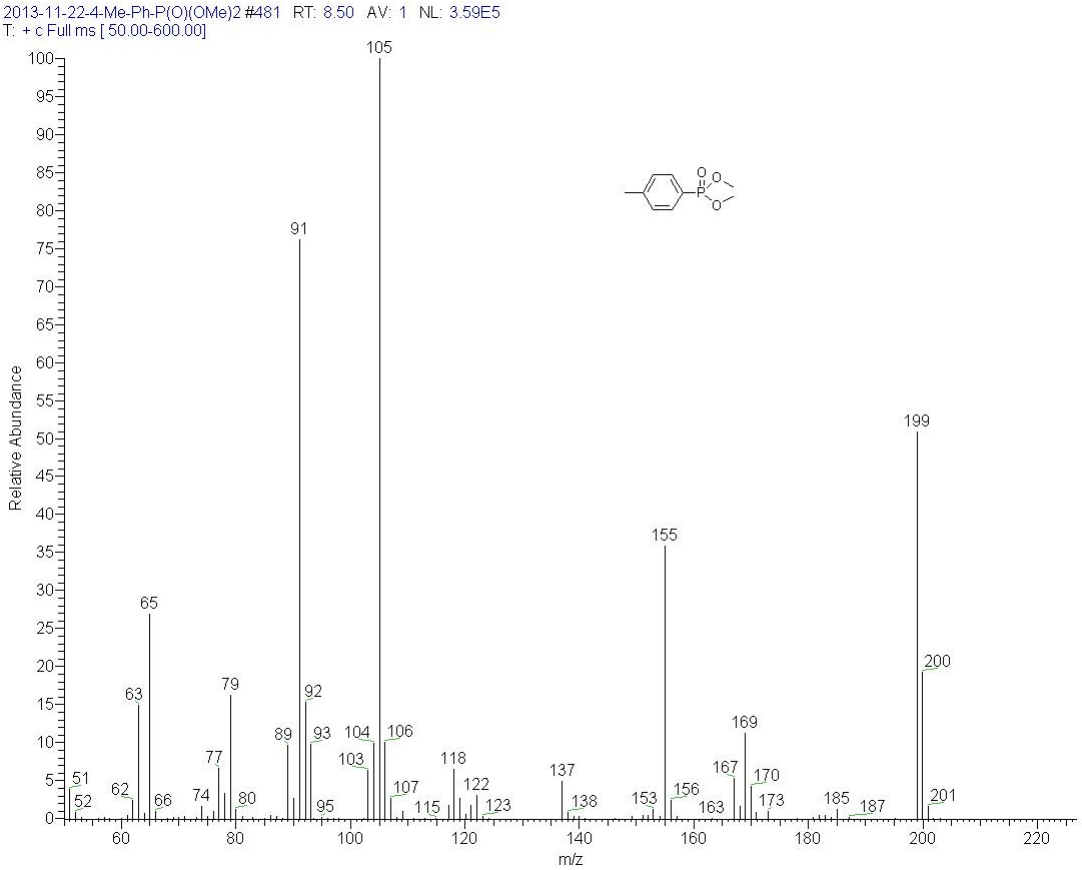
3b


PMDM
PMDM
7.72
7.70
7.69
7.67
7.30
7.29
7.28
7.27
7.27
3.76
3.73
2.41
-0.00
2.06
2.06
3.02
3.00
3.06
f1 (ppm)

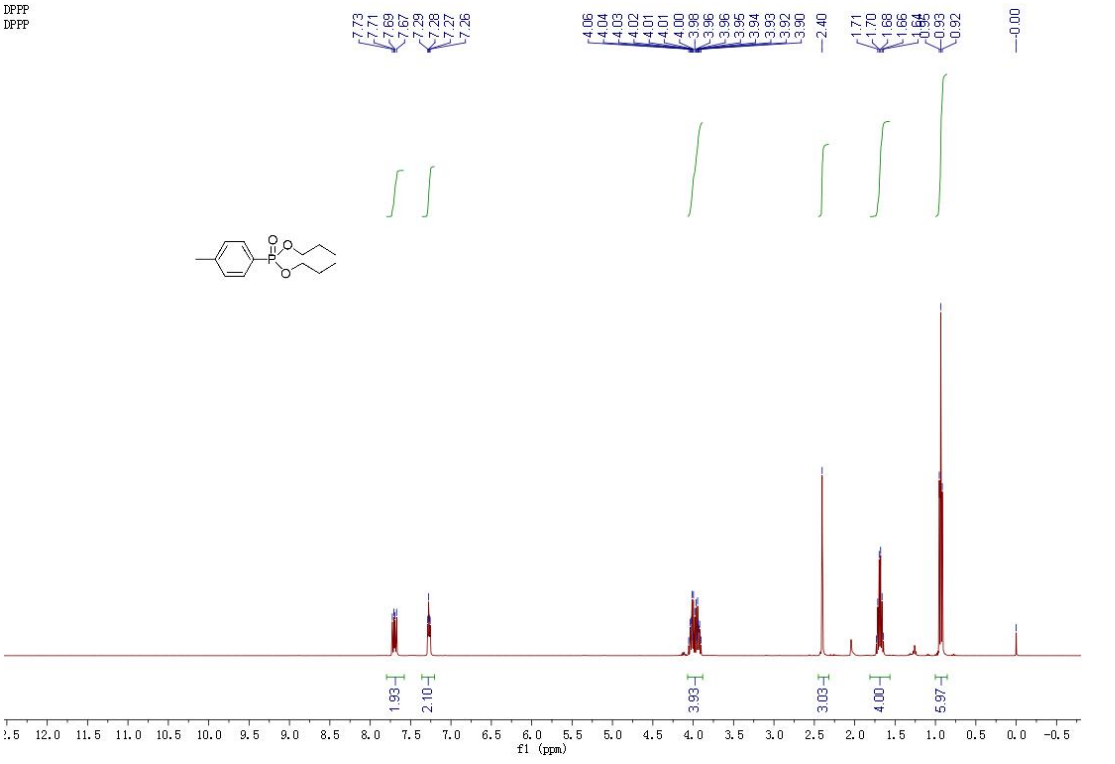

PMDM-13C
PMDM-13C
143.42
143.39
132.14
132.04
129.52
129.37
124.63
122.73
77.48
77.16
76.84
52.75
52.70
21.81
21.80
f1 (ppm)

3b


PMDM-31P
PMDM-31P
22.39
f1 (ppm)


2013-11-22-4-Me-Ph-P(O)(OMe)2 #481 RT: 8.50 AV: 1 NL: 3.59E5
T: + c Full ms [50.00-600.00]
Relative Abundance
m/z

3c


DPPP
DPPP
7.73
7.71
7.69
7.67
7.29
7.28
7.27
7.26
4.06
4.04
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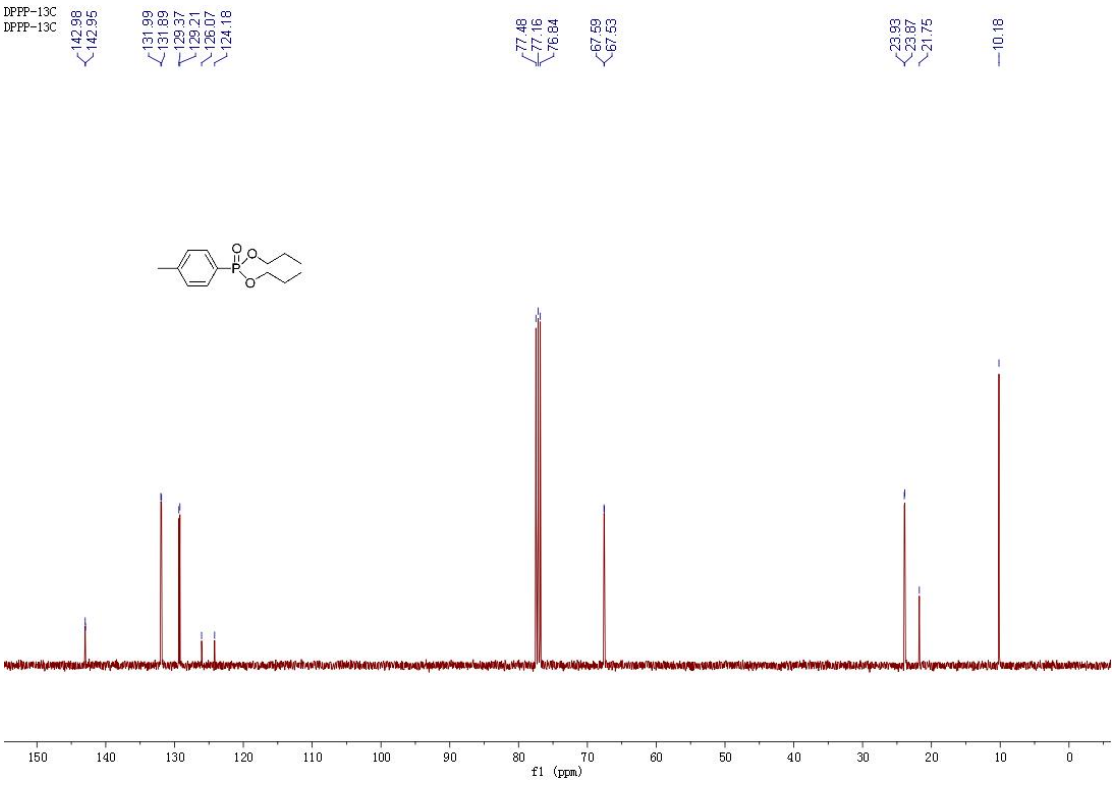
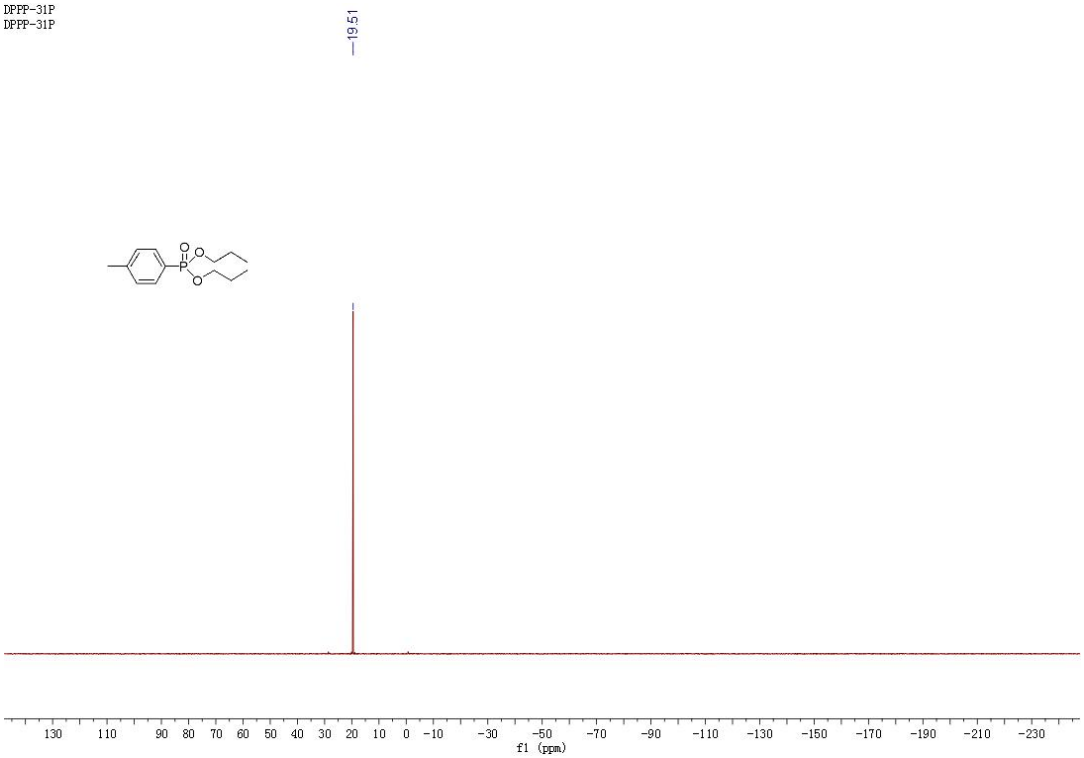
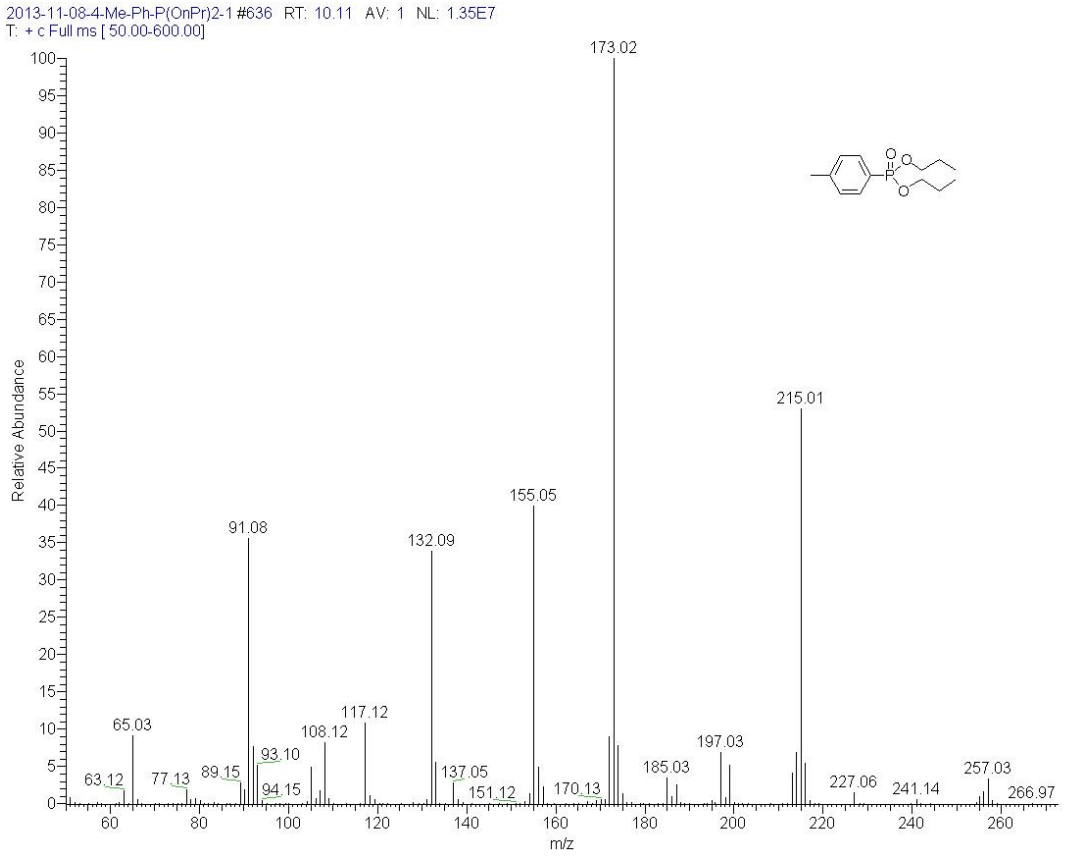
4.02
4.01
4.01
4.00
3.98
3.96
3.96
3.95
3.94
3.93
3.92
3.90
2.40
1.71
1.70
1.68
1.66
1.65
0.93
0.92
0.00
1.93
2.10
3.93
3.03
4.00
5.97
f1 (ppm)

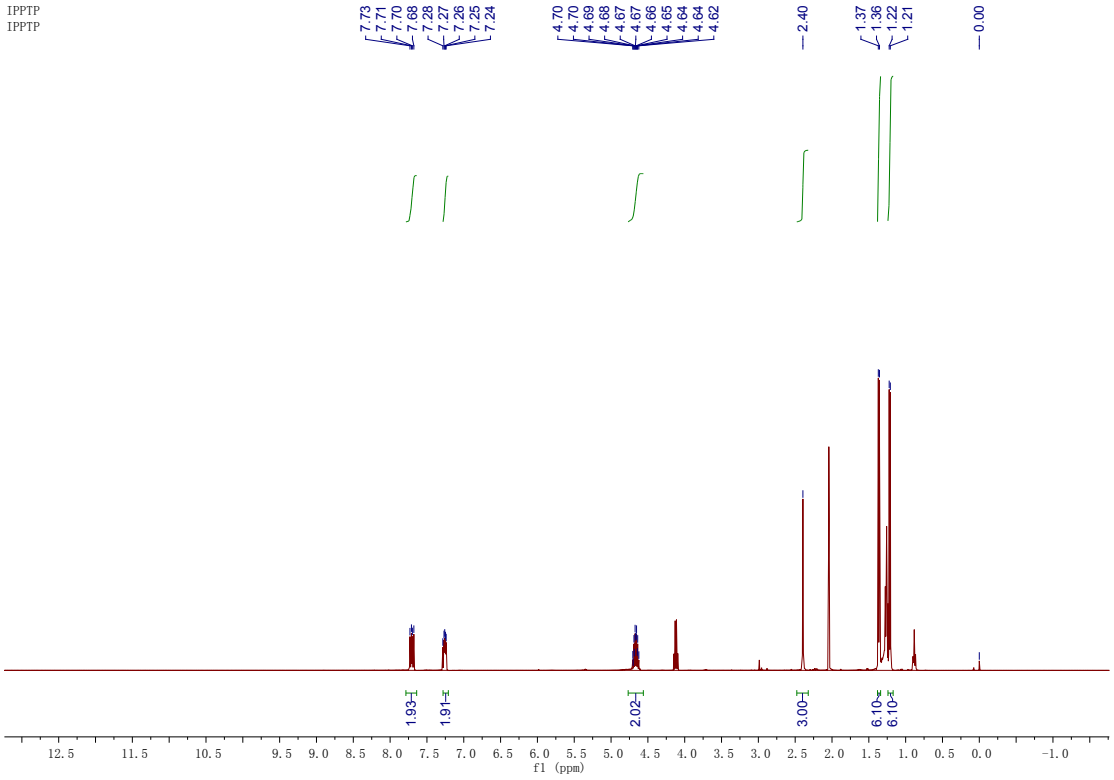

DPPP-13C
DPPP-13C
142.98
142.95
131.99
131.89
129.37
129.21
126.07
124.18
77.48
77.16
76.84
67.59
67.53
23.93
23.87
21.75
10.18
f1 (ppm)

3c


DPPP-31P
DPPP-31P
19.51
f1 (ppm)


2013-11-08-4-Me-Ph-P(OnPr)2-1 #636 RT: 10.11 AV: 1 NL: 1.35E7
T: + c Full ms [50.00-600.00]
Relative Abundance
m/z
173.02
215.01
155.05
91.08
132.09
117.12
65.03
108.12
93.10
197.03
137.05
185.03
257.03
63.12
77.13
89.15
94.15
151.12
170.13
227.06
241.14
266.97

3d


IPPTP
IPPTP
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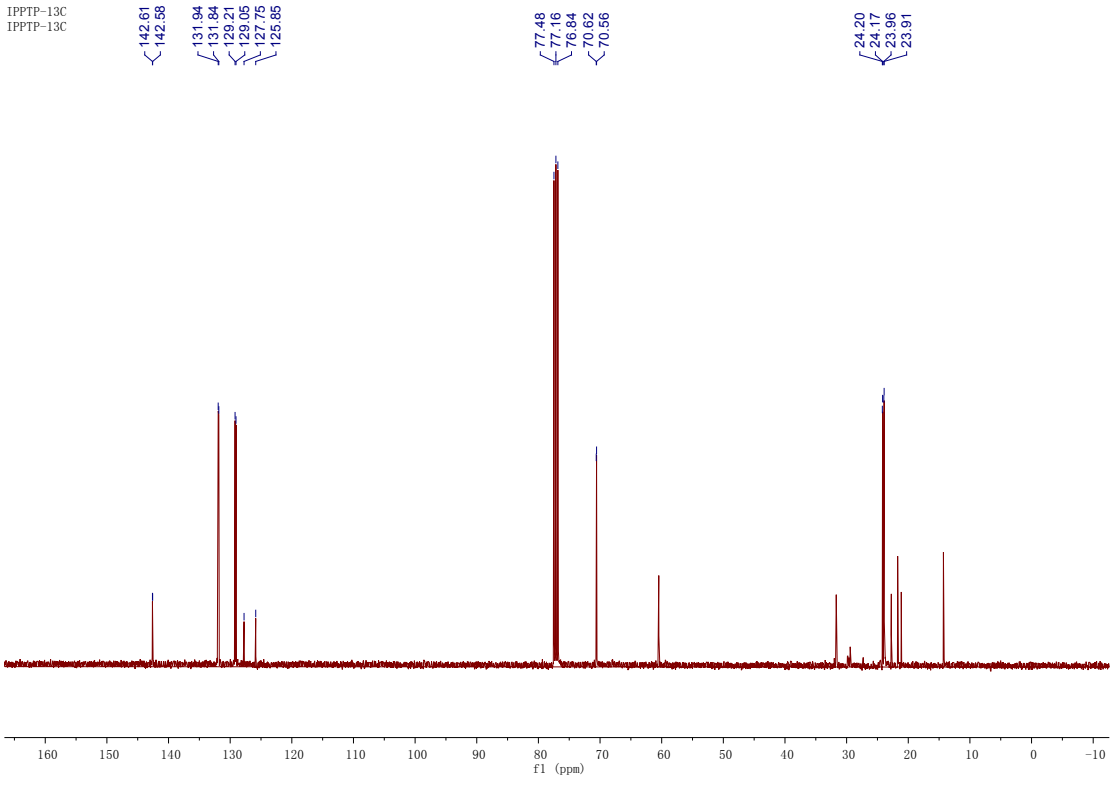
7.71
7.70
7.68
7.28
7.27
7.26
7.25
7.24
4.70
4.70
4.69
4.68
4.67
4.67
4.66
4.65
4.64
4.64
4.62
2.40
1.37
1.36
1.22
1.21
0.00
1.93
1.91
2.02
3.00
6.10
6.10
f1 (ppm)


IPPTP-13C
IPPTP-13C
142.61
142.58
131.94
131.84
129.21
129.05
127.75
125.85
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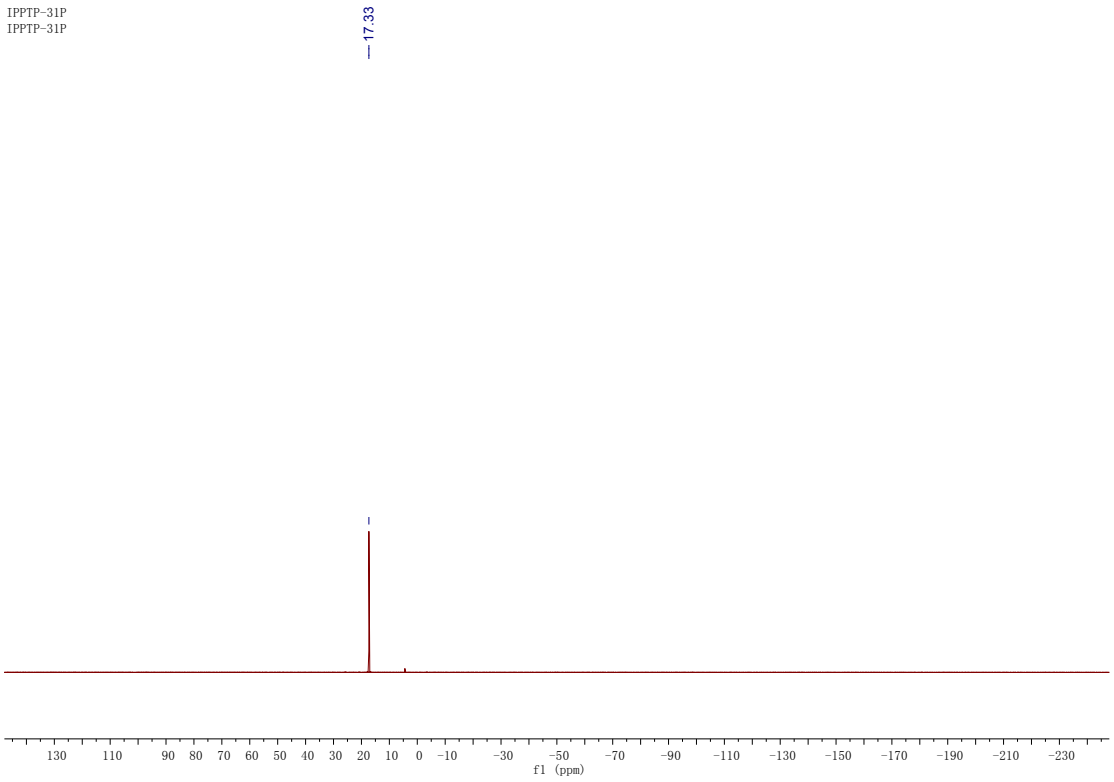
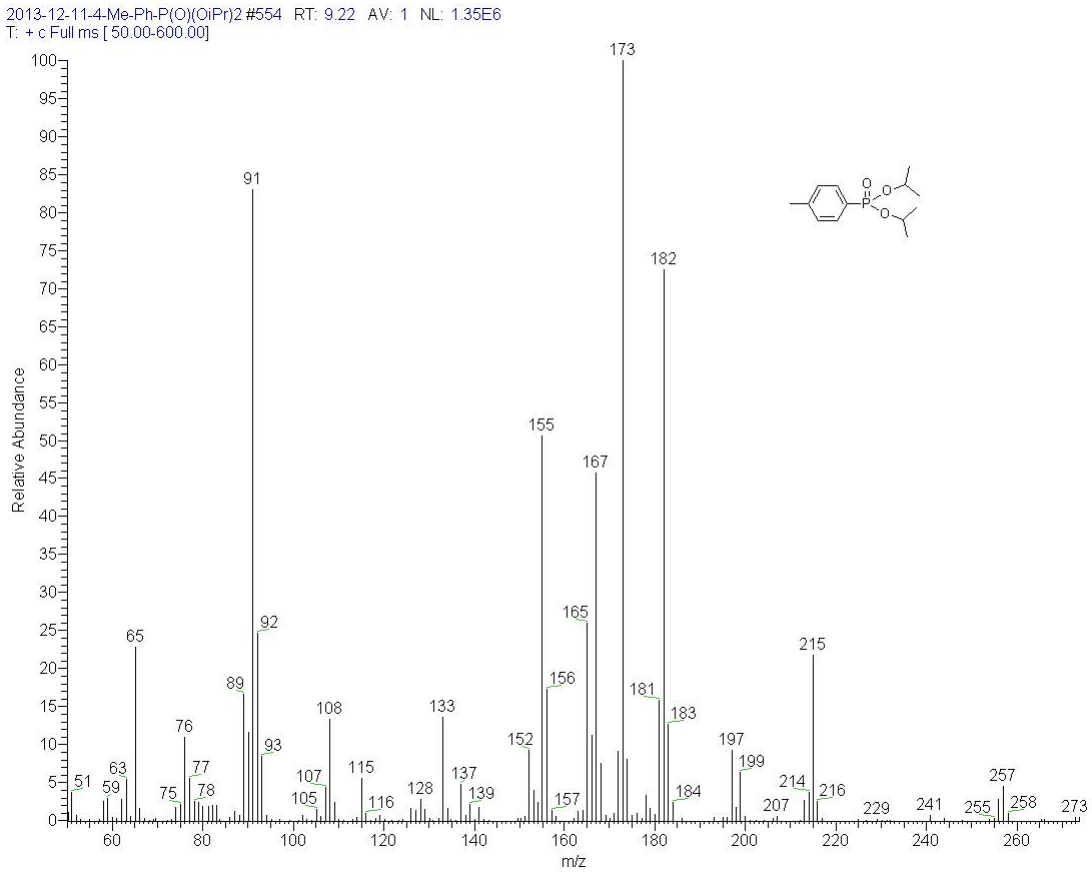
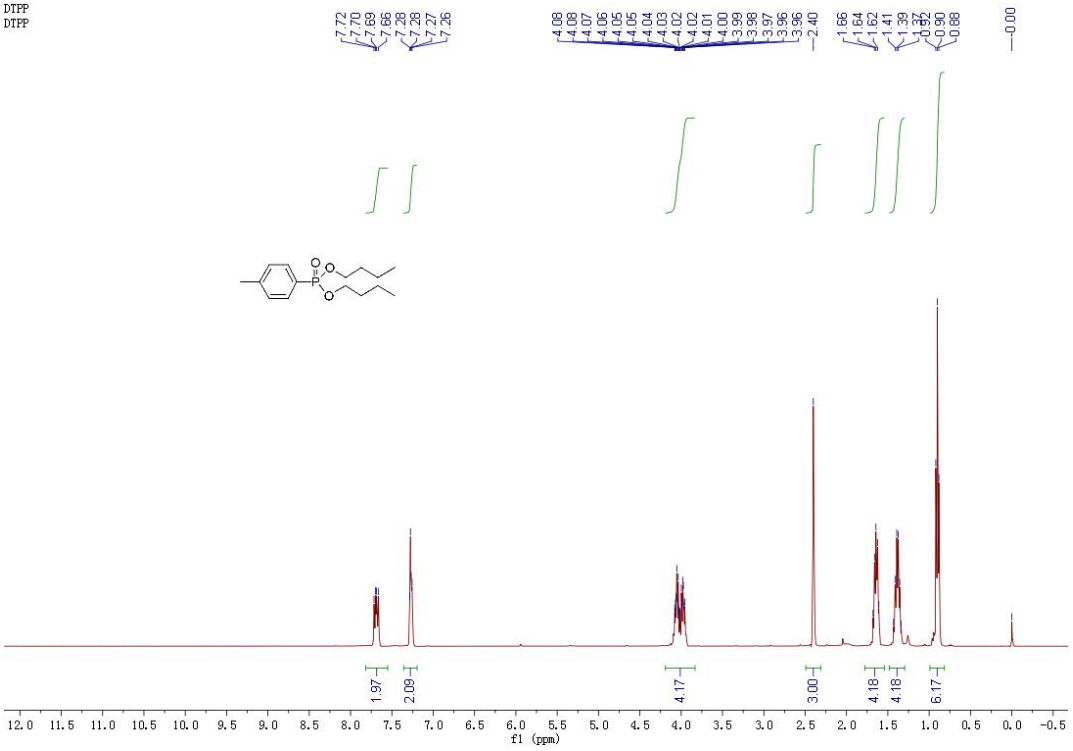
76.84
70.62
70.56
24.20
24.17
23.96
23.91
f1 (ppm)

3d


IPPTP-31P
IPPTP-31P
17.33
f1 (ppm)


2013-12-11-4-Me-Ph-P(O)(OiPr)2 #554 RT: 9.22 AV: 1 NL: 1.35E6
T: + c Full ms [50.00-600.00]
Relative Abundance
m/z

3e


DTPP
DTPP
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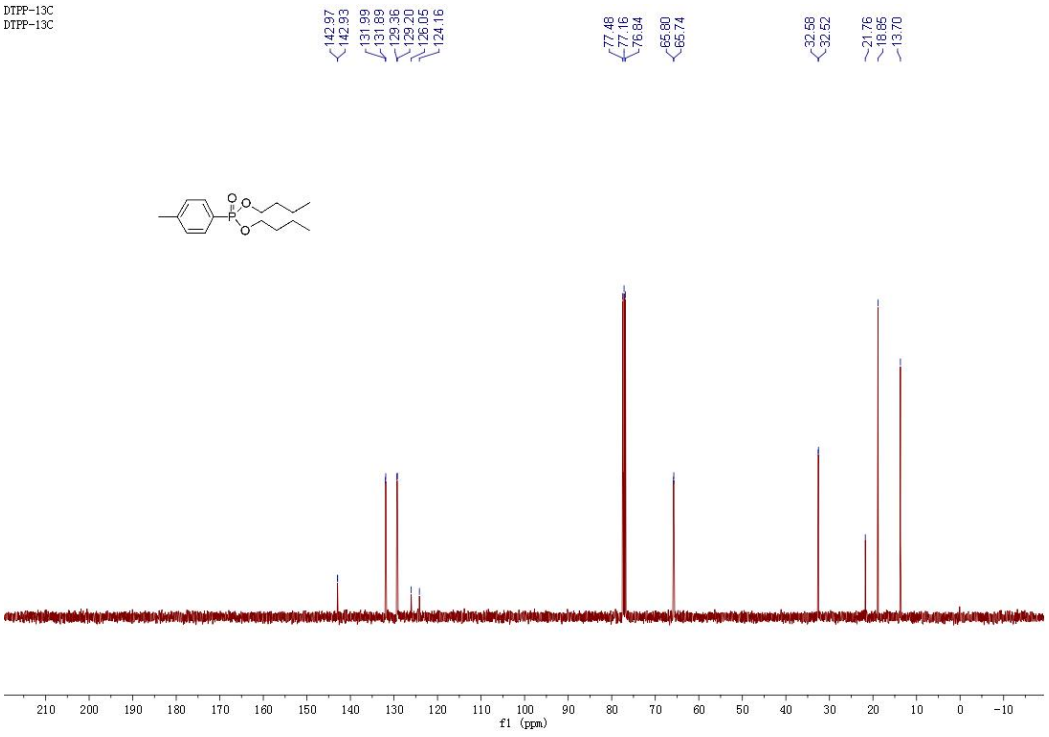
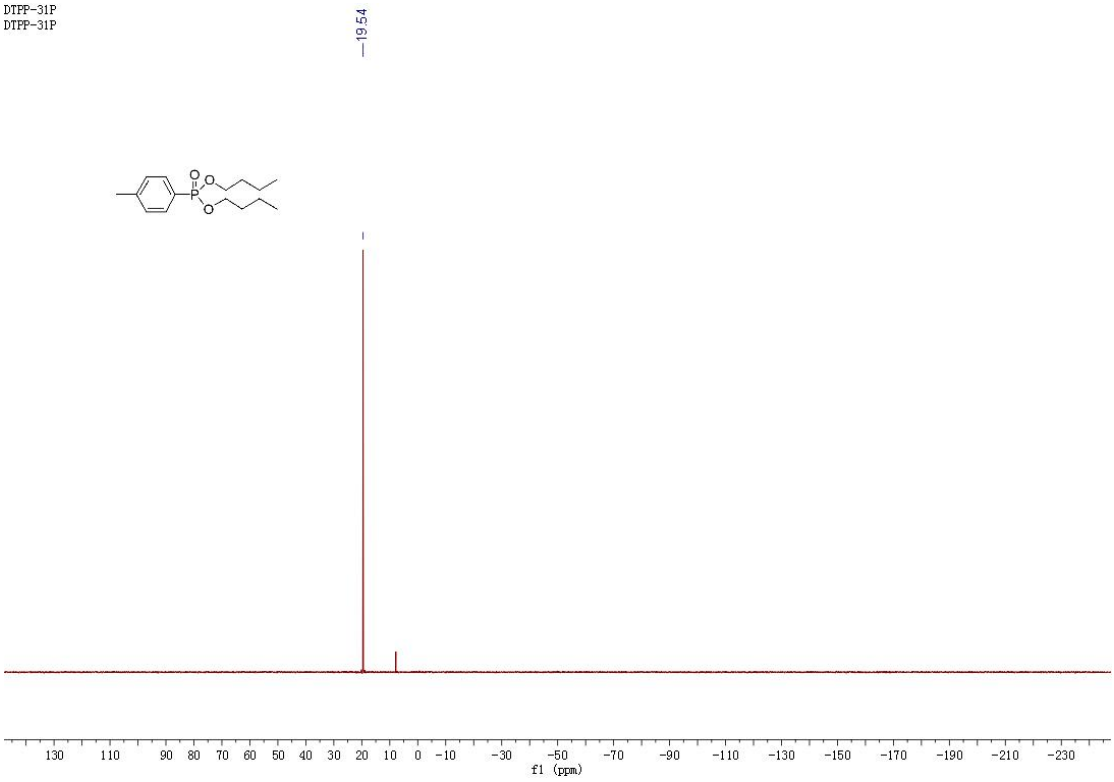
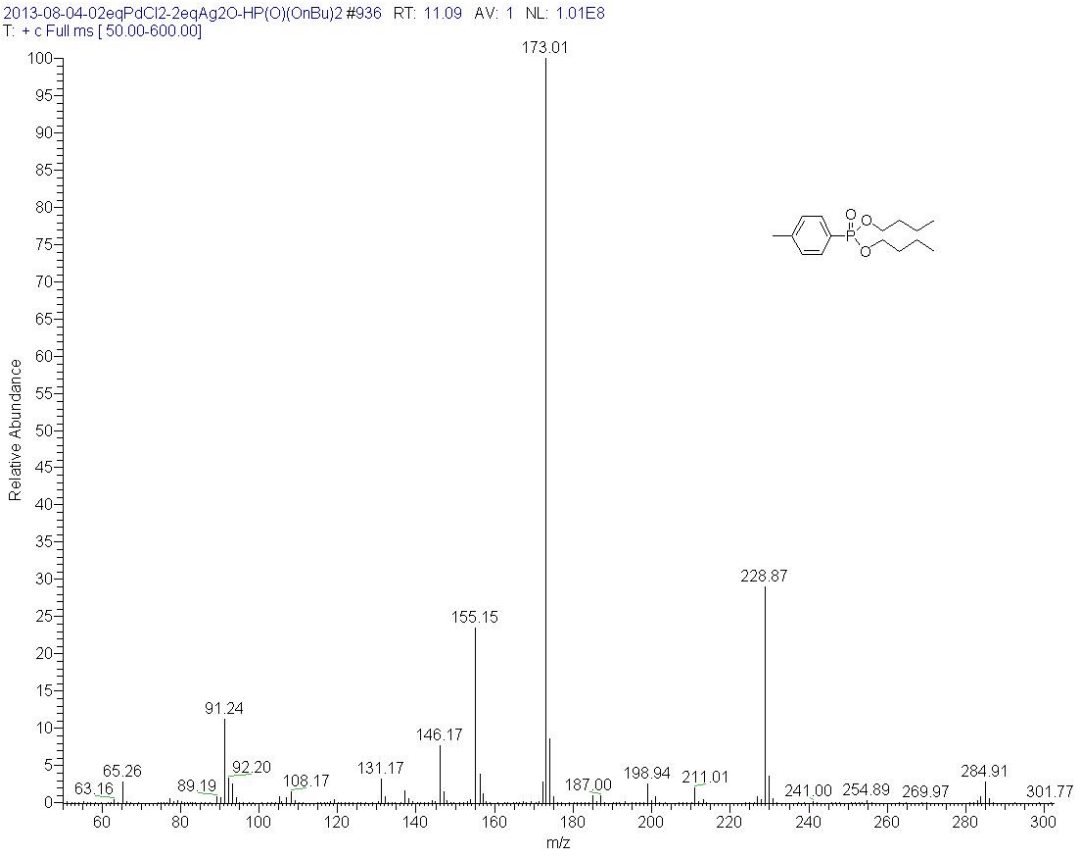
7.70
7.69
7.66
7.28
7.28
7.27
7.26
4.08
4.08
4.07
4.06
4.05
4.05
4.04
4.03
4.02
4.02
4.01
4.00
3.99
3.98
3.97
3.96
3.96
2.40
1.66
1.64
1.62
1.41
1.39
0.92
0.90
0.88
0.00
1.97
2.09
4.17
3.00
4.18
4.18
6.17
f1 (ppm)

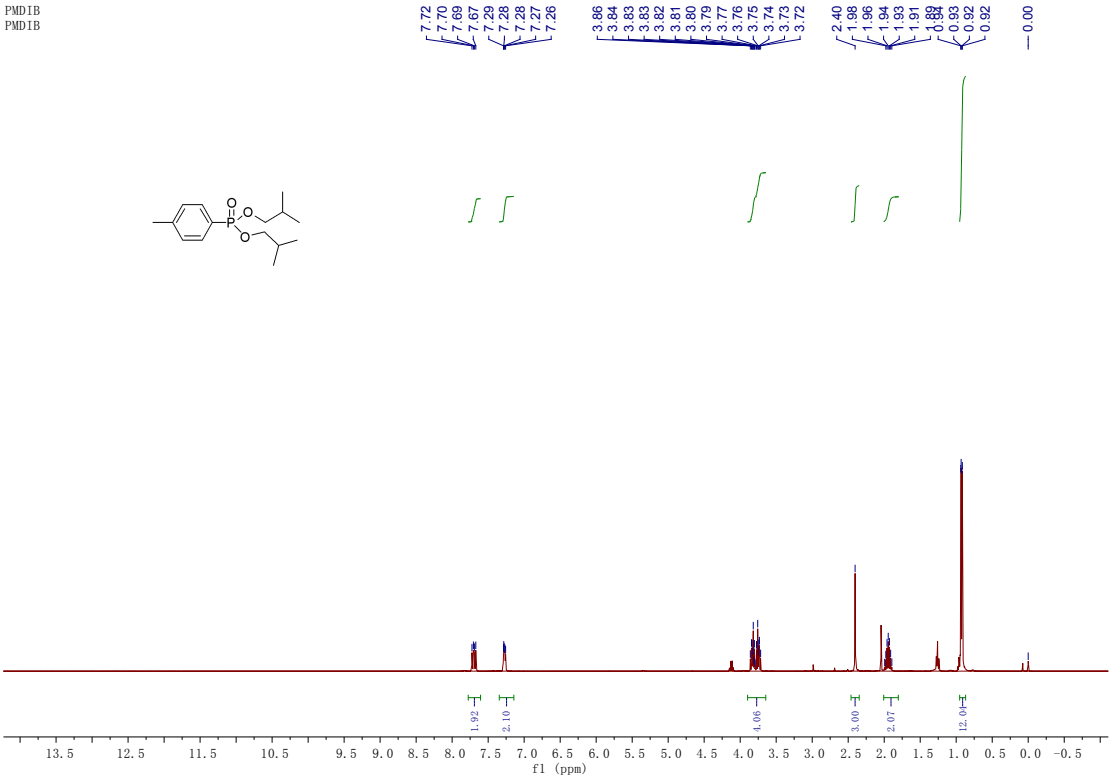

DTPP-13C
DTPP-13C
142.97
142.93
131.99
131.89
129.36
129.20
126.05
124.16
77.48
77.16
76.84
65.80
65.74
32.58
32.52
21.76
18.85
13.70
f1 (ppm)

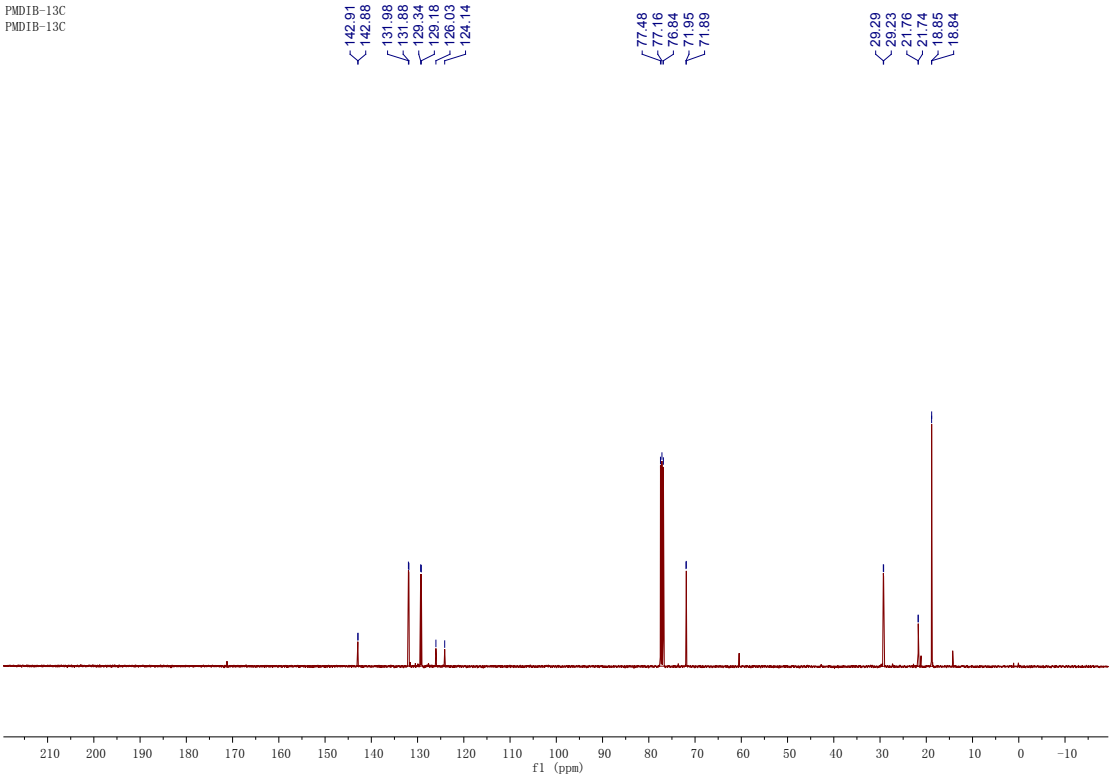
3e


DTPP-31P
DTPP-31P
19.54
f1 (ppm)

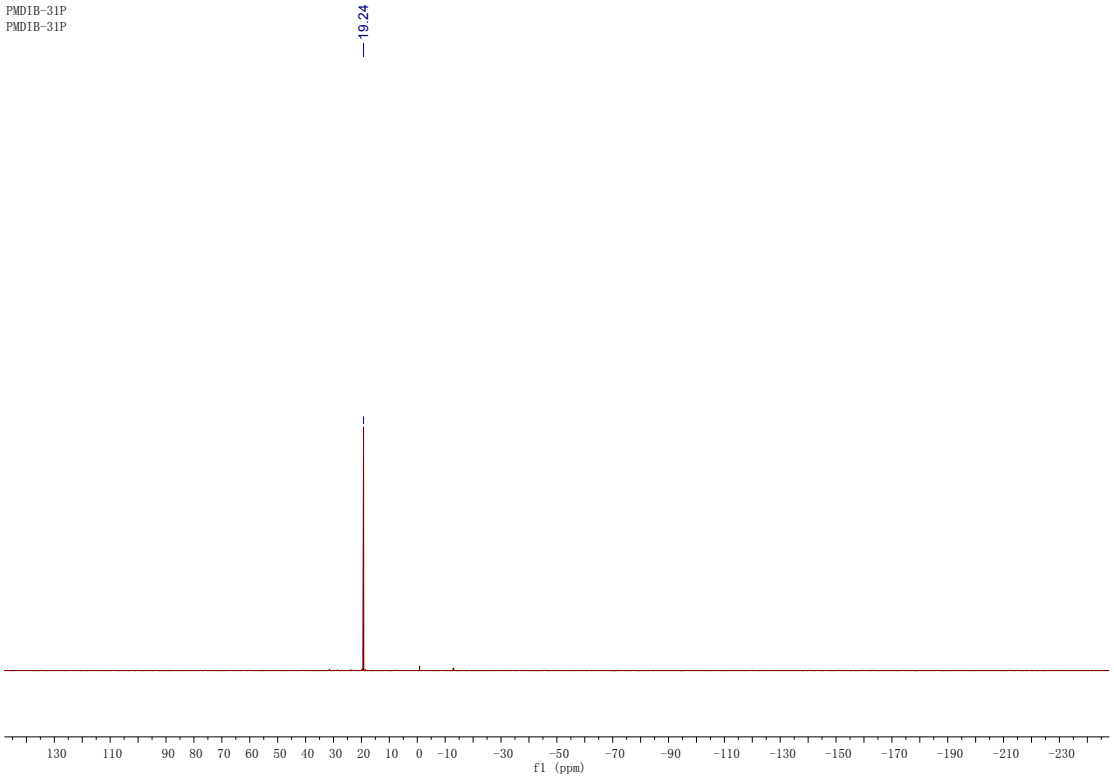

2013-08-04-02eqPdCl2-2eqAg2O-HP(O)(OnBu)2 #936 RT: 11.09 AV: 1 NL: 1.01E8
T: + c Full ms [50.00-600.00]
Relative Abundance
m/z
173.01
228.87
155.15
91.24
146.17
65.26
63.16
89.19
92.20
108.17
131.17
187.00
198.94
211.01
241.00
254.89
269.97
284.91
301.77

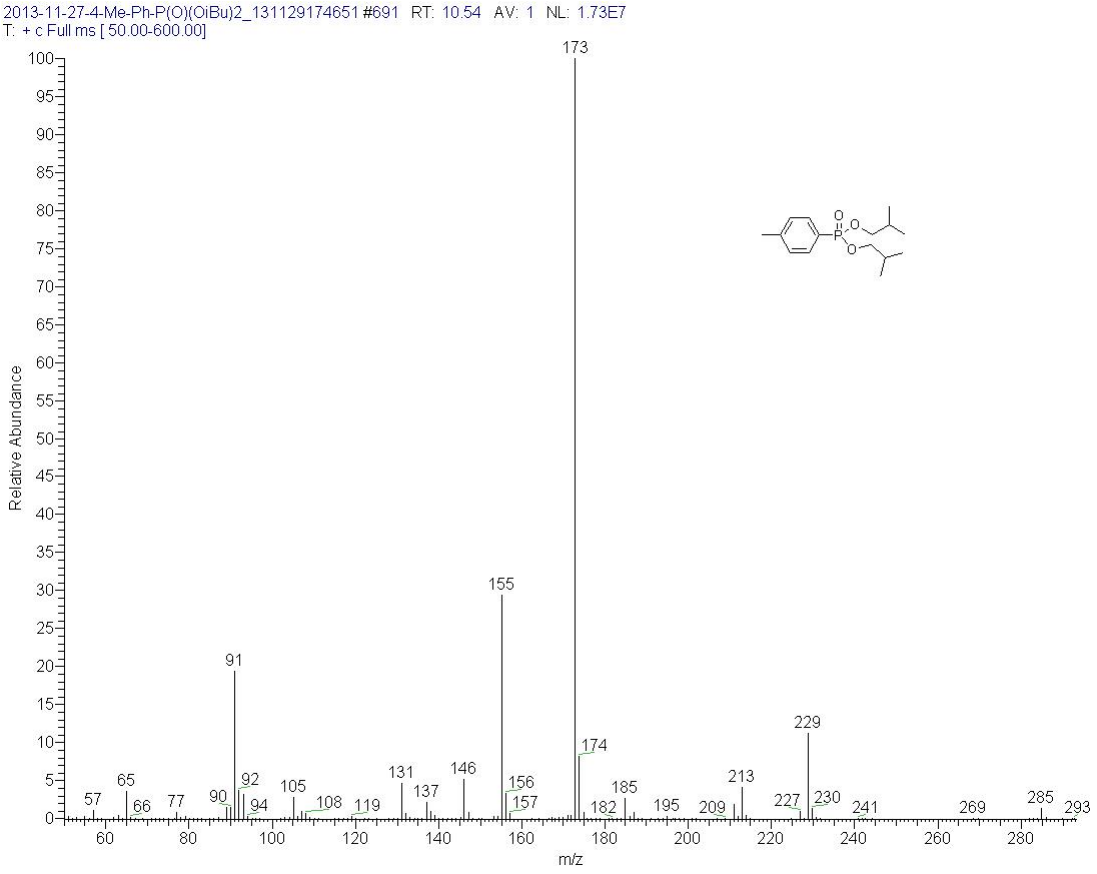
3f


PMDIB
PMDIB
7.72
7.70
7.69
7.67
7.29
7.28
7.28
7.27
7.26
3.86
3.84
3.83
3.83
3.82
3.81
3.80
3.79
3.77
3.76
3.75
3.74
3.73
3.72
2.40
1.98
1.96
1.94
1.93
1.91
0.93
0.92
0.92
0.00
1.92
2.10
4.06
3.00
2.07
12.00
f1 (ppm)

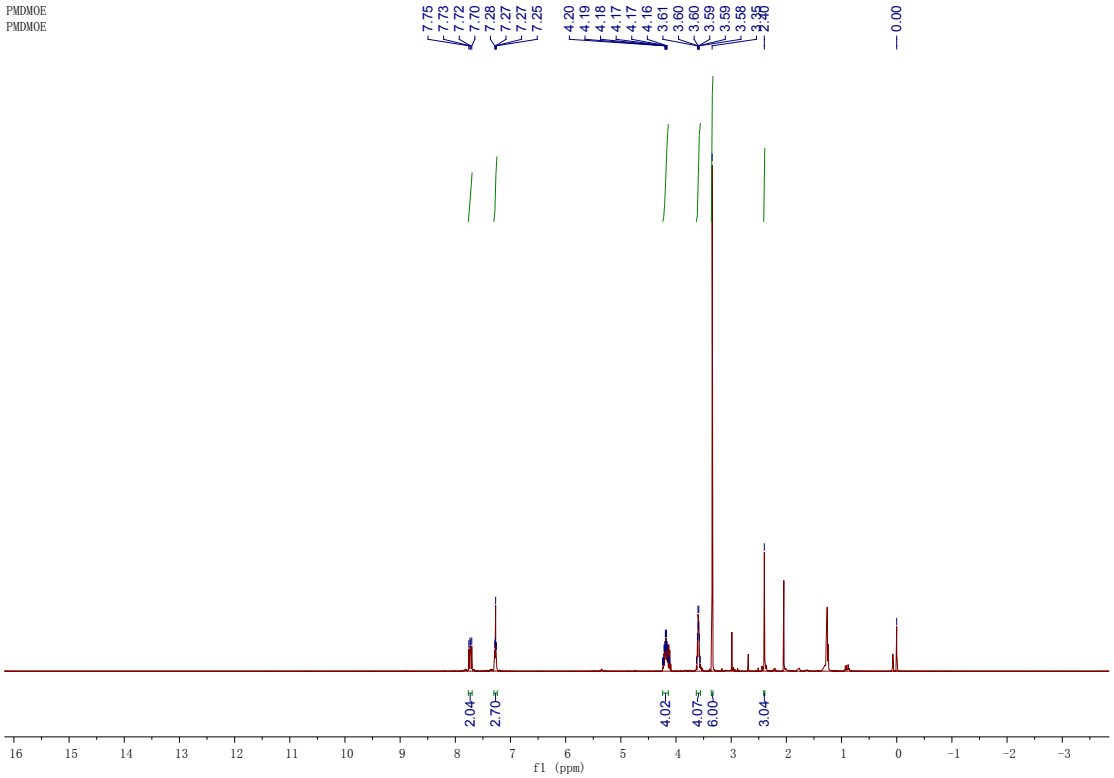

PMDIB-13C
PMDIB-13C
142.91
142.88
131.98
131.88
129.34
129.18
126.03
124.14
77.48
77.16
76.84
71.95
71.89
29.29
29.23
21.76
21.74
18.85
18.84
f1 (ppm)

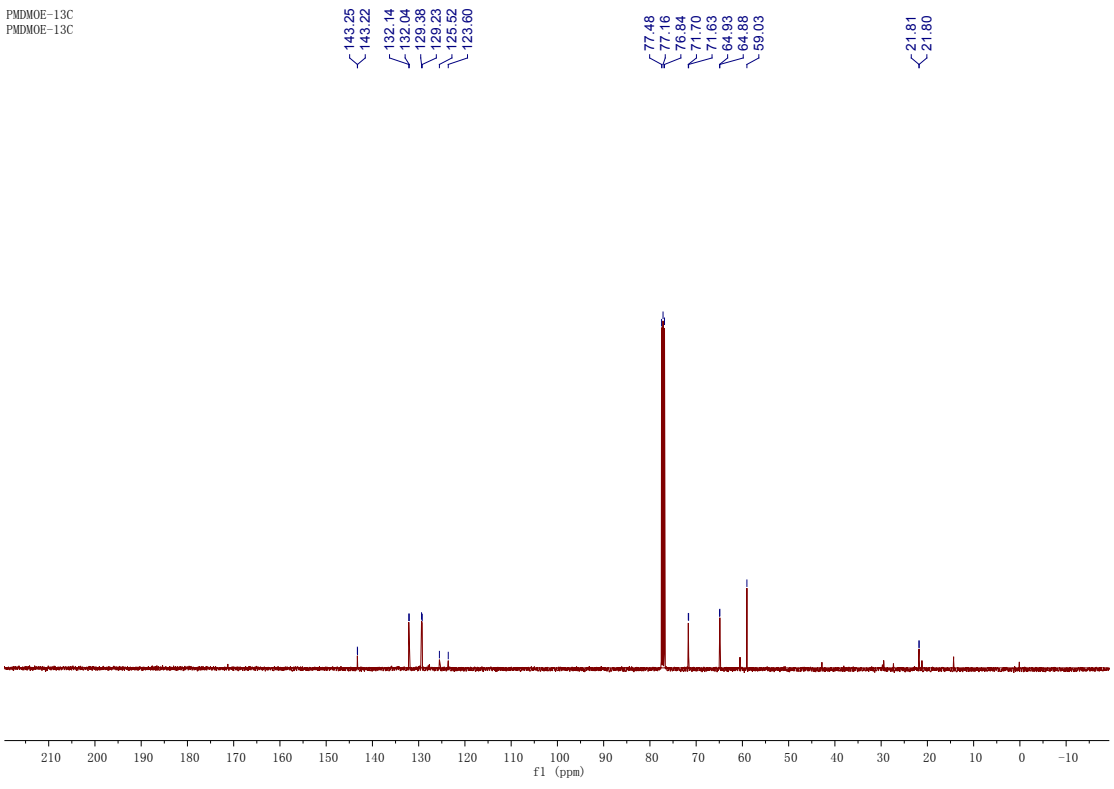
3f


PMDIB-31P
PMDIB-31P
19.24
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

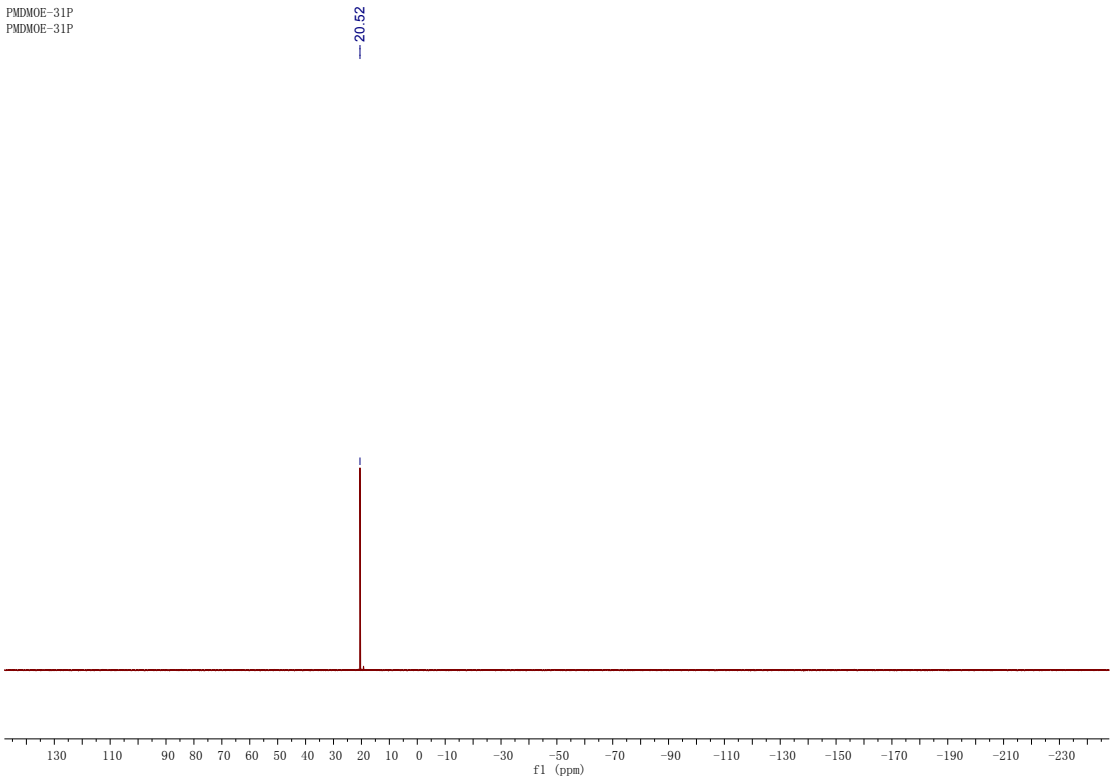

2013-11-27-4-Me-Ph-P(O)(OiBu)2_131129174651 #691 RT: 10.54 AV: 1 NL: 1.73E7
T: + c Full ms [50.00-600.00]
Relative Abundance
m/z
173
155
91
229
174
131
146
105
92
65
57
66
77
90
94
108
119
137
156
157
185
182
195
209
213
227
230
241
269
285
293

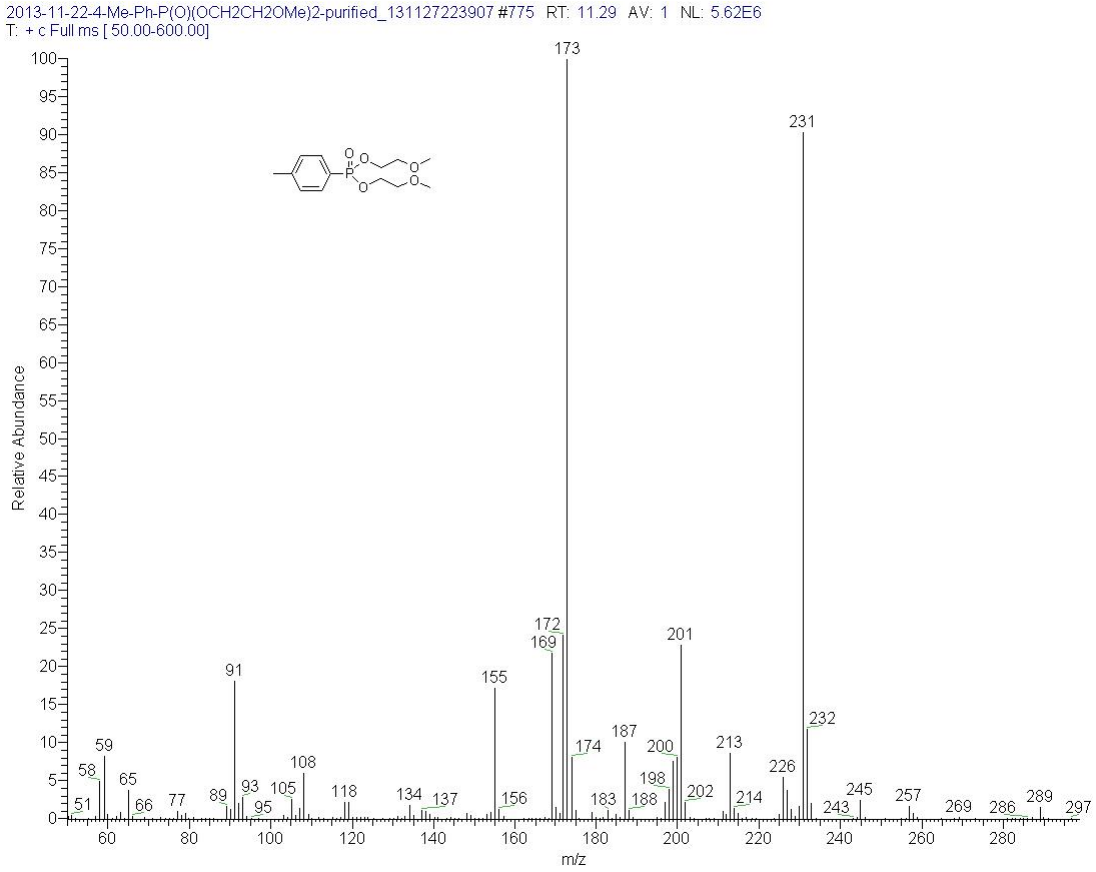
3h


PMDMOE
PMDMOE
7.75
7.73
7.72
7.70
7.28
7.27
7.27
7.25
4.20
4.19
4.18
4.17
4.17
4.16
3.61
3.60
3.60
3.59
3.59
3.58
3.35
2.40
0.00
2.04
2.70
4.02
4.07
6.00
3.04
f1 (ppm)

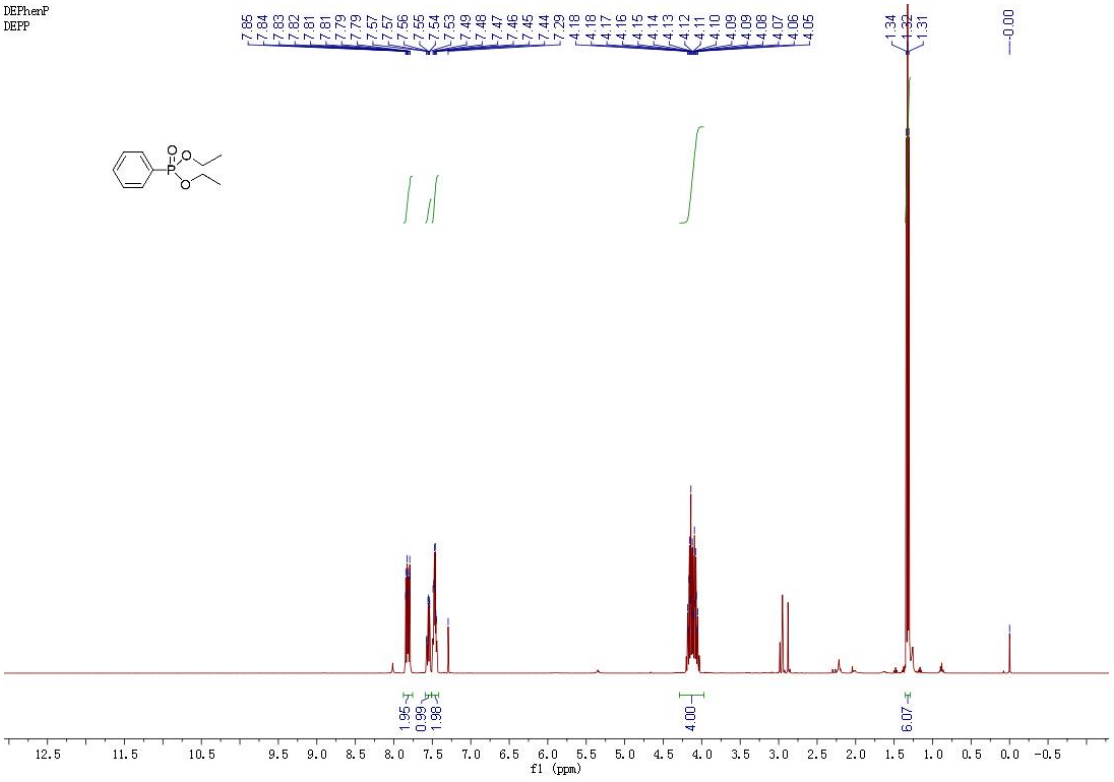

PMDMOE-13C
PMDMOE-13C
143.25
143.22
132.14
132.04
129.38
129.23
125.52
123.60
77.48
77.16
76.84
71.70
71.63
64.93
64.88
59.03
21.81
21.80
f1 (ppm)

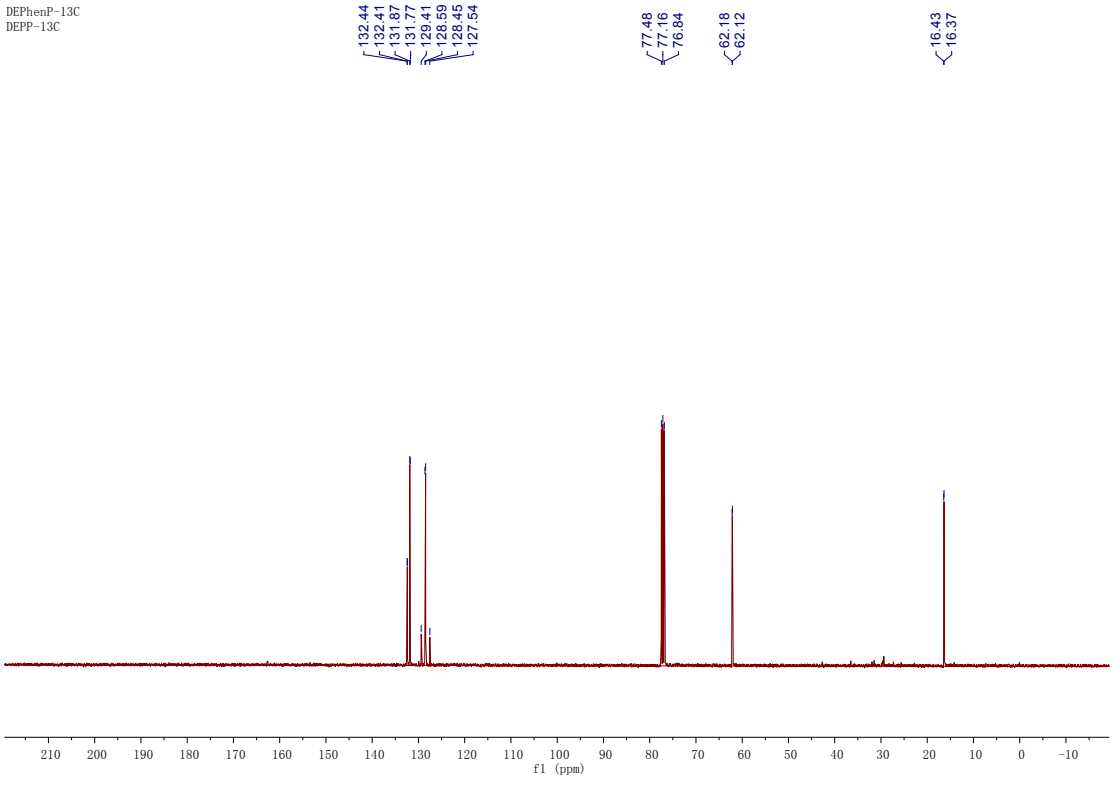
3h


PMDMOE-31P
PMDMOE-31P
20.52
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

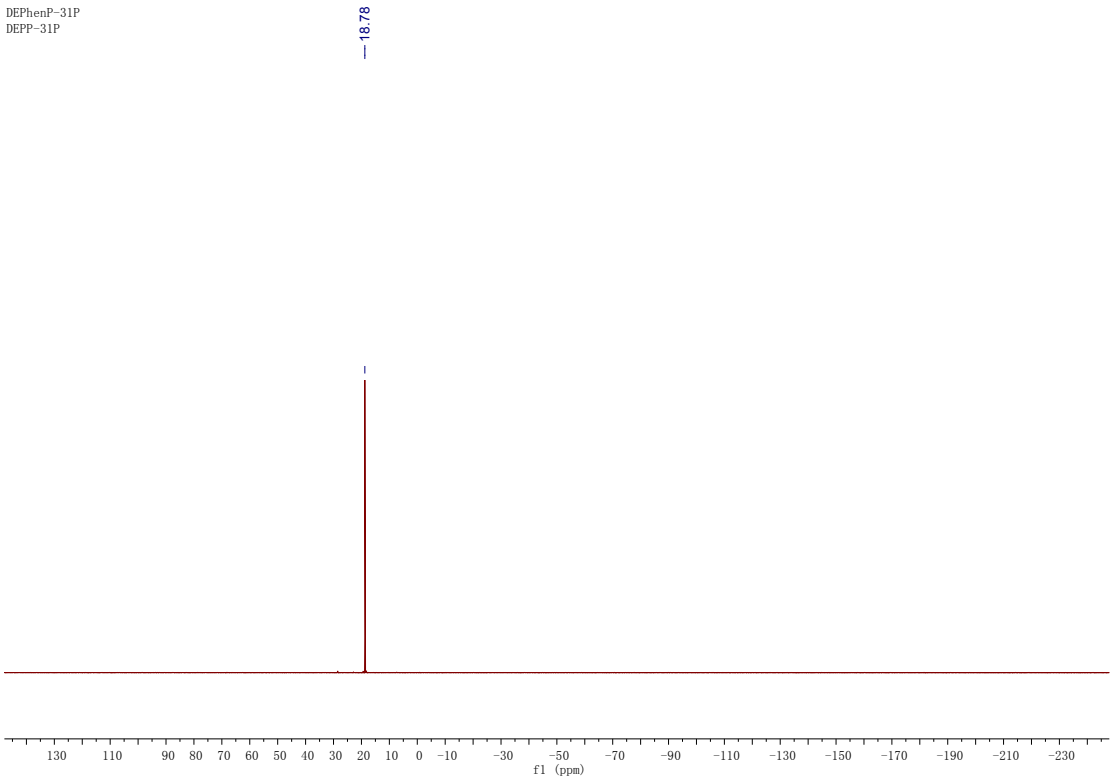

2013-11-22-4-Me-Ph-P(O)(OCH2CH2OMe)2-purified_131127223907 #775 RT: 11.29 AV: 1 NL: 5.62E6
T: + c Full ms [50.00-600.00]
Relative Abundance
m/z
173
231
91
155
172
169
201
232
59
58
65
108
174
187
200
213
226
198
202
214
245
257
269
286
289
297
51
66
77
89
93
95
105
118
134
137
156
183
188
243

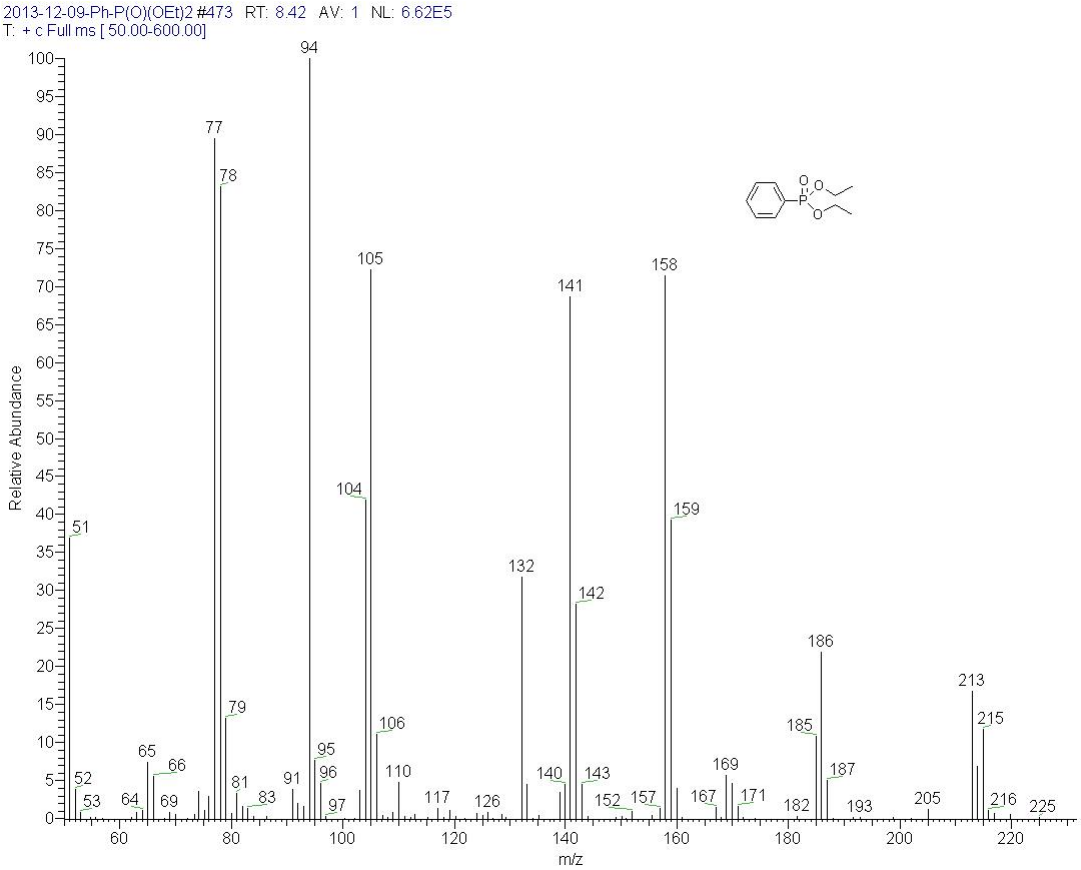
3i


DEPhenP
DEPP
7.85
7.84
7.83
7.82
7.81
7.81
7.79
7.79
7.57
7.57
7.56
7.55
7.54
7.54
7.53
7.49
7.48
7.47
7.46
7.45
7.44
7.29
4.18
4.18
4.17
4.16
4.15
4.14
4.13
4.12
4.11
4.10
4.09
4.09
4.08
4.07
4.06
4.05
1.34
1.32
1.31
0.00
1.95
0.99
1.98
4.00
6.07
f1 (ppm)

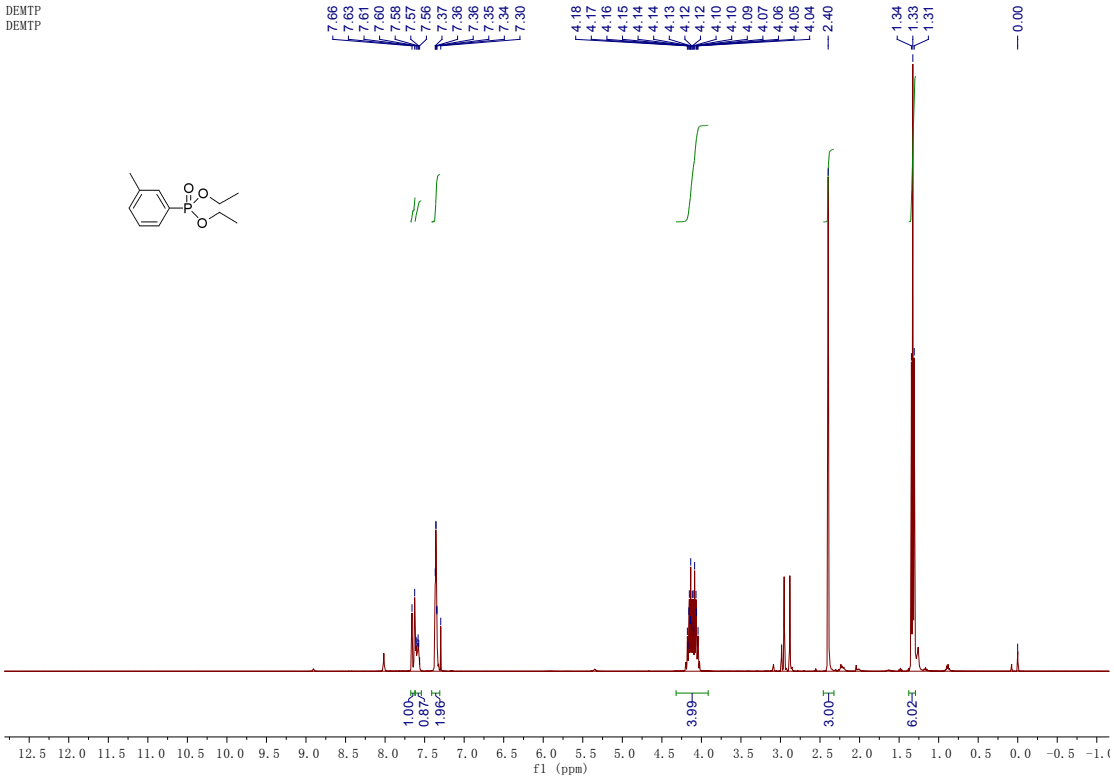

DEPhenP-13C
DEPP-13C
132.44
132.41
131.87
131.77
129.41
128.59
128.45
127.54
77.48
77.16
76.84
62.18
62.12
16.43
16.37
f1 (ppm)

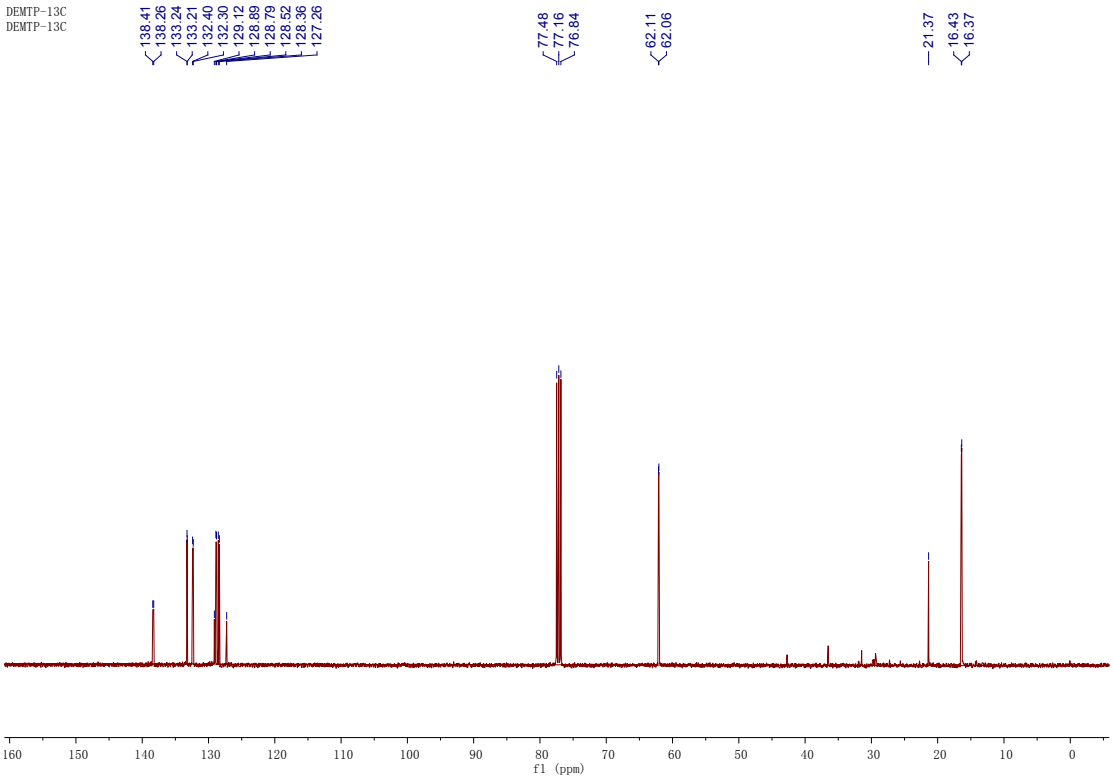
3i


DEPhenP-31P
DEPP-31P
18.78
f1 (ppm)

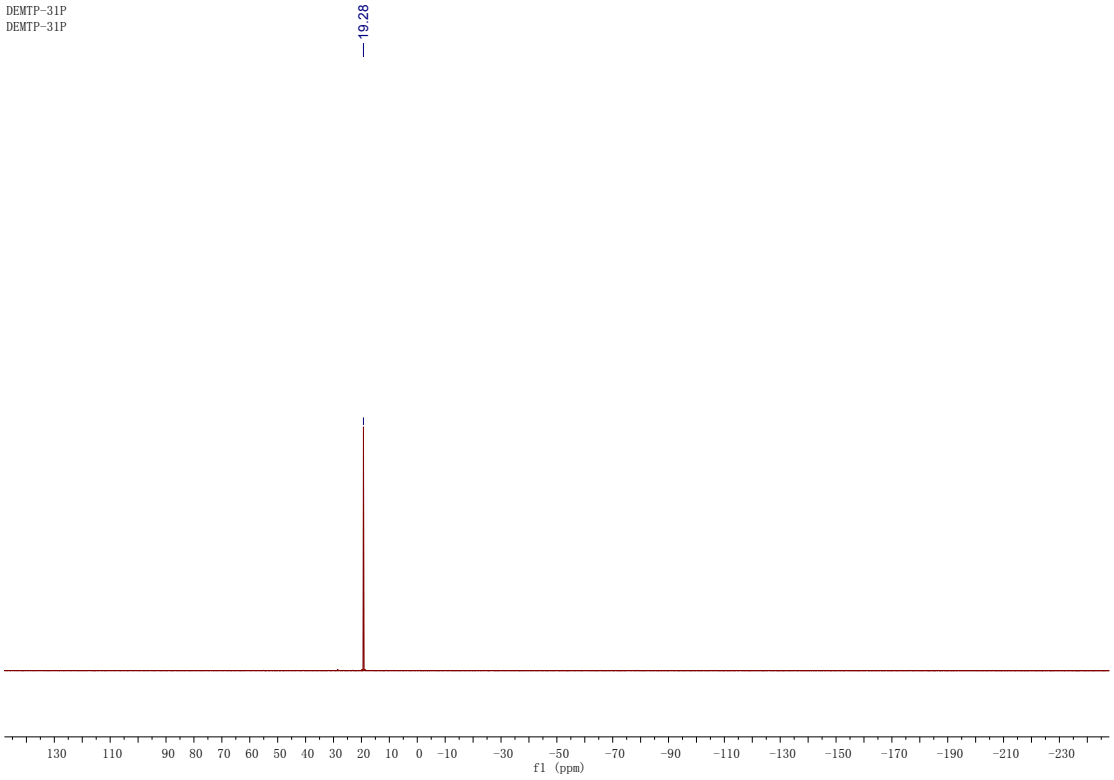

2013-12-09-Ph-P(O)(OEt)2 #473 RT: 8.42 AV: 1 NL: 6.62E5
T: + c Full ms [50.00-600.00]
Relative Abundance
m/z

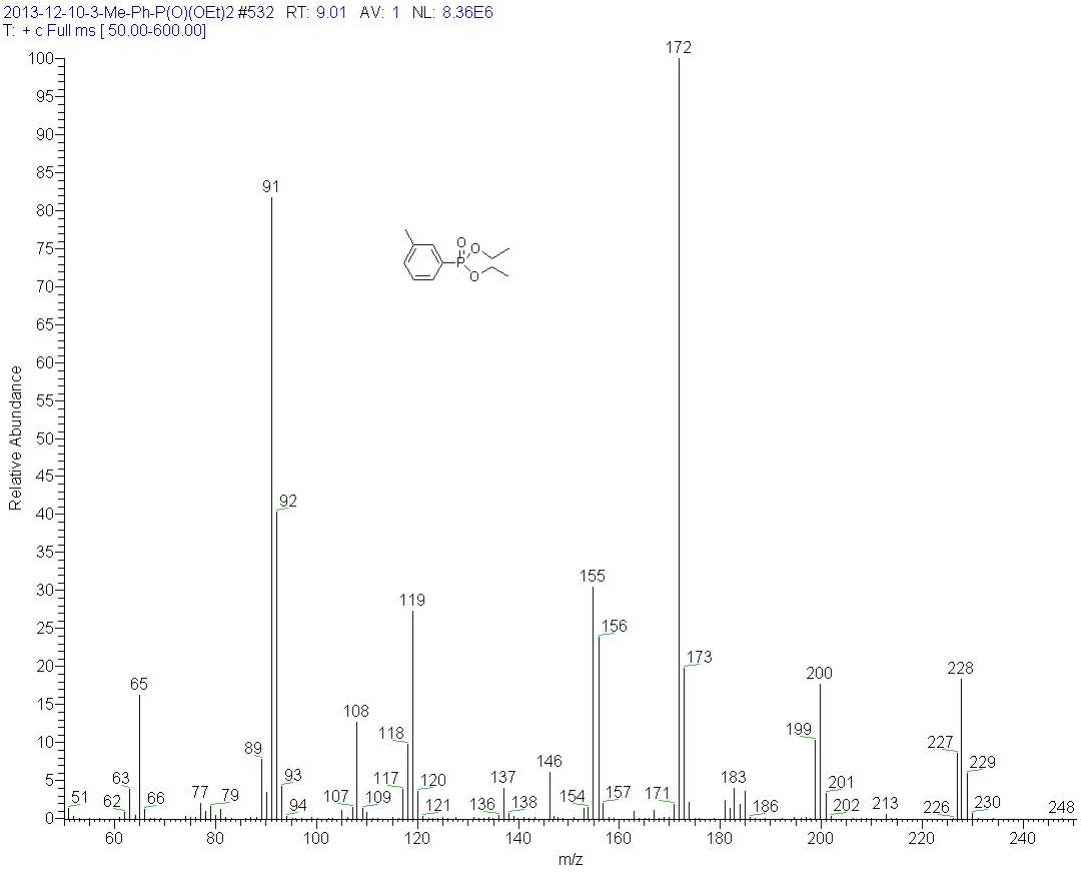
3j


DEMTP
DEMTP
7.66
7.63
7.61
7.60
7.58
7.57
7.56
7.37
7.36
7.36
7.35
7.34
7.30
4.18
4.17
4.16
4.15
4.14
4.14
4.13
4.12
4.12
4.10
4.10
4.09
4.07
4.06
4.05
4.04
2.40
1.34
1.33
1.31
0.00
1.00
0.87
1.96
3.99
3.00
6.02
f1 (ppm)

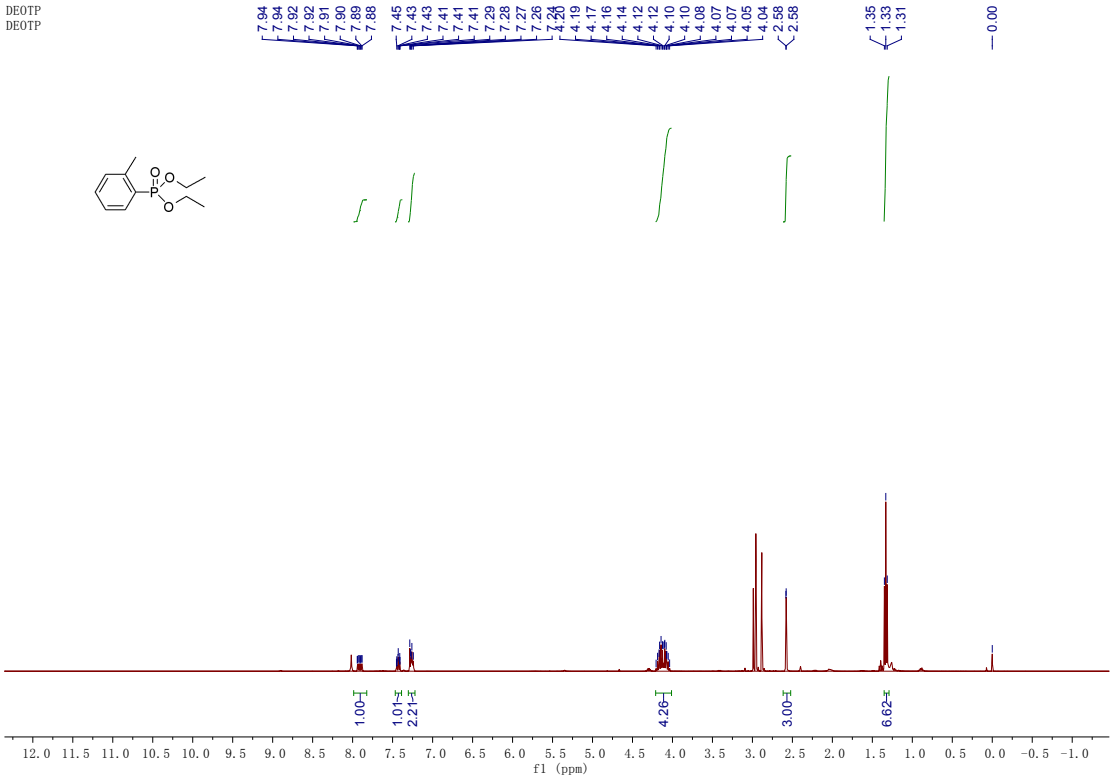

DEMTP-13C
DEMTP-13C
138.41
138.26
133.24
133.21
132.40
132.30
129.12
128.89
128.79
128.52
128.36
127.26
77.48
77.16
76.84
62.11
62.06
21.37
16.43
16.37
f1 (ppm)

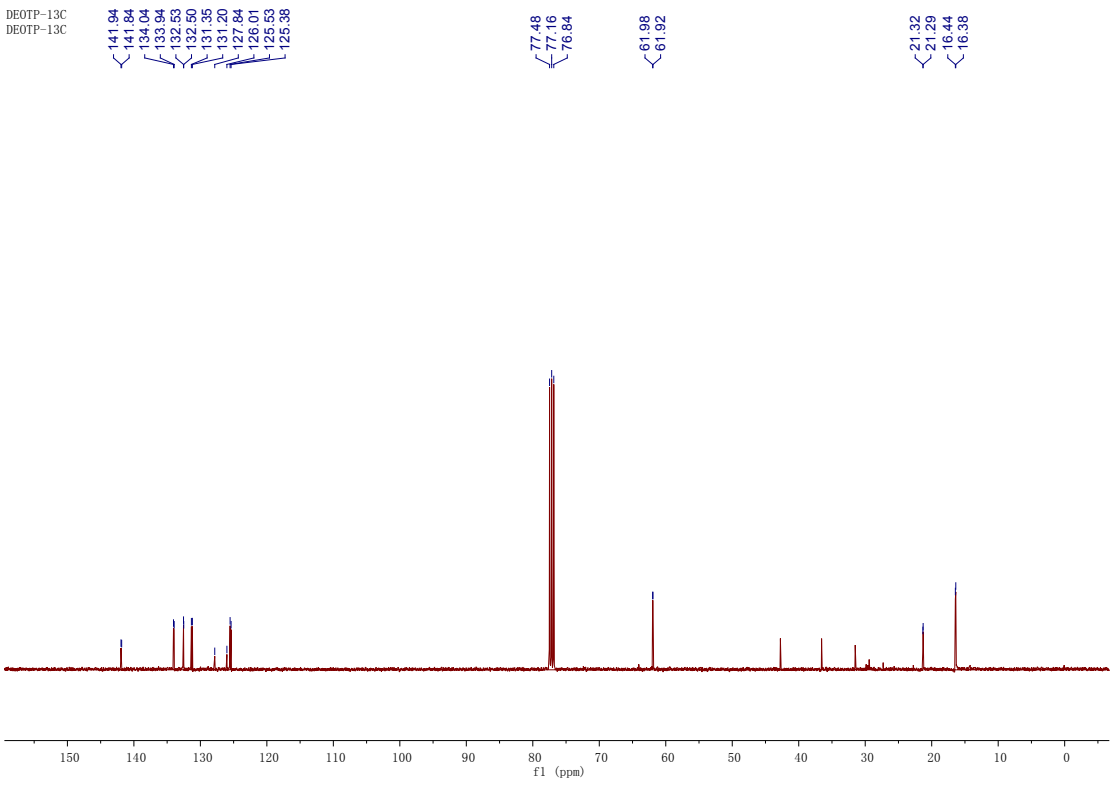
3j


DEMTP-31P
DEMTP-31P
19.28
f1 (ppm)

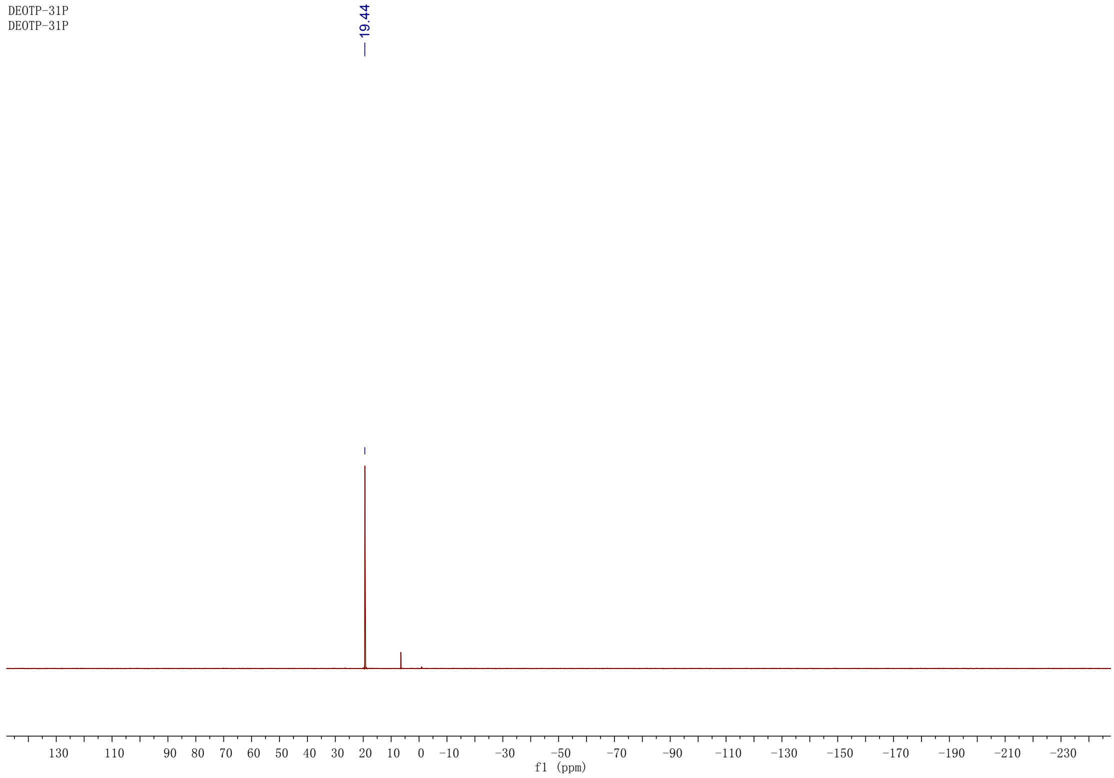

2013-12-10-3-Me-Ph-P(O)(OEt)2 #532 RT: 9.01 AV: 1 NL: 8.36E6
T: + c Full ms [50.00-600.00]
Relative Abundance
m/z

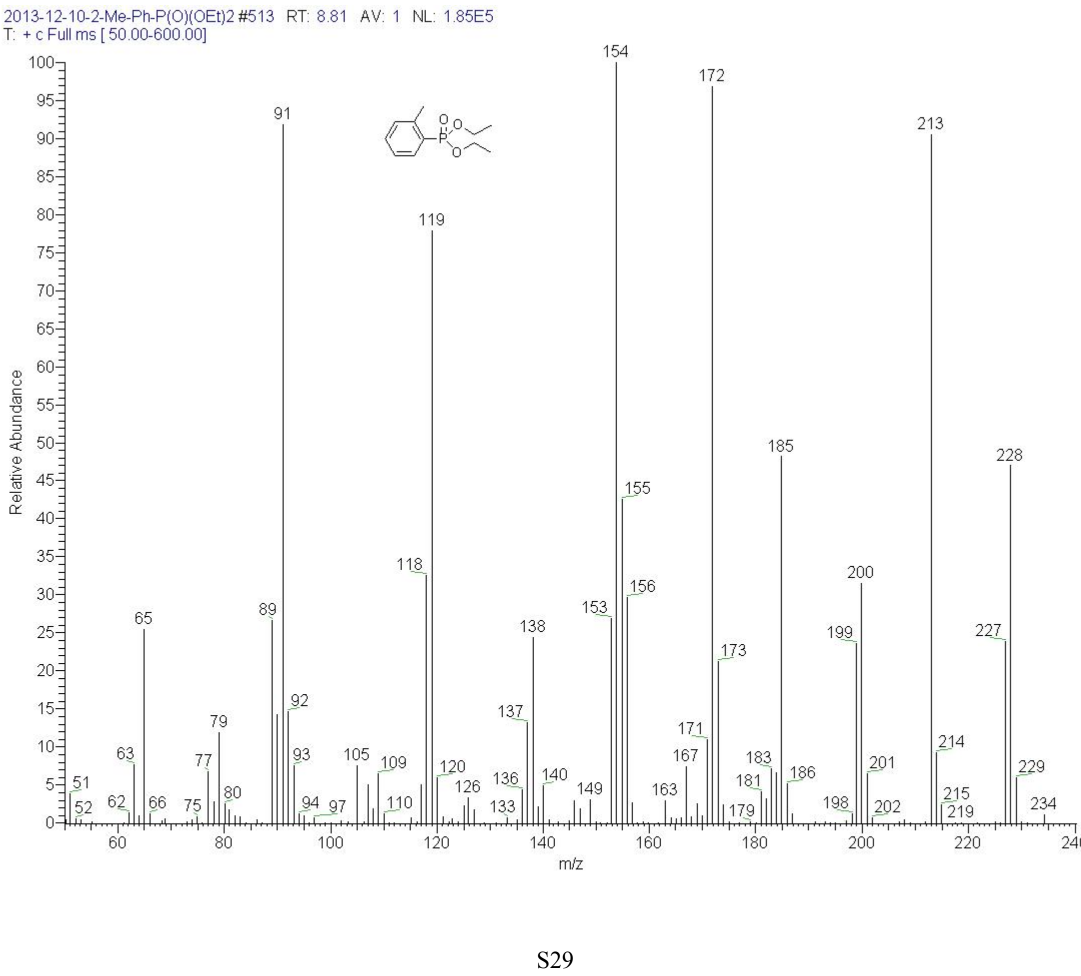
3k


DEOTP
DEOTP
7.94
7.94
7.92
7.92
7.91
7.90
7.89
7.88
7.45
7.43
7.43
7.41
7.41
7.41
7.29
7.28
7.27
7.26
4.19
4.17
4.16
4.14
4.12
4.12
4.10
4.10
4.08
4.07
4.07
4.05
4.04
2.58
2.58
1.35
1.33
1.31
0.00
1.00
1.01
2.21
4.26
3.00
6.62
f1 (ppm)

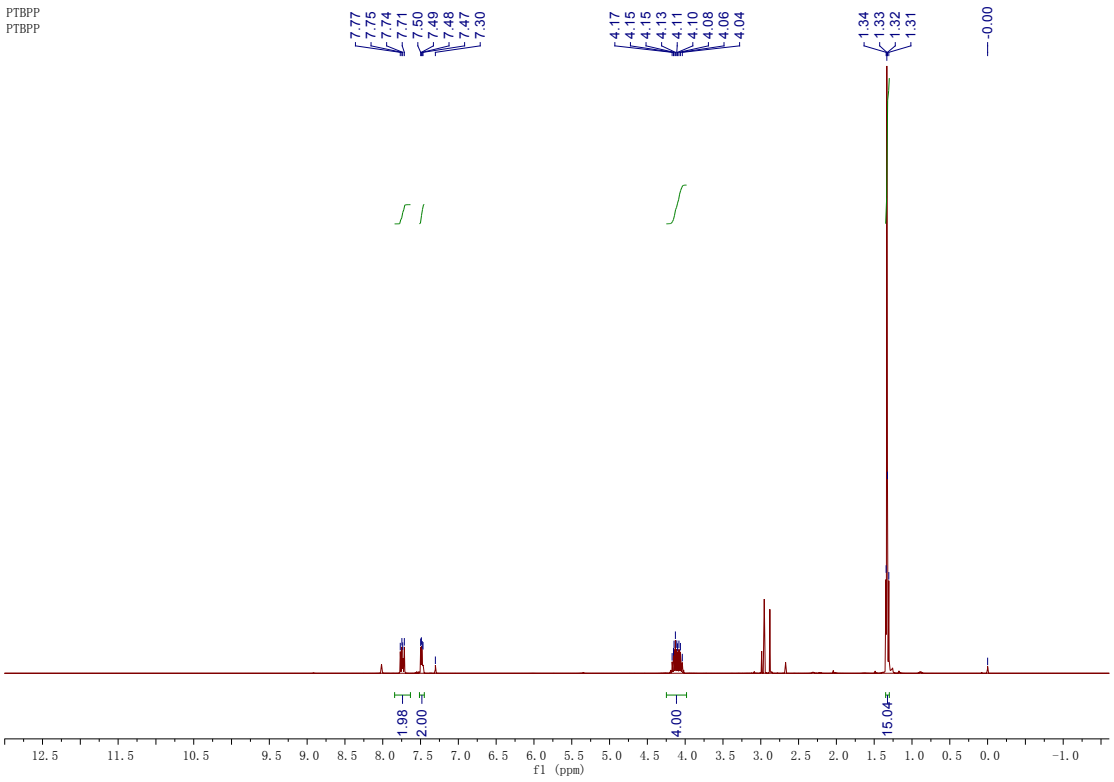

DEOTP-13C
DEOTP-13C
141.94
141.84
134.04
133.94
132.53
132.50
131.35
131.20
127.84
126.01
125.53
125.38
77.48
77.16
76.84
61.98
61.92
21.32
21.29
16.44
16.38
f1 (ppm)

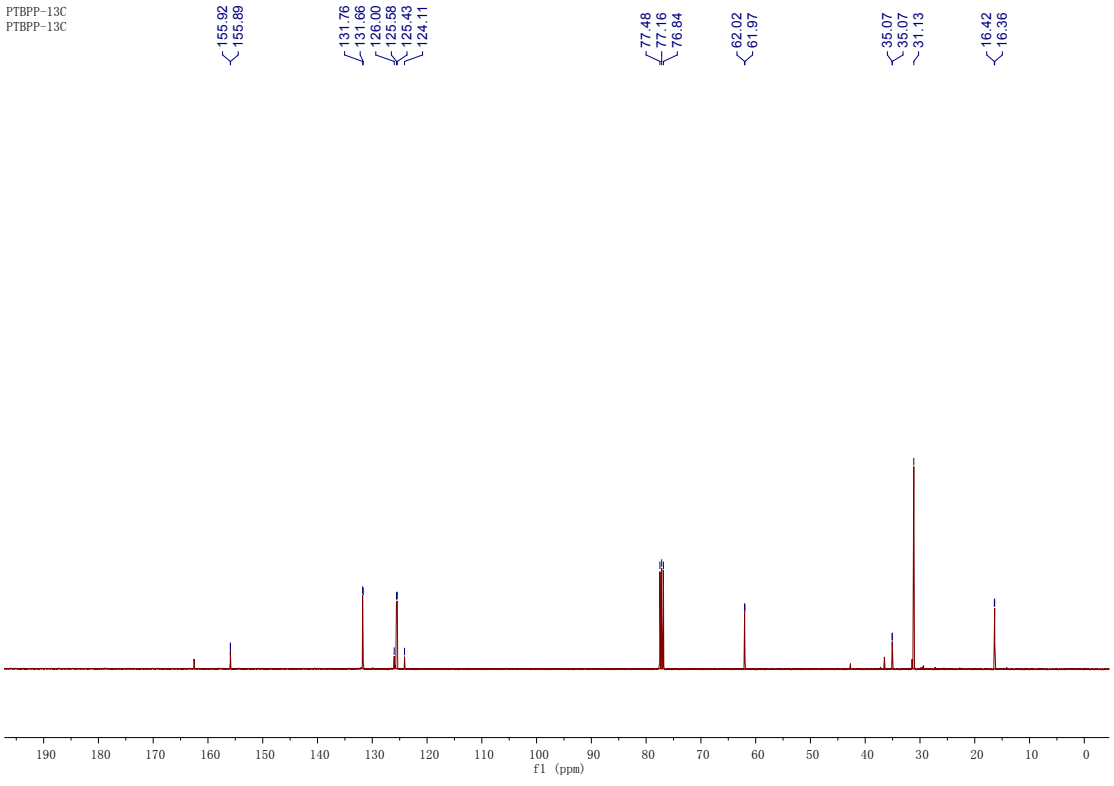
3k


DEOTP-31P
DEOTP-31P
19.44
130 110 90 80 70 60 50 40 30 20 10 0 −10 −30 −50 −70 −90 −110 −130 −150 −170 −190 −210 −230
f1 (ppm)

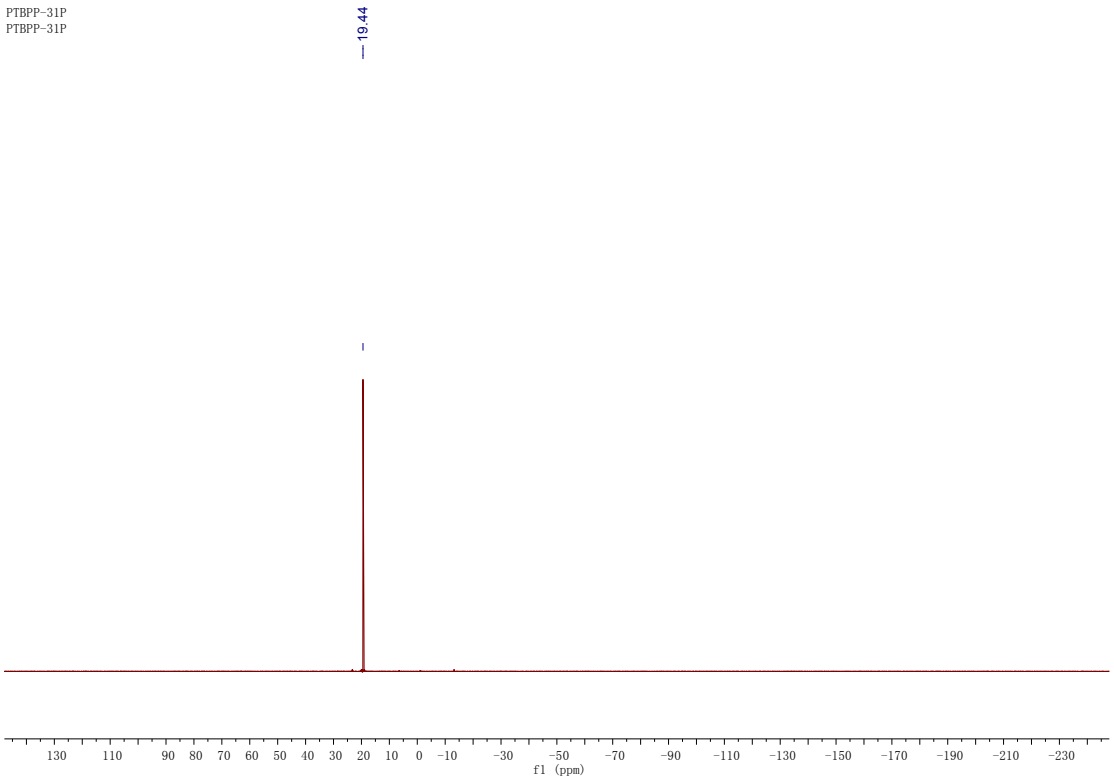

2013-12-10-2-Me-Ph-P(O)(OEt)2 #513 RT: 8.81 AV: 1 NL: 1.85E5
T: + c Full ms [50.00-600.00]
Relative Abundance
m/z
154
172
213
91
119
185
228
155
118
200
156
153
89
65
138
199
227
173
92
137
79
171
214
63
93
105
109
167
183
77
120
201
229
140
136
186
51
126
149
181
163
80
62
66
215
234
75
94
97
110
133
198
202
52
179
219

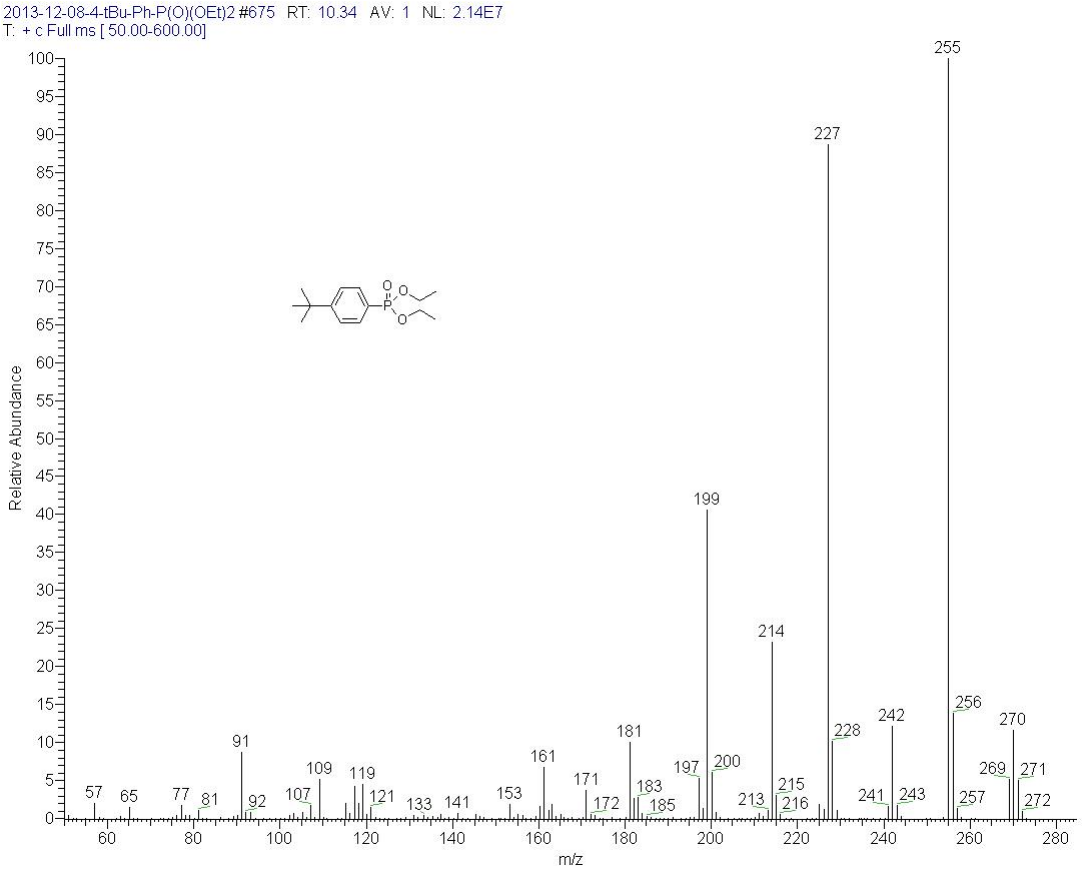
3l


PTBPP
PTBPP
7.77
7.75
7.74
7.71
7.50
7.49
7.48
7.47
7.30
4.17
4.15
4.15
4.13
4.11
4.10
4.08
4.06
4.04
1.34
1.33
1.32
1.31
-0.00
1.98
2.00
4.00
15.04
f1 (ppm)

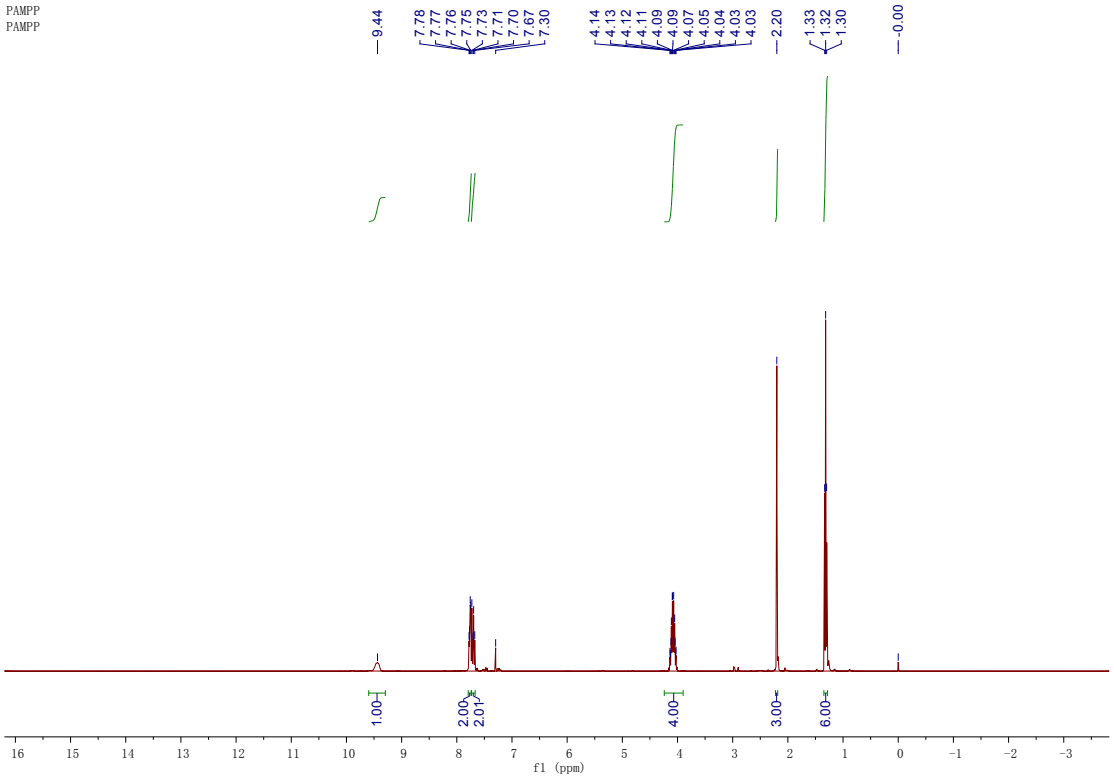

PTBPP-13C
PTBPP-13C
155.92
155.89
131.76
131.66
126.00
125.58
125.43
124.11
77.48
77.16
76.84
62.02
61.97
35.07
35.07
31.13
16.42
16.36
f1 (ppm)

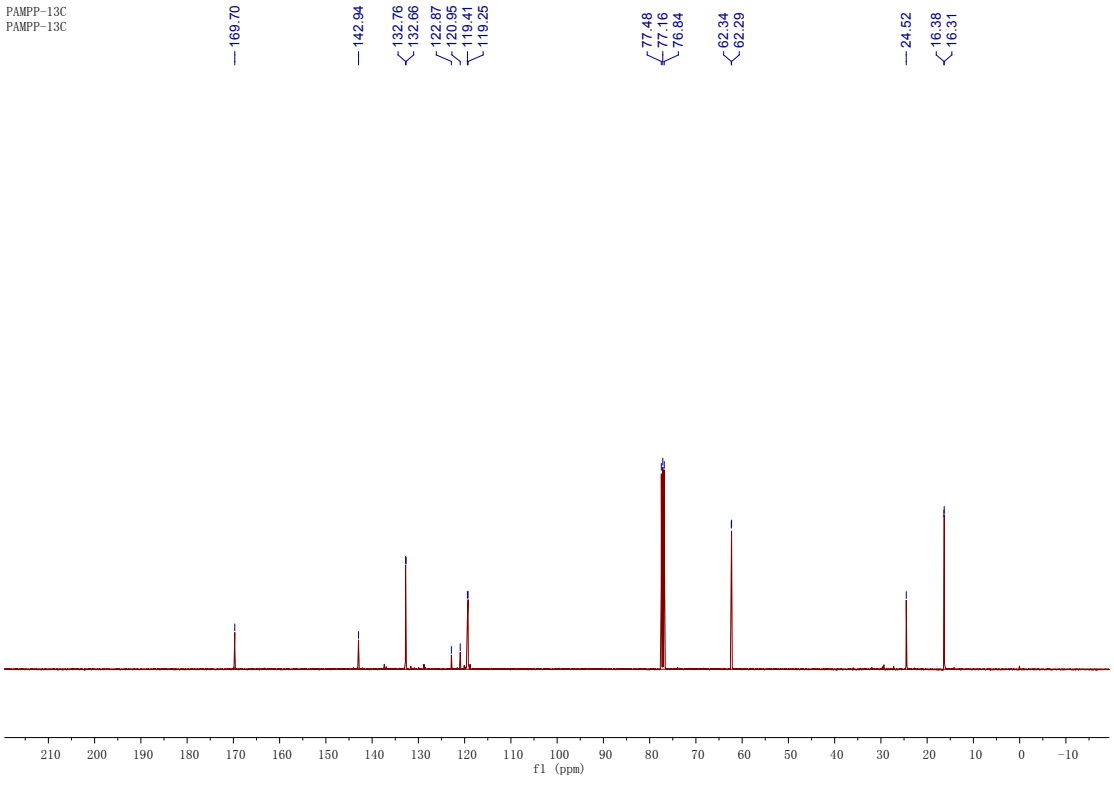
3l


PTBPP-31P
PTBPP-31P
19.44
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

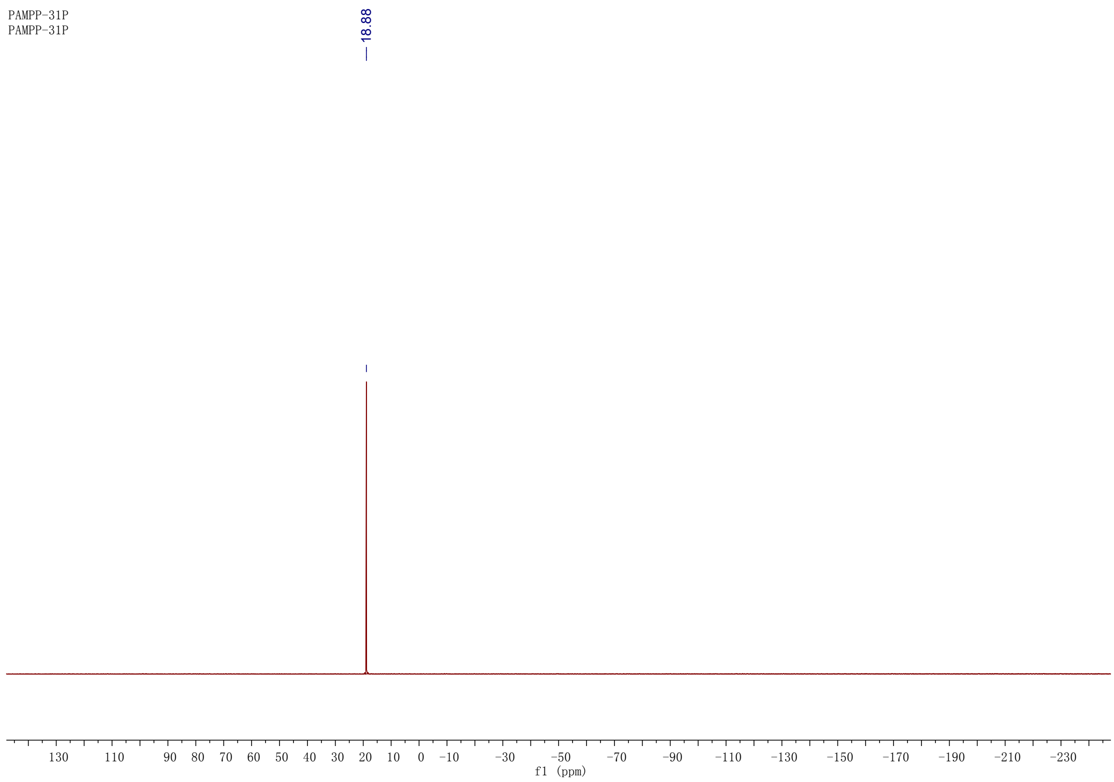

2013-12-08-4-tBu-Ph-P(O)(OEt)2 #675 RT: 10.34 AV: 1 NL: 2.14E7
T: + c Full ms [50.00-600.00]
Relative Abundance
m/z
255
227
199
214
256
242
228
270
181
161
91
109
119
171
197
200
183
215
269
271
57 65 77 81 92 107 121 133 141 153 172 185 213 216 241 243 257 272

3m

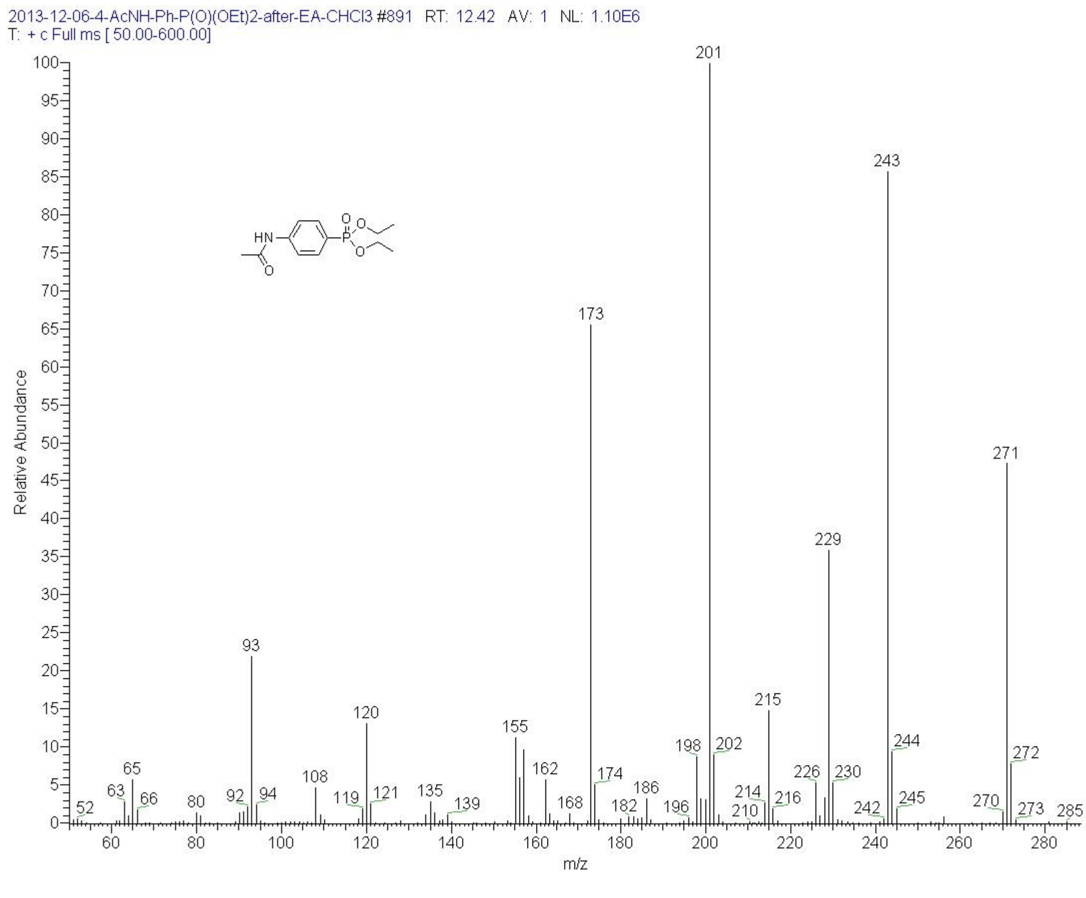

PAMPP
PAMPP
9.44
7.78
7.77
7.76
7.75
7.73
7.71
7.70
7.67
7.30
4.14
4.13
4.12
4.11
4.09
4.09
4.07
4.05
4.04
4.03
4.03
2.20
1.33
1.32
1.30
-0.00
1.00
2.00
2.01
4.00
3.00
6.00
f1 (ppm)


PAMPP-13C
PAMPP-13C
169.70
142.94
132.76
132.66
122.87
120.95
119.41
119.25
77.48
77.16
76.84
62.34
62.29
24.52
16.38
16.31
f1 (ppm)

3m

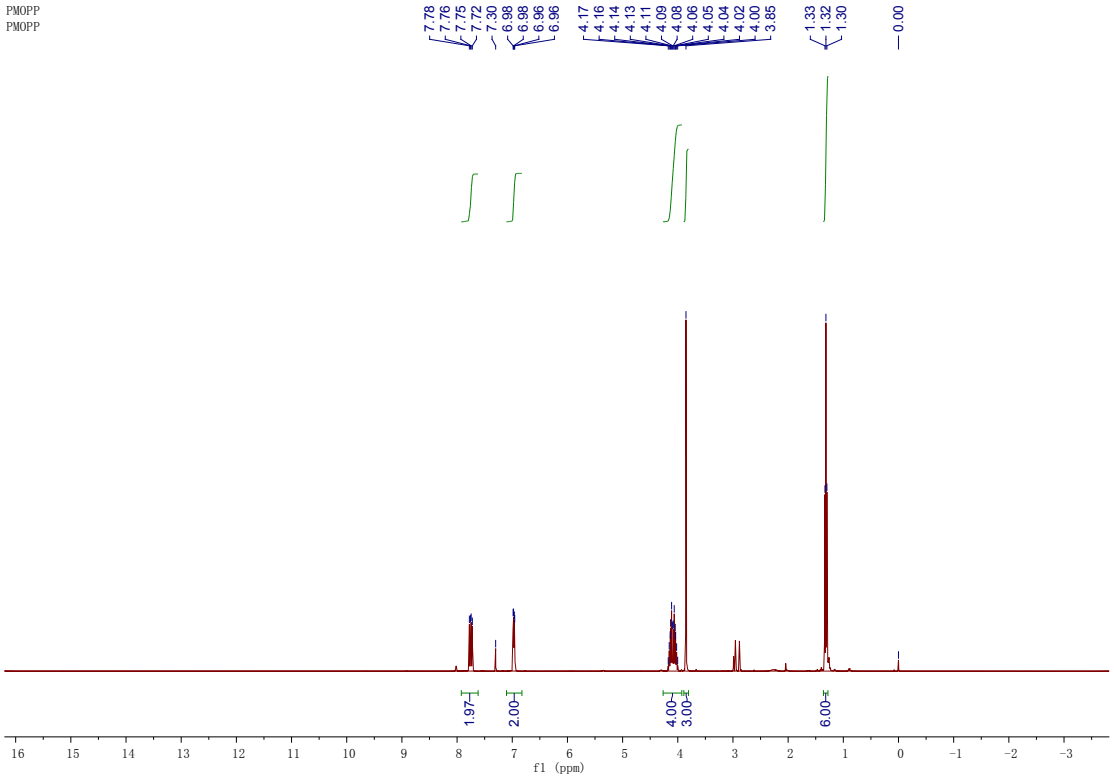


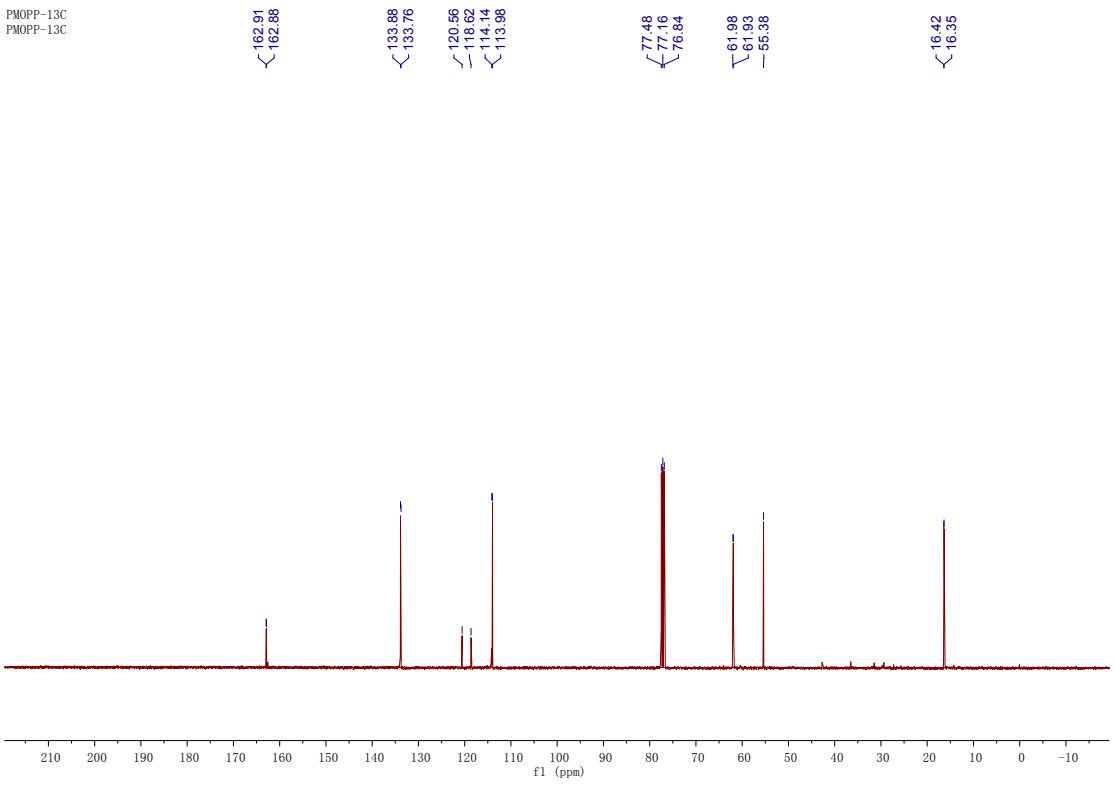
PAMPP-31P
PAMPP-31P
18.88
130 110 90 80 70 60 50 40 30 20 10 0 −10 −30 −50 −70 −90 −110 −130 −150 −170 −190 −210 −230
f1 (ppm)



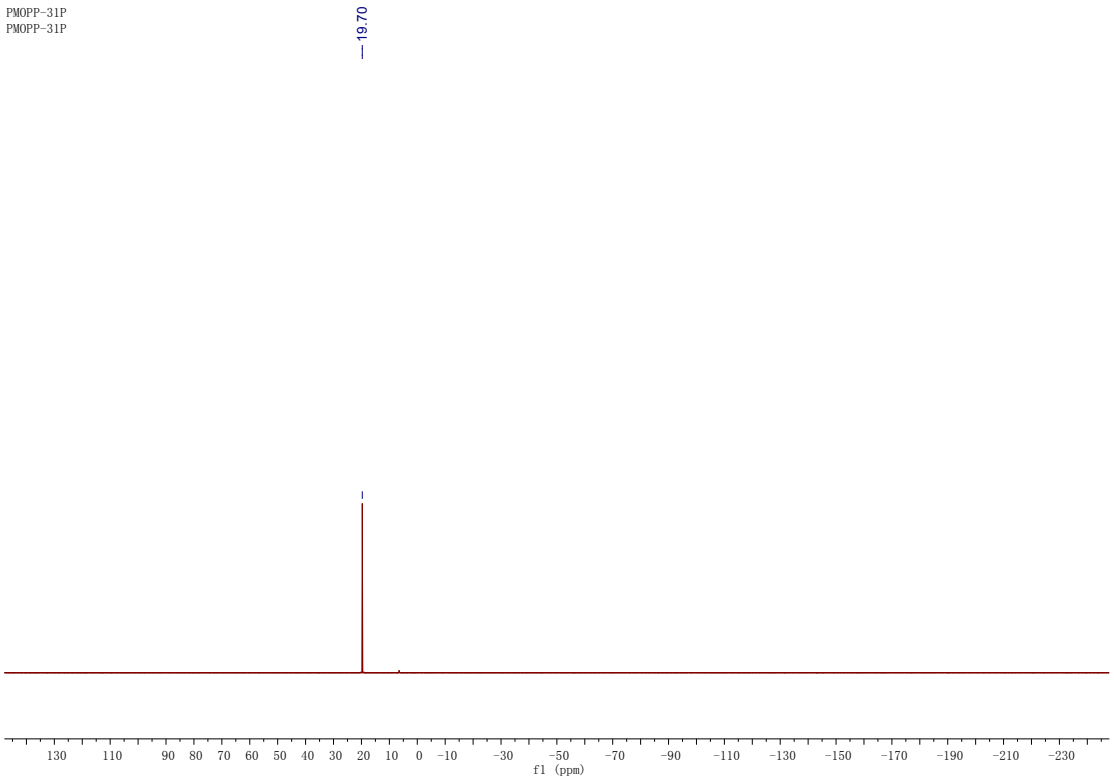
2013-12-06-4-AcNH-Ph-P(O)(OEt)2-after-EA-CHCl3 #891 RT: 12.42 AV: 1 NL: 1.10E6
T: + c Full ms [50.00-600.00]
Relative Abundance
m/z
201
243
173
271
229
93
215
120
155
198 202
244
272
162
174
186
226 230
65
63 66
52
80
92 94
108
119 121
135
139
168
182
196
214
210
216
242
245
270
273
285
HN
O
P

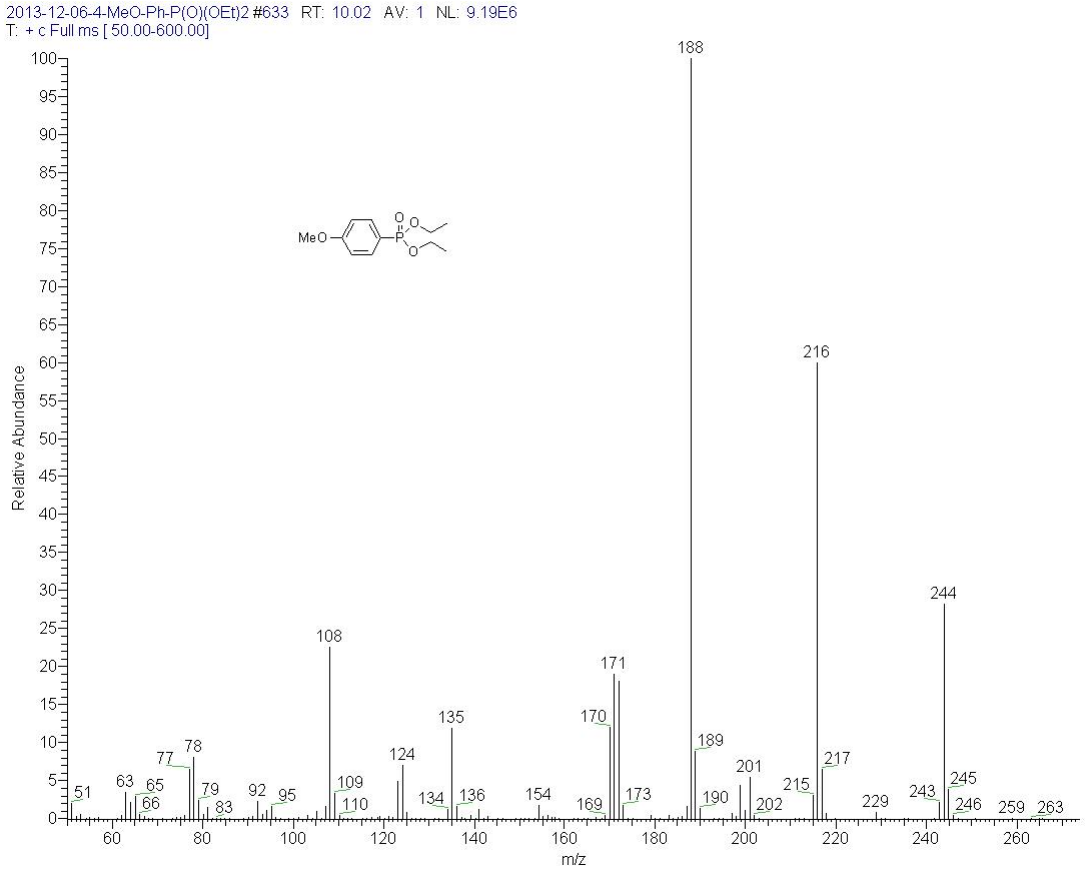
3n


PMOPP
PMOPP
7.78
7.76
7.75
7.72
7.30
6.98
6.98
6.96
6.96
4.17
4.16
4.14
4.13
4.11
4.09
4.08
4.06
4.05
4.04
4.02
4.00
3.85
1.33
1.32
1.30
0.00
1.97
2.00
4.00
3.00
6.00
f1 (ppm)

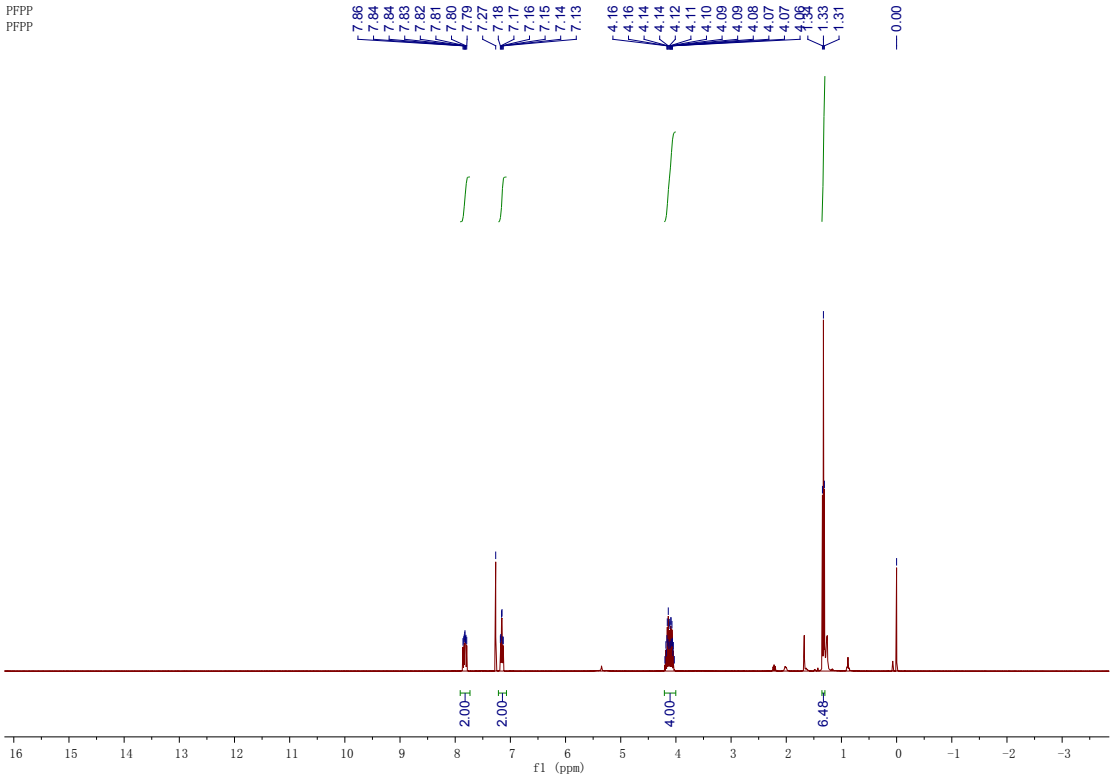

PMOPP-13C
PMOPP-13C
162.91
162.88
133.88
133.76
120.56
118.62
114.14
113.98
77.48
77.16
76.84
61.98
61.93
55.38
16.42
16.35
f1 (ppm)

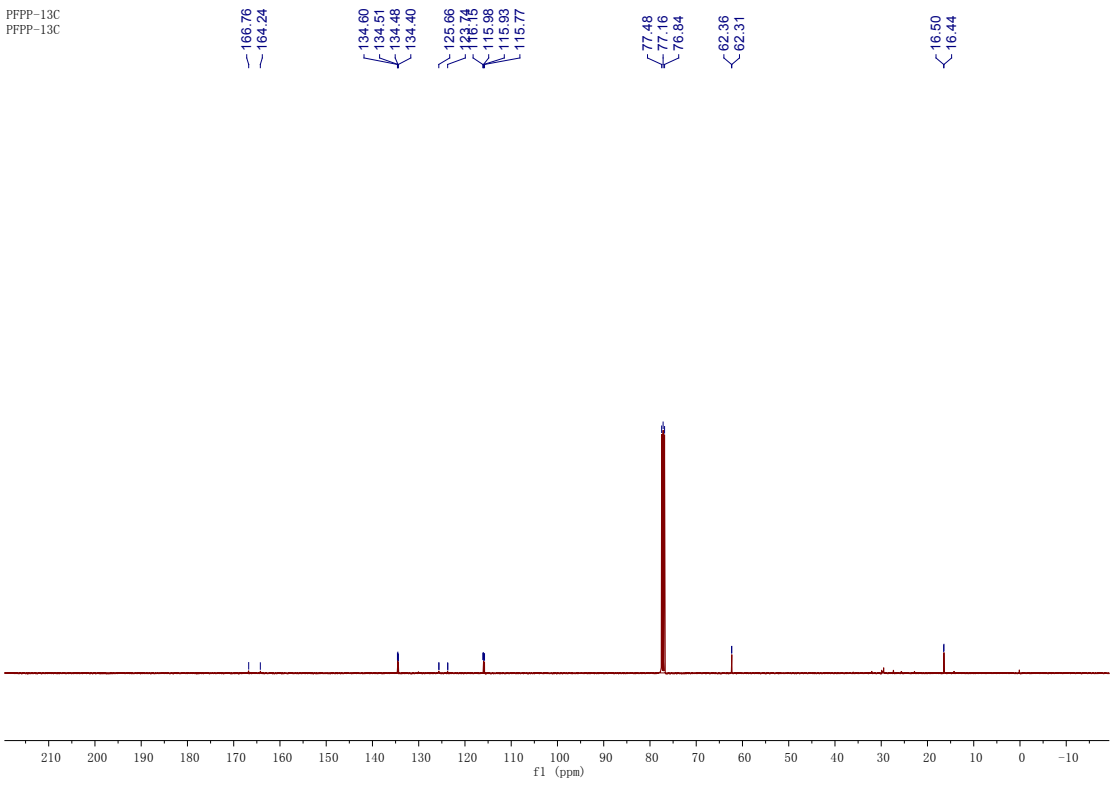
3n


PMOPP-31P
PMOPP-31P
19.70
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

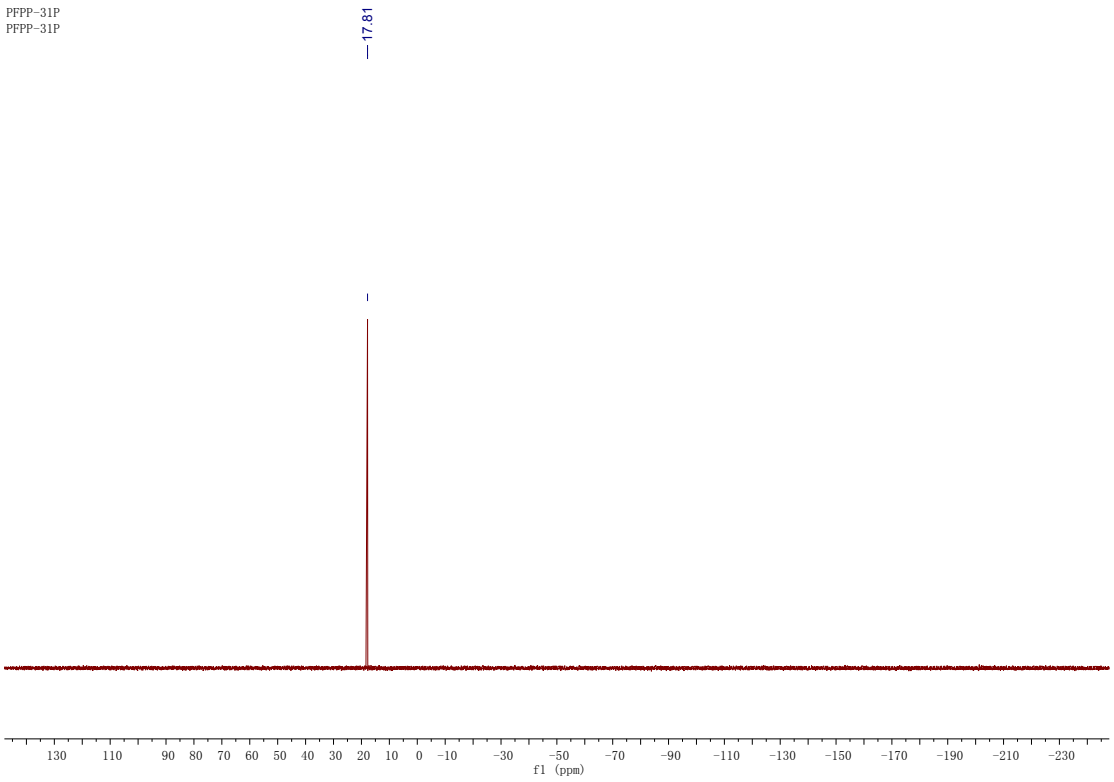

2013-12-06-4-MeO-Ph-P(O)(OEt)2 #633 RT: 10.02 AV: 1 NL: 9.19E6
T: + c Full ms [50.00-600.00]
MeO
Relative Abundance
m/z
188
216
244
108
171
135
170
124
78
77
189
201
217
109
63
65
66
79
83
92
95
110
134
136
154
169
173
190
202
215
229
243
245
246
259
263
51

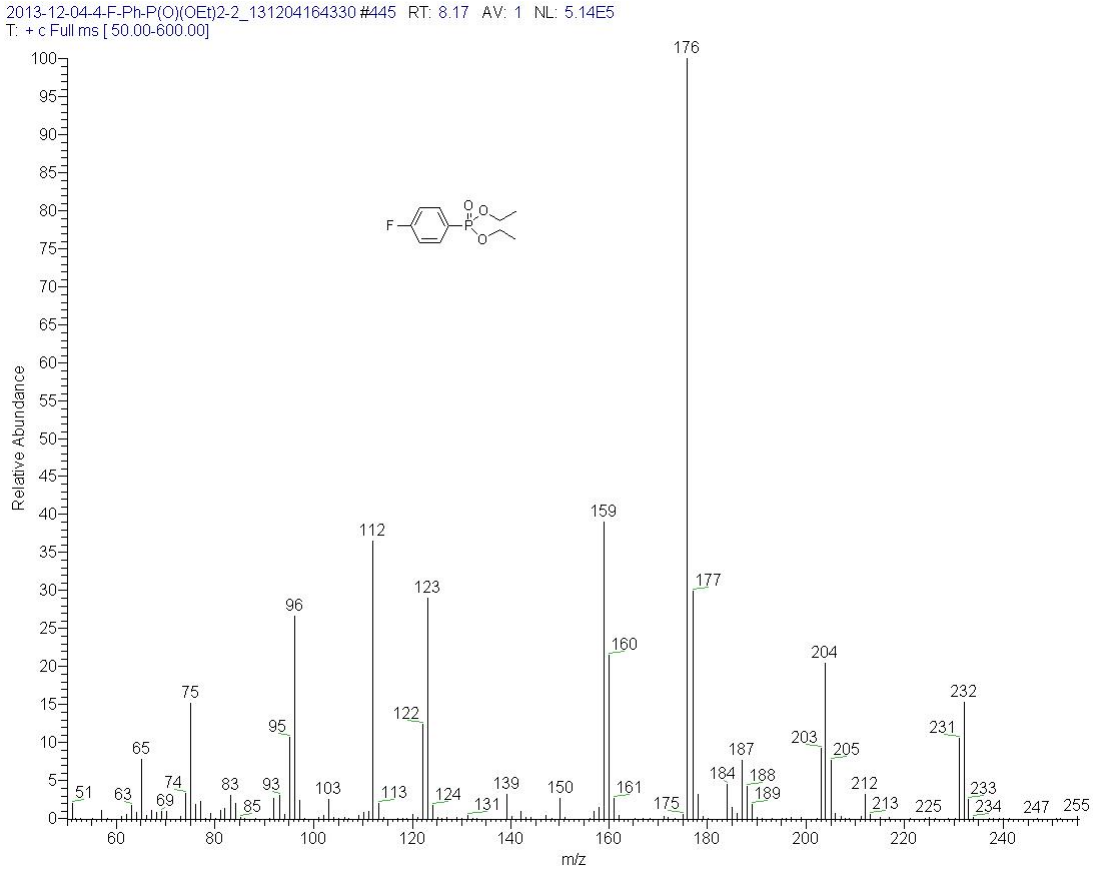
3o


PFPP
PFPP
7.86
7.84
7.84
7.83
7.82
7.81
7.80
7.79
7.27
7.18
7.17
7.16
7.15
7.14
7.13
4.16
4.16
4.14
4.14
4.12
4.11
4.10
4.09
4.09
4.08
4.07
4.07
1.33
1.31
0.00
2.00
2.00
4.00
6.48
f1 (ppm)

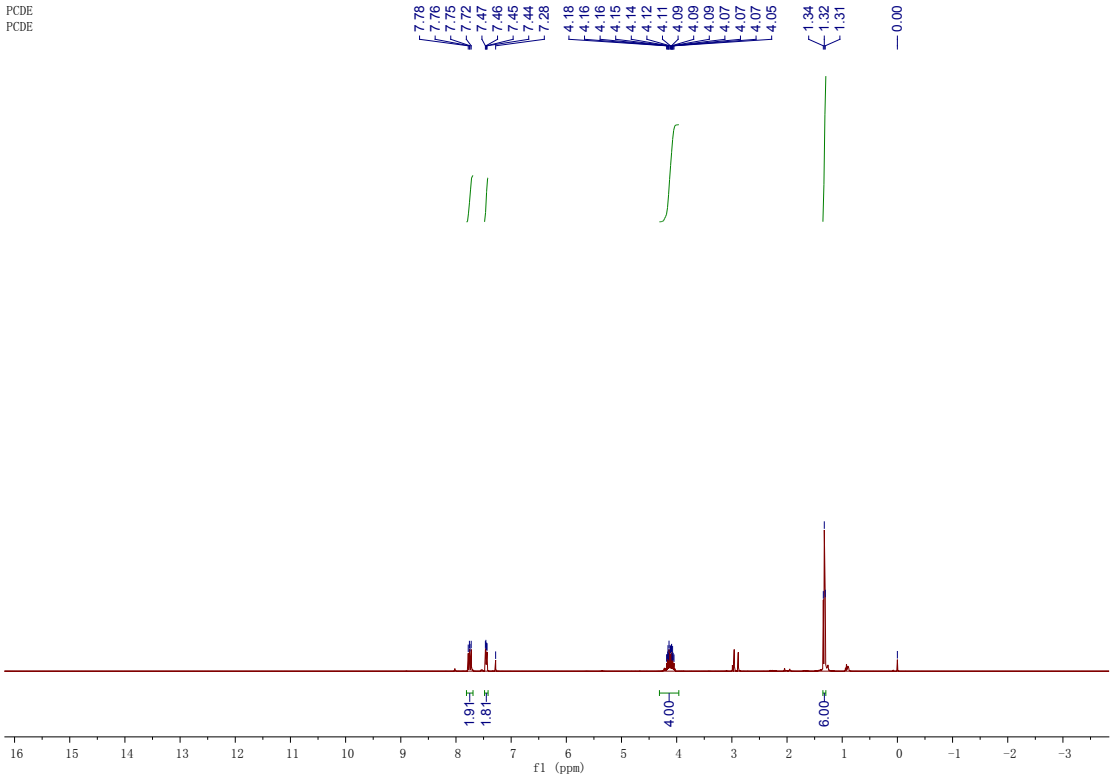

PFPP-13C
PFPP-13C
166.76
164.24
134.60
134.51
134.48
134.40
125.66
115.98
115.93
115.77
77.48
77.16
76.84
62.36
62.31
16.50
16.44
f1 (ppm)

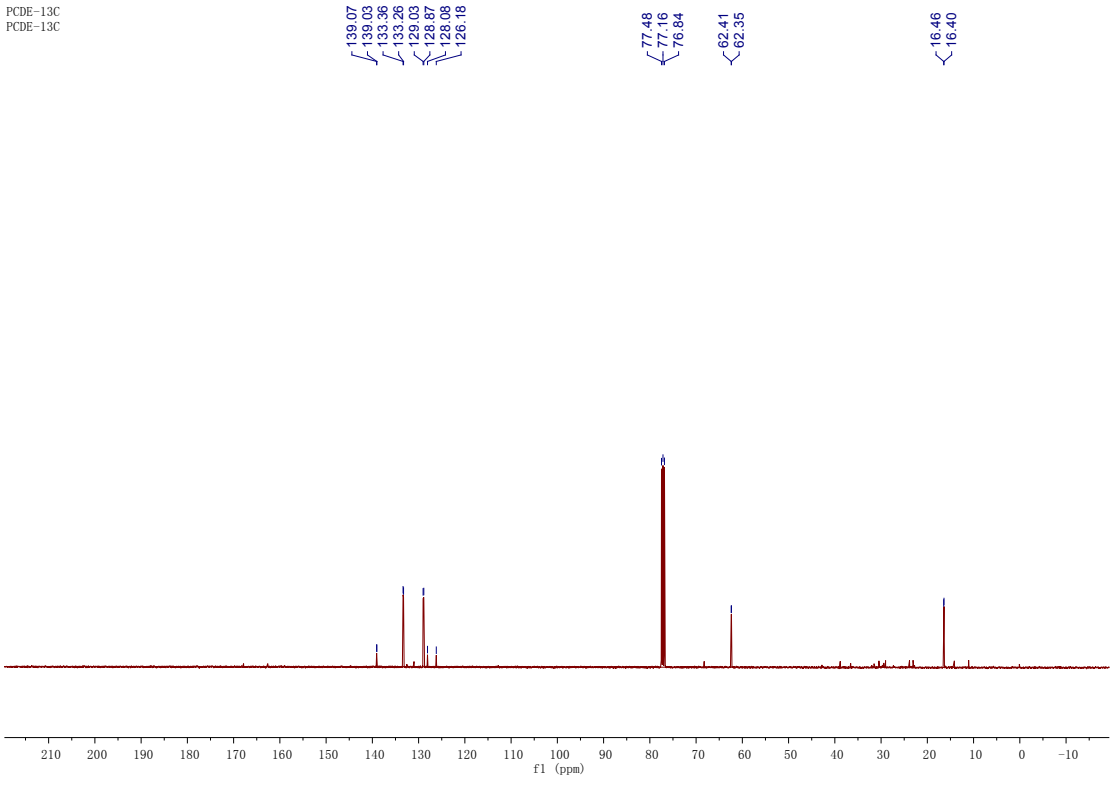
3o


PFPP-31P
PFPP-31P
17.81
f1 (ppm)

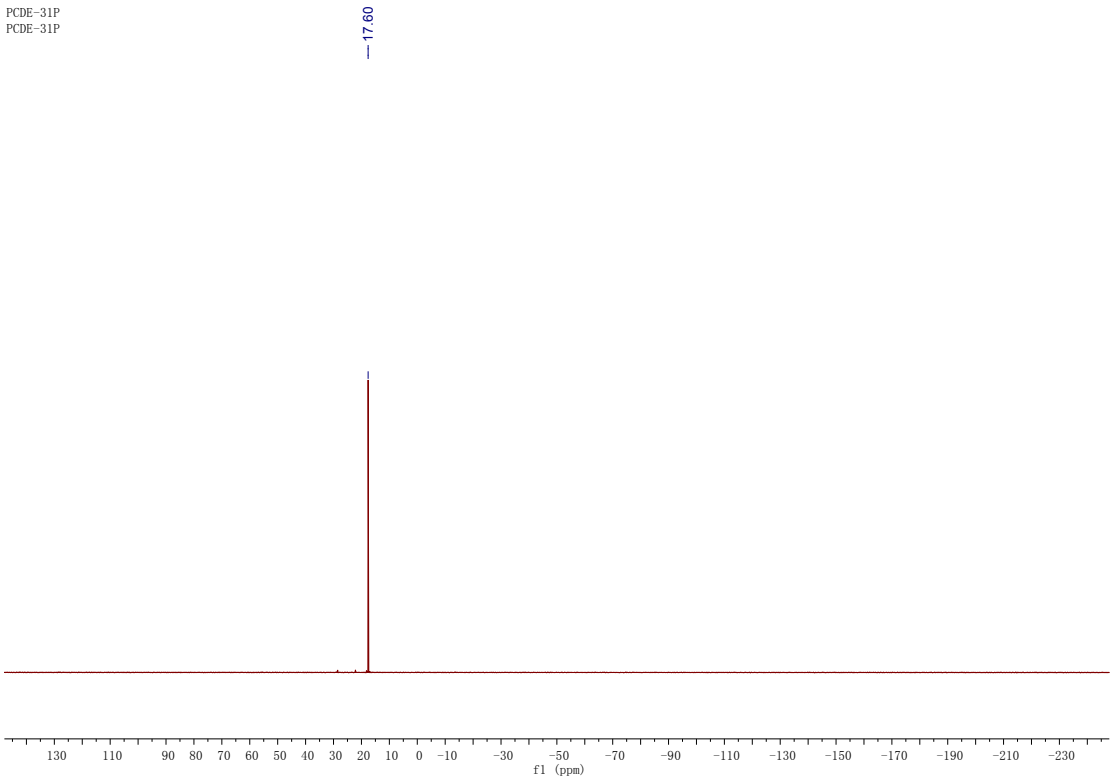

2013-12-04-4-F-Ph-P(O)(OEt)2-2_131204164330 #445 RT: 8.17 AV: 1 NL: 5.14E5
T: + c Full ms [50.00-600.00]
Relative Abundance
m/z

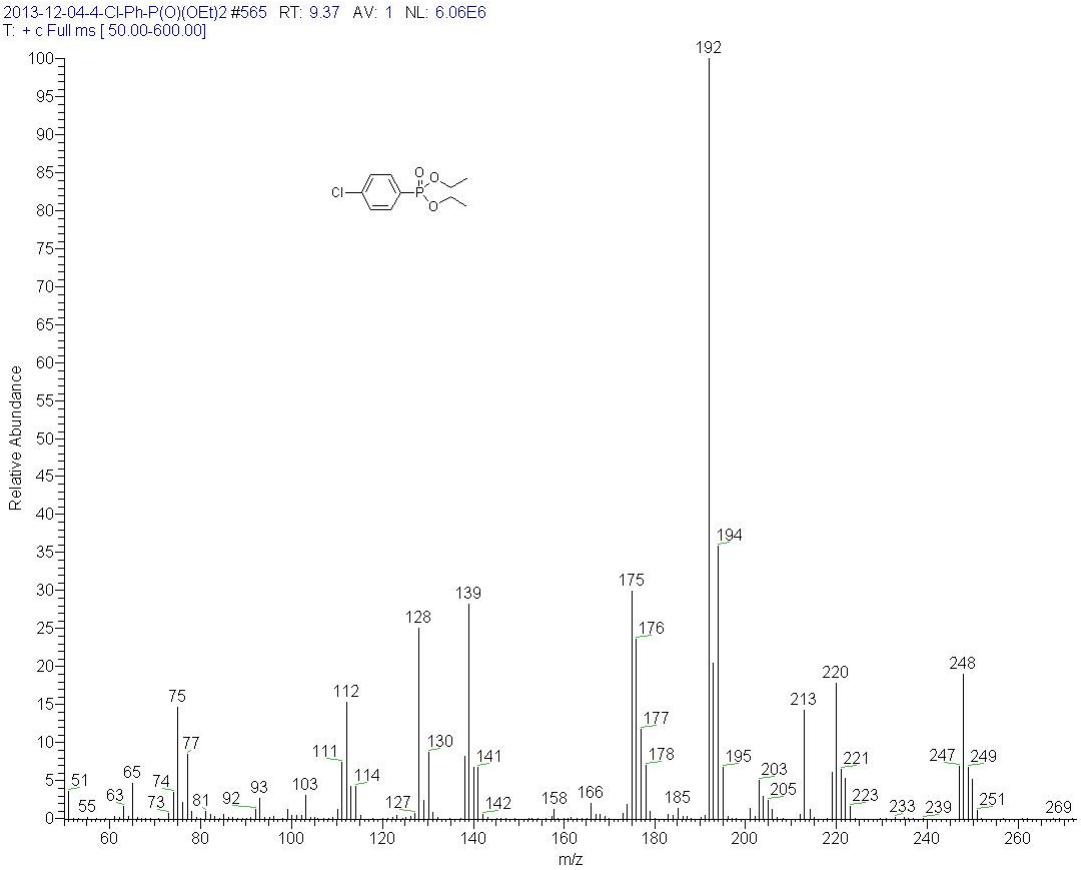
3p


PCDE
PCDE
7.78
7.76
7.75
7.72
7.47
7.46
7.45
7.44
7.28
4.18
4.16
4.16
4.15
4.14
4.12
4.11
4.09
4.09
4.09
4.07
4.07
4.07
4.05
1.34
1.32
1.31
0.00
1.91
1.81
4.00
6.00
f1 (ppm)

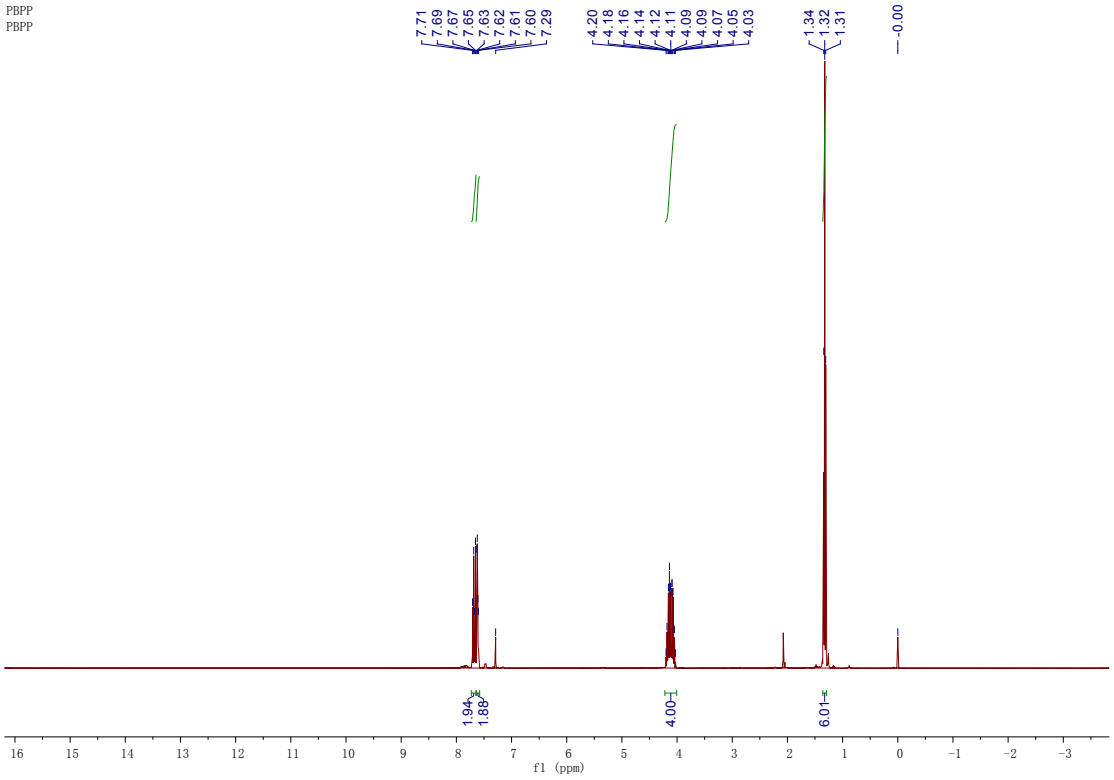

PCDE-13C
PCDE-13C
139.07
139.03
133.36
133.26
129.03
128.87
128.08
126.18
77.48
77.16
76.84
62.41
62.35
16.46
16.40
f1 (ppm)

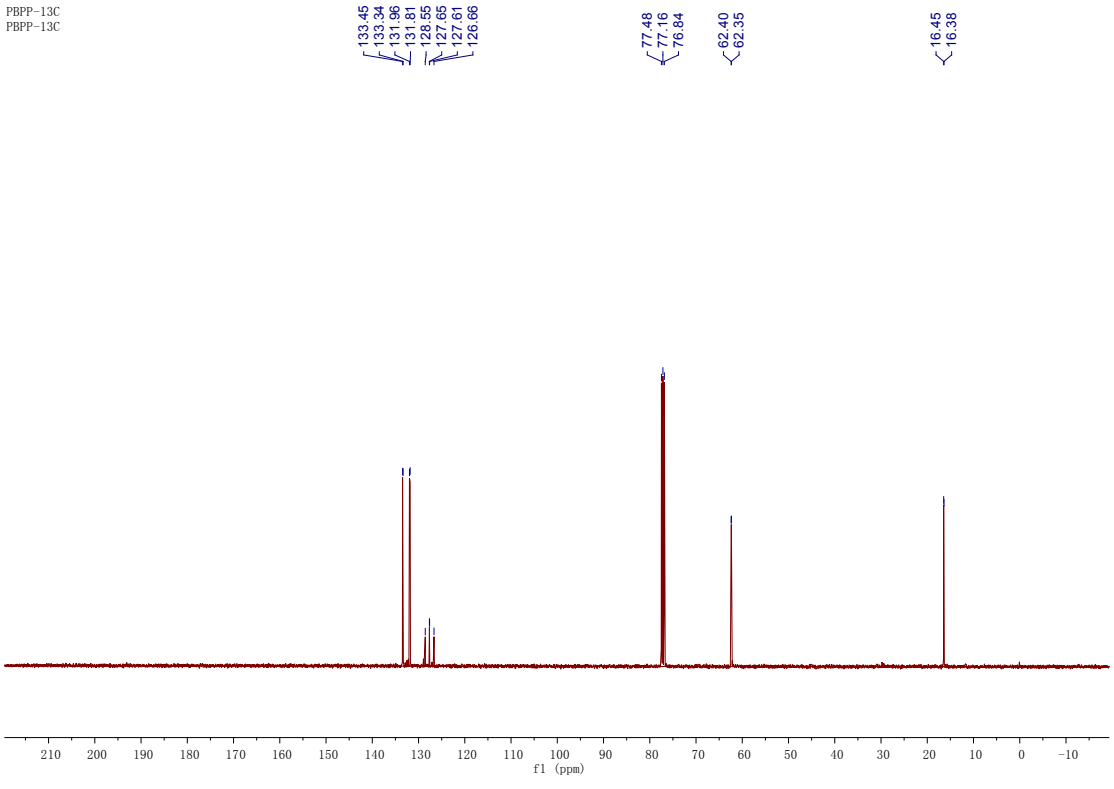
3p


PCDE-31P
PCDE-31P
17.60
f1 (ppm)

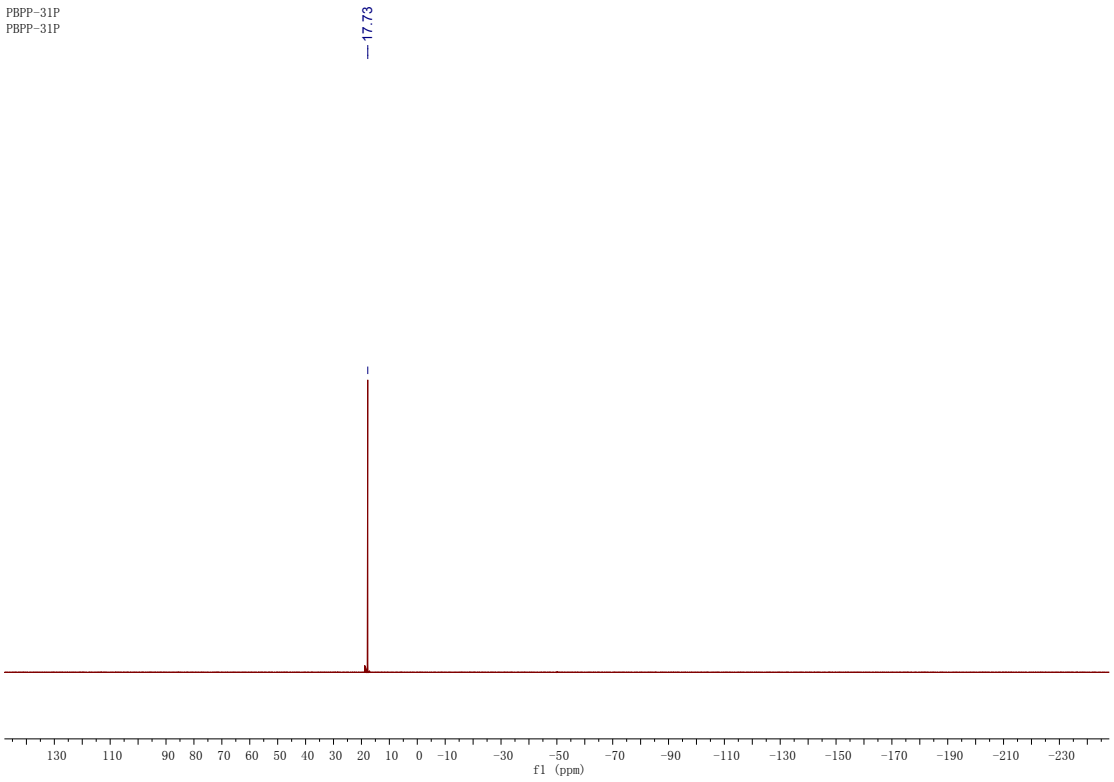

2013-12-04-4-Cl-Ph-P(O)(OEt)2 #565 RT: 9.37 AV: 1 NL: 6.06E6
T: + c Full ms [50.00-600.00]
Relative Abundance
m/z

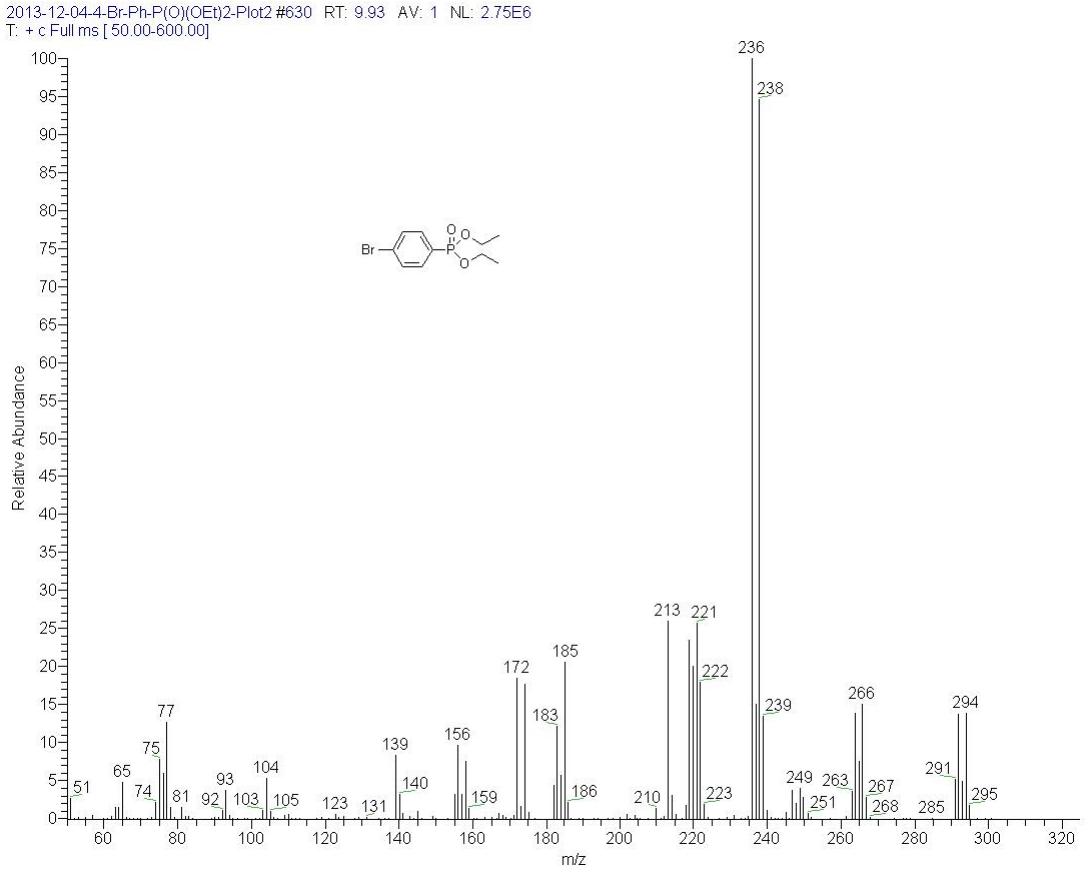
3q


PBPP
PBPP
7.71
7.69
7.67
7.65
7.63
7.62
7.61
7.60
7.29
4.20
4.18
4.16
4.14
4.12
4.11
4.09
4.09
4.07
4.05
4.03
1.34
1.32
1.31
0.00
1.94
1.88
4.00
6.01
f1 (ppm)

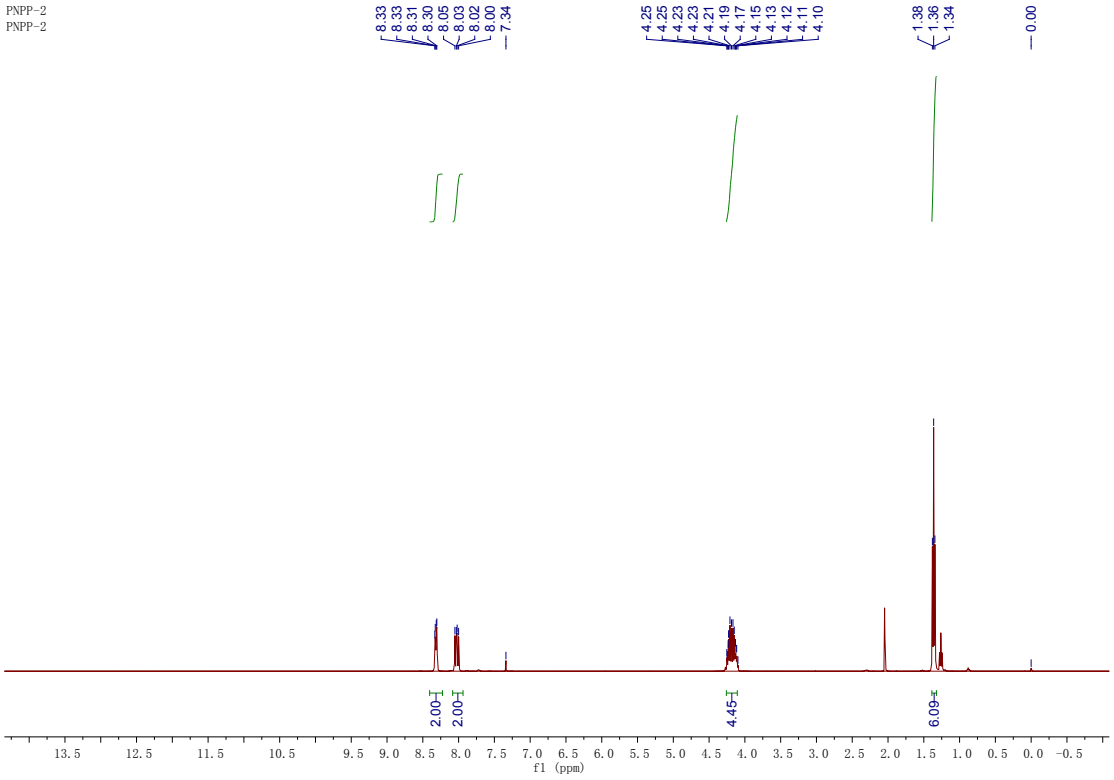

PBPP-13C
PBPP-13C
133.45
133.34
131.96
131.81
128.55
127.65
127.61
126.66
77.48
77.16
76.84
62.40
62.35
16.45
16.38
f1 (ppm)

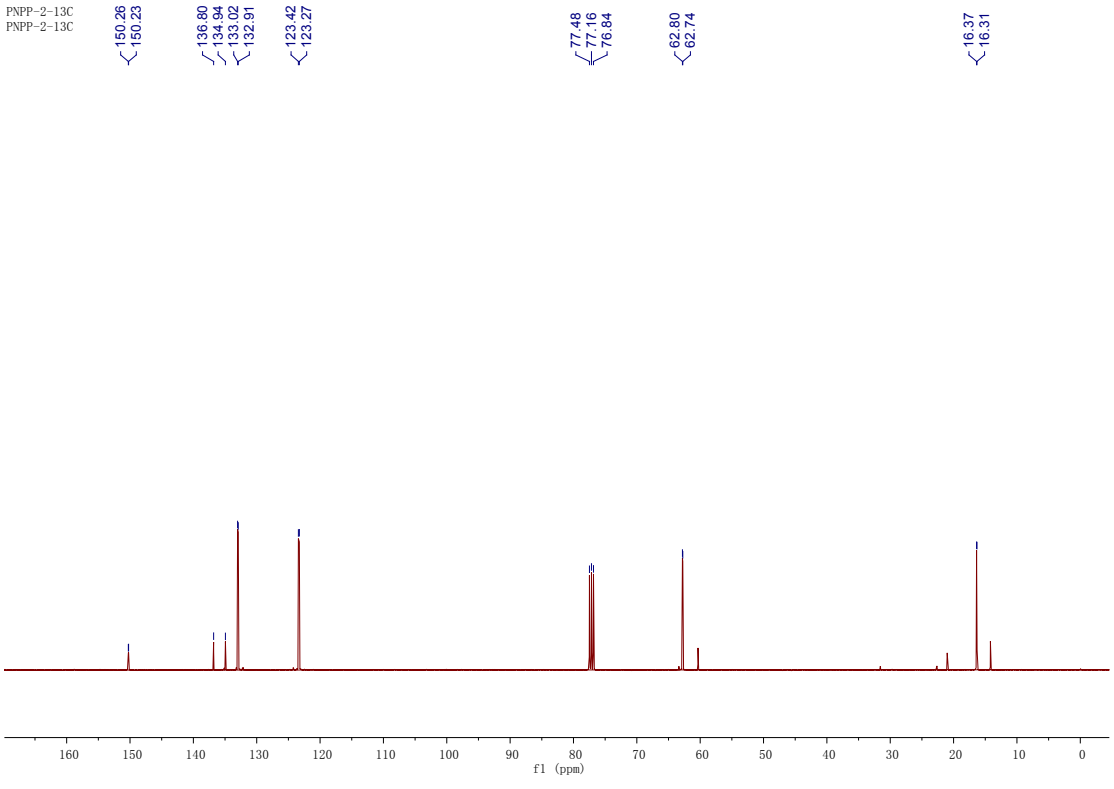
3q


PBPP-31P
PBPP-31P
17.73
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

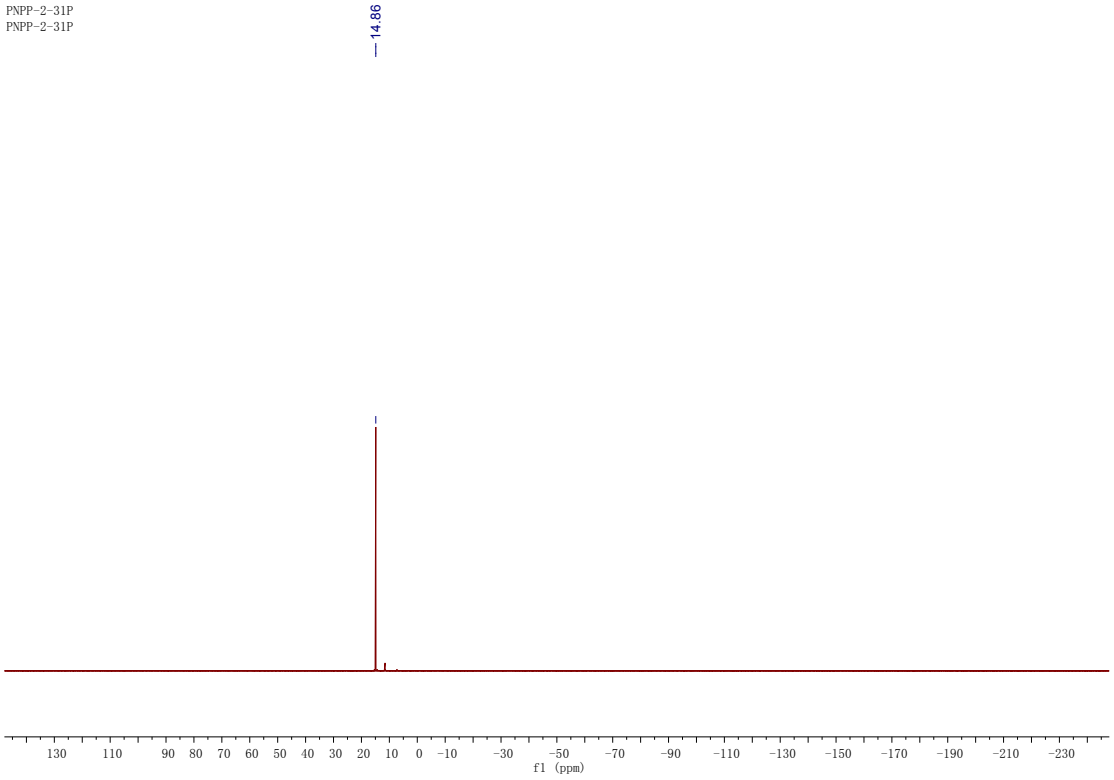

2013-12-04-4-Br-Ph-P(O)(OEt)2-Plot2 #630 RT: 9.93 AV: 1 NL: 2.75E6
T: + c Full ms [50.00-600.00]
Br
O
P
O
O
Relative Abundance
m/z
236
238
213
221
222
185
172
183
239
266
294
77
75
65
74
81
92
93
103
104
105
123
131
139
140
156
159
186
210
223
249
251
263
267
268
285
291
295

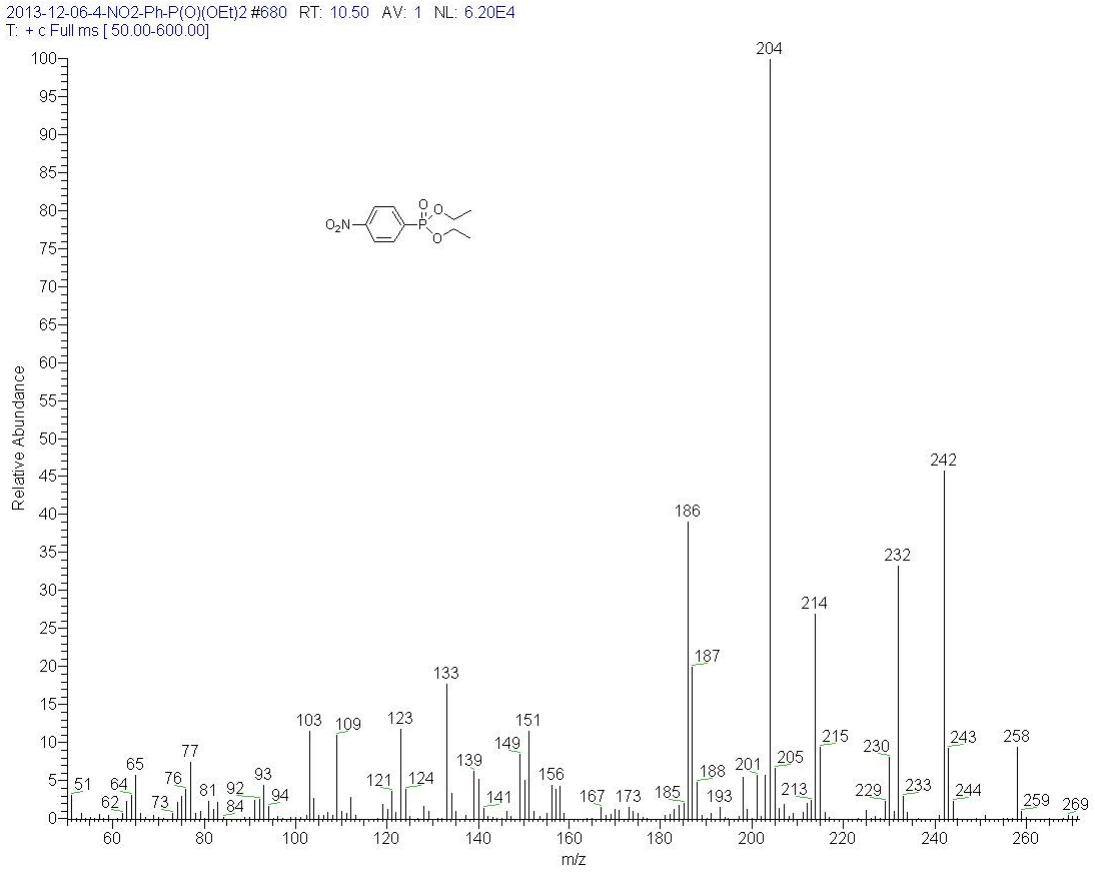
3r


PNPP-2
PNPP-2
8.33
8.33
8.31
8.30
8.05
8.03
8.02
8.00
7.34
4.25
4.25
4.23
4.23
4.21
4.19
4.17
4.15
4.13
4.12
4.11
4.10
1.38
1.36
1.34
0.00
2.00
2.00
4.45
6.09
f1 (ppm)

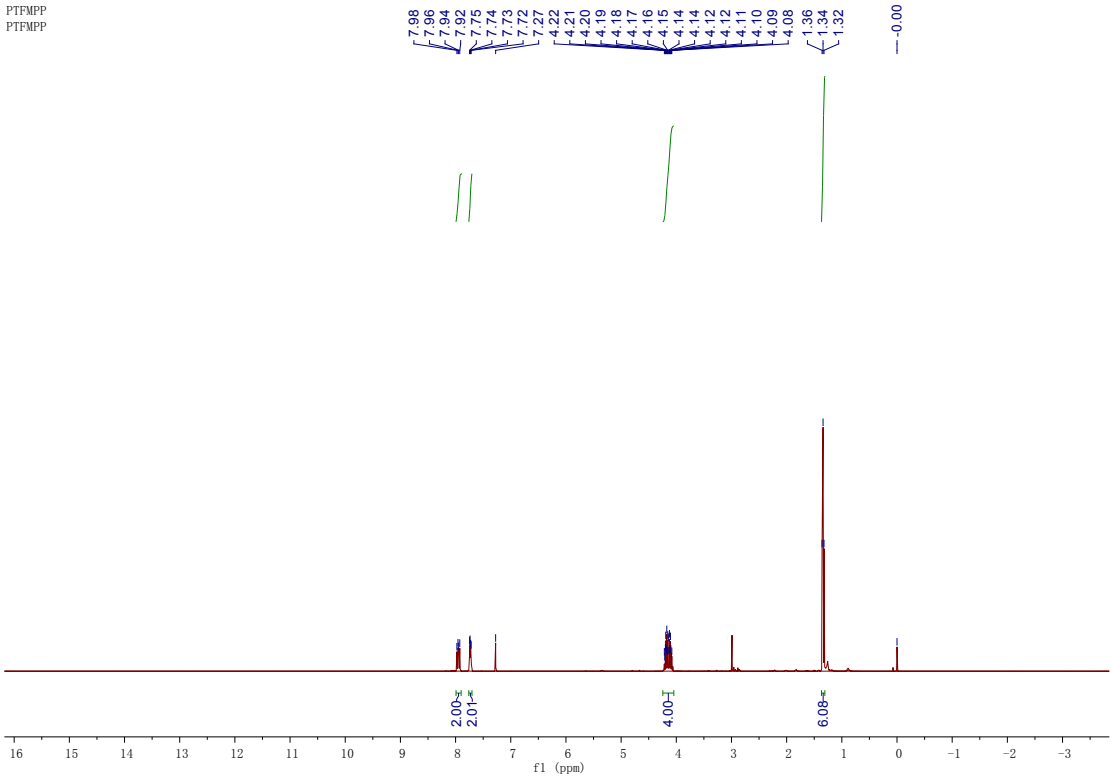

PNPP-2-13C
PNPP-2-13C
150.26
150.23
136.80
134.94
133.02
132.91
123.42
123.27
77.48
77.16
76.84
62.80
62.74
16.37
16.31
f1 (ppm)

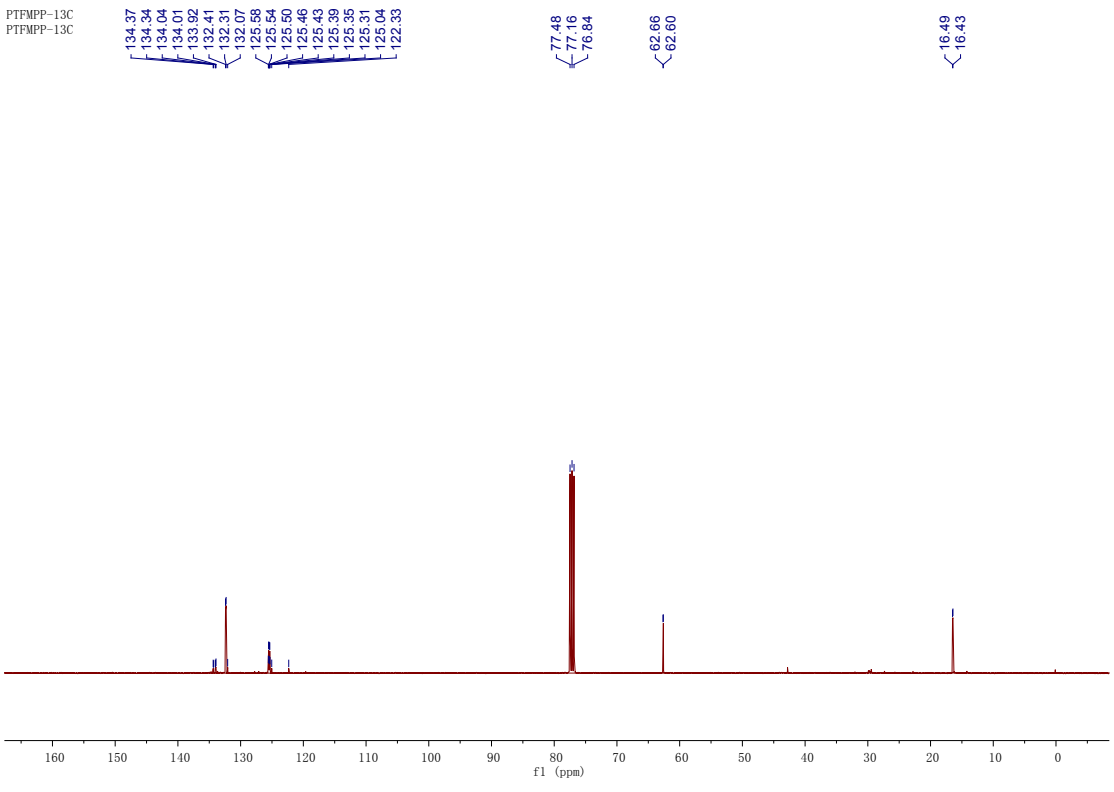
3r


PNPP-2-31P
PNPP-2-31P
14.86
f1 (ppm)

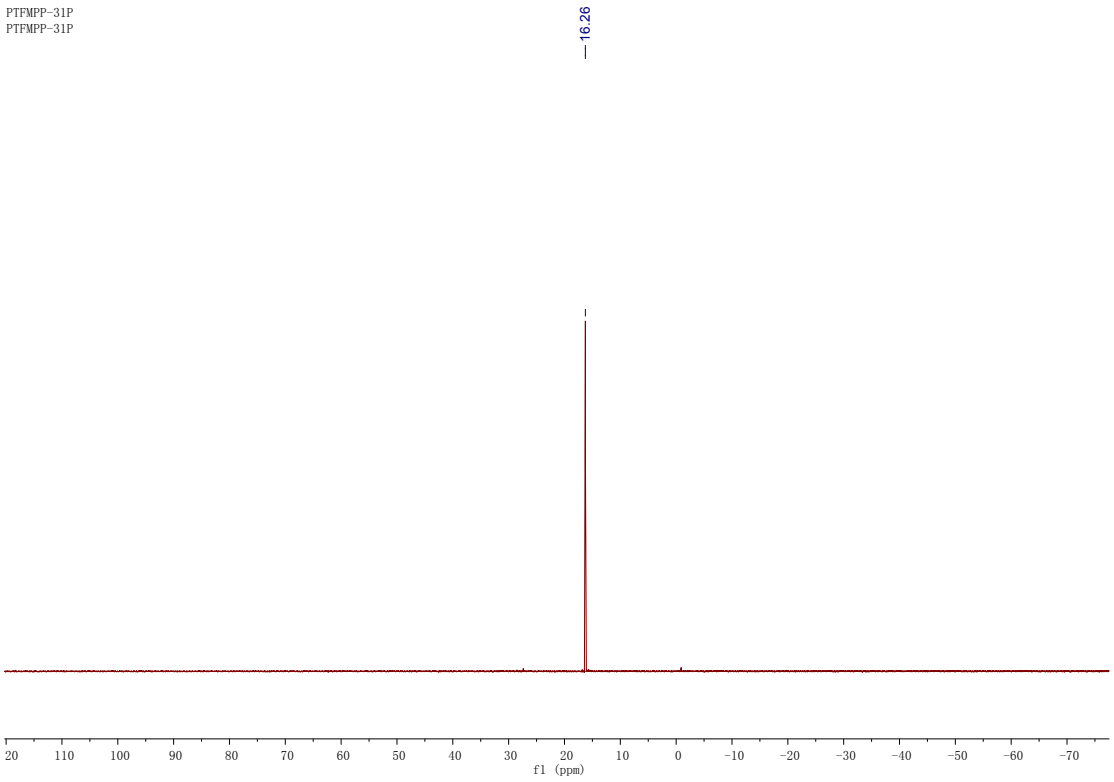

2013-12-06-4-NO2-Ph-P(O)(OEt)2 #680 RT: 10.50 AV: 1 NL: 6.20E4
T: + c Full ms [50.00-600.00]
Relative Abundance
m/z

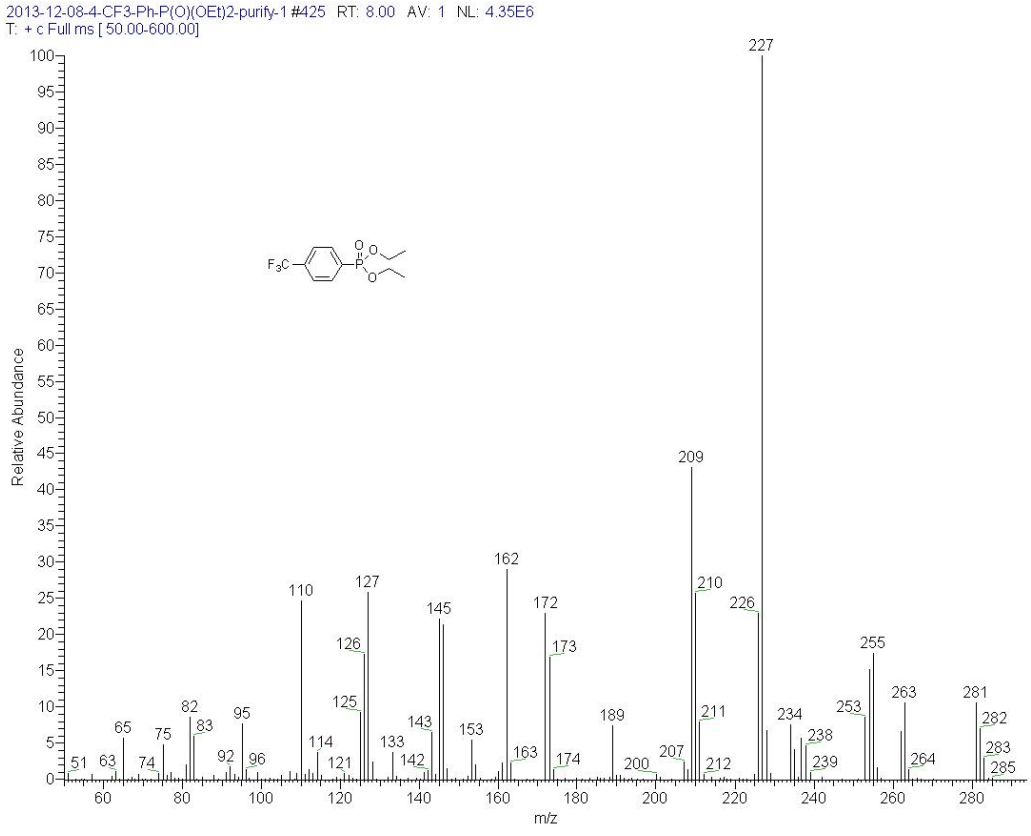
3s


PTFMPP
PTFMPP
7.98
7.96
7.94
7.92
7.75
7.74
7.73
7.72
7.27
4.22
4.21
4.20
4.19
4.18
4.17
4.16
4.15
4.14
4.14
4.12
4.12
4.11
4.10
4.09
4.08
1.36
1.34
1.32
0.00
2.00
2.01
4.00
6.08
f1 (ppm)

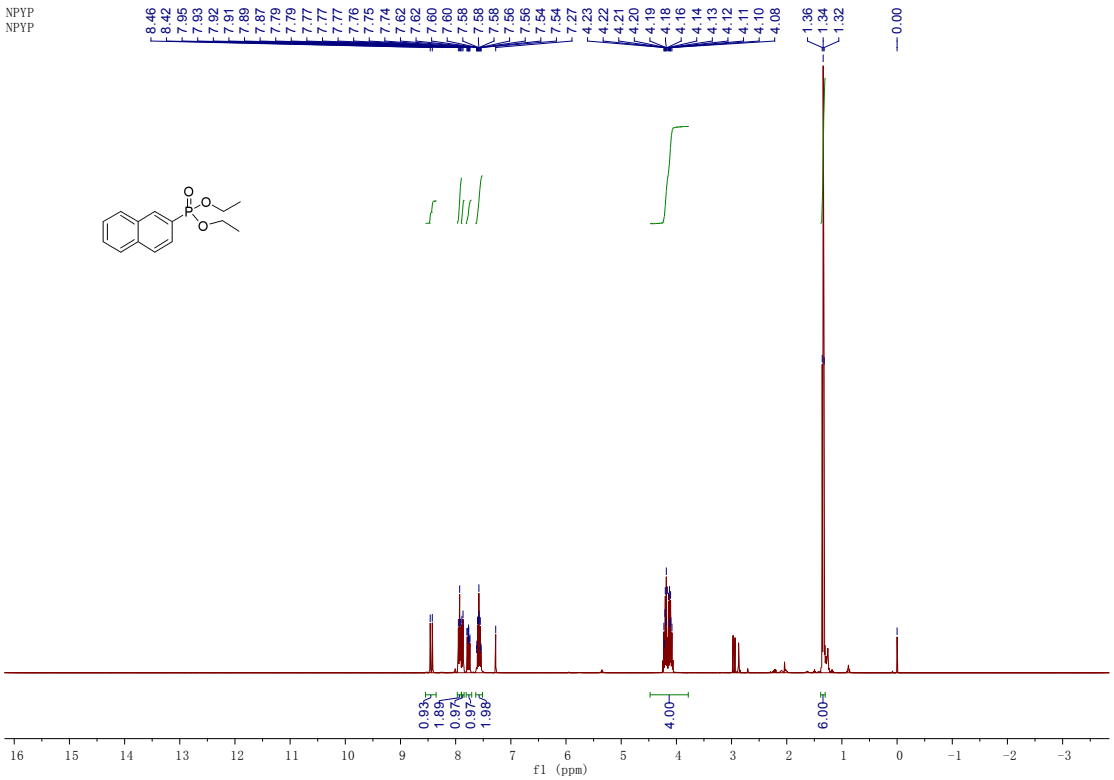

PTFMPP-13C
PTFMPP-13C
134.37
134.34
134.04
134.01
133.92
132.41
132.31
132.07
125.58
125.54
125.50
125.46
125.43
125.39
125.35
125.31
125.04
122.33
77.48
77.16
76.84
62.66
62.60
16.49
16.43
f1 (ppm)

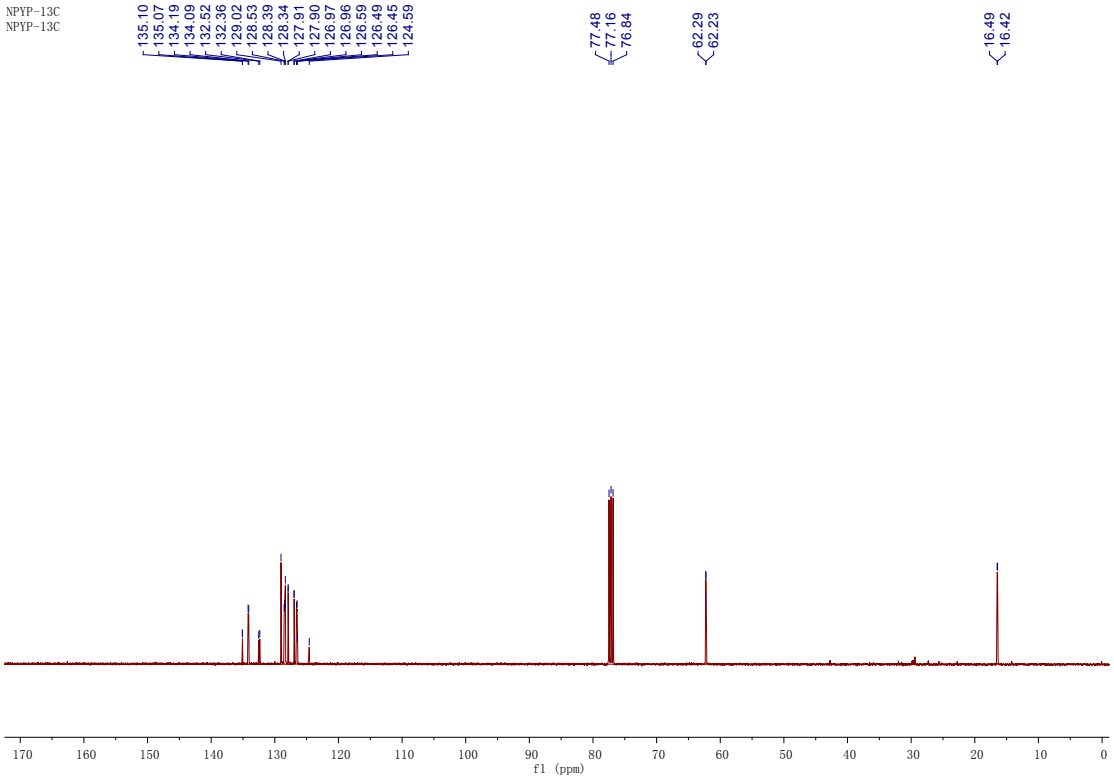
3s


PTFMPP-31P
PTFMPP-31P
16.26
f1 (ppm)

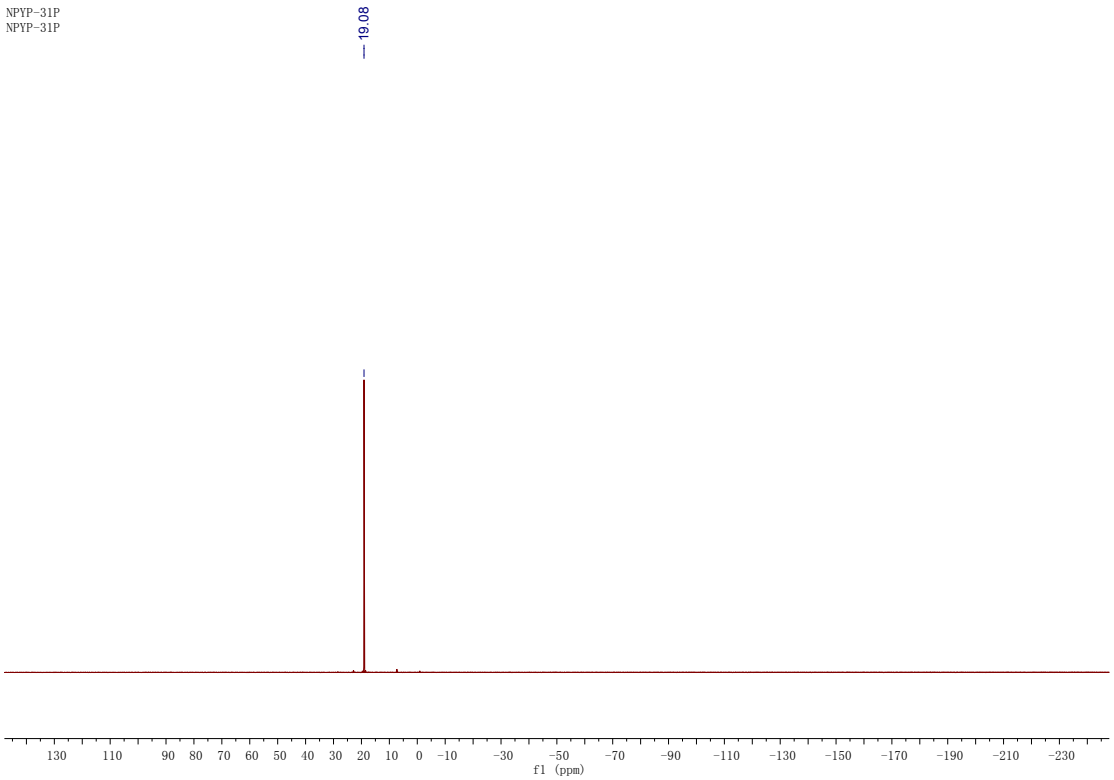

2013-12-08-4-CF3-Ph-P(O)(OEt)2-purify-1 #425 RT: 8.00 AV: 1 NL: 4.35E6
T: + c Full ms [50.00-600.00]
Relative Abundance
m/z

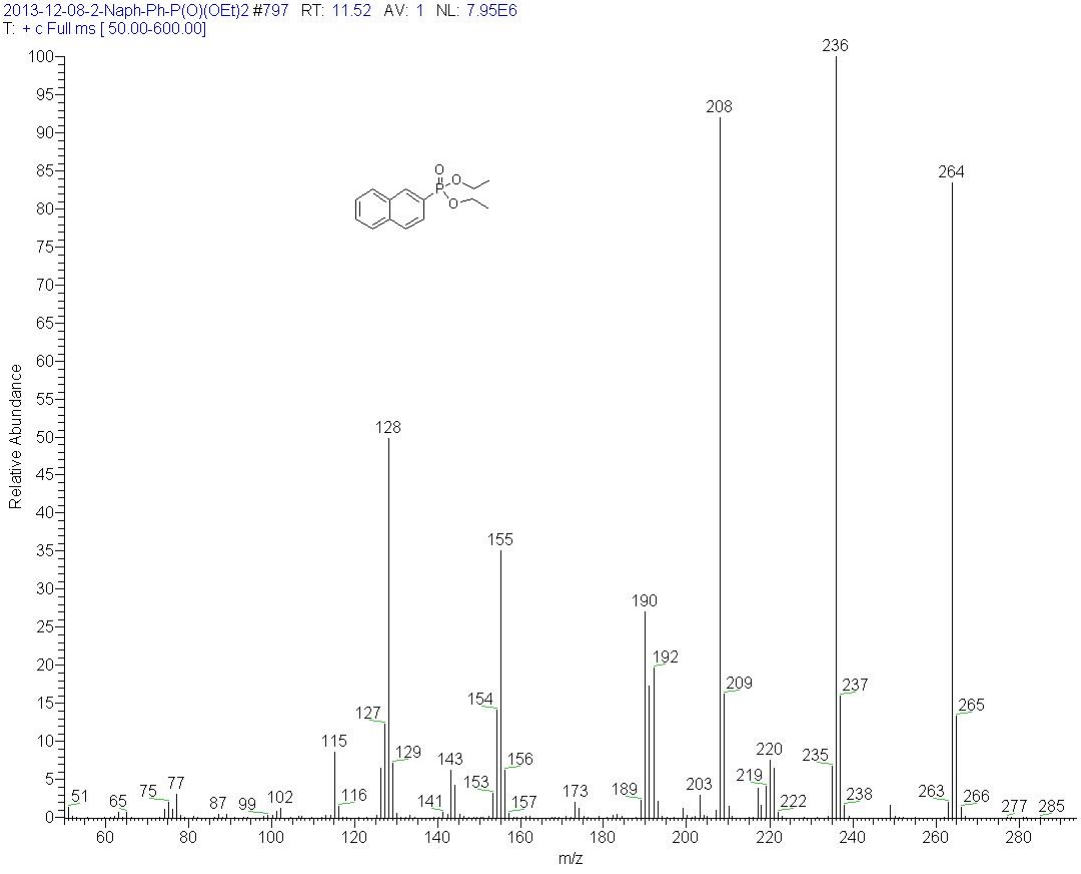
3t


NPYP
NPYP
8.46
8.42
7.95
7.93
7.92
7.91
7.89
7.87
7.79
7.79
7.77
7.77
7.77
7.76
7.75
7.74
7.62
7.62
7.60
7.60
7.58
7.58
7.58
7.56
7.56
7.54
7.54
7.27
4.23
4.22
4.21
4.20
4.19
4.18
4.16
4.14
4.13
4.12
4.11
4.10
4.08
1.36
1.34
1.32
0.00
0.93
1.89
0.97
0.97
1.98
4.00
6.00
f1 (ppm)


NPYP-13C
NPYP-13C
135.10
135.07
134.19
134.09
132.52
132.36
129.02
128.53
128.39
128.34
127.91
127.90
126.97
126.96
126.59
126.49
126.45
124.59
77.48
77.16
76.84
62.29
62.23
16.49
16.42
f1 (ppm)

3t


NPYP-31P
NPYP-31P
19.08
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)


2013-12-08-2-Naph-Ph-P(O)(OEt)2 #797 RT: 11.52 AV: 1 NL: 7.95E6
T: + c Full ms [50.00-600.00]
Relative Abundance
m/z
236
208
264
128
155
190
192
209
237
265
127
154
115
129
143
156
220
235
51 65 75 77 87 99 102 116 141 153 157 173 189 203 219 222 238 263 266 277 285