The Construction of Phosphonate Triazoles Using Furan Dearomatized Cycloaddition

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I. General information

Dichloromethane was refluxed with CaH_2 and freshly distilled prior to use. THF was refluxed with Na and freshly distilled prior to use. 1,1,1,3,3,3-Hexafluoro-2-propanol (HFIP) was purchased from Aladdin Chemicals. All reactions under standard conditions were monitored by thin-layer chromatography (TLC) on gel F254 plates. The silica gel (200-300 meshes) was used for column chromatography, and the distillation range of petroleum ether was 60-90℃. High-resolution mass spectra (HRMS) were obtained on a Bruker MAXIS and are reported as m/z (relative intensity). Accurate masses are reported for the molecular ion $[M+Na]^+$ or $[M+H]^+$. ^1H and ^{13}C NMR spectra were recorded on *Bruker* AM-600 MHz, *Bruker* AM-400 MHz and *JEOL* JNM-ECZ-400 MHz instruments, and spectral data were reported in *ppm* relative to tetramethylsilane (TMS) as internal standard. Data for ^1H NMR are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, p = quintet, m = multiplet, dd = doublet of doublets, dt = doublet of triplets etc, br = broad), coupling constant (Hz) and integration. All ^{13}C NMR and ^{31}P NMR spectra are reported in ppm.

II. General procedure for Cu-catalyzed [3+2] cycloaddition of furfuryl phosphonate alcohols and organo azides

A sealed tube (25 mL) equipped with a stirring bar was loaded with furfuryl phosphonate alcohols (0.2 mmol, 1.0 equiv), organo aizdes (0.2 mmol, 1.0 equiv) and $CuCl_2$ (0.01 mmol, 5 mmol%) in HFIP (2 mL), the mixture was stirred at 80°C for 1h under a nitrogen atmosphere. After the completion of reaction, the mixture was quenched with water and extracted three times with dichloromethene (25 mL). The organic layer was dried with Na_2SO_4 and concentrated under vacuum. The oily crude product was purified by silica gel with petroleum ether/ethyl acetate as eluent to give the corresponding phosphonates triazoles in noted yields.

III. Typical preparation of furfuryl phosphonate alcohol substrates

To a round bottom flask (25 mL) equipped with a stirring bar was loaded with 5-methyl furfuraldehyde (0.91 mmol, 1.0 equiv) and alkyl phosphonates (1.09 mmol, 1.2 equiv), and then was added triethylamine (0.045 mmol, 0.05 equiv) dropwise. The resulted mixture was stirred at room temperature for 8h. After the completion of reaction via TLC detection, the mixture was quenched with 0.1 N HCl and extracted three times with dichloromethene (25ml). The organic layer was dried with Na_2SO_4 and concentrated under vacuum. The oily crude product was purified by silica gel with petroleum ether/ethyl acetate as eluent to give the corresponding furfuryl phosphonate alcohols in noted yields.

IV. Preparation of (3a) by multi-components reaction

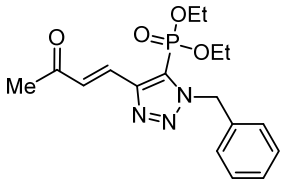
A sealed tube (25 mL) equipped with a stirring bar was loaded with 5-methyl furfuryldehyde (0.2 mmol, 1.0 equiv), alkyl phosphonates (1.2 equiv) and triethylamine (0.1 equiv). The mixture was stirred at room temperature for 6h under a nitrogen atmosphere. After that, a solution of benzyl aizde **2a** (1.0 equiv) and $CuCl_2$ (0.05 equiv) in HFIP (5 mL) was added and the mixture was stirred at 80°C for 1h. After the completion of reaction via TLC detection, the mixture was quenched with water and extracted three times with dichloromethene (25 mL). The organic layer was dried over Na_2SO_4 and concentrated under vacuum. The oily crude product was purified by silica gel with petroleum ether/ethyl acetate as eluent to give the corresponding phosphonate triazole **3a** in 71% yields.

V. Gram-scale reaction

A sealed tube (100 mL) equipped with a stirring bar was loaded with furfuryl phosphonate alcohols **1a** (1 g, 4.05 mmol, 1.0 equiv), benzyl azide **2a** (0.54 g, 4.05 mmol, 1.0 equiv) and $CuCl_2$ (27 mg, 0.2 mmol, 5 mmol%) in HFIP (15 mL), the mixture was stirred at 80°C for 1h

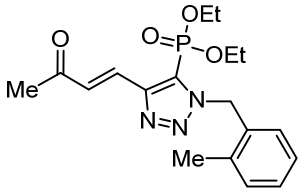
under a nitrogen atmosphere. After the completion of reaction, the mixture was quenched with water and extracted three times with dichloromethene (75 mL). The organic layer was dried with Na_2SO_4 and concentrated under vacuum. The oily crude product was purified by silica gel with petroleum ether/ethyl acetate as eluent to give the phosphonate triazole **3a** (1.28 g, 3.52 mmol) in 87% yield.

VI. Spectroscopic data of products and substrates



Diethyl(E)-(1-benzyl-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phosphonate

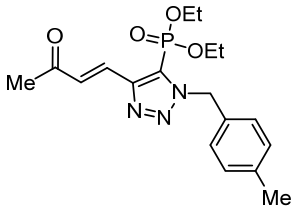
(3a) Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3a** as a yellow oil (62 mg, 86% yield), 0.2 mmol 5-methylfurfurylphosphonate (**1a)** and benzyl azide **(2a)** were used as starting materials.**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 16.1 Hz, 1H), 7.36 – 7.31 (m, 1H), 7.31 – 7.27 (m, 4H), 5.87 (s, 2H), 4.05 (m, 2H), 3.94 – 3.83 (m, 2H), 2.38 (s, 3H), 1.20 (t, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.90, 148.65 (d, J = 20.0 Hz), 134.90, 130.23, 129.24, 128.61, 128.40, 127.84, 125.02 (d, J = 214.9 Hz), 63.41 (d, *J* = 5.2 Hz), 53.78, 28.14, 15.93 (d, *J* = 6.6 Hz). **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.42. **HRMS (ESI-TOF) m/z**: calcd for $C_{17}H_{22}N_3O_4PNa$ $[M+Na]^+$ 386.1240; found 386.1240.



Diethyl

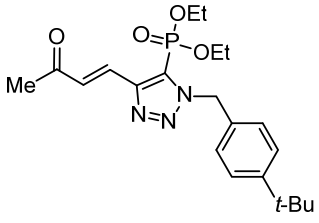
(E)-(1-(2-methylbenzyl)-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phosphonate (3b)

Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3b** as a yellow oil (64 mg, 85% yield), 0.2 mmol 5-methylfurfurylphosphonate **(1a)** and 2-methylbenzyl azide **(2b)** were used as starting material. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 16.1 Hz, 1H), 7.35 – 7.27 (m, 1H), 7.20 (d, *J* = 4.1 Hz, 2H), 7.10 (m, 1H), 6.73 (d, *J* = 7.6 Hz, 1H), 5.86 (s, 2H), 4.10 – 4.00 (m, 2H), 3.94 – 3.83 (m, 2H), 2.44 (s, 3H), 2.40 (s, 3H), 1.20 (t, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.98, 148.61, 135.68, 133.50, 130.43, 130.33, 129.38, 128.18, 126.78, 126.52, 126.21, 124.38, 63.48 (d, *J* = 5.3 Hz), 51.51, 28.14, 19.22, 15.96 (d, *J* = 6.6 Hz). **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.50. **HRMS** (ESI-TOF) m/z: calcd for $C_{18}H_{24}N_3O_4PNa$ $[M+Na]^+$ 400.1397; found 400.1399.



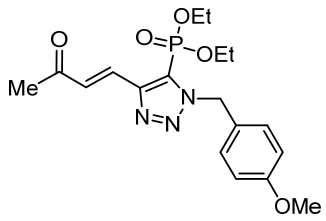
Diethyl(E)-(1-(4-methylbenzyl)-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phosphonate (3c)

Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3c** as a yellow oil (64 mg, 85% yield), 0.2 mmol 5-methylfurfurylphosphonate **(1a)** and 4-methylbenzyl azide **(2c)** were used as starting material.**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 16.1 Hz, 1H), 7.27 – 7.21 (m, 1H), 7.18 (m, 2H), 7.09 (m, 2H), 5.77 (s, 2H), 4.07 – 3.97 (m, 2H), 3.85 (m, 2H), 2.34 (s, 3H), 2.27 (s, 3H), 1.17 (t, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.95, 148.57 (d, *J* = 20.0 Hz) 138.25, 131.85, 130.16, 129.30, 129.21, 127.90, 126.09 – 123.54 (m), 63.40 (d, *J* = 5.2 Hz), 53.59, 28.13, 21.03, 15.93 (d, *J* = 6.6 Hz). **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.51. **HRMS** (ESI-TOF) m/z: calcd for $C_{18}H_{24}N_3O4PNa$ $[M+Na]^+$ 400.1397; found 400.1397.



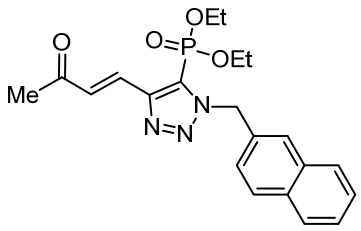
Diethyl(E)-(1-(4-(tert-butyl)benzyl)-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phospho nate (3d)

Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3d** as a yellow oil (68 mg, 81% yield), 0.2 mmol 5-methylfurfurylphosphonate **(1a)** and 4-(tert-butyl)benzyl azide **(2d)** were used as starting material.**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.76 – 7.69 (m, 1H), 7.35 (d, *J* = 8.3 Hz, 2H), 7.28 (t, *J* = 5.5 Hz, 1H), 7.27 – 7.24 (m, 2H), 5.83 (s, 2H), 4.08 – 3.97 (m, 2H), 3.91 – 3.80 (m, 2H), 2.38 (s, 3H), 1.28 (s, 9H), 1.18 (t,6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.91, 151.56, 148.67 (d, *J* = 20.1 Hz), 131.99, 130.21, 129.33, 127.71, 126.01, 124.70 (d, *J* = 165.6 Hz), 63.36 (d, *J* = 5.1 Hz), 53.60, 34.51, 31.18, 28.16, 15.94 (d, *J* = 6.6 Hz). **^{31}P** (162 MHz, $CDCl_3$) δ 3.45. **HRMS** (ESI-TOF) m/z: calcd for $C_{21}H_{30}N_3O_4PNa$ $[M+Na]^+$ 442.4515; found 442.4576.



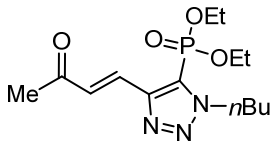
Diethyl(E)-(1-(4-methoxybenzyl)-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phosphona te (3e)

Isolated by column chromatography over silica gel (eluent, 1:1 petroleum ether/ethyl acetate) to afford **3e** as a yellow oil (51 mg, 64% yield), 0.2 mmol 5-methylfurfurylphosphonate **(1a)** and 4-methoxybenzyl azide **(2e)** were used as starting material. **^{1}H NMR** (400 MHz,) δ (400 MHz, $CDCl_3$) δ 7.73 – 7.67 (m, 1H), 7.34 – 7.21 (m, 3H), 6.89 – 6.80 (m, 2H), 5.79 (s, 2H), 4.14 – 4.04 (m, 2H), 3.98 – 3.88 (m, 2H), 3.78 (s, 3H), 2.38 (s, 2H), 1.23 (t, *J* = 7.1 Hz, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.89, 159.62, 148.51 (d, *J* = 20.0 Hz),130.12, 129.55, 129.25, 126.91, 124.75 (d, *J* = 214.9 Hz), 113.86, 63.45, 55.21, 53.32, 28.14, 15.96 (d, *J* = 6.5 Hz). **^{31}P** (162 MHz, $CDCl_3$) δ 3.58. **HRMS** (ESI-TOF) m/z: calcd for $C_{18}H_{24}N_3O_5PNa$ $[M+Na]^+$ 416.1346; found 416.1348.



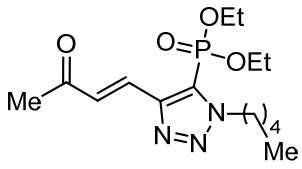
Diethyl(E)-(1-(naphthalen-1-ylmethyl)-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phosphonate (3f)

Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3f** as a yellow oil (60 mg, 73% yield), 0.2 mmol 5-methylfurfurylphosphonate **(1a)** and 1-naphthylmethyl azide **(2f)** were used as starting material. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81 (m, 4H), 7.72 (d, *J* = 16.1 Hz, 1H), 7.52 – 7.43 (m, 3H), 7.30 (d, *J* = 16.1 Hz, 1H), 6.03 (s, 2H), 4.01 (m, 2H), 3.87 (m, 2H), 2.38 (s, 3H), 1.11 (t, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 198.09, 148.73, 138.07, 133.12, 132.39, 130.41, 129.36, 128.72, 128.03, 127.78, 127.50, 126.67, 126.34, 125.85, 125.43, 63.66, 54.17, 28.42, 16.04 (d, *J* = 6.6 Hz). **^{31}P** (162 MHz, $CDCl_3$) δ 3.48. **HRMS** (ESI-TOF) m/z: calcd for $C_{21}H_{24}N_3O_4PNa$ $[M+Na]^+$ 436.1397; found 436.1398.



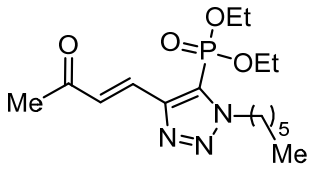
Diethyl (E)-(1-butyl-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phosphonate (3g)

Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3g** as a yellow oil (46 mg, 70% yield), 0.2 mmol 5-methylfurfurylphosphonate **(1a)** and n-butyl azide **(2g)** were used as starting material. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 16.1 Hz, 1H), 7.29 (s, 1H), 4.62 (t, 2H), 4.29 – 4.11 (m, 4H), 2.39 (s, 3H), 1.99 – 1.90 (m, 2H), 1.39 (m, 8H), 0.97 (t, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 198.02, 148.27 (d, *J* = 21.0 Hz), 130.11, 129.48, 123.88 (s), 63.54 (d, *J* = 5.4 Hz), 50.55, 32.43, 28.11, 19.63, 16.16 (d, *J* = 6.5 Hz), 13.47. **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.95. **HRMS** (ESI-TOF) m/z: calcd for $C_{14}H_{24}N_3O_4PNa$ $[M+Na]^+$ 352.1397; found 352.1397.



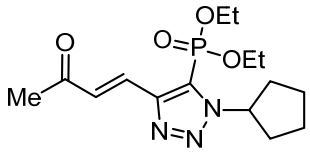
Diethyl(E)-(4-(3-oxobut-1-en-1-yl)-1-pentyl-1H-1,2,3-triazol-5-yl)phosphonate (3h)

Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3h** as a yellow oil (53 mg, 77% yield), 0.2 mmol 5-methylfurfurylphosphonate **(1a)** and n-pently azide **(1h)** were used as starting material. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 16.1 Hz), 7.27 (d, *J* = 15.6 Hz), 4.61 (t, *J* = 7.5 Hz), 4.29 – 4.11 (m), 2.39 (s), 2.02 – 1.91 (m), 1.43 – 1.24 (m), 0.98 – 0.87 (m). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 198.01, 148.27 (d, *J* = 20.8 Hz), 130.10, 129.48, 124.96 (d, *J* = 217.0 Hz), 63.53 (d, *J* = 5.5 Hz), 50.79, 30.19, 28.49, 28.11, 22.08, 16.16 (d, *J* = 6.5 Hz), 13.80. **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.95. **HRMS** (ESI-TOF) m/z: calcd for $C_{15}H_{26}N_3O_4PNa$ $[M+Na]^+$ 366.1553; found 366.1560



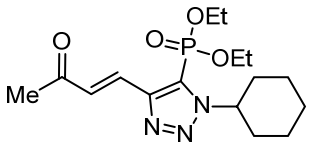
Diethyl(E)-(1-hexyl-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phosphonate (3i)

Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3i** as a yellow oil (57 mg, 80% yield), 0.2 mmol 5-methylfurfurylphosphonate **(1a)** and n- hexyl azide **(2i)** were used as starting material. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 16.1 Hz, 1H), 7.27 (d, *J* = 16.5 Hz), 4.61 (t, *J* = 7.3 Hz), 4.28 – 4.12 (m), 2.39 (s), 1.95 (d, *J* = 7.0 Hz), 1.37 (dd, *J* = 17.1, 10.1 Hz), 0.89 (s). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.86, 148.12 (d, *J* = 20.7 Hz), 129.96, 129.34, 125.89 (s), 63.38 (d, *J* = 5.3 Hz), 58.46, 50.68, 30.65 (d, *J* = 67.5 Hz), 25.93, 22.23, 16.01 (d, *J* = 6.4 Hz), 13.74. **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.06. **HRMS** (ESI-TOF) m/z: calcd for $C_{15}H_{26}N_3O_4PNa$ $[M+Na]^+$ 380.1710; found 380.1716



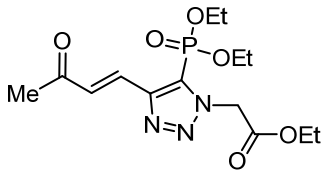
Diethyl(E)-(1-cyclopentyl-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phosphonate (3j)

Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3j** as a yellow oil (53 mg, 78% yield), 0.2 mmol 5-methylfurfurylphosphonate **(1a)** and cyclopentyl azide **(2j)** were used as starting material. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 16.0 Hz, 1H), 7.20 (d, *J* = 16.1 Hz, 1H), 5.31 – 5.24 (m, 1H), 4.24 – 4.07 (m, 4H), 2.33 (s, 3H), 2.18 – 2.11 (m, 4H), 2.02 – 1.94 (m, 2H), 1.74 – 1.67 (m, 2H), 1.32 (t, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 198.13, 148.15 (d, *J* = 21.2 Hz),130.11, 129.87, 124.95 (d, *J* = 216.2 Hz), 63.45 (d, *J* = 5.3 Hz), 62.11, 33.75, 27.79, 24.66, 16.13 (d, *J* = 6.4 Hz), 16.10. **^{31}P NMR** (162 MHz, $CDCl_3$) δ 4.23. **HRMS** (ESI-TOF) m/z: calcd for $C_{15}H_{24}N_3O_4PNa$ $[M+Na]^+$ 364.1397; found 364.1396.



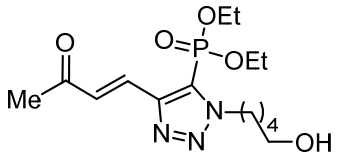
Diethyl(E)-(1-cyclohexyl-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phosphonate (3k)

Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3k** as a yellow oil (61 mg, 81% yield), 50.2 mmol 5-methylfurfurylphosphonate **(1a)** and cyclohexyl azide (3k) were used as starting material. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.83 (d, J = 16.1 Hz, 1H), 7.29 (d, J = 1.2 Hz, 1H), 4.83 – 4.74 (m, 1H), 4.21 (m, 4H), 2.39 (s, 3H), 2.12 – 2.05 (m, 4H), 1.96 (m, 2H), 1.76 (m, 1H), 1.50 – 1.41 (m, 2H), 1.38 (t, 7H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 198.17, 147.70, 130.14, 129.86, 124.45, 63.48, 61.35, 60.91, 55.60, 33.39, 32.68, 27.94, 26.62, 25.44, 24.93, 22.30, 16.22, 16.19 (d, *J* = 6.5 Hz). **^{31}P NMR** (162 MHz, $CDCl_3$) δ 4.25. **HRMS** (ESI-TOF) m/z: calcd for $C_{16}H_{26}N_3O_4PNa$ $[M+Na]^+$ 378.1553; found 378.1558.



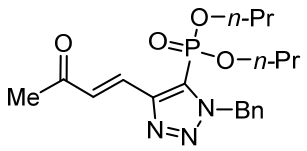
Diethyl(E)-2-(5-(diethoxyphosphoryl)-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-1-yl)acetat e (3l)

Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3l** as a yellow oil (47 mg, 66% yield), 0.2 mmol 5-methylfurfurylphosphonate **(1a)** and ethyl azidoacetate **(2l)** were used as starting material. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 16.0 Hz, 1H), 7.30 (d, *J* = 15.4 Hz, 1H), 5.47 (s, 2H), 4.31 – 4.19 (m, 4H), 4.17 – 4.06 (m, 2H), 2.40 (s, 3H), 1.34 (m, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.75, 149.82 – 146.87 (m), 166.51, 130.35, 128.64, 128.04 – 124.49 (m) 63.80 (d, *J* = 4.9 Hz), 62.36, 51.41, 28.44, 16.09 (d, *J* = 6.6 Hz), 14.02. **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.95 **HRMS** (ESI-TOF) m/z: calcd for $C_{14}H_{22}N_3O_4PNa$ $[M+Na]^+$ 382.1138; found 382.1139.



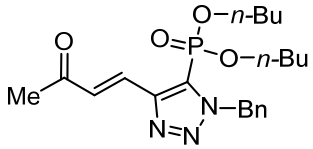
Diethyl(E)-(1-(6-hydroxyhexyl)-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phosphonat e (3m)

Isolated by column chromatography over silica gel (eluent, 1:1 petroleum ether/ethyl acetate) to afford **3m** as a yellow oil (56 mg, 75% yield), 0.2 mmol 5-methylfurfurylphosphonate **(1a)** and 6-hydroxyhexyl aizde **(2m)** were used as starting material.**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 16.0 Hz, 1H), 7.30 – 7.25 (m, 1H), 4.66 – 4.60 (m, 1H), 4.28 – 4.13 (m, 4H), 3.64 (t, 2H), 2.39 (s, 3H), 1.97 (m, 2H), 1.63 – 1.56 (m, 5H), 1.38 (t, 7H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.85, 153.81, 129.90, 129.80, 129.14, 122.64, 63.46, 62.32, 50.42, 32.10, 30.17, 28.08, 25.87, 24.81, 16.03, 15.96. **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.53. **HRMS** (ESI-TOF) m/z: calcd for $C_{16}H_{28}N_3O_5PNa$ $[M+Na]^+$ 396.1659; found 396.1661.



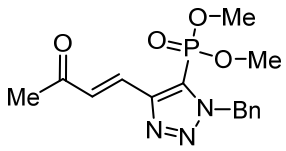
Dipropyl(E)-(1-benzyl-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phosphonate (3n)

Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3n** as a yellow oil (61 mg, 78% yield), 0.2 mmol **1b** and benzyl azide **(2a)** were used as starting material was used. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.71 (d, *J* = 16.0 Hz, 1H), 7.33 – 7.26 (m, 5H), 5.88 (s, 2H), 3.93 (m, 2H), 3.73 (m, 2H), 2.38 (s, 3H), 1.55 (m, 4H), 0.84 (t, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.82, 148.58 (d, *J* = 19.9 Hz), 134.99, 130.21, 129.20, 128.62, 128.40, 127.89, 123.97 (s), 68.74 (d, *J* = 5.6 Hz), 53.77, 28.16, 23.40 (d, *J* = 6.6 Hz), 9.80. **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.44. **HRMS** (ESI-TOF) m/z: calcd for $C_{19}H_{26}N_3O_4PNa$ $[M+Na]^+$ 414.1553; found 414.1559.



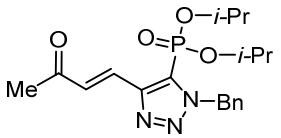
Dibutyl(E)-(1-benzyl-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phosphonate (3o)

Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3o** as a yellow oil (65 mg, 78% yield), 0.2 mmol **1c** and benzyl azide **(2a)** were used as starting material was used. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 16.1 Hz, 1H), 7.32 – 7.28 (m, 5H), 5.87 (s, 2H), 3.97 (m, 2H), 3.82 – 3.72 (m, 2H), 2.37 (S, 3H), 1.50 (m, 4H), 1.32 – 1.18 (m, 4H), 0.85 (t, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.86, 148.60 (d, *J* = 19.8 Hz), 134.99, 130.24, 129.21, 128.63, 128.41, 127.88, 126.33 – 123.67 (m), 67.03 (d, *J* = 5.6 Hz), 53.77, 31.96 (d, *J* = 6.5 Hz), 28.14, 18.47, 13.39. **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.96. **HRMS** (ESI-TOF) m/z: calcd for $C_{21}H_{30}N_3O_4PNa$ $[M+Na]^+$ 442.1866; found 442.1863.



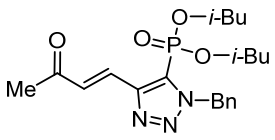
Dimethyl(E)-(1-benzyl-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phosphonate(3p)

Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3p** as a yellow oil (54 mg, 81% yield), 0.2 mmol **1d** and benzyl azide **(2a)** were used as starting material was used. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 16.0 Hz, 1H), 7.34 – 7.29 (m, 5H), 5.85 (s, 2H), 3.59 (d, 6H), 2.38 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.88, 149.55 – 148.70 (m), 134.92, 130.59, 128.86, 128.72, 128.54, 127.92, 125.11 – 122.18 (m), 53.98, 53.28 (d, *J* = 5.3 Hz), 28.30. **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.57. **HRMS** (ESI-TOF) m/z: calcd for $C_{15}H_{18}N_3O_4PNa$ $[M+Na]^+$ 358.0927; found 358.0921.



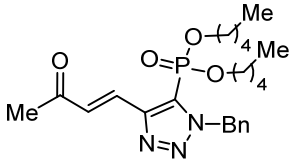
Diisopropyl(E)-(1-benzyl-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phosphonate (3q)

Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3q** as a yellow oil (62 mg, 79% yield), 0.2 mmol **1e** and benzyl azide **(2a)** were used as starting material was used. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 16.1 Hz, 1H), 7.33 (m, 5H), 5.89 (s, 2H), 4.60 – 4.50 (m, 2H), 2.38 (s, 3H), 1.31 (m, 6H), 1.05 (m, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.96, 148.14(m) 139.60, 135.04, 129.95, 129.66, 128.60, 128.38, 128.03, 127.67 – 124.95 (m), 73.08 (d, *J* = 5.5 Hz), 53.69, 28.06, 23.86 (d, *J* = 4.1 Hz), 23.36 (d, *J* = 4.9 Hz). **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.98. **HRMS** (ESI-TOF) m/z: calcd for $C_{19}H_{26}N_3O_4PNa$ $[M+Na]^+$ 414.1553; found 414.1553.



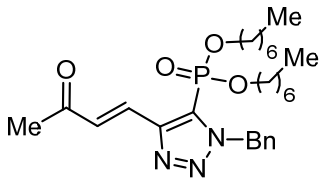
Diisobutyl(E)-(1-benzyl-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phosphonate (3r)

Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3r** as a yellow oil (64 mg, 79% yield), 0.2 mmol **1f** and benzyl azide **(2a)** were used as starting material was used. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 16.1 Hz, 1H), 7.34 – 7.28 (m, 5H), 5.89 (s, 2H), 3.73 (m, 2H), 3.48 (m, 2H), 2.37 (s, 3H), 1.79 (m, 2H), 0.83 (m, 12H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.81, δ 149.29 – 147.95 (m), 135.08, 130.20, 129.15, 128.68, 128.46, 127.98, 126.85 – 122.67 (m) 72.96, 53.79, 28.84 (d, *J* = 6.8 Hz), 28.23, 18.46 (s). **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.46. **HRMS** (ESI-TOF) m/z: calcd for $C_{21}H_{30}N_3O_4PNa$ $[M+Na]^+$ 442.1866; found 442.1870.



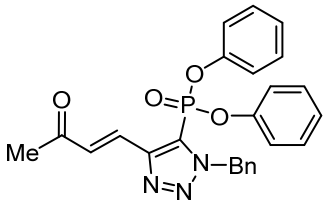
Dipentyl(E)-(1-benzyl-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phosphonate (3s)

Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3s** as a yellow oil (69 mg, 87% yield), 0.2 mmol **1g** and benzyl azide **(2a)** were used as starting material was used. **^{1}H NMR** (400 MHz, $CDCl_3$) δ δ 7.69 – 7.61 (m, 1H), 7.34 – 7.26 (m, 5H), 5.84 (s, 2H), 3.92 (m, 2H), 3.79 – 3.66 (m, 2H), 2.35 (s, 3H), 1.54 – 1.45 (m, 4H), 1.36 – 1.29 (m, 2H), 1.26 – 1.10 (m, 9H), 0.83 (t, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.84, 148.87 – 148.30 (m), 136.12, 131.90, 130.27, 129.25, 128.66, 128.44, 127.93, 126.23 – 123.50 (m), 67.20 (d, *J* = 5.6 Hz), 53.79, 29.56 (d, *J* = 6.4 Hz), 28.17, 27.37, 22.03, 13.83. **^{31}P NMR** (162 MHz, $CDCl_3$) δ 4.06. **HRMS** (ESI-TOF) m/z: calcd for $C_{23}H_{34}N_3O_4PNa$ $[M+Na]^+$ 470.2179; found 470.2181.



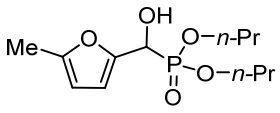
Diheptyl(E)-(1-benzyl-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phosphonate(3t)

Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3t** as a yellow oil (81 mg, 81% yield), 0.2 mmol **1h** and benzyl azide **(2a)** were used as starting material was used. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 16.1 Hz, 1H), 7.35 – 7.30 (m, 5H), 7.26 (s, 1H), 5.87 (s, 2H), 3.95 (m, 2H), 3.75 (m, 2H), 2.38 (s, 3H), 1.56 – 1.45 (m, 4H), 1.29 – 1.14 (m, 16H), 0.87 (t, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.82, 149.10 – 148.29 (m), 135.00, 130.26, 129.26, 128.63, 128.42, 127.90, 126.19 – 123.87 (m), 67.36 (d, *J* = 5.6 Hz), 53.76, 31.53, 30.01 (d, *J* = 6.6 Hz), 28.60, 28.11, 25.20, 22.46, 13.99. **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.08. **HRMS** (ESI-TOF) m/z: calcd for $C_{27}H_{42}N_3O_4PNa$ $[M+Na]^+$ 540.2962; found 540.2962.



Diphenyl (E)-(1-benzyl-4-(3-oxobut-1-en-1-yl)-1H-1,2,3-triazol-5-yl)phosphonate (3u)

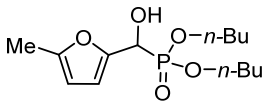
Isolated by column chromatography over silica gel (eluent, 2:1 petroleum ether/ethyl acetate) to afford **3u** as a yellow oil (66 mg, 71% yield), 0.2 mmol **1i** and benzyl azide **(2a)** were used as starting material was used.as starting material.**^{1}H NMR** (400 MHz,) δ 7.30 – 7.14 (m, 11H), 6.92 (m, 5H), 6.29 (d, *J* = 12.7 Hz, 1H), 5.85 (d, *J* = 19.6 Hz, 2H), 2.30 (s, 3H). **^{13}C NMR** (101 MHz,) δ 202.47, 149.23 (d, *J* = 7.3 Hz), 134.71 (d, *J* = 10.0 Hz), 130.13, 128.87, 128.61, 128.22, 126.15, 121.00, 120.34, 120.30, 54.19, 30.42. **^{31}P NMR** (162 MHz, $CDCl_3$) δ -3.78. **HRMS** (ESI-TOF) m/z: calcd for $C_{25}H_{22}N_3O_4PNa$ $[M+Na]^+$ 482.1240; found 482.1248.



dipropyl (hydroxy(5-methylfuran-2-yl) methyl) phosphonate (1b)

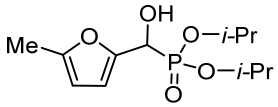
Isolated by column chromatography over silica gel (eluent, 6:1 petroleum ether/ethyl acetate) 0.91 mmol 5-methyl furfuraldehyde and dipropyl phosphonates (1.2 equiv) were used as starting materials to afford **1b** as a yellow oil (241 mg, 96% yield) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.39 (t, *J* = 2.8 Hz, 1H), 5.95 (d, *J* = 2.7 Hz, 1H), 4.95 (d, *J* = 13.3 Hz, 1H), 4.12 –

3.98 (m, 4H), 2.29 (s, 3H), 1.74 – 1.59 (m, 4H), 0.98 – 0.85 (m, 7H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ152.19, 148.20, 110.05 (d, *J* = 6.0 Hz), 106.47, 68.53 (dd, *J* = 28.2, 7.0 Hz), 64.32, (d, *J* = 168.0 Hz)23.66 (t, *J* = 5.0 Hz), 9.76 (d, *J* = 2.3 Hz). **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.74. **HRMS** (ESI-TOF) m/z: calcd for $C_{12}H_{21}O_5PNa$ $[M+Na]^+$ 299.1019; found 299.1026.



dibutyl (hydroxy(5-methylfuran-2-yl) methyl) phosphonate (1c)

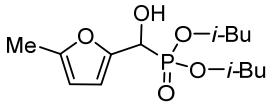
Isolated by column chromatography over silica gel (eluent, 6:1 petroleum ether/ethyl acetate) 0.91 mmol 5-methyl furfuraldehyde and dibuyl phosphonates (1.2 equiv) were used as starting materials to afford **1c** (254 mg, 92% yield). **^{1}H NMR** (600 MHz, $CDCl_3$) δ 6.39 (d, *J* = 1.9 Hz, 1H), 5.93 (s, 1H), 4.96 (d, *J* = 13.3 Hz, 1H), 4.18 – 3.94 (m, 4H), 2.27 (s, 3H), 1.68 – 1.47 (m, 4H), 1.42 – 1.29 (m, 4H), 1.00 – 0.69 (m, 7H).**^{13}C NMR** (151 MHz, $CDCl_3$) δ 151.96, 148.30, 109.87 (d, *J* = 6.1 Hz), 106.37, 66.65 (dd, *J* = 44.6, 7.2 Hz), 32.23 (t, *J* = 5.7 Hz), 18.32 (d, *J* = 4.2 Hz), 13.24 (d, *J* = 6.9 Hz). **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.76. **HRMS** (ESI-TOF) m/z: calcd for $C_{14}H_{25}O_5PNa$ $[M+Na]^+$ 327.1332; found 327.1341.



diisopropyl(hydroxy(5-methylfuran-2-yl)methyl) phosphonate (1e)

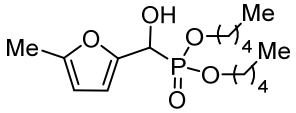
Isolated by column chromatography over silica gel (eluent, 6:1 petroleum ether/ethyl acetate) 0.91 mmol 5-methyl furfuraldehyde and diisopropyl phosphonates (1.2 equiv) were used as starting materials to afford **1d** as a yellow oil (243 mg, 94% yield) Isolated by column chromatography over silica gel (eluent, 6:1 petroleum ether/ethyl acetate) to afford **1e** as a yellow oil (236 mg, 94% yield) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.39 (t, *J* = 2.8 Hz, 1H), 5.96 (t, *J* = 5.7 Hz, 1H), 4.87 (d, *J* = 13.4 Hz, 1H), 4.72 (m, 2H), 2.29 (s, 3H), 1.36 – 1.30 (m, 9H), 1.19 (m, 3H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 152.45, 148.14, 110.32 (d, *J* = 6.0 Hz), 106.64, 72.05 (dd, *J* = 30.8, 7.2 Hz), 64.91 (d, *J* = 168.2 Hz), 24.64 – 23.01 (m), 13.54. **^{31}P NMR** (162

MHz, $CDCl_3$) δ 3.59. **HRMS** (ESI-TOF) m/z: calcd for $C_{12}H_{21}O_5PNa$ $[M+Na]^+$ 299.1019; found 299.1007



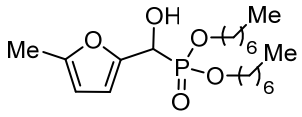
diisobutyl (hydroxy(5-methylfuran-2-yl)methyl)phosphonate (1f)

Isolated by column chromatography over silica gel (eluent, 6:1 petroleum ether/ethyl acetate) 0.91 mmol 5-methyl furfuraldehyde and diisobuyl phosphonates (1.2 equiv) were used as starting materials to afford **1e** (249 mg, 90% yield) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.39 (t, *J* = 2.7 Hz, 1H), 5.95 (d, *J* = 2.4 Hz, 1H), 4.97 (d, *J* = 13.2 Hz, 1H), 3.92 – 3.74 (m, 4H), 2.28 (s, 3H), 1.98 – 1.81 (m, 2H), 0.97 – 0.83 (m, 12H). **^{13}C NMR** (101 MHz, $CDCl_3$) Unknown NMR (101 MHz,) δ 152.19, 148.20, 110.09 (d, *J* = 6.3 Hz), 106.47, 72.83 (dd, *J* = 25.8, 7.4 Hz), 64.37 (d, *J* = 168.0 Hz), 29.16 – 28.66 (m), 18.36 (d, *J* = 5.0 Hz). 13.37. **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.58. **HRMS** (ESI-TOF) m/z: calcd for $C_{14}H_{25}O_5PNa$ $[M+Na]^+$ 327.1332; found 327.1332.



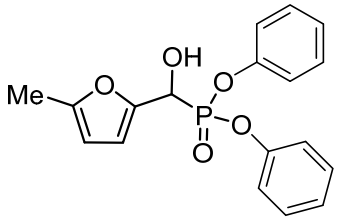
dipentyl (hydroxy(5-methylfuran-2-yl)methyl)phosphonate (1g)

Isolated by column chromatography over silica gel (eluent, 6:1 petroleum ether/ethyl acetate) 0.91 mmol 5-methyl furfuraldehyde and dipentyl phosphonates (1.2 equiv) were used as starting materials to afford **1f** as a yellow oil (271 mg, 89% yield) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.39 (s, 1H), 5.96 (s, 1H), 4.93 (d, *J* = 13.3 Hz, 1H), 4.16 – 3.95 (m, 4H), 2.29 (s, 3H), 2.17 (s, 3H), 1.73 – 1.52 (m, 5H), 1.40 – 1.19 (m,10H), 0.89 (m,7H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 152.29, 148.04, 110.15, 110.12 (d, *J* = 6.0 Hz), 67.08 (dd, *J* = 25.8, 7.2 Hz), 64.36 (d, *J* = 167.4 Hz), 29.99 (t, *J* = 5.5 Hz), 27.33 (d, *J* = 3.1 Hz), 22.01, 13.57 (d, *J* = 37.3 Hz). **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.74. **HRMS** (ESI-TOF) m/z: calcd for $C_{16}H_{29}O_5PNa$ $[M+Na]^+$ 355.1635; found 355.1635.



diheptyl (hydroxy(5-methylfuran-2-yl)methyl)phosphonate (1h)

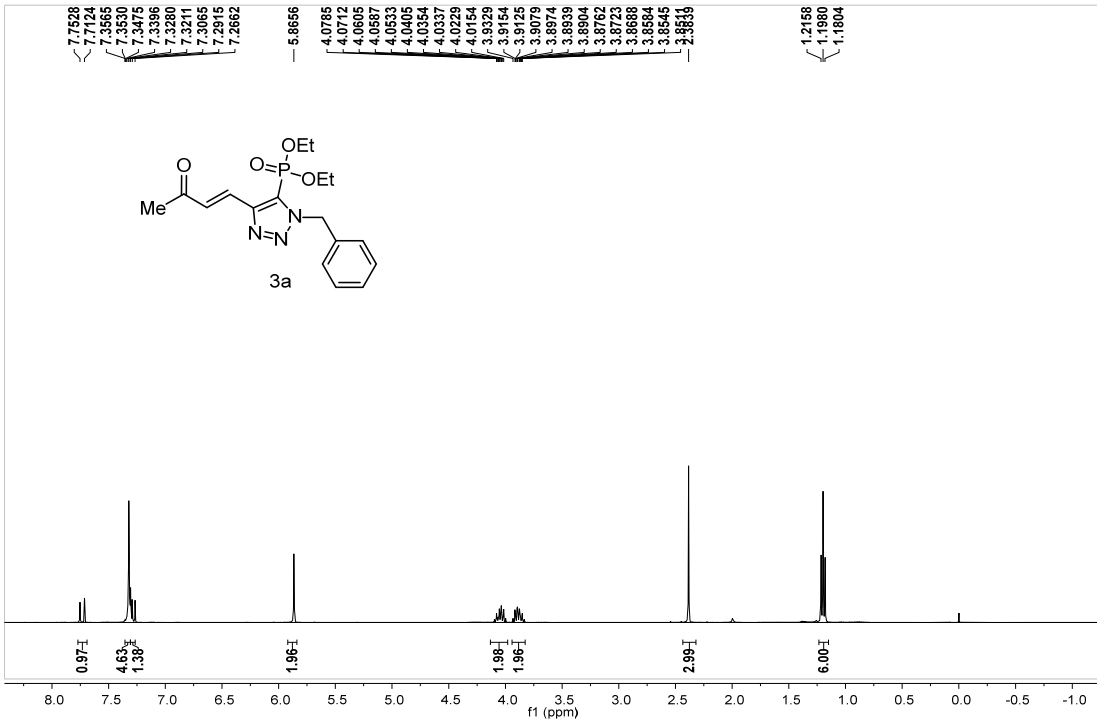
Isolated by column chromatography over silica gel (eluent, 6:1 petroleum ether/ethyl acetate) 0.91 mmol 5-methyl furfuraldehyde and diheptyl phosphonates (1.2 equiv) were used as starting materials to afford **1g** as a yellow oil (317 mg, 90% yield)**^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.39 (t, 1H), 5.94 (d, *J* = 2.7 Hz, 2H), 4.94 (d, *J* = 13.2 Hz, 2H), 4.17 – 3.95 (m, 4H), 2.28 (s, 3H), 1.73 – 1.53 (m, 4H), 1.29 (m, 17H) (t, 6H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 152.39, 148.14, 110.23 (d, *J* = 6.2 Hz), 106.60, δ67.21 (dd, *J* = 26.1, 7.2 Hz), 64.46 (d, *J* = 167.2 Hz), 31.62, 30.48, 30.42, 30.37, 28.74, 25.26 (d, *J* = 3.2 Hz), 22.48, 13.97, 13.49. **^{31}P NMR** (162 MHz, $CDCl_3$) δ 3.80. **HRMS** (ESI-TOF) m/z: calcd for $C_{20}H_{37}O_5PNa$ $[M+Na]^+$ 422.2271; found 422.2251.

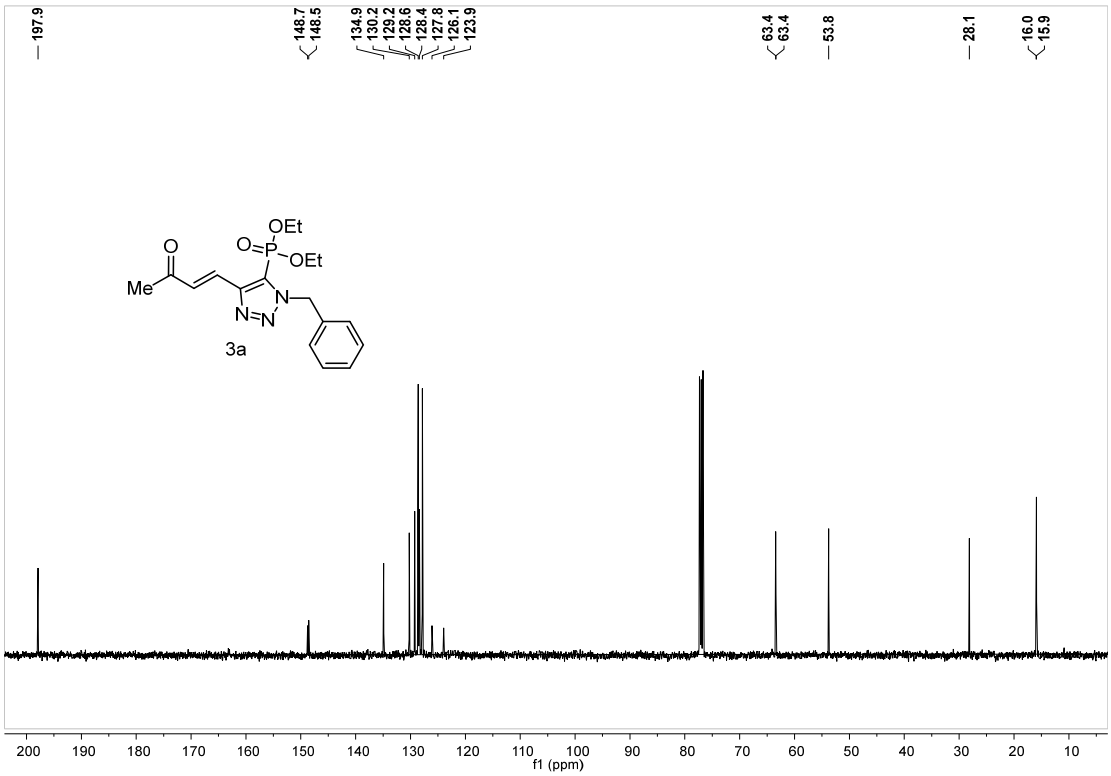


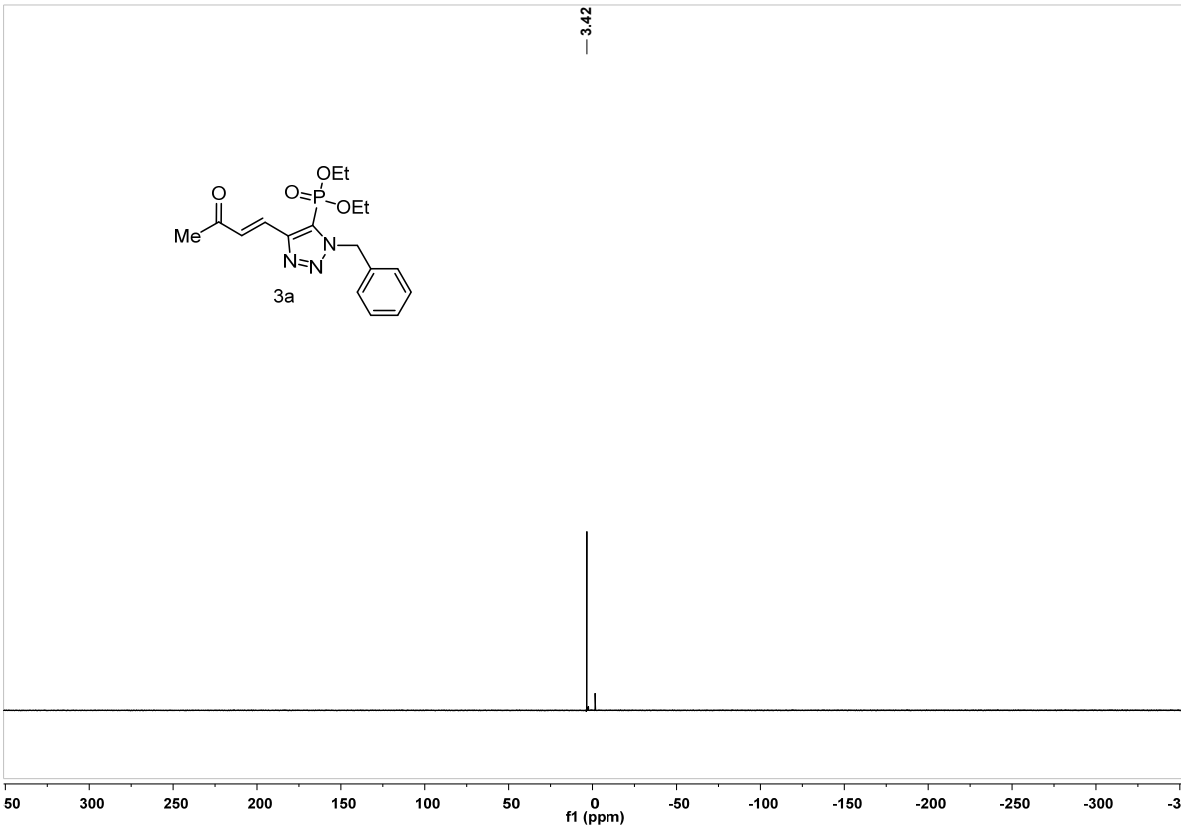
Diphenyl (hydroxy(5-methylfuran-2-yl)methyl)phosphonate(1i)

Isolated by column chromatography over silica gel (eluent, 4:1 petroleum ether/ethyl acetate) 0.91 mmol 5-methyl furfuraldehyde and diphenyl phosphonates (1.2 equiv) were used as starting materials to afford **1g** as a yellow oil (161 mg, 51% yield). **^{1}H NMR** (400 MHz,) δ 7.30 – 7.17 (m, 4H), 7.17 – 6.96 (m, 6H), 6.39 (t, 1H), 5.88 (d, *J* = 2.7 Hz, 1H), 5.17 (d, *J* = 12.6 Hz, 1H), 2.17 (s, 3H). **^{13}C NMR** (101 MHz,) δ 153.45, 150.36 (d, *J* = 12.8 Hz), 146.58, δ129.75 (d, *J* = 7.9 Hz), 125.40 (d, *J* = 9.2 Hz), 120.79 (d, *J* = 3.9 Hz), 120.63 (d, *J* = 3.9 Hz), 111.61, 107.05, 64.55 (d, *J* = 169.8 Hz), 13.63. **^{31}P NMR** (162 MHz, $CDCl_3$) δ 12.69. **HRMS** (ESI-TOF) m/z: calcd for $C_{18}H_{17}N_3O_5PNa$ $[M+Na]^+$ 367.0706; found 367.0709.

VII. Copies of ^{1}H, ^{13}C and ^{31}P NMR Spectra for products and substrates



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O
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Me
N
N=N
3a
50
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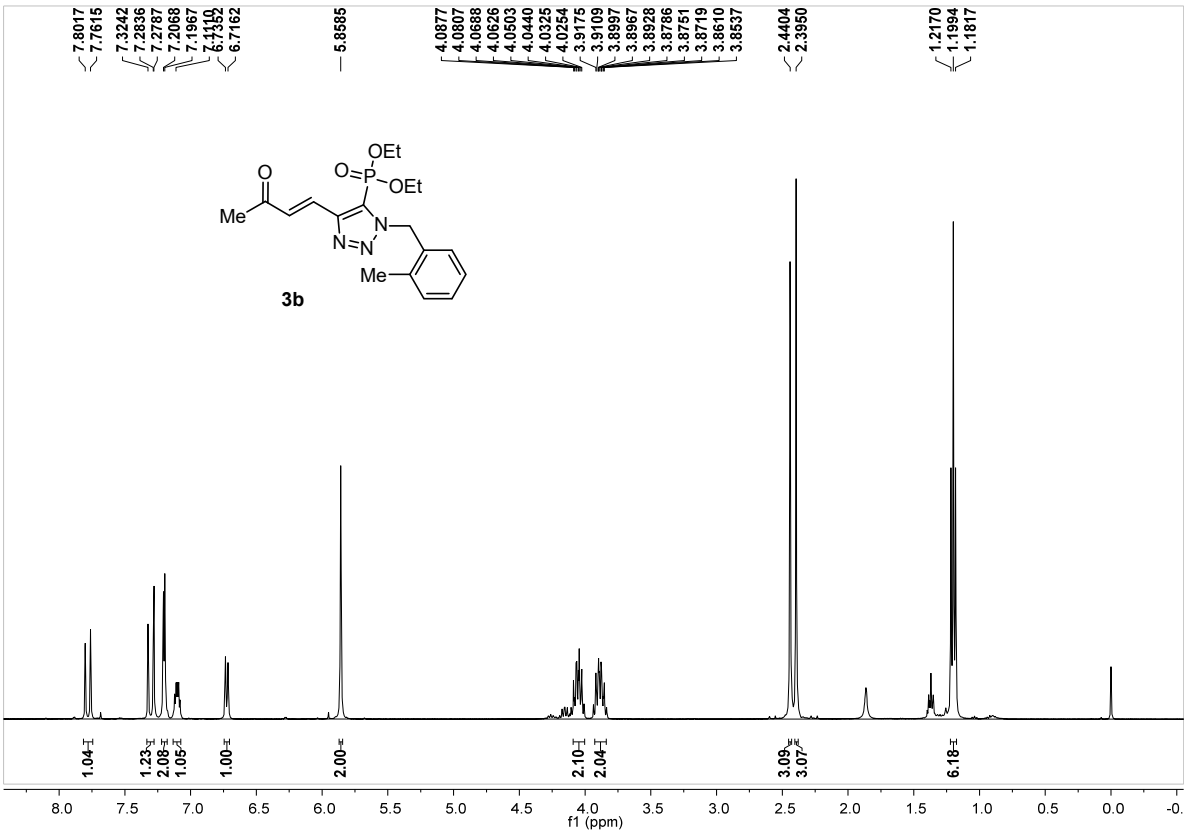
200
150
100
50
0
-50
-100
-150
-200
-250
-300
-3
f1 (ppm)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7.2787
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7.1967
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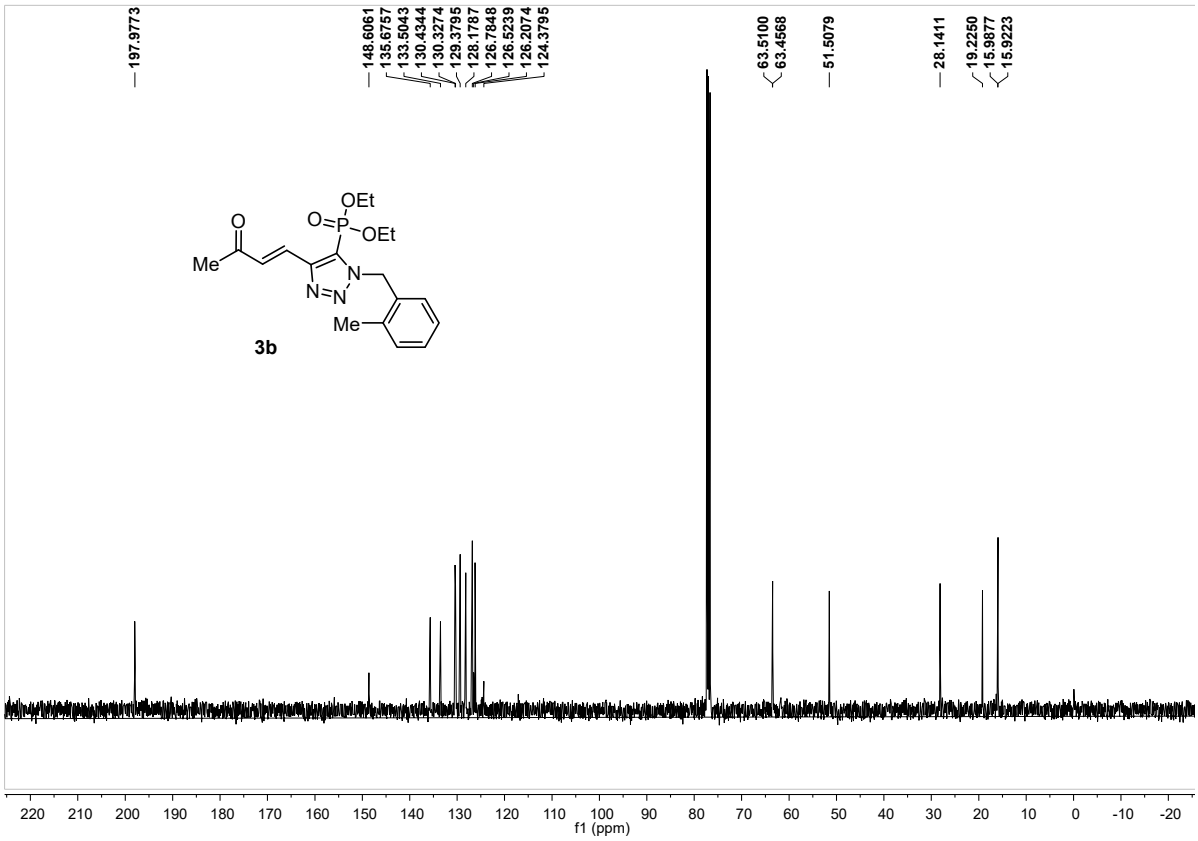
7.1352
6.7162
5.8585
4.0877
4.0807
4.0626
4.0503
4.0440
4.0325
4.0254
3.9175
3.9109
3.8997
3.8967
3.8928
3.8786
3.8751
3.8719
3.8610
3.8537
2.4404
2.3950
1.2170
1.1994
1.1817
OEt
O
O=P
OEt
Me
N
N=N
Me
3b
1.04
1.23
2.08
1.05
1.00
2.00
2.10
2.04
3.09
3.07
6.18
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)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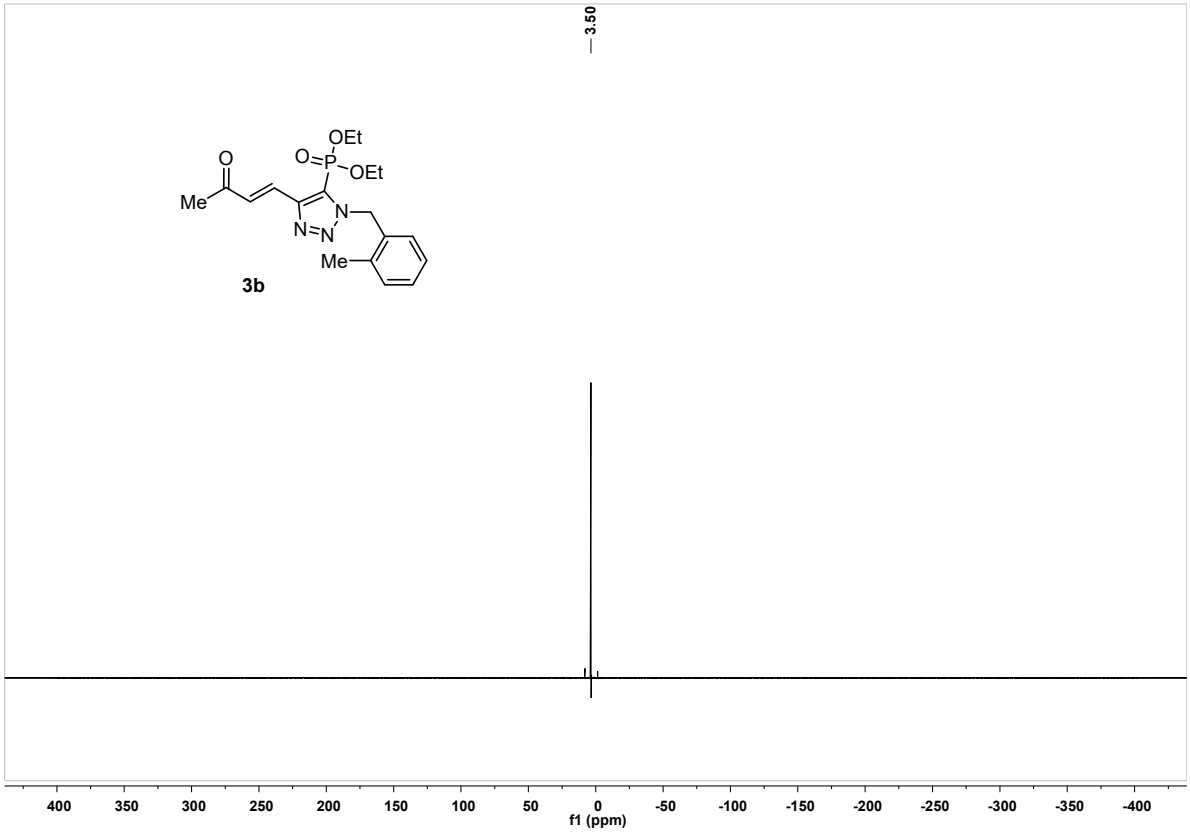
135.6757
133.5043
130.4344
130.3274
129.3795
128.1787
126.7848
126.5239
126.2074
124.3795
63.5100
63.4568
51.5079
28.1411
19.2250
15.9877
15.9223
OEt
O
O=P
OEt
Me
N
N=N
Me
3b
f1 (ppm)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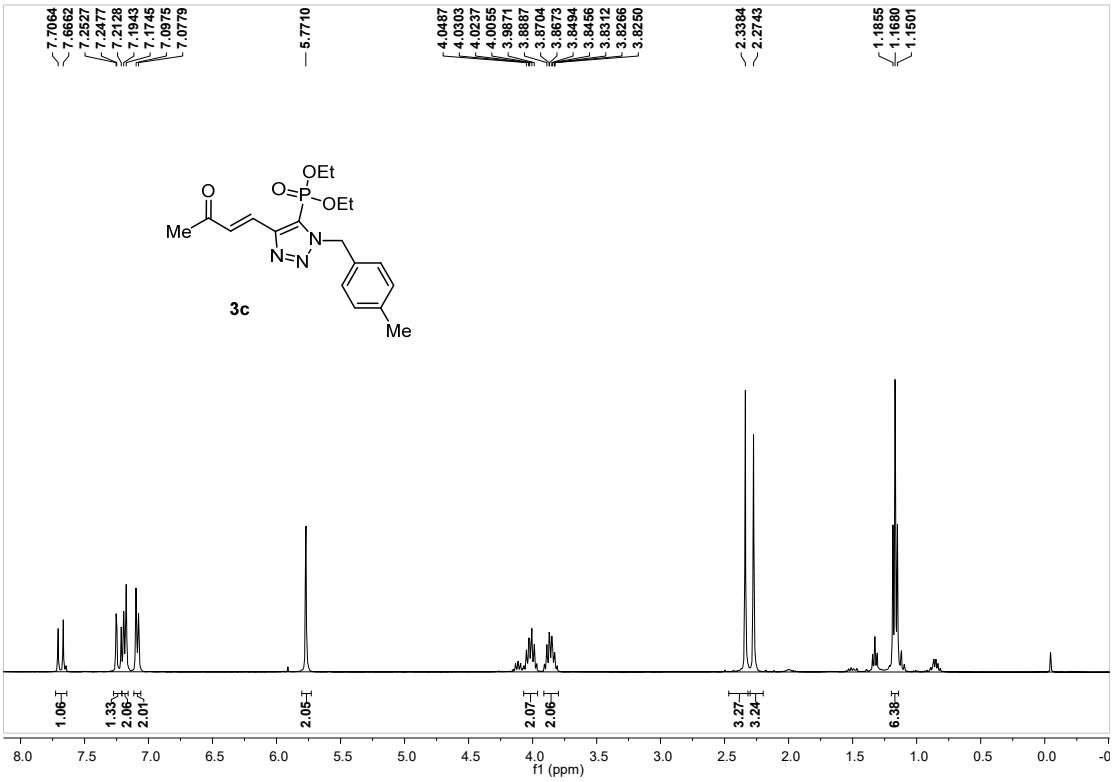
OEt
Me
N
N=N
Me
3b
f1 (ppm)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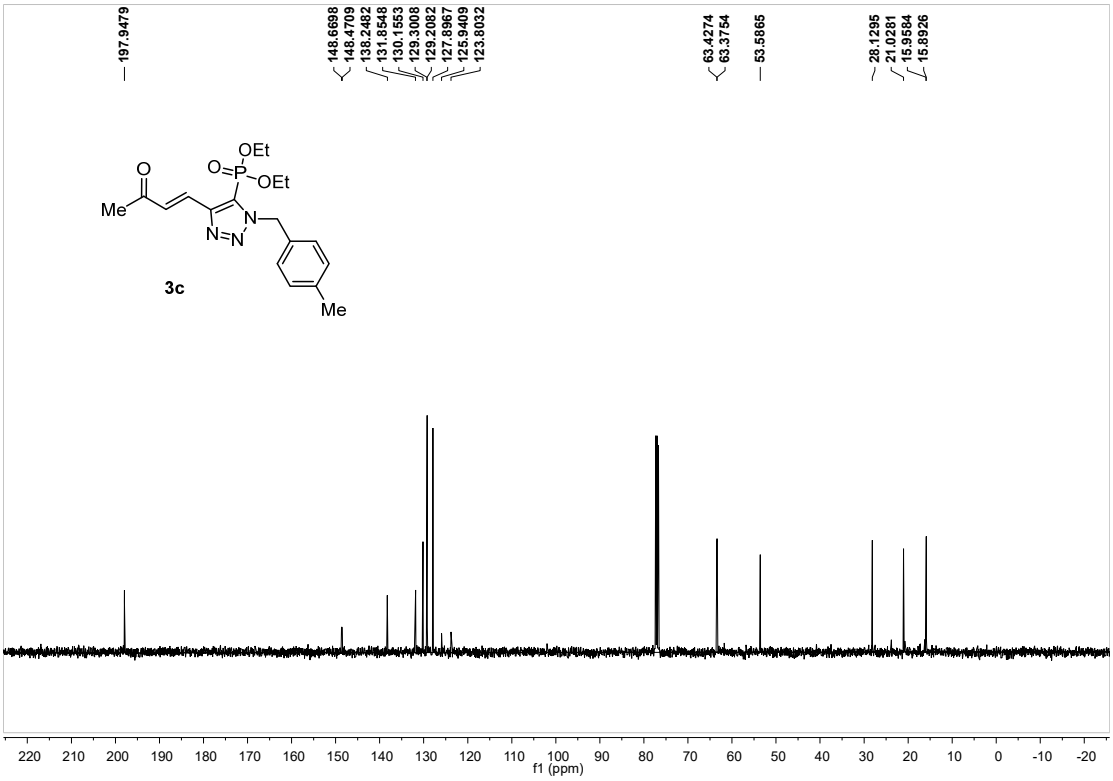
7.6662
7.2527
7.2477
7.2128
7.1943
7.1745
7.0975
7.0779
5.7710
4.0487
4.0303
4.0237
4.0055
3.9871
3.8887
3.8704
3.8673
3.8494
3.8456
3.8312
3.8266
3.8250
2.3384
2.2743
1.1855
1.1680
1.1501
OEt
O
P
OEt
Me
N
N=N
3c
Me
1.06
1.33
2.06
2.01
2.05
2.07
2.06
3.27
3.24
6.38
f1 (ppm)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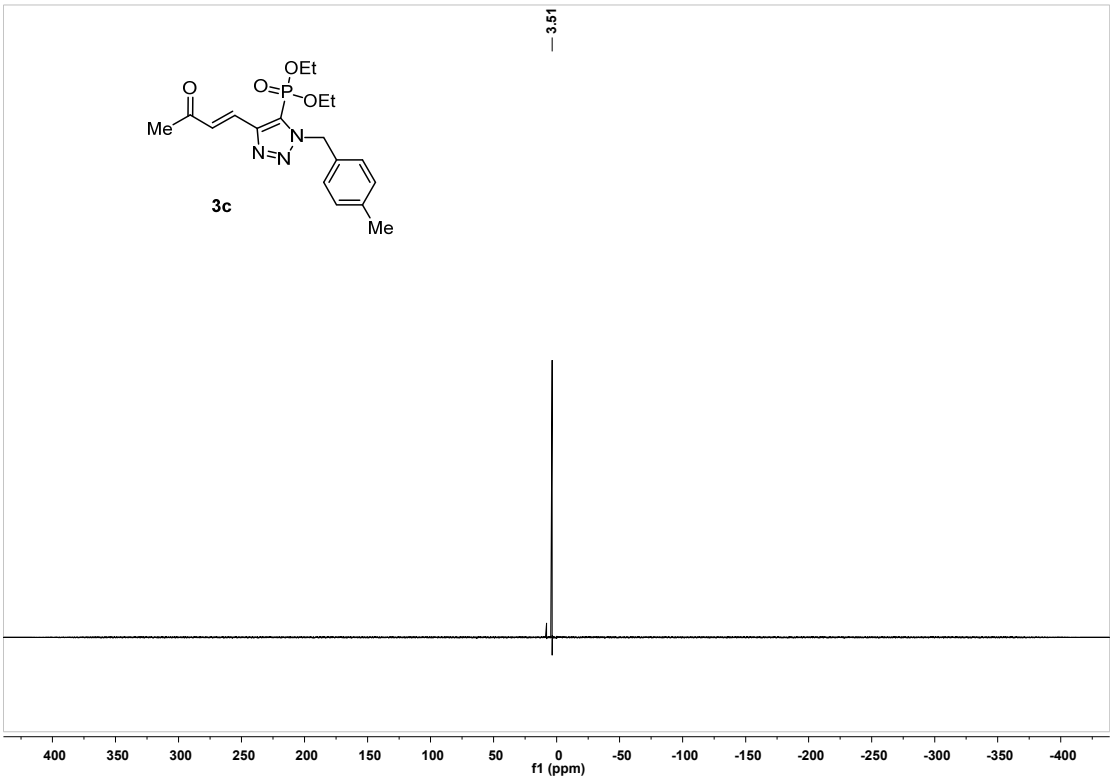
130.1553
129.3008
129.2082
127.8967
125.9409
123.8032
63.4274
63.3754
53.5865
28.1295
21.0281
15.9584
15.8926
OEt
O
P
OEt
Me
N
N=N
3c
Me
f1 (ppm)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O
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OEt
Me
N
N=N
Me
3c
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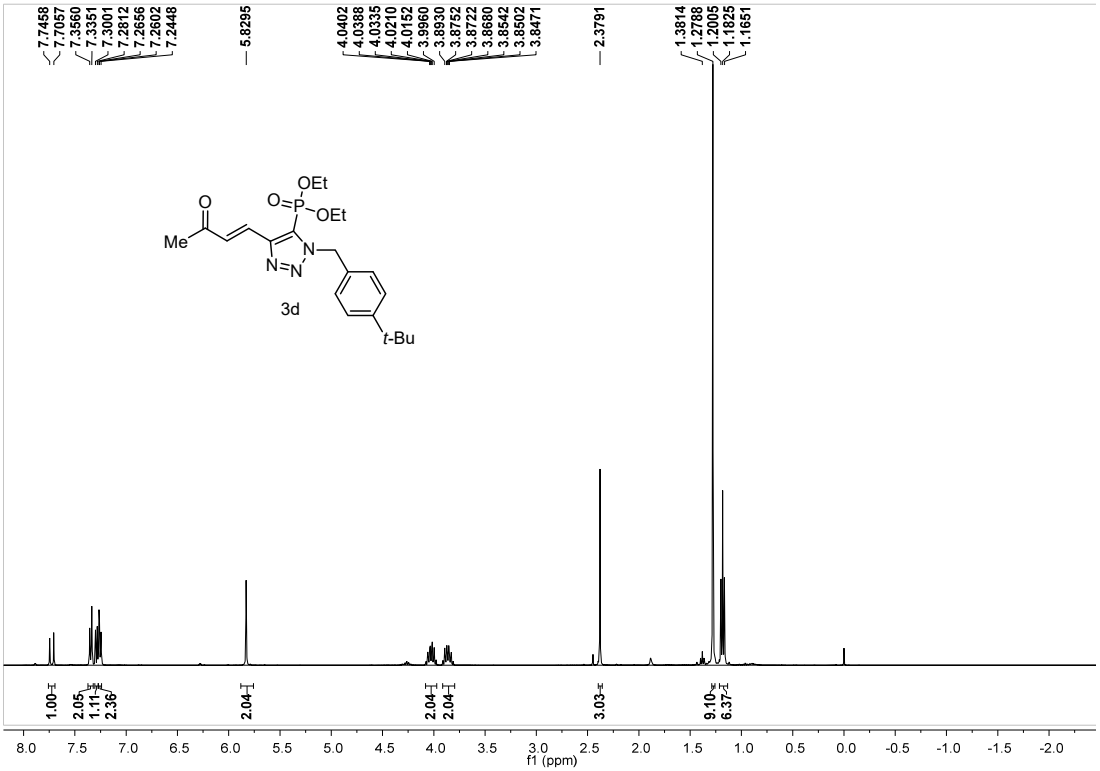
350
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)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7.3560
7.3351
7.3001
7.2812
7.2656
7.2602
7.2448
5.8295
4.0402
4.0388
4.0335
4.0210
4.0152
3.9960
3.8930
3.8752
3.8722
3.8680
3.8542
3.8502
3.8471
2.3791
1.3814
1.2788
1.2005
1.1825
1.1651
OEt
O
O=P
OEt
Me
N
N=N
3d
t-Bu
1.00
2.05
1.11
2.36
2.04
2.04
2.04
3.03
9.10
6.37
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
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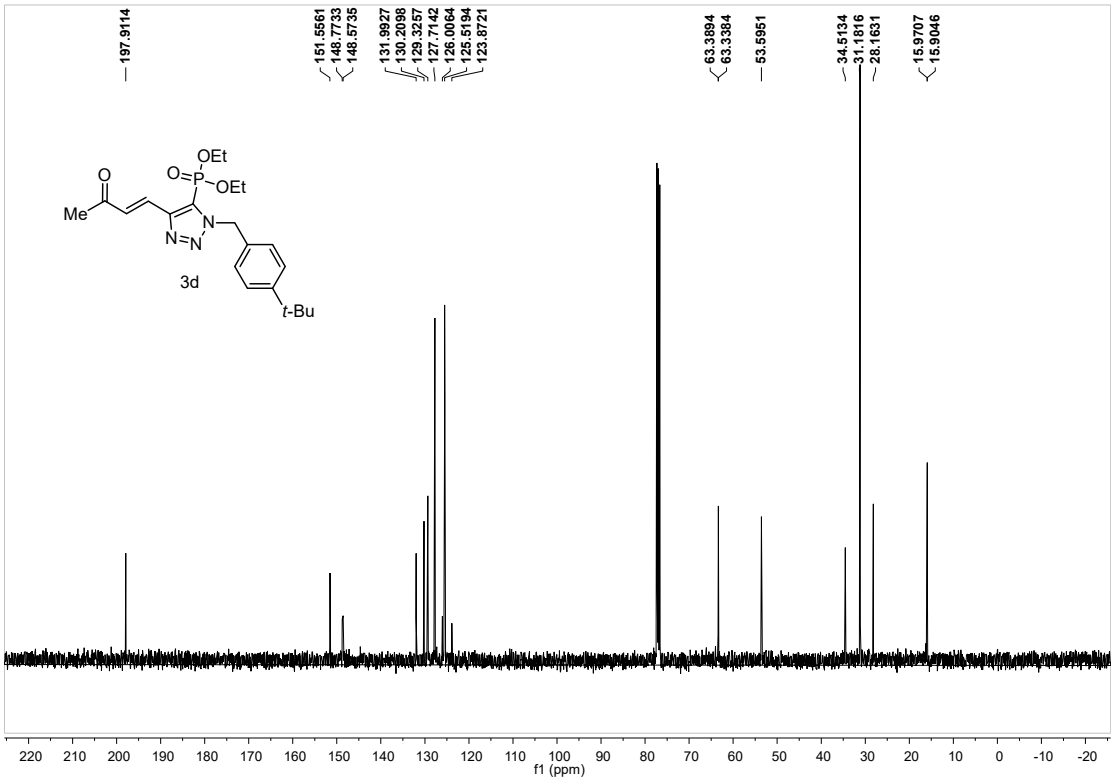
0.0
-0.5
-1.0
-1.5
-2.0
f1 (ppm)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129.3257
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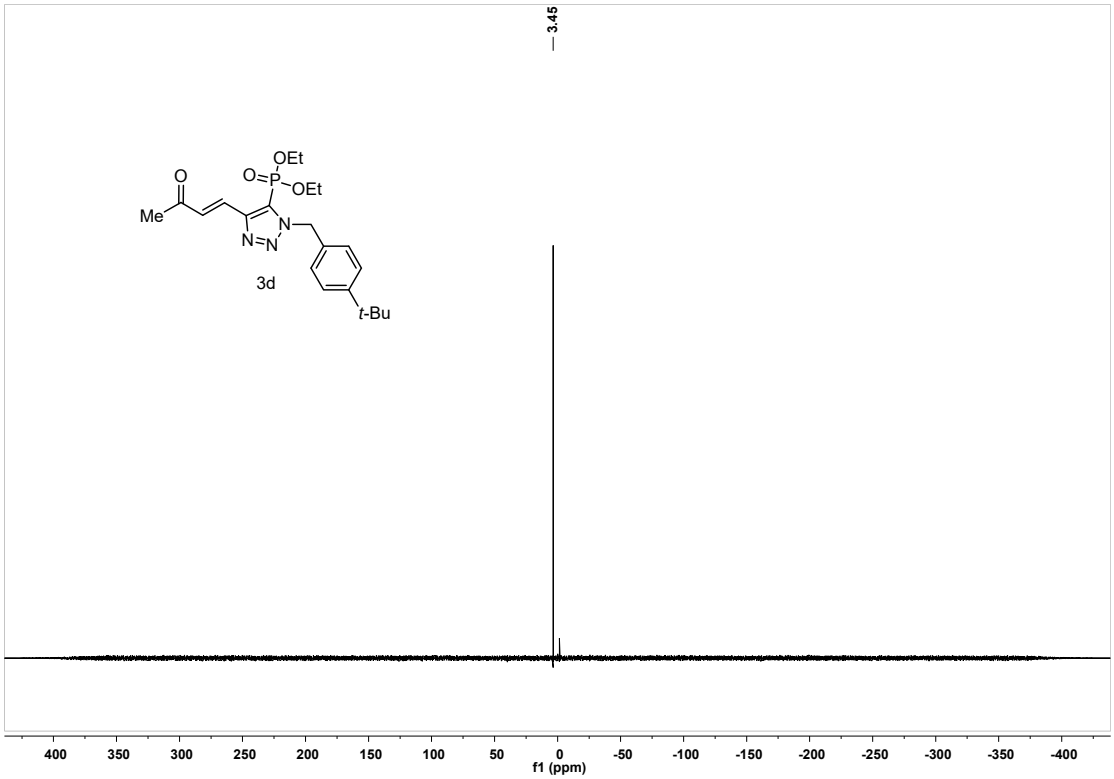
125.5194
123.8721
63.3894
63.3384
53.5951
34.5134
31.1816
28.1631
15.9707
15.9046
OEt
O
O=P
OEt
Me
N
N=N
3d
t-Bu
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)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OEt
Me
N
N=N
3d
t-Bu
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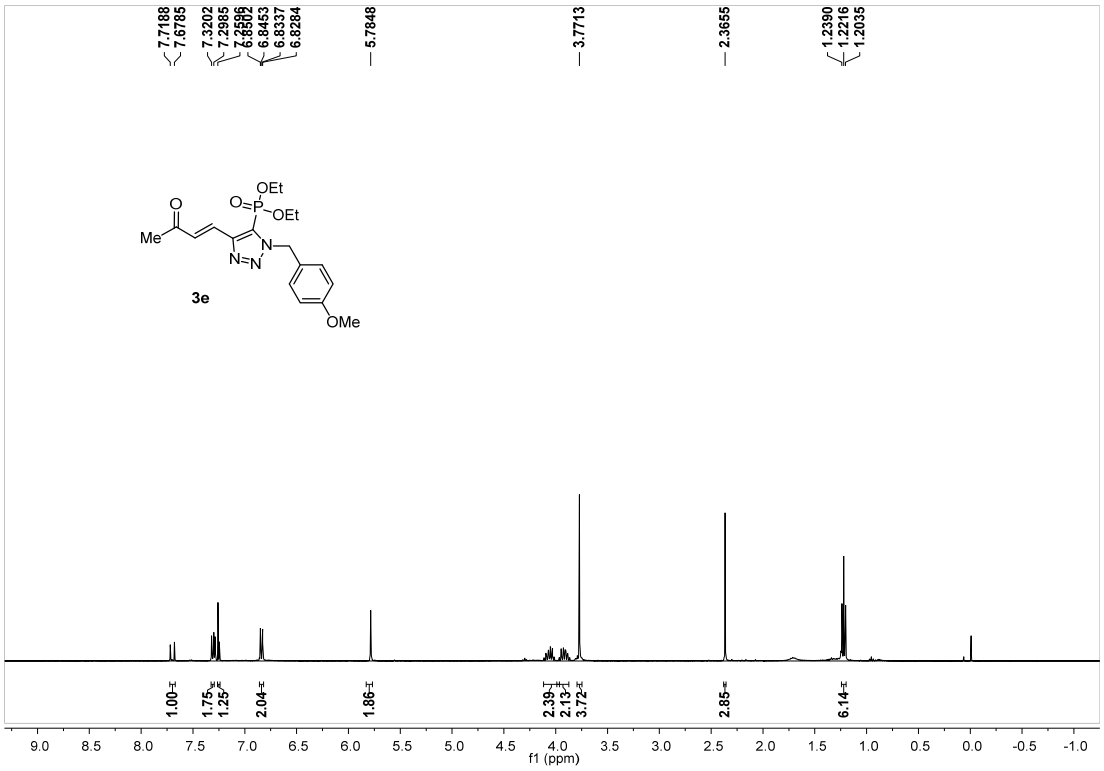
f1 (ppm)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7.2985
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6.8453
6.8337
6.8284
5.7848
3.7713
2.3655
1.2390
1.2216
1.2035
OEt
O
P
OEt
Me
N
N
N
3e
OMe
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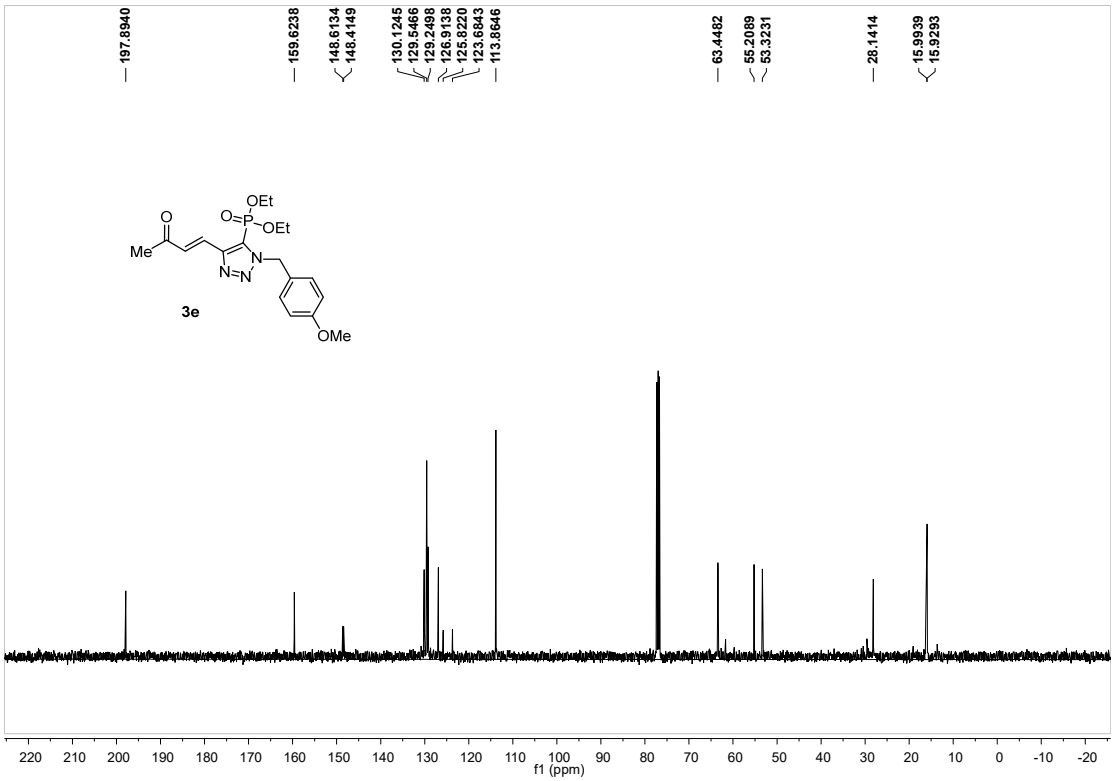
1.25
2.04
1.86
2.39
2.13
3.72
2.85
6.14
f1 (ppm)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129.5466
129.2498
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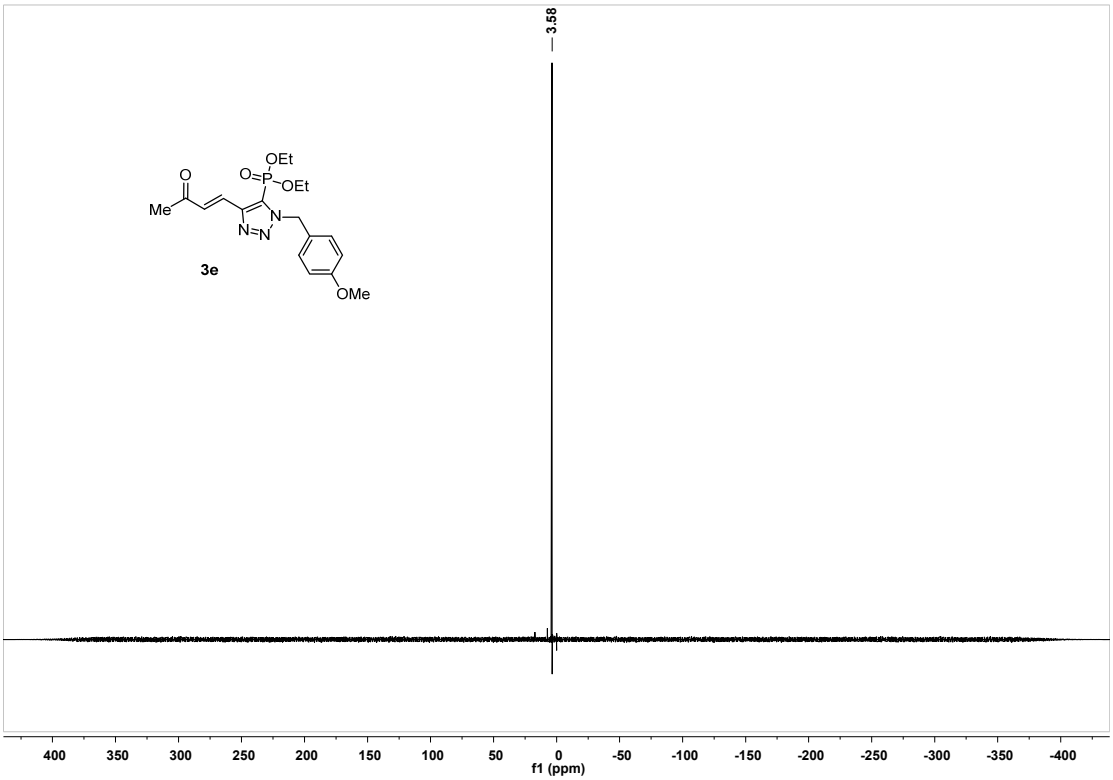
125.8220
123.6843
113.8646
63.4482
55.2089
53.3231
28.1414
15.9939
15.9293
OEt
O
P
OEt
Me
N
N
N
3e
OMe
f1 (ppm)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O
O=P
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N
N=N
OMe
3e
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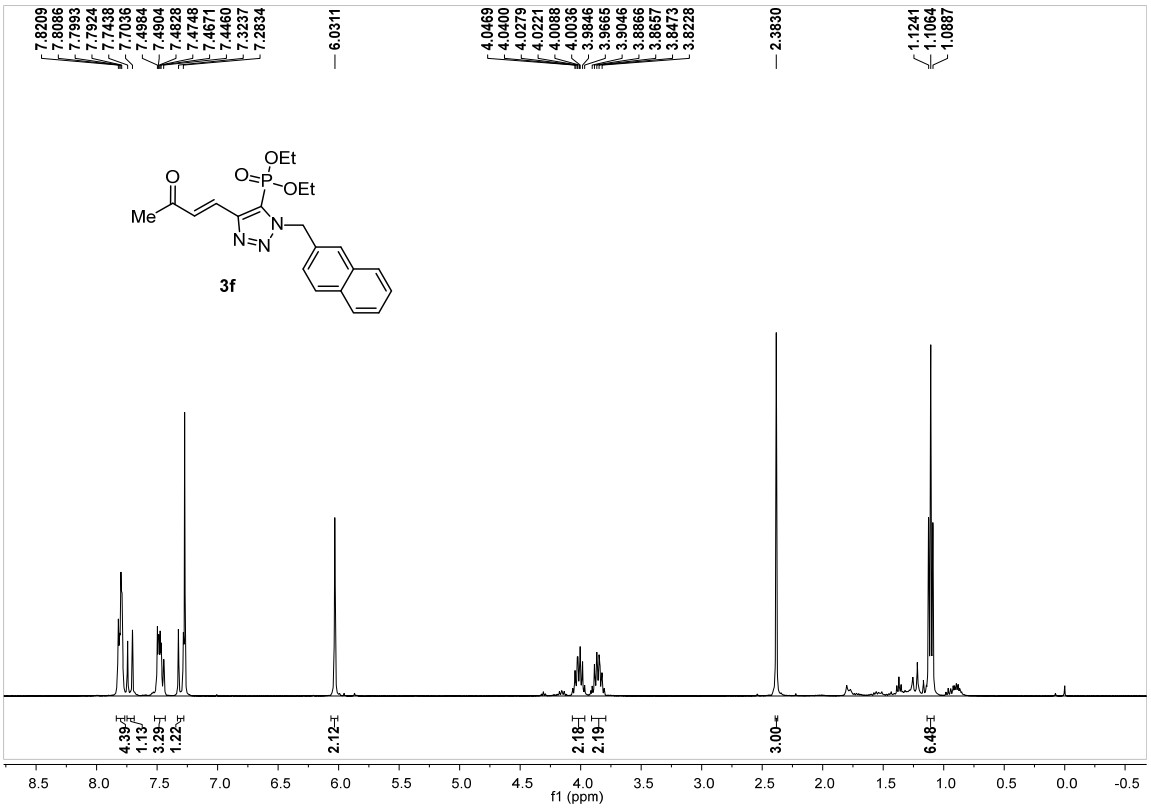
f1 (ppm)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7.7438
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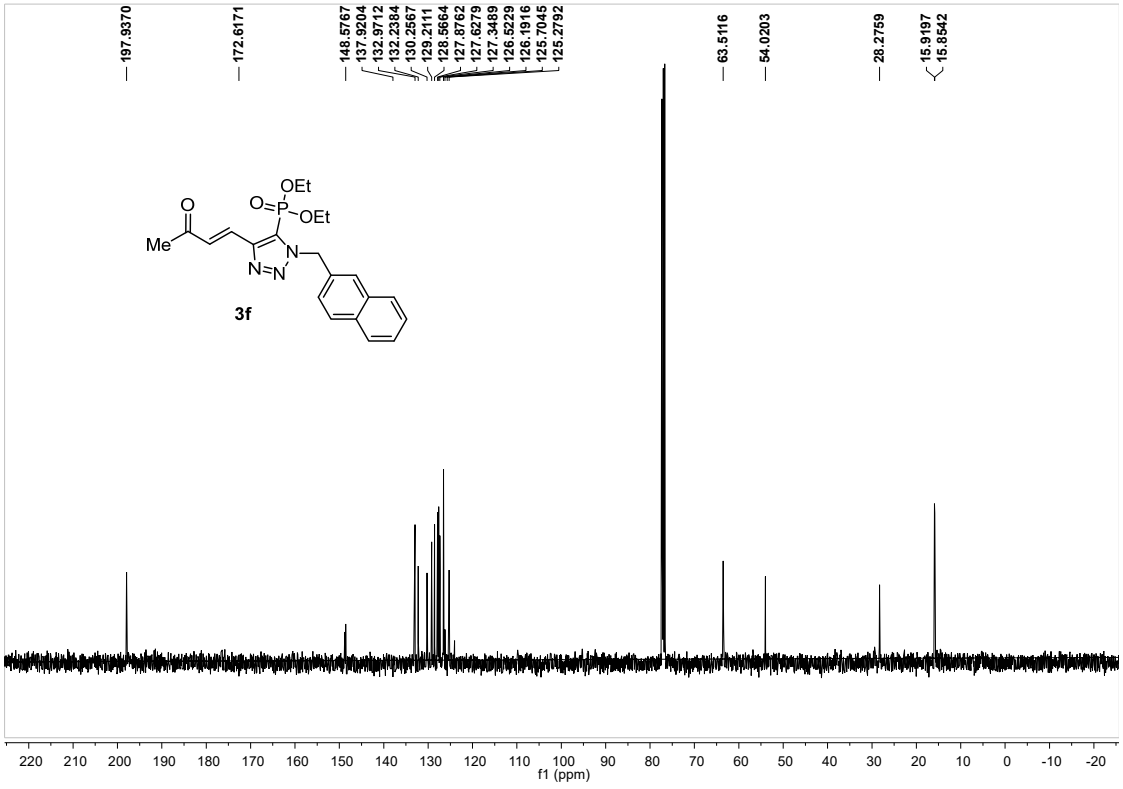
7.4671
7.4460
7.3237
7.2834
6.0311
4.0469
4.0400
4.0279
4.0221
4.0088
4.0036
3.9846
3.9665
3.9046
3.8866
3.8657
3.8473
3.8228
2.3830
1.1241
1.1064
1.0887
OEt
O
O=P
OEt
Me
N
N=N
3f
4.39
1.13
3.29
1.22
2.12
2.18
2.19
3.00
6.48
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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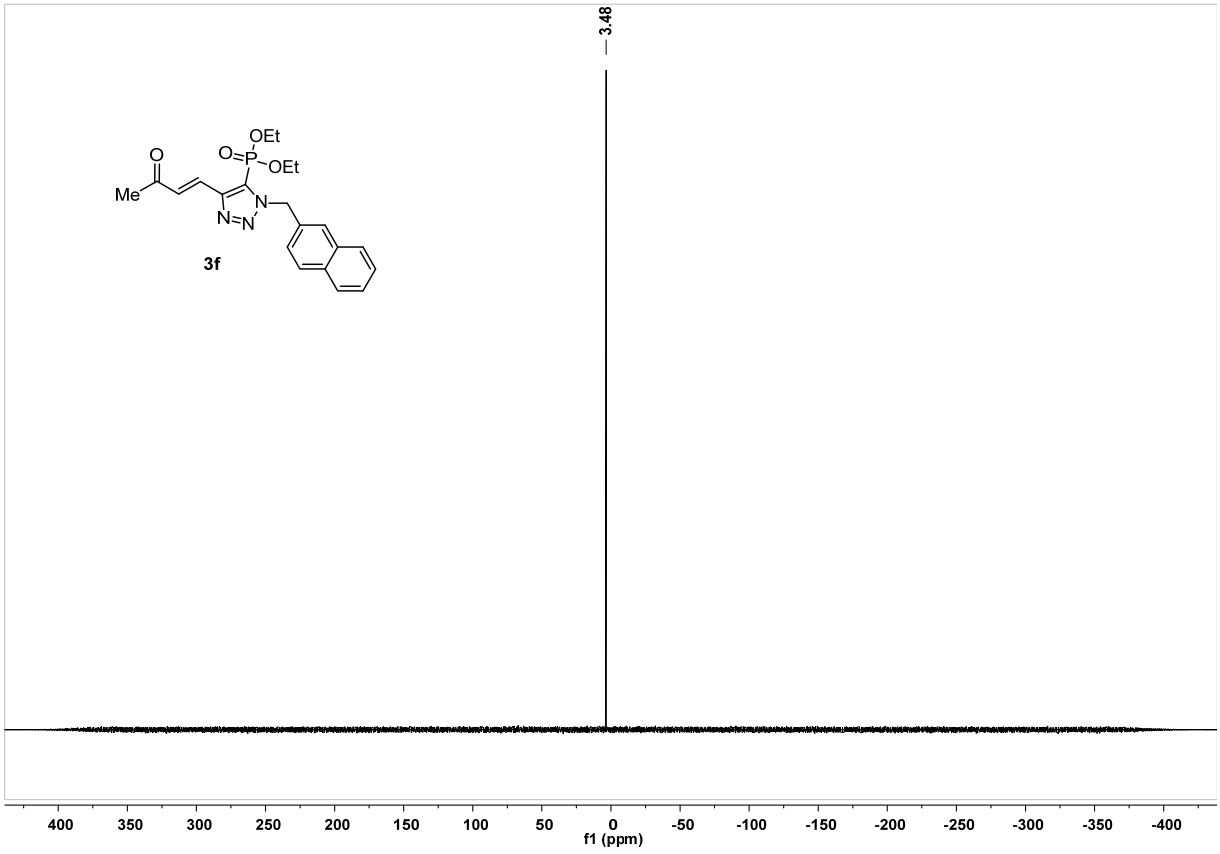
127.3489
126.5229
126.1916
125.7045
125.2792
63.5116
54.0203
28.2759
15.9197
15.8542
OEt
O
O=P
OEt
Me
N
N=N
3f
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)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OEt
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Me
N
N=N
3f
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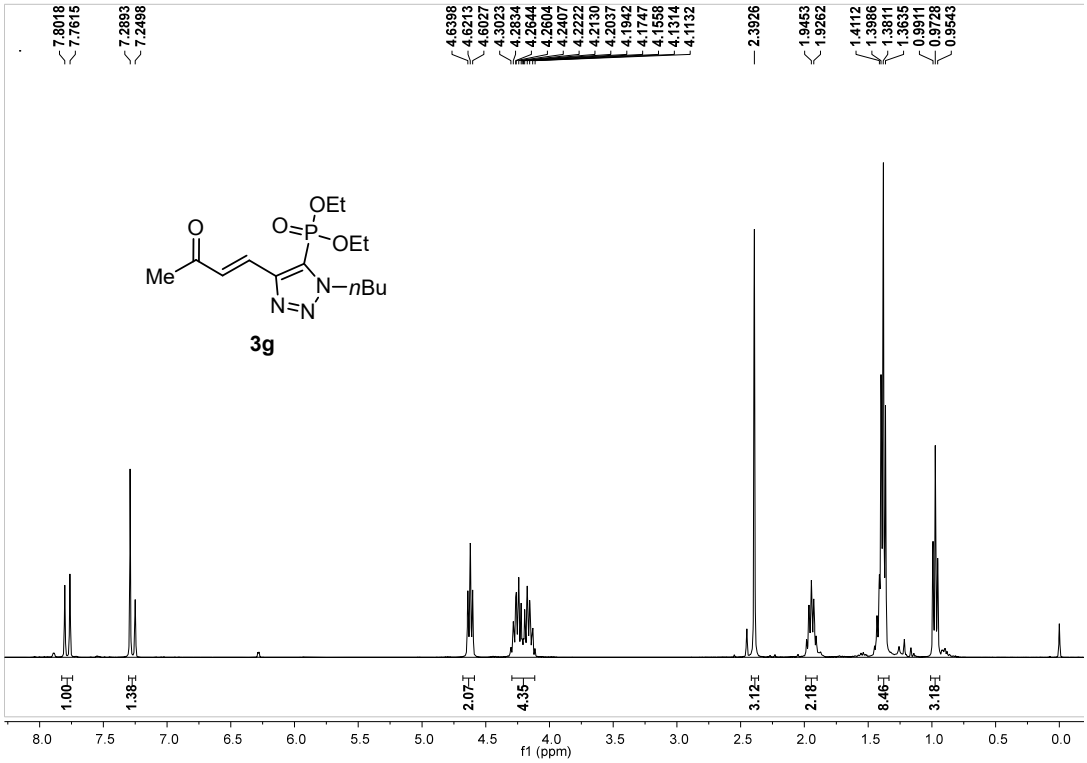
f1 (ppm)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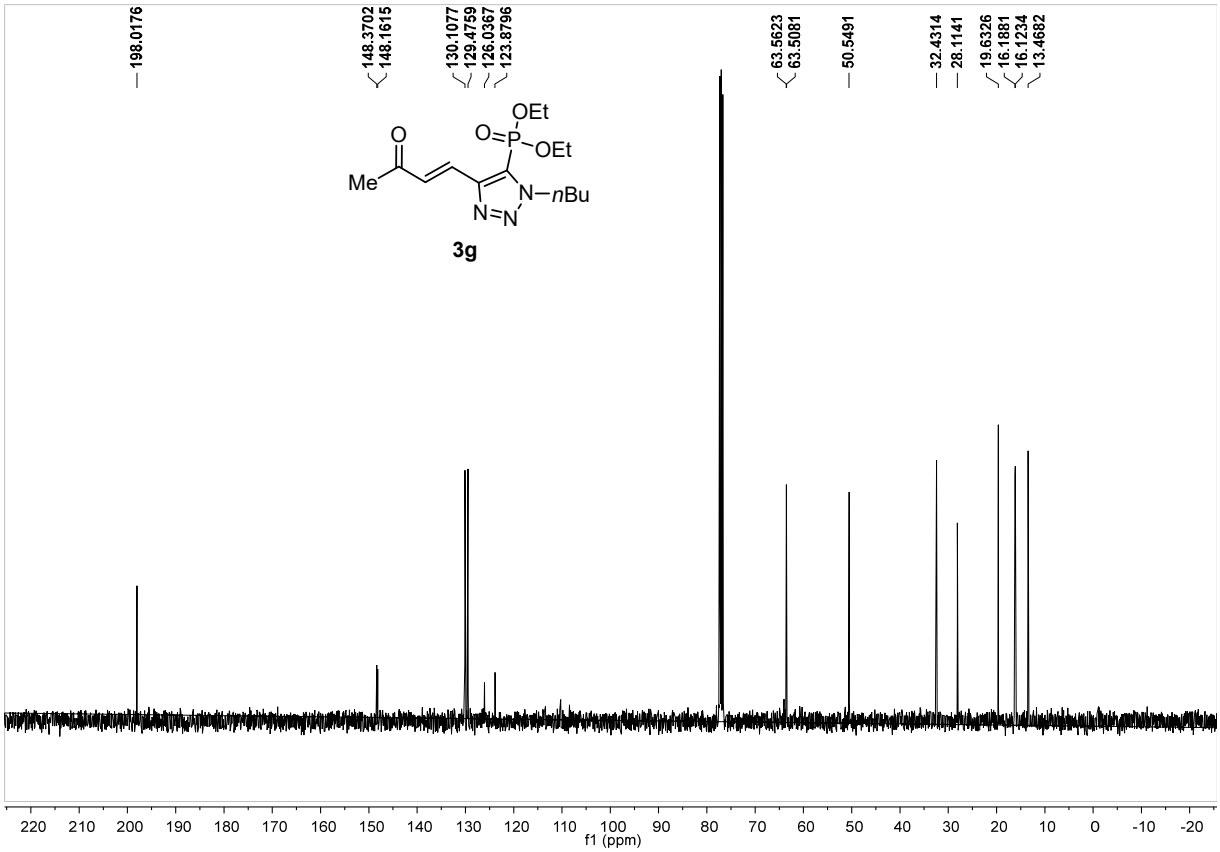
4.2604
4.2407
4.2222
4.2130
4.2037
4.1942
4.1747
4.1558
4.1314
4.1132
2.3926
1.9453
1.9262
1.4112
1.3986
1.3811
1.3635
0.9911
0.9728
0.9543
OEt
O=P
OEt
O
Me
N–nBu
N=N
3g
1.00
1.38
2.07
4.35
3.12
2.18
8.46
3.18
f1 (ppm)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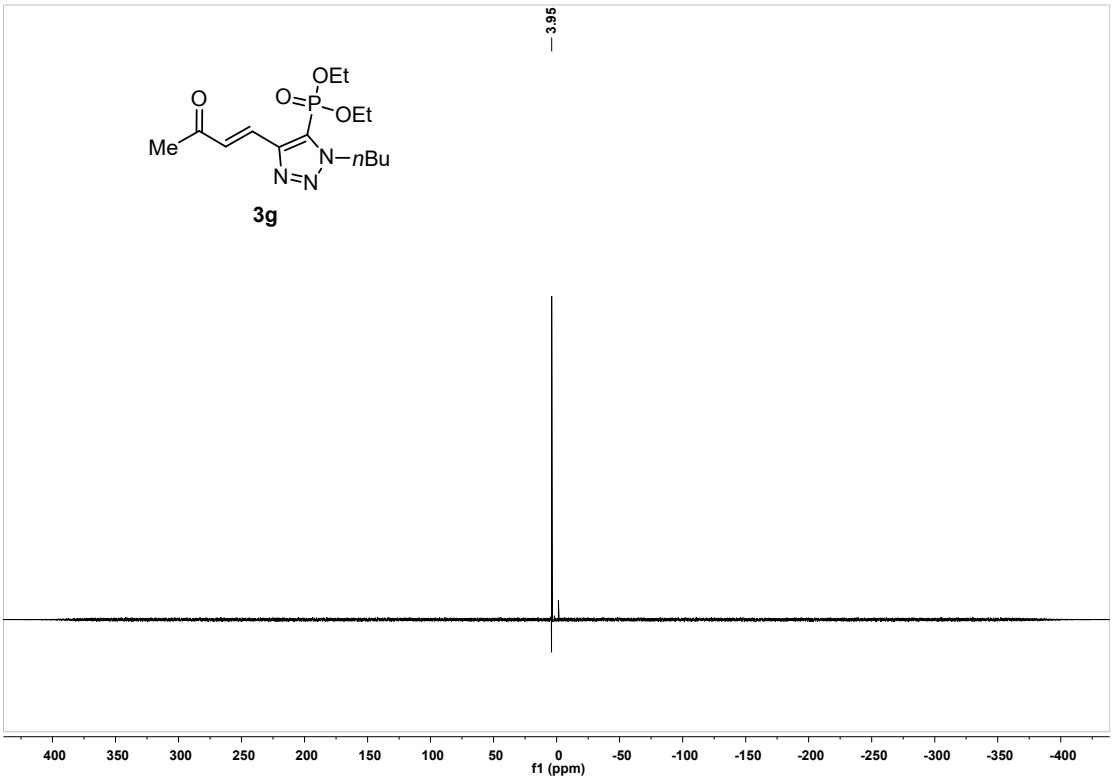
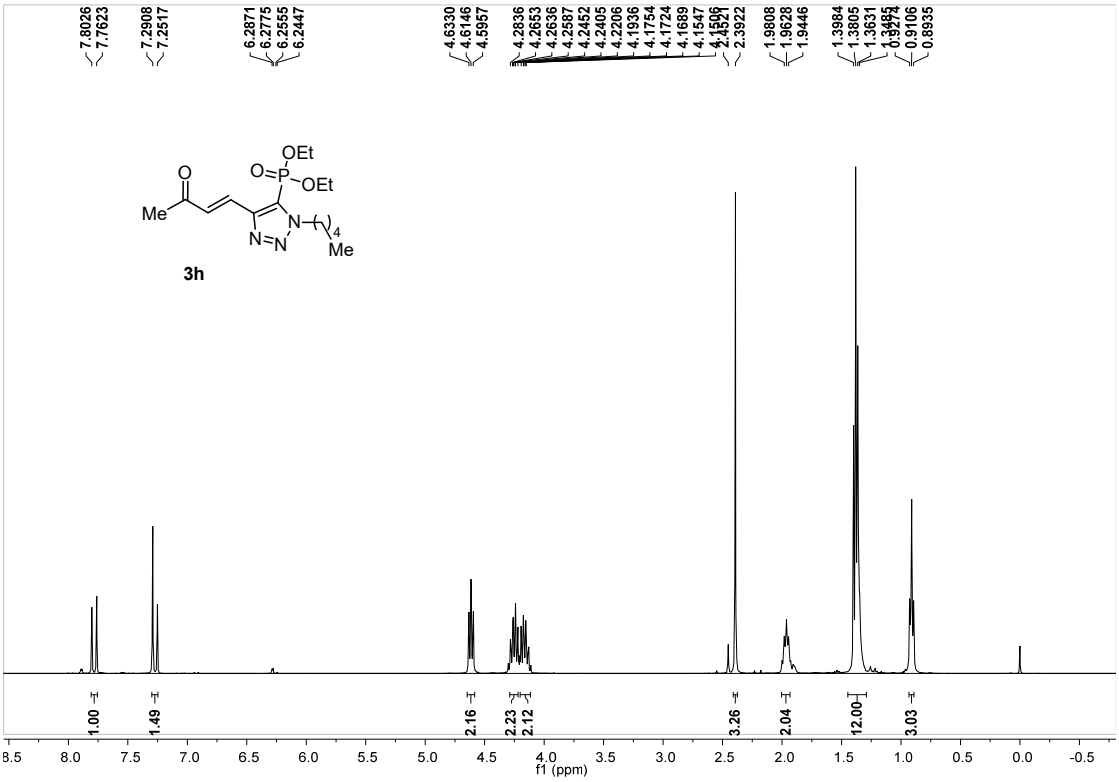
63.5081
50.5491
32.4314
28.1141
19.6326
16.1881
16.1234
13.4682
OEt
O=P
OEt
O
Me
N–nBu
N=N
3g
f1 (ppm)


3.95
OEt
O
O=P
OEt
Me
N–nBu
N=N
400 350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400
f1 (ppm)

3g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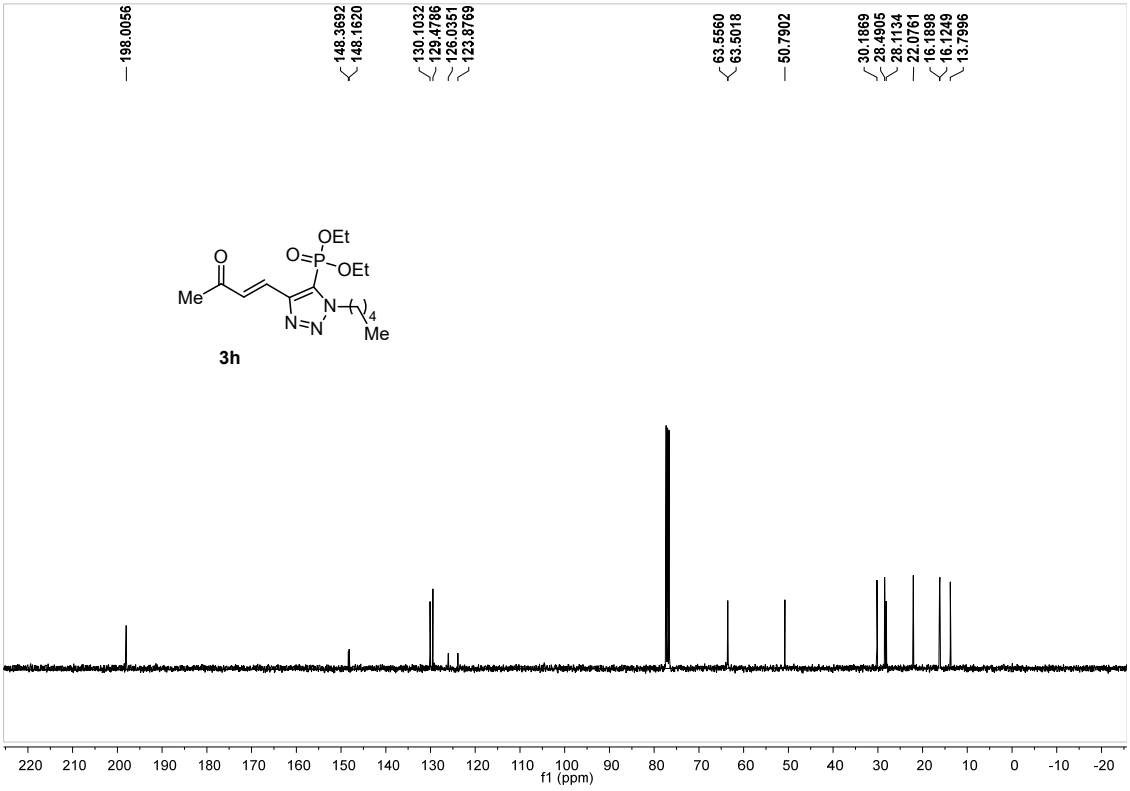
6.2775
6.2555
6.2447
4.6330
4.6146
4.5957
4.2836
4.2653
4.2636
4.2587
4.2452
4.2405
4.2206
4.1936
4.1754
4.1724
4.1689
4.1547
2.3922
1.9808
1.9628
1.9446
1.3984
1.3805
1.3631
0.9106
0.8935
OEt
O
O=P
OEt
Me
N–(CH2)4–Me
N=N
1.00
1.49
2.16
2.23
2.12
3.26
2.04
12.00
3.03
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

3h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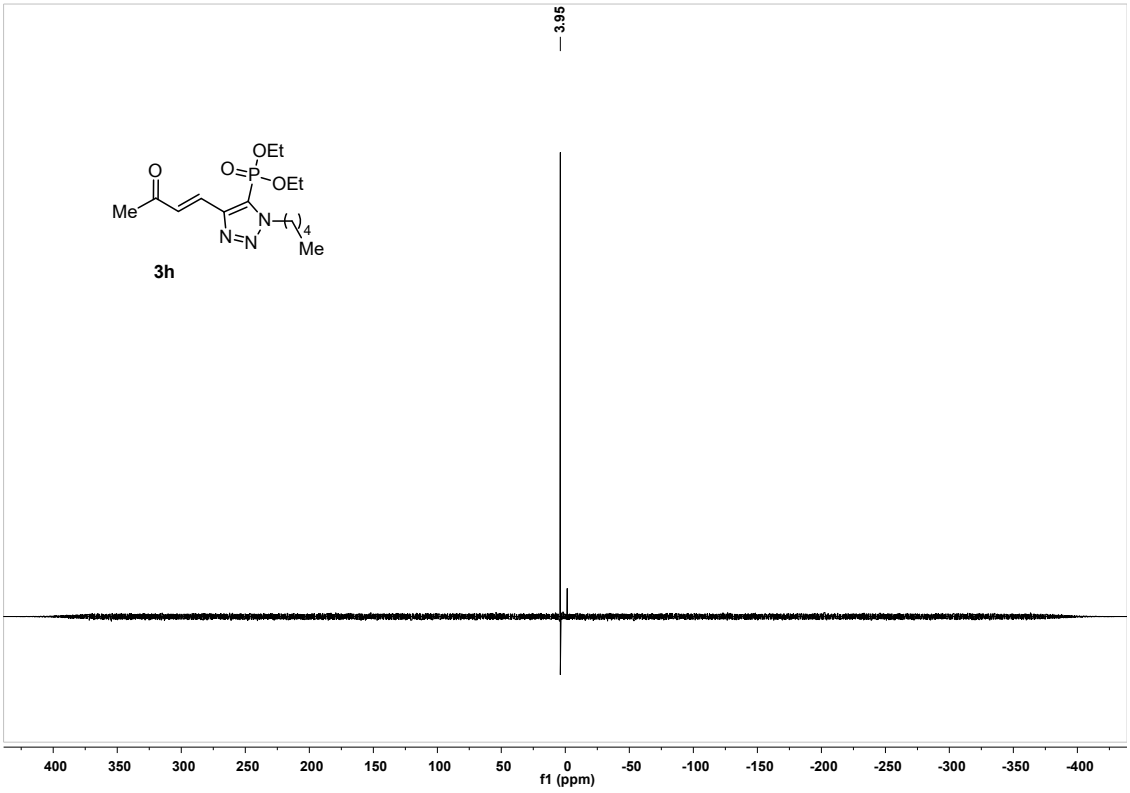
50.7902
30.1869
28.4905
28.1134
22.0761
16.1898
16.1249
13.7996
OEt
O=P
OEt
O
Me
N
N=N
4
Me
3h
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)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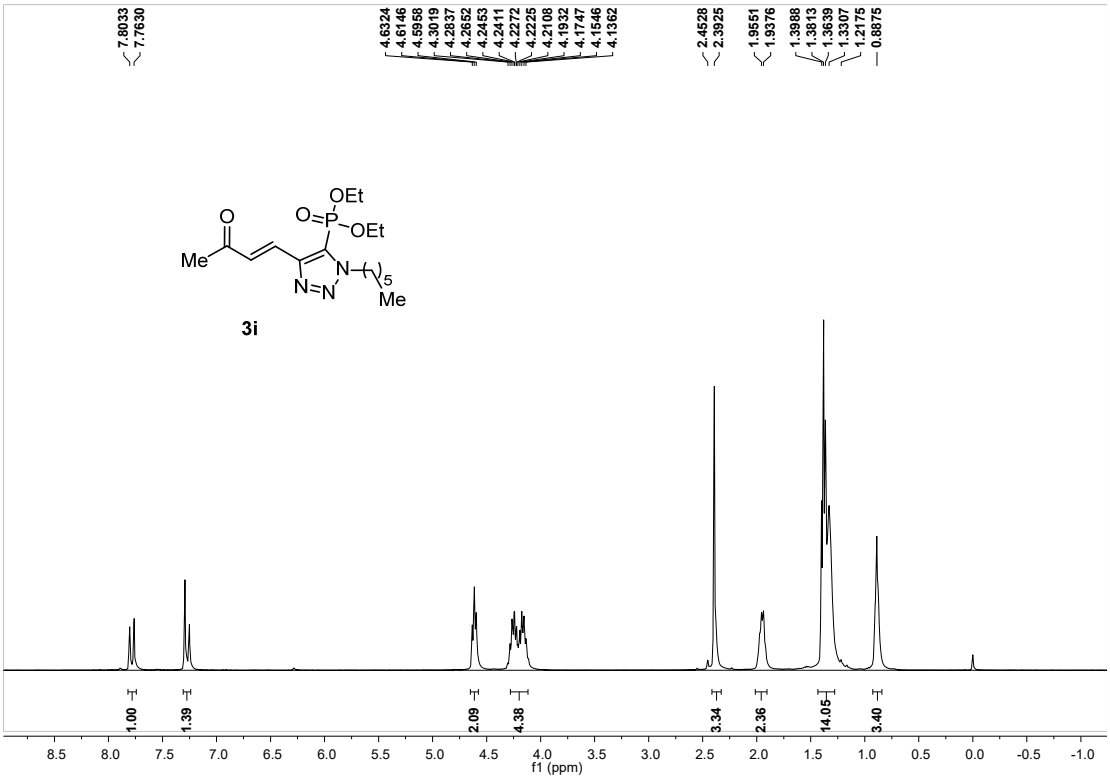
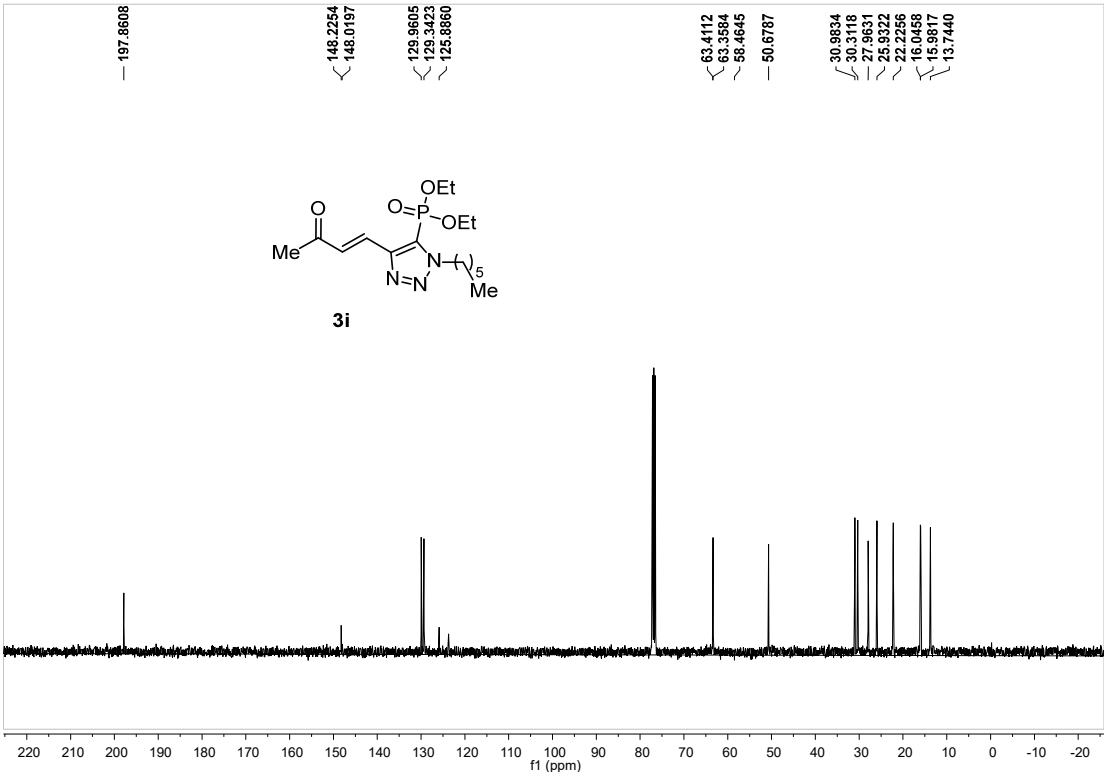
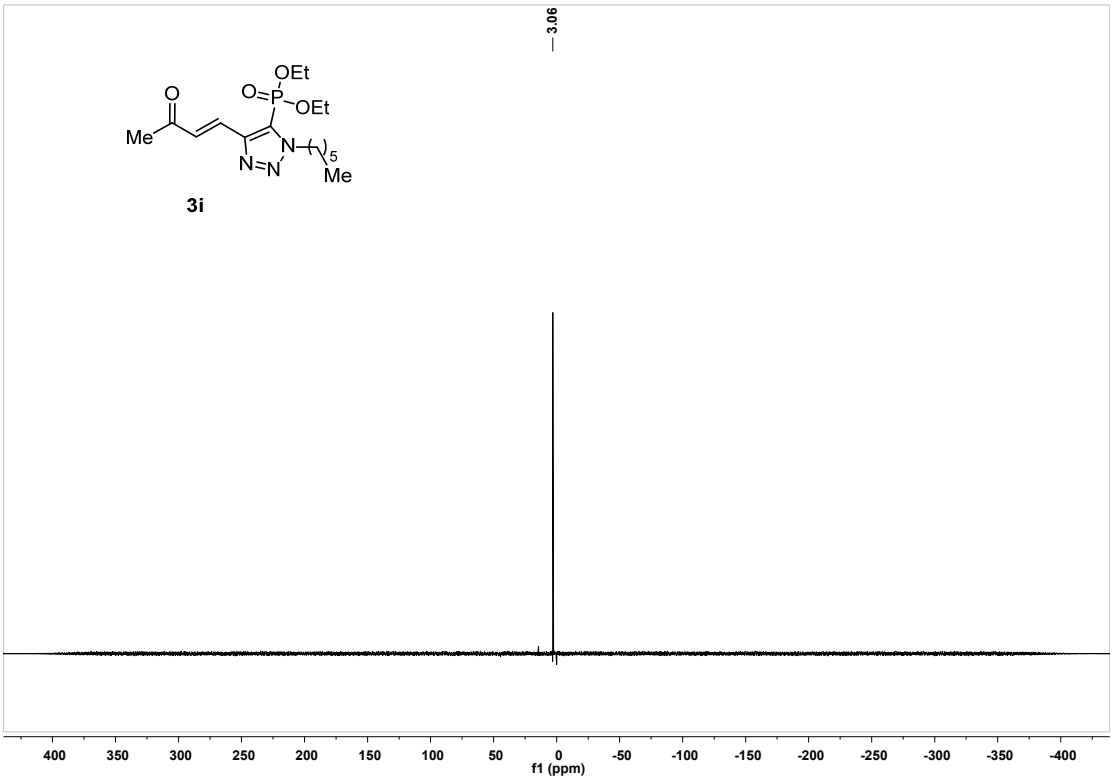
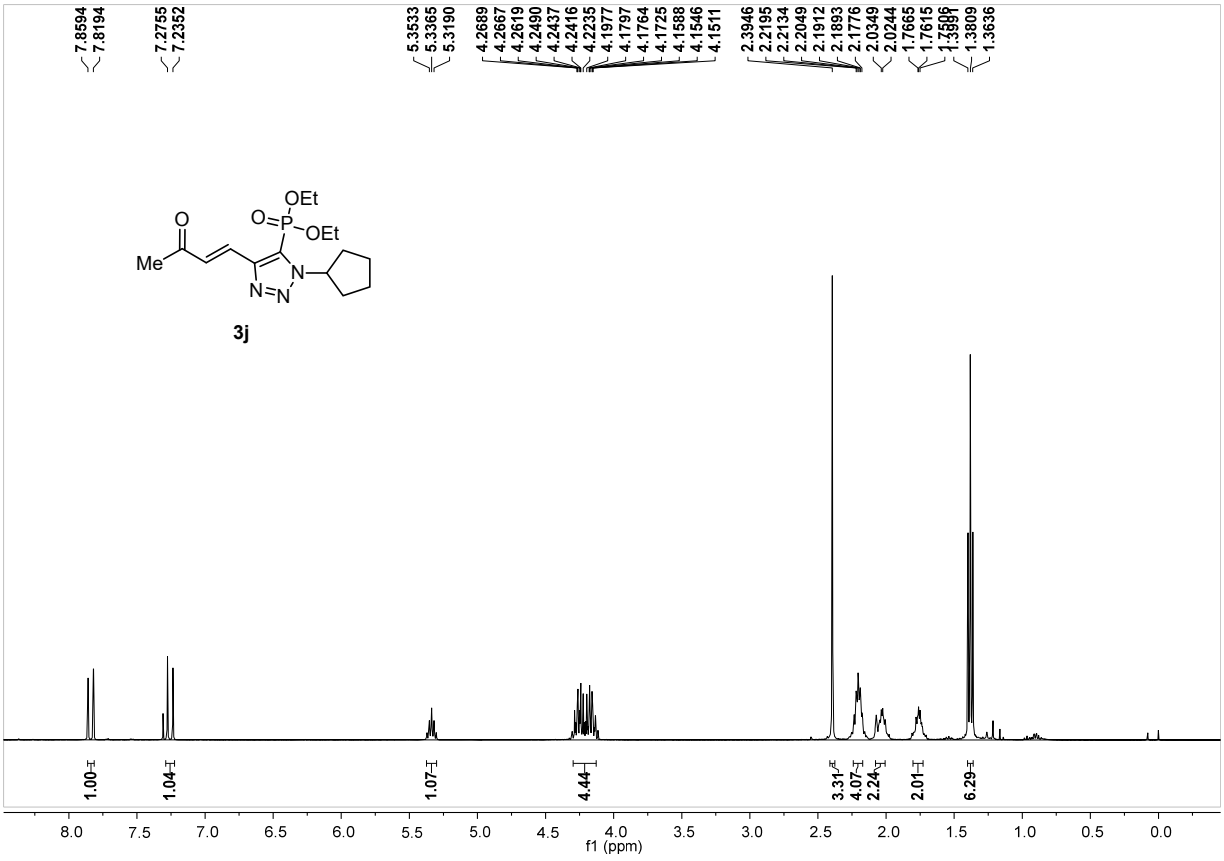
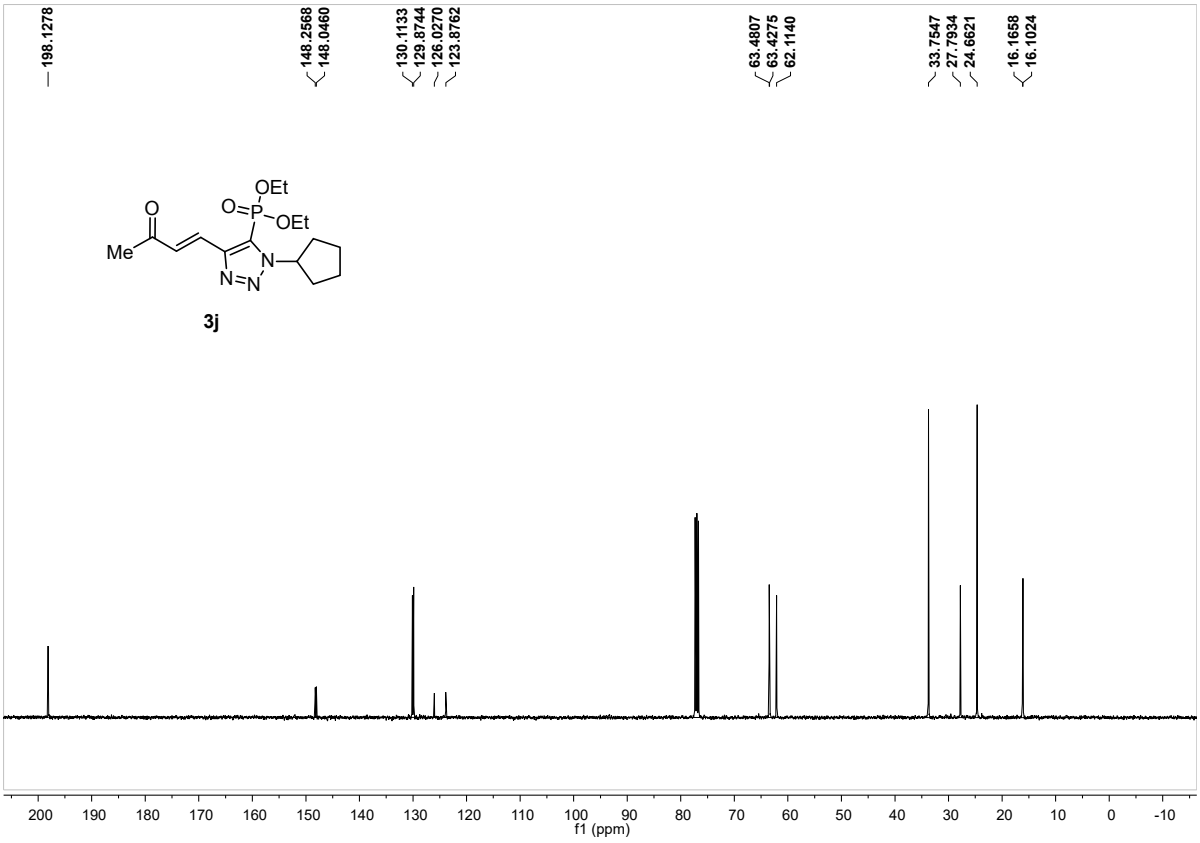
OEt
O
Me
N
N=N
4
Me
3h
400 350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400
f1 (ppm)


7.8033
7.7630
4.6324
4.6146
4.5958
4.3019
4.2837
4.2652
4.2453
4.2411
4.2272
4.2225
4.2108
4.1932
4.1747
4.1546
4.1362
2.4528
2.3925
1.9551
1.9376
1.3988
1.3813
1.3639
1.3307
1.2175
0.8875
OEt
O
O=P
OEt
Me
N
N=N
5
Me
3i
1.00
1.39
2.09
4.38
3.34
2.36
14.05
3.40
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
-1.0
f1 (ppm)


197.8608
148.2254
148.0197
129.9605
129.3423
125.8860
63.4112
63.3584
58.4645
50.6787
30.9834
30.3118
27.9631
25.9322
22.2256
16.0458
15.9817
13.7440
OEt
O
O=P
OEt
Me
N
N=N
5
Me
3i
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)


3.06
OEt
O
O=P
OEt
Me
N
N=N
5
Me
3i
400 350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400
f1 (ppm)


7.8594
7.8194
7.2755
7.2352
5.3533
5.3365
5.3190
4.2689
4.2667
4.2619
4.2490
4.2437
4.2416
4.2235
4.1977
4.1797
4.1764
4.1725
4.1588
4.1546
4.1511
2.3946
2.2195
2.2134
2.2049
2.1912
2.1893
2.1776
2.0349
2.0244
1.7665
1.7615
1.3991
1.3809
1.3636
OEt
O
O=P
OEt
Me
N
N=N
3j
1.00
1.04
1.07
4.44
3.31
4.07
2.24
2.01
6.29
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
f1 (ppm)


198.1278
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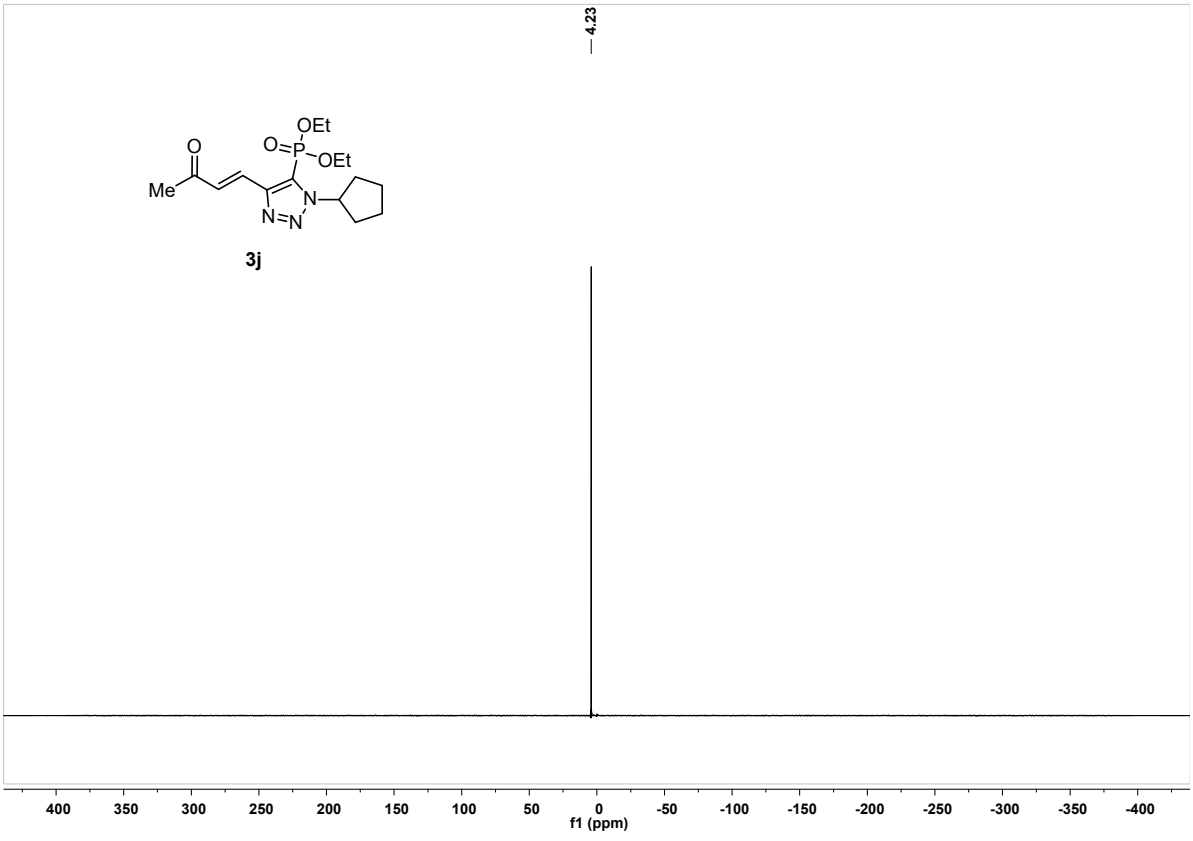
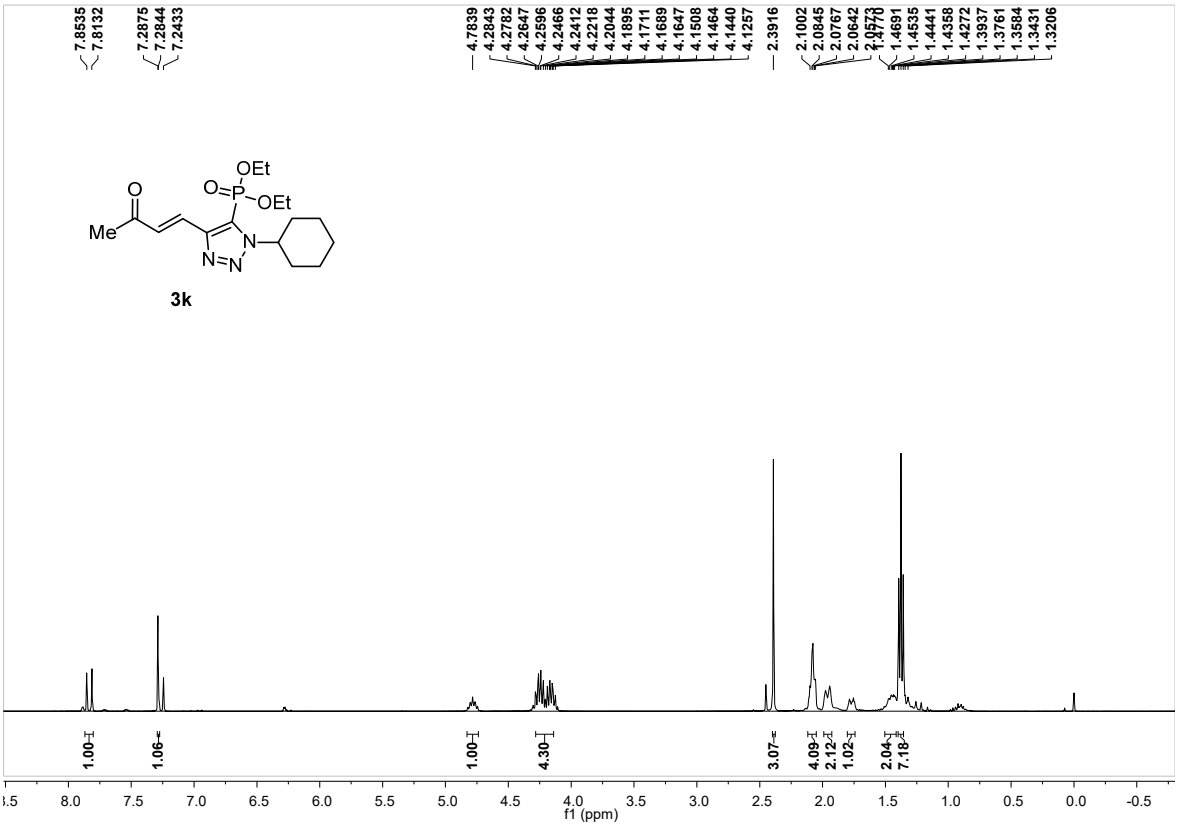
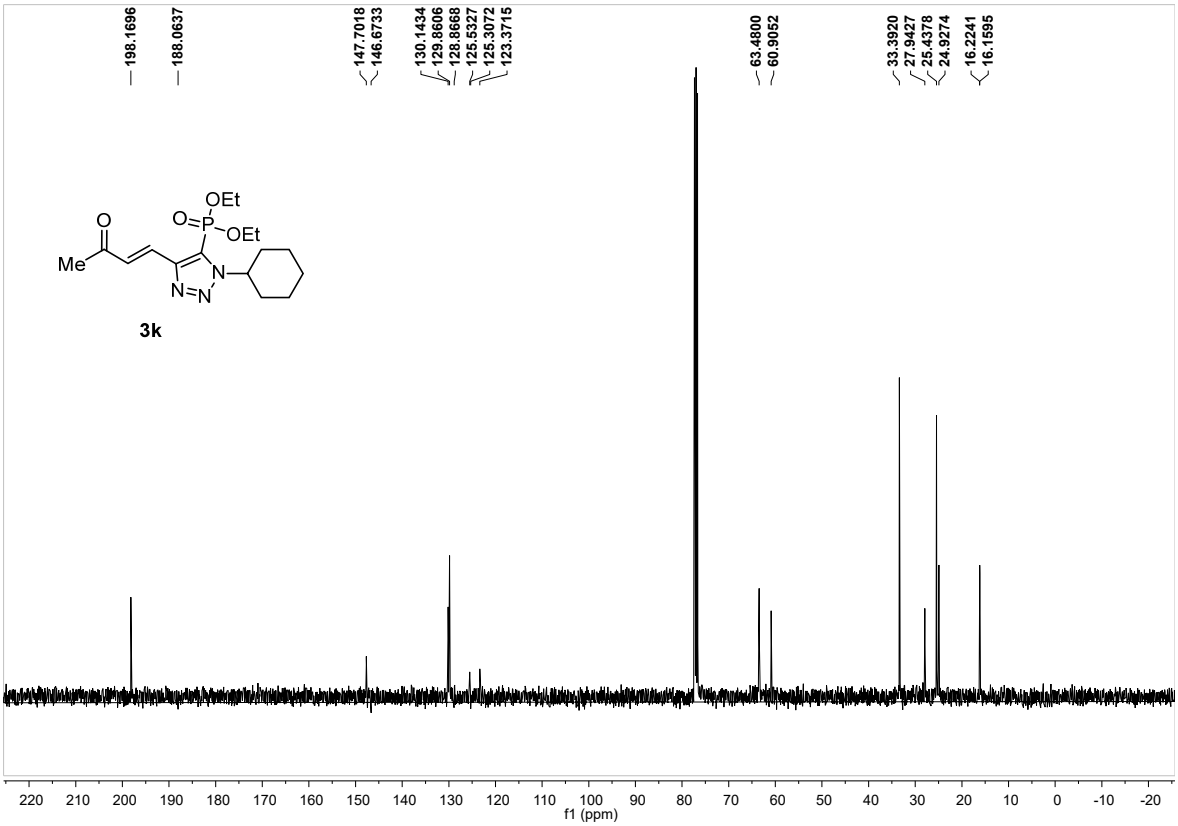
148.0460
130.1133
129.8744
126.0270
123.8762
63.4807
63.4275
62.1140
33.7547
27.7934
24.6621
16.1658
16.1024
OEt
O
O=P
OEt
Me
N
N=N
3j
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

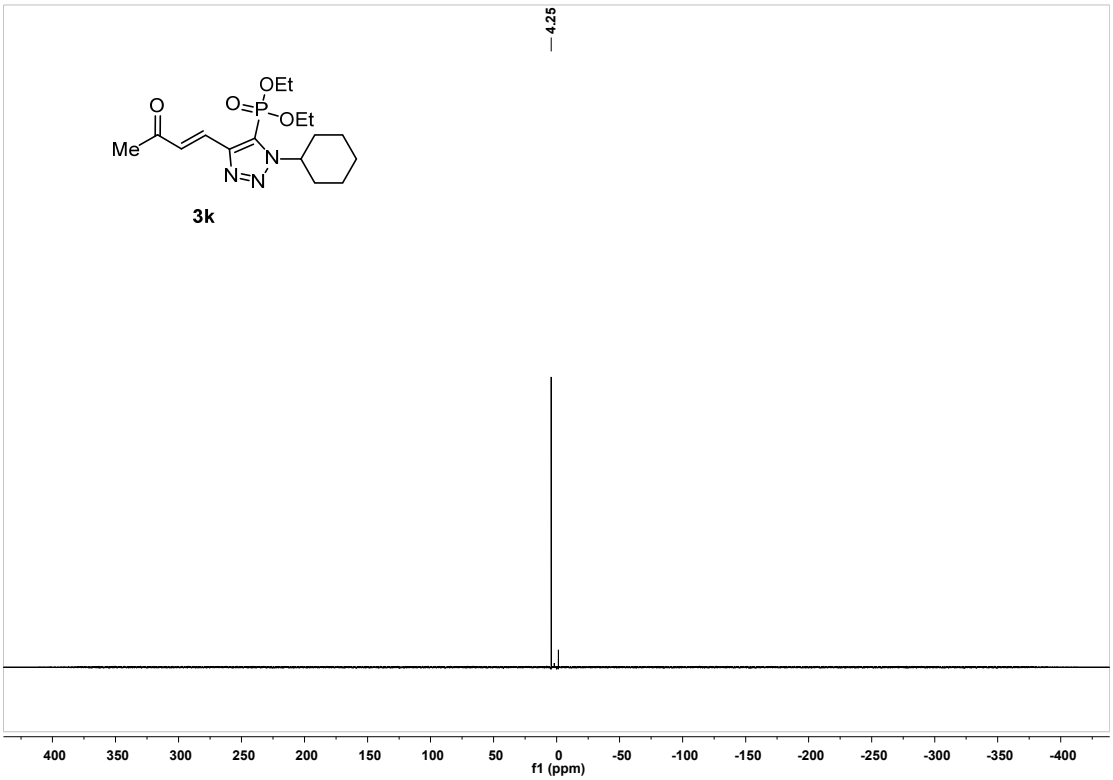

4.23
OEt
O
O=P
OEt
Me
N
N=N
3j
400 350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400
f1 (ppm)

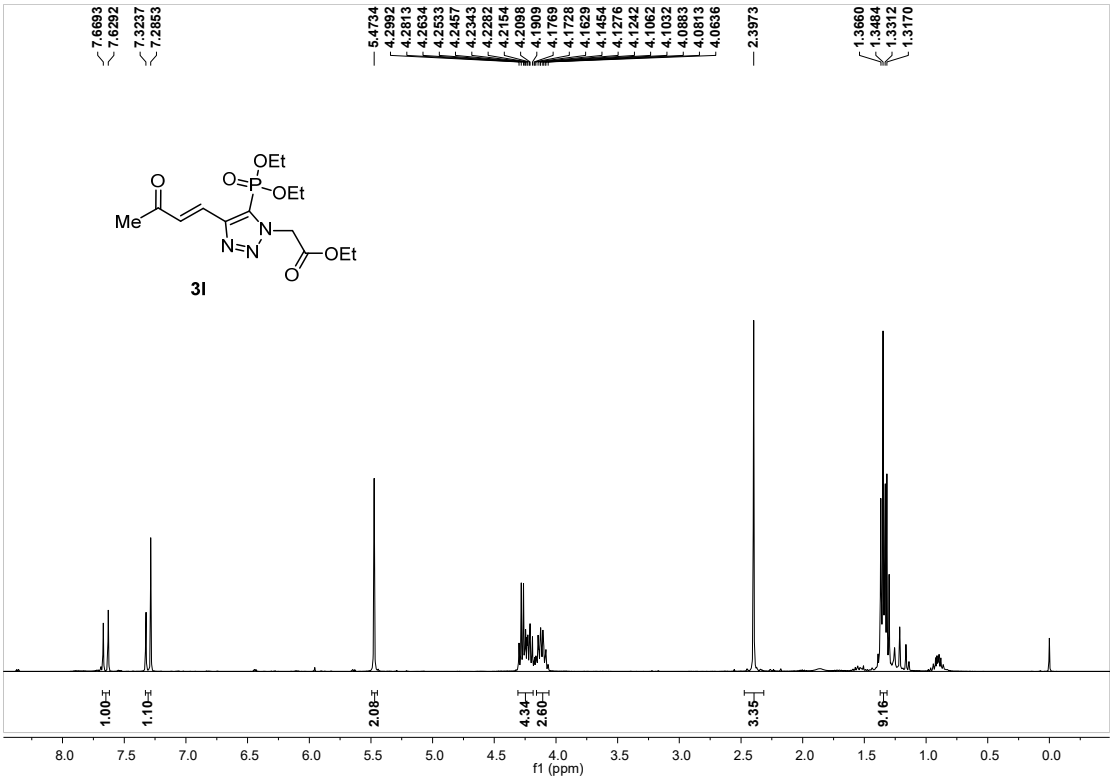

7.8535
7.8132
7.2875
7.2844
7.2433
4.7839
4.2843
4.2782
4.2647
4.2596
4.2466
4.2412
4.2218
4.2044
4.1895
4.1711
4.1689
4.1647
4.1508
4.1464
4.1440
4.1257
2.3916
2.1002
2.0845
2.0767
2.0642
1.4691
1.4535
1.4441
1.4358
1.4272
1.3937
1.3761
1.3584
1.3431
1.3206
OEt
O
O=P
OEt
Me
N
N=N
1.00
1.06
1.00
4.30
3.07
4.09
2.12
1.02
2.04
7.18
f1 (ppm)

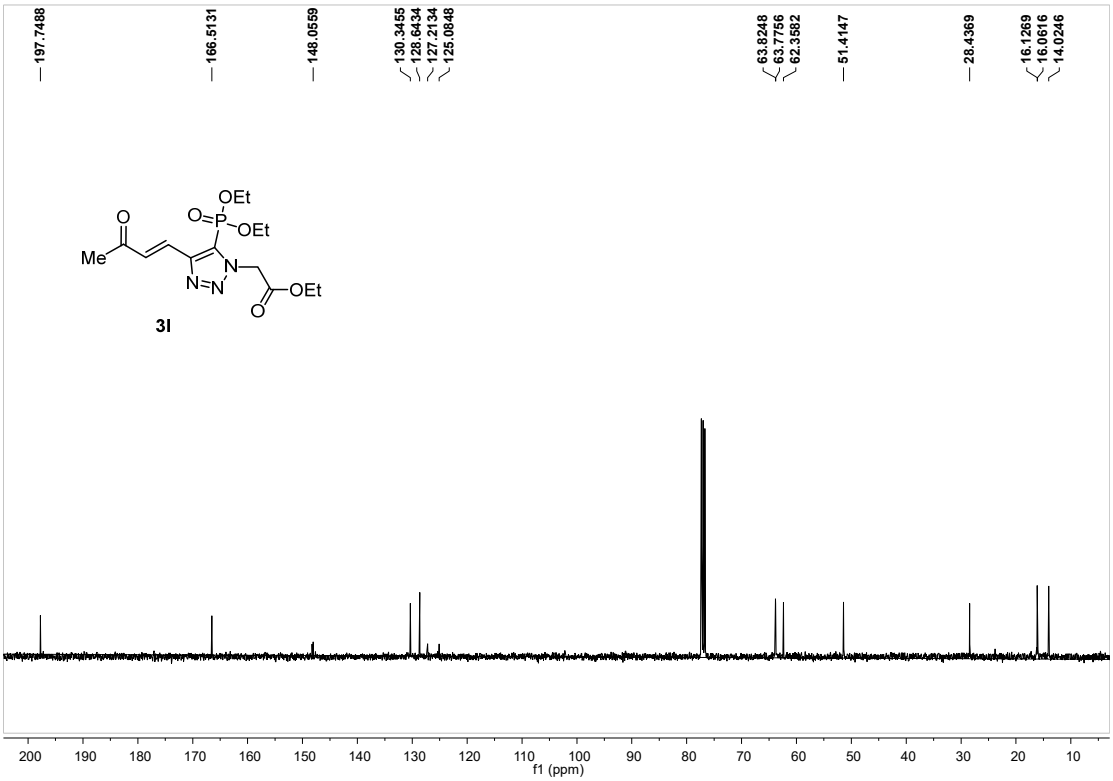
3k

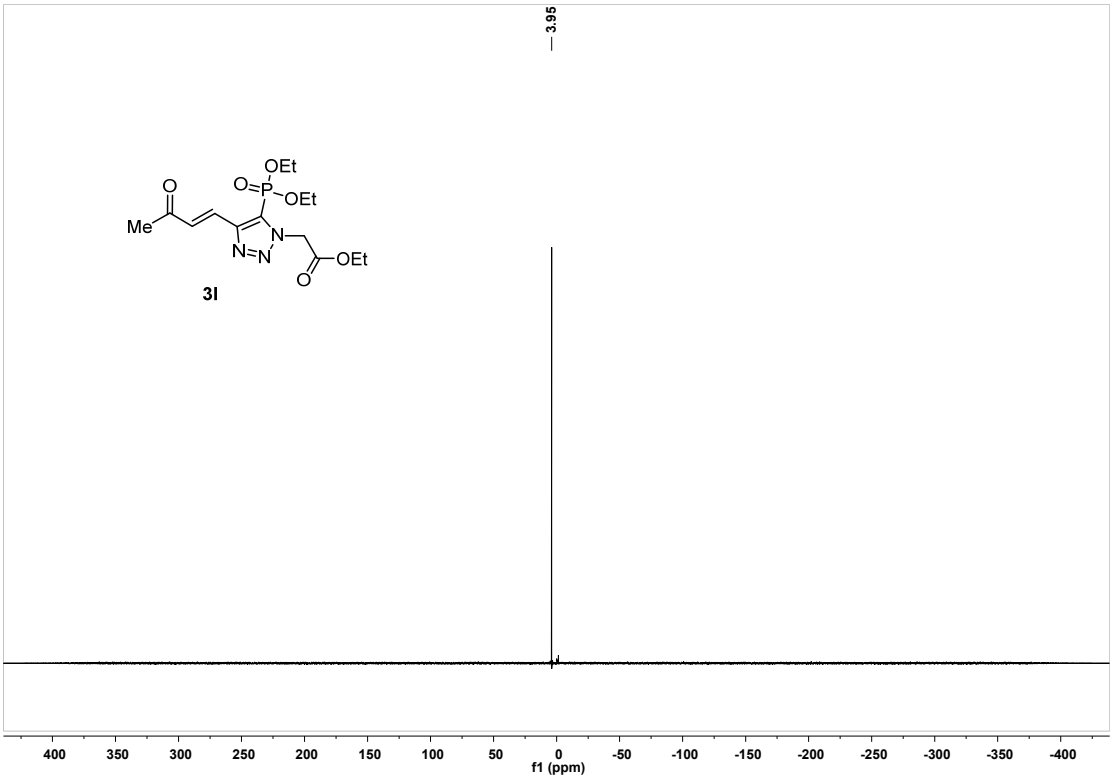

198.1696
188.0637
147.7018
146.6733
130.1434
129.8606
128.8668
125.5327
125.3072
123.3715
63.4800
60.9052
33.3920
27.9427
25.4378
24.9274
16.2241
16.1595
OEt
O
O=P
OEt
Me
N
N=N
f1 (ppm)

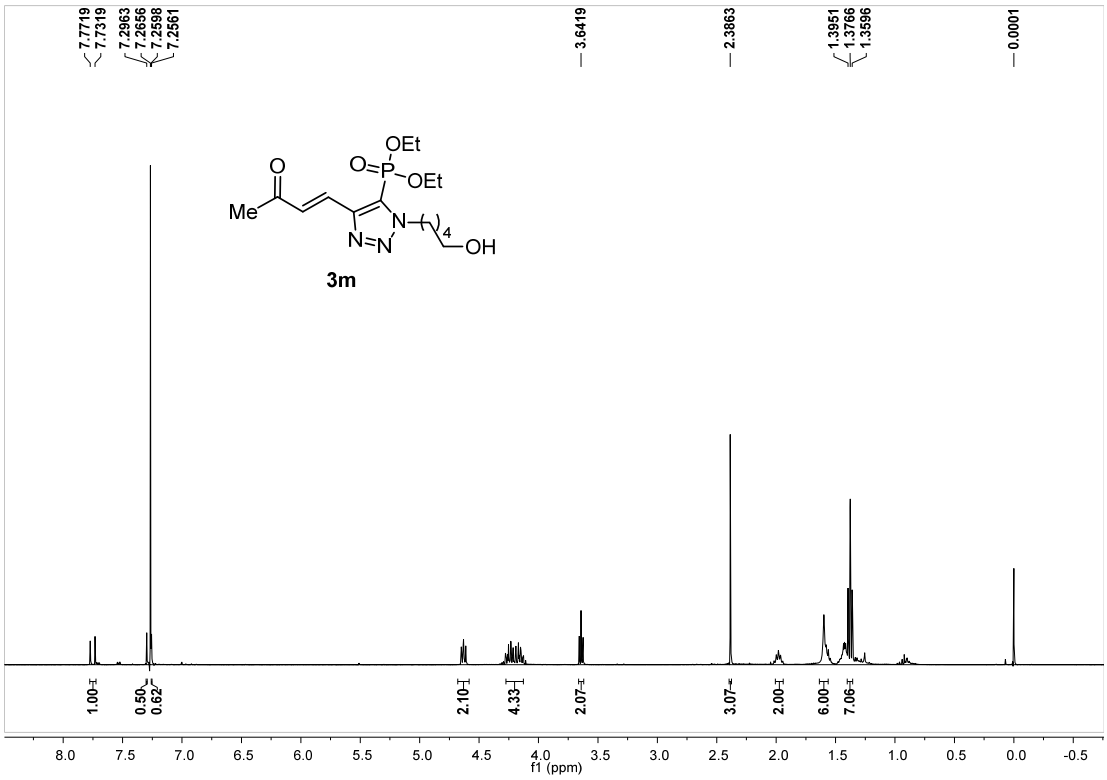
3k

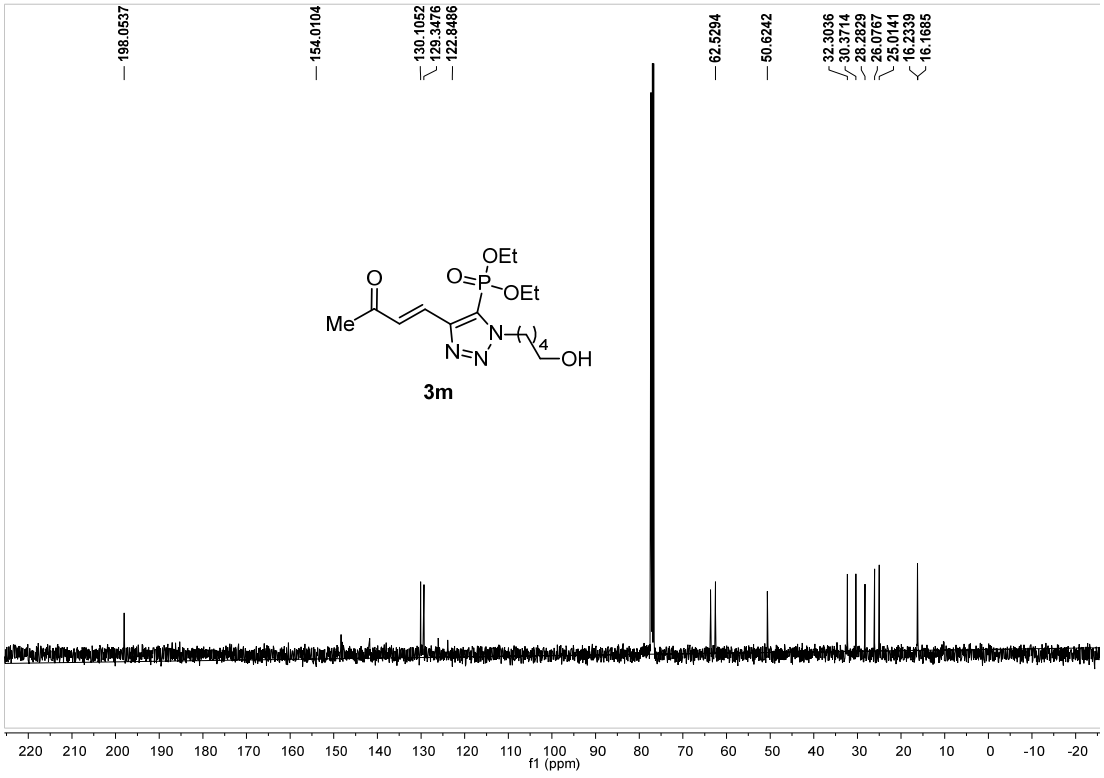

4.25
OEt
O
O=P
OEt
Me
N
N=N
3k
400 350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400
f1 (ppm)

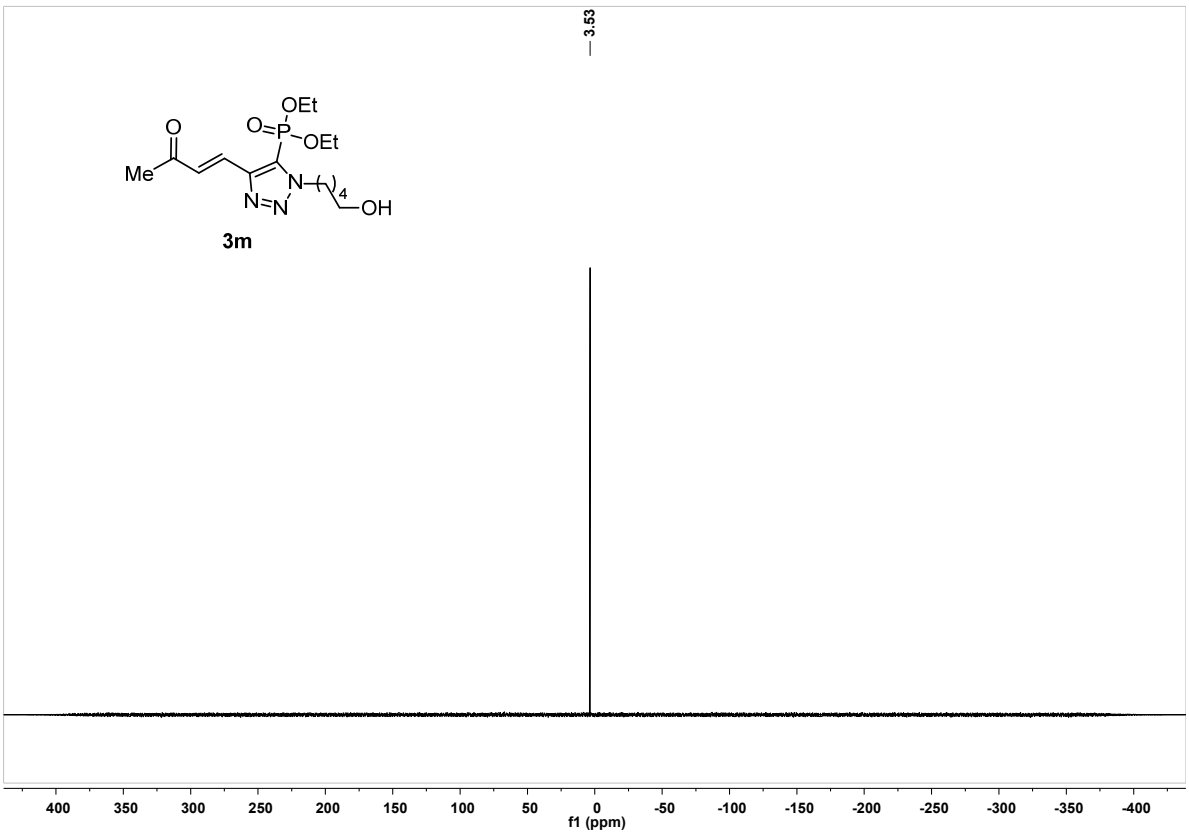

7.6693
7.6292
7.3237
7.2853
5.4734
4.2992
4.2813
4.2634
4.2533
4.2457
4.2343
4.2282
4.2154
4.2098
4.1909
4.1769
4.1728
4.1629
4.1454
4.1276
4.1242
4.1062
4.1032
4.0883
4.0813
4.0636
2.3973
1.3660
1.3484
1.3312
1.3170
OEt
O
O=P
OEt
Me
N
N=N
OEt
O
3l
1.00
1.10
2.08
4.34
2.60
3.35
9.16
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

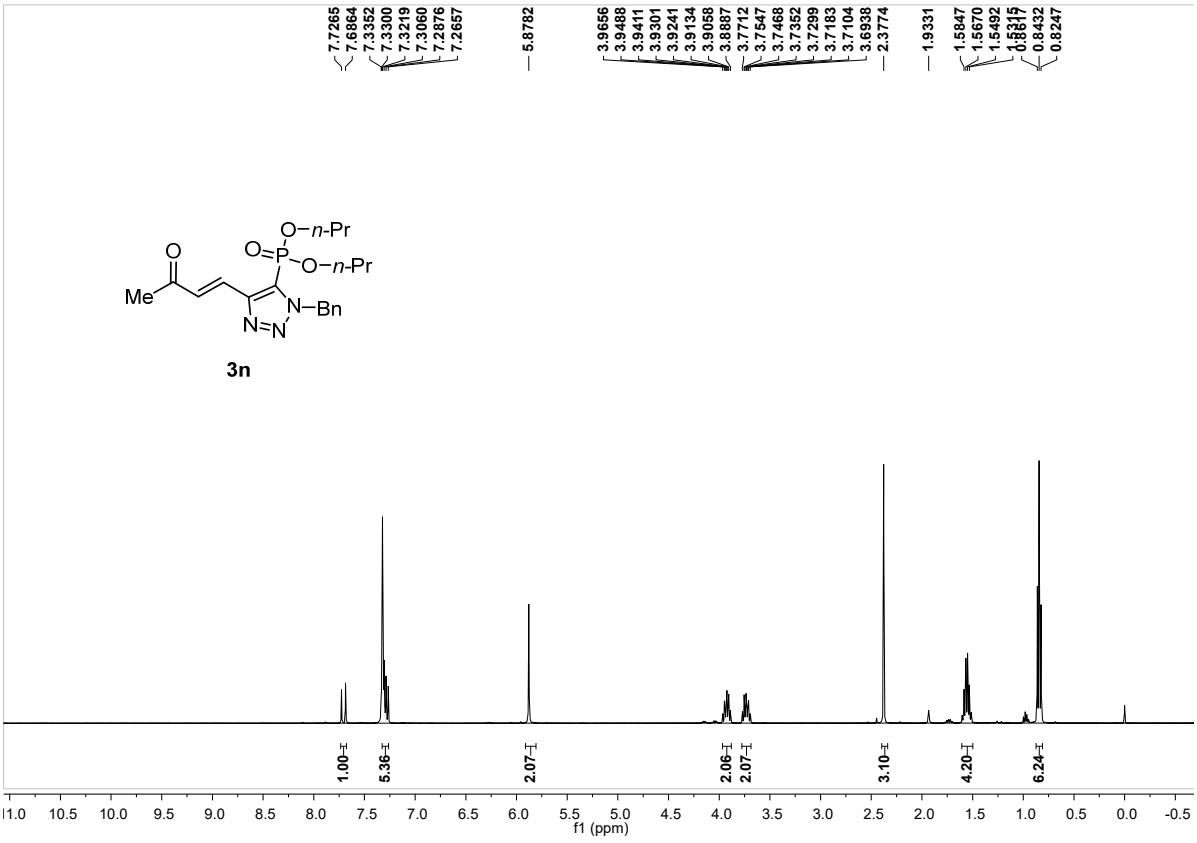

197.7488
166.5131
148.0559
130.3455
128.6434
127.2134
125.0848
63.8248
63.7756
62.3582
51.4147
28.4369
16.1269
16.0616
14.0246
OEt
O
O=P
OEt
Me
N
N=N
OEt
O
3l
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

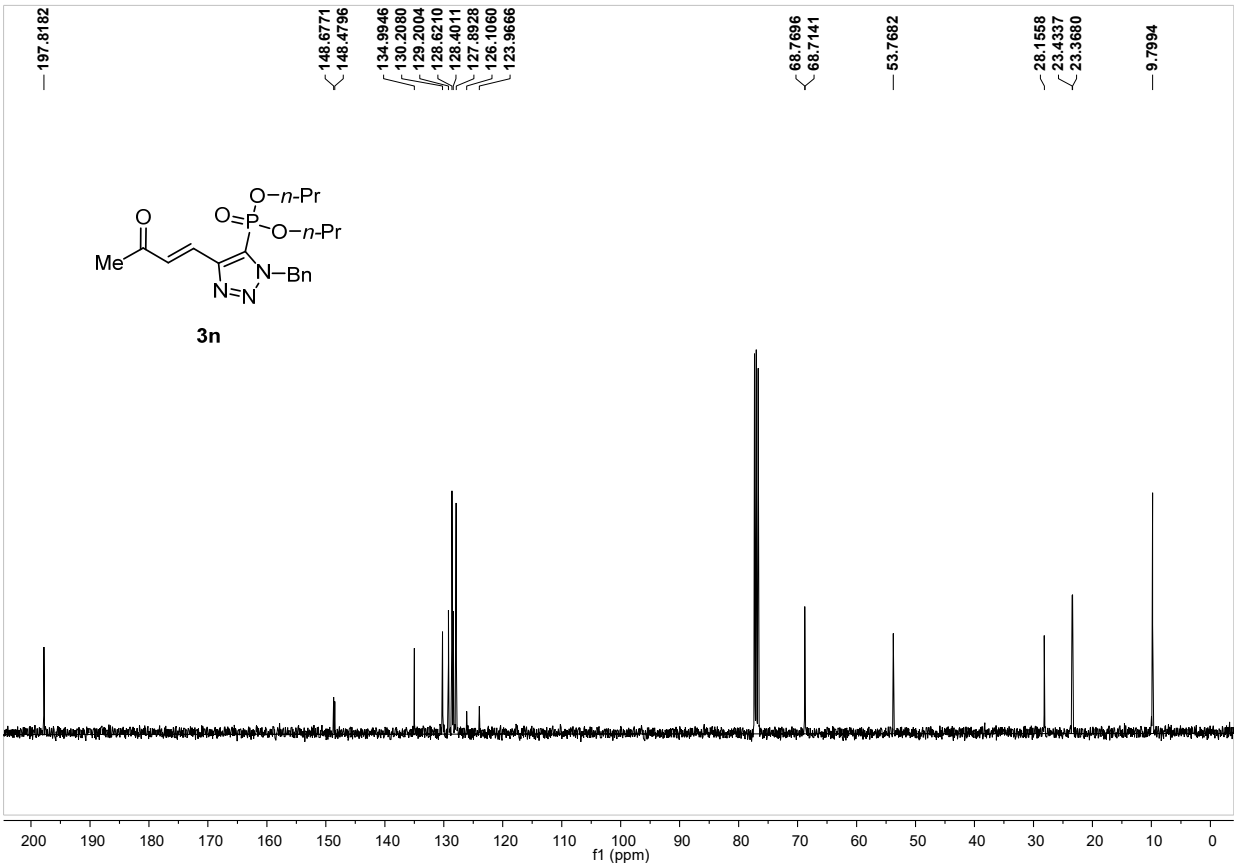

3.95
OEt
O
O=P
OEt
Me
N
N=N
OEt
O
3l
400 350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400
f1 (ppm)


7.7719
7.7319
7.2963
7.2656
7.2598
7.2561
3.6419
2.3863
1.3951
1.3766
1.3596
0.0001
OEt
O
O=P
OEt
Me
N
N=N
4
OH
3m
1.00
0.50
0.62
2.10
4.33
2.07
3.07
2.00
6.00
7.06
f1 (ppm)


198.0537
154.0104
130.1052
129.3476
122.8486
62.5294
50.6242
32.3036
30.3714
28.2829
26.0767
25.0141
16.2339
16.1685
OEt
O
O=P
OEt
Me
N
N=N
4
OH
3m
f1 (ppm)


3.53
OEt
O
O=P
OEt
Me
N
4
N=N
OH
3m
400 350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400
f1 (ppm)


7.7265
7.6864
7.3352
7.3300
7.3219
7.3060
7.2876
7.2657
5.8782
3.9656
3.9488
3.9411
3.9301
3.9241
3.9134
3.9058
3.8887
3.7712
3.7547
3.7468
3.7352
3.7299
3.7183
3.7104
3.6938
2.3774
1.9331
1.5847
1.5670
1.5492
0.8432
0.8247
O−n-Pr
O
O=P
O−n-Pr
Me
N−Bn
N=N
3n
1.00
5.36
2.07
2.06
2.07
3.10
4.20
6.24
f1 (ppm)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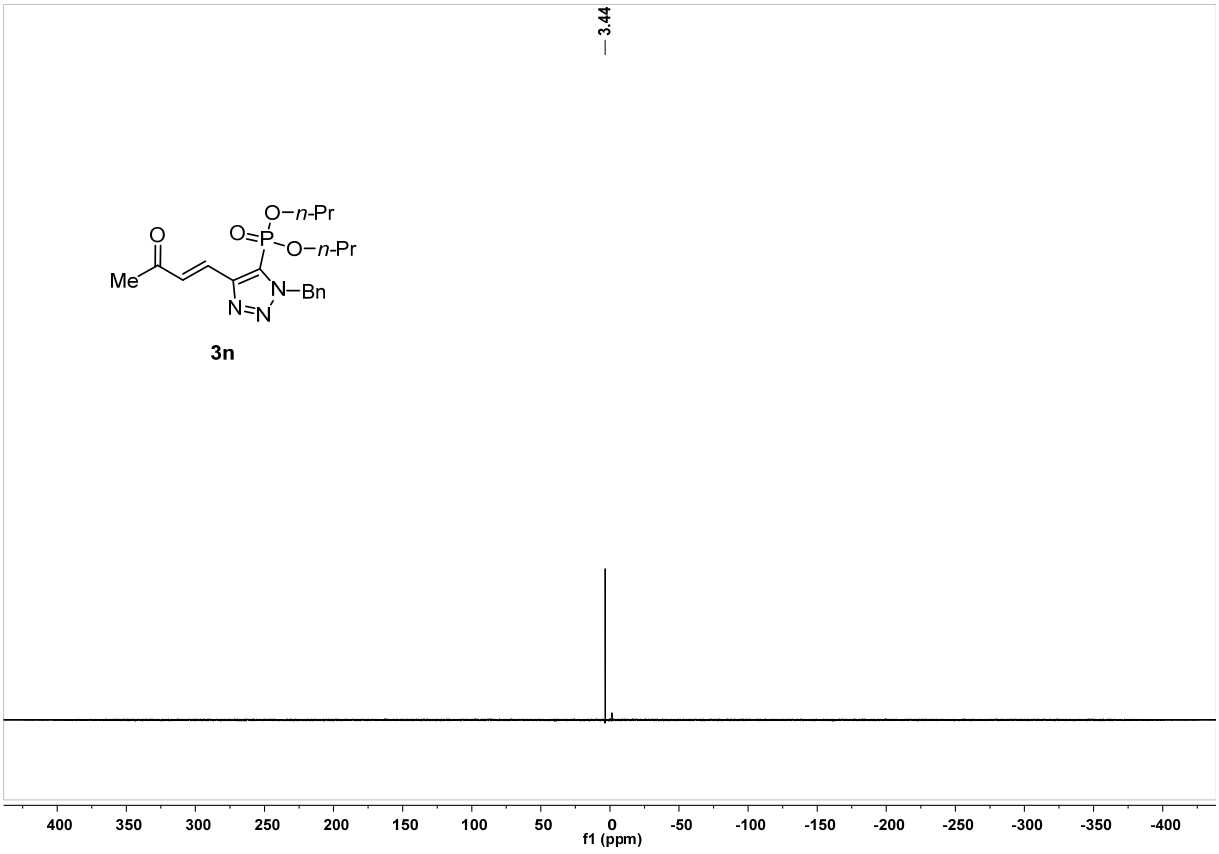
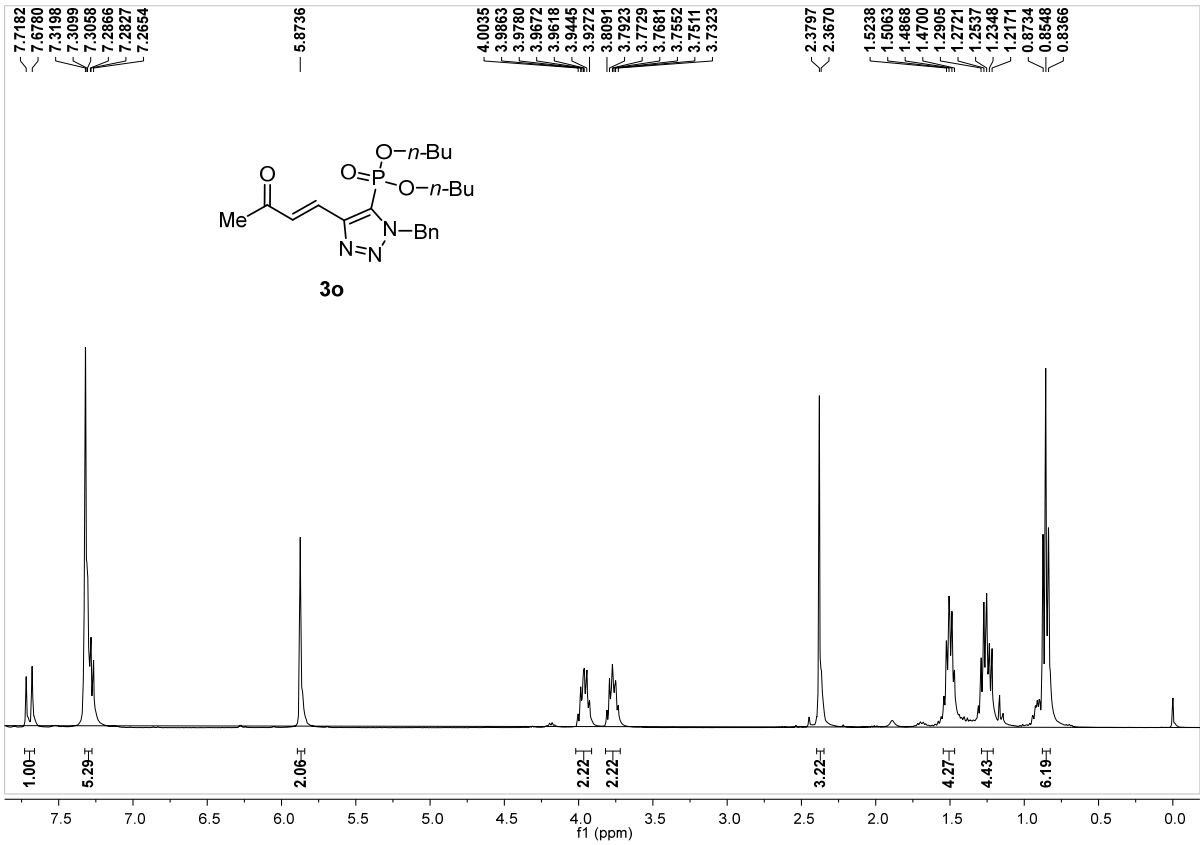
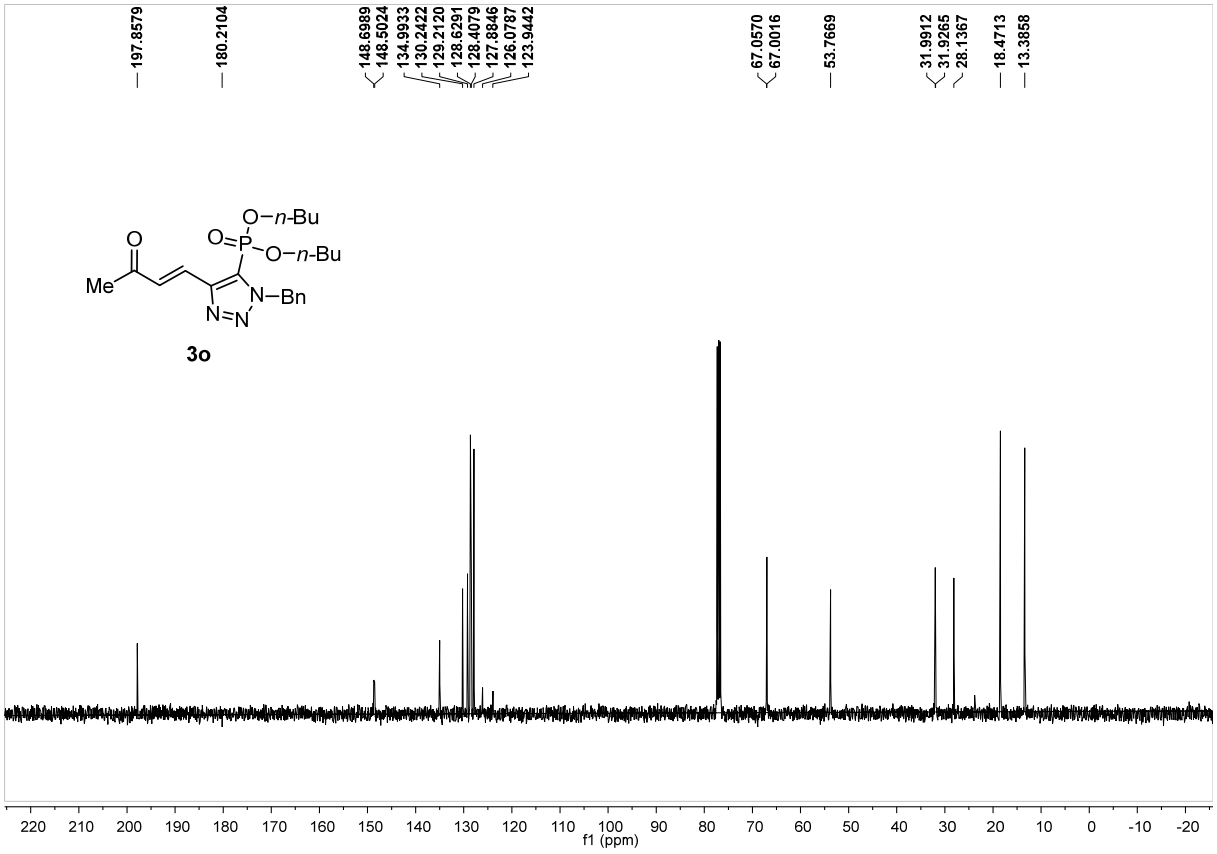
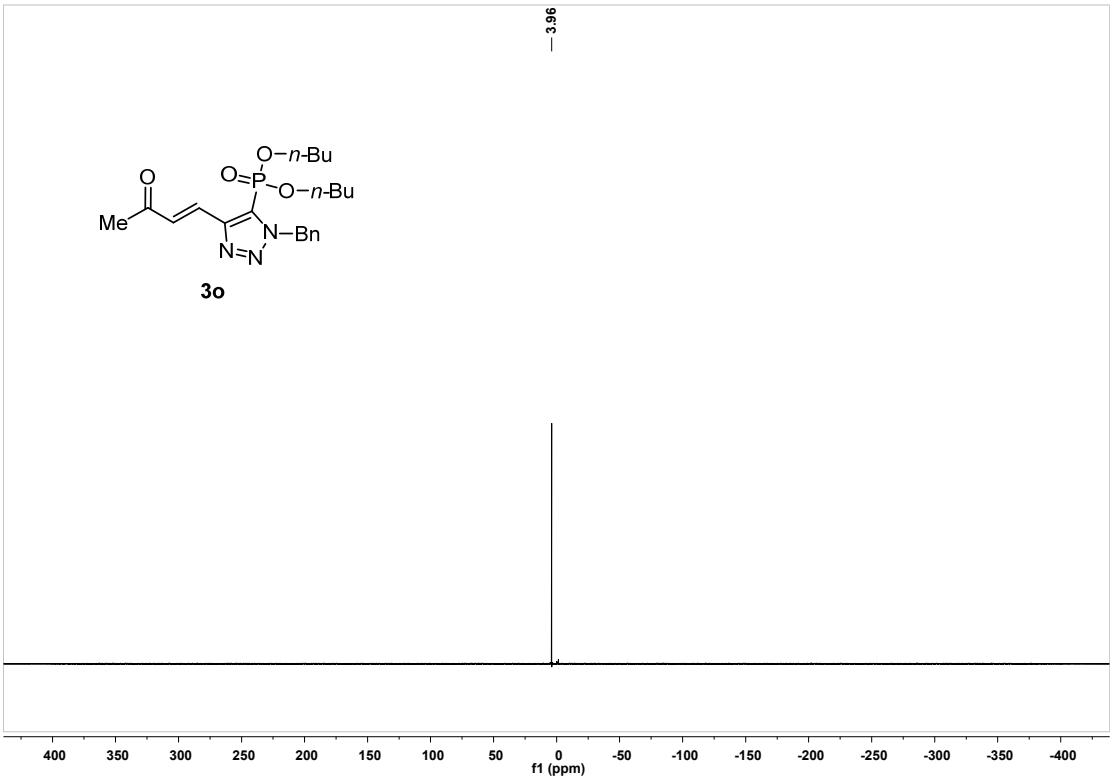
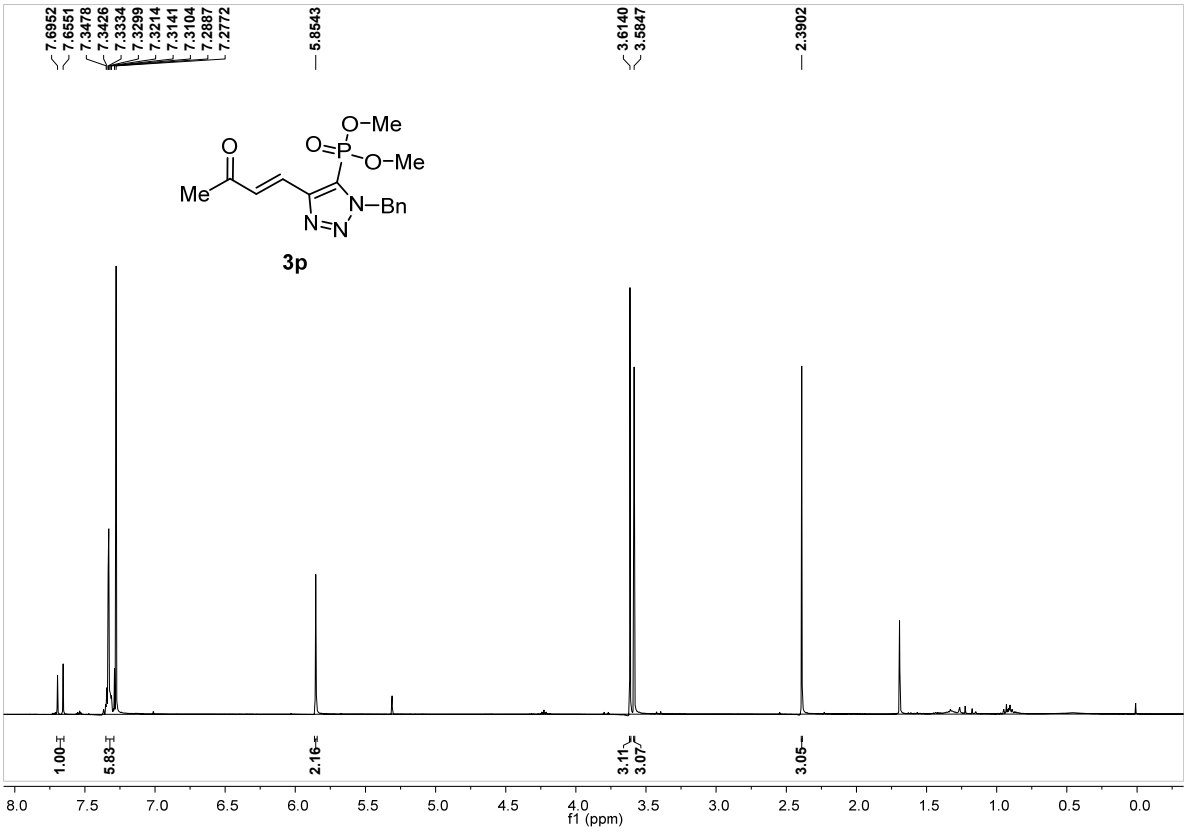
148.6771
148.4796
134.9946
130.2080
129.2004
128.6210
128.4011
127.8928
126.1060
123.9666
68.7696
68.7141
53.7682
28.1558
23.4337
23.3680
9.7994
O–n-Pr
O=P
O–n-Pr
O
Me
N–Bn
N=N
3n
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

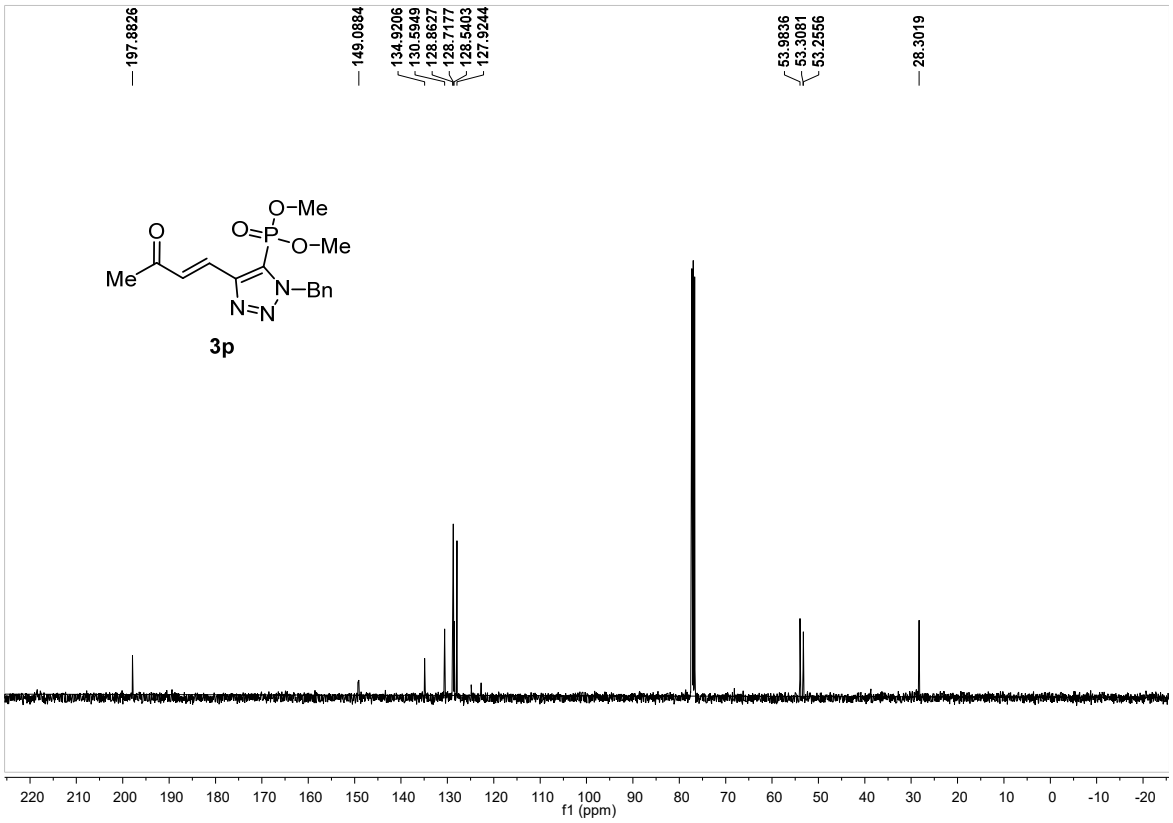

3.44
O–n-Pr
O=P
O–n-Pr
O
Me
N–Bn
N=N
3n
400 350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400
f1 (ppm)


7.7182
7.6780
7.3198
7.3099
7.3058
7.2866
7.2827
7.2654
5.8736
4.0035
3.9863
3.9780
3.9672
3.9618
3.9445
3.9272
3.8091
3.7923
3.7729
3.7681
3.7552
3.7511
3.7323
2.3797
2.3670
1.5238
1.5063
1.4868
1.4700
1.2905
1.2721
1.2537
1.2348
1.2171
0.8734
0.8548
0.8366
O−n-Bu
O
O=P
O−n-Bu
Me
N−Bn
N=N
3o
1.00
5.29
2.06
2.22
2.22
3.22
4.27
4.43
6.19
f1 (ppm)


197.8579
180.2104
148.6989
148.5024
134.9933
130.2422
129.2120
128.6291
128.4079
127.8846
126.0787
123.9442
67.0570
67.0016
53.7669
31.9912
31.9265
28.1367
18.4713
13.3858
O−n-Bu
O
O=P
O−n-Bu
Me
N−Bn
N=N
3o
f1 (ppm)


3.96
O–n-Bu
O
P
O–n-Bu
O
Me
N–Bn
N=N
3o
400 350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400
f1 (ppm)


7.6952
7.6551
7.3478
7.3426
7.3334
7.3299
7.3214
7.3141
7.3104
7.2887
7.2772
5.8543
3.6140
3.5847
2.3902
O–Me
O
P
O–Me
O
Me
N–Bn
N=N
3p
1.00
5.83
2.16
3.11
3.07
3.05
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
f1 (ppm)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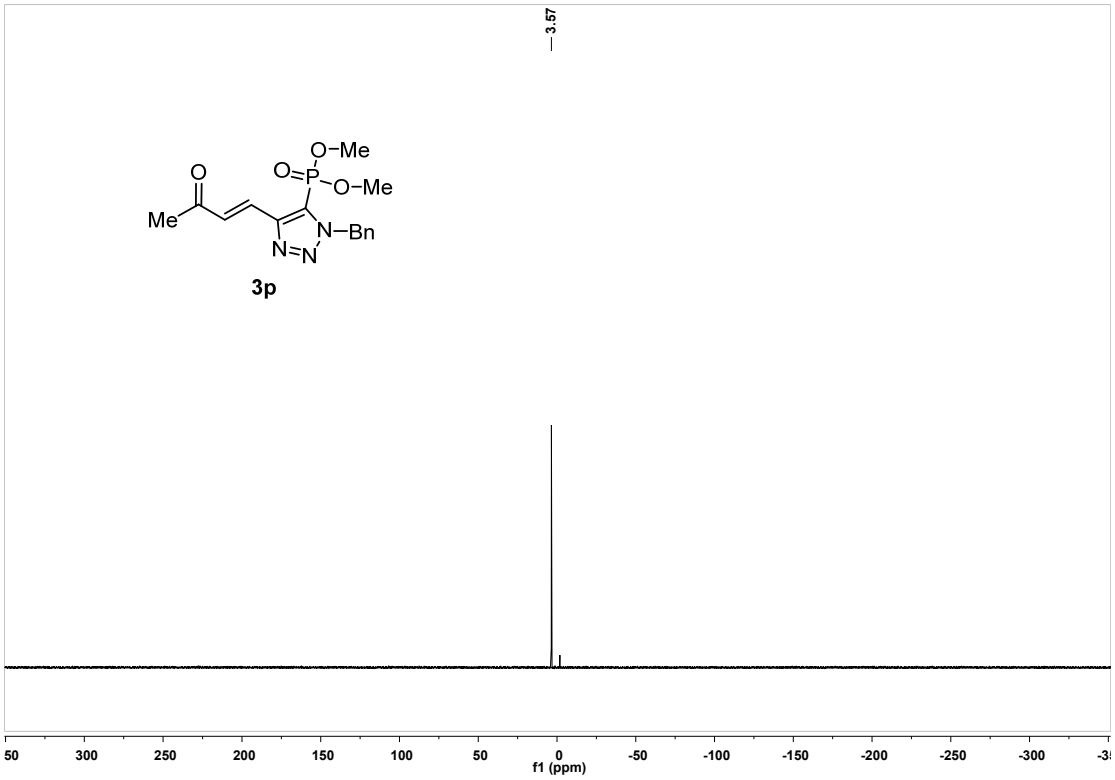
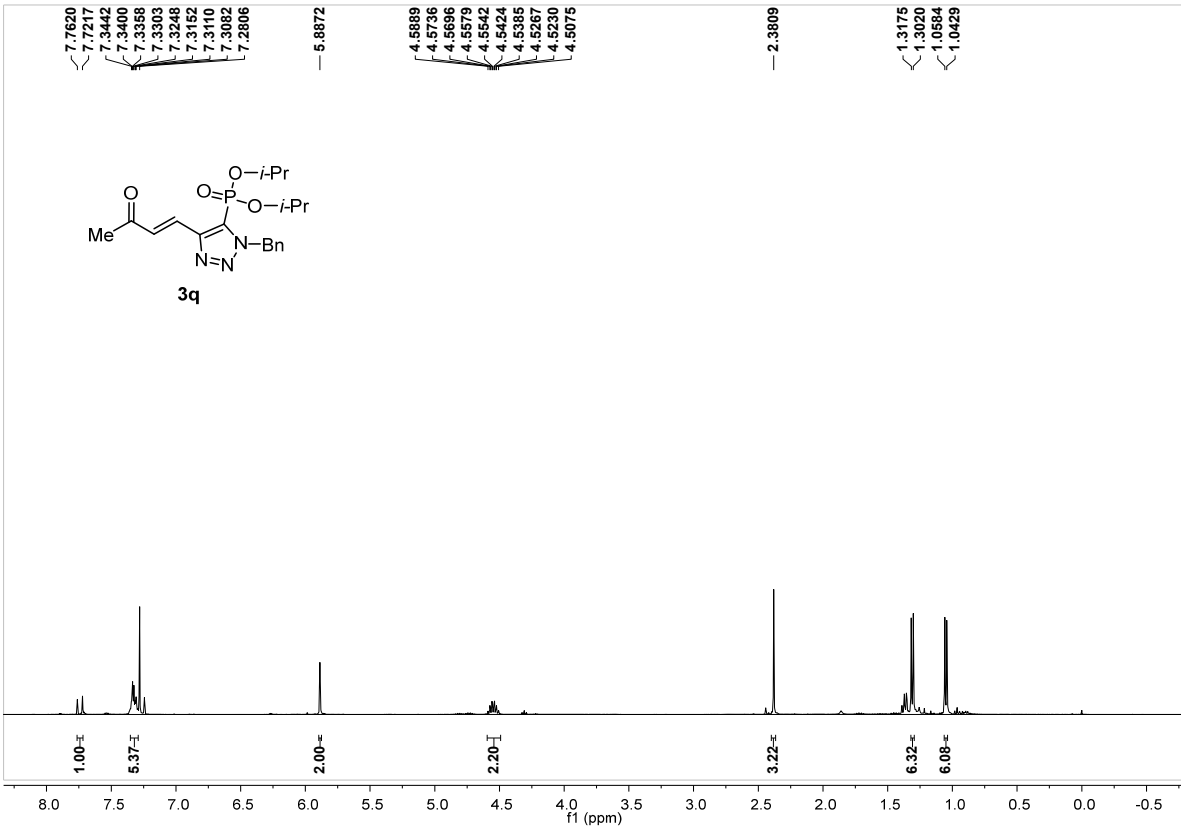
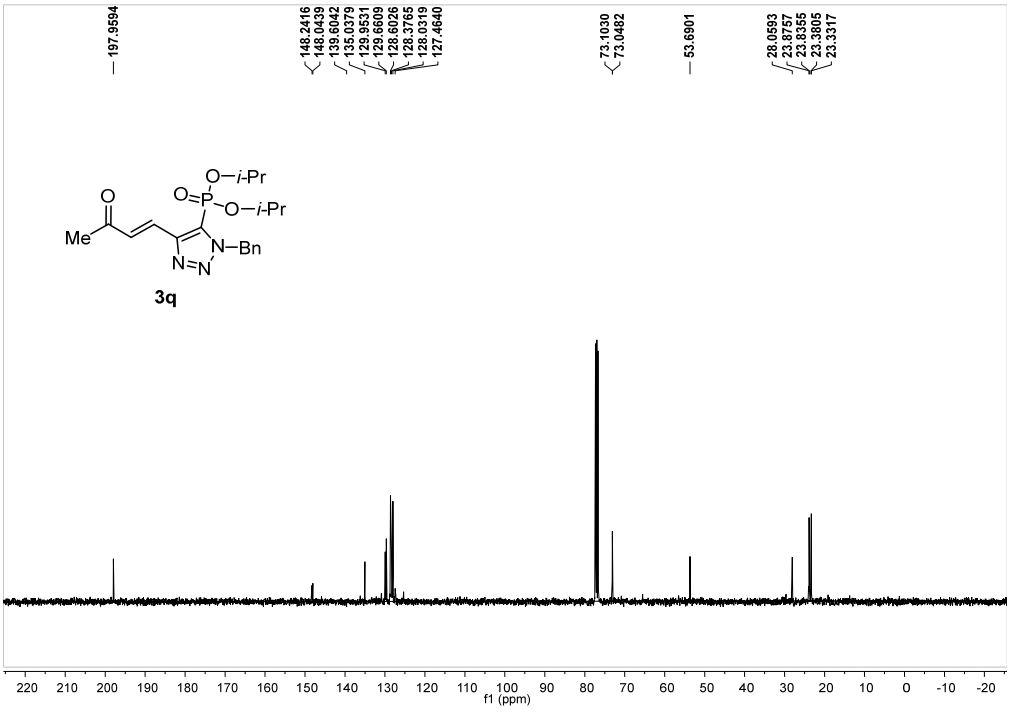
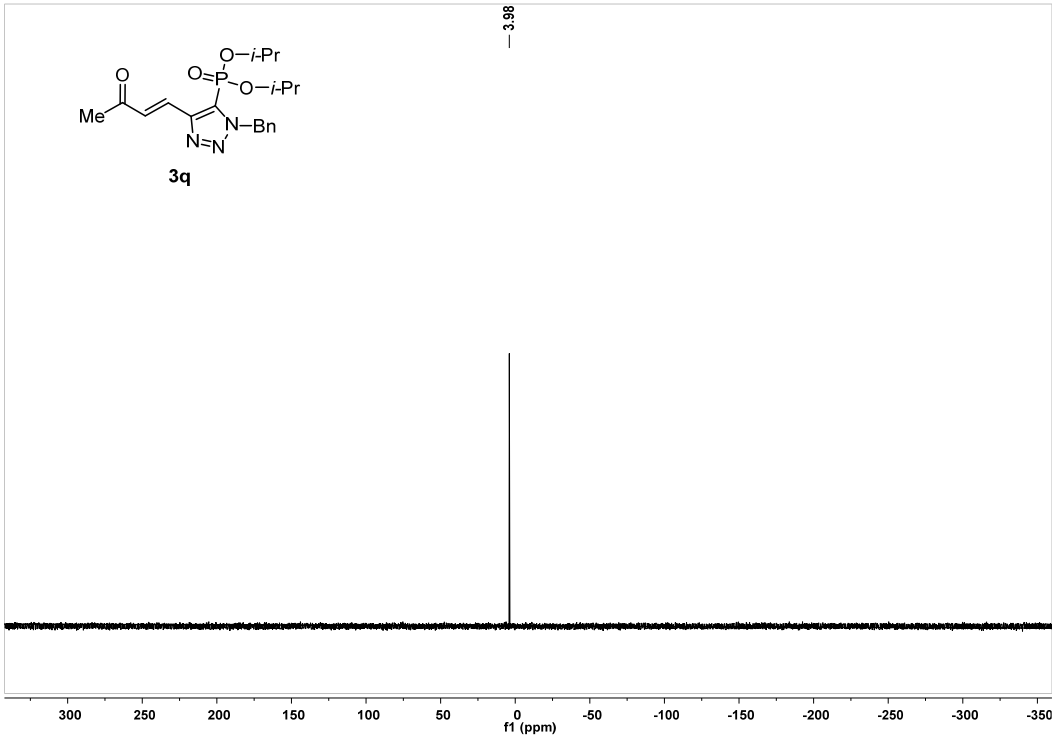
149.0884
134.9206
130.5949
128.8627
128.7177
128.5403
127.9244
53.9836
53.3081
53.2556
28.3019
O
Me
O=P
O-Me
O-Me
N-Bn
N=N
3p
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

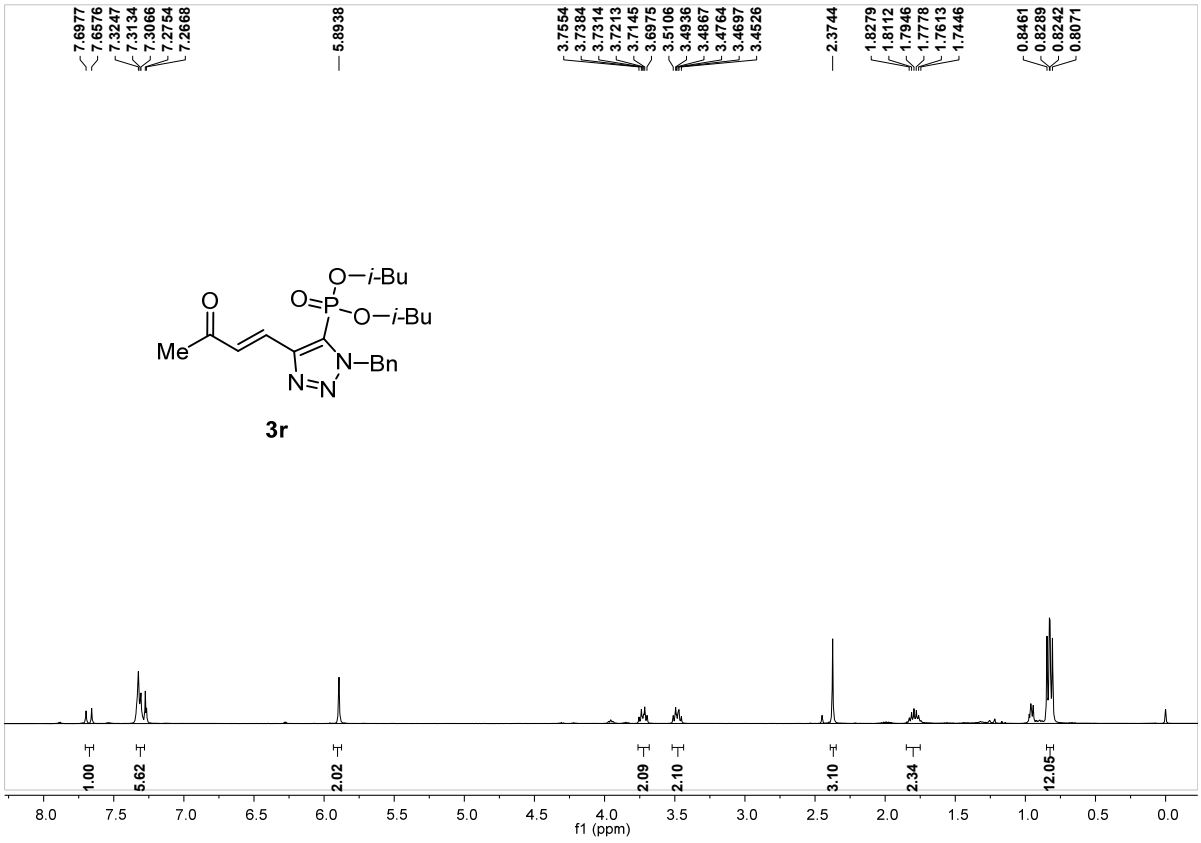

3.57
O
Me
O=P
O-Me
O-Me
N-Bn
N=N
3p
50 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300
f1 (ppm)


7.7620
7.7217
7.3442
7.3400
7.3358
7.3303
7.3248
7.3152
7.3110
7.3082
7.2806
5.8872
4.5889
4.5736
4.5696
4.5579
4.5542
4.5424
4.5385
4.5267
4.5230
4.5075
2.3809
1.3175
1.3020
1.0584
1.0429
O–i-Pr
O
O=P
O–i-Pr
Me
N–Bn
N=N
3q
1.00
5.37
2.00
2.20
3.22
6.32
6.08
f1 (ppm)

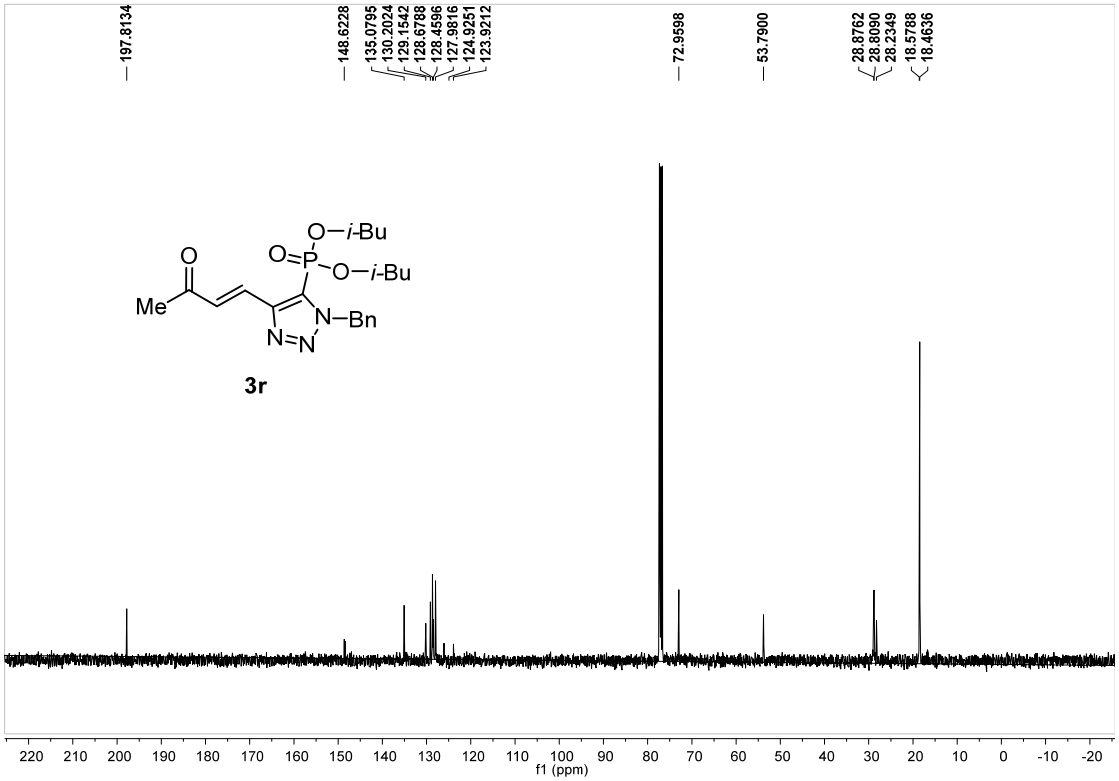

197.9594
148.2416
148.0439
139.6042
135.0379
129.9531
129.6609
128.6026
128.3765
128.0319
127.4640
73.1030
73.0482
53.6901
28.0593
23.8757
23.8355
23.3805
23.3317
O–i-Pr
O
O=P
O–i-Pr
Me
N–Bn
N=N
3q
f1 (ppm)

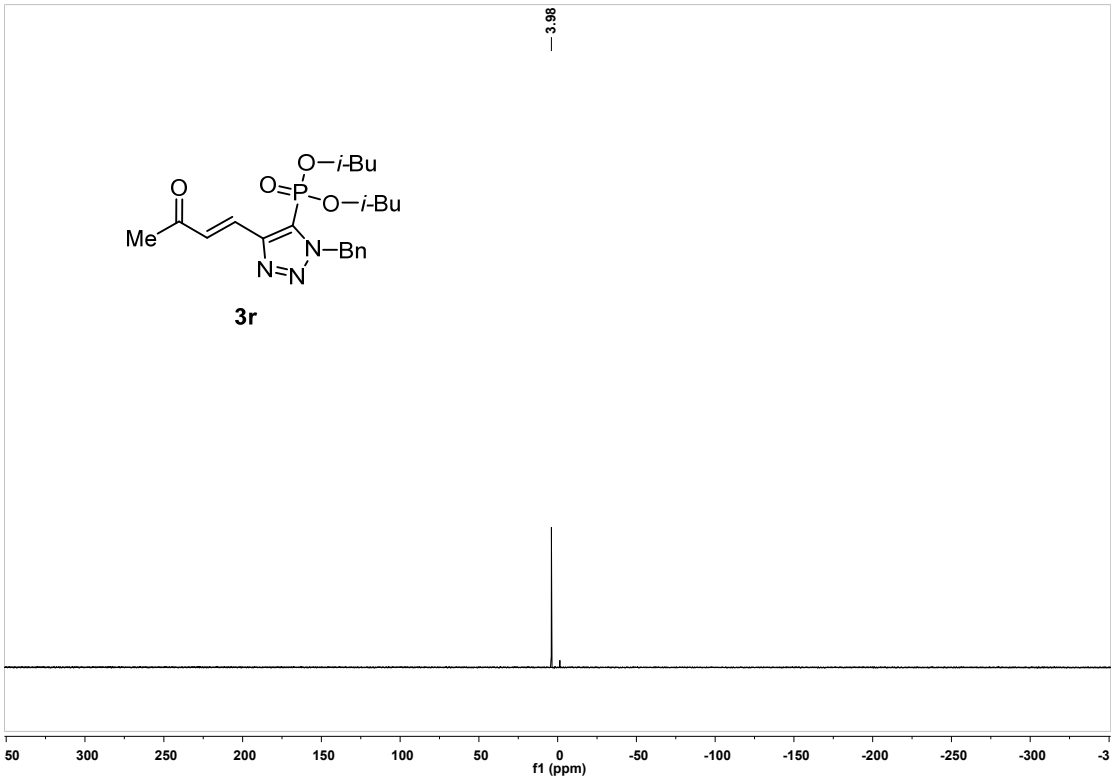

3.98
O
Me
O—i-Pr
O=P
O—i-Pr
N—Bn
N=N
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
-350
f1 (ppm)

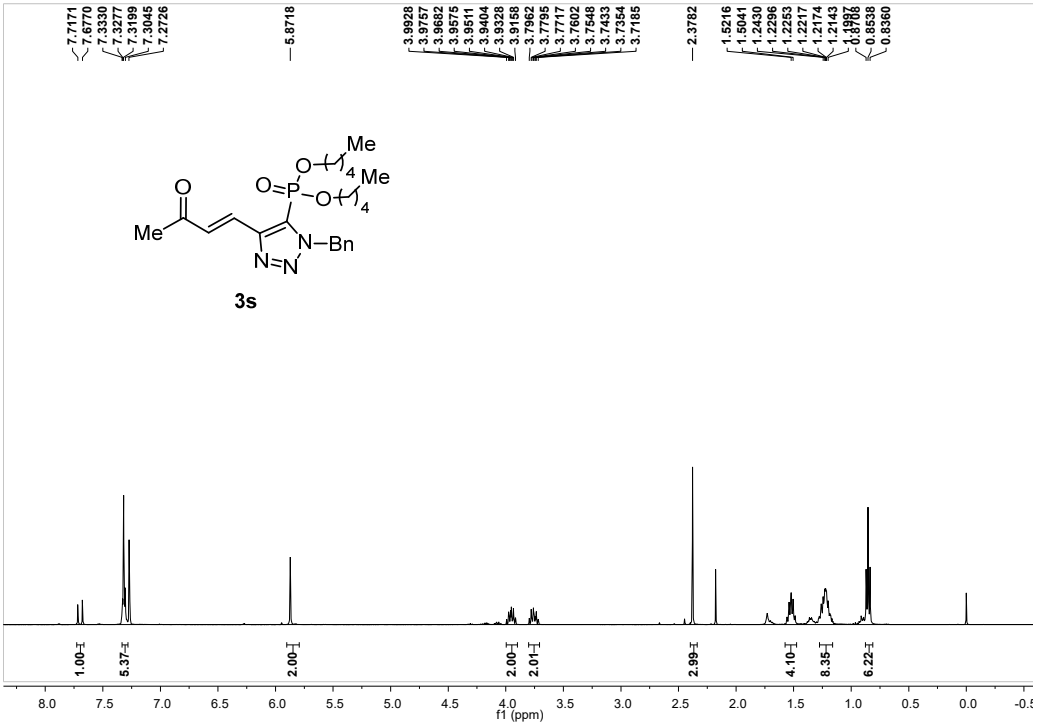
3q

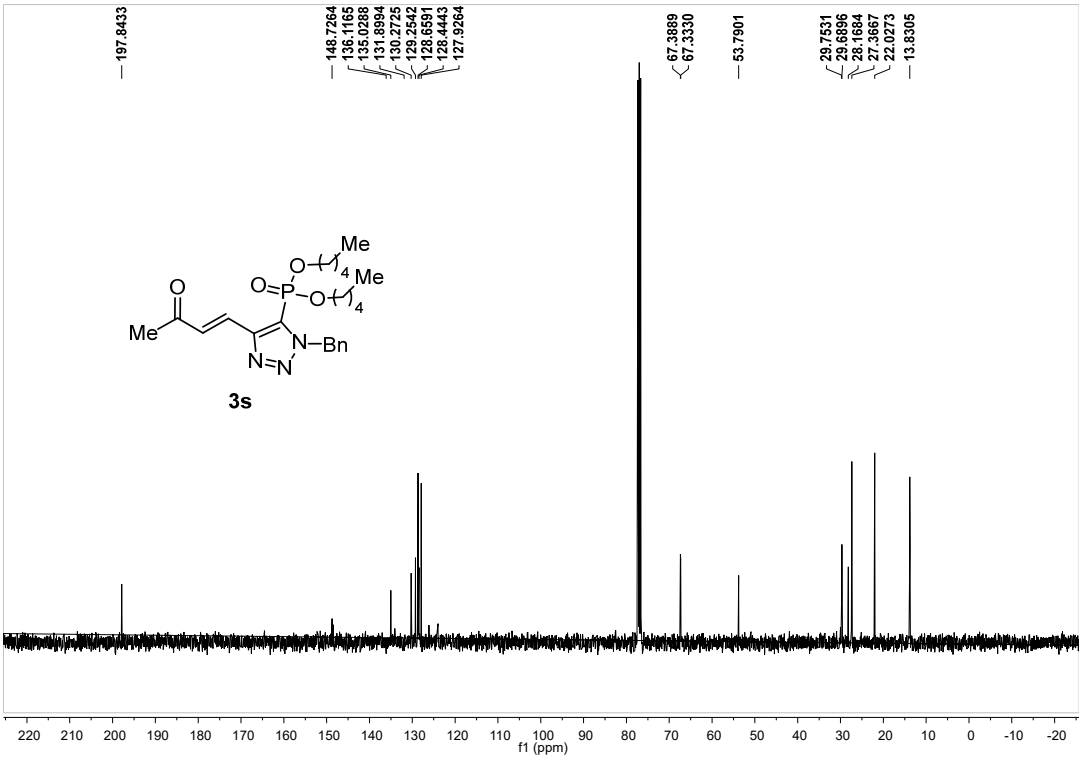

7.6977
7.6576
7.3247
7.3134
7.3066
7.2754
7.2668
5.8938
3.7554
3.7384
3.7314
3.7213
3.7145
3.6975
3.5106
3.4936
3.4867
3.4764
3.4697
3.4526
2.3744
1.8279
1.8112
1.7946
1.7778
1.7613
1.7446
0.8461
0.8289
0.8242
0.8071
O
Me
O—i-Bu
O=P
O—i-Bu
N—Bn
N=N
1.00
5.62
2.02
2.09
2.10
3.10
2.34
12.05
8.0
7.5
7.0
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
f1 (ppm)

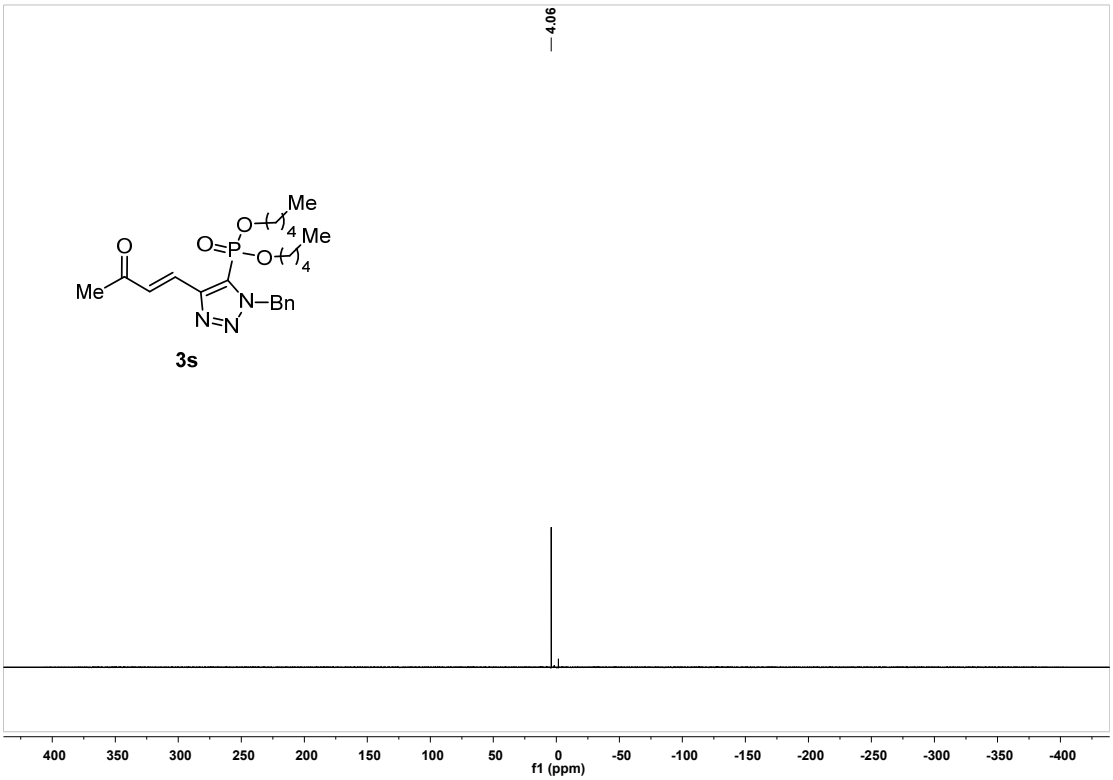
3r

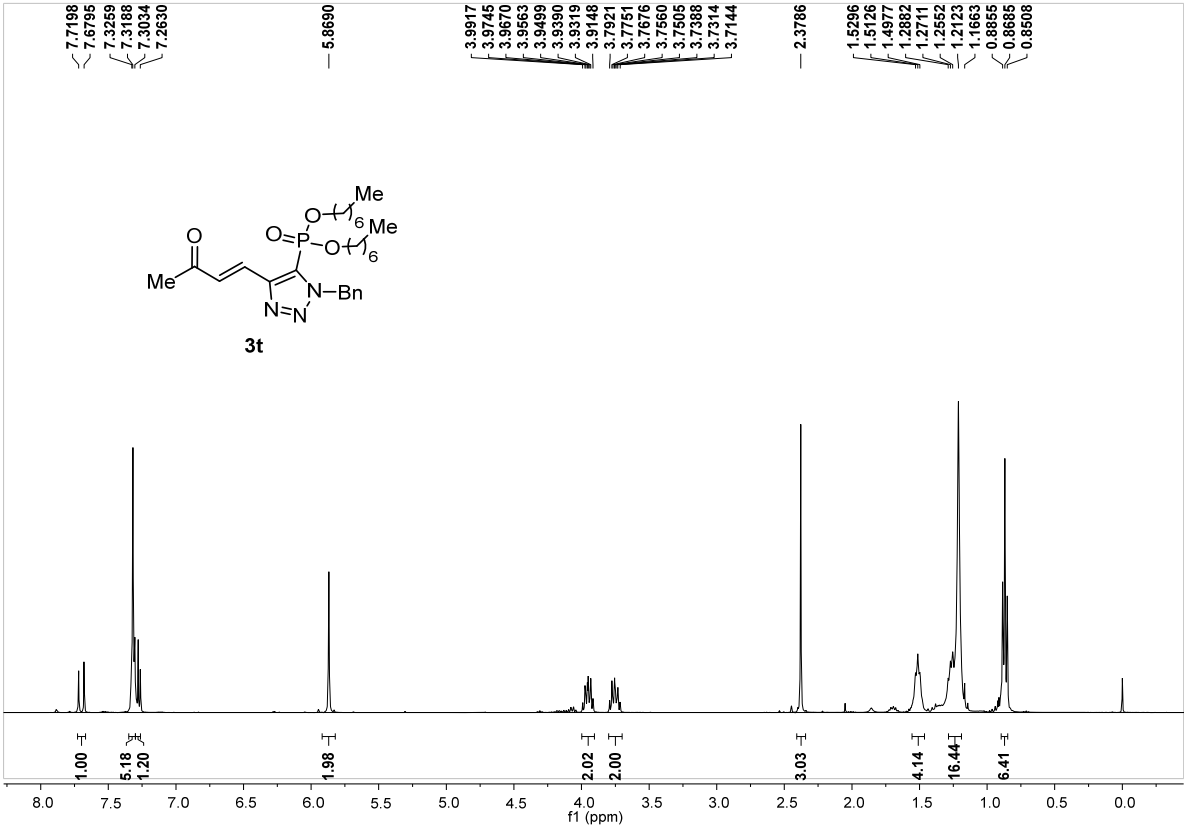

197.8134
148.6228
135.0795
130.2024
129.1542
128.6788
128.4596
127.9816
124.9251
123.9212
72.9598
53.7900
28.8762
28.8090
28.2349
18.5788
18.4636
O
O=P
O—i-Bu
O—i-Bu
Me
N—Bn
N=N
3r
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

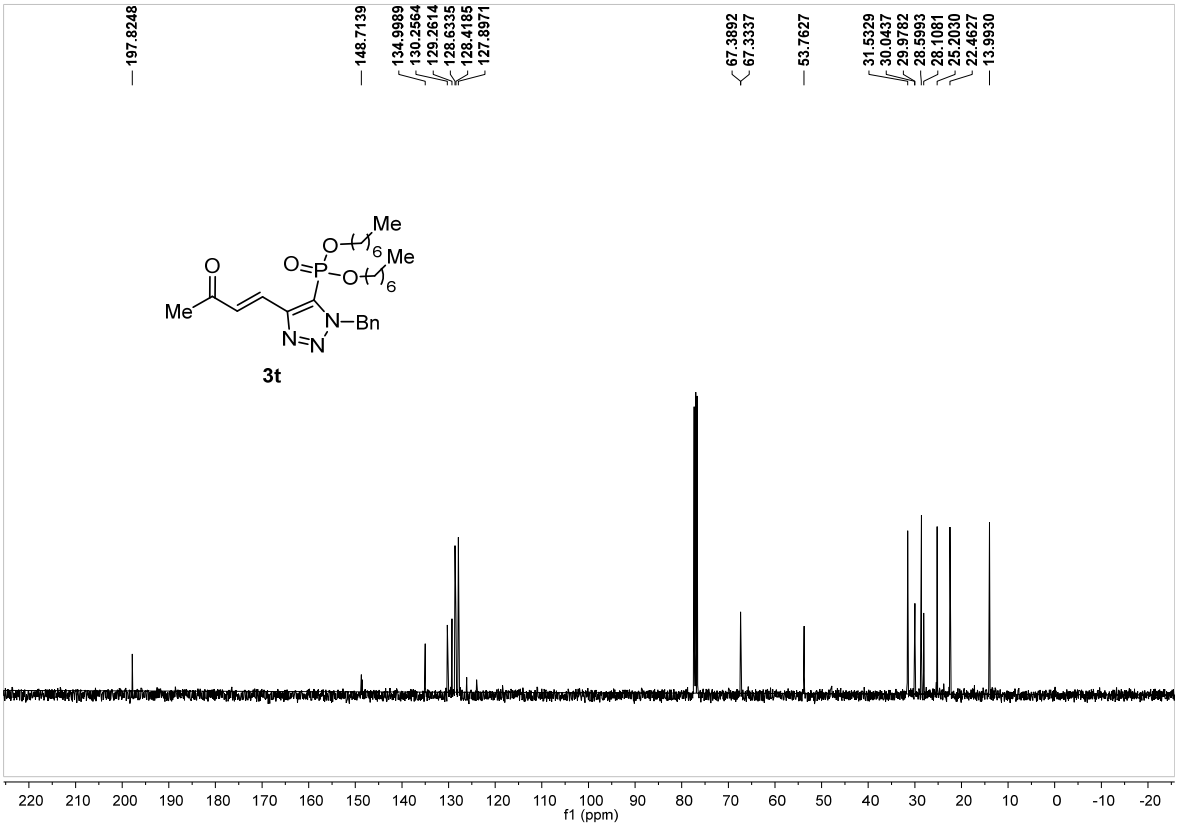

3.98
O
O=P
O—i-Bu
O—i-Bu
Me
N—Bn
N=N
3r
50 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -3
f1 (ppm)

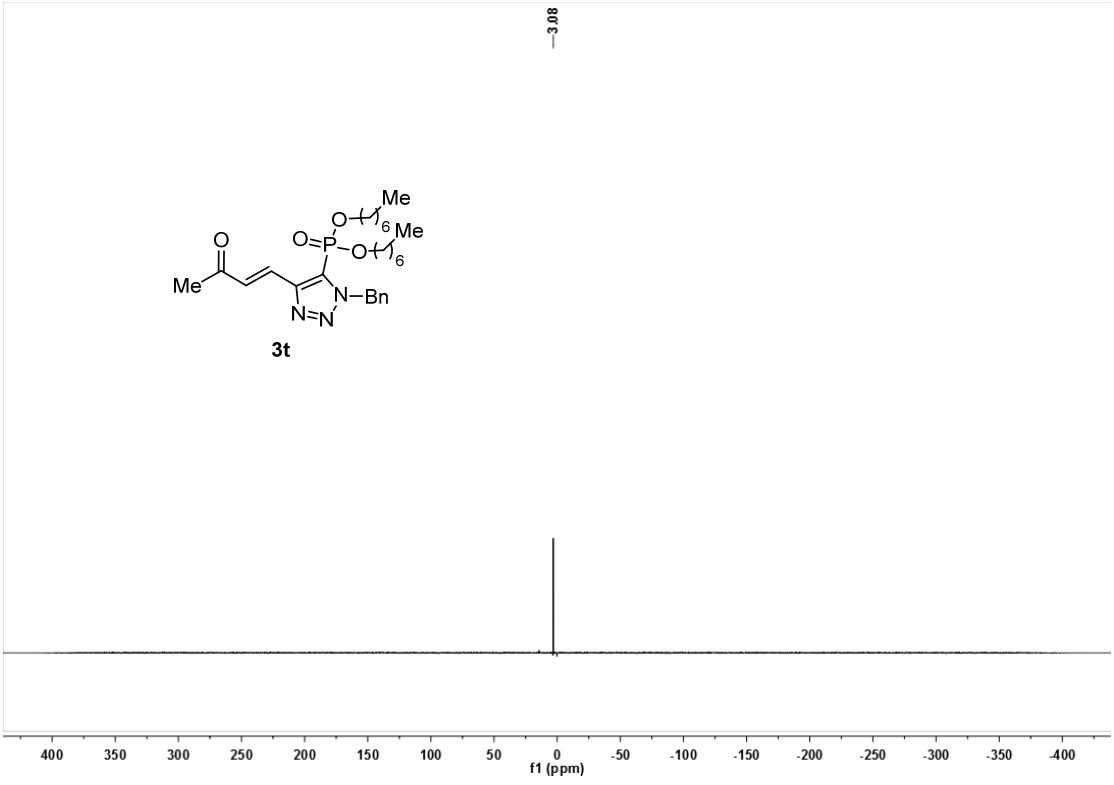

7.7171
7.6770
7.3330
7.3277
7.3199
7.3045
7.2726
5.8718
3.9928
3.9757
3.9682
3.9575
3.9511
3.9404
3.9328
3.9158
3.7962
3.7795
3.7717
3.7602
3.7546
3.7433
3.7354
3.7185
2.3782
1.5216
1.5041
1.2430
1.2296
1.2253
1.2217
1.2174
1.2143
0.8708
0.8538
0.8360
Me
O
4
Me
O=P
O
4
O
Me
N–Bn
N=N
3s
1.00
5.37
2.00
2.00
2.01
2.99
4.10
8.35
6.22
f1 (ppm)

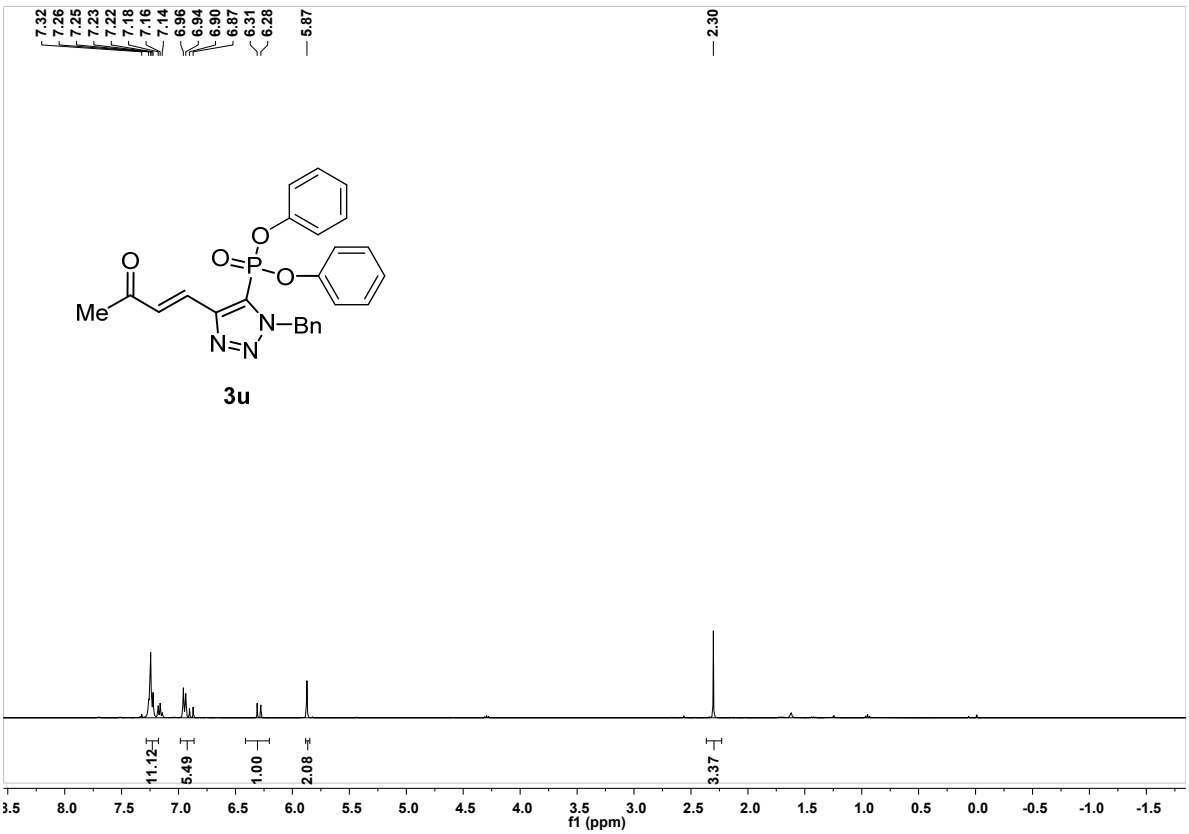

197.8433
148.7264
136.1165
135.0288
131.8994
130.2725
129.2542
128.6591
128.4443
127.9264
67.3889
67.3330
53.7901
29.7531
29.6896
28.1684
27.3667
22.0273
13.8305
Me
O
4
Me
O=P
O
4
O
Me
N–Bn
N=N
3s
f1 (ppm)


4.06
Me
O
O
P
O
4
Me
4
O
Me
N–Bn
N=N
3s
400
350
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)

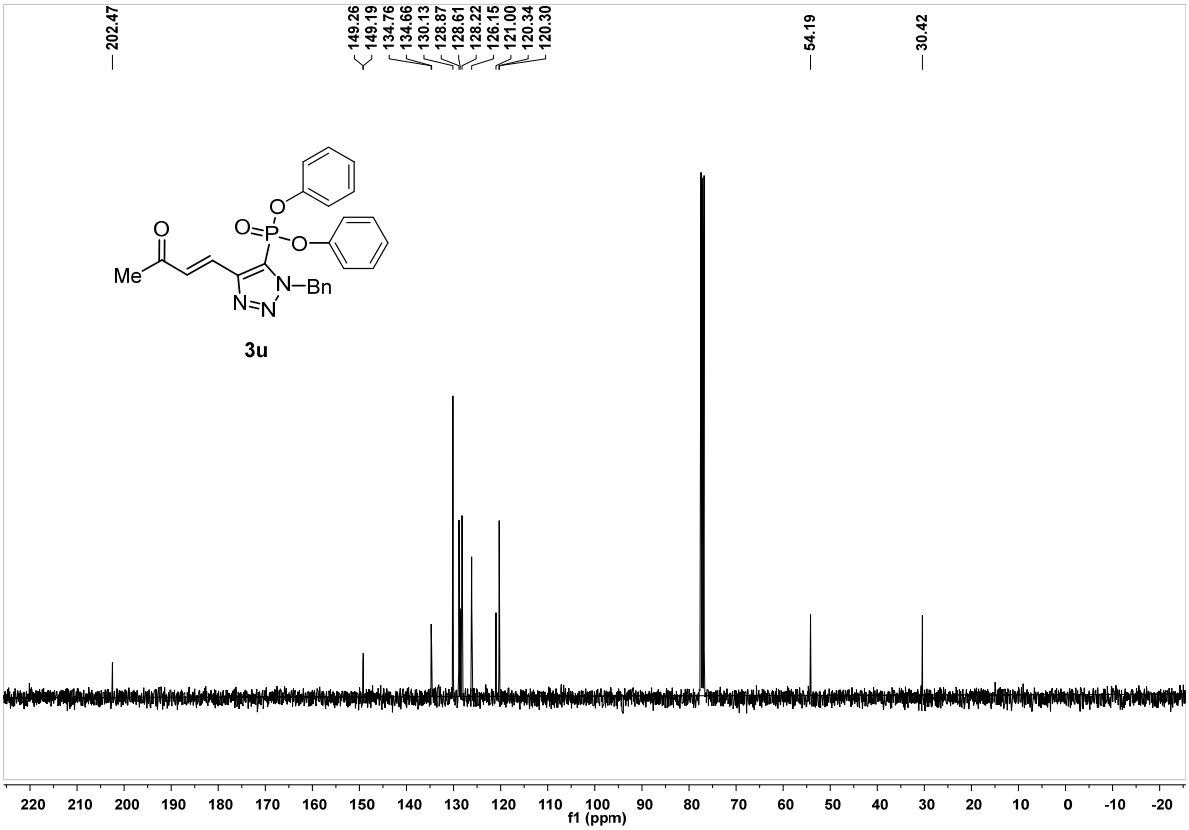

7.7198
7.6795
7.3259
7.3188
7.3034
7.2630
5.8690
3.9917
3.9745
3.9670
3.9563
3.9499
3.9390
3.9319
3.9148
3.7921
3.7751
3.7676
3.7560
3.7505
3.7388
3.7314
3.7144
2.3786
1.5296
1.5126
1.4977
1.2882
1.2711
1.2552
1.2123
1.1663
0.8855
0.8685
0.8508
Me
O
O
P
O
6
Me
6
O
Me
N–Bn
N=N
3t
1.00
5.18
1.20
1.98
2.02
2.00
3.03
4.14
16.44
6.41
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
f1 (ppm)


197.8248
148.7139
134.9989
130.2564
129.2614
128.6335
128.4185
127.8971
67.3892
67.3337
53.7627
31.5329
30.0437
29.9782
28.5993
28.1081
25.2030
22.4627
13.9930
Me
O
6
Me
O
P
O
6
Me
N–Bn
N=N
3t
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

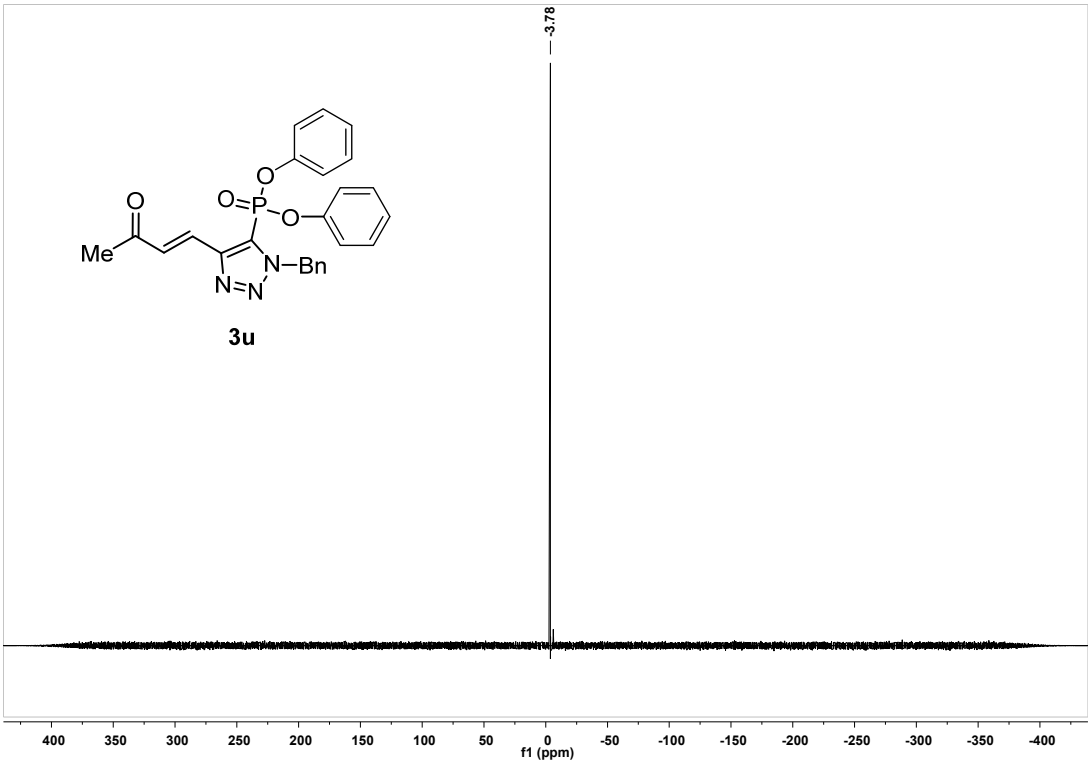

3.08
Me
O
6
Me
O
P
O
6
Me
N–Bn
N=N
3t
400 350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400
f1 (ppm)

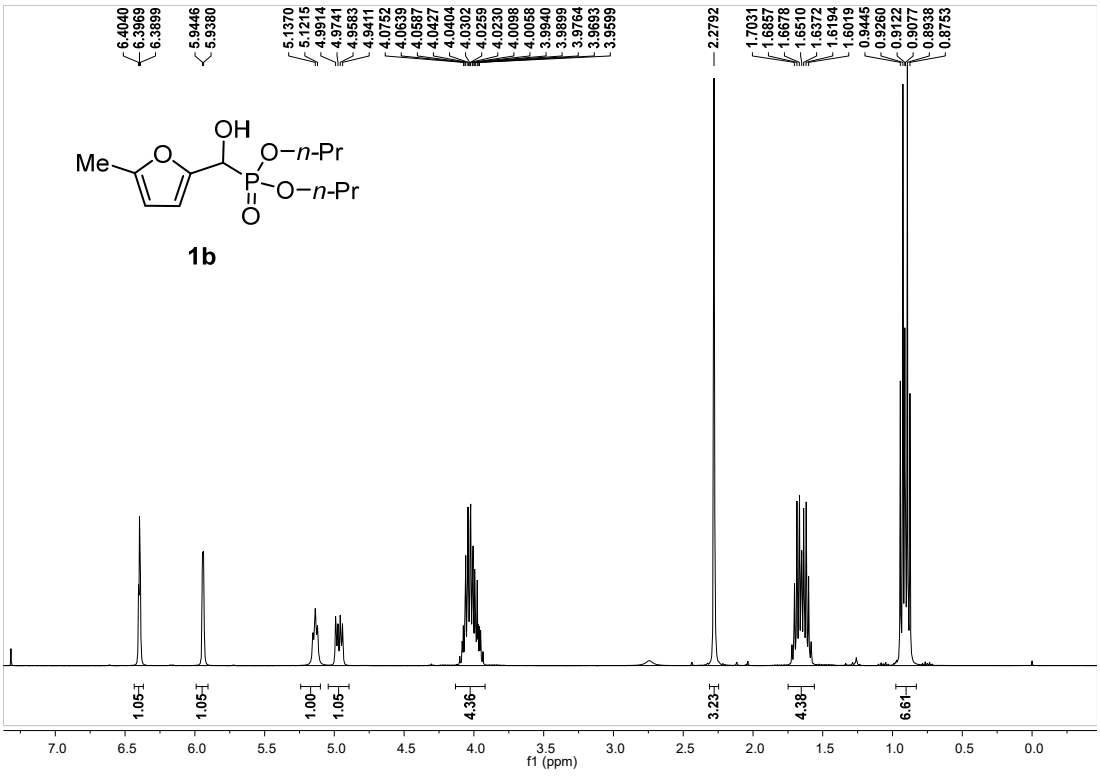

7.32
7.26
7.25
7.23
7.22
7.18
7.16
7.14
6.96
6.94
6.90
6.87
6.31
6.28
5.87
2.30
O
O=P
O
O
Me
N–Bn
N=N
11.12
5.49
1.00
2.08
3.37
f1 (ppm)

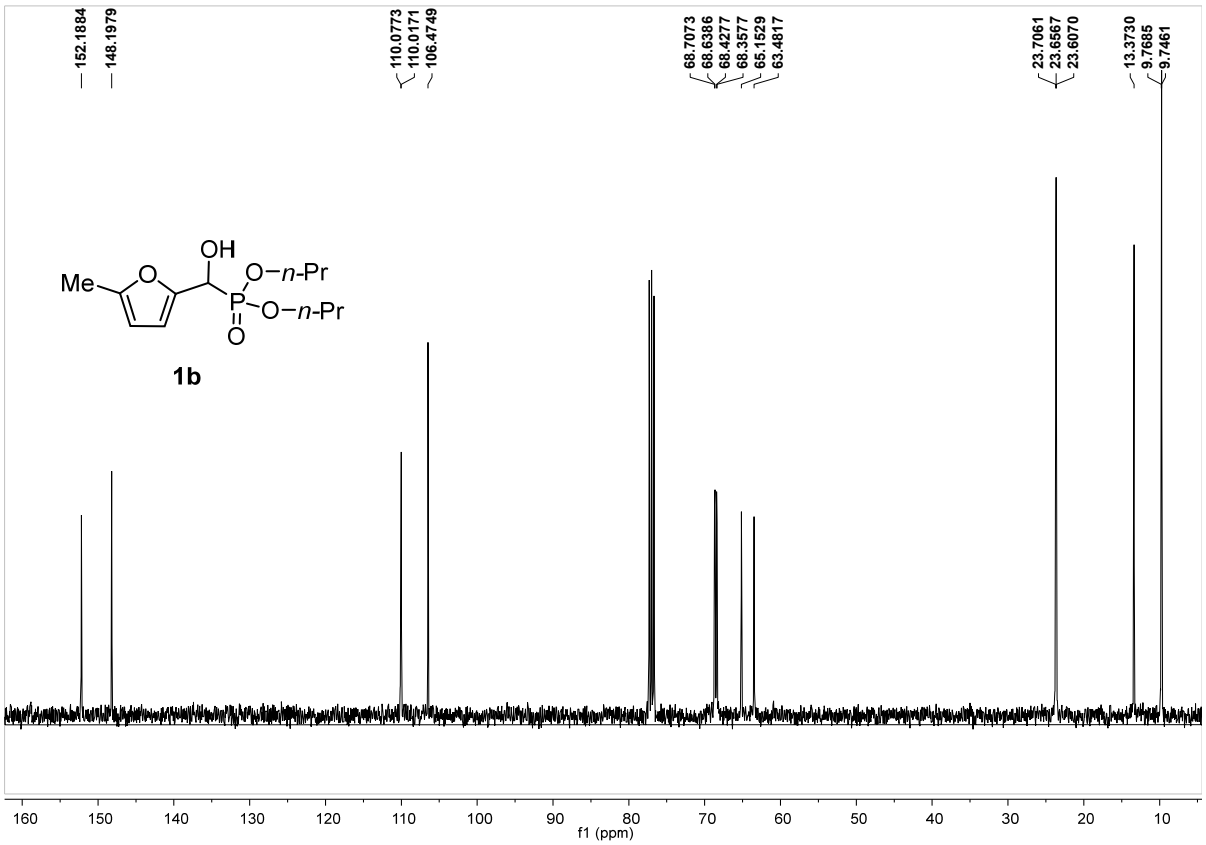
3u

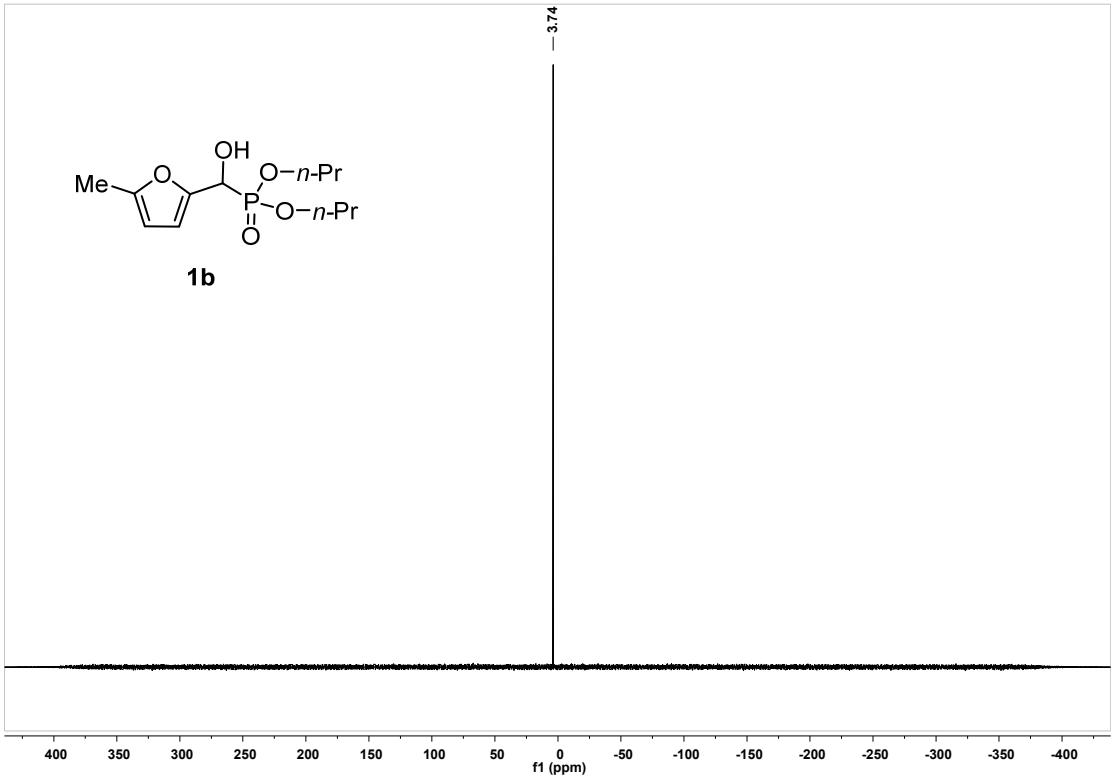

202.47
149.26
149.19
134.76
134.66
130.13
128.87
128.61
128.22
126.15
121.00
120.34
120.30
54.19
30.42
O
O=P
O
O
Me
N–Bn
N=N
f1 (ppm)

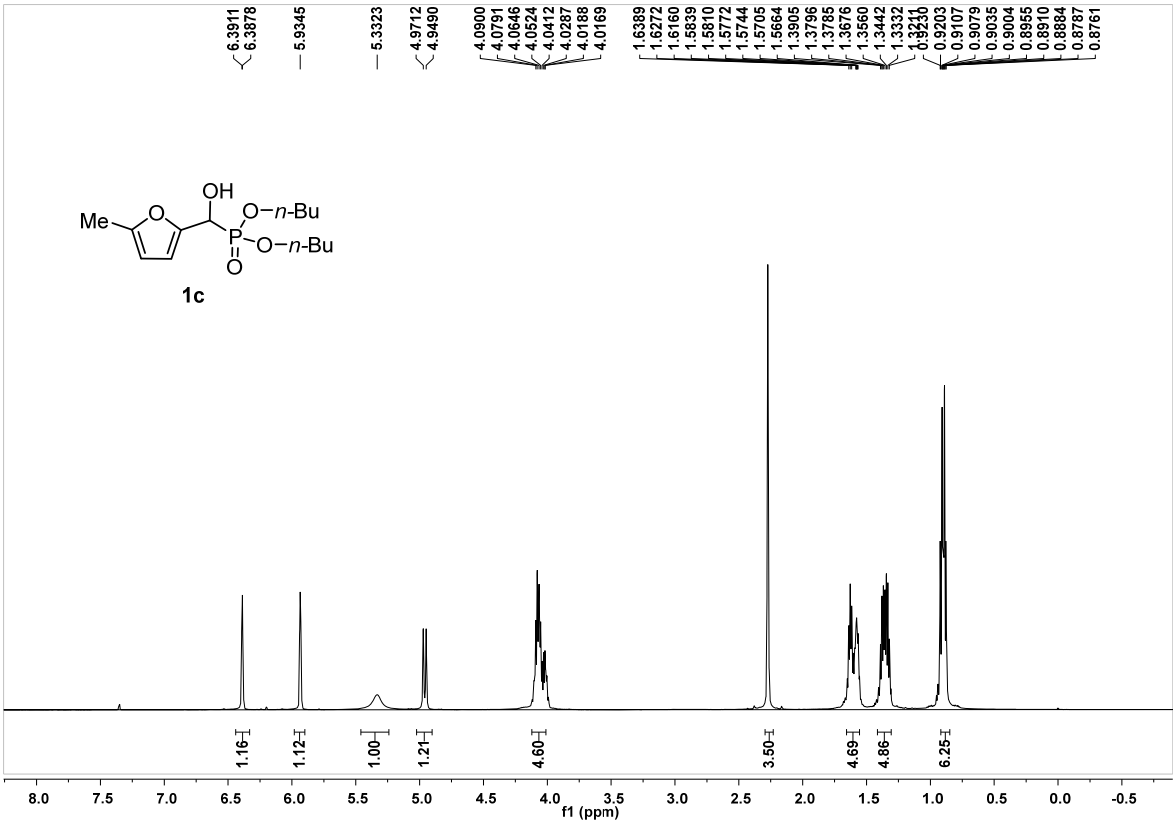
3u

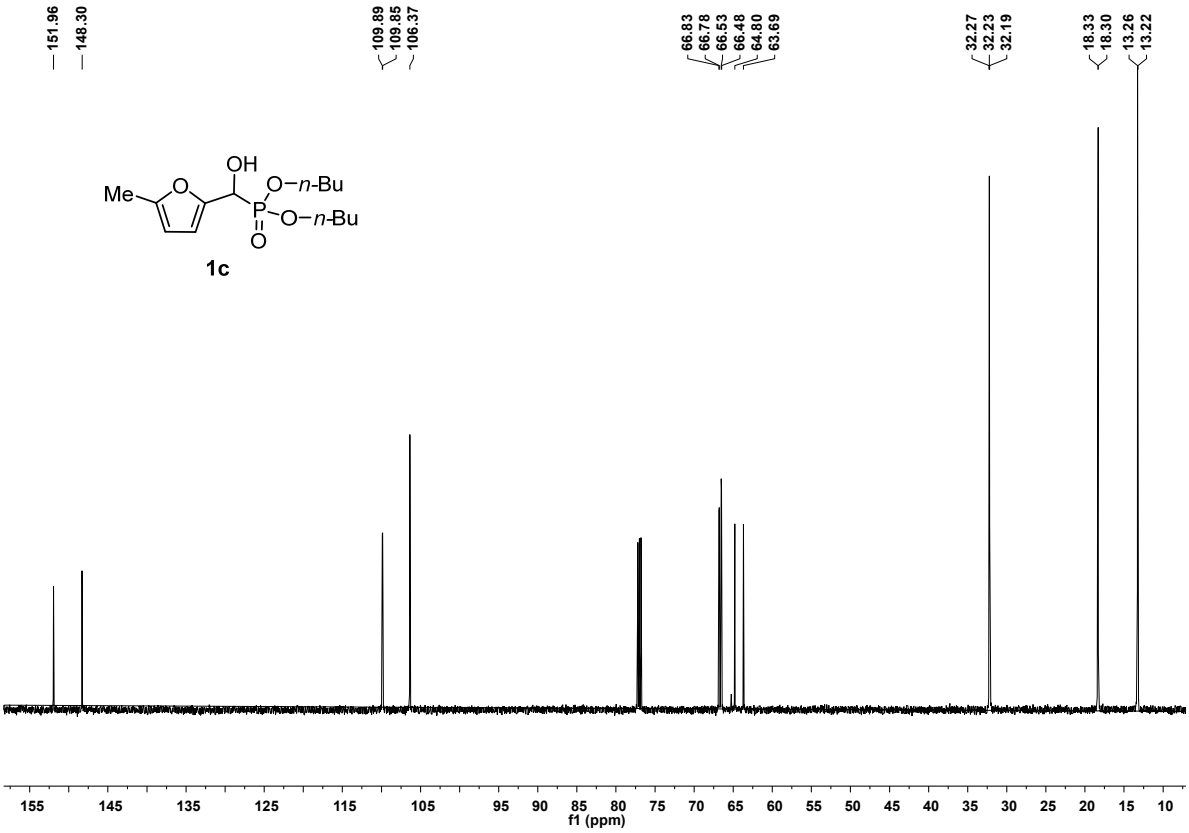

-3.78
O
O=P
O
Me
N–Bn
N=N
3u
400 350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400
f1 (ppm)

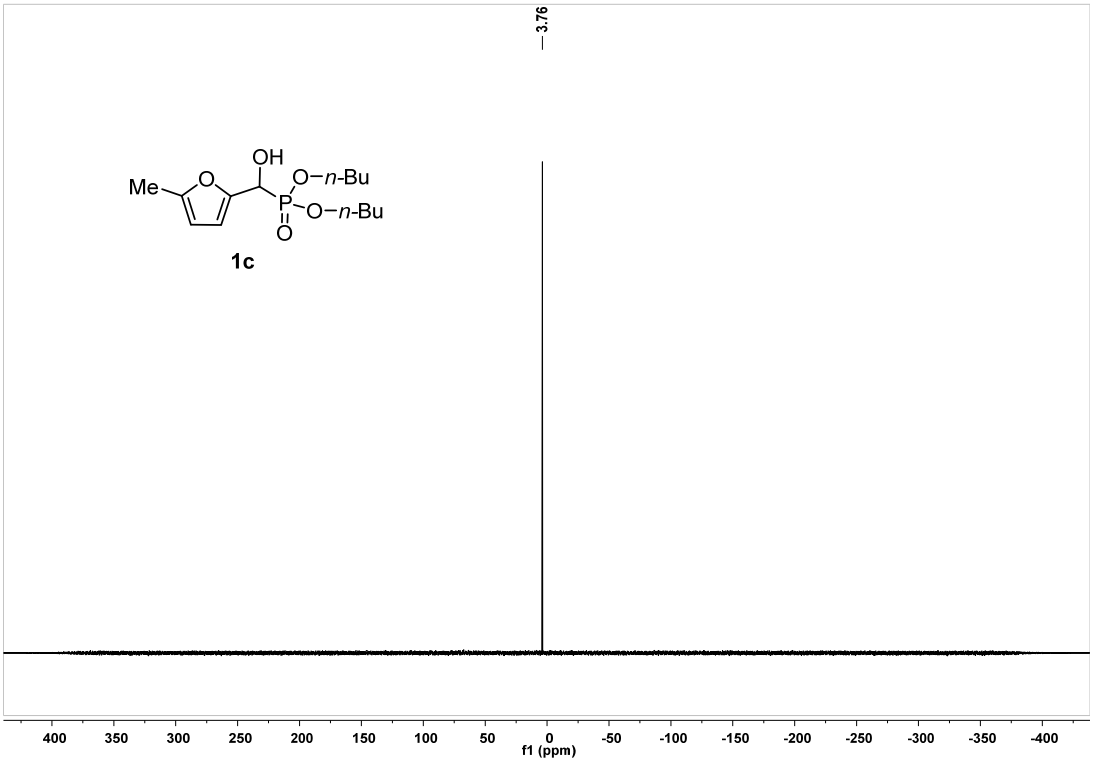

6.4040
6.3969
6.3899
5.9446
5.9380
5.1370
5.1215
4.9914
4.9741
4.9583
4.9411
4.0752
4.0639
4.0587
4.0427
4.0404
4.0302
4.0259
4.0230
4.0098
4.0058
3.9940
3.9899
3.9764
3.9693
3.9599
2.2792
1.7031
1.6857
1.6678
1.6510
1.6372
1.6194
1.6019
0.9445
0.9260
0.9122
0.9077
0.8938
0.8753
OH
Me
O
O–n-Pr
P
O–n-Pr
O
1b
1.05
1.05
1.00
1.05
4.36
3.23
4.38
6.61
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
f1 (ppm)

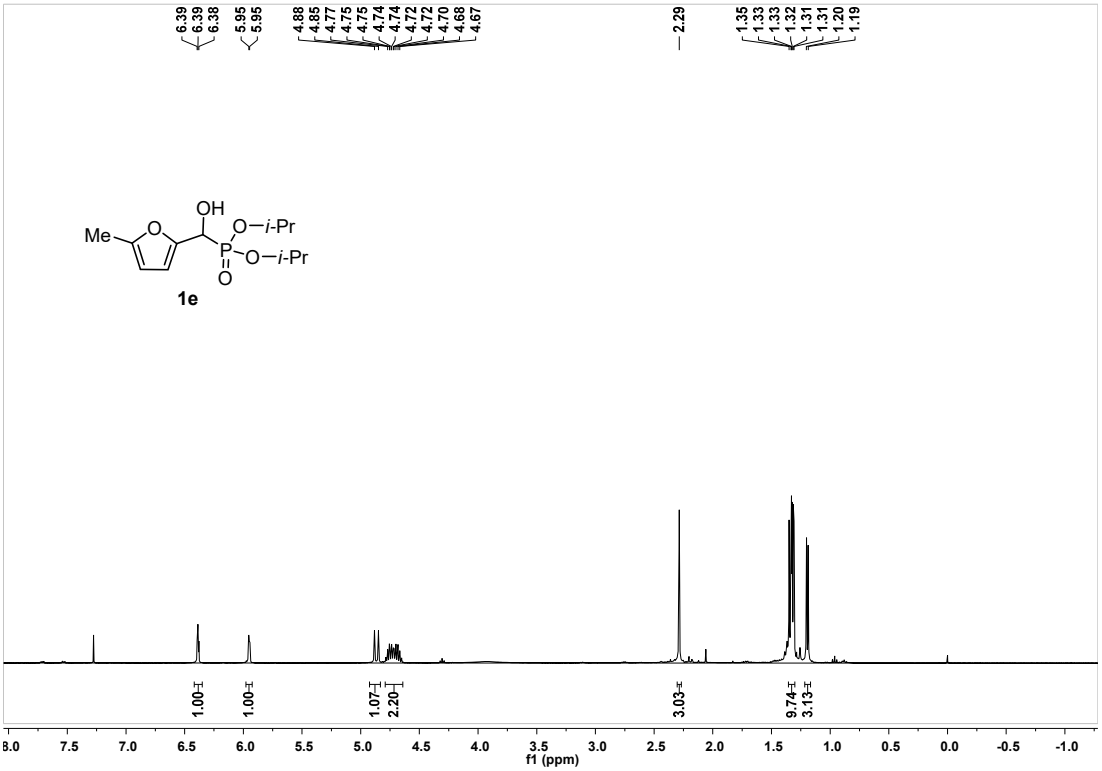

152.1884
148.1979
110.0773
110.0171
106.4749
68.7073
68.6386
68.4277
68.3577
65.1529
63.4817
23.7061
23.6567
23.6070
13.3730
9.7685
9.7461
OH
Me
O
O–n-Pr
P
O–n-Pr
O
1b
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

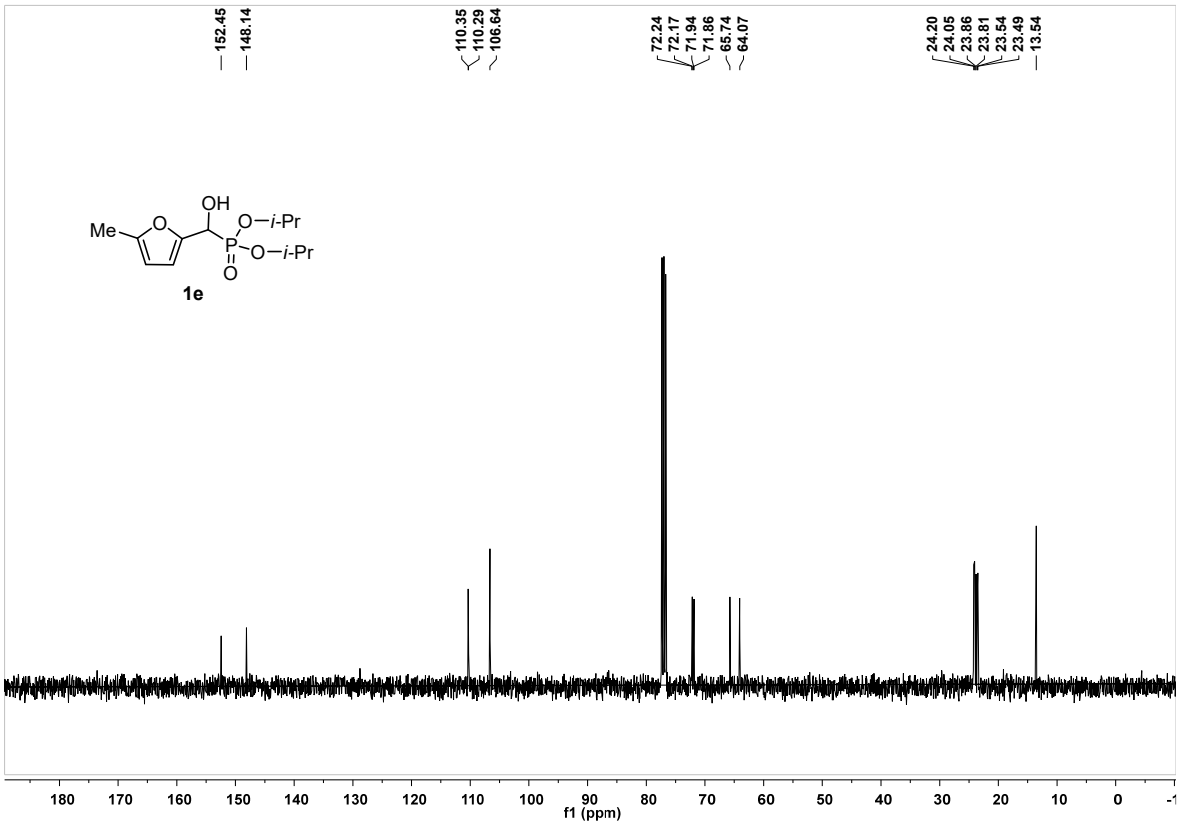

3.74
OH
Me
O
O–n-Pr
P
O–n-Pr
O
1b
400
350
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)

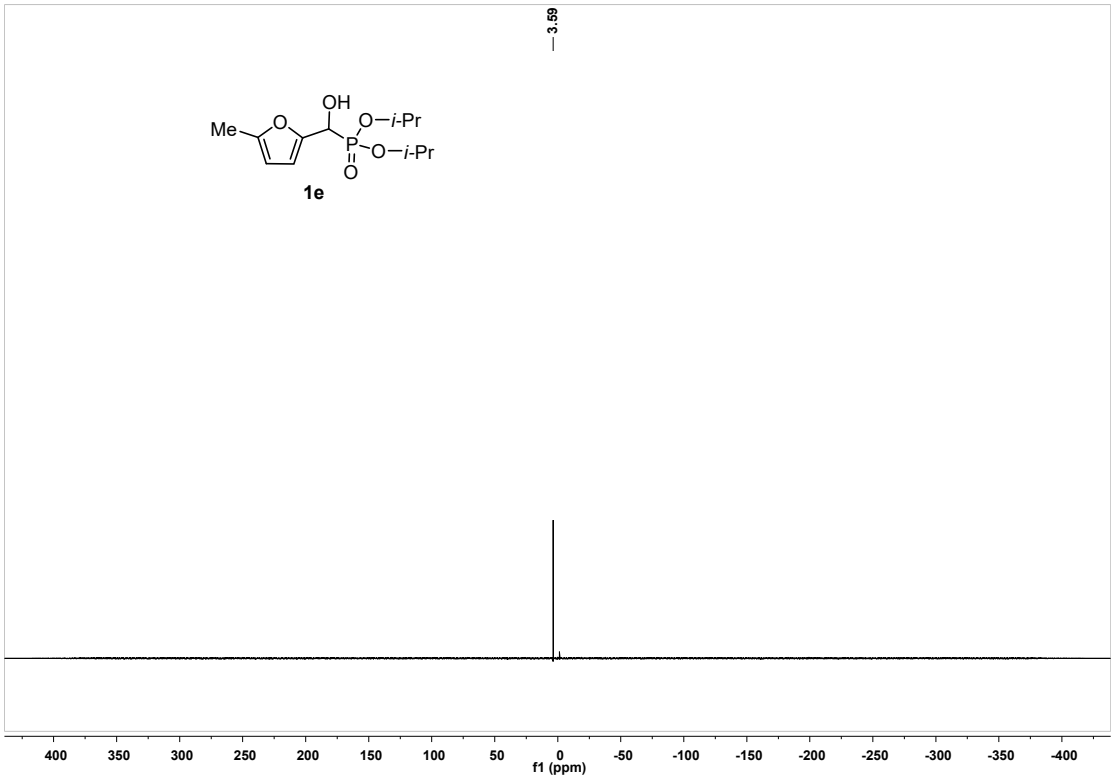

6.3911
6.3878
5.9345
5.3323
4.9712
4.9490
4.0900
4.0791
4.0646
4.0524
4.0412
4.0287
4.0188
4.0169
1.6389
1.6272
1.6160
1.5839
1.5810
1.5772
1.5744
1.5705
1.5664
1.3905
1.3796
1.3785
1.3676
1.3560
1.3442
1.3332
0.9203
0.9107
0.9079
0.9035
0.9004
0.8955
0.8910
0.8884
0.8787
0.8761
OH
Me
O
O−n-Bu
P
O−n-Bu
O
1c
1.16
1.12
1.00
1.21
4.60
3.50
4.69
4.86
6.25
f1 (ppm)

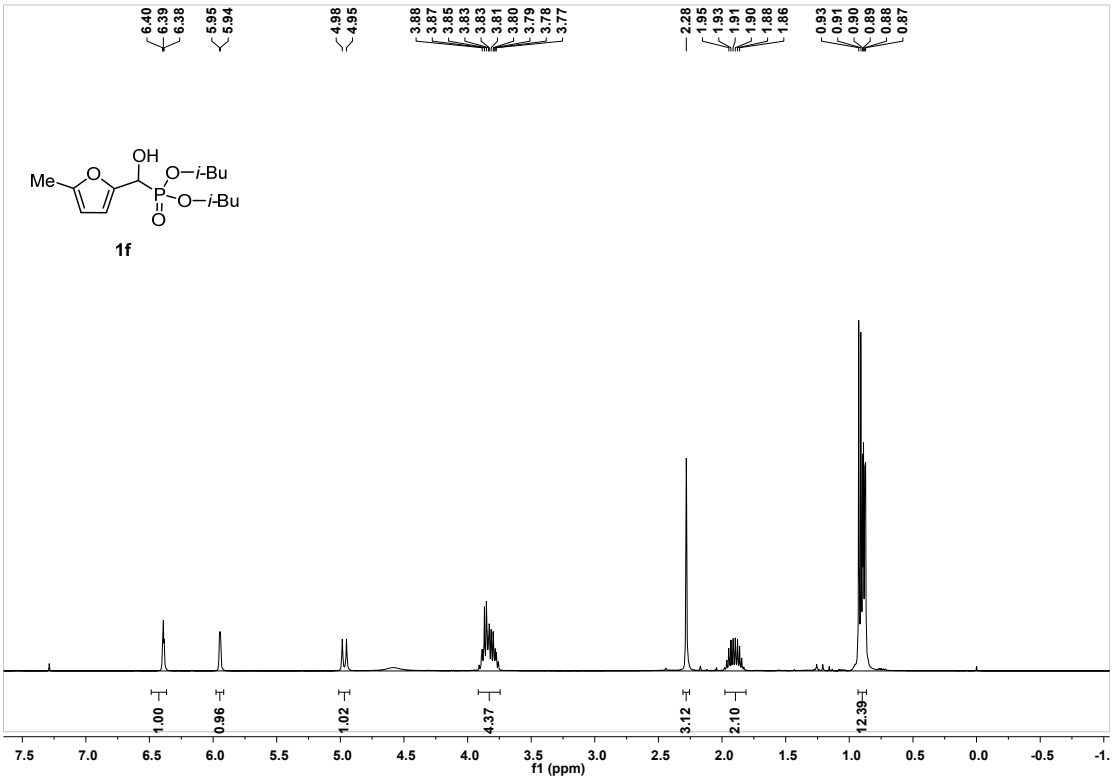

151.96
148.30
109.89
109.85
106.37
66.83
66.78
66.53
66.48
64.80
63.69
32.27
32.23
32.19
18.33
18.30
13.26
13.22
OH
Me
O
O−n-Bu
P
O−n-Bu
O
1c
f1 (ppm)

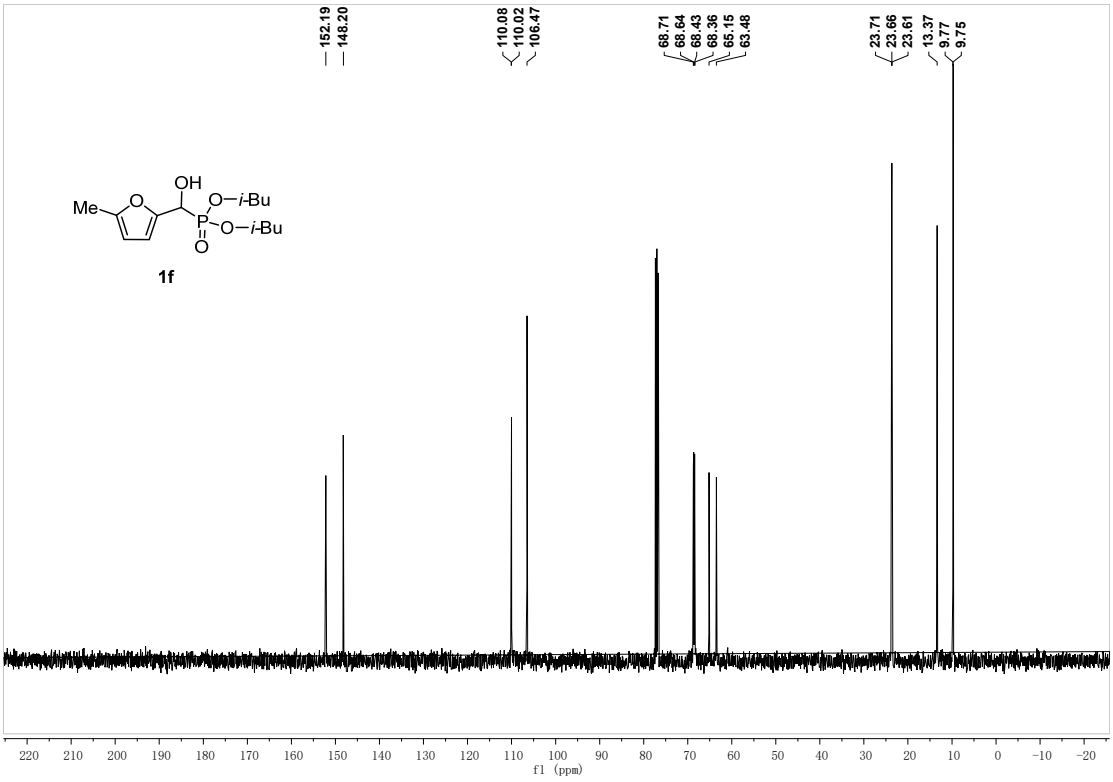

3.76
OH
Me
O
O−n-Bu
P
O−n-Bu
O
1c
400 350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400
f1 (ppm)

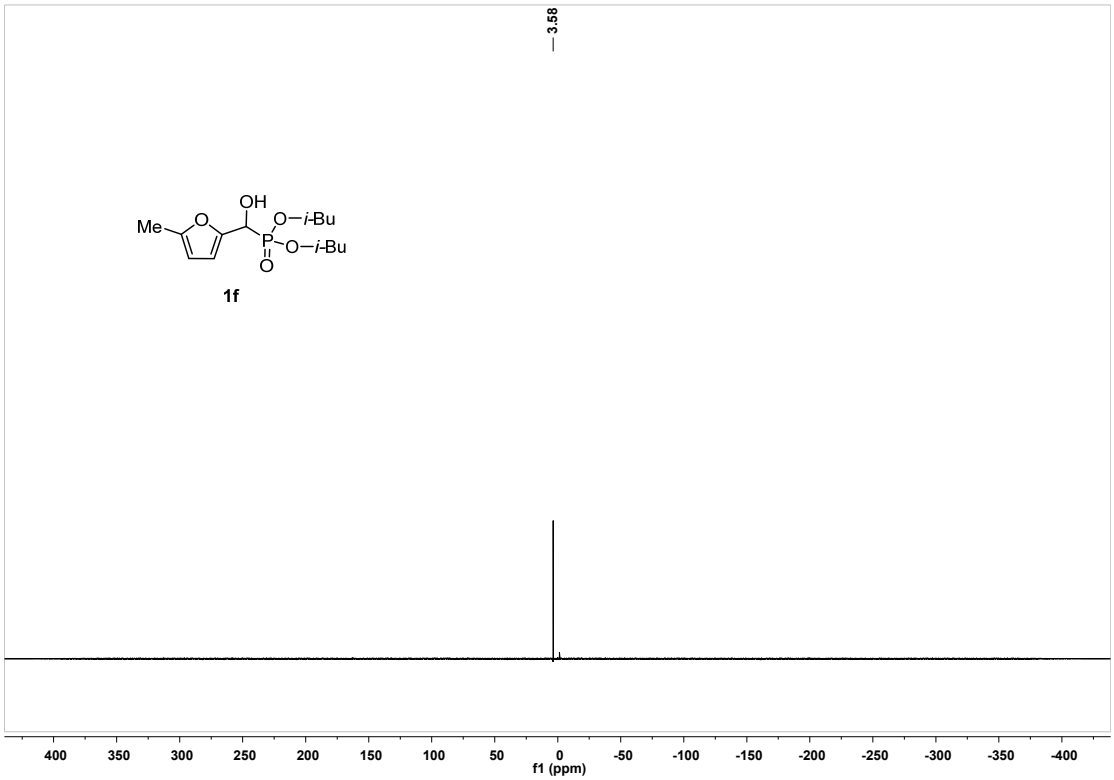

6.39 6.39 6.38
5.95 5.95
4.88 4.85 4.77 4.75 4.75 4.74 4.74 4.72 4.72 4.70 4.68 4.67
2.29
1.35 1.33 1.33 1.32 1.31 1.31 1.20 1.19
OH
Me
O
O−i-Pr
P
O−i-Pr
O
1e
1.00 1.00 1.07 2.20 3.03 9.74 3.13
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

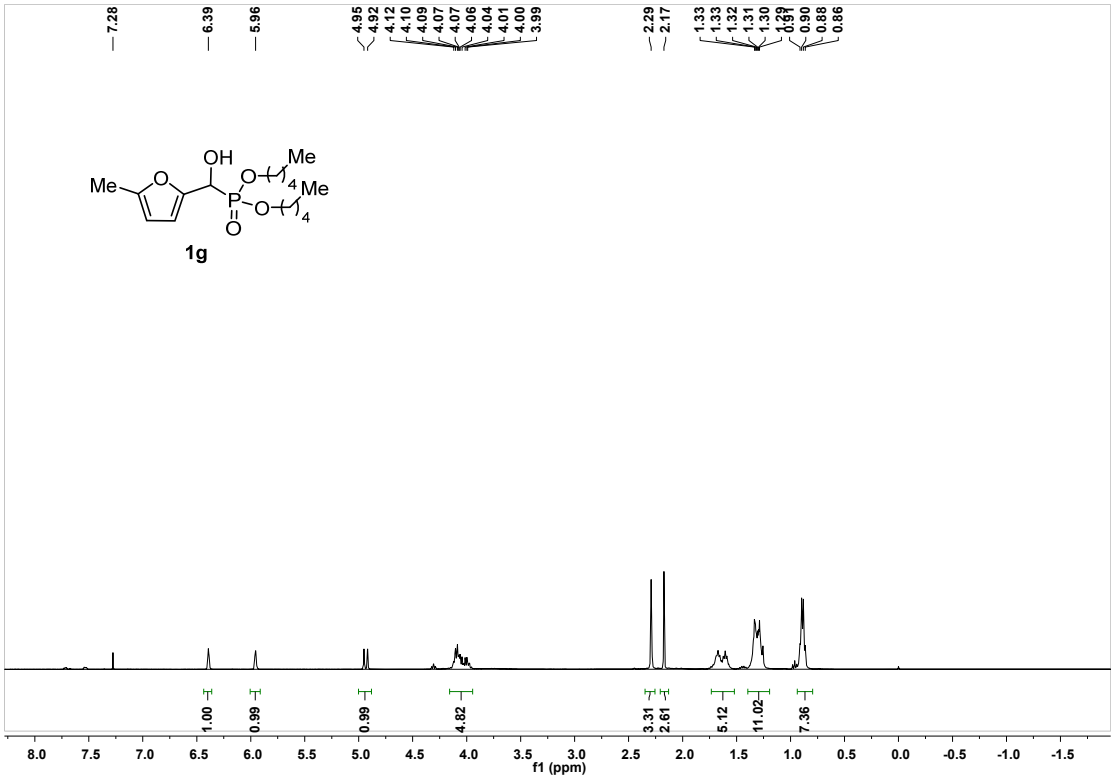

152.45
148.14
110.35
110.29
106.64
72.24
72.17
71.94
71.86
65.74
64.07
24.20
24.05
23.86
23.81
23.54
23.49
13.54
OH
Me
O
O—i-Pr
P
O—i-Pr
O
1e
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

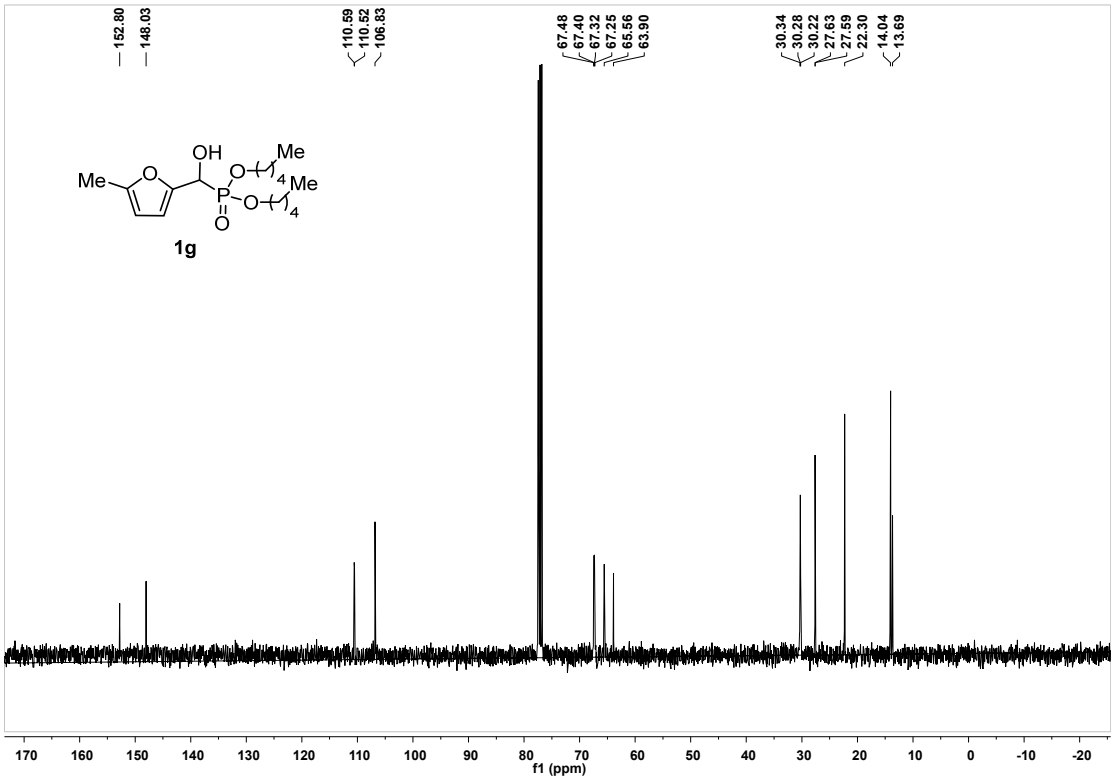

3.59
OH
Me
O
O—i-Pr
P
O—i-Pr
O
1e
400 350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400
f1 (ppm)

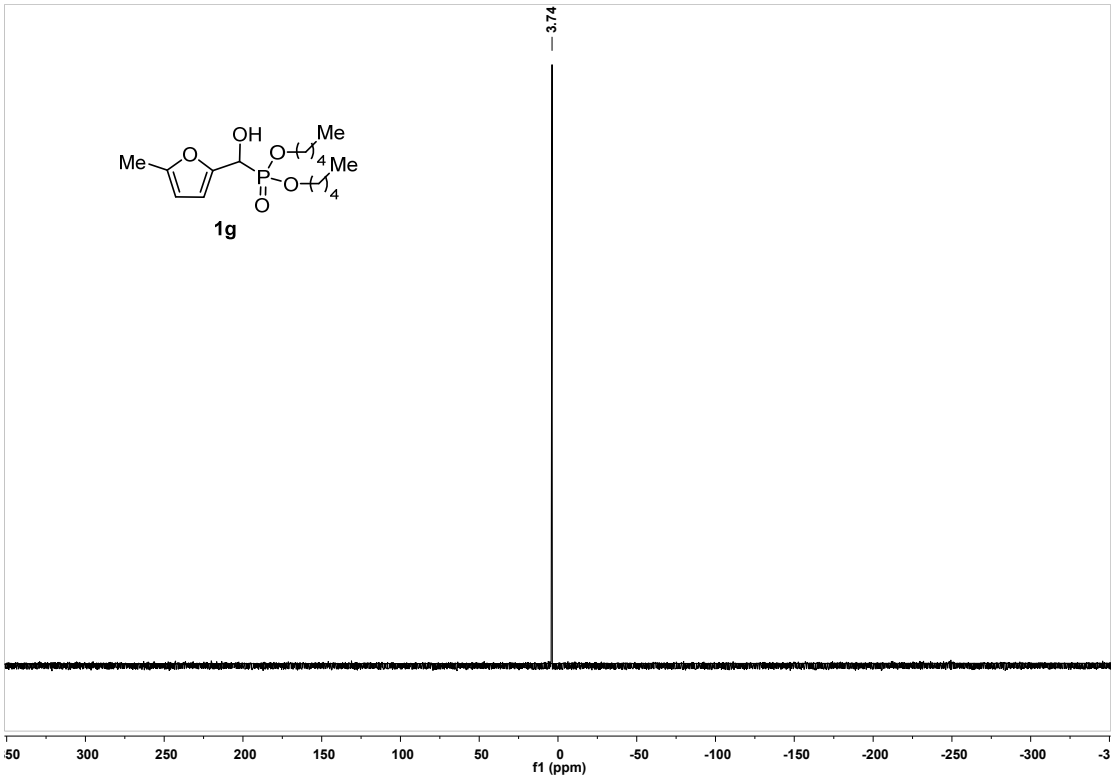

6.40
6.39
6.38
5.95
5.94
4.98
4.95
3.88
3.87
3.85
3.83
3.83
3.81
3.80
3.79
3.78
3.77
2.28
1.95
1.93
1.91
1.90
1.88
1.86
0.93
0.91
0.90
0.89
0.88
0.87
OH
Me
O
O—i-Bu
P
O—i-Bu
O
1f
1.00
0.96
1.02
4.37
3.12
2.10
12.39
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

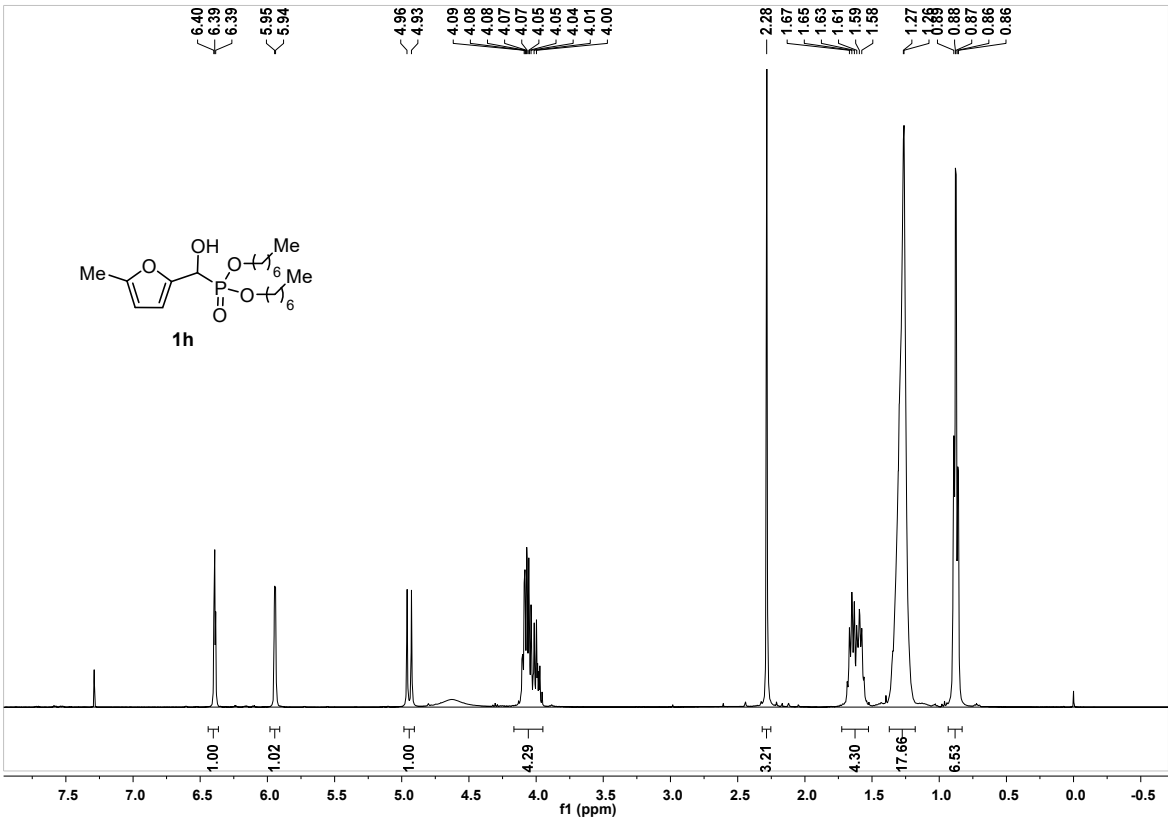

152.19
148.20
110.08
110.02
106.47
68.71
68.64
68.43
68.36
65.15
63.48
23.71
23.66
23.61
13.37
9.77
9.75
OH
Me
O
O—i-Bu
P
O—i-Bu
O
1f
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

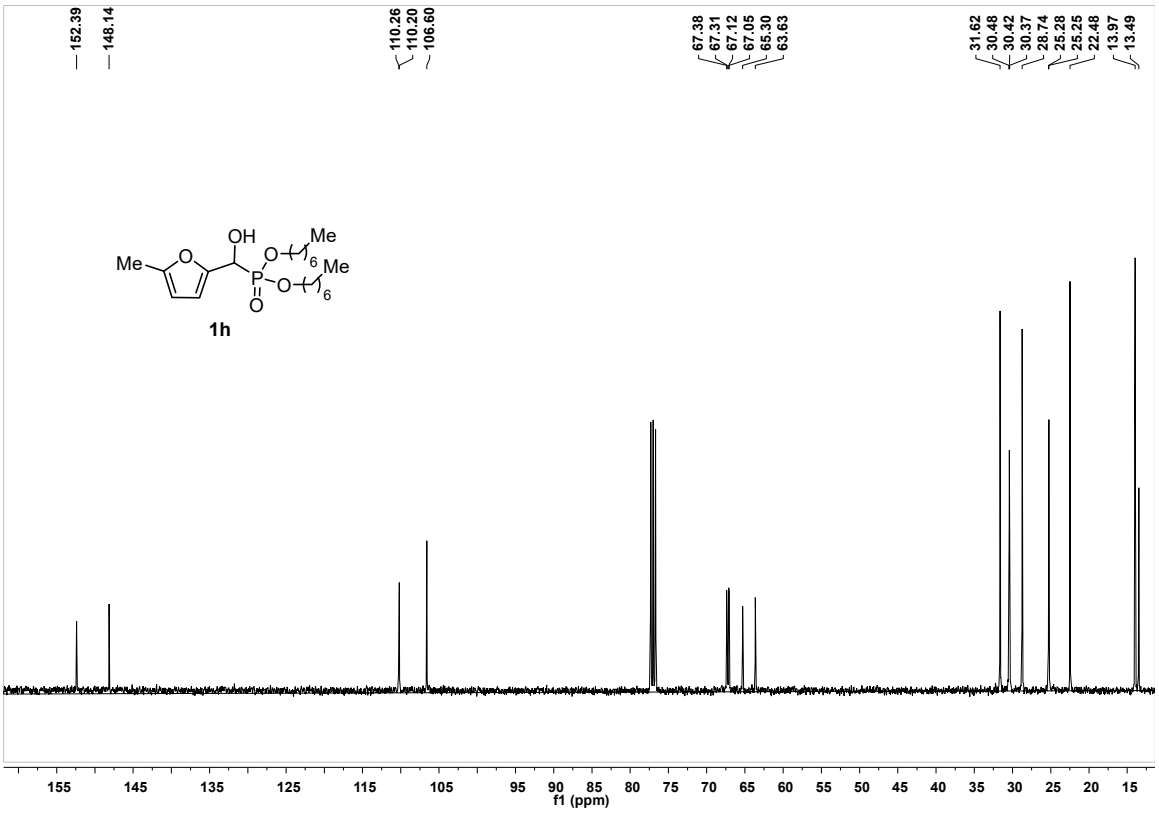

3.58
OH
Me
O
O—i-Bu
P
O—i-Bu
O
1f
400 350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400
f1 (ppm)

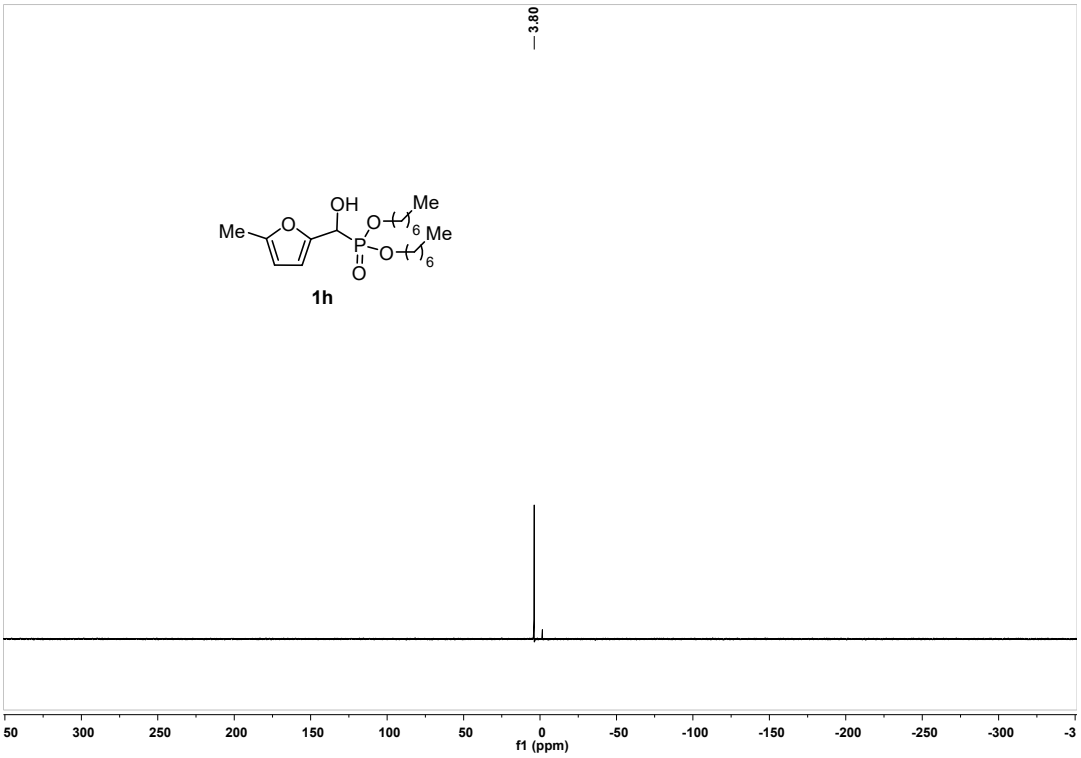

7.28
6.39
5.96
4.95 4.92 4.12 4.10 4.09 4.07 4.07 4.06 4.04 4.01 4.00 3.99
2.29 2.17
1.33 1.33 1.32 1.31 1.30 1.29 0.91 0.90 0.88 0.86
OH
Me
O
Me
P
O
O
4
Me
4
1g
1.00 0.99 0.99 4.82 3.31 2.61 5.12 11.02 7.36
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

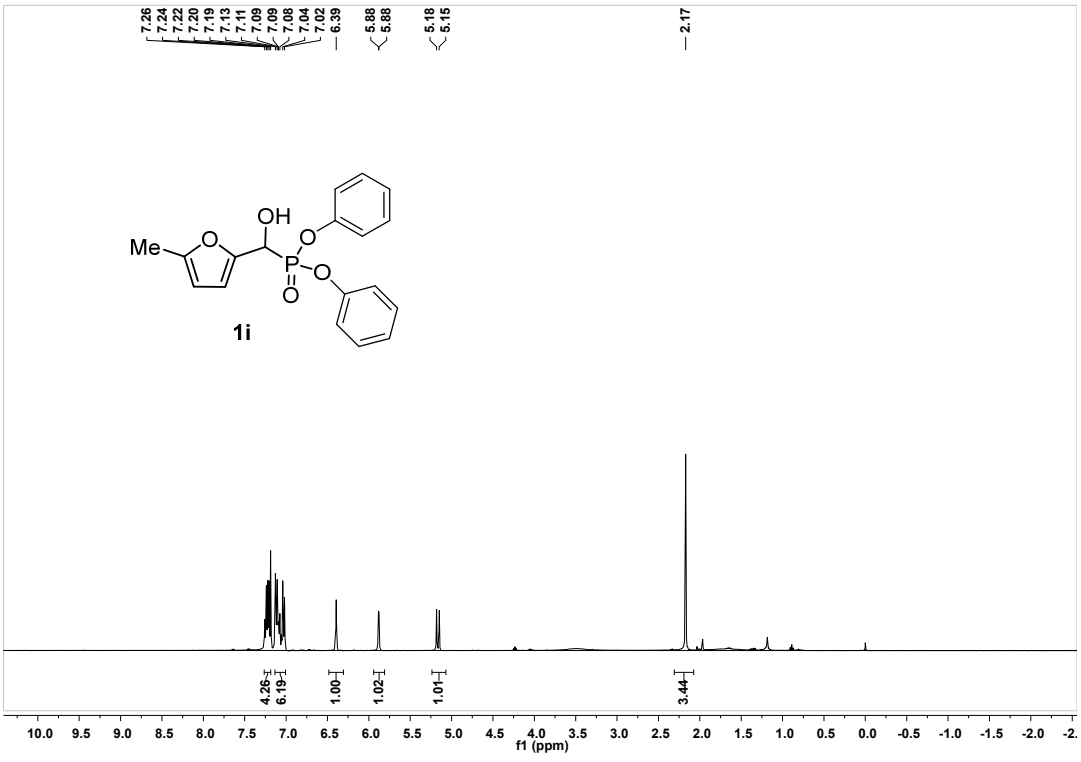

152.80
148.03
110.59
110.52
106.83
67.48
67.40
67.32
67.25
65.56
63.90
30.34
30.28
30.22
27.63
27.59
22.30
14.04
13.69
OH
Me
Me
O
P
O
O
4
4
Me
1g
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

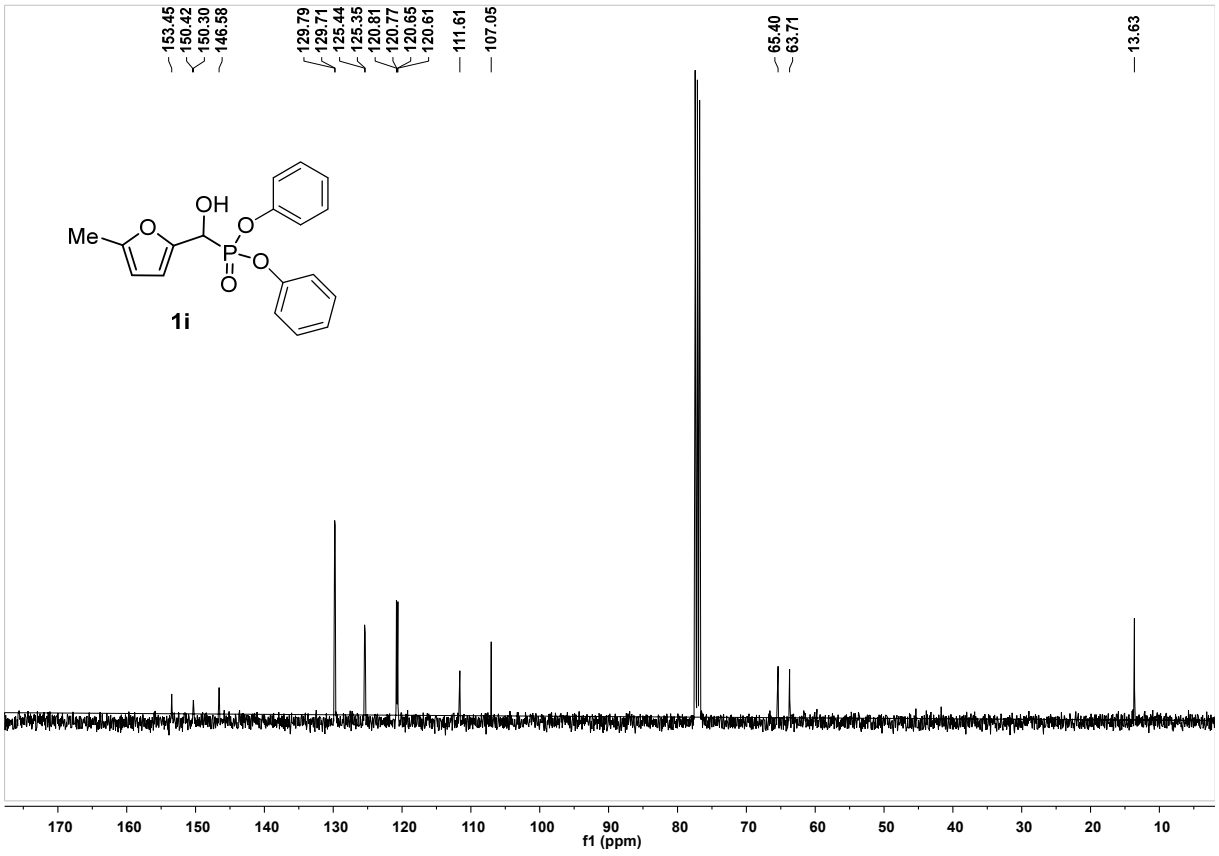

3.74
OH
Me
Me
O
P
O
O
4
4
Me
1g
50 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -3
f1 (ppm)

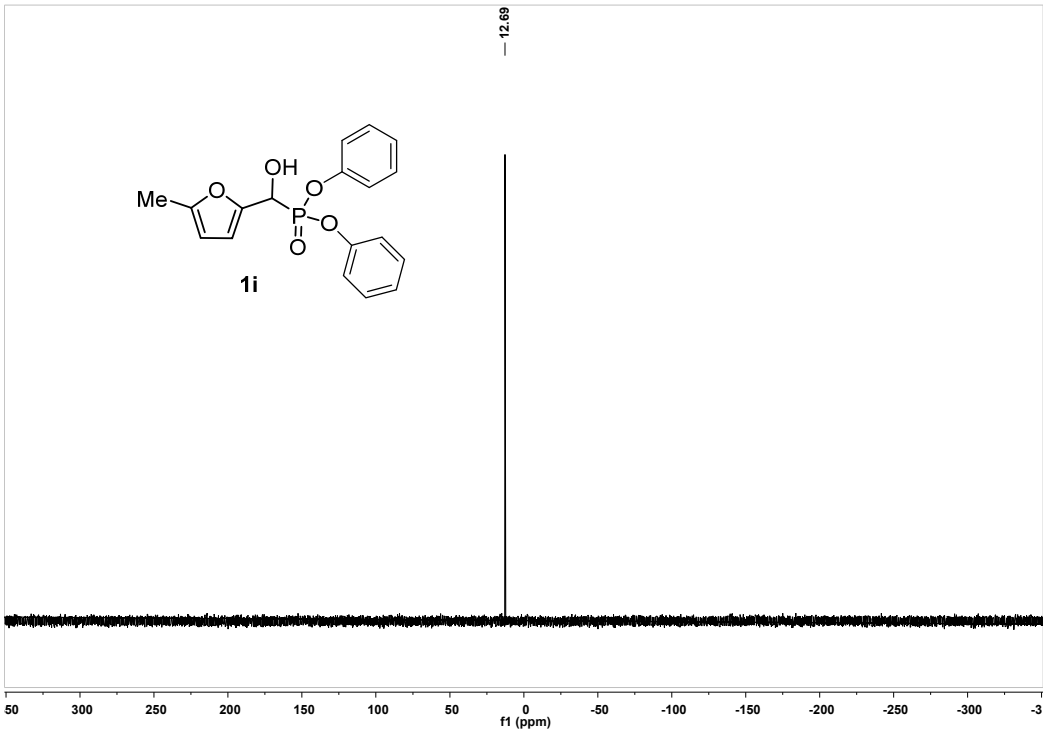

6.40
6.39
6.39
5.95
5.94
4.96
4.93
4.09
4.08
4.08
4.07
4.07
4.05
4.05
4.04
4.01
4.00
2.28
1.67
1.65
1.63
1.61
1.59
1.58
1.27
0.88
0.87
0.86
0.86
OH
Me
O
Me
O
P
O
O
6
Me
6
1h
1.00
1.02
1.00
4.29
3.21
4.30
17.66
6.53
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


152.39
148.14
110.26
110.20
106.60
67.38
67.31
67.12
67.05
65.30
63.63
31.62
30.48
30.42
30.37
28.74
25.28
25.25
22.48
13.97
13.49
OH
Me
O
Me
O
P
O
O
6
Me
6
1h
155
145
135
125
115
105
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
f1 (ppm)


3.80
OH
Me
O
Me
P
O
O
6
Me
O
6
1h
50
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
-3
f1 (ppm)


7.26
7.24
7.22
7.20
7.19
7.13
7.11
7.09
7.09
7.08
7.04
7.02
6.39
5.88
5.88
5.18
5.15
2.17
OH
O
Me
O
P
O
O
1i
4.26
6.19
1.00
1.02
1.01
3.44
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
-1.0
-1.5
-2.0
-2.
f1 (ppm)


153.45
150.42
150.30
146.58
129.79
129.71
125.44
125.35
120.81
120.77
120.65
120.61
111.61
107.05
65.40
63.71
13.63
OH
Me
O
P
O
O
O
1i
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


12.69
OH
Me
O
P
O
O
O
1i
50
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
-3
f1 (ppm)