Electronic Supplementary Material (ESI) for ChemComm.

o-Phthalaldehyde Catalyzed Hydrolysis of Organophosphinic Amides and Other P(=O)-NH Containing Compounds

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Supporting Information

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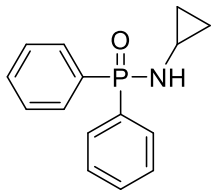
1. General Information

Unless otherwise mentioned, all commercially available materials were purchased from Alfa Aesar, Aldrich or Combi Blocks and used without further purification. Solvents were obtained from LC Technology solvent system or distilled before using. All reactions involving moisture sensitive reactants were executed under an argon atmosphere using oven dried and/or flame dried glassware. Flash column chromatography was performed using 40 – 63 μm Silicycle silica gel. Reactions were monitored by analytical thin layer chromatography (TLC), using glass or aluminium based plates, and cut to size. TLC visualization was achieved by UV, I_2 stain and $KMnO_4$ stain. Bruker AVANCE 300 MHz and 400 MHz spectrometers were used to obtain ^1H NMR and ^{13}C NMR spectra at ambient temperature. Spectral data is reported in ppm using solvents as the reference ($CDCl_3$ at 7.26 ppm, CD_3OD at 3.31 ppm and DMSO at 2.50 ppm for ^1H NMR/$CDCl_3$ at 77.16 ppm, CD_3OD at 49.00 ppm and DMSO at 39.52 ppm for ^{13}C NMR). ^1H NMR data was reporting as: multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant(s) in Hz, and integration. Infrared (IR) spectra were obtained as neat solids on an Agilent Cary 630 Fourier transform infrared spectrometer. Electron impact and electrospray ionisation high-resolution mass spectroscopy (EI-HRMS/ESI-HRMS) were recorded on a Kratos Concept 11-A mass spectrometer at the Ottawa-Carleton Mass Spectrometry Centre.

2. Synthesis and Spectral Data of Diphenylphosphinic Amides and Diphenylphosphoramidats.

Diphenylphosphinic amides and *O*,*O*-diphenyl phosphoramidates were synthesized according to a reported general procedure.[1] To a stirred solution of amines (10 mmol) in THF (25 mL) was added trimethylamine (21 mmol) at room temperature. Diphenylphosphinic choride (12 mmol) or *O*,*O*-diphenyl chlorophosphate (12 mmol) in 25 mL of THF was added to the solution at 0 °C. After being stirred for 15 min at 0 °C, the reaction solution was allowed back to ambient temperature and stirred for overnight. The resulting mixture was cooled in ice bath, and diluted with $CHCl_3$ and water. The product was extracted with $CHCl_3$ and combined organic layer was washed by brine, 1 N HCl, saturated $NaHCO_3$ and brine. The combined organic phases were dried over Na_2SO_4 and filtered and concentrated *in vacuo*. The crude product was purified by column chromatography using CH_2Cl_2-MeOH (95:5) as eluent to give the corresponding phosphinic amides or phosphoramidates. **1a**[2], **1b**[3], **1c**[4], **1d**[5], **1f**[6], **1h**[7], **1i**[8], **1j**[9], **1k**[10], **1n**[11], **1s**[12], **1t**[13], **1z**[12], **1aa**[14], **1ab**[15] were prepared according to the known procedures and spectral data was found to be in good agreement with literature.

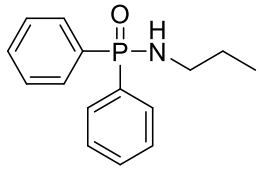
N-Cyclopropyl-*P*,*P*-diphenylphosphinic amide (1g)



Synthesized according to general procedure. The title compound was isolated as white solid (1.49 g, 58% yield). ^1H NMR (400 MHz, $CDCl_3$) δ 7.91 – 7.79 (m, 4H), 7.52 – 7.42 (m, 2H), 7.42 – 7.36 (m, 4H), 3.54 (s, 1H), 2.44 (tq, J = 7.1, 3.6 Hz, 1H), 0.53-0.57 (m, 2H), 0.40-0.46 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 132.7 (d, J_{P-C} = 128.2 Hz), 131.9 (d, J_{P-C} = 9.6 Hz), 131.6 (d, J_{P-C} = 2.7 Hz), 128.3 (d, J_{P-C} = 12.6 Hz), 23.0, 7.3 (d, J_{P-C} = 5.1 Hz). ^{31}P NMR

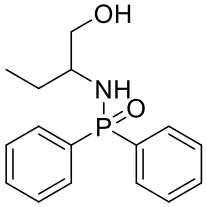
(121 MHz, $CDCl_3$) δ 20.7. IR (ATR Diamond): 3173, 3053, 1438, 1421, 1405, 1356, 1190, 1157, 1113, 1070, 1039, 1019, 1007, 995, 878, 819, 749, 721, 693 cm^{-1}. HRMS (EI): Exact mass calculated for $C_{15}H_{16}NOP$ $[M]^+$: 257.09695, found 257.09772.

_N_-Propyl-_P_,_P_-diphenylphosphinic amide (1e)



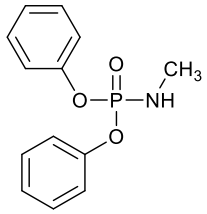
Synthesized according to general procedure. The title compound was isolated as white solid (1.40 g, 54% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.98 – 7.76 (m, 4H), 7.60 – 7.35 (m, 6H), 3.09 (s, 1H), 2.98 – 2.82 (m, 2H), 1.68 – 1.46 (m, 2H), 0.90 (t, J = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 132.5 (d, $J_{P\text{-}C}$ = 129.3 Hz), 132.1 (d, $J_{P\text{-}C}$ = 9.3 Hz), 131.8 (d, $J_{P\text{-}C}$ = 2.8 Hz), 128.5 (d, $J_{P\text{-}C}$ = 12.5 Hz), 42.6 (d, $J_{P\text{-}C}$ = 2.0 Hz), 25.3 (d, $J_{P\text{-}C}$ = 7.3 Hz), 11.3. ^{31}P NMR (121 MHz, $CDCl_3$) δ 20.6. IR (ATR Diamond): 3171, 2963, 2842, 1590, 1435, 1179, 1124, 1102, 1074, 1010, 995, 885, 842, 749, 773, 701, 692 cm^{-1}. HRMS (EI) for: Exact mass calculated for $C_{15}H_{18}NOP$ $[M]^+$: 259.11260, found 259.11133.

_N_-(1-Hydroxybutan-2-yl)-_P_,_P_-diphenylphosphinic amide (1l)



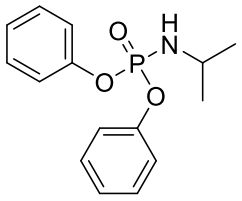
Synthesized according to general procedure. The title compound was isolated as white solid (1.62 g, 56% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 – 7.80 (m, 4H), 7.55 – 7.35 (m, 6H), 4.85 (s, 1H), 3.62 (dd, J = 11.7, 2.6 Hz, 1H), 3.43 (dd, J = 11.6, 7.3 Hz, 1H), 3.13 (s, 1H), 2.99 – 2.80 (m, 1H), 1.65 – 1.35 (m, 2H), 0.96 (t, J = 7.5 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 132.6 (d, $J_{P\text{-}C}$ = 9.5 Hz), 132.3 (d, $J_{P\text{-}C}$ = 148.2 Hz), 132.0 (d, $J_{P\text{-}C}$ = 2.5 Hz), 131.9 (d, $J_{P\text{-}C}$ = 2.6 Hz), 131.7 (d, $J_{P\text{-}C}$ = 9.4 Hz), 130.2, 128.6 (d, $J_{P\text{-}C}$ = 1.5 Hz), 128.5 (d, $J_{P\text{-}C}$ = 1.8 Hz), 67.0, 56.8 (d, $J_{P\text{-}C}$ = 2.2 Hz), 26.5 (d, $J_{P\text{-}C}$ = 9.6 Hz), 10.8. ^{31}P NMR (121 MHz, $CDCl_3$) δ 23.7. IR (ATR Diamond): 3249, 3187, 1142, 1436, 1169, 1112, 1093, 1063, 1021, 995, 946, 873, 851, 754, 748, 724, 694 cm^{-1}. HRMS (ESI) for: Exact mass calculated for $C_{16}H_{21}NO_2P$ $[M+H]^+$ 290.1310, found 290.1300.

Diphenyl methylphosphoramidate (1u)



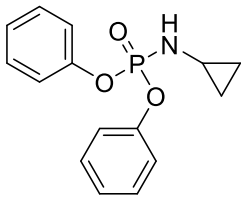
Synthesized according to general procedure. The title compound was isolated as yellow solid (1.58 g, 60% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.31 (m, 4H), 7.26 (dq, J = 7.9, 1.2 Hz, 5H), 7.21 – 7.14 (m, 2H), 3.03 (s, 1H), 2.76 (d, J = 12.5 Hz, 3H). ^{13} C NMR (75 MHz, $CDCl_3$) δ 150.7 (d, $J_{P\text{-}C}$ = 6.7 Hz), 129.7, 124.9, 120.2 (d, $J_{P\text{-}C}$ = 4.7 Hz), 27.9. ^{31} P NMR (121 MHz, CDCl3) δ 0.42. IR (ATR Diamond): 3244, 3069, 2928, 2824, 1587, 1488, 1456, 1424, 1256, 1227, 1104, 1163, 1110, 1081, 1024, 1008, 949, 926, 905, 857, 750, 687, 613, 591, 568 cm^{-1} HRMS (EI) for: Exact mass calculated for $C_{13}H_{14}NO_3P$ $[M]^+$: 263.07113, found 263.07396.

Diphenyl isopropylphosphoramidate (1w)



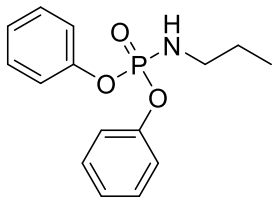
Synthesized according to general procedure. The title compound was isolated as white solid (2.03 g, 70% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.27 (m, 4H), 7.24 – 7.26 (m, 4H), 7.16 – 7.10 (m, 2H), 3.65 – 3.51 (m, 1H), 2.99 (t, J = 11.0 Hz, 1H), 1.14 (d, J = 1.0 Hz, 3H), 1.13 (d, J = 1.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.9 (d, $J_{P\text{-}C}$ = 6.9 Hz), 129.6, 124.7 (d, $J_{P\text{-}C}$ = 1.1 Hz), 120.2 (d, $J_{P\text{-}C}$ = 5.1 Hz), 44.5, 25.1 (d, $J_{P\text{-}C}$ = 5.8 Hz). ^{31}P NMR (121 MHz, $CDCl_3$) δ -4.6. IR (ATR Diamond): 3261, 2967, 1590, 1484, 1464, 1422, 1385, 1369, 1245, 1220, 1191, 1163, 1144, 1126, 1042, 1026, 924, 906, 767, 751, 691 cm^{-1}. HRMS (ESI) for: Exact mass calculated for $C_{15}H_{18}NO_3PNa$ $[M+Na]^+$ 314.0922, found 314.0933

Diphenyl cyclopropylphosphoramidate (1x)



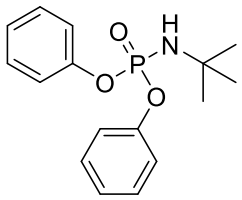
Synthesized according to general procedure. The title compound was isolated as white solid (2.60 g, 90% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31 (dd, J = 8.7, 6.9 Hz, 4H), 7.26 – 7.20 (m, 4H), 7.19 – 7.09 (m, 2H), 3.65 (d, J = 14.9 Hz, 1H), 2.43 – 2.51 (m, 1H), 0.74 – 0.41 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.8 (d, $J_{P\text{-}C}$ = 6.6 Hz), 129.6, 124.8 (d, $J_{P\text{-}C}$ = 1.1 Hz), 120.1 (d, $J_{P\text{-}C}$ = 5.1 Hz), 23.2, 7.1 (d, $J_{P\text{-}C}$ = 5.3 Hz). ^{31}P NMR (121 MHz, $CDCl_3$) δ -3.6. IR (ATR Diamond): 3218, 1591, 1483, 1453, 1433, 1417, 1249, 1209, 1190, 1176, 1162, 1101, 1025, 1005, 929, 903, 777, 766, 752, 691 cm^{-1}. HRMS (ESI) for: Exact mass calculated for $C_{15}H_{16}NO_3PNa$ $[M+Na]^+$ 312.0766, found 312.0774.

Diphenyl propylphosphoramidate (1v)



Synthesized according to general procedure. The title compound was isolated as white solid (2.47 g, 85% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (t, J = 7.9 Hz, 4H), 7.25 (d, J = 3.5 Hz, 3H), 7.23 (s, 1H), 7.14 (t, J = 7.4 Hz, 2H), 3.12 – 3.00 (m, 2H), 2.98 (d, J = 14.0 Hz, 1H), 1.48 (h, J = 7.3 Hz, 2H), 0.86 (t, J = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.9 (d, $J_{P\text{-}C}$ = 6.7 Hz), 129.6, 124.9, 120.2 (d, $J_{P\text{-}C}$ = 5.0 Hz), 43.6, 24.7 (d, $J_{P\text{-}C}$ = 6.3 Hz), 11.0. ^{31}P NMR (121 MHz, $CDCl_3$) δ -3.3. IR (ATR Diamond): 3174, 3055, 2963, 2932, 2875, 1589, 1486, 1456, 1250, 1233, 1213, 1187, 1167, 1104, 1069, 1036, 1021, 1006, 949, 927, 915, 773, 749, 688, 586, 545 cm^{-1}. HRMS (ESI): Exact mass calculated for $C_{15}H_{18}NO_3PNa$ $[M+Na]^+$ 314.0922, found 314.0920.

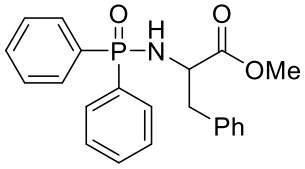
Diphenyl *tert*-butylphosphoramidate (1y)



Synthesized according to general procedure. The title compound was isolated as white solid (2.28g, 75% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.27 (m, 4H), 7.24 (dq, J = 7.4, 1.2 Hz, 4H), 7.10 – 7.13 (m, 2H), 3.24 (s, 1H), 1.32 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.1 (d, $J_{P\text{-}C}$ = 7.0 Hz), 129.5, 124.6 (d, $J_{P\text{-}C}$ = 1.3 Hz), 120.1 (d, $J_{P\text{-}C}$ = 5.2 Hz), 51.7 (d, $J_{P\text{-}C}$ = 1.5 Hz), 31.2 (d, $J_{P\text{-}C}$ = 4.9 Hz). ^{31}P NMR (121 MHz, $CDCl_3$) δ -6.2. IR (ATR

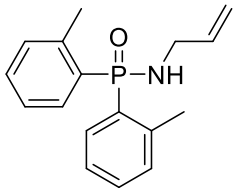
Diamond): 3264, 2974, 1588, 1484, 1409, 1388, 1365, 1260, 1219, 1192, 1156, 1047, 1021, 923, 867, 784, 757, 721, 690 cm^{-1}. HRMS (ESI): Exact mass calculated for $C_{16}H_{21}NO_3P$ $[M+H]^+$ 306.1255, found 306.1259

Methyl (diphenylphosphoryl)phenylalaninate (1p)



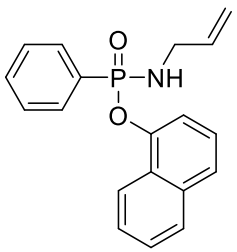
Synthesized according to general procedure. The title compound was isolated as white solid (2.27g, 60% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83-7.78 (m, 2H), 7.69-7.63 (m, 2H), 7.51 – 7.38 (m, 4H), 7.36-7.32 (m, 2H), 7.30 – 7.22 (m, 3H), 7.13 (dd, J = 7.7, 1.8 Hz, 2H), 4.09-4.01 (m, 1H), 3.64 (s, 3H), 3.49 (dd, J = 11.2, 6.9 Hz, 1H), 3.08 (dd, J = 6.0, 1.8 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.2 (d, J_{P-C} = 5.6 Hz), 135.9 , 132.6 (d, J_{P-C} = 11.7 Hz), 132.1 (d, J_{P-C} = 9.7 Hz), 132.0 (d, J_{P-C} = 2.8 Hz), 131.9 (d, J_{P-C} = 9.8 Hz), 131.9 (d, J_{P-C} = 2.8 Hz), 131.3 (d, J_{P-C} = 10.1 Hz), 129.7, 128.5 (d, J_{P-C} = 2.7 Hz), 128.5 (d, J_{P-C} = 22.8 Hz), 128.5, 127.0, 54.7, 52.2, 41.2 (d, J_{P-C} = 5.0 Hz). ^{31}P NMR (121 MHz, $CDCl_3$) δ 20.3 IR (ATR Diamond): 3145, 1732, 1659, 1591, 1433, 1282, 1243, 1182, 1125, 1108, 1096, 1043, 1016, 940, 918, 748, 726, 691 cm^{-1}. HRMS (ESI) for: Exact mass calculated for $C_{22}H_{22}NO_3PNa$ $[M+Na]^+$ 402.1235, found 402.1241.

***N*-Allyl-*P*,*P*-di-*o*-tolylphosphinic amide (1m)**



Synthesized according to general procedure. The title compound was isolated as white solid (1.99 g, 70% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73-7.67 (m, 2H), 7.40-7.35 (m, 2H), 7.24 – 7.16 (m, 4H), 6.03-5.93 (m, 1H), 5.33 – 5.04 (m, 2H), 3.70-3.64 (m, 2H), 2.73 (d, J = 7.4 Hz, 1H), 2.46 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 142.3 (d, J_{P-C} = 9.9 Hz), 136.5 (d, J_{P-C} = 7.3 Hz), 132.9 (d, J_{P-C} = 10.7 Hz), 131.8, 131.7 (d, J_{P-C} = 15.2 Hz), 131.0 (d, J_{P-C} = 123.5 Hz), 125.4 (d, J_{P-C} = 12.6 Hz), 115.8, 43.5, 21.5 (d, J_{P-C} = 4.0 Hz). ^{31}P NMR (121 MHz, $CDCl_3$) δ 25.5. IR (ATR Diamond): 3117, 3062, 1645, 1591, 1452, 1433, 1275, 1201, 1166, 1136, 1076, 1030, 998, 910, 866, 806, 762, 716, 691, 671 cm^{-1}. HRMS (ESI): Exact mass calculated for $C_{17}H_{20}NOPNa$ $[M+Na]^+$ 308.1180 , found 308.1181.

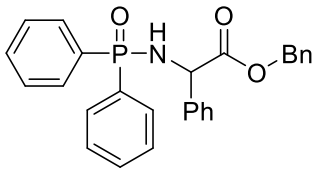
Naphthalen-1-yl-*N*-allyl-*P*-phenylphosphonamidate (1r)



Synthesized according to general procedure. The title compound was isolated as white solid (2.58 g, 80% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 – 8.10 (m, 1H), 8.03 – 7.92 (m, 2H), 7.87 – 7.78 (m, 1H), 7.61 (dd, J = 7.9, 1.2 Hz, 2H), 7.59 – 7.52 (m, 1H), 7.48 (ddt, J = 8.3, 6.9, 2.8 Hz, 4H), 7.35 (t, J = 8.0 Hz, 1H), 5.67 (ddt, J = 17.2, 10.6, 5.5 Hz, 1H), 5.23 – 4.78 (m, 2H), 3.57 (ddt, J = 9.2, 5.5, 1.6 Hz, 2H), 3.03 (s, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 146.7 (d, J_{P-C} = 8.4 Hz), 135.5 (d, J_{P-C} = 6.3 Hz), 134.8, 132.3 (d, J_{P-C} = 3.2 Hz), 131.4 (d, J_{P-C} = 10.0 Hz), 131.2 , 129.4 , 128.6 (d, J_{P-C} = 14.7 Hz), 127.9 , 126.6 (d, J_{P-C} = 5.9 Hz), 126.3 (d, J_{P-C} = 28.5 Hz), 125.7 (d, J_{P-C} = 0.9 Hz), 124.3 , 121.5 , 115.9 , 115.3 (d, J_{P-C} = 4.0 Hz), 43.6. ^{31}P NMR (121 MHz, $CDCl_3$) δ 17.8. IR

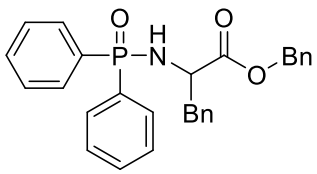
(ATR Diamond): 3207, 1574, 1462, 1437, 1392, 1262, 1214, 1125, 1077, 1046, 913, 853, 795, 771, 750, 718, 694 cm^{-1}. HRMS (ESI): Exact mass calculated for $C_{19}H_{18}NO_2PNa$ $[M+Na]^+$ 346.0973, found 346.0971.

Benzyl 2-((diphenylphosphoryl)amino)-2-phenylacetate (1o)



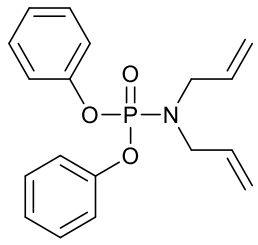
Synthesized according to general procedure. The title compound was isolated as white solid (2.96 g, 67% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 – 7.81 (m, 2H), 7.78 – 7.70 (m, 2H), 7.54 – 7.48 (m, 1H), 7.47 – 7.40 (m, 3H), 7.34 – 7.26 (m, 10H), 7.17 – 7.11 (m, 2H), 5.16 – 5.09 (m, 2H), 4.99 (dd, J = 10.7, 9.7 Hz, 1H), 4.30 (dd, J = 9.7, 6.6 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.0 (d, J_{P-C} = 5.7 Hz), 138.1 (d, J_{P-C} = 4.4 Hz), 135.1, 132.5 (d, J_{P-C} = 48.5 Hz), 132.3 (d, J_{P-C} = 9.8 Hz), 132.1 (d, J_{P-C} = 2.8 Hz), 131.9, 131.9 (d, J_{P-C} = 9.6 Hz), 131.2 (d, J_{P-C} = 51.7 Hz), 129.2 , 128.6 (d, J_{P-C} = 12.7 Hz), 128.6 (d, J_{P-C} = 23.9 Hz), 128.3 (d, J_{P-C} = 24.4 Hz), 128.3, 128.1, 127.9, 127.0, 67.5, 57.0. IR (ATR Diamond): 3059, 1743, 1591, 1538, 1436, 1248, 1222, 1160, 1065, 1040, 1019, 955, 741, 754, 717, 692 cm^{-1}. HRMS (ESI): Exact mass calculated for $C_{27}H_{25}NO_3P$ $[M+H]^+$ 442.1572 , found 442.1594.

Benzyl (diphenylphosphoryl)phenylalaninate (1q)



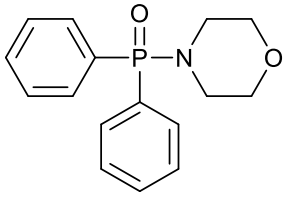
Synthesized according to general procedure. The title compound was isolated as white solid (3.19 g, 70% yield). ^{1}H NMR (300 MHz, CD_3OD) δ 7.82 – 7.66 (m, 2H), 7.61 – 7.40 (m, 6H), 7.40 – 7.23 (m, 10H), 7.21 – 7.11 (m, 2H), 5.17 – 4.98 (m, 2H), 3.85 (td, J = 8.4, 6.5 Hz, 1H), 3.17 – 2.94 (m, 2H). ^{13}C NMR (75 MHz, CD_3OD) δ 172.8 (d, J_{P-C} = 3.2 Hz), 136.9, 135.7, 132.5, 132.0 (d, J_{P-C} = 2.7 Hz), 131.9, 131.8, 131.7 (d, J_{P-C} = 1.4 Hz), 131.6, 130.7, 130.1, 129.4, 128.3, 128.1, 128.1 (d, J_{P-C} = 1.8 Hz), 127.9, 126.6, 66.5, 55.7, 40.1 (d, J_{P-C} = 7.2 Hz). ^{31}P NMR (121 MHz, $CDCl_3$) δ 22.93. IR (ATR Diamond): 3172, 3059, 1738, 1434, 1383, 1159, 1177, 1125, 1106, 1091, 919, 902, 742, 727, 693 cm^{-1}. HRMS (ESI): Exact mass calculated for $C_{28}H_{26}NO_3PNa$ $[M+Na]^+$ 478.1548 , found 478.1541.

Diphenyl diallylphosphoramidate



Synthesized according to general procedure. The title compound was isolated as white solid (2.87 g, 87% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 – 7.09 (m, 10H), 5.56 (ddt, J = 17.5, 9.7, 6.3 Hz, 2H), 5.19 – 5.07 (m, 4H), 3.75 (ddt, J = 11.3, 6.3, 1.3 Hz, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.19 (d, J_{P-C} = 6.7 Hz), 133.94 (d, J_{P-C} = 2.6 Hz), 129.92, 125.12 (d, J_{P-C} = 1.1 Hz), 120.52 (d, J_{P-C} = 5.0 Hz), 118.78, 48.12 (d, J_{P-C} = 4.4 Hz). ^{31}P NMR (121 MHz, $CDCl_3$) δ -2.77. IR (ATR Diamond): 3452, 1588, 1485, 1254, 1227, 1186, 1162, 1111, 1085, 1023, 993, 923, 910, 775, 762, 613 cm^{-1}. HRMS (EI): Exact mass calculated for $C_{18}H_{20}NO_3P$ $[M]^+$: 329.11808, found 329.11611

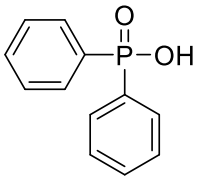
Morpholinodiphenylphosphine oxide



Synthesized according to general procedure. The title compound was isolated as colorless solid (1.60 g, 56 % yield). ^{1}H NMR (300 MHz, CD_3OD) δ 8.02 – 7.73 (m, 1H), 7.69 – 7.24 (m, 1H), 3.77 – 3.58 (m, 1H), 3.09 – 2.94 (m, 1H). ^{13}C NMR (75MHz, CD_3OD): δ 133.72 (d, $J_{P\text{-}C}$ = 2.8 Hz), 133.45 (d, $J_{P\text{-}C}$ = 9.4 Hz), 131.20 (d, $J_{P\text{-}C}$ = 131.4 Hz), 130.14 (d, $J_{P\text{-}C}$ = 12.7 Hz), 68.05 (d, $J_{P\text{-}C}$ = 6.6 Hz), 46.19. ^{31} P NMR (121 MHz, $CDCl_3$) δ 28.6. IR (ATR Diamond): 3053, 2961, 2848, 1723, 1440, 1367, 1294, 1255, 1196, 1111, 1082, 1067, 1019, 957, 914, 846, 764, 702, 694, 669, 611, 598, 552 cm^{-1}. HRMS (ESI): Exact mass calculated for $C_{16}H_{18}NO_2PNa$ $[M+Na]^+$ 310.0973, found 310.0983.

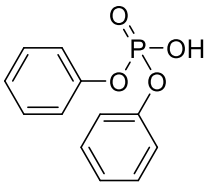
3. Spectral Data of Products.

Diphenylphosphinic acid



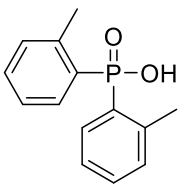
White solid, ^{1}H NMR (400 MHz, CD_3OD) δ 7.85 – 7.71 (m, 4H), 7.59 – 7.50 (m, 2H), 7.50 – 7.41 (m, 4H). ^{13}C NMR (101 MHz, CD_3OD) δ 133.1 (d, $J_{P\text{-}C}$ = 138.4 Hz), 131.8 (d, $J_{P\text{-}C}$ = 2.9 Hz), 130.9 (d, $J_{P\text{-}C}$ = 10.3 Hz), 128.2 (d, $J_{P\text{-}C}$ = 13.2 Hz). Spectral data is in good agreement with literature data.[16]

Diphenyl hydrogen phosphate



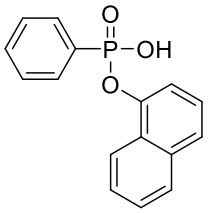
White solid, ^{1}H NMR (300 MHz, CD_3OD) δ 7.34 (dddd, J = 9.0, 6.5, 1.2, 0.6 Hz, 4H), 7.23 – 7.13 (m, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.3 (d, $J_{P\text{-}C}$ = 7.1 Hz), 129.7, 125.4, 120.2 (d, $J_{P\text{-}C}$ = 4.9 Hz). Spectral data is in good agreement with literature data.[17]

Di-*o*-tolylphosphinic acid



colorless oil. ^{1}H NMR (400 MHz, CD_3OD) δ 7.94-7.88 (m, 2H), 7.44 (tt, J = 7.6, 1.5 Hz, 2H), 7.33-7.28 (m, 2H), 7.26 – 7.18 (m, 2H), 2.27 (s, 6H). ^{13}C NMR (101 MHz, CD_3OD) δ 141.1 (d, $J_{P\text{-}C}$ = 11.4 Hz), 132.5 (d, $J_{P\text{-}C}$ = 9.8 Hz), 132.1, 132.0, 131.1 (d, $J_{P\text{-}C}$ = 12.5 Hz), 125.2 (d, $J_{P\text{-}C}$ = 12.6 Hz), 20.0 (d, $J_{P\text{-}C}$ = 4.6 Hz). ^{31}P NMR (121 MHz, CD_3OD) δ 25.56. IR (ATR Diamond): 3183, 2919, 2848, 1645, 1594, 1276, 939, 715, 699 cm^{-1}. HRMS (ESI): Exact mass calculated for $C_{14}H_{15}O_2PNa$ $[M+Na]^+$ 269.0707 , found 269.0726.

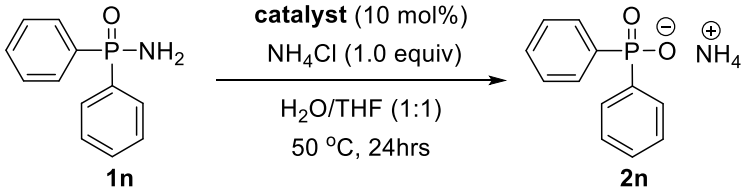
Naphthalen-1-yl hydrogen phenyl phosphonate

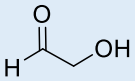
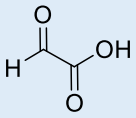
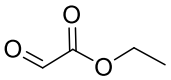
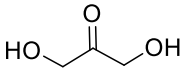
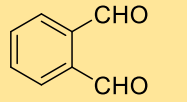
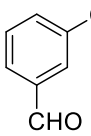
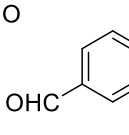
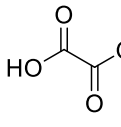
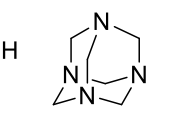
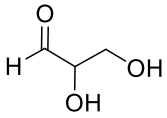
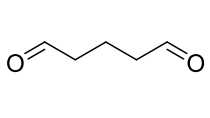
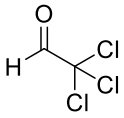
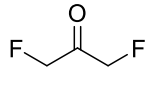
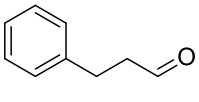


colorless oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.50 (s, 1H), 8.06 (dd, J = 8.3, 1.2 Hz, 1H), 7.89-7.83 (m, 2H), 7.78 – 7.71 (m, 1H), 7.56 – 7.46 (m, 2H), 7.45 – 7.28 (m, 5H), 7.16 (t, J = 7.9 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 146.4 (d, $J_{P\text{-}C}$ = 8.4 Hz), 134.7, 132.7 (d, $J_{P\text{-}C}$ = 3.1 Hz), 131.5 (d, $J_{P\text{-}C}$ = 10.4 Hz), 129.0, 128.4 (d, $J_{P\text{-}C}$ = 15.8 Hz), 127.5, 127.0, 126.8 (d, $J_{P\text{-}C}$ = 5.4 Hz), 126.3 (d, $J_{P\text{-}C}$ = 32.6 Hz), 125.4 (d, $J_{P\text{-}C}$ = 1.6 Hz), 124.7 (d, $J_{P\text{-}C}$ = 1.5 Hz), 122.1, 115.6 (d, $J_{P\text{-}C}$ = 3.6 Hz). ^{31}P NMR (121 MHz, $CDCl_3$) δ 17.3. IR (ATR Diamond): 3056, 1595, 1574, 1507, 1462, 1439, 1390, 1258, 1226, 1154, 1132, 1082, 1043, 977, 914, 815, 795, 769, 749, 719, 691, 638, 596, 565, 544 cm^{-1}. HRMS (ESI): Exact mass calculated for $C_{16}H_{13}O_3PNa$ $[M+Na]^+$ 307.0500 , found 307.0506.

4. Details for Optimization of the Reaction Conditions.

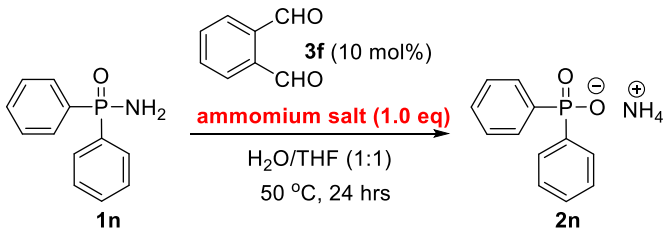
Table S1. Survey of catalysts for the hydrolysis of diphenylphosphinic amide **1n**[a]



									
85%	77%	71%	45%	32%					
3a	**3b**	**3c**	**3d**	**3e**					
83%	NR	NR	30%	23%					
3f	**3g**	**3h**	**3i**	**3j**					
									
0.1 eq 15% 0.2 eq 57%	7%	6%	4%	3%					
3k	**3l**	**3m**	**3n**	**3o**					

[a] Reaction conditions: **1n** (0.2 mmol), NH_4Cl (0.2 mmol) and **catalyst** (0.02 mmol) in 0.2 mL of THF and 0.2 mL of H_2O at 50 °C for 24 h in a sealed tube. The yield was determined by ^1H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard.

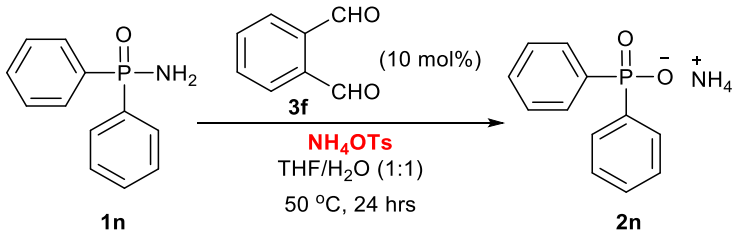
Table S2. Screening of additives of the hydrolysis of diphenylphosphinic amide **1n**[a]

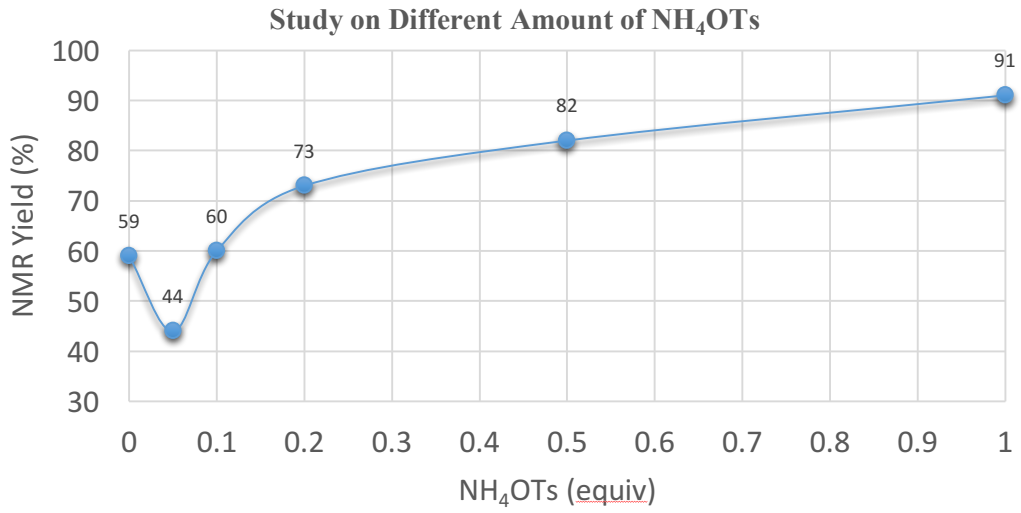


Entry[a]	Ammonium salt	Yield (%)[b]
1	NH_4OTs	91
2	NH_4Cl	83
3	NH_4I	74
4	NH_4OMs	70
5	NH_4O_2CH	23
6	NH_4OAc	N.R.
7	NH_4OH	N.R.

[a] Reaction conditions: **1n** (0.2 mmol), ammonia salt (0.2 mmol) and **3f** (0.02 mmol) in 0.2 mL of solvent and 0.2 mL of H_2O at 50 °C for 24 h in a sealed tube. [b] Determined by ^1H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard.

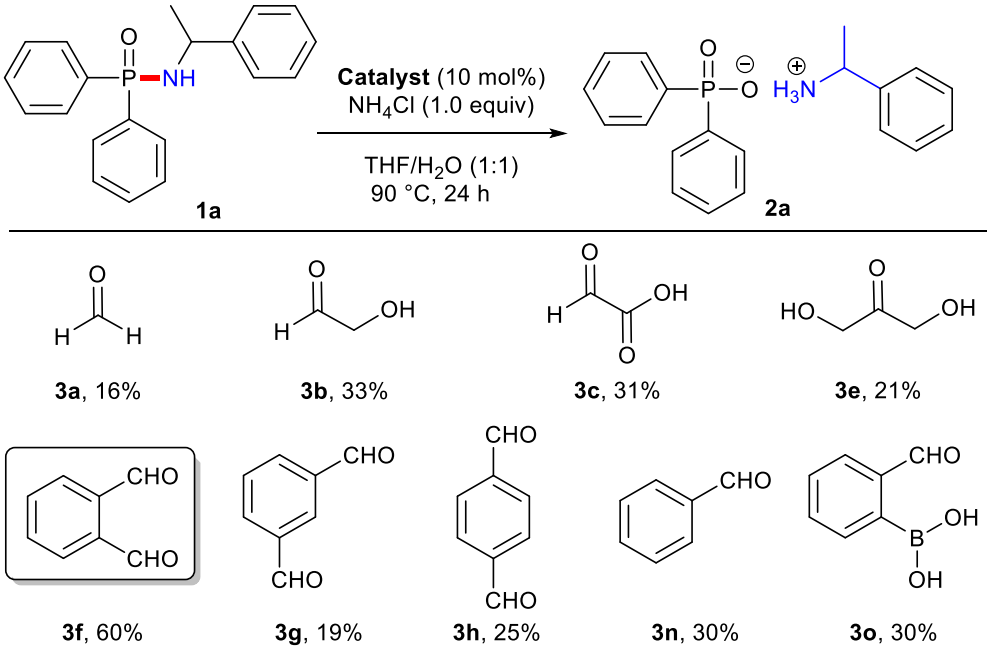
Table S3. Study on the amount of NH_4OTs[a]





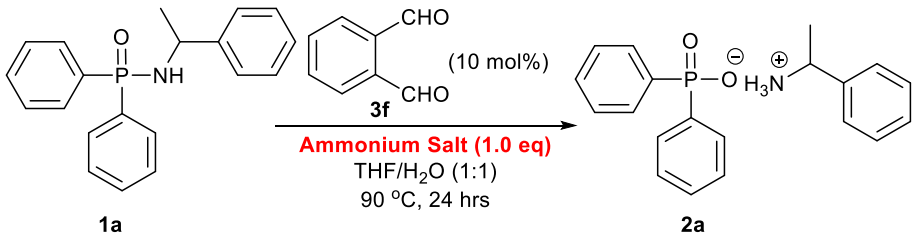
[a] Reaction conditions: **1n** (0.2 mmol), NH_4OTs and **3f** (0.02 mmol) in 0.2 mL of THF and 0.2 mL of H_2O at 50 °C for 24 h in a sealed tube. The yield was determined by ^1H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard.

Table S4. Survey of catalysts for the hydrolysis of diphenylphosphinic amide **1a**[a]



[a] Reaction conditions: **1a** (0.2 mmol), NH_4Cl (0.2 mmol) and **3** (0.02 mmol) in 0.2 mL of THF and 0.2 mL of H_2O at 90 °C for 24 h in a sealed tube. The yield was determined by ^1H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard.

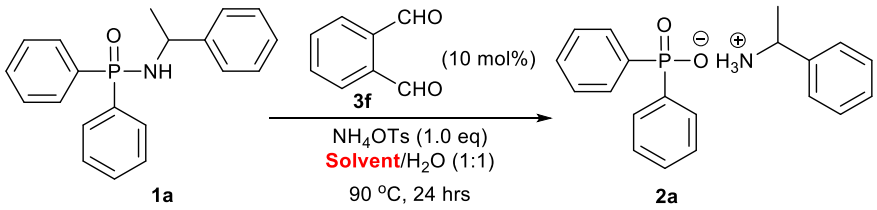
Table S5 Screen of additives for the hydrolysis of diphenylphosphinic amide **1a**[a]



Entry[a]	Ammonium salt	Solvent	Yield (%)[b]
1	NH_4Cl	THF	60
2	$MeNH_2$•HCl	THF	40
3	Pyrrolidine•HCl	THF	42
2	$(n\text{-Bu})_3N$•HCl	THF	52
3	**NH_4OTs**	**THF**	**75**
4	NH_4OAc	THF	10
5	NH_4COOH	THF	N.R.
6	-	THF	36

[a]Reaction conditions: **1a** (0.2 mmol), ammonia salt (0.2 mmol) and **3f** (0.02 mmol) in 0.2 mL of solvent and 0.2 mL of H_2O at 90 °C for 24 h in a sealed tube. [b]Determined by ^1H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard.

Table S6. Solvent screen for the hydrolysis of diphenylphosphinic amide **1a**[a]



Entry[a]	Ammonium salt	Solvent	Yield (%)[b]
1	NH_4OTs	THF	75
2	NH_4OTs	DMF	38
3	NH_4OTs	DMSO	59
4	NH_4OTs	1,4-Dioxane	69
5	NH_4OTs	MeCN	82
6[c]	**NH_4OTs**	**MeCN**	**99 (91)**[d]
7[e]	NH_4OTs	MeCN	4

[a] Reaction conditions: **1a** (0.2 mmol), ammonia salt (0.2 mmol) and **3f** (0.02 mmol) in 0.2 mL of solvent and 0.2 mL of H_2O at 90 °C for 24 h in a sealed tube. [b] Determined by ^1H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard. [c] **3f** (0.04 mmol) was used. [d] Isolated yield based on diphenylphosphinic acid. [e] Without *o*-phthalaldehyde.

5. General Procedure for the Hydrolysis Reaction

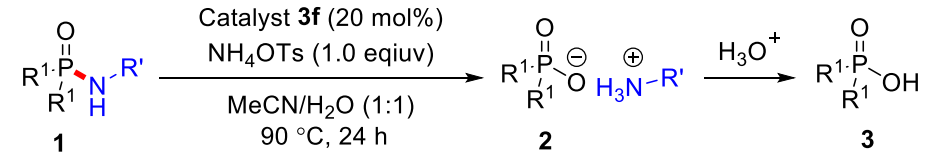
5.1 Isolation of diphenylphosphinic acid.

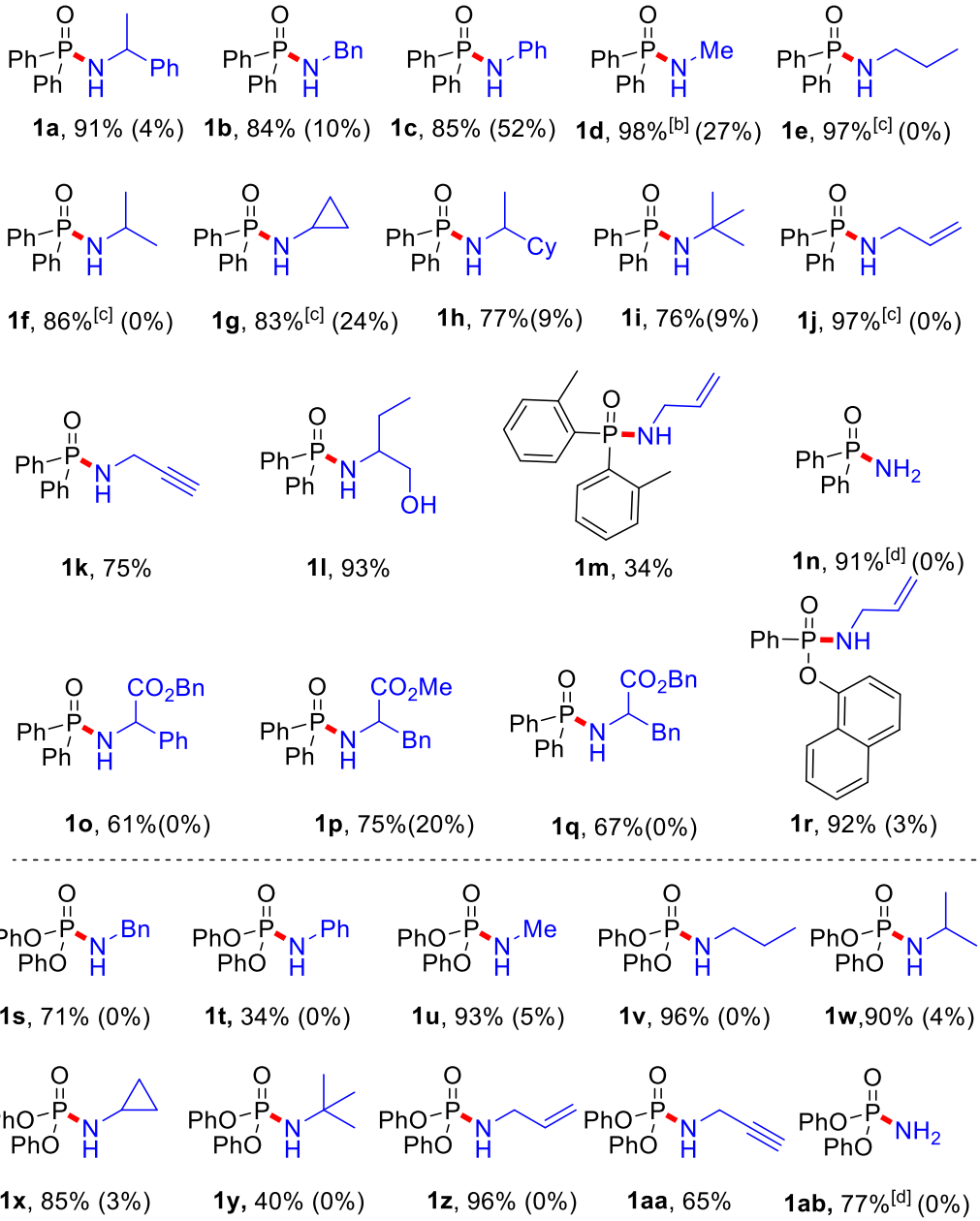
Diphenylphosphinic amide **1** (0.2 mmol), phthalaldehyde **3f** (0.04mmol) and NH_4OTs (0.2 mmol) were added into a sealed tube. 0.2 mL MeCN and 0.2 mL H_2O were followed. Then the reaction mixture was stirred under 90 °C for 24 hours. Then the solvent was removed by high vacuum and following adding H_2O (10 mL) and toluene (10 mL). The mixture was stirred for 10 minutes and the aqueous layer was collected. After acidification of the aqueous layer by 1 M HCl to pH = 1~2, then the aqueous layer was extracted using 5% methanol in dichloromethane (20 mL x 3). The organic layers was combined, dried over Na_2SO_4 and filtered and concentrated *in vacuo*.

5.2 Isolation of amine component.

Diphenylphosphinic amide **1** (0.2 mmol), phthalaldehyde **3f** (0.04mmol) and NH_4OTs (0.2 mmol) were added into a sealed tube. 0.2 mL MeCN and 0.2 mL H_2O were followed. Then the reaction mixture was stirred under 90 °C for 24 hours. Then the solvent was removed by high vacuum and following adding H_2O (10 mL) and toluene (10 mL). The mixture was stirred for 10 minutes and the aqueous layer was collected. After basification of the aqueous layer by 1 M NaOH to pH = 8~9, then the aqueous layer was extracted using ethyl acetate (20 mL x 3). The organic layers was combined, dried over Na_2SO_4 and filtered and concentrated *in vacuo*.

Table S7. Substrate scope for the catalytic hydrolysis of P(=O)-NHR reagents showing the results in the absence of *o*-phthalaldehyde[a]

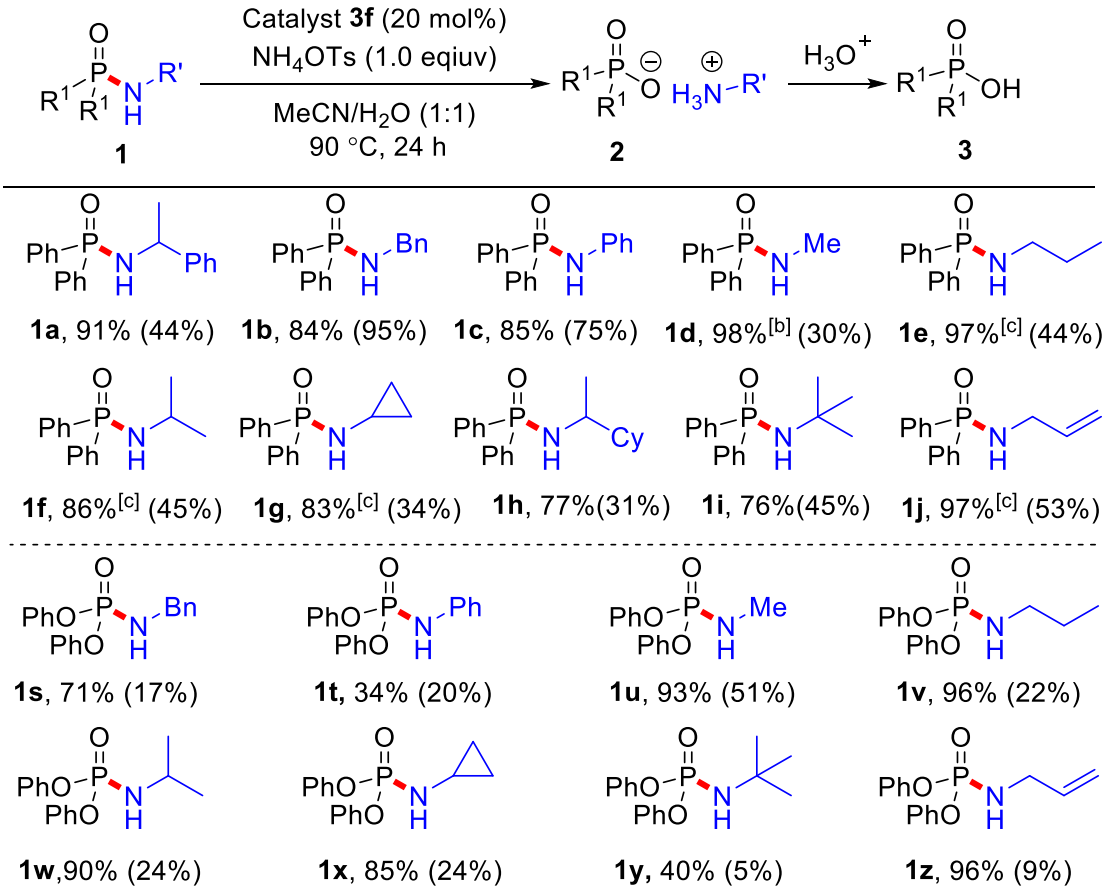




[a] Reaction conditions: **1** (0.2 mmol), NH_4OTs (0.2 mmol) and **3f** (0.04 mmol) in 0.2 mL of solvent and 0.2 mL of H_2O at 90 °C for 24 h in a sealed tube. Results between parentheses related to similar reactions performed without adding **3f**. Amine component of hydrolysis reaction of **1a** was isolated in 87% yield. [b] NH_4OTs (0.04 mmol) was used at r.t. [c] NH_4OTs (0.04 mmol) and **3f** (0.04 mmol) were used at 50 °C. [d] Performed in THF (0.2 mL) and H_2O (0.2 mL) at 50 °C.

6. Comparison between phthalaldehyde and formaldehyde[a].

Table S8. Comparison between phthalaldehyde and formaldehyde[a]



[a] Reaction conditions: **1a** (0.2 mmol), NH_4OTs (0.2 mmol) and **3f** (0.04 mmol) in 0.2 mL of solvent and 0.2 mL of H_2O at 90 °C for 24 h in a sealed tube. Results between parentheses related to similar reactions performed using 20 mol% of formaldehyde. [b] NH_4OTs (0.04 mmol) was used at r.t. [c] NH_4OTs (0.04 mmol) and **3f** (0.04 mmol) were used at 50 °C.

Table S9. Control experiments

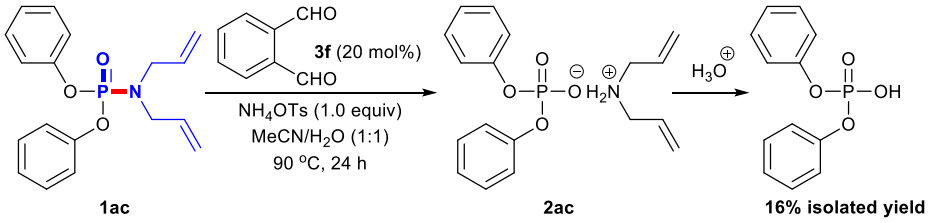
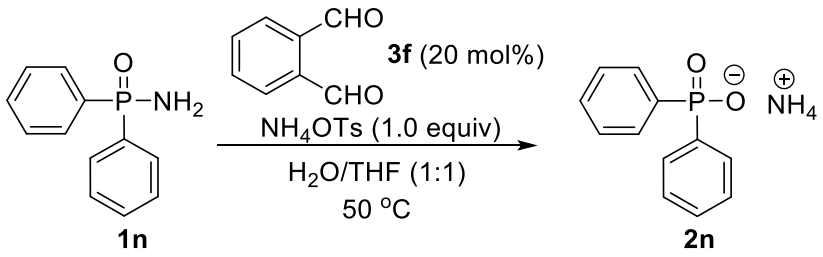
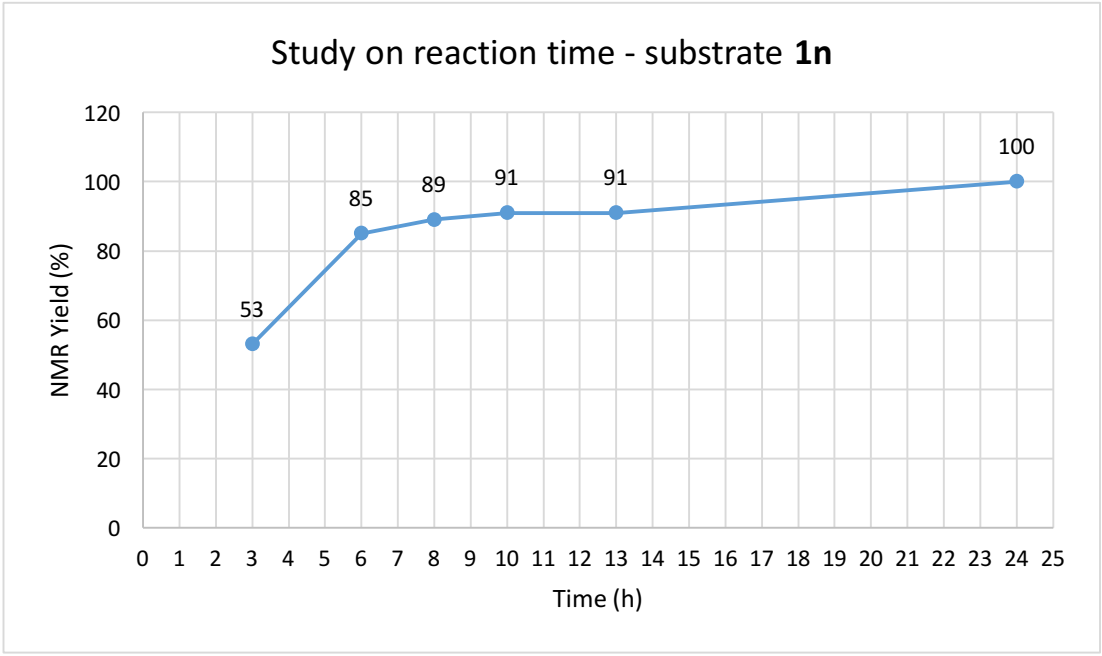
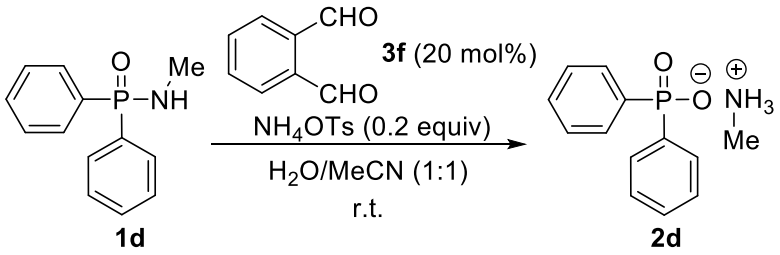


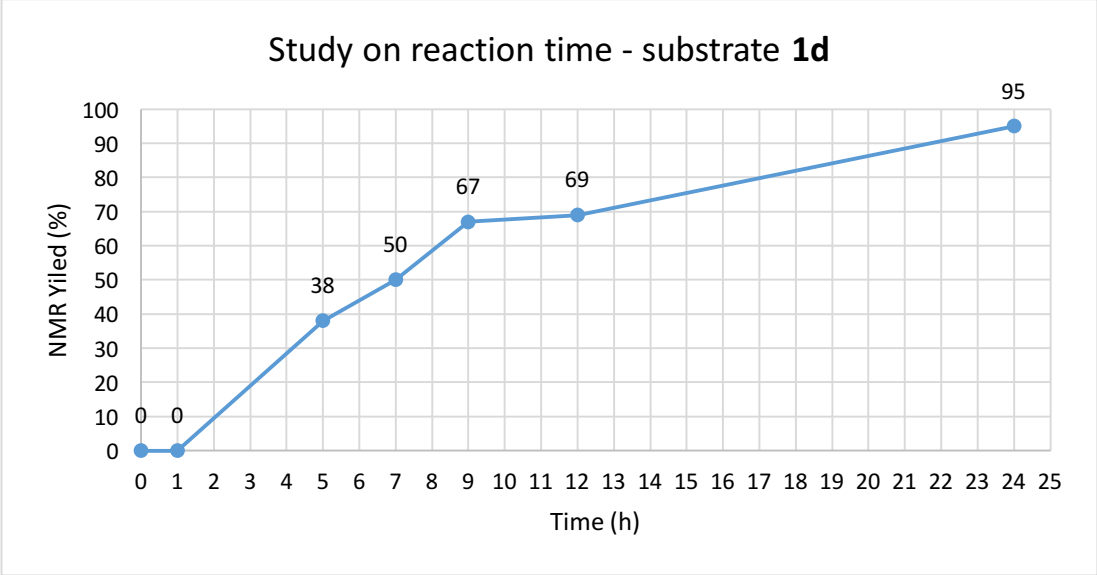
Table S10. Monitor the reaction time.



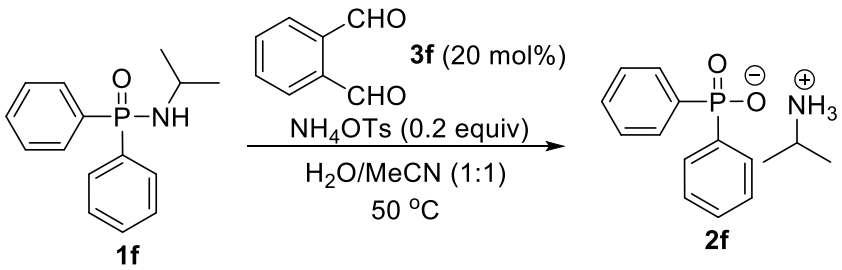


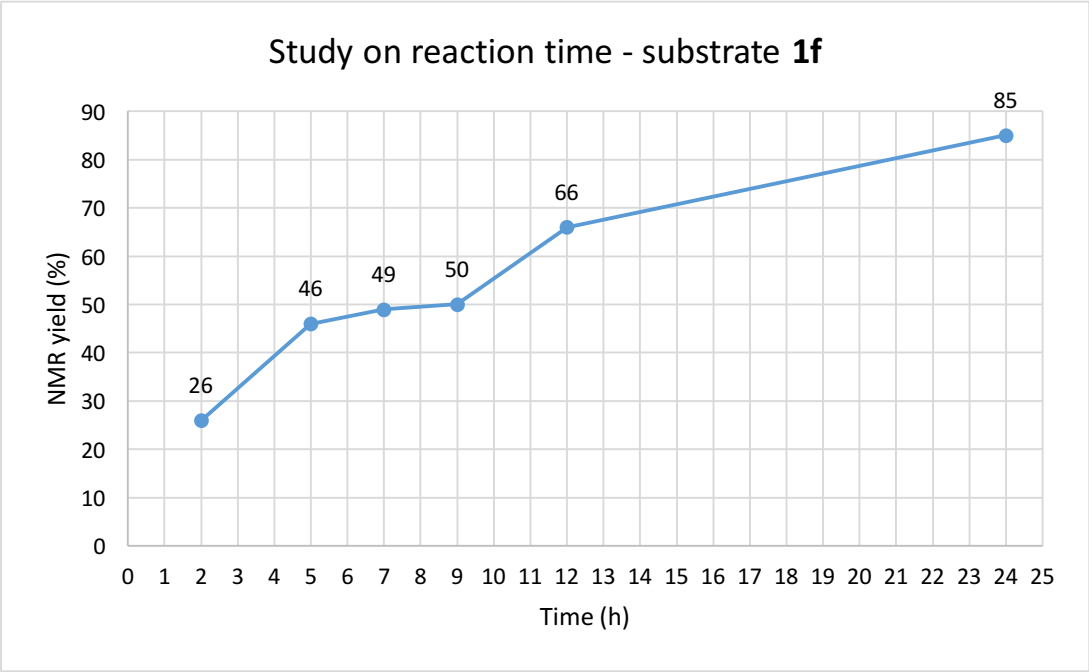
Reaction conditions: **1n** (0.2 mmol), NH_4OTs (0.2 mmol) and **3f** (0.04 mmol) in 0.2 mL of THF and 0.2 mL of H_2O at 50 °C for 24 h in a sealed tube. The reaction was monitored by ^1H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard.





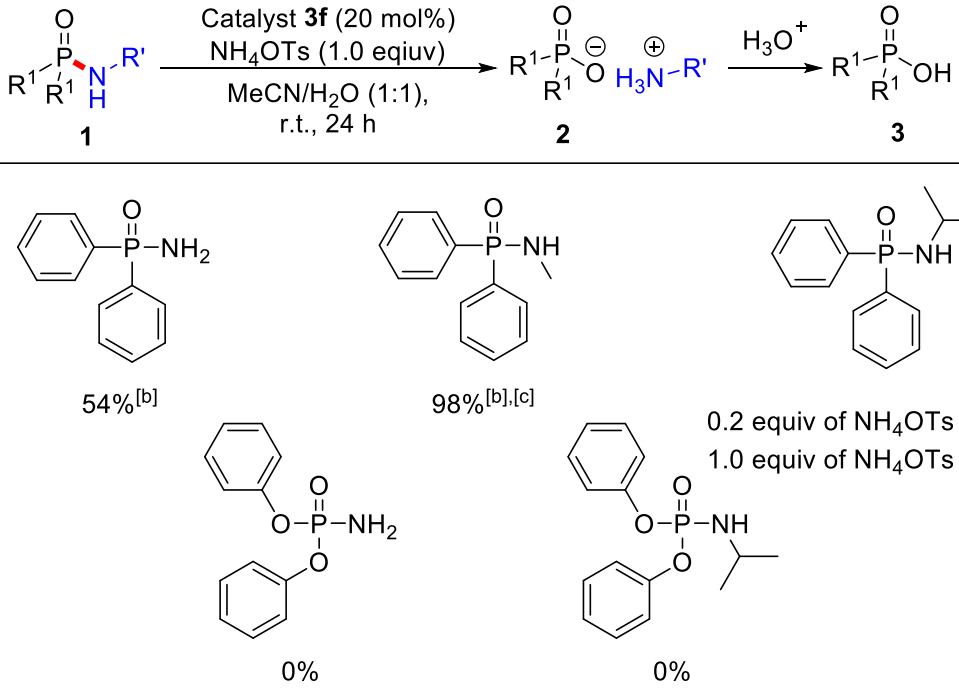
Reaction conditions: **1d** (0.2 mmol), NH_4OTs (0.04 mmol) and **3f** (0.04 mmol) in 0.2 mL of MeCN and 0.2 mL of H_2O at r.t. for 24 h in a sealed tube. The yield was determined by ^1H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard.





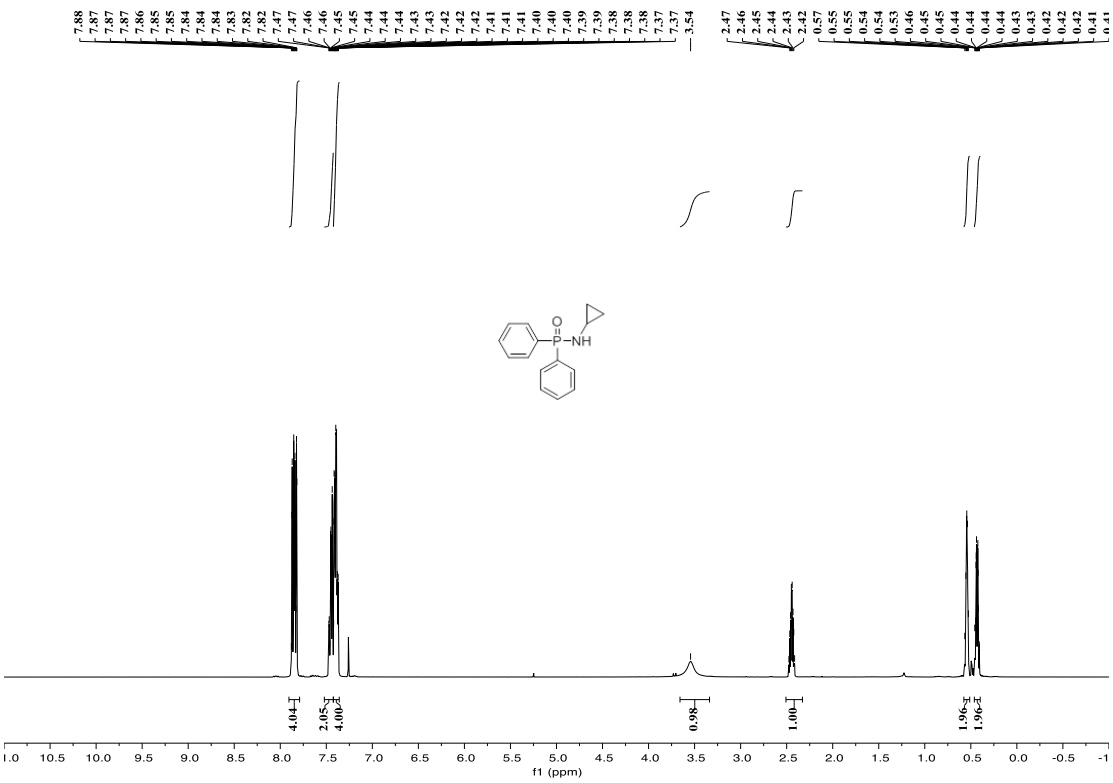
Reaction conditions: **1f** (0.2 mmol), NH_4OTs (0.04 mmol) and **3f** (0.04 mmol) in 0.2 mL of MeCN and 0.2 mL of H_2O at 50 °C for 24 h in a sealed tube. The yield was determined by ^1H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard.

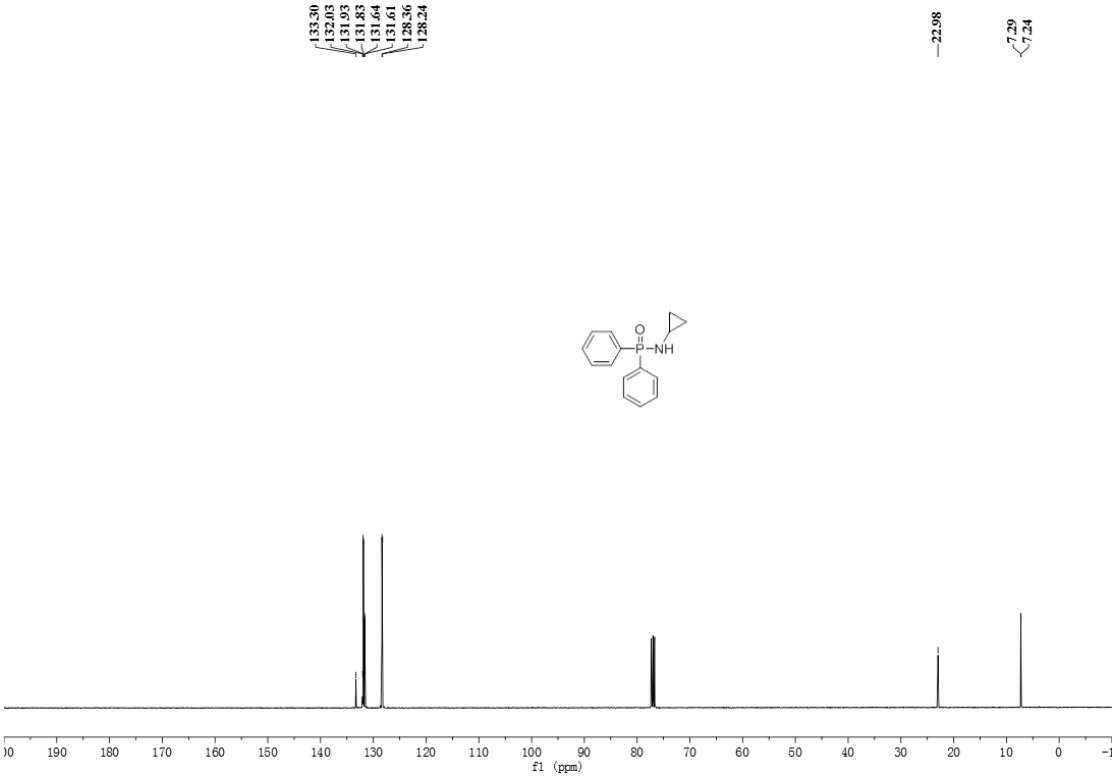
Table S11. Study the reaction at room temperature[a]

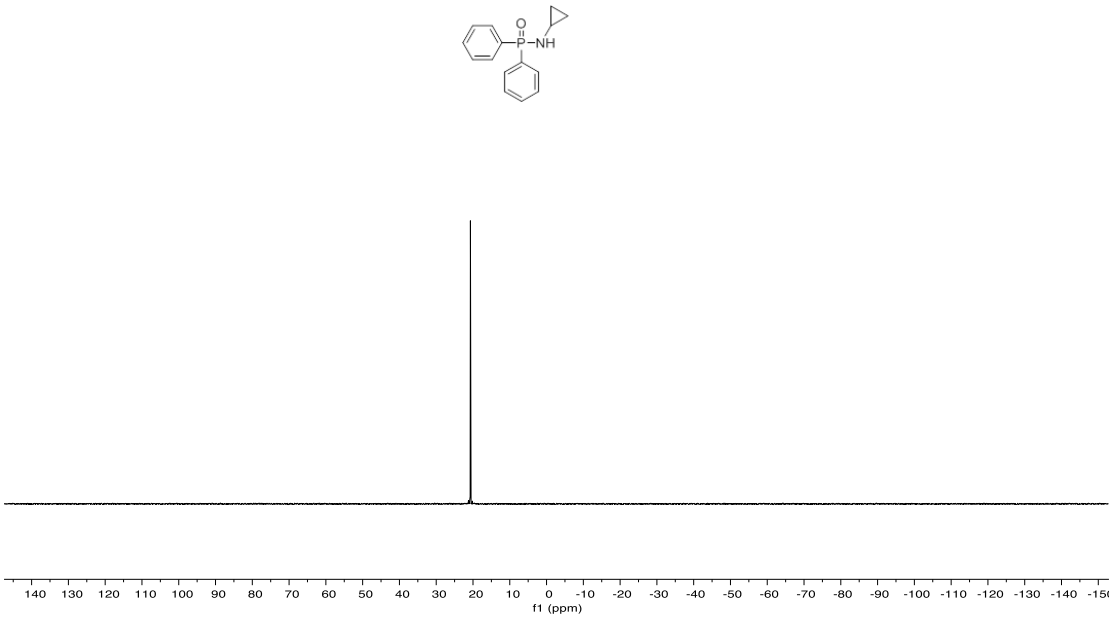


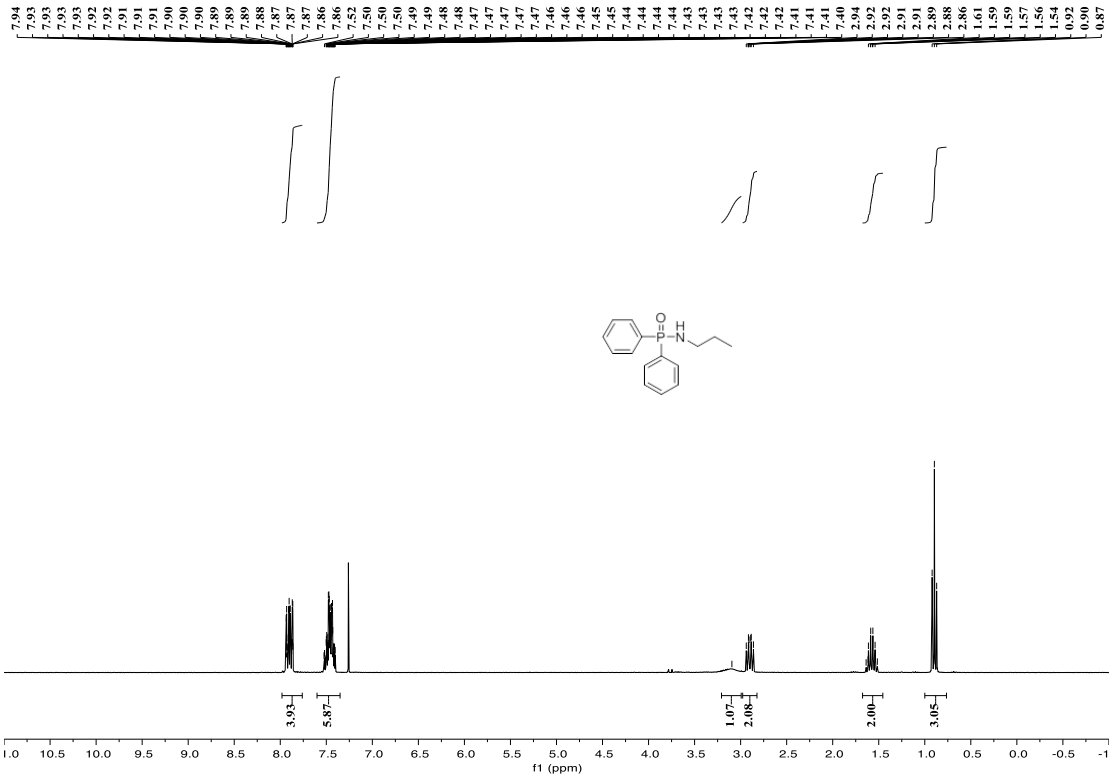
[a] Reaction conditions: **1** (0.2 mmol), NH_4OTs (0.2 mmol) and **3f** (0.04 mmol) in 0.2 mL of MeCN and 0.2 mL of H_2O at room temperature for 24 h in a sealed tube. [b] Isolated yield based on diphenylphosphinic acid. [c] NH_4OTs (0.04 mmol) was used.

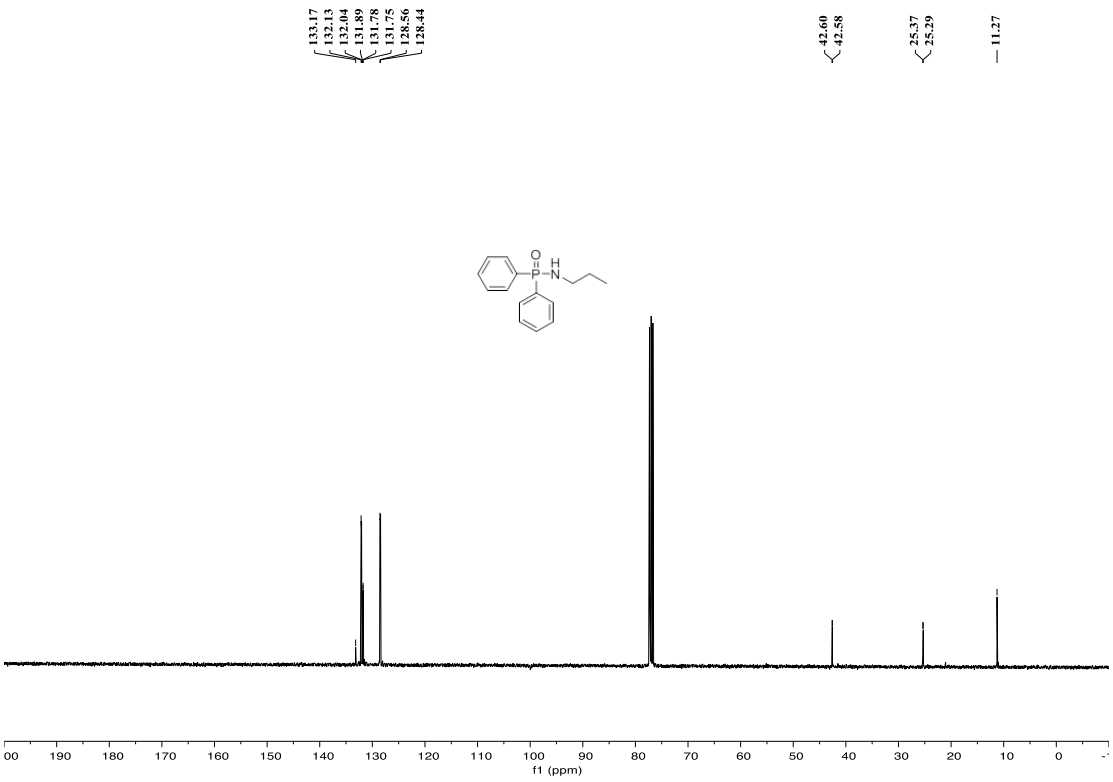
7. NMR spectra






O
P
NH
140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140
f1 (ppm)


7.94 7.93 7.93 7.93 7.93 7.92 7.92 7.91 7.91 7.91 7.90 7.90 7.90 7.89 7.89 7.89 7.88 7.87 7.87 7.87 7.86 7.86 7.52 7.50 7.50 7.50 7.49 7.49 7.48 7.48 7.47 7.47 7.47 7.47 7.47 7.46 7.46 7.46 7.45 7.45 7.44 7.44 7.44 7.44 7.43 7.43 7.43 7.43 7.42 7.42 7.42 7.41 7.41 7.41 7.40 2.94 2.92 2.92 2.91 2.91 2.89 2.88 2.86 1.61 1.59 1.59 1.57 1.56 1.54 0.92 0.90 0.87
O
P
H
N
3.93
5.87
1.07
2.08
2.00
3.05
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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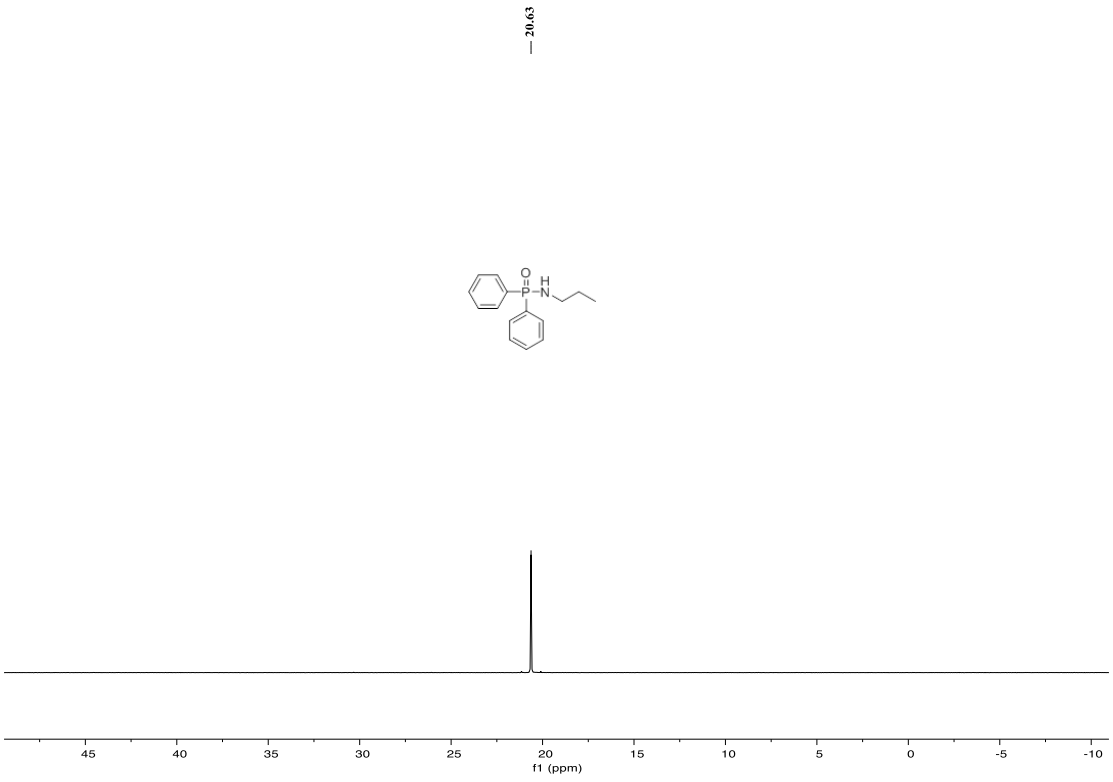
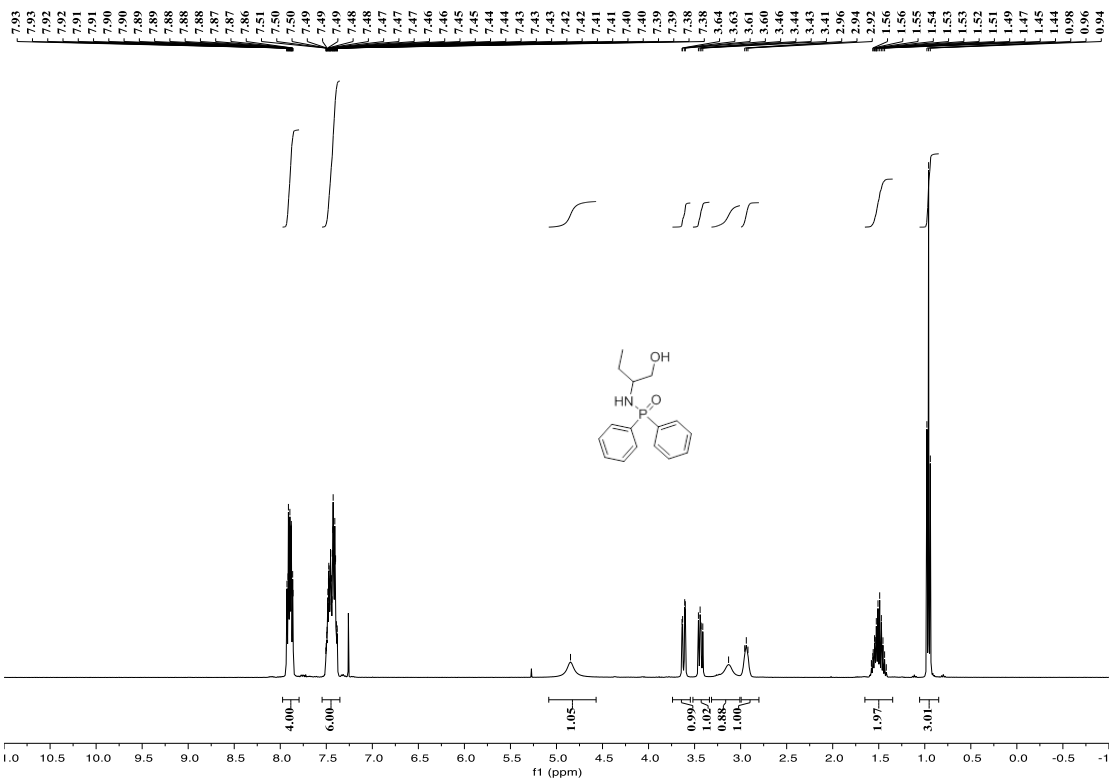
132.04
131.89
131.78
131.75
128.56
128.44
42.60
42.58
25.37
25.29
11.27
f1 (ppm)


20.63
f1 (ppm)


7.93 7.93 7.92 7.92 7.91 7.91 7.90 7.90 7.89 7.89 7.88 7.88 7.88 7.87 7.87 7.86 7.51 7.50 7.50 7.49 7.49 7.49 7.48 7.48 7.47 7.47 7.47 7.46 7.46 7.45 7.45 7.44 7.44 7.43 7.43 7.43 7.42 7.42 7.41 7.41 7.40 7.40 7.39 7.39 7.38 7.38 3.64 3.63 3.61 3.60 3.46 3.44 3.43 3.41 2.96 2.94 2.92 1.56 1.56 1.55 1.54 1.53 1.53 1.52 1.51 1.49 1.47 1.45 1.44 0.98 0.96 0.94
OH
HN
P
O
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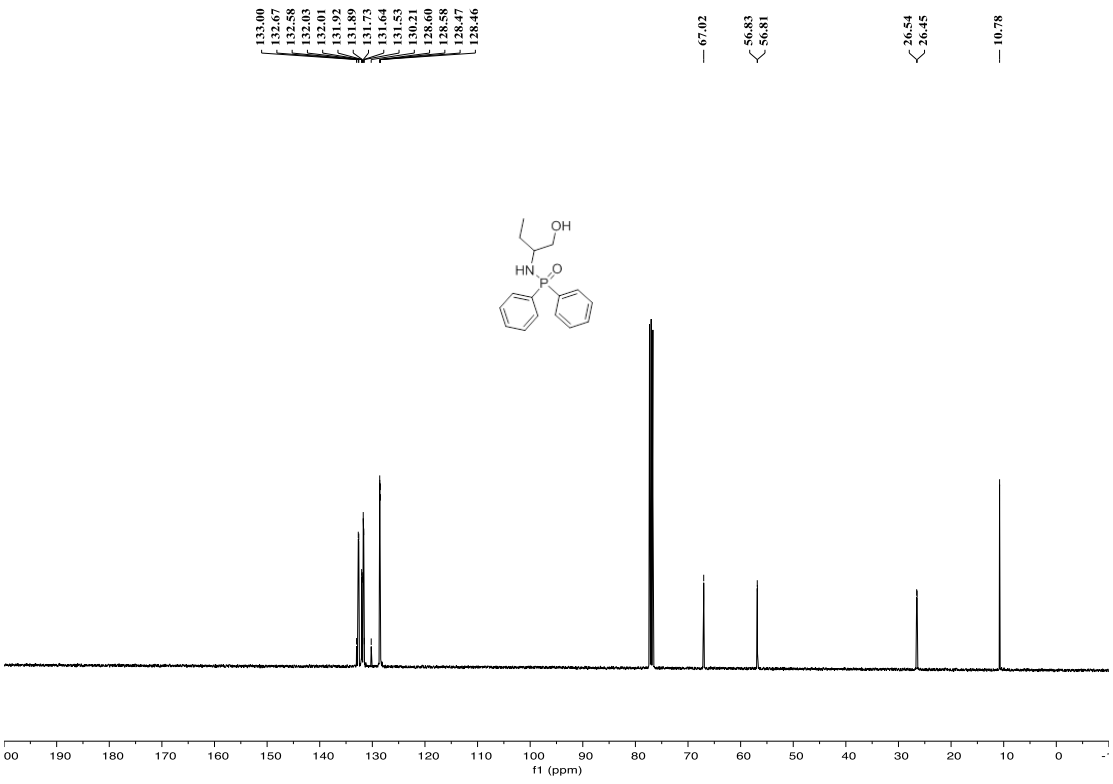
6.00
1.05
0.99
1.02
0.88
1.00
1.97
3.01
f1 (ppm)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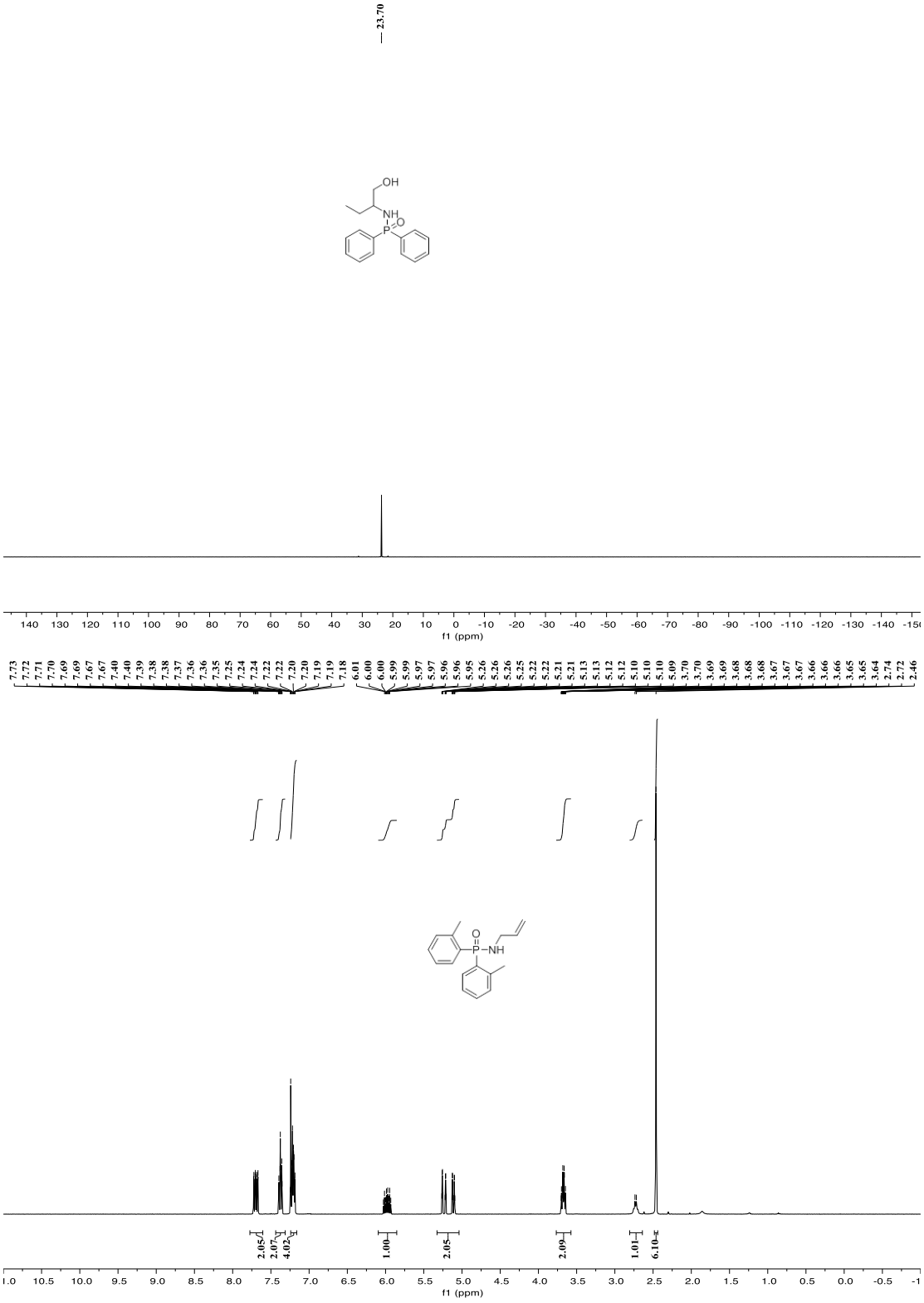
67.02
56.83 56.81
26.54 26.45
10.78
OH
HN
P
O
f1 (ppm)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OH
NH
O
P
f1 (ppm)
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6.01 6.00 6.00 5.99 5.99 5.97 5.97 5.96 5.96 5.95 5.26 5.26 5.25 5.22 5.22 5.21 5.21 5.13 5.13 5.12 5.12 5.10 5.10 5.10 5.09 3.70 3.70 3.69 3.69 3.68 3.68 3.68 3.67 3.67 3.67 3.66 3.66 3.66 3.65 3.65 3.64 2.74 2.72 2.46
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P
NH
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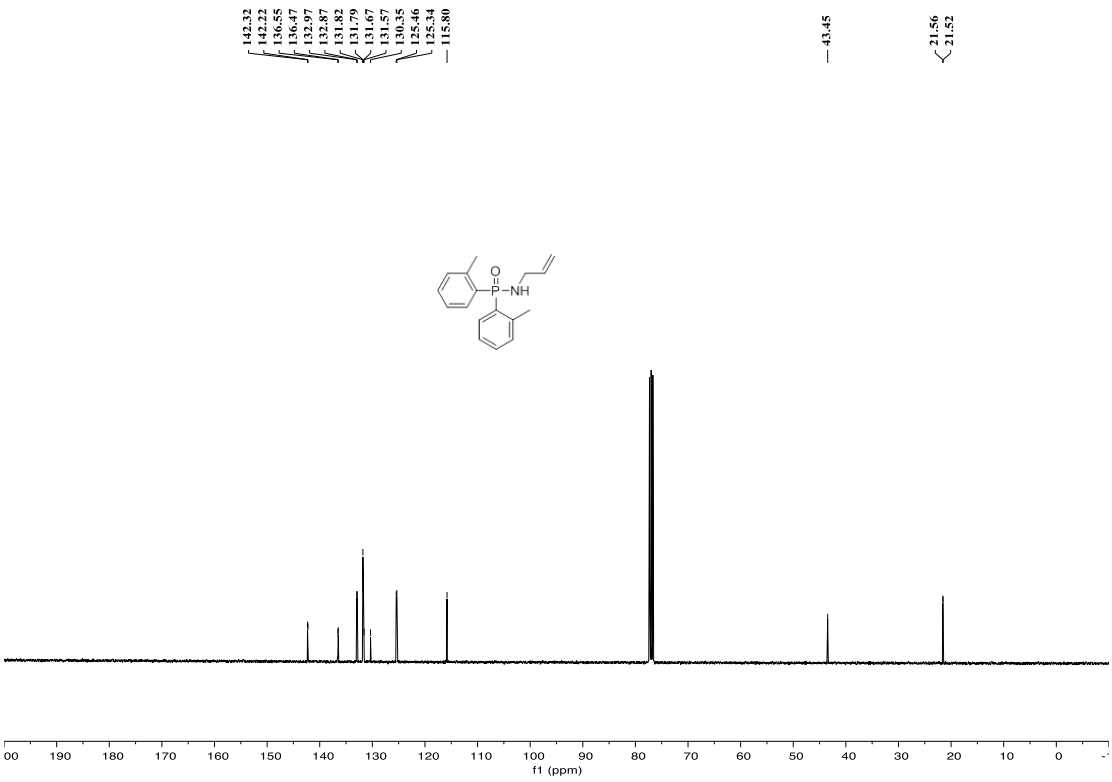
4.02
1.00
2.05
2.09
1.01
6.10
f1 (ppm)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132.87
131.82
131.79
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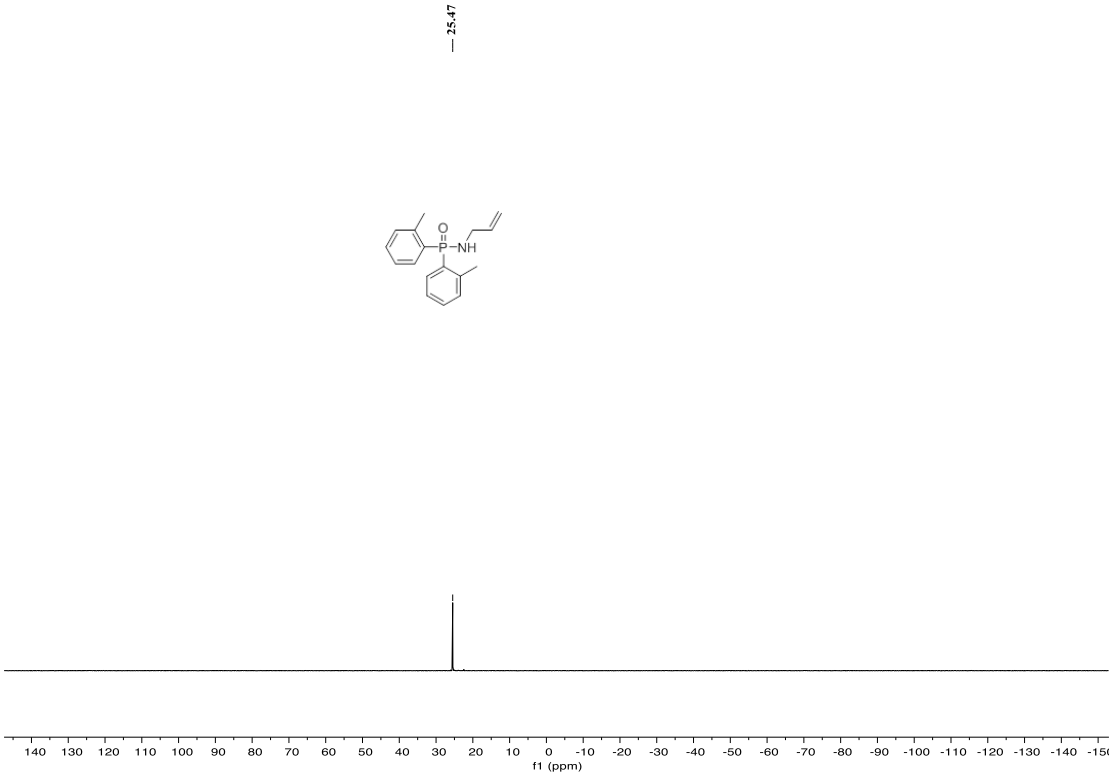
131.57
130.35
125.46
125.34
115.80
43.45
21.56
21.52
O
P
NH
f1 (ppm)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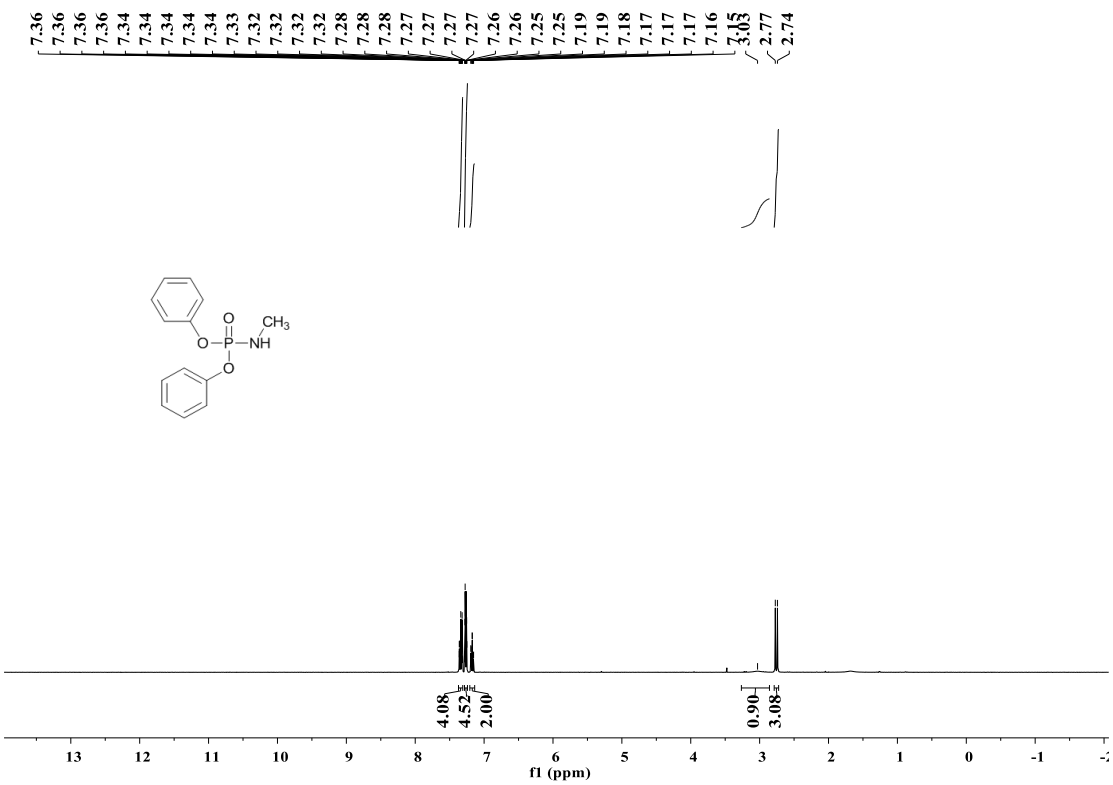
NH
f1 (ppm)


7.36
7.36
7.36
7.34
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7.33
7.32
7.32
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7.16
7.15
3.03
2.77
2.74
O
O
P
NH
CH3
O
4.08
4.52
2.00
0.90
3.08
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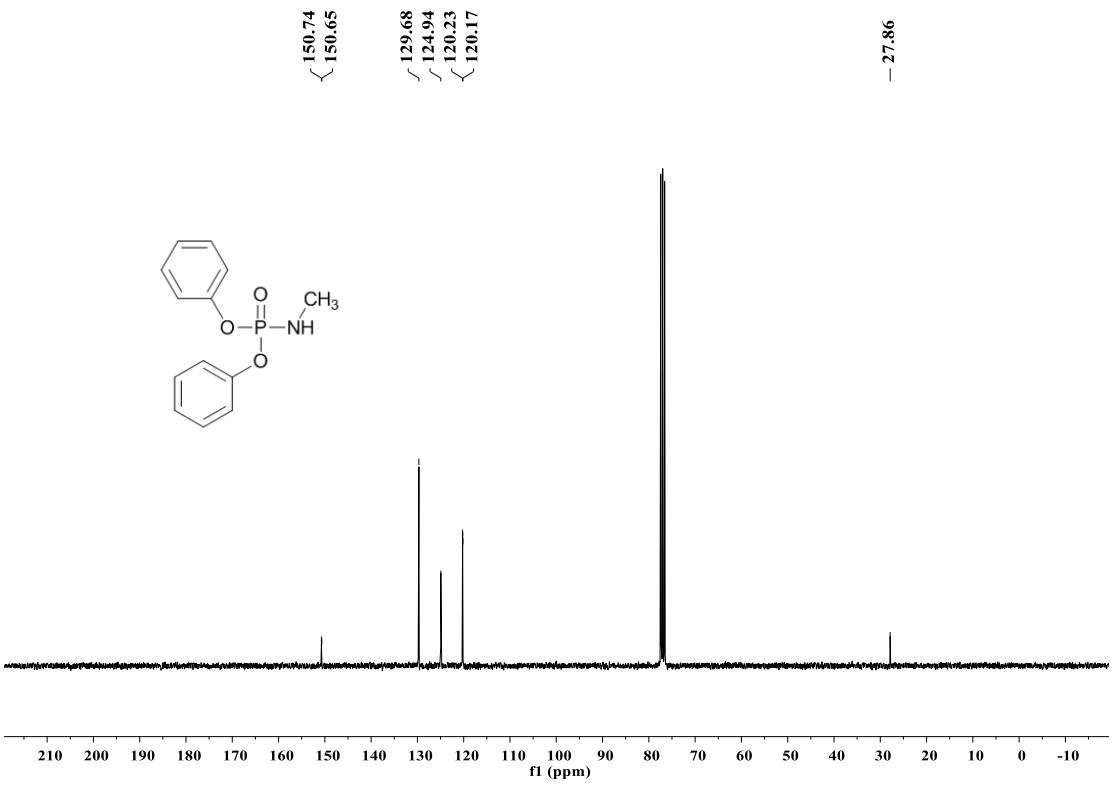
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)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124.94
120.23
120.17
27.86
O
O
P
NH
CH3
O
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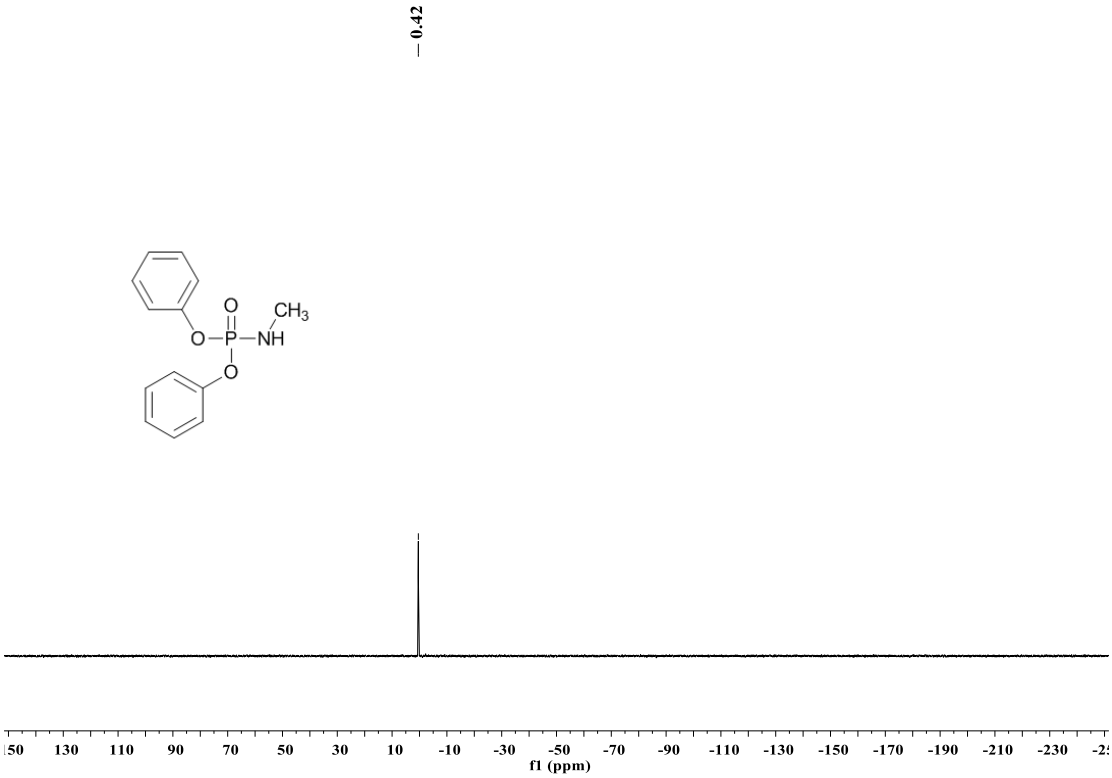
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


— 0.42
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CH3
O—P—NH
O
f1 (ppm)
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7.29
7.25
7.24
7.23
7.16
7.15
7.13
3.07
3.06
3.04
3.03
3.02
3.01
3.00
2.96
1.53
1.51
1.49
1.47
1.46
1.44
0.88
0.86
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NH
4.09
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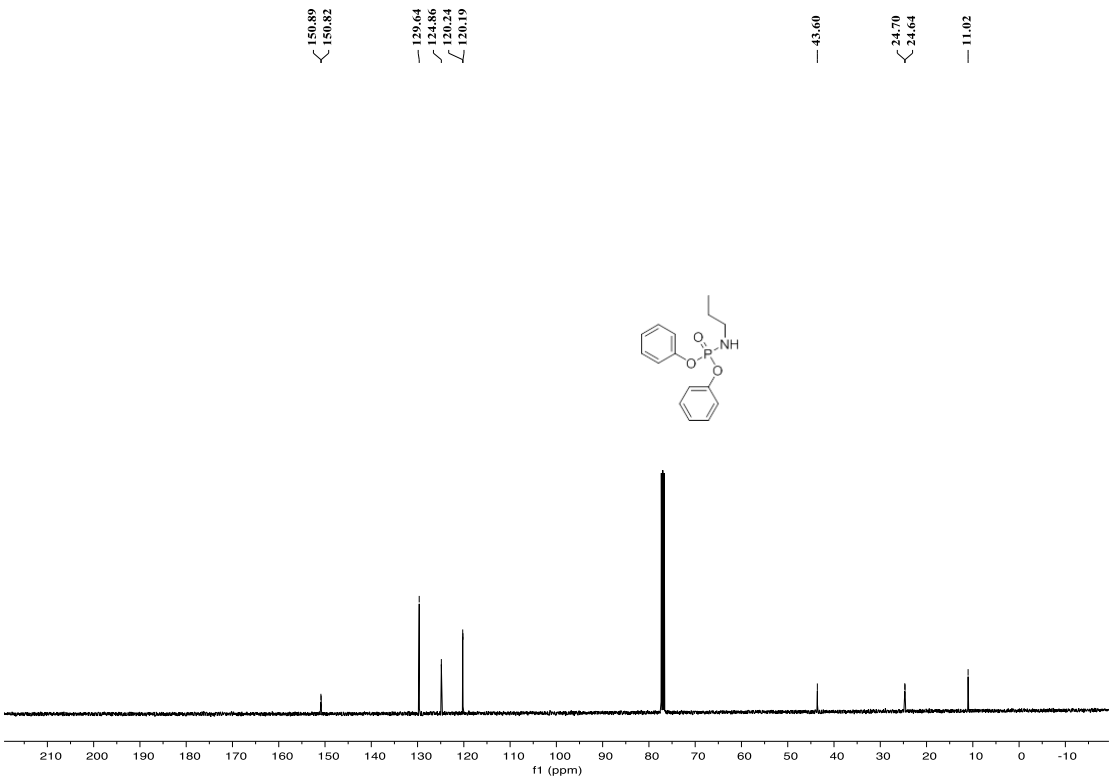
1.20
2.00
2.07
0.93
2.11
3.11
f1 (ppm)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24.64
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O
P
O
O
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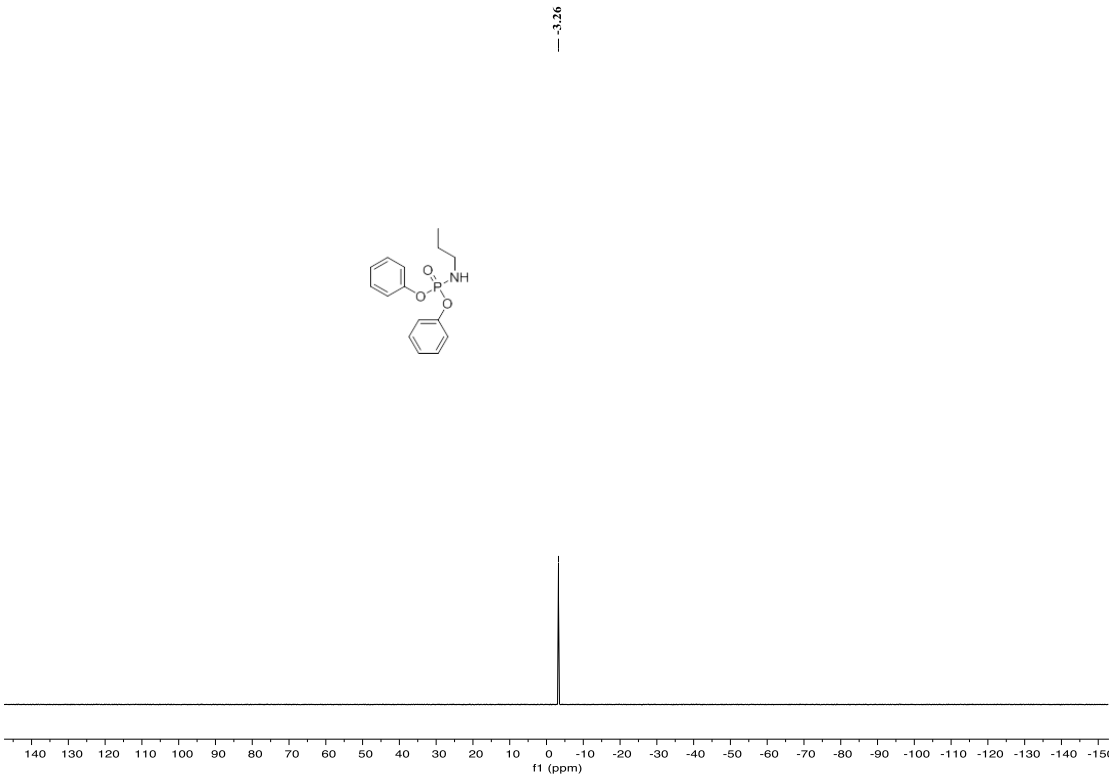
f1 (ppm)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O
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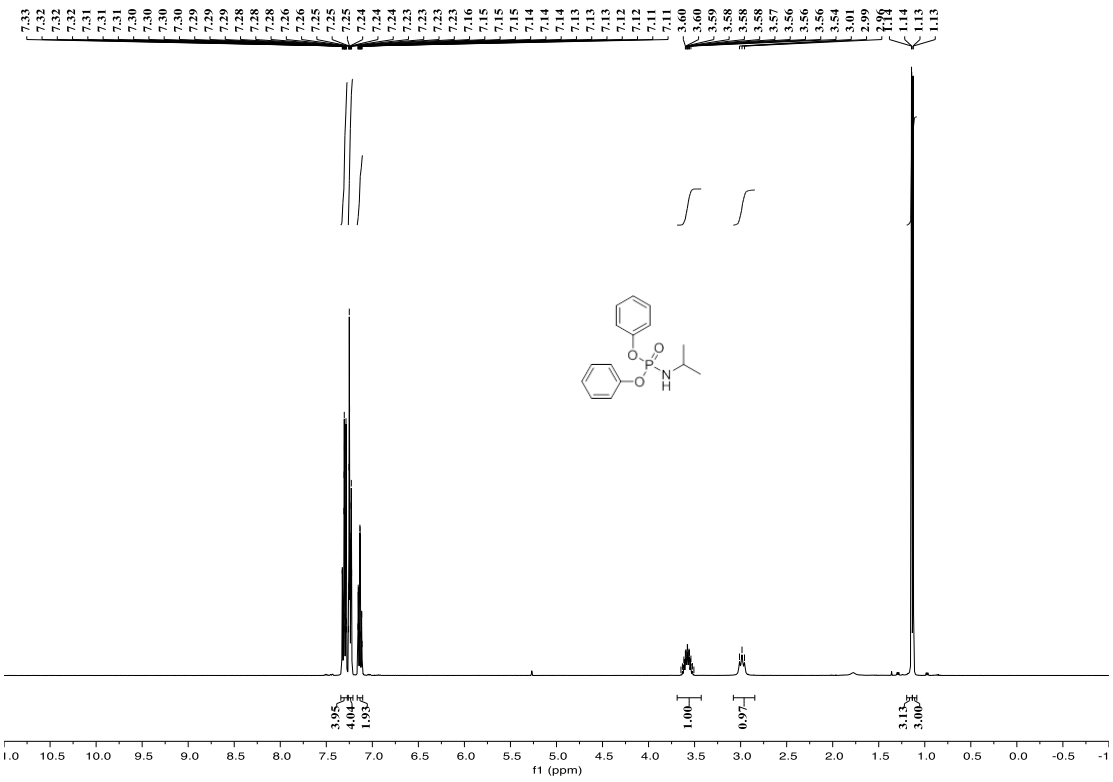
f1 (ppm)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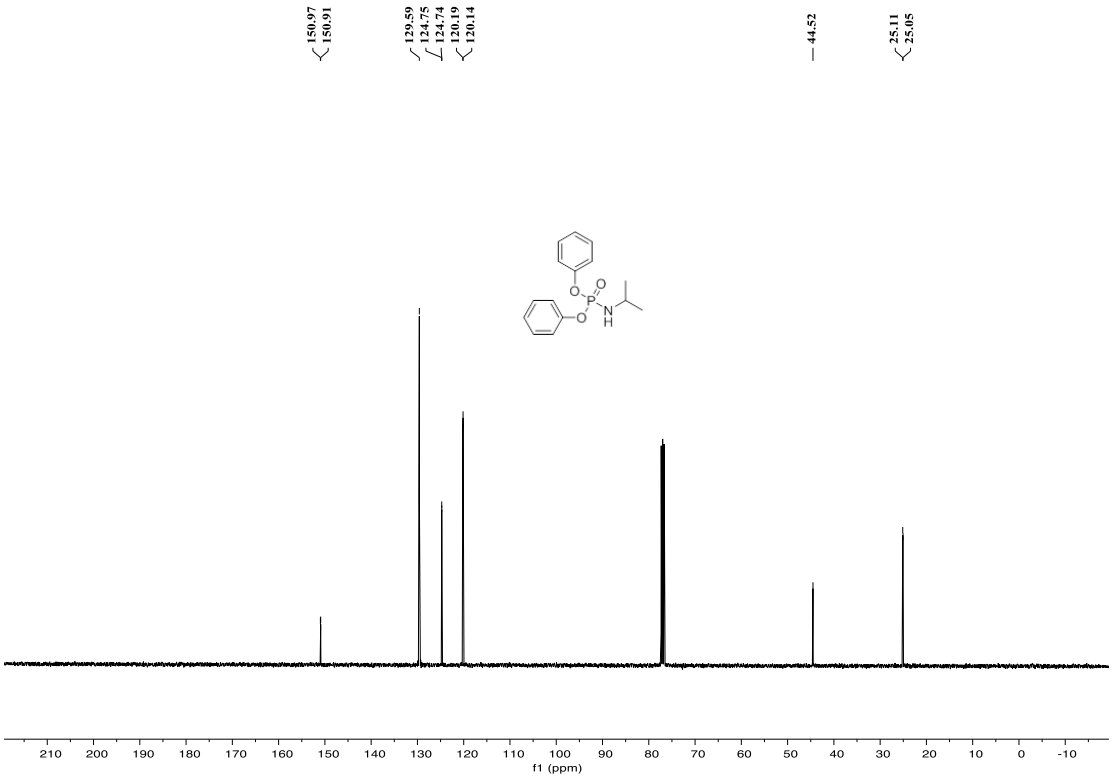
3.60 3.59 3.58 3.58 3.58 3.57 3.56 3.56 3.56 3.54 3.01 2.99 2.96 1.14 1.14 1.13 1.13
3.95 4.04 1.93 1.00 0.97 3.13 3.00
f1 (ppm)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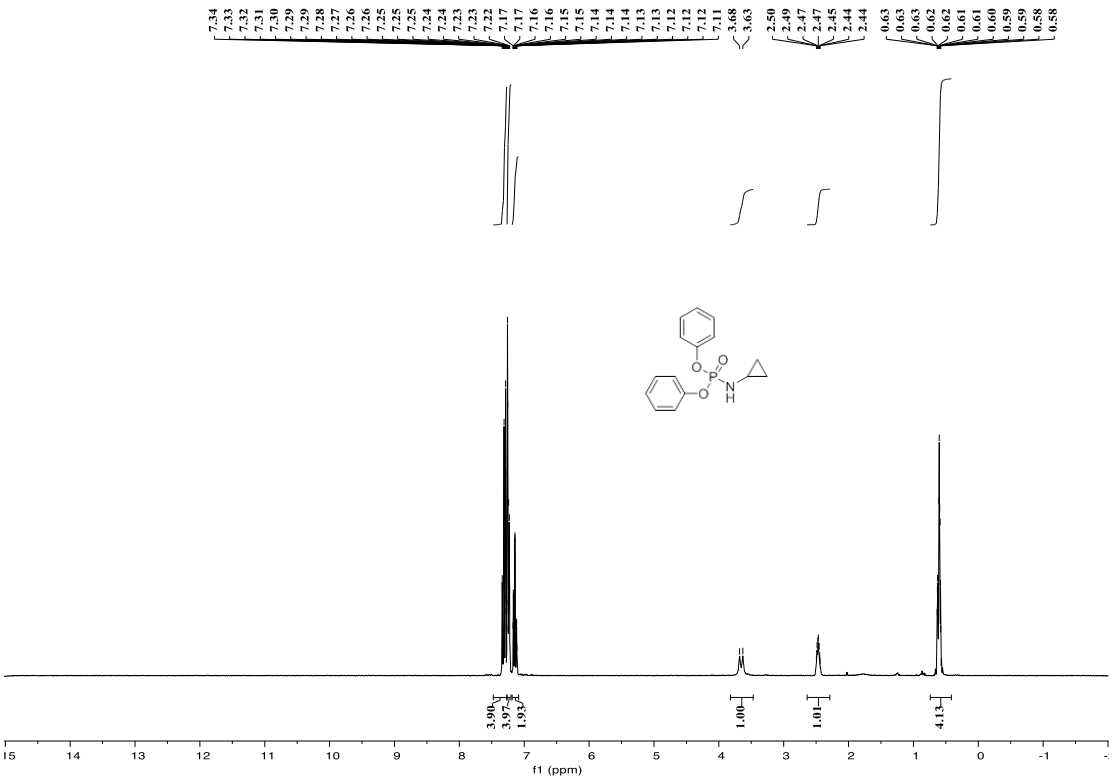
f1 (ppm)


-4.60
f1 (ppm)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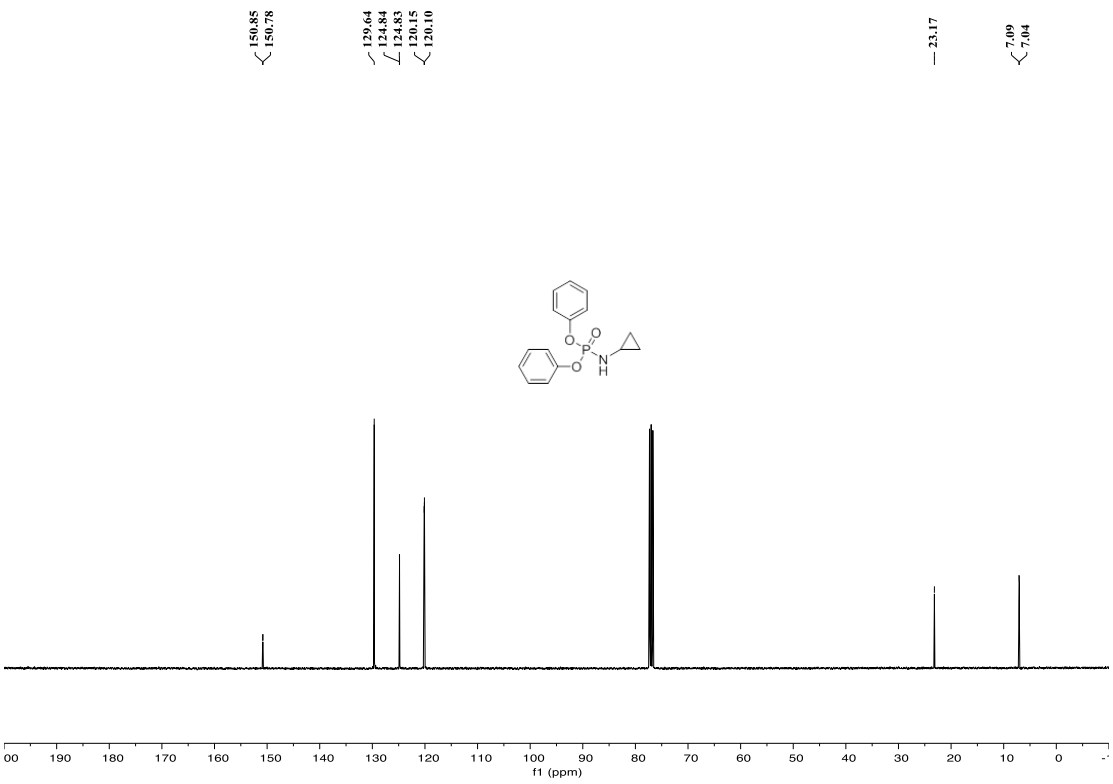
3.68 3.63
2.50 2.49 2.47 2.47 2.45 2.44 2.44
0.63 0.63 0.63 0.62 0.62 0.61 0.61 0.60 0.59 0.59 0.58 0.58
3.90 3.97 1.93 1.00 1.01 4.13
f1 (ppm)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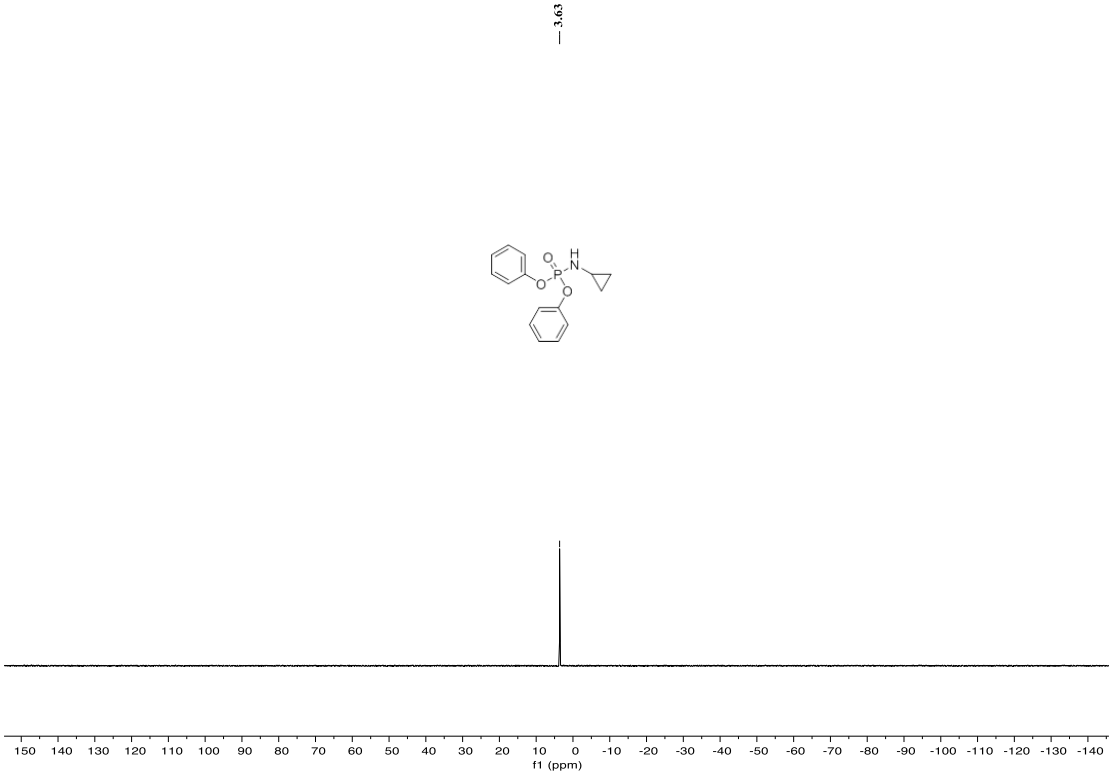
23.17
7.09
7.04
f1 (ppm)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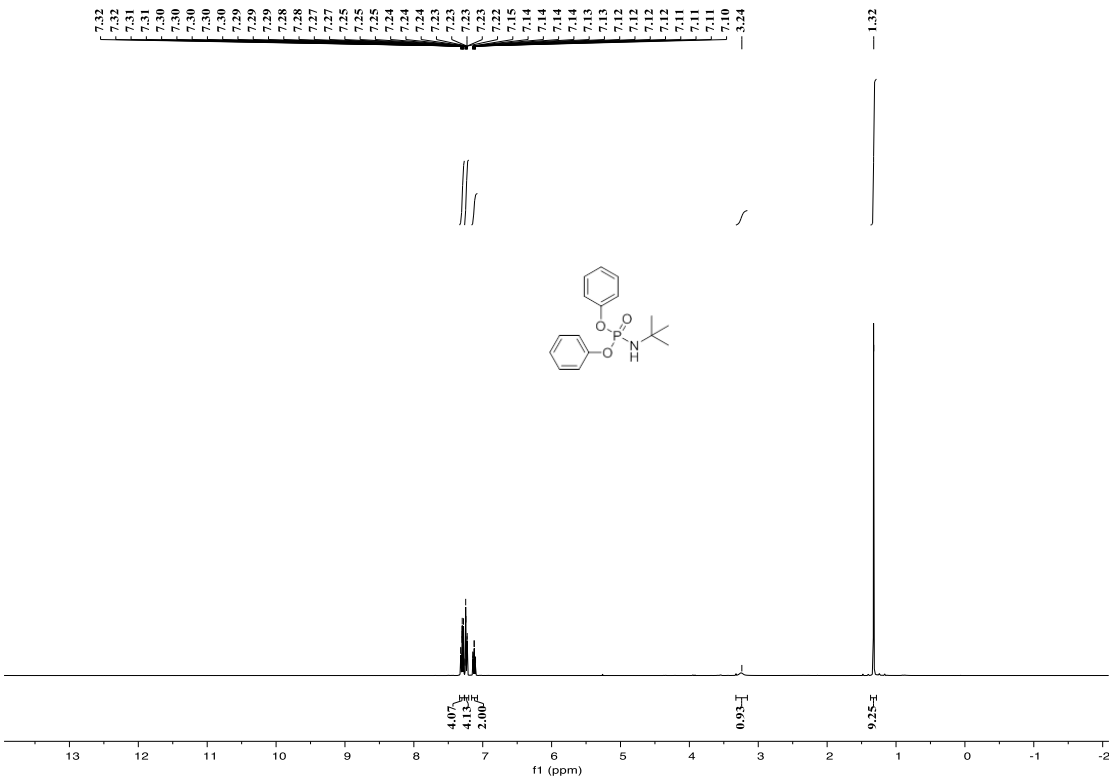
7.32
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7.12
7.12
7.12
7.11
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7.10
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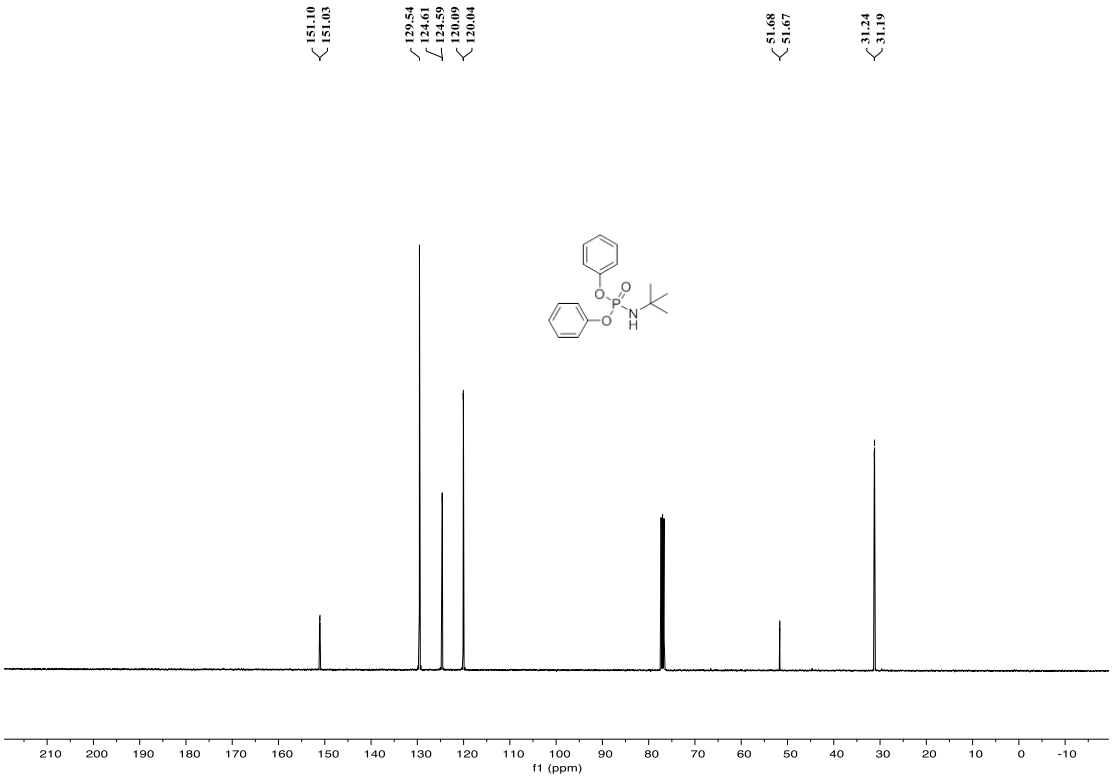
4.13
2.00
0.93
9.25
f1 (ppm)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151.03
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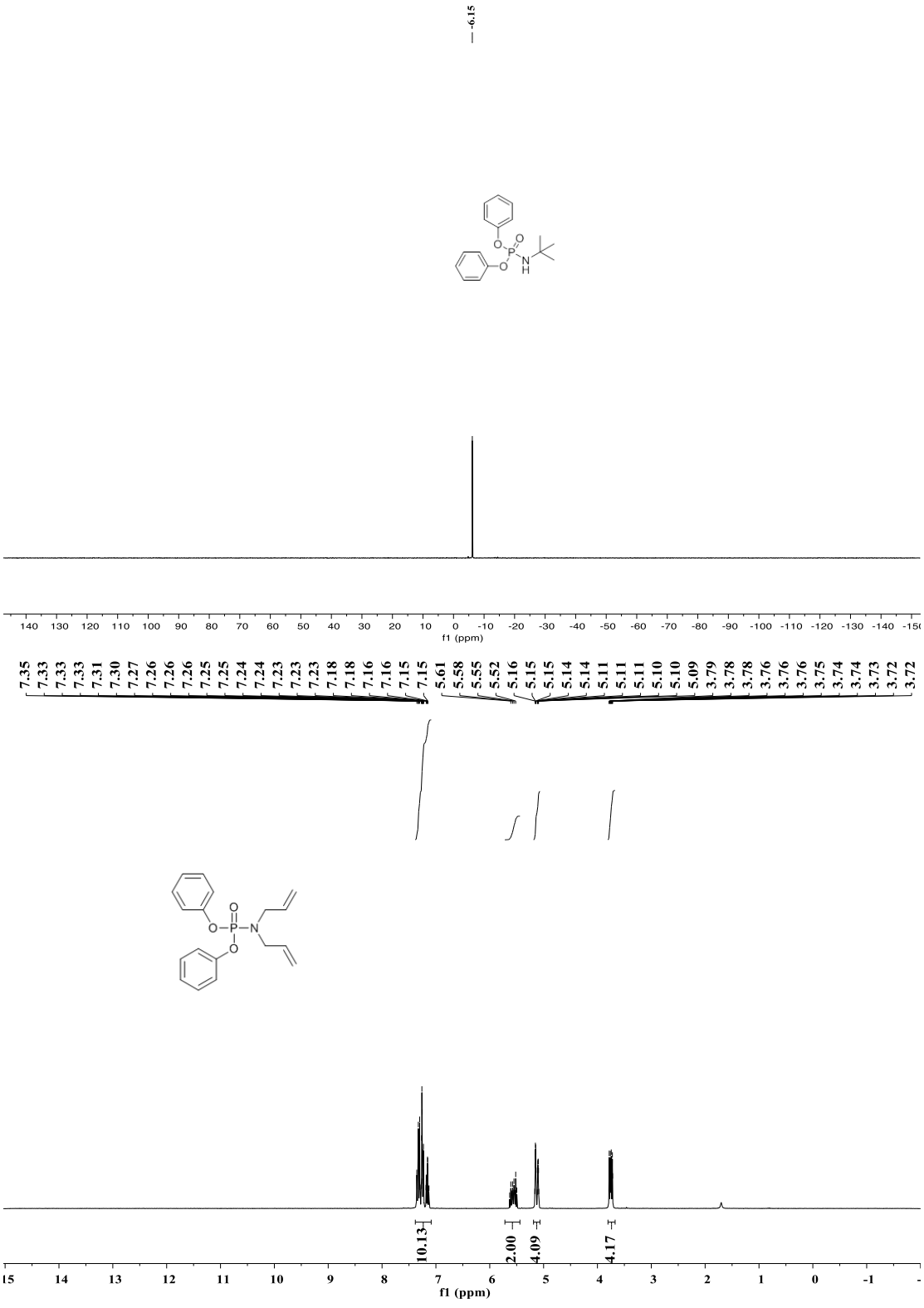
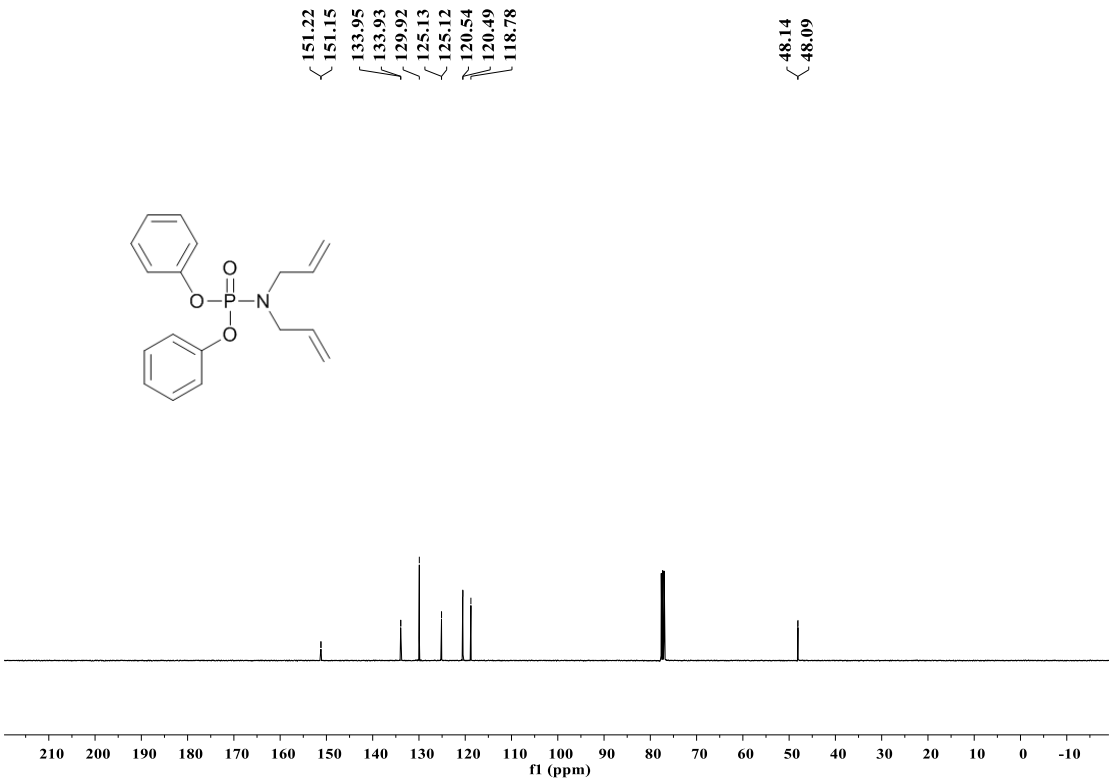
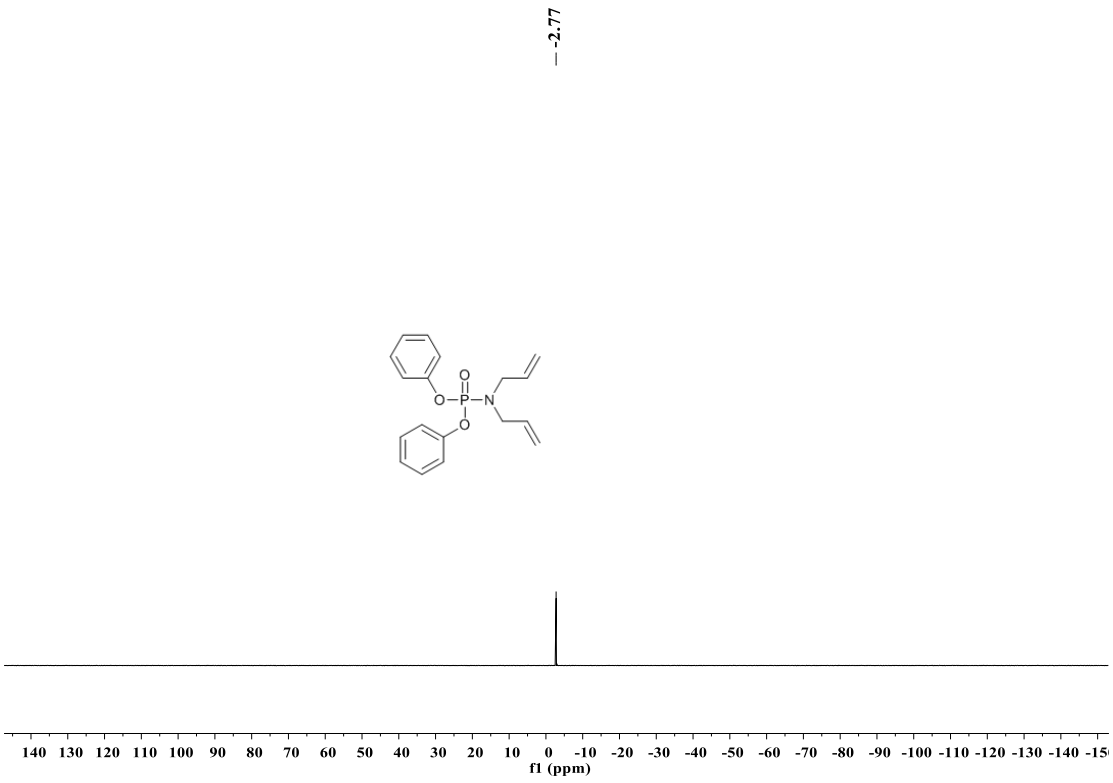
124.59
120.09
120.04
51.68
51.67
31.24
31.19
f1 (ppm)


6.15
f1 (ppm)
7.35 7.33 7.33 7.33 7.31 7.30 7.27 7.26 7.26 7.26 7.25 7.25 7.24 7.24 7.23 7.23 7.23 7.18 7.18 7.16 7.16 7.15 7.15 7.15
5.61 5.58 5.55 5.52 5.16 5.15 5.15 5.14 5.14 5.11 5.11 5.11 5.10 5.10 5.09 3.79 3.78 3.78 3.76 3.76 3.76 3.75 3.74 3.74 3.73 3.72 3.72
10.13
2.00
4.09
4.17
f1 (ppm)


151.22
151.15
133.95
133.93
129.92
125.13
125.12
120.54
120.49
118.78
48.14
48.09
O
O
P
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-2.77
O
O
P
N
O
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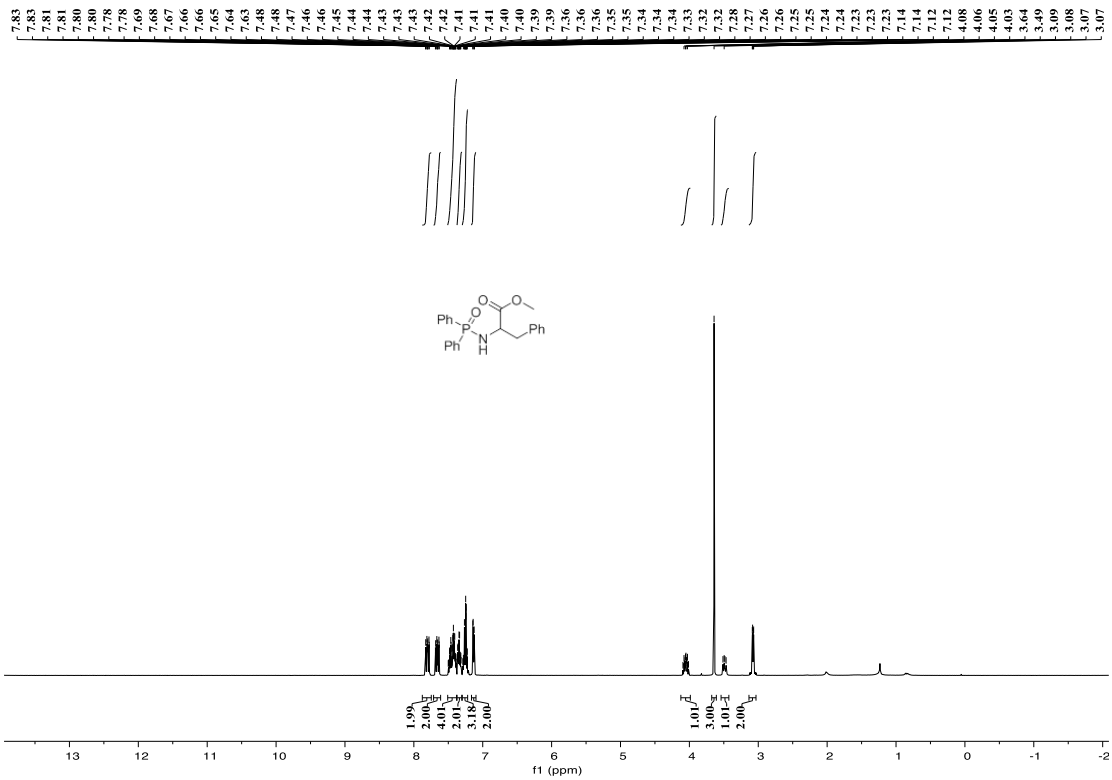
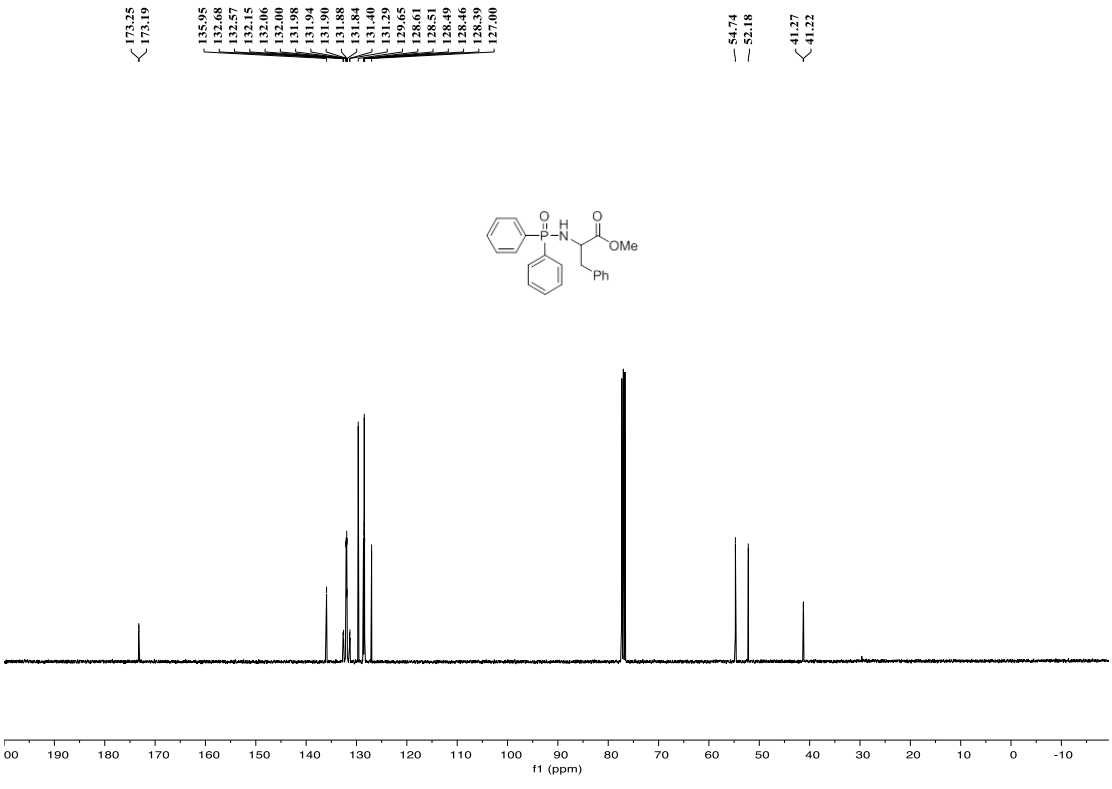
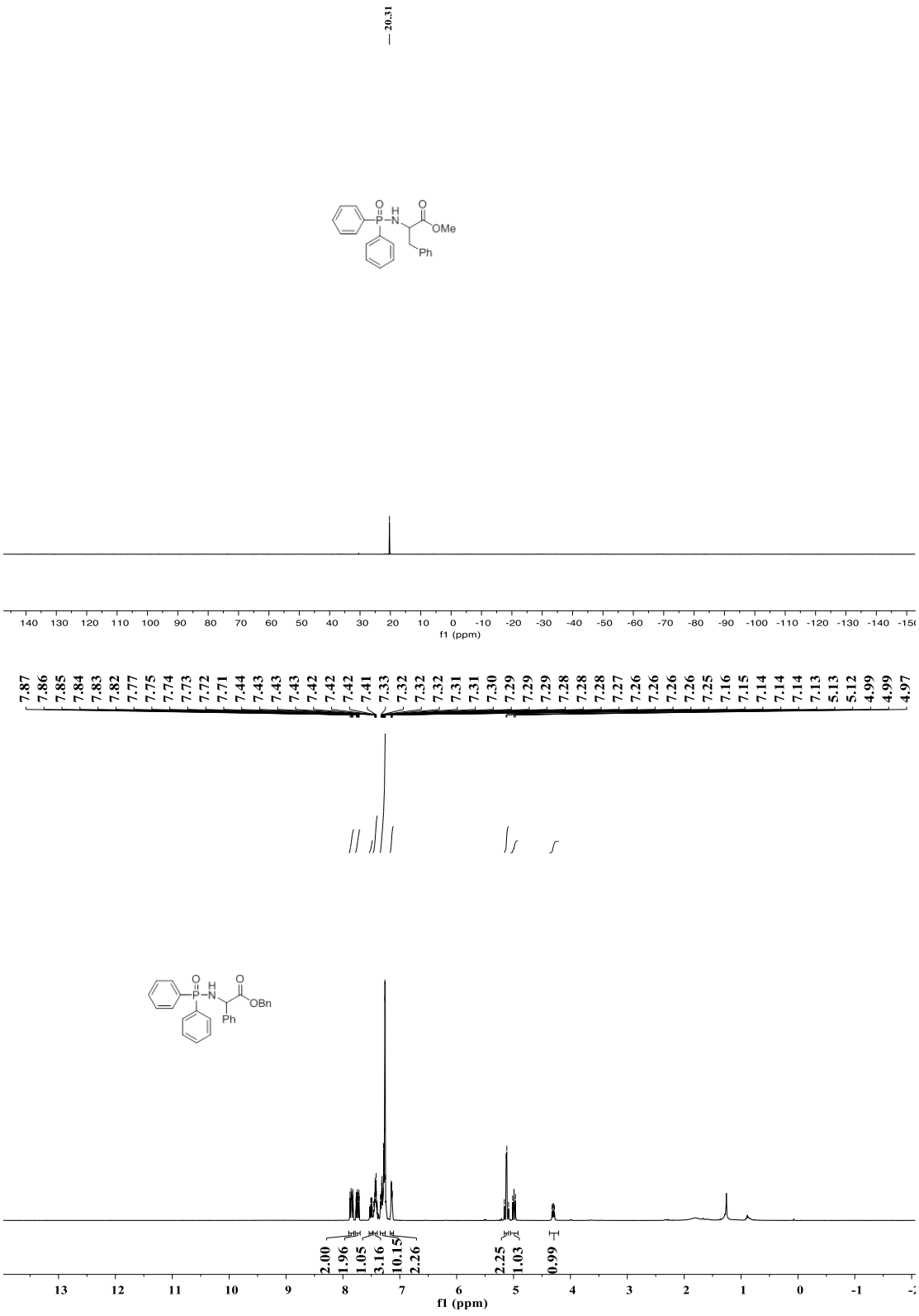
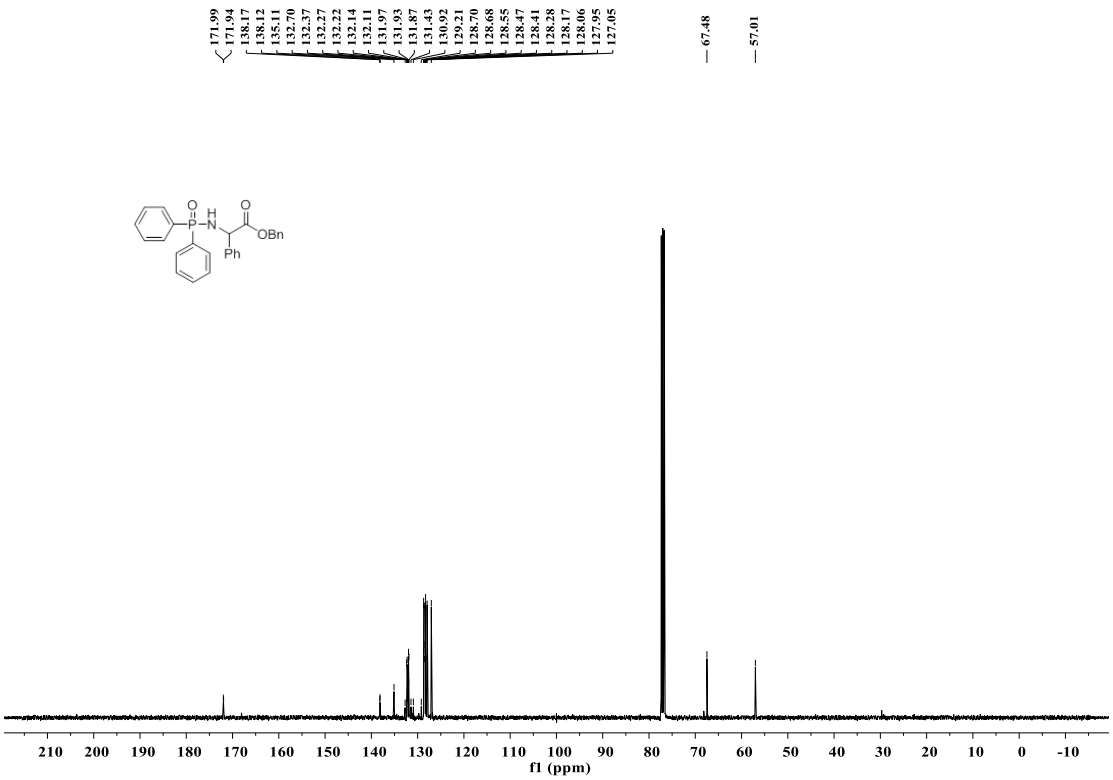
f1 (ppm)


7.83 7.83 7.81 7.81 7.80 7.80 7.78 7.78 7.69 7.68 7.67 7.66 7.66 7.65 7.64 7.63 7.48 7.48 7.47 7.46 7.46 7.45 7.44 7.44 7.43 7.43 7.42 7.42 7.41 7.41 7.41 7.40 7.40 7.39 7.39 7.36 7.36 7.36 7.35 7.35 7.34 7.34 7.34 7.33 7.32 7.28 7.27 7.26 7.26 7.25 7.25 7.24 7.24 7.23 7.23 7.14 7.14 7.12 4.08 4.06 4.05 4.03 3.64 3.49 3.09 3.08 3.07 3.07
Ph
P
O
O
O
N
H
Ph
Ph
1.99 2.00 4.01 2.01 3.18 2.00 1.01 3.00 1.01 2.00
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2
f1 (ppm)


173.25 173.19 135.95 132.68 132.57 132.15 132.06 132.00 131.98 131.94 131.90 131.88 131.84 131.40 131.29 129.65 128.61 128.51 128.49 128.46 128.39 127.00 54.74 52.18 41.27 41.22
O
P
H
N
O
OMe
Ph
00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


20.31
OMe
Ph
140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140
f1 (ppm)
7.87 7.86 7.85 7.84 7.83 7.82 7.77 7.75 7.74 7.73 7.72 7.71 7.44 7.43 7.43 7.43 7.42 7.42 7.42 7.41 7.33 7.32 7.32 7.32 7.31 7.31 7.30 7.29 7.29 7.29 7.28 7.28 7.28 7.27 7.26 7.26 7.26 7.25 7.16 7.15 7.14 7.14 7.14 7.13 5.13 5.12 4.99 4.99 4.97
OBn
Ph
2.00 1.96 1.05 3.16 10.15 2.26 2.25 1.03 0.99
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)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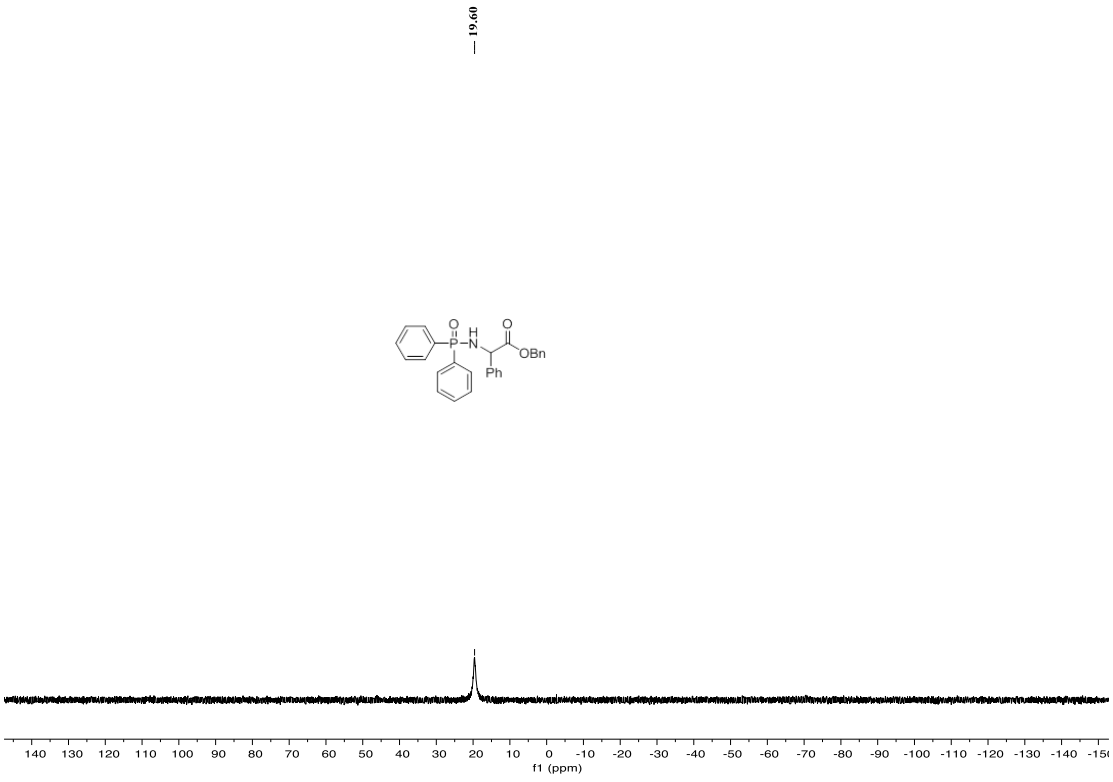
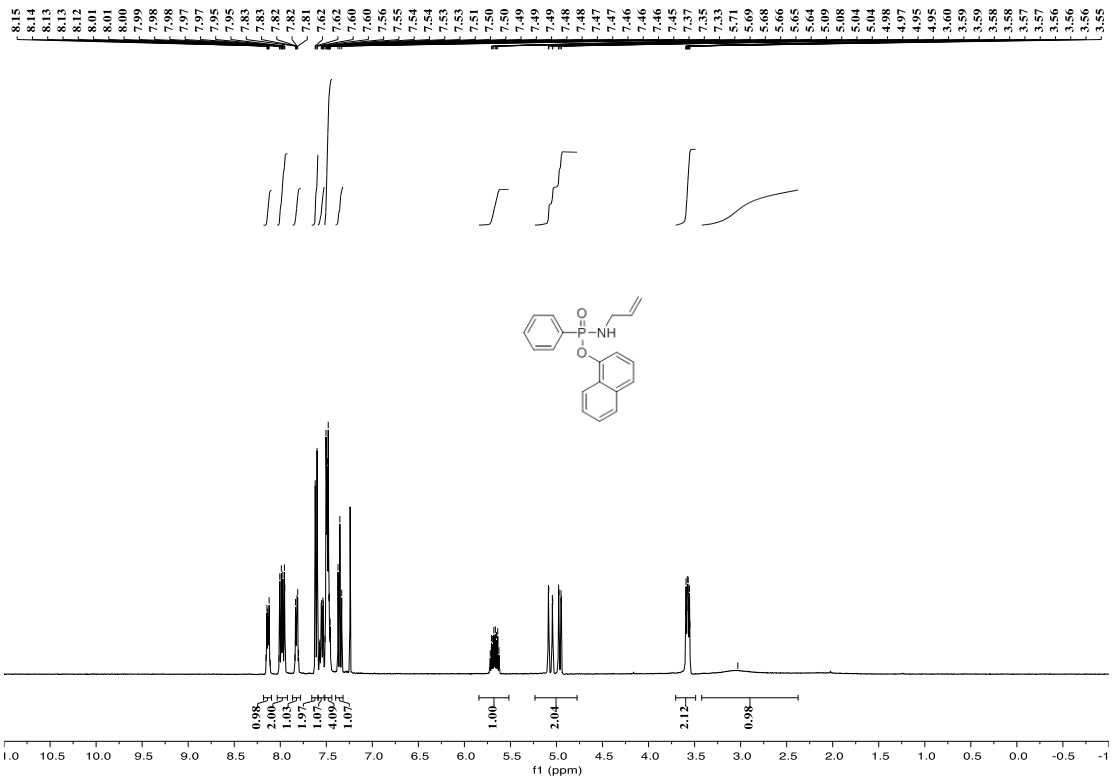
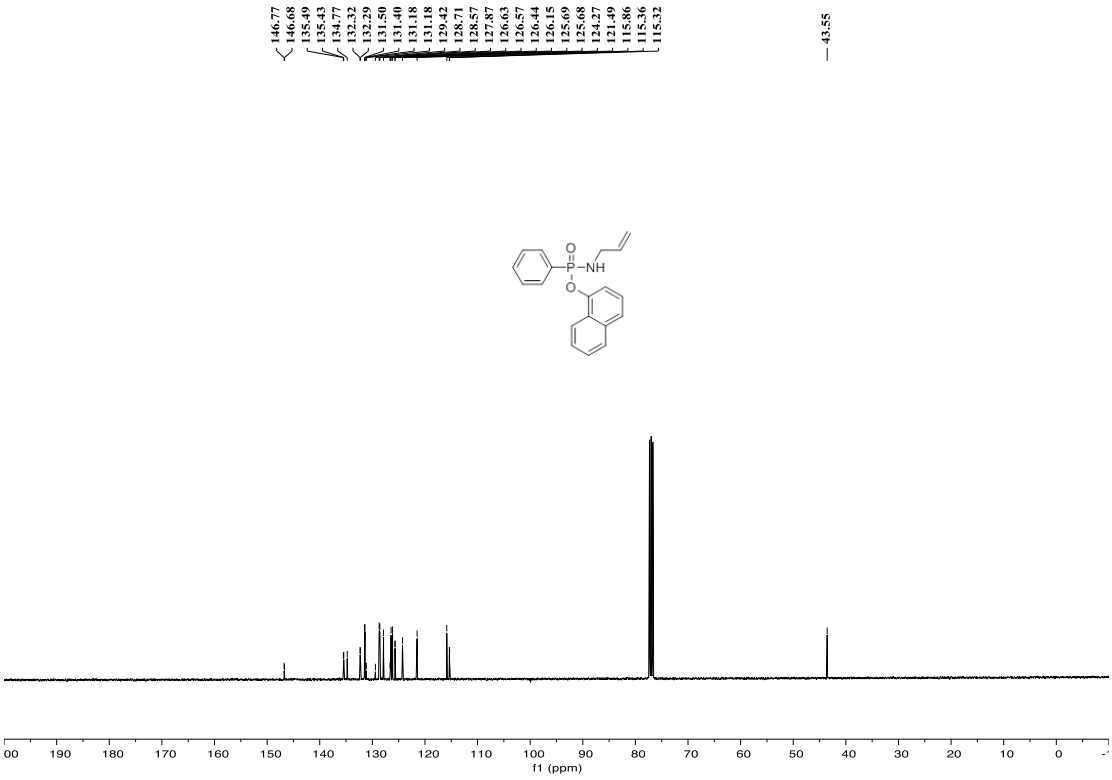
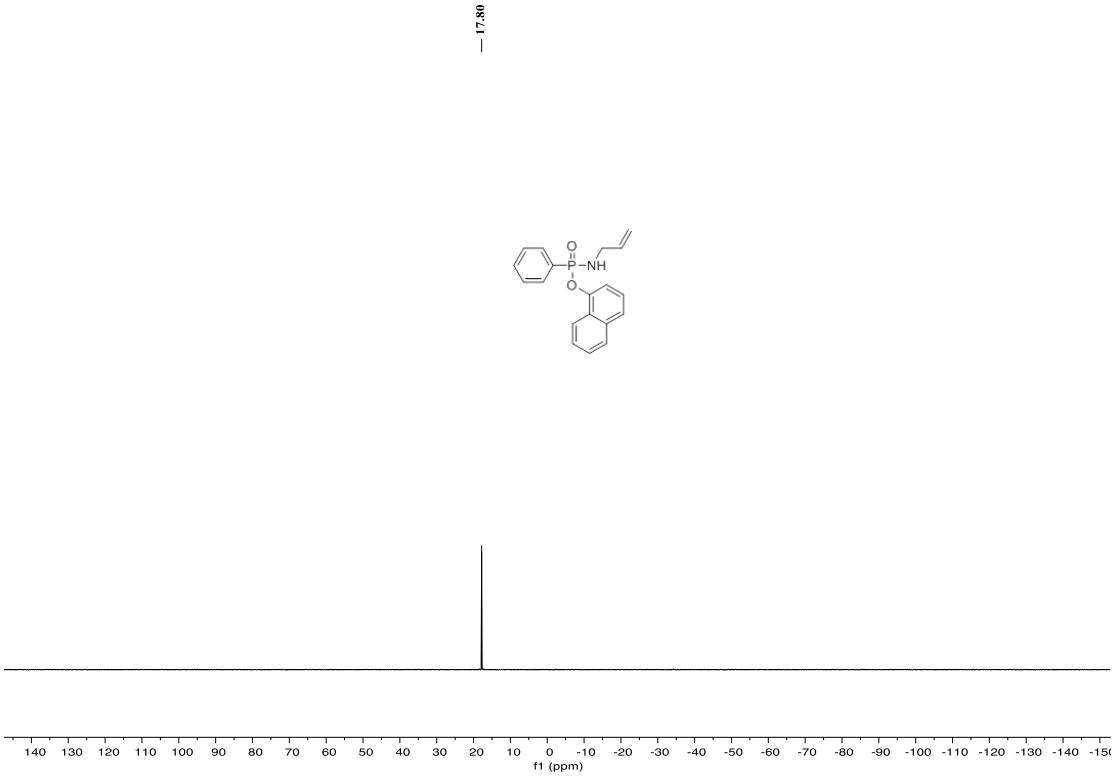
171.94
138.17
138.12
135.11
132.70
132.37
132.27
132.22
132.14
132.11
131.97
131.93
131.87
131.43
130.92
129.21
128.70
128.68
128.55
128.47
128.41
128.28
128.17
128.06
127.95
127.05
67.48
57.01
OBn
Ph
f1 (ppm)


19.60
OBn
Ph
f1 (ppm)


8.15 8.14 8.13 8.13 8.12 8.01 8.01 8.00 7.99 7.98 7.98 7.97 7.97 7.95 7.95 7.83 7.82 7.82 7.81 7.62 7.62 7.60 7.60 7.56 7.55 7.54 7.54 7.53 7.53 7.51 7.50 7.50 7.49 7.49 7.49 7.48 7.48 7.47 7.47 7.46 7.46 7.46 7.45 7.37 7.35 7.33 5.71 5.69 5.68 5.66 5.65 5.64 5.09 5.08 5.04 5.04 4.98 4.97 4.95 4.95 3.60 3.59 3.59 3.58 3.58 3.57 3.57 3.56 3.56 3.56 3.55
O
P
NH
O
0.98 2.00 1.03 1.97 1.07 4.09 1.07 1.00 2.04 2.12 0.98
f1 (ppm)


146.77 146.68 135.49 135.43 134.77 132.32 132.29 131.50 131.40 131.18 131.18 129.42 128.71 128.57 127.87 126.63 126.57 126.44 126.15 125.69 125.68 124.27 121.49 115.86 115.36 115.32
43.55
O
P
NH
O
f1 (ppm)


17.80
O
P
NH
O
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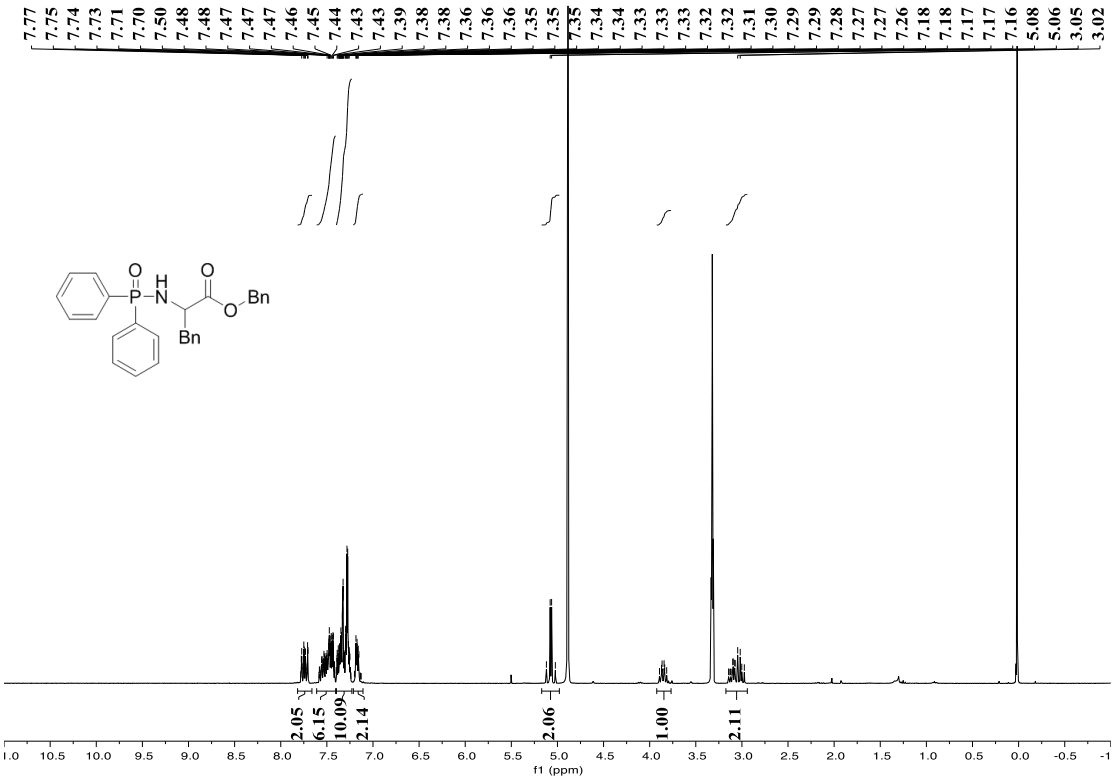
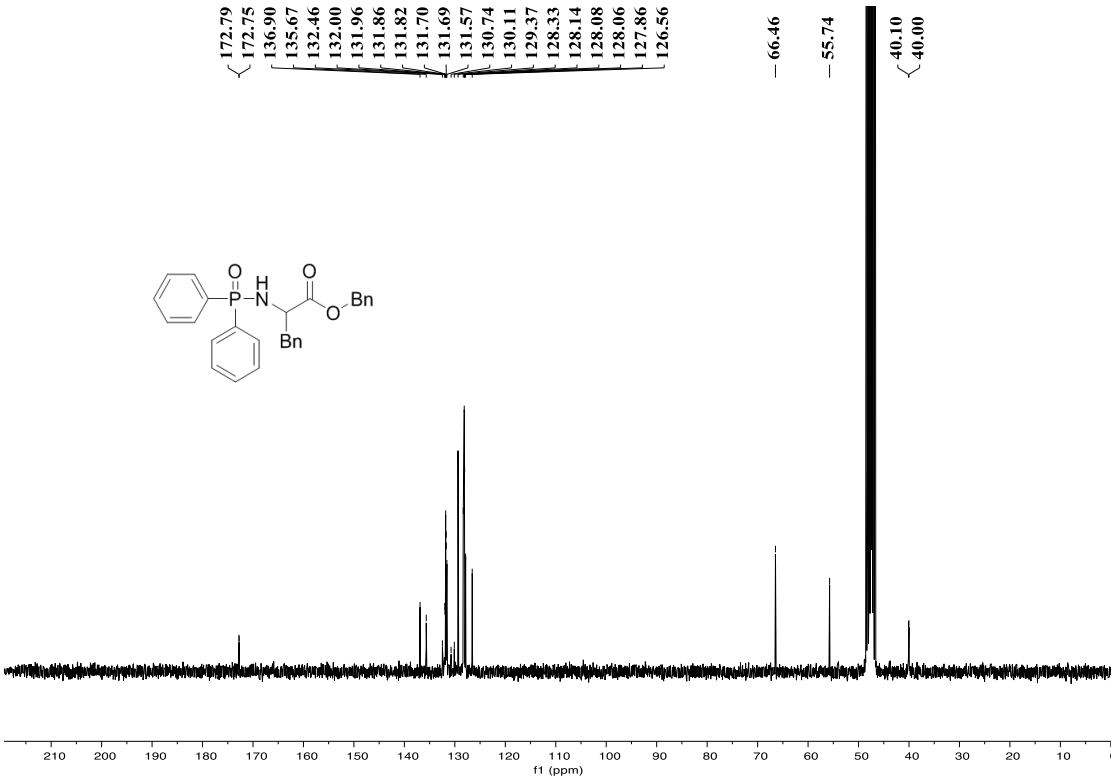
f1 (ppm)


7.77 7.75 7.74 7.73 7.71 7.70 7.50 7.48 7.48 7.47 7.47 7.47 7.46 7.45 7.44 7.43 7.43 7.39 7.38 7.38 7.36 7.36 7.36 7.35 7.35 7.35 7.34 7.34 7.33 7.33 7.33 7.32 7.32 7.31 7.30 7.29 7.29 7.28 7.27 7.27 7.26 7.18 7.18 7.17 7.17 7.16 5.08 5.06 3.05 3.02
O
H
O
P
N
O
Bn
Bn
2.05
6.15
10.09
2.14
2.06
1.00
2.11
f1 (ppm)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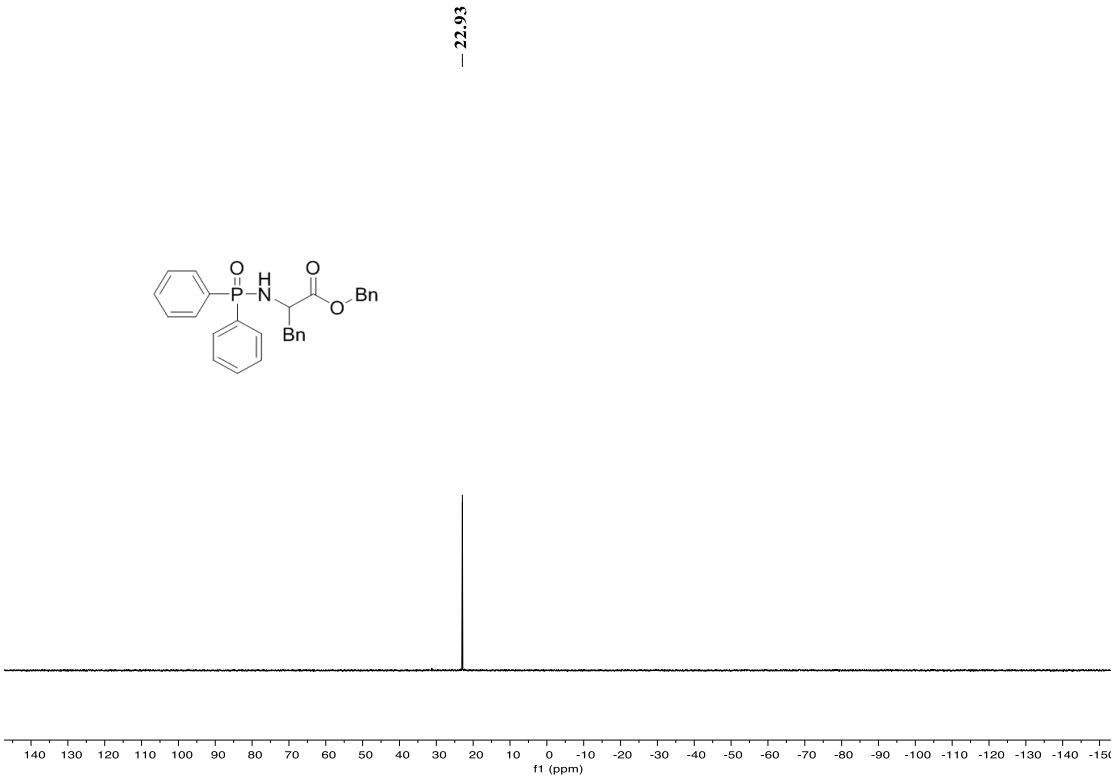
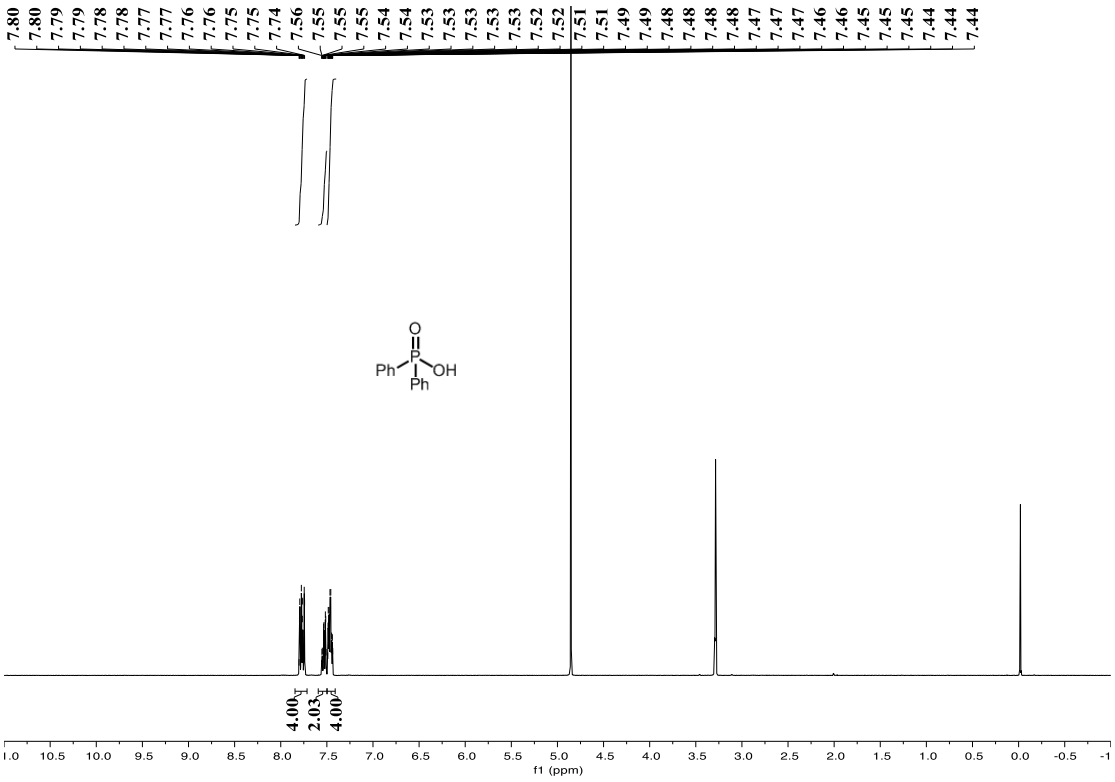
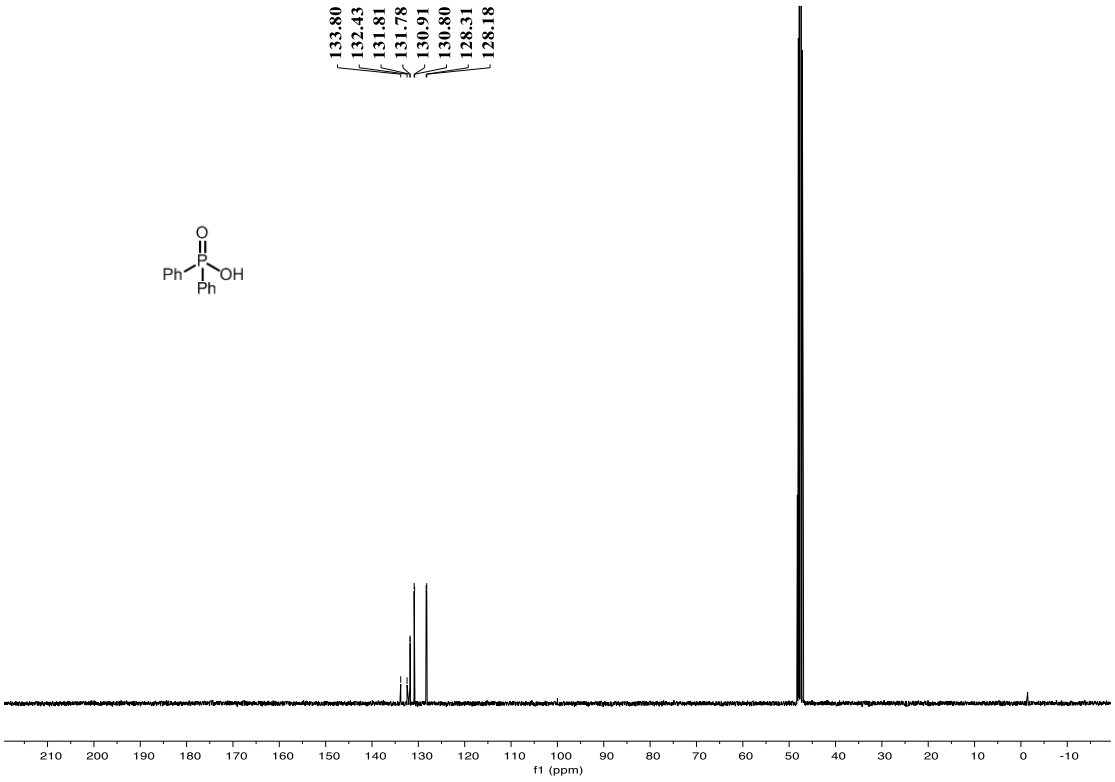
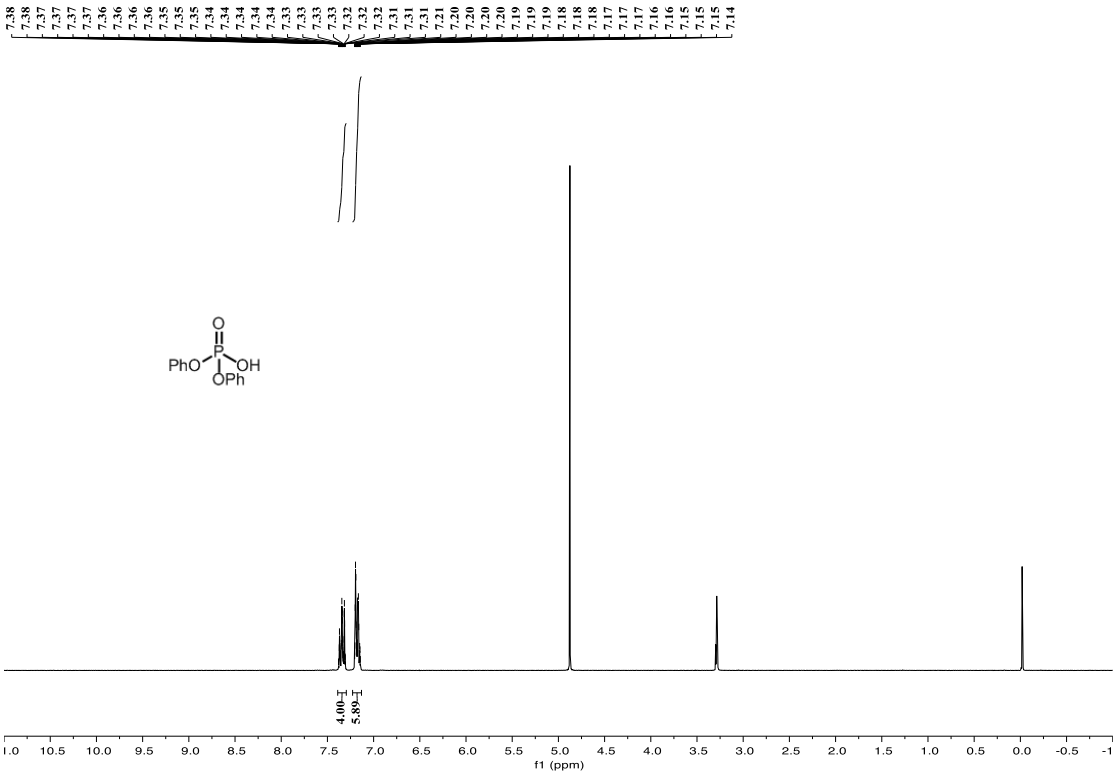
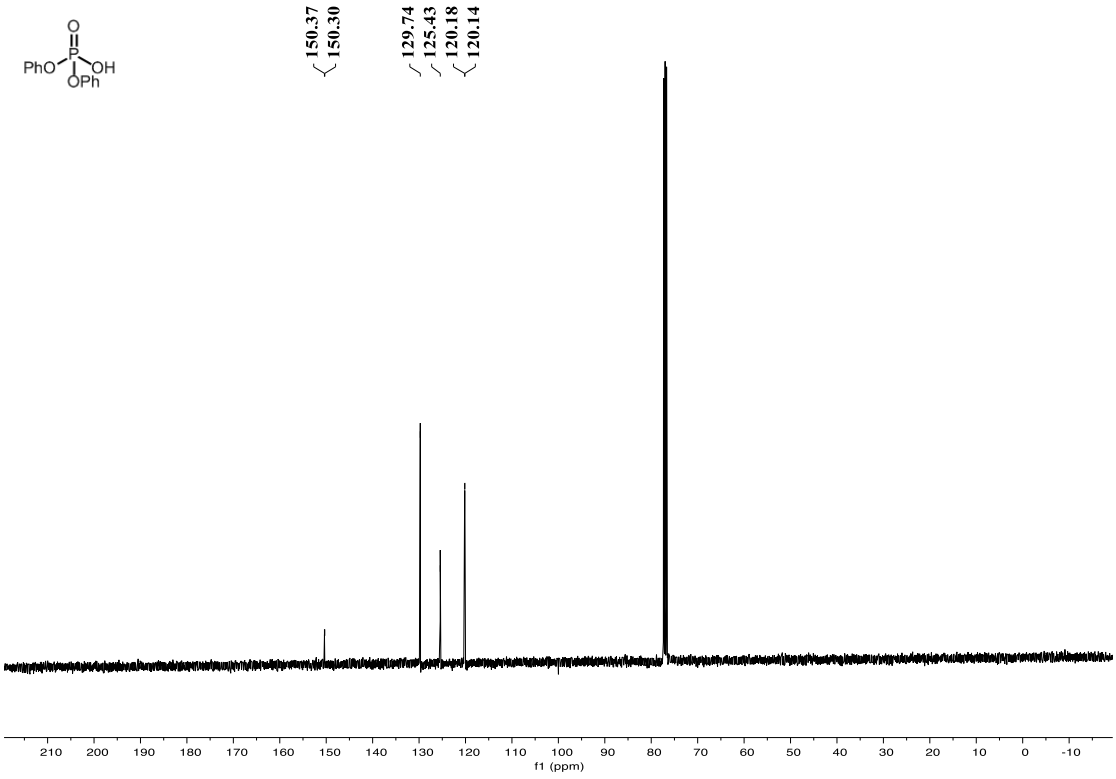
172.75
136.90
135.67
132.46
132.00
131.96
131.86
131.82
131.70
131.69
131.57
130.74
130.11
129.37
128.33
128.14
128.08
128.06
127.86
126.56
66.46
55.74
40.10
40.00
O
H
P
N
O
O
Bn
Bn
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

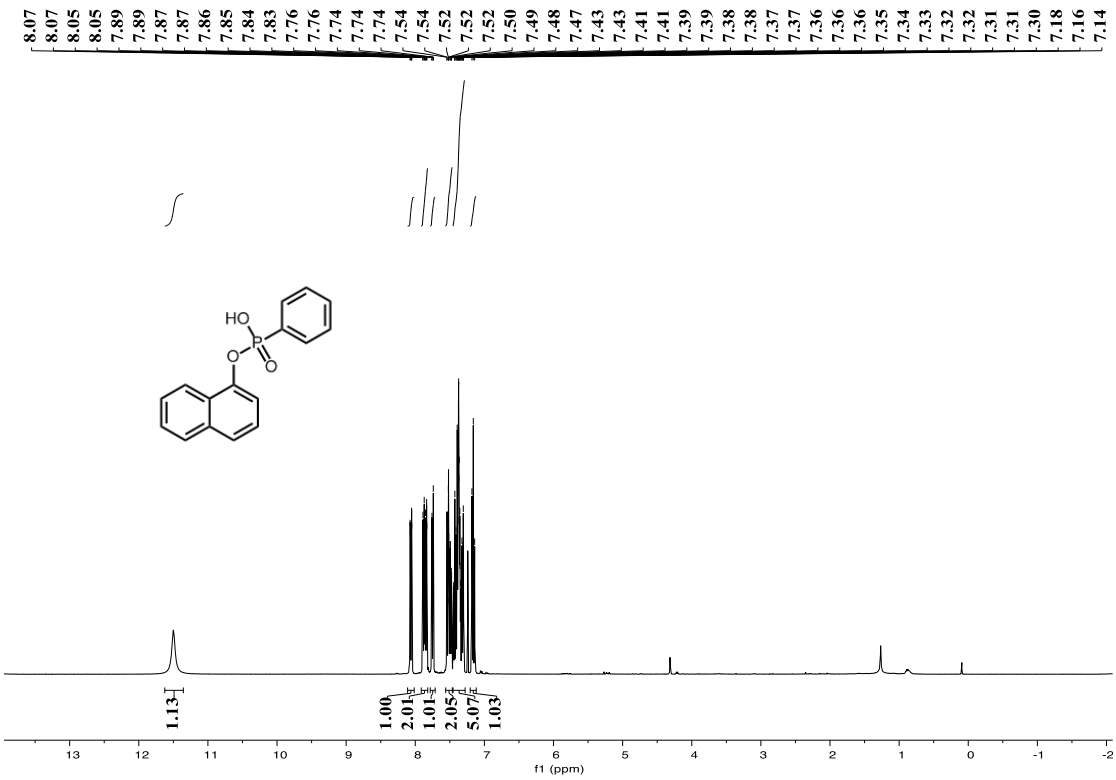

22.93
O
H
P
N
O
O
Bn
Bn
140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -15
f1 (ppm)

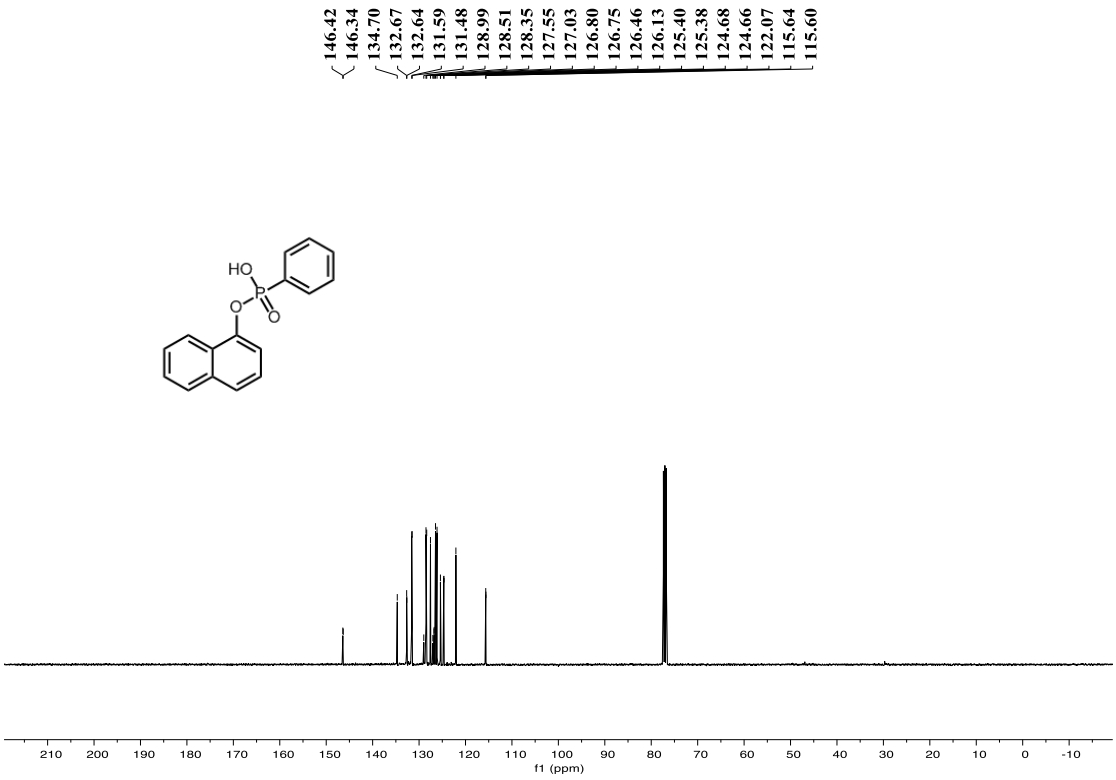

7.80
7.80
7.79
7.79
7.78
7.78
7.77
7.77
7.76
7.76
7.75
7.75
7.74
7.56
7.55
7.55
7.55
7.54
7.54
7.53
7.53
7.53
7.53
7.52
7.52
7.51
7.51
7.49
7.49
7.48
7.48
7.48
7.47
7.47
7.47
7.46
7.46
7.45
7.45
7.45
7.44
7.44
7.44
O
P
Ph
OH
Ph
4.00
2.03
4.00
f1 (ppm)


133.80
132.43
131.81
131.78
130.91
130.80
128.31
128.18
O
P
Ph
OH
Ph
f1 (ppm)

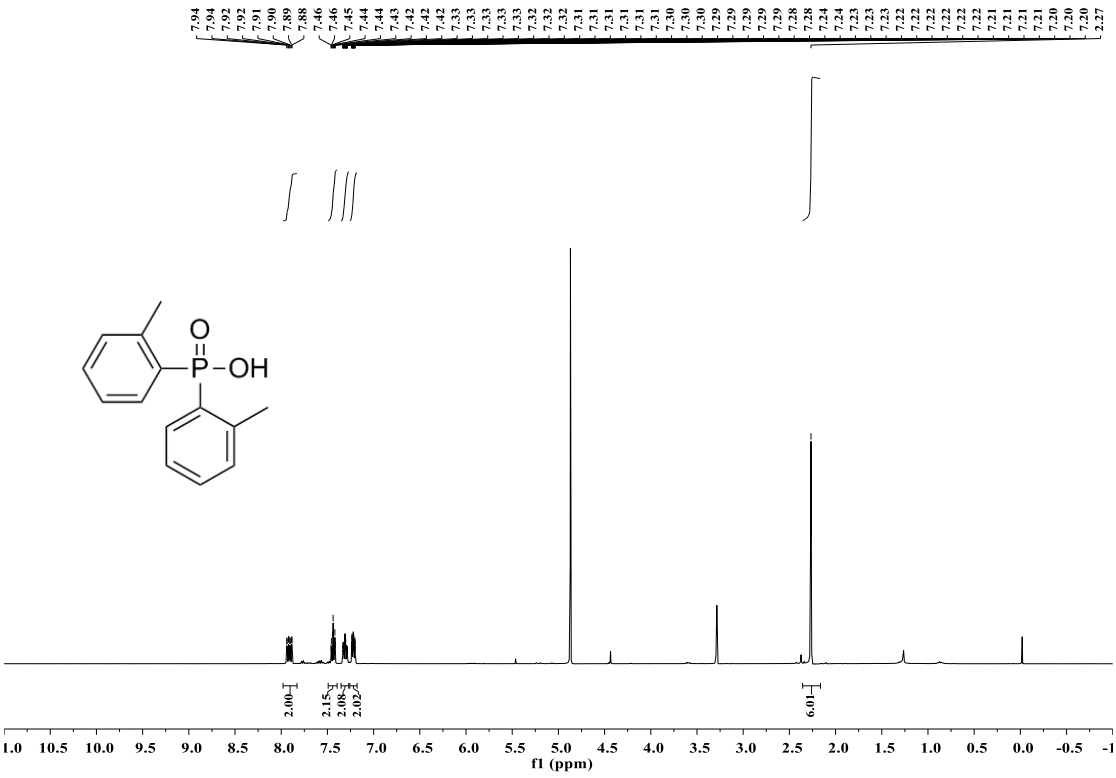

7.38 7.38 7.37 7.37 7.37 7.37 7.36 7.36 7.36 7.35 7.35 7.35 7.34 7.34 7.34 7.34 7.33 7.33 7.33 7.32 7.32 7.32 7.31 7.31 7.31 7.21 7.20 7.20 7.20 7.19 7.19 7.19 7.18 7.18 7.18 7.17 7.17 7.17 7.16 7.16 7.15 7.15 7.15 7.14
O
PhO
P
OH
OPh
4.00
5.89
f1 (ppm)

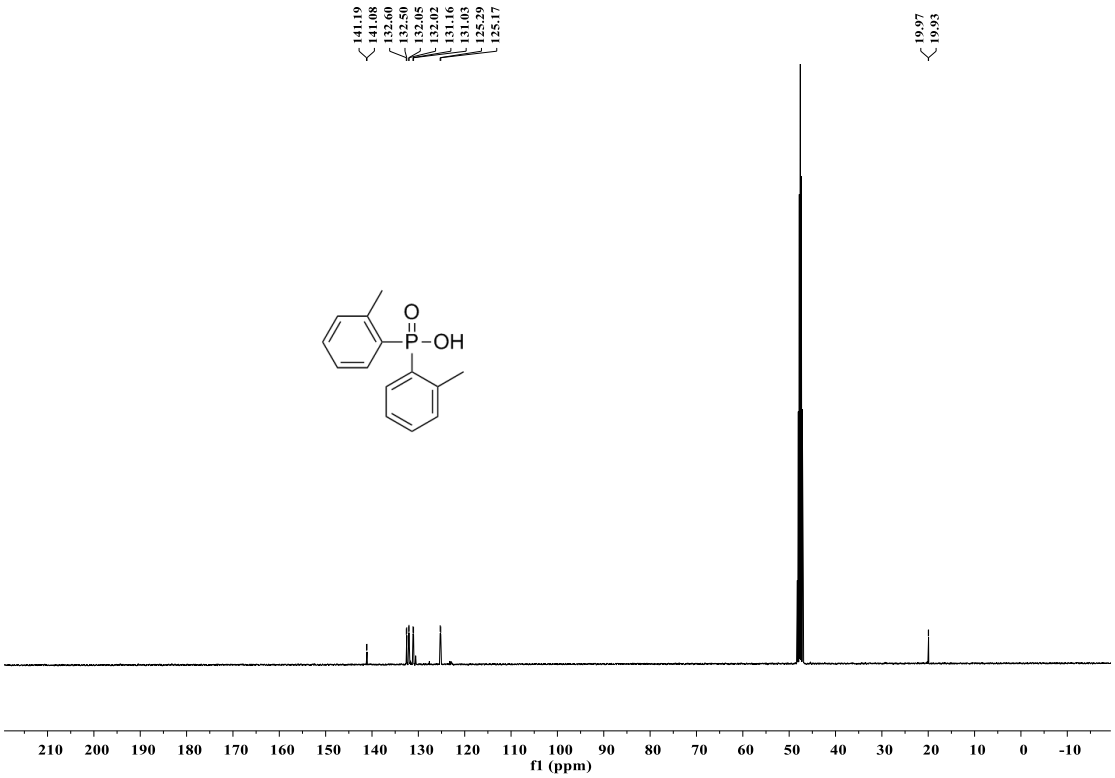

O
PhO
P
OH
OPh
150.37
150.30
129.74
125.43
120.18
120.14
f1 (ppm)

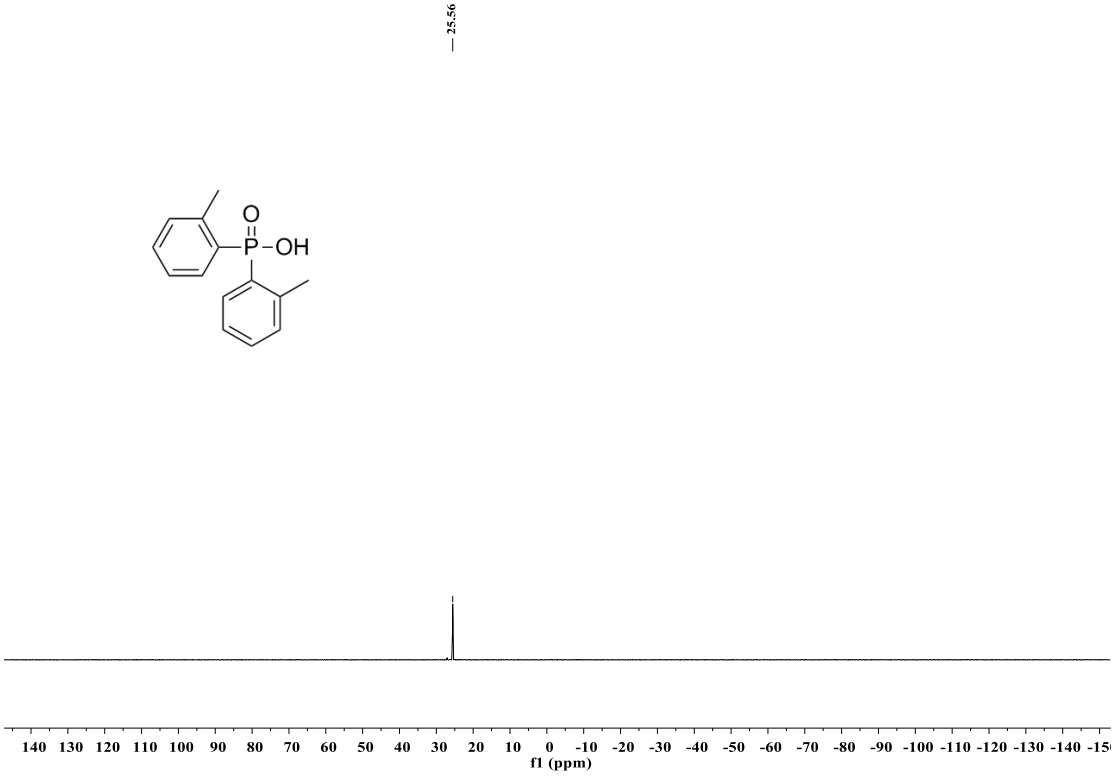

8.07 8.07 8.05 8.05 7.89 7.89 7.87 7.87 7.86 7.85 7.84 7.83 7.76 7.76 7.74 7.74 7.74 7.54 7.54 7.52 7.52 7.52 7.50 7.49 7.48 7.47 7.43 7.43 7.41 7.41 7.39 7.39 7.38 7.38 7.37 7.37 7.36 7.36 7.36 7.35 7.34 7.33 7.32 7.32 7.31 7.31 7.30 7.18 7.16 7.14
HO
O
P
O
1.13
1.00
2.01
1.01
2.05
5.07
1.03
f1 (ppm)


146.42 146.34 134.70 132.67 132.64 131.59 131.48 128.99 128.51 128.35 127.55 127.03 126.80 126.75 126.46 126.13 125.40 125.38 124.68 124.66 122.07 115.64 115.60
HO
O
P
O
f1 (ppm)


17.32
HO
P
O
O
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240
f1 (ppm)


7.94 7.94 7.92 7.92 7.91 7.90 7.89 7.88 7.46 7.45 7.44 7.44 7.43 7.42 7.42 7.42 7.33 7.33 7.33 7.33 7.32 7.32 7.32 7.31 7.31 7.31 7.31 7.31 7.31 7.30 7.30 7.30 7.29 7.29 7.29 7.29 7.29 7.28 7.28 7.24 7.24 7.23 7.23 7.23 7.22 7.22 7.22 7.22 7.22 7.22 7.21 7.21 7.21 7.21 7.20 7.20 7.20 2.27
O
P–OH
2.00
2.15
2.08
2.02
6.01
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)





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