

Supporting Information for:

Visible Light–Mediated Access to Phosphate Esters

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1. General Methods and Materials

1.1 General

Proton nuclear magnetic resonance (^1H NMR) spectra were recorded at ambient temperature on a Bruker 500 (500 MHz) spectrometer. Chemical shifts (δ_{H}) are reported in ppm and quoted to the nearest 0.01 ppm relative to the residual protons in CDCl_3 (7.26 ppm), and coupling constants (J) are quoted in Hertz. Data are reported as follows: Chemical shift (number of protons, multiplicity, coupling constants). Coupling constants were quoted to the nearest 0.1 Hz and multiplicity reported according to the following convention: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad, and associated combinations for example: dd=doublet of doublet, dt=doublet of triplet. Carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded at ambient temperature on a Bruker 500 (126 MHz) spectrometer. Chemical shift (δ_{C}) was measured in ppm and quoted to the nearest 0.1 ppm relative to the residual solvent peaks in CDCl_3 (77.16 ppm).

Flash column chromatography were carried out with Merck silica gel 60 (0.040–0.063 mm). Chromatography fractions and stated reactions were monitored by TLC on Merck silica gel 60 F254 aluminum plates. The spots were visualized under UV light at 254 nm. UV-Visible spectra were recorded on a Perkin Elmer Lambda 40 Spectrophotometer. The high-resolution mass spectrometry (HRMS) analyses were performed using a Xevo G2-XS QToF WATERS mass spectrometer equipped with an electrospray ion source (ESI) operated in positive ion mode.

THF, diethylether, dichloromethane, toluene and acetonitrile were purified using an Innovative Technology PURESOLV® solvent purification system, ethyl acetate was dried over activated 3 Å MS. All Commercially available reagents were purchased (from Sigma Aldrich, Fischer, Alfa Aesar, Fisher Scientific, TCI Europe) and used without further purification.

1.2 EPR-ST experiments

EPR-ST experiments were carried out using an X-Band spectrometer (MS 400 Magnettech). The EPR spectra simulations were carried out using the WINSIM software.

2. Determination of Quantum Yield

Dicronica LED GU10-456 nm was used for measurement of quantum yield (Fig S1).

According to a procedure previously reported by Yoon,^[1] the photon flux of the blue LED was determined by standard ferrioxalate actinometry. A 0.15 M solution of ferrioxalate was prepared by dissolving 0.737 g of potassium ferrioxalate hydrate in 10 mL of 0.05 M H₂SO₄. A buffered solution of phenanthroline was prepared by dissolving 5.0 mg of phenanthroline and 1.13 g of sodium acetate in 5.0 mL of 0.5 M H₂SO₄. Both solutions were stored in the dark. To determine the photon flux of the spectrophotometer, 2.0 mL of the ferrioxalate solution was placed in a cuvette and irradiated for 90.0 seconds at $\lambda = 455$ nm. After irradiation, 0.35 mL of the phenanthroline solution was added to the cuvette. The solution was then allowed to rest for 1 h to allow the ferrous ions to completely coordinate to the phenanthroline. The absorbance of the solution was measured at 510 nm. A non-irradiated sample was also prepared and the absorbance at 510 nm measured (Fig S2). Conversion was calculated using **Eq 1**,

$$\text{mol of Fe}^{2+} = \frac{V \cdot \Delta A}{l \cdot \epsilon} \quad (1)$$

$$\text{mol of Fe}^{2+} = \frac{(0.00235 \text{ L})(0.676)}{1 \text{ cm} \cdot 11100 \frac{\text{L}}{\text{mol cm}^{-1}}} = 1.431 \cdot 10^{-7} \text{ mol}$$

where **V** is the total volume (0.00235 L) of the solution after addition of phenanthroline, **ΔA** is the difference in absorbance at 510 nm between the irradiated and non-irradiated solutions, **l** is the path length (1.000 cm), and **ε** is the molar absorptivity at 510 nm (11,100 L mol⁻¹ cm⁻¹). The photon flux can be calculated using **Eq 2**,

$$\text{Photon flux} = \frac{\text{mol of Fe}^{2+}}{\Phi \cdot t \cdot f} \quad (2)$$

where **Φ** is the quantum yield for the ferrioxalate actinometer (0.84 at $\lambda = 455$ nm)^[2], **t** is the time (90 s), and **f** is the fraction of light absorbed calculated using **Eq 3**, where **A** is the measured absorbance at 456 nm

$$f = 1 - 10^{-A} = 0.967 \quad (3)$$

$$\text{Photon flux} = \frac{1.431 \cdot 10^{-7} \text{ mol}}{0.84 \cdot 90 \text{ s} \cdot 0.967} = 1.957 \cdot 10^{-9} \text{ einstein/s}$$

Reaction quantum yield

A dry and argon-flushed Schlenk-flask, equipped with a magnetic stirring bar, was charged with 1-(3-phenylpropoxy)pyridin-1-ium 4-methylbenzenesulfonate (1 eq) and *fac*-Ir(ppy)₃ (0.02 eq). Dry acetonitrile (0.1M) and trimethylphosphite (3 eq.) were added and the reaction mixture was stirred at room temperature for 80 min under blue LED irradiation ($\lambda = 455$ nm). The solvent was removed in vacuo and the yield of formed product was determined by ¹H NMR based on tetrachloroethan as internal standard to be 71% (0.071 mmol).

The quantum yield was determined using **Eq 4**.

$$\Phi = \frac{\text{mol product}}{\text{flux} \cdot t \cdot f} \quad (4)$$

$$\Phi = \frac{0.071 \cdot 10^{-3}}{1.957 \cdot 10^{-9} \cdot 4800 \cdot 0.939} = 8.05$$

f is the fraction of light absorbed by the catalyst calculated using **Eq 3**, where **A** is the measured absorbance at 455 nm indicating that the fraction of absorbed light by the photocatalyst **f**=0.939 (Fig S3).

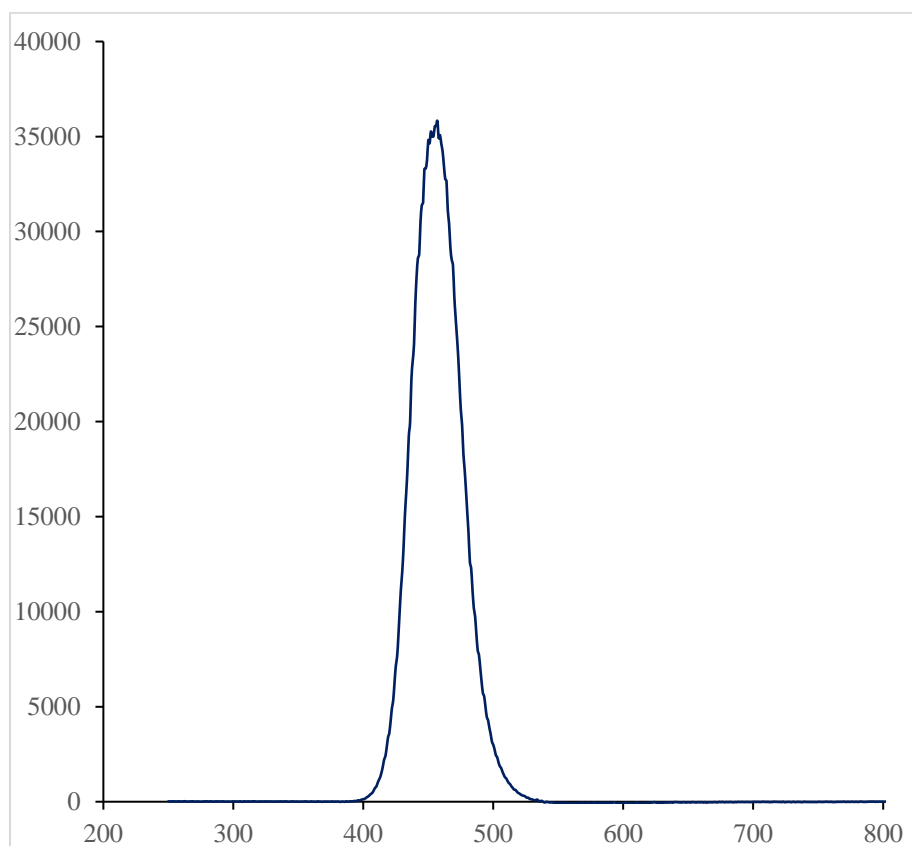


Figure S1. Emission spectrum of the blue LED lamp used in the screening experiments.

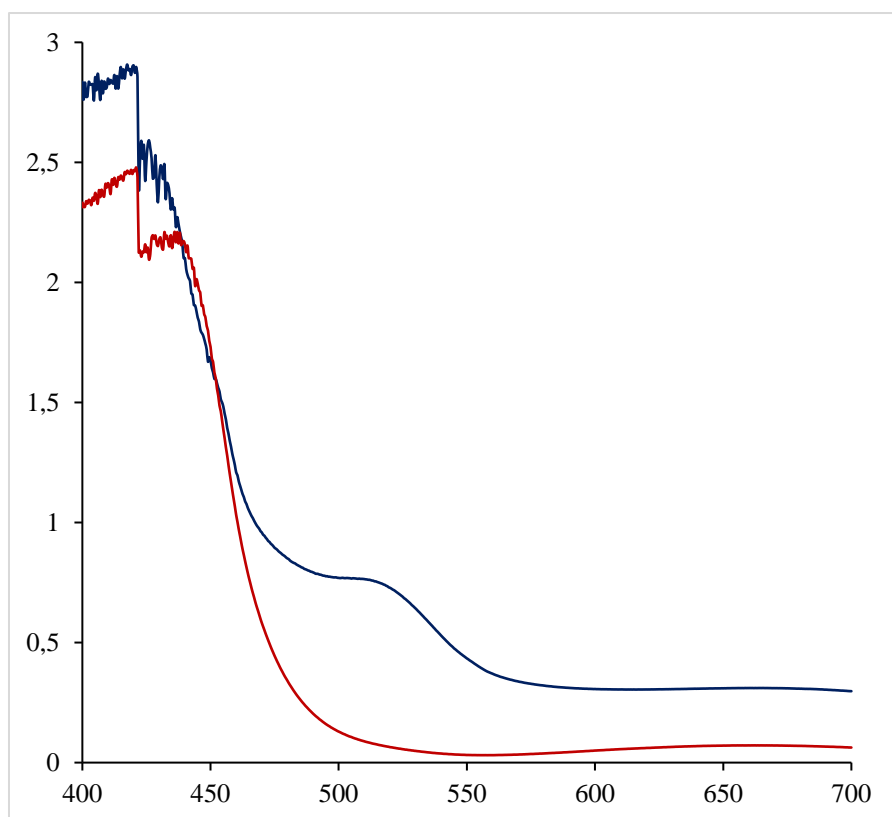


Figure S2. Absorption spectra of irradiation and non-irradiation experiments.

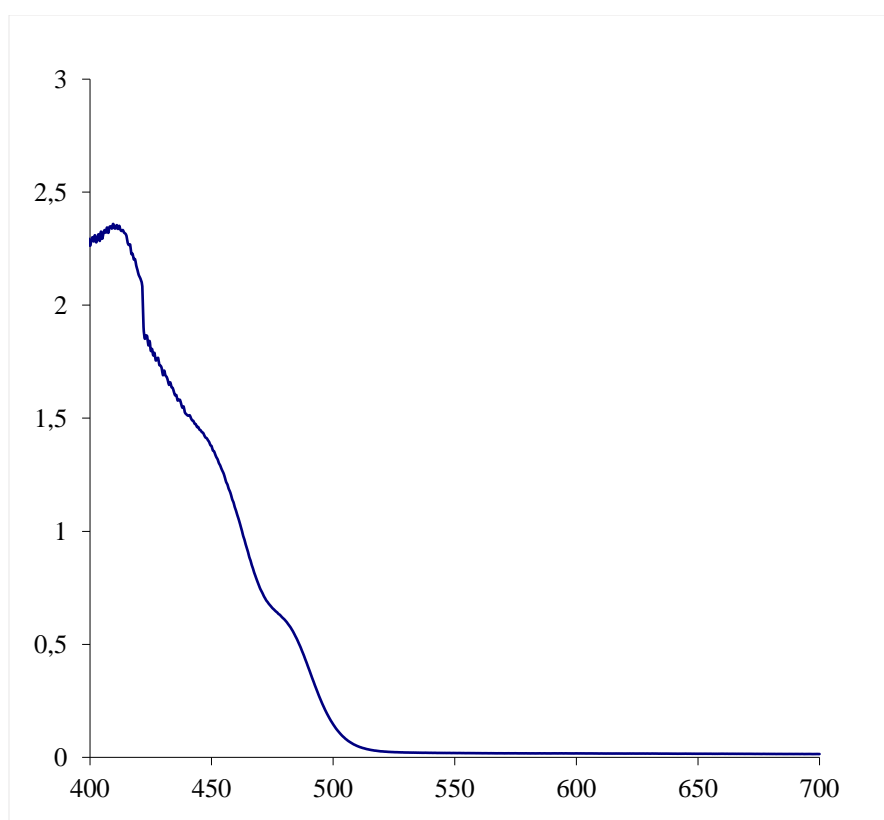
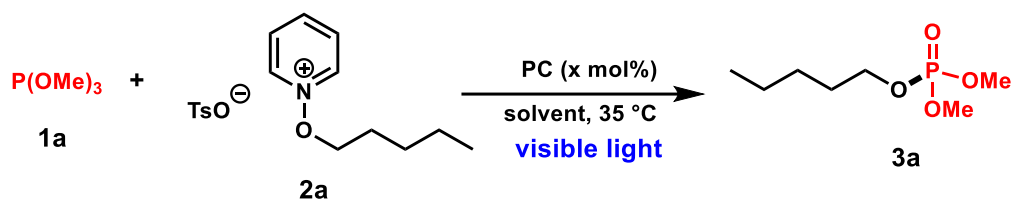


Figure S3. Absorption spectrum of *fac*-Ir(ppy)₃ [0.001 M] in acetonitrile.

3. Optimization of the Reaction Conditions

Table S1



entry ^a	Photocatalyst (PC)	catalyst loading (%)	solvent	3a , yield [%] ^b
1	Ru(bpy) ₃	5	MeCN	65
2	Eosin Y	5	MeCN	67
3	Rose Bengal	5	MeCN	63
4	<i>fac</i> -Ir(ppy) ₃	5	MeCN	82
5	—	—	MeCN	traces
6	<i>fac</i> -Ir(ppy) ₃ ^c	5	MeCN	72
7	<i>fac</i> -Ir(ppy) ₃ ^d	5	MeCN	80
8	<i>fac</i> -Ir(ppy) ₃	2	MeCN	81
9	<i>fac</i> -Ir(ppy) ₃	2	EtOAc	78
10	<i>fac</i> -Ir(ppy) ₃	2	CH ₂ Cl ₂	77
11	<i>fac</i> -Ir(ppy) ₃	2	DMF	47
12	<i>fac</i> -Ir(ppy) ₃	2	MeOH	75
13	<i>fac</i> -Ir(ppy) ₃	2	Toluene	75
14	<i>fac</i> -Ir(ppy) ₃	2	THF	56

^a Reaction conditions: trimethylphosphite **1a** (0.3 mmol, 3 equiv), *N*-alkoxypyridinium tosylate **2a** (0.1 mmol, 1 equiv), solvent (1 mL), blue LEDs (5W), 15 h. ^b NMR yields are determined from ³¹P NMR spectroscopy using tributyl phosphate as internal standard. ^c trimethylphosphite **1a** (0.1 mmol, 1 equiv), *N*-alkoxypyridinium tosylate **2a** (0.1 mmol, 1 equiv). ^d trimethylphosphite **1a** (0.1 mmol, 1 equiv), *N*-alkoxypyridinium tosylate **2a** (0.3 mmol, 3 equiv).

4. NMR Investigations

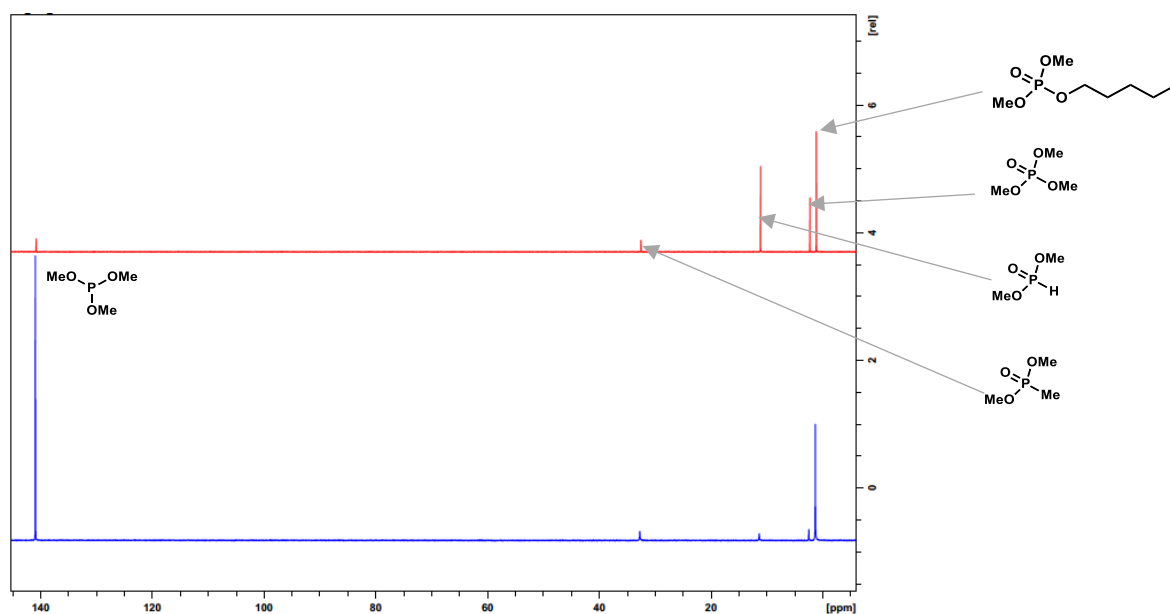


Figure S4. ^{31}P NMR spectra of the reaction of the pyridinium salt **1a** with $\text{P}(\text{OMe})_3$ under optimized reaction conditions. In blue: with distilled $\text{P}(\text{OMe})_3$; in red: with standard $\text{P}(\text{OMe})_3$.



Figure S5. ^{31}P NMR spectrum of the crude of the optimized reaction using $\text{OP}(\text{OnBu})_3$ as internal standard

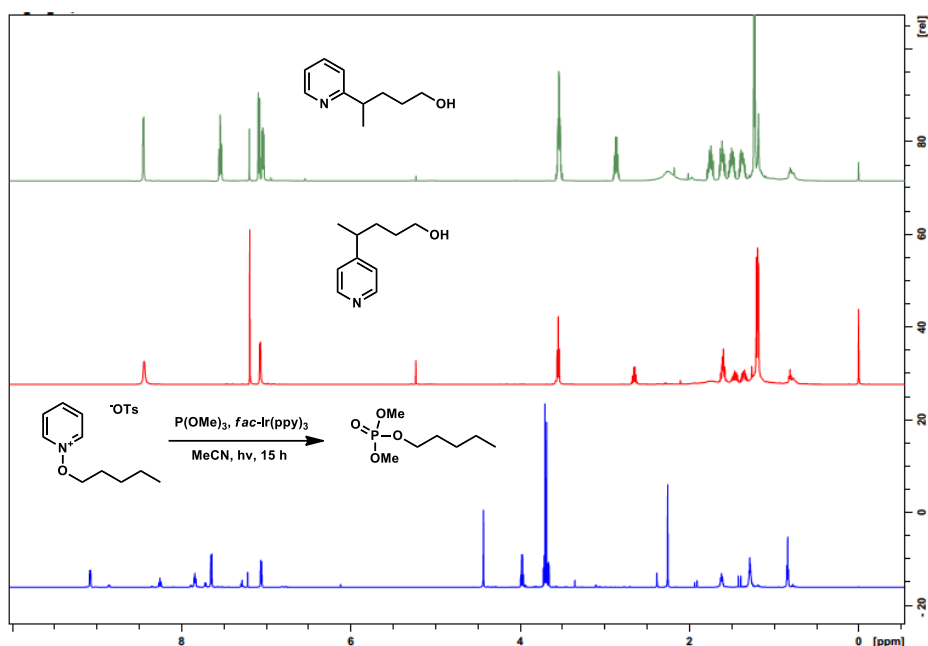


Figure S6. ^1H NMR experiments, showing the absence of the Minisci adduct.

5. Synthesis of Starting Materials and Products

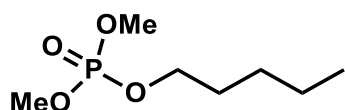
5.1 General procedure for the synthesis of pyridinium salts

All pyridinium salts were synthesized according to literature procedures reported by Hong.^[3]

5.2 General procedure for the synthesis of phosphates

A dry and argon-flushed Schlenk-flask, equipped with a magnetic stirring bar, was charged with appropriate pyridinium salt (1 equiv.) and *fac*-Ir(ppy)₃ (2 mol%). Dry acetonitrile (4 mL) and phosphite (3 equiv.) were added and the reaction mixture was irradiated for 15 h with a GU10 5W LED lamp (5 W; $\lambda = 455$ nm ; 5 cm away). After that, the crude mixture was evaporated under reduced pressure. Dichloromethane (10 mL) was added and the mixture was washed with 1N HCl (4 mL) and subsequently with brine (10 mL). The organic layers were dried over Na₂SO₄, concentrated under reduced pressure and purified by flash column chromatography (ethyl acetate/*n*-pentane) to afford the title compound in the stated yield.

Dimethyl pentyl phosphate (3a)



Prepared according to General Procedure (GP) using 1-(pentyloxy)pyridin-1-ium 4-methylbenzenesulfonate **2a** (135.0

mg, 0.4 mmol) and trimethylphosphite **1a** (141.8 μ L, 1.2 mmol), purified by flash chromatography on silica gel (ethyl acetate/*n*-pentane : 3/7) to afford **3a** (51.2 mg, 66 % yield) as a yellow oil.

¹H NMR (500 MHz, CDCl₃): δ 4.04 (quad, 2H, *J* = 6.8 Hz), 3.75 (d, 6H, *J* = 11.0 Hz), 1.64-1.72 (m, 2H), 1.32-1.38 (m, 4H), 0.90 (t, 3H, *J* = 6.9 Hz).

¹³C NMR (125.7 MHz, CDCl₃): δ 68.0 (d, *J* = 6.0 Hz), 54.2 (d, *J* = 6.0 Hz), 29.9 (d, *J* = 6.6 Hz), 27.5, 22.2, 14.0.

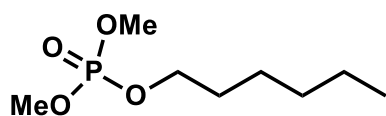
³¹P NMR (202.5 MHz, CDCl₃): δ 1.41.

HRMS (ESI, positive) = [C₇H₁₇O₄NaP⁺] Calculated mass: 219.0762 g/mol, found mass: 219.0764 g/mol.

Scale up of the reaction to 5 mmol starting material

A 250 mL dry and argon-flushed Schlenk-flask, equipped with a magnetic stirring bar, was charged with 1-(pentyloxy)pyridin-1-ium 4-methylbenzenesulfonate **2a** (1.69 g, 5 mmol) and *fac*-Ir(ppy)₃ (2 mol%). Dry acetonitrile (50 mL) and trimethylphosphite **1a** (1.77 mL, 15 mmol) were added and the reaction mixture was irradiated for 15 h with a GU10 5W LED lamp (5 W; λ = 455 nm ; 5 cm away). After that, the crude mixture was evaporated under reduced pressure. Then dichloromethane (125 mL) was added and the mixture was washed with 1N HCl (50 mL) and then with Brine (125 mL). The organic layers were dried over Na₂SO₄, concentrated under reduced pressure and purified by flash chromatography on silica gel (ethyl acetate/*n*-pentane : 3/7) to afford **3a** (667.1 mg, 68 % yield) as a yellow oil. ¹H-NMR data are in good agreement with the those previously reported.

Dimethyl hexyl phosphate (3b)



Prepared according to General Procedure (GP) using 1-(hexyloxy)pyridin-1-ium 4-methylbenzenesulfonate **2b** (140.6 mg, 0.4 mmol) and trimethylphosphite **1a** (141.8 μ L, 1.2

mmol), purified by flash chromatography on silica gel (ethyl acetate/*n*-pentane : 3/7) to afford **3b** (64.2 mg, 76 % yield) as a yellow oil.

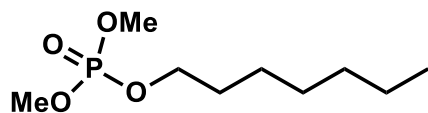
¹H NMR (500 MHz, CDCl₃): δ 4.03 (quad, 2H, *J* = 6.8 Hz), 3.75 (d, 6H, *J* = 11.1 Hz), 1.66 (quint, 2H, *J* = 7.0 Hz), 1.22-1.40 (m, 6H), 0.87 (t, 3H, *J* = 6.7 Hz).

¹³C NMR (125.7 MHz, CDCl₃): δ 68.0 (d, *J* = 6.1 Hz), 54.2 (d, *J* = 6.1 Hz), 31.3, 30.2 (d, *J* = 6.7 Hz), 25.1, 22.5, 14.0.

³¹P NMR (202.5 MHz, CDCl₃): δ 1.41.

HRMS (ESI) m/z: [M + Na]⁺ Calcd for C₈H₁₉O₄NaP 233.0919; Found 233.0915.

Dimethyl heptyl phosphate (3c)



Prepared according to General Procedure (GP) using 1-(heptyloxy)pyridin-1-ium 4-methylbenzenesulfonate **2c** (146.2 mg, 0.4 mmol) and trimethylphosphite **1a** (141.8

μL, 1.2 mmol), purified by flash chromatography on silica gel (ethyl acetate/*n*-pentane : 3/7) to afford **3c** (70.6 mg, 79 % yield) as a yellow oil.

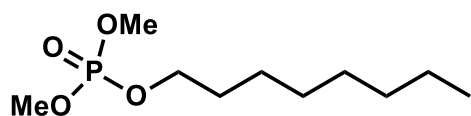
¹H NMR (500 MHz, CDCl₃): δ 4.04 (quad, 2H, *J* = 6.8 Hz), 3.76 (d, 6H, *J* = 11.0 Hz), 1.67 (quint, 2H, *J* = 7.0 Hz), 1.22-1.40 (m, 8H), 0.87 (t, 3H, *J* = 6.7 Hz).

¹³C NMR (125.7 MHz, CDCl₃): δ 68.0 (d, *J* = 6.2 Hz), 54.2 (d, *J* = 6.2 Hz), 31.7, 30.3 (d, *J* = 6.6 Hz), 28.8, 25.4, 22.6, 14.1.

³¹P NMR (202.5 MHz, CDCl₃): δ 1.38.

HRMS (ESI) m/z: [M + Na]⁺ Calcd for C₉H₂₁O₄NaP 247.1075; Found 247.1077.

Dimethyl octyl phosphate (3d)



Prepared according to General Procedure (GP) using 1-(octyloxy)pyridin-1-ium 4-methylbenzenesulfonate **2d** (151.8 mg, 0.4 mmol) and trimethylphosphite **1a**

(141.8 μL, 1.2 mmol), purified by flash chromatography on silica gel (ethyl acetate/*n*-pentane : 3/7) to afford **3d** (65.1 mg, 68 % yield) as a yellow oil.

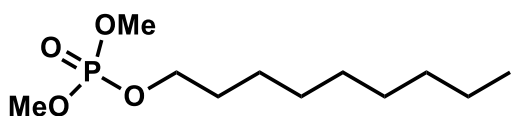
¹H NMR (500 MHz, CDCl₃): δ 4.03 (quad, 2H, *J* = 6.8 Hz), 3.76 (d, 6H, *J* = 11.0 Hz), 1.66 (quint, 2H, *J* = 7.1 Hz), 1.19-1.39 (m, 10H), 0.86 (t, 3H, *J* = 6.8 Hz).

¹³C NMR (125.7 MHz, CDCl₃): δ 68.0 (d, *J* = 6.0 Hz), 54.2 (d, *J* = 5.7 Hz), 31.8, 30.3 (d, *J* = 6.4 Hz), 29.2, 29.1, 25.4, 22.6, 14.1.

³¹P NMR (202.5 MHz, CDCl₃): δ 1.37.

HRMS (ESI) m/z: [M + Na]⁺ Calcd for C₁₀H₂₃O₄NaP 261.1232; Found 261.1236.

Dimethyl nonyl phosphate (3e)



Prepared according to General Procedure (GP) using 1-(nonyloxy)pyridin-1-ium 4-methylbenzenesulfonate **2e** (157.4 mg, 0.4 mmol)

and trimethylphosphite **1a** (141.8 μ L, 1.2 mmol), purified by flash chromatography on silica gel (ethyl acetate/*n*-pentane : 3/7) to afford **3e** (70.8 mg, 70 % yield) as a yellow oil.

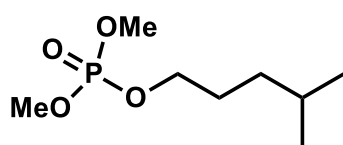
^1H NMR (500 MHz, CDCl_3): δ 4.03 (quad, 2H, J = 6.8 Hz), 3.76 (d, 6H, J = 11.1 Hz), 1.67 (quint, 2H, J = 7.0 Hz), 1.20-1.40 (m, 12H), 0.87 (t, 3H, J = 6.7 Hz).

^{13}C NMR (125.7 MHz, CDCl_3): δ 68.0 (d, J = 5.6 Hz), 54.2 (d, J = 6.3 Hz), 31.8, 30.3 (d, J = 6.9 Hz), 29.5, 29.2, 29.1, 25.4, 22.6, 14.1.

^{31}P NMR (202.5 MHz, CDCl_3): δ 1.38.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{11}\text{H}_{25}\text{O}_4\text{NaP}$ 275.1388; Found 275.1390.

Dimethyl hexyl phosphate (**3f**)



Prepared according to General Procedure (GP) using 1-((4-methylpentyl)oxy)pyridin-1-ium **2f** (140.6 mg, 0.4 mmol) and trimethylphosphite **1a** (141.8 μ L, 1.2 mmol), purified by flash

chromatography on silica gel (ethyl acetate/pentane : 3/7) to afford **3f** (56.0 mg, 67 % yield) as a yellow oil.

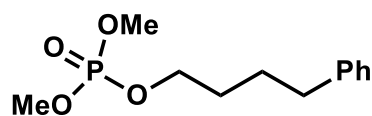
^1H NMR (500 MHz, CDCl_3): δ 4.02 (quad, 2H, J = 6.8 Hz), 3.75 (d, 6H, J = 11.0 Hz), 1.63-1.71 (m, 2H), 1.55 (non, 1H, J = 6.7 Hz), 1.21-1.28 (m, 2H), 0.88 (t, 6H, J = 6.6 Hz).

^{13}C NMR (125.7 MHz, CDCl_3): δ 68.3 (d, J = 6.2 Hz), 54.2 (d, J = 6.2 Hz), 34.5, 28.2 (d, J = 6.7 Hz), 27.7, 22.5.

^{31}P NMR (202.5 MHz, CDCl_3): δ 1.36.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_8\text{H}_{19}\text{O}_4\text{NaP}$ 233.0919; Found 233.0922.

Dimethyl (4-phenylbutyl) phosphate (**3g**)



Prepared according to General Procedure (GP) using 1-(4-phenylbutoxy)pyridin-1-ium 4-methylbenzenesulfonate **2g** (159.8 mg, 0.4 mmol) and trimethylphosphite **1a** (141.8 μ L,

1.2 mmol), purified by flash chromatography on silica gel (ethyl acetate/pentane : 5/5) to afford **3g** (66.4 mg, 64 % yield) as a yellow oil.

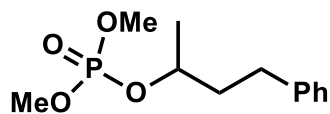
^1H NMR (500 MHz, CDCl_3): δ 7.25-7.31 (m, 2H), 7.15-7.21 (m, 3H), 4.04-4.10 (m, 2H), 3.76 (d, 6H, J = 11.0 Hz), 2.62-2.68 (m, 2H), 1.69-1.76 (m, 4H).

^{13}C NMR (125.7 MHz, CDCl_3): δ 141.9, 128.4, 128.3, 125.9, 67.7 (d, J = 5.7 Hz), 54.3 (d, J = 6.0 Hz), 35.3, 29.8 (d, J = 6.8 Hz), 27.2.

^{31}P NMR (202.5 MHz, CDCl_3): δ 1.39.

HRMS (ESI) m/z : $[M + Na]^+$ Calcd for $C_{12}H_{19}O_4NaP$ 281.0919; Found 281.0919.

Dimethyl (4-phenylbutan-2-yl) phosphate (3h)



Prepared according to General Procedure (GP) using 1-((4-phenylbutan-2-yl)oxy)pyridin-1-ium 4-methylbenzenesulfonate **2h** (159.8 mg, 0.4 mmol) and trimethylphosphite **1a** (141.8 μ L,

1.2 mmol), purified by flash chromatography on silica gel (ethyl acetate/*n*-pentane : 5/5) to afford **3h** (68.2 mg, 66 % yield) as a yellow oil.

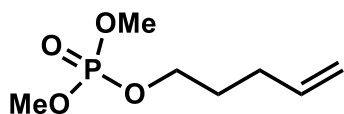
1H NMR (500 MHz, $CDCl_3$): δ 7.25-7.31 (m, 2H), 7.15-7.22 (m, 3H), 4.54 (sept, 1H, $J = 6.3$ Hz), 3.76 (dd, 6H, $J = 11.2, 3.6$ Hz), 2.72-2.80 (m, 1H), 2.63-2.71 (m, 1H), 1.94-2.03 (m, 1H), 1.79-1.89 (m, 1H), 1.38 (d, 3H, $J = 6.2$ Hz).

^{13}C NMR (125.7 MHz, $CDCl_3$): δ 141.4, 128.5, 128.4, 126.0, 75.8 (d, $J = 6.0$ Hz), 54.2 (d, $J = 6.4$ Hz), 54.1 (d, $J = 6.7$ Hz), 39.2 (d, $J = 6.4$ Hz), 31.5, 21.6 (d, $J = 2.3$ Hz).

^{31}P NMR (202.5 MHz, $CDCl_3$): δ 0.63.

HRMS (ESI) m/z : $[M + Na]^+$ Calcd for $C_{12}H_{19}O_4NaP$ 281.0919; Found 281.0918.

Dimethyl pent-4-en-1-yl phosphate (3i)



Prepared according to General Procedure (GP) using 1-(pent-4-en-1-yloxy)pyridin-1-ium 4-methylbenzenesulfonate **2i** (134.2 mg, 0.4 mmol) and trimethylphosphite **1a** (472.4 μ L, 4 mmol),

purified by flash chromatography on silica gel (ethyl acetate/ *n*-pentane : 5/5) to afford **3i** (51.3 mg, 66 % yield) as a yellow oil.

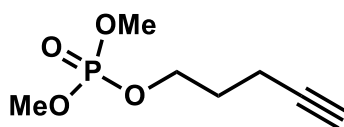
1H NMR (500 MHz, $CDCl_3$): δ 5.78 (ddt, 1H, $J = 17.0, 10.2, 6.7$ Hz), 5.04 (d, 1H, $J = 16.9$ Hz), 4.99 (d, 1H, $J = 10.1$ Hz), 4.15 (quad, 2H, $J = 6.7$ Hz), 3.75 (d, 6H, $J = 11.1$ Hz), 2.15 (quad, 2H, $J = 7.1$ Hz), 1.78 (quint, 2H, $J = 7.0$ Hz).

^{13}C NMR (125.7 MHz, $CDCl_3$): δ 137.2, 115.6, 67.2 (d, $J = 5.9$ Hz), 54.2 (d, $J = 5.9$ Hz), 29.5, 29.4 (d, $J = 6.9$ Hz).

^{31}P NMR (202.5 MHz, $CDCl_3$): δ 1.36.

HRMS (ESI) m/z : $[M + Na]^+$ Calcd for $C_7H_{15}O_4NaP$ 217.0606; Found 217.0603.

Dimethyl pent-4-yn-1-yl phosphate (3j)



Prepared according to General Procedure (GP) using 1-(pent-4-yn-1-yloxy)pyridin-1-ium 4-methylbenzenesulfonate **2j** (133.4 mg, 0.4 mmol) and trimethylphosphite **1a** (141.8 μ L, 1.2 mmol),

purified by flash chromatography on silica gel (ethyl acetate/*n*-pentane : 7/3) to afford **3j** (62.0 mg, 81 % yield) as a yellow oil.

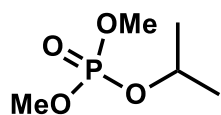
¹H NMR (500 MHz, CDCl₃): δ 4.15 (quad, 2H, *J* = 6.3 Hz), 3.76 (d, 6H, *J* = 11.1 Hz), 2.32 (dt, 2H, *J* = 6.9, 2.3 Hz), 1.95-1.98 (m, 1H), 1.89 (quint, 2H, *J* = 6.5 Hz).

¹³C NMR (125.7 MHz, CDCl₃): δ 82.7, 69.2, 66.2 (d, *J* = 5.6 Hz), 54.3 (d, *J* = 5.6 Hz), 29.0 (d, *J* = 7.4 Hz), 14.7.

³¹P NMR (202.5 MHz, CDCl₃): δ 1.27.

HRMS (ESI) *m/z*: [M + H]⁺ Calcd for C₇H₁₄O₄P 193.0630; Found 193.0631.

Isopropyl dimethyl phosphate (**3k**)



Prepared according to General Procedure (GP) using 1-

(isopropoxy)pyridin-1-ium 4-methylbenzenesulfonate **2k** (123.8 mg, 0.4 mmol) and trimethylphosphite **1a** (141.8 μL, 1.2 mmol), purified by flash

chromatography on silica gel (ethyl acetate/ *n*-pentane : 5/5) to afford **3k** (39.7 mg, 59 % yield) as a yellow oil.

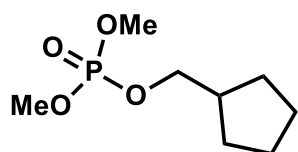
¹H NMR (500 MHz, CDCl₃): δ 4.64 (oct, 1H, *J* = 6.3 Hz), 3.73 (d, 6H, *J* = 11.0 Hz), 1.32 (d, 6H, *J* = 6.3 Hz).

¹³C NMR (125.7 MHz, CDCl₃): δ 72.8 (d, *J* = 6.0 Hz), 54.1 (d, *J* = 6.1 Hz), 23.6 (d, *J* = 5.0 Hz).

³¹P NMR (202.5 MHz, CDCl₃): δ 0.39.

HRMS (ESI) *m/z*: [M + Na]⁺ Calcd for C₅H₁₃O₄NaP 191.0449; Found 191.0452.

Cyclopentylmethyl dimethyl phosphate (**3l**)



Prepared according to General Procedure (GP) using 1-

(cyclopentylmethoxy)pyridin-1-ium 4-methylbenzenesulfonate **2l** (139.8 mg, 0.4 mmol) and trimethylphosphite **1a** (141.8 μL, 1.2

mmol), purified by flash chromatography on silica gel (ethyl acetate/ *n*-pentane : 3/7) to afford **3l** (70.2 mg, 84 % yield) as a yellow oil.

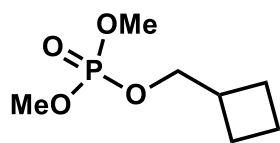
¹H NMR (500 MHz, CDCl₃): δ 3.91 (t, 2H, *J* = 6.8 Hz), 3.75 (d, 6H, *J* = 11.0 Hz), 2.24 (sept, 1H, *J* = 6.8 Hz), 1.69-1.80 (m, 2H), 1.48-1.64 (m, 4H), 1.20-1.33 (m, 2H).

¹³C NMR (125.7 MHz, CDCl₃): δ 71.7 (d, *J* = 6.1 Hz), 54.2 (d, *J* = 6.1 Hz), 39.8 (d, *J* = 6.7 Hz), 28.9, 25.4.

³¹P NMR (202.5 MHz, CDCl₃): δ 1.28.

HRMS (ESI) m/z : $[M + Na]^+$ Calcd for $C_8H_{17}O_4NaP$ 231.0762; Found 231.0764.

Cyclobutylmethyl dimethyl phosphate (**3m**)



Prepared according to General Procedure (GP) using 1-(cyclobutylmethoxy)pyridin-1-ium 4-methylbenzenesulfonate **2m** (134.2 mg, 0.4 mmol) and trimethylphosphite **1a** (141.8 μ L, 1.2

mmol), purified by flash chromatography on silica gel (ethyl acetate/ *n*-pentane : 3/7) to afford **3m** (55.4 mg, 71 % yield) as a yellow oil.

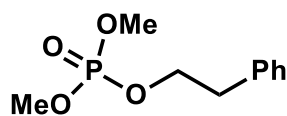
1H NMR (500 MHz, $CDCl_3$): δ 3.98 (t, 2H, $J = 6.8$ Hz), 3.74 (d, 6H, $J = 11.1$ Hz), 2.63 (sept, 1H, $J = 6.8$ Hz), 1.99-2.09 (m, 2H), 1.72-1.96 (m, 4H).

^{13}C NMR (125.7 MHz, $CDCl_3$): δ 71.5 (d, $J = 6.2$ Hz), 54.2 (d, $J = 6.0$ Hz), 35.2 (d, $J = 6.9$ Hz), 24.2, 18.2.

^{31}P NMR (202.5 MHz, $CDCl_3$): δ 1.47.

HRMS (ESI) m/z : $[M + Na]^+$ Calcd for $C_8H_{17}O_4NaP$ 217.0606; Found 217.0606.

Dimethyl (3-phenylethyl) phosphate (**3n**)



Prepared according to General Procedure (GP) using 1-(2-phenylethoxy)pyridin-1-ium 4-methylbenzenesulfonate **2n** (148.6 mg, 0.4 mmol) and trimethylphosphite **1a** (141.8 μ L, 1.2 mmol),

purified by flash chromatography on silica gel (ethyl acetate/ *n*-pentane : 7/3) to afford **3n** (58.4 mg, 63 % yield) as a yellow oil.

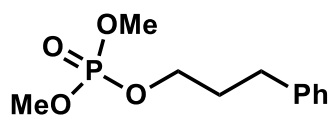
1H NMR (500 MHz, $CDCl_3$): δ 7.28-7.33 (m, 2H), 7.21-7.26 (m, 3H), 4.25 (quad, 2H, $J = 7.1$ Hz), 3.68 (d, 6H, $J = 11.1$ Hz), 2.73 (t, 2H, $J = 7.0$ Hz).

^{13}C NMR (125.7 MHz, $CDCl_3$): δ 137.1, 129.1, 128.6, 68.2 (d, $J = 5.9$ Hz), 54.2 (d, $J = 5.9$ Hz), 36.7 (d, $J = 6.6$ Hz).

^{31}P NMR (202.5 MHz, $CDCl_3$): δ 1.08.

HRMS (ESI) m/z : $[M + Na]^+$ Calcd for $C_{10}H_{15}O_4NaP$ 253.0606; Found 253.0609.

Dimethyl (3-phenylpropyl) phosphate (**3o**)



Prepared according to General Procedure (GP) using 1-(3-phenylpropoxy)pyridin-1-ium 4-methylbenzenesulfonate **2o** (154.2 mg, 0.4 mmol) and trimethylphosphite **1a** (141.8 μ L, 1.2

mmol), purified by flash chromatography on silica gel (ethyl acetate/ *n*-pentane : 4/6) to afford **3o** (80.9 mg, 83 % yield) as a yellow oil.

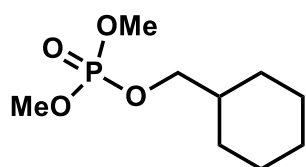
¹H NMR (500 MHz, CDCl₃): δ 7.26-7.31 (m, 2H), 7.18-7.22 (m, 3H), 4.08 (quad, 2H, *J* = 6.7 Hz), 3.78 (d, 6H, *J* = 11.2 Hz), 2.73 (t, 2H, *J* = 7.7 Hz), 2.02 (quint, 2H, *J* = 7.2 Hz).

¹³C NMR (125.7 MHz, CDCl₃): δ 140.9, 128.5, 128.4, 126.1, 67.1 (d, *J* = 5.5 Hz), 54.3 (d, *J* = 6.2 Hz), 31.9 (d, *J* = 6.8 Hz), 31.6.

³¹P NMR (202.5 MHz, CDCl₃): δ 1.42.

HRMS (ESI) *m/z*: [M + Na]⁺ Calcd for C₁₁H₁₇O₄NaP 267.0762; Found 267.0764.

Cyclohexylmethyl dimethyl phosphate (**3p**)



Prepared according to General Procedure (GP) using 1-(cyclohexylmethoxy)pyridin-1-ium 4-methylbenzenesulfonate **2p** (145.4 mg, 0.4 mmol) and trimethylphosphite **1a** (141.8 μL, 1.2

mmol), purified by flash chromatography on silica gel (ethyl acetate/ *n*-pentane : 3/7) to afford **3p** (78.6 mg, 88 % yield) as a yellow oil.

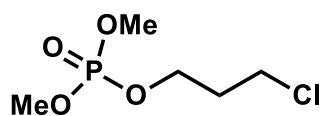
¹H NMR (500 MHz, CDCl₃): δ 3.83 (t, 2H, *J* = 6.4 Hz), 3.75 (d, 6H, *J* = 11.1 Hz), 1.60-1.80 (m, 6H), 1.09-1.29 (m, 3H), 0.90-1.01 (m, 2H).

¹³C NMR (125.7 MHz, CDCl₃): δ 72.9 (d, *J* = 6.2 Hz), 54.2 (d, *J* = 5.9 Hz), 38.3 (d, *J* = 6.9 Hz), 29.2, 26.3, 25.5.

³¹P NMR (202.5 MHz, CDCl₃): δ 1.42.

HRMS (ESI) *m/z*: [M + Na]⁺ Calcd for C₉H₁₉O₄NaP 245.0919; Found 245.0921.

3-chloropropyl dimethyl phosphate (**3q**)



Prepared according to General Procedure (GP) using 1-(3-chloropropoxy)pyridin-1-ium 4-methylbenzenesulfonate **2q** (137.5 mg, 0.4 mmol) and trimethylphosphite **1a** (162.5 μL, 1.2 mmol),

purified by flash chromatography on silica gel (ethyl acetate/pentane : 4/6) to afford **3q** (67.2 mg, 83 % yield) as a yellow oil.

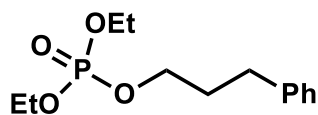
¹H NMR (500 MHz, CDCl₃): δ 4.20 (quad, 2H, *J* = 6.3 Hz), 3.77 (d, 6H, *J* = 11.1 Hz), 3.66 (t, 2H, *J* = 6.2 Hz), 2.12 (quint, 2H, *J* = 6.0 Hz).

¹³C NMR (125.7 MHz, CDCl₃): δ 64.3 (d, *J* = 5.7 Hz), 54.4 (d, *J* = 5.9 Hz), 40.6, 33.0 (d, *J* = 67.2 Hz).

³¹P NMR (202.5 MHz, CDCl₃): δ 1.24.

HRMS (ESI) m/z : $[M + H]^+$ Calcd for $C_5H_{13}O_4ClP$ 203.0240; Found 203.0238.

Diethyl (3-phenylpropyl) phosphate (**3r**)



Prepared according to General Procedure (GP) using 1-(3-phenylpropoxy)pyridin-1-ium 4-methylbenzenesulfonate **2o** (154.2 mg, 0.4 mmol) and triethylphosphite **1b** (205.7 μ L, 1.2 mmol), purified by flash chromatography on silica gel (ethyl acetate/ *n*-pentane : 4/6) to afford **3r** (67.1 mg, 62 % yield) as a yellow oil.

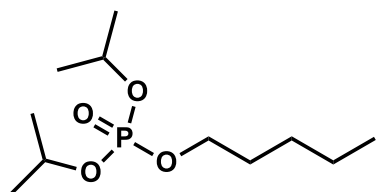
1H NMR (500 MHz, $CDCl_3$): δ 7.27-7.32 (m, 2H), 7.17-7.22 (m, 3H), 4.12 (quint, 4H, $J = 7.2$ Hz), 4.06 (quad, 2H, $J = 6.5$ Hz), 2.73 (t, 2H, $J = 7.7$ Hz), 2.01 (quint, 2H, $J = 6.9$ Hz), 1.34 (t, 6H, $J = 7.0$ Hz).

^{13}C NMR (125.7 MHz, $CDCl_3$): δ 141.0, 128.5, 126.1, 66.8 (d, $J = 5.9$ Hz), 63.7 (d, $J = 5.9$ Hz), 31.9 (d, $J = 7.1$ Hz), 31.7, 16.2 (d, $J = 6.9$ Hz).

^{31}P NMR (202.5 MHz, $CDCl_3$): δ -0.80.

HRMS (ESI) m/z : $[M + Na]^+$ Calcd for $C_{13}H_{21}O_4NaP$ 295.1075; Found 295.1078.

Diisopropyl pentyl phosphate (**3s**)



Prepared according to General Procedure (GP) using 1-(pentyloxy)pyridin-1-ium 4-methylbenzenesulfonate **2a** (135.0 mg, 0.4 mmol) and triisopropylphosphite **1c** (296.1 μ L, 1.2 mmol), purified by flash chromatography on silica gel (ethyl acetate/ *n*-pentane : 3/7) to afford **3s** (57.0 mg, 56 % yield) as a yellow oil.

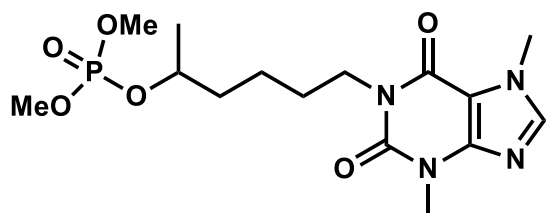
1H NMR (500 MHz, $CDCl_3$): δ 4.61 (oct, 2H, $J = 6.3$ Hz), 3.98 (quad, 2H, $J = 6.7$ Hz), 1.32 (d, 12H, $J = 6.3$ Hz), 1.30-1.35 (m, 2H), 1.25 (t, 2H, $J = 7.1$ Hz), 0.89 (t, 3H, $J = 6.9$ Hz).

^{13}C NMR (125.7 MHz, $CDCl_3$): δ 72.2 (d, $J = 6.1$ Hz), 67.3 (d, $J = 6.4$ Hz), 30.0 (d, $J = 7.1$ Hz), 27.6, 23.7 (d, $J = 4.9$ Hz), 22.2, 14.0.

^{31}P NMR (202.5 MHz, $CDCl_3$): δ -2.48.

HRMS (ESI) m/z : $[M + Na]^+$ Calcd for $C_{11}H_{25}O_4NaP$ 275.1388; Found 275.1388.

5-(3,7-dimethyl-2,6-dioxo-2,3,6,7-tetrahydro-1H-purin-1-yl)hexan-2-yl dimethyl phosphate (3t)



Prepared according to General Procedure (GP) using 1-((5-(3,7-dimethyl-2,6-dioxo-2,3,6,7-tetrahydro-1H-purin-1-yl)hexan-2-yl)oxy)pyridin-1-ium 4-methylbenzenesulfonate

2r (212.0 mg, 0.4 mmol) and trimethylphosphite **1a** (472.4 μ L, 4 mmol), purified by flash chromatography on silica gel (methanol/ethyl acetate : 1/9) to afford **3t** (94.8 mg, 61 % yield) as a yellow oil.

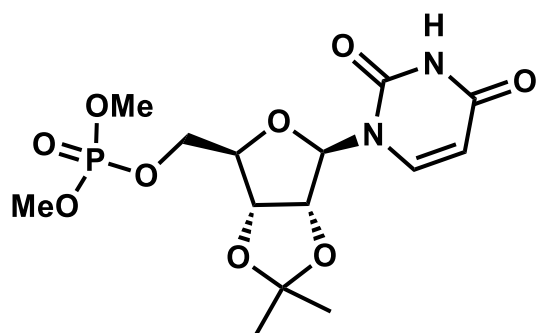
¹H NMR (500 MHz, CDCl₃): δ 7.49 (s, 1H), 4.47 (sept, 1H, J = 6.4 Hz), 3.99 (t, 2H, J = 7.6 Hz), 3.97 (s, 3H), 3.74 (dd, 6H, J = 11.0, 5.1 Hz), 3.56 (s, 3H), 1.54-1.75 (m, 4H), 1.37-1.53 (m, 2H), 1.32 (d, 3H, J = 6.3 Hz).

¹³C NMR (125.7 MHz, CDCl₃): δ 155.3, 151.3, 148.8, 141.4, 107.7, 76.2 (d, J = 6.1 Hz), 54.2 (d, J = 6.3 Hz), 54.1 (d, J = 6.6 Hz), 41.2, 37.1 (d, J = 6.4 Hz), 33.6, 29.7, 27.8, 21.6 (d, J = 2.8 Hz).

³¹P NMR (202.5 MHz, CDCl₃): δ 0.59.

HRMS (ESI) m/z : [M + Na]⁺ Calcd for C₁₂H₁₉O₄NaP 411.1409; Found 411.1414.

(6-(2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)-2,2-dimethyltetrahydrofuro[3,4-d][1,3]dioxol-4-yl)methyl dimethyl phosphate (3u)



Prepared according to General Procedure (GP) using 1-((6-(2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)-2,2-dimethyltetrahydrofuro[3,4-d][1,3]dioxol-4-yl)methoxy)pyridin-1-ium 4-methylbenzenesulfonate **2t** (53.3 mg, 0.1 mmol) and trimethylphosphite **1a** (118.1 μ L, 0.3 mmol),

purified by flash chromatography on silica gel (methanol/ethyl acetate : 1/9) to afford **3u** (30.7 mg, 78 % yield) as a yellow oil.

¹H NMR (500 MHz, CDCl₃): δ 8.77 (br s, 1H), 7.36 (d, 1H, J = 8.1 Hz), 5.72-5.77 (m, 1H), 4.83-4.84 (m, 2H), 4.33-4.36 (m, 1H), 4.22-4.31 (m, 2H), 3.79 (d, 3H, J = 3.8 Hz), 3.77 (d, 3H, J = 3.8 Hz), 1.67 (br s, 1H), 1.57 (s, 3H), 1.35 (s, 3H).

¹³C NMR (125.7 MHz, CDCl₃): δ 162.9, 149.9, 141.8, 114.7, 102.7, 94.0, 85.4 (d, J = 7.4 Hz), 84.4, 80.6, 67.0 (d, J = 5.6 Hz), 54.6 (dd, J = 5.6, 1.8 Hz), 52.3 (d, J = 6.5 Hz), 27.1, 25.3.

³¹P NMR (202.5 MHz, CDCl₃): δ 1.26.

HRMS (ESI) m/z: [M + Na]⁺ Calcd for C₁₄H₂₁N₂O₉NaP 415.0882; Found 415.0884.

References:

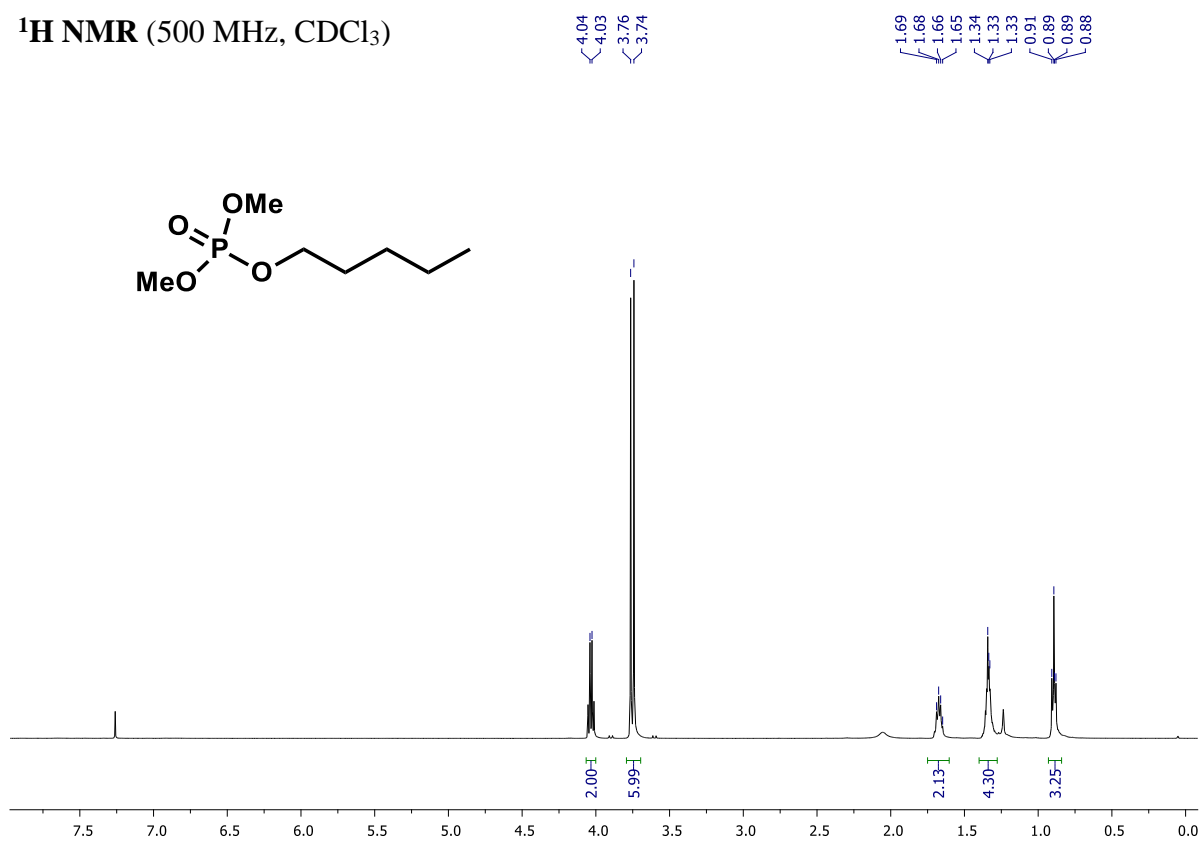
[1] Cismesia, M. A.; Yoon, T. P. *Chem. Sci.*, **2015**, 6, 5426-5434

[2] Walkinshaw, A. J.; Xu, W.; Suero, M. G.; Gaunt, M. J. *J. Am. Chem. Soc.* **2013**, 135, 12532-12535.

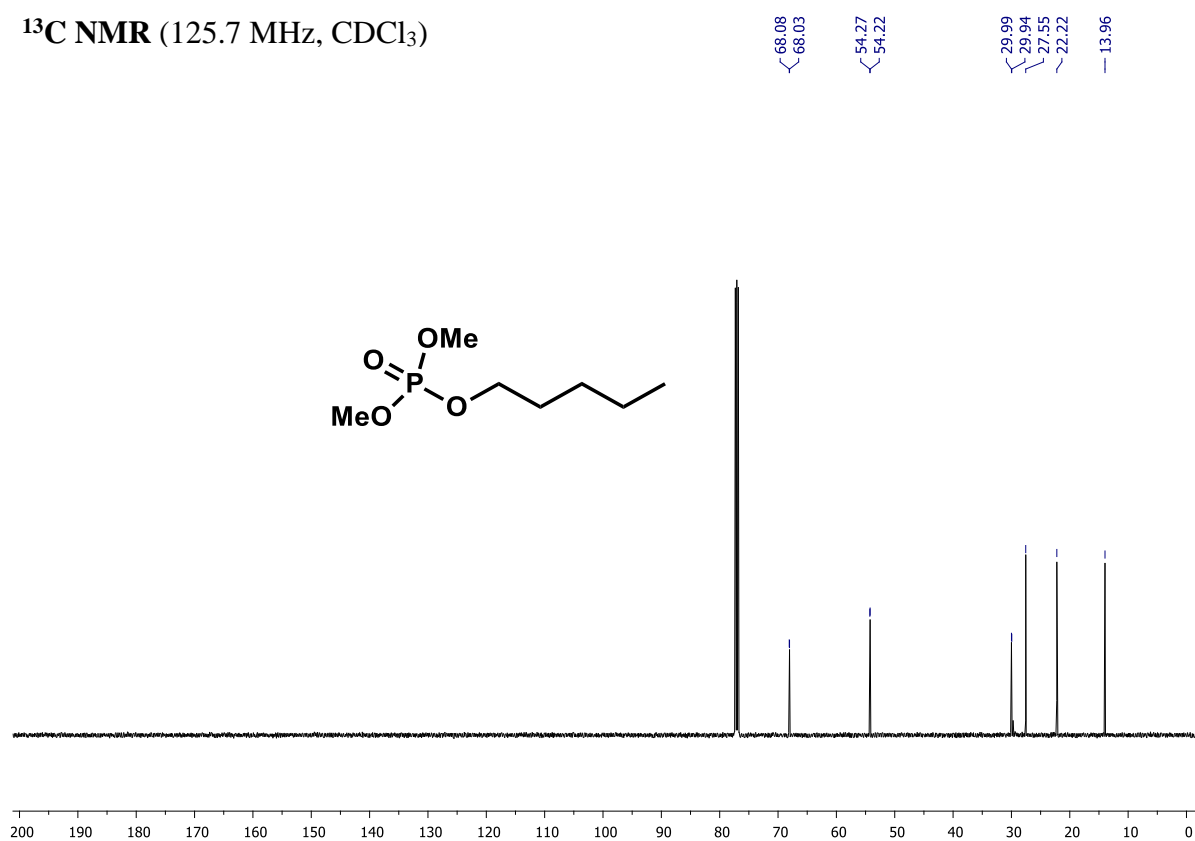
[3] Kim, I.; Park, B.; Kang, G.; Kim, J.; Jung, H.; Lee, H.; Baik, M.-H.; Hong, S. *Angew. Chem. Int. Ed.* **2018**, 57, 15517-15522.

Dimethyl pentyl phosphate (3a)

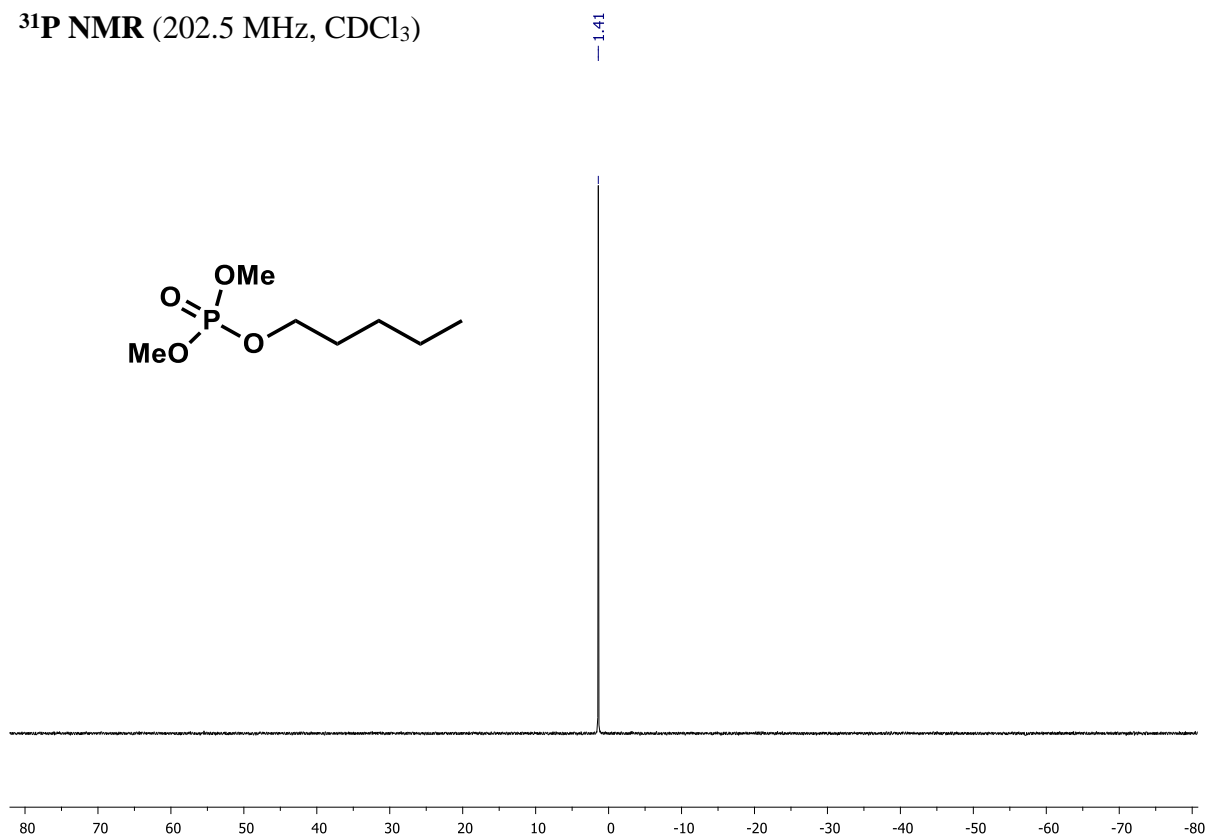
^1H NMR (500 MHz, CDCl_3)



^{13}C NMR (125.7 MHz, CDCl_3)

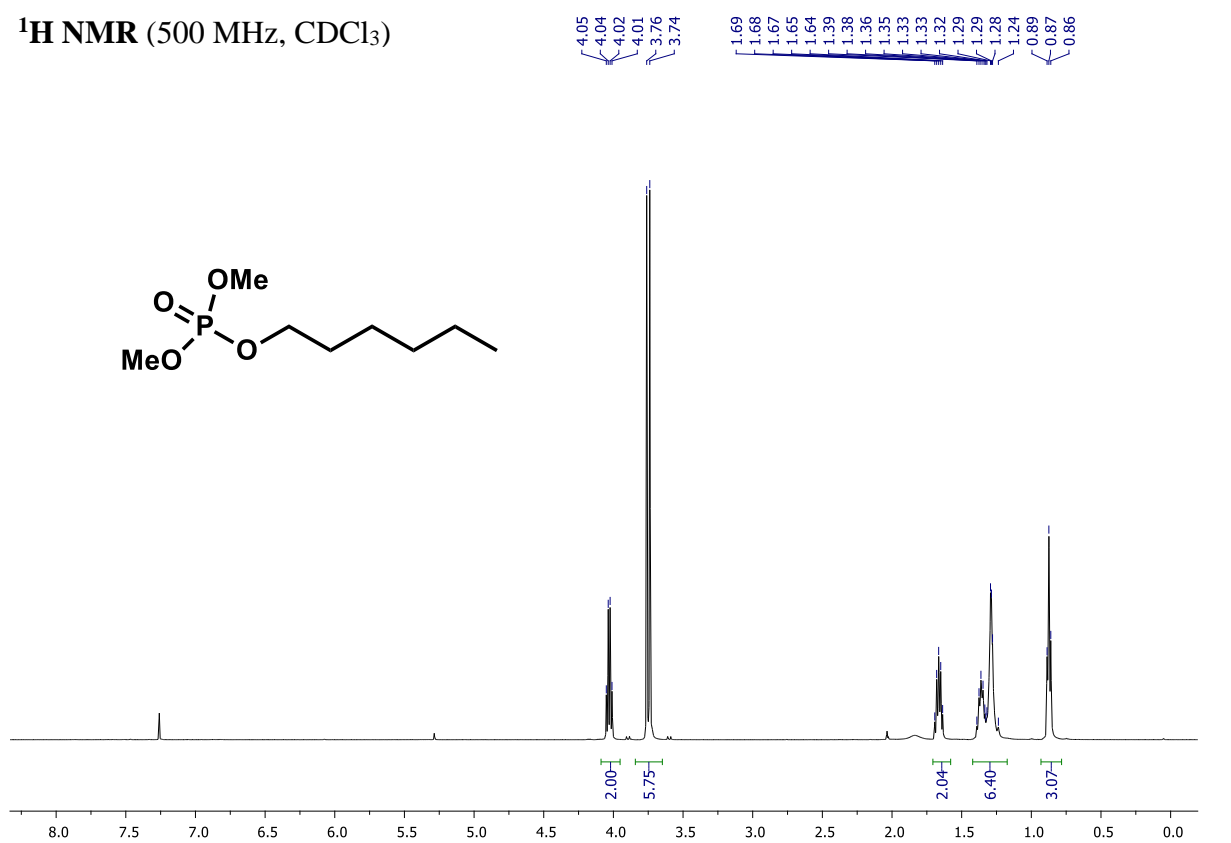


^{31}P NMR (202.5 MHz, CDCl_3)

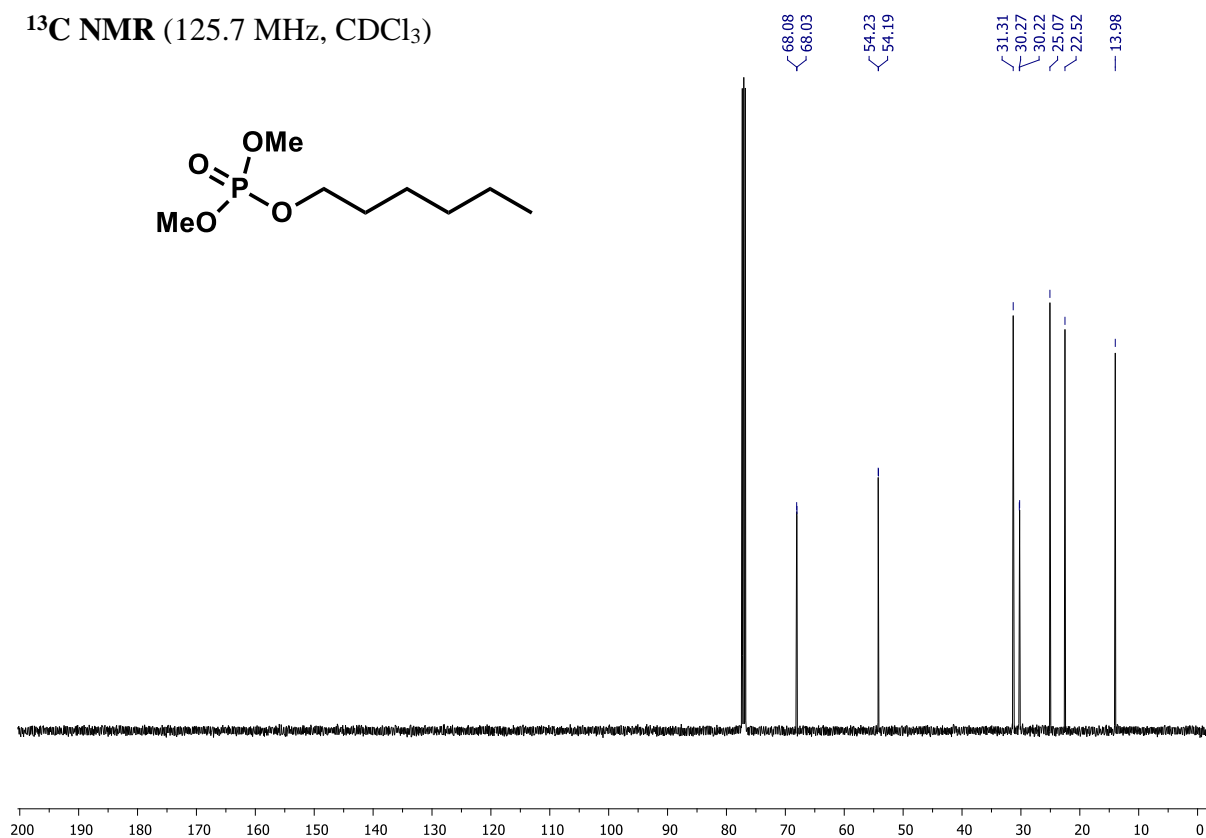


Dimethyl hexyl phosphate (3b)

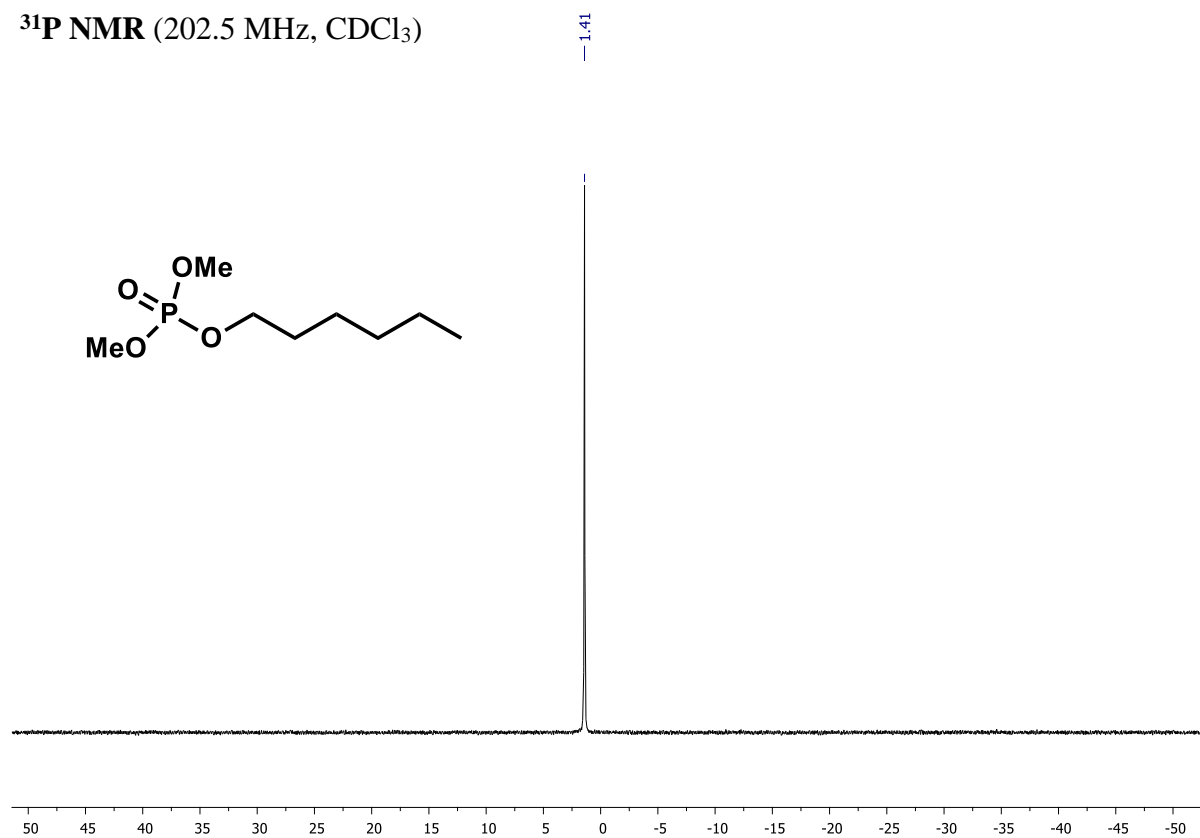
^1H NMR (500 MHz, CDCl_3)



^{13}C NMR (125.7 MHz, CDCl_3)

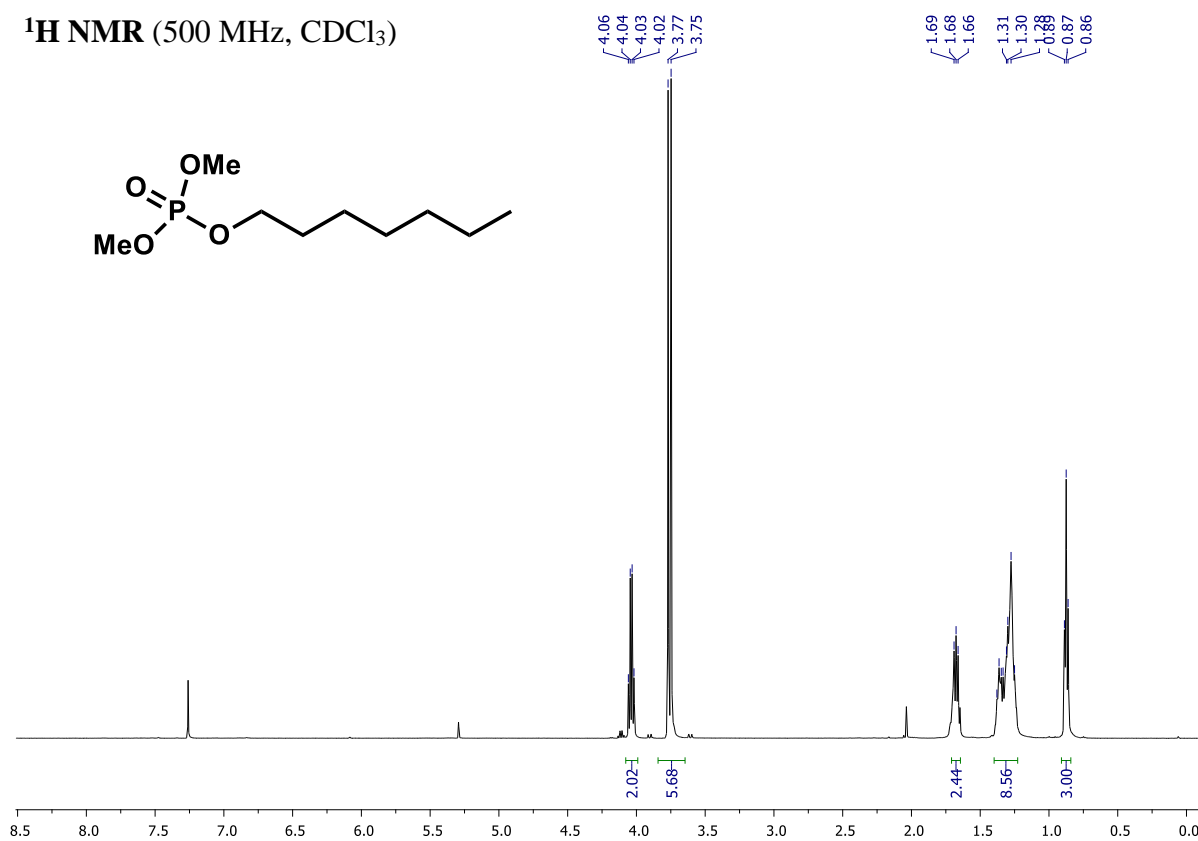


^{31}P NMR (202.5 MHz, CDCl_3)

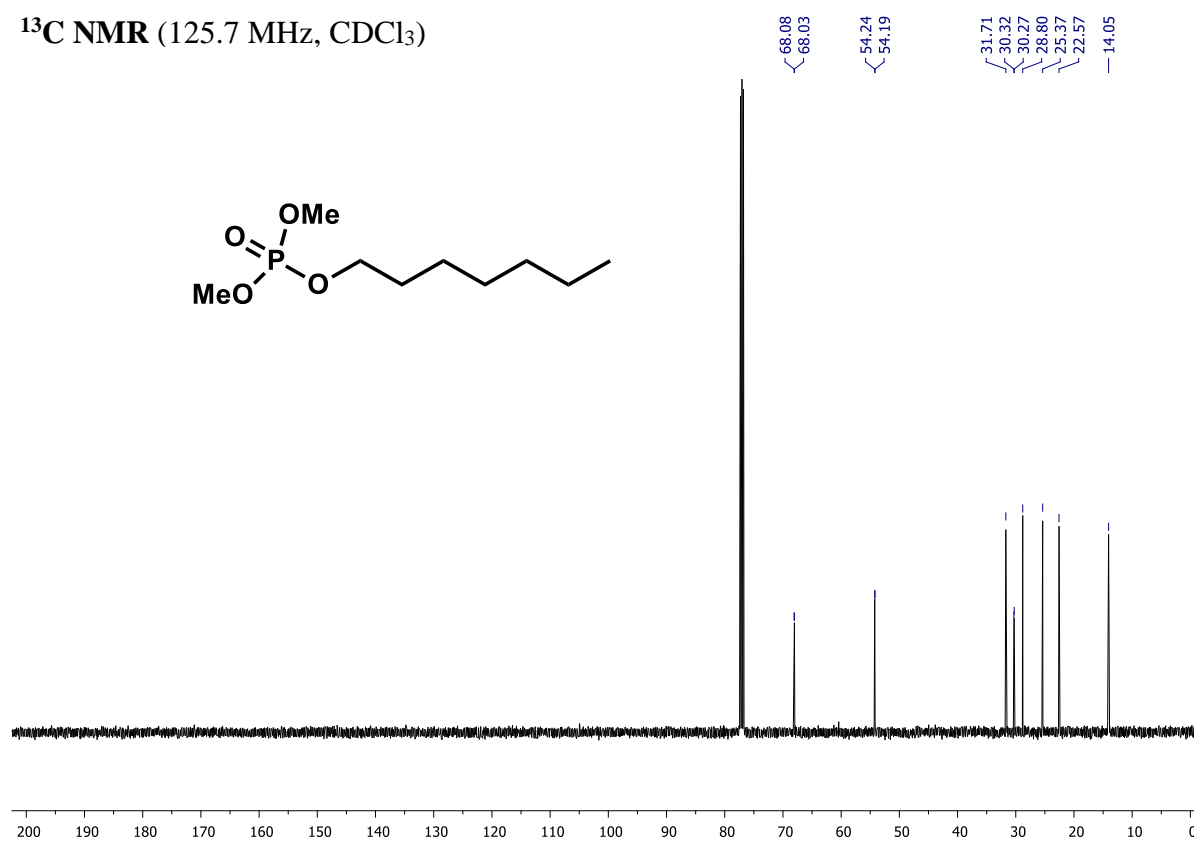


Dimethyl heptyl phosphate (3c)

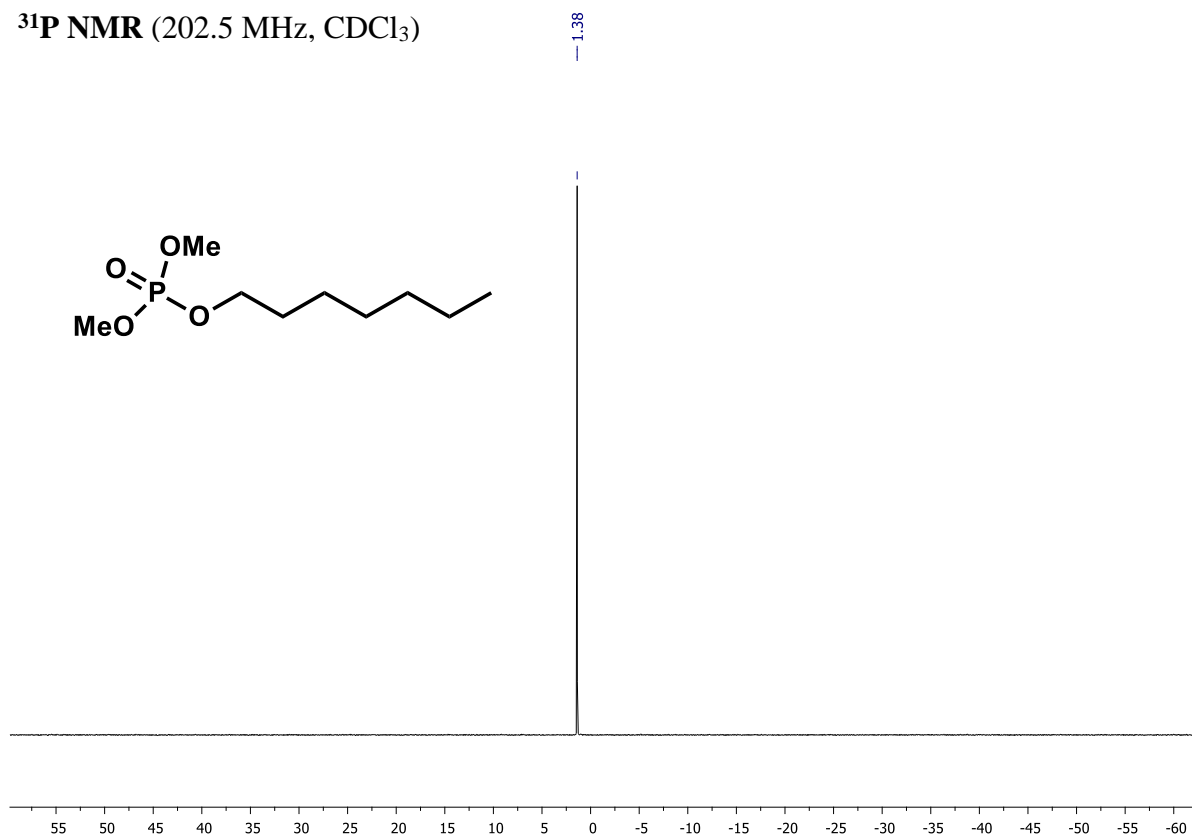
^1H NMR (500 MHz, CDCl_3)



^{13}C NMR (125.7 MHz, CDCl_3)

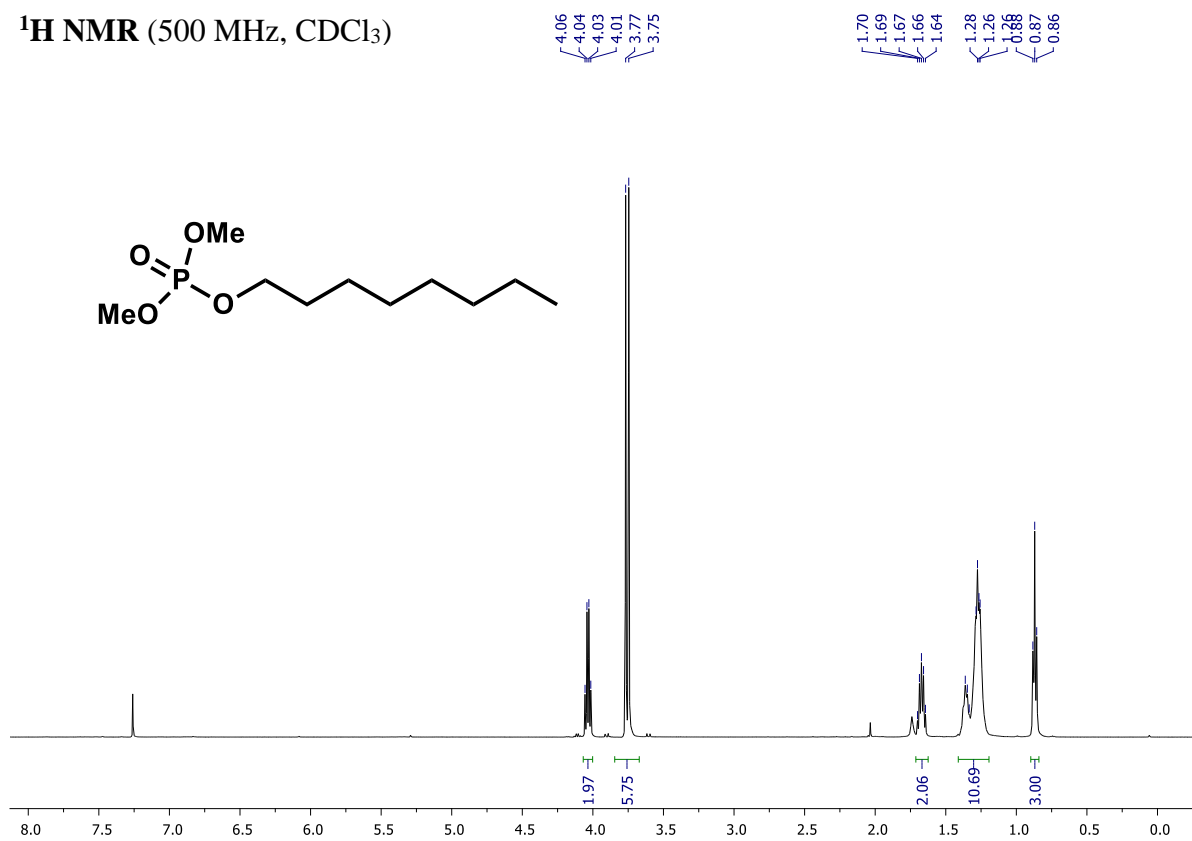


^{31}P NMR (202.5 MHz, CDCl_3)

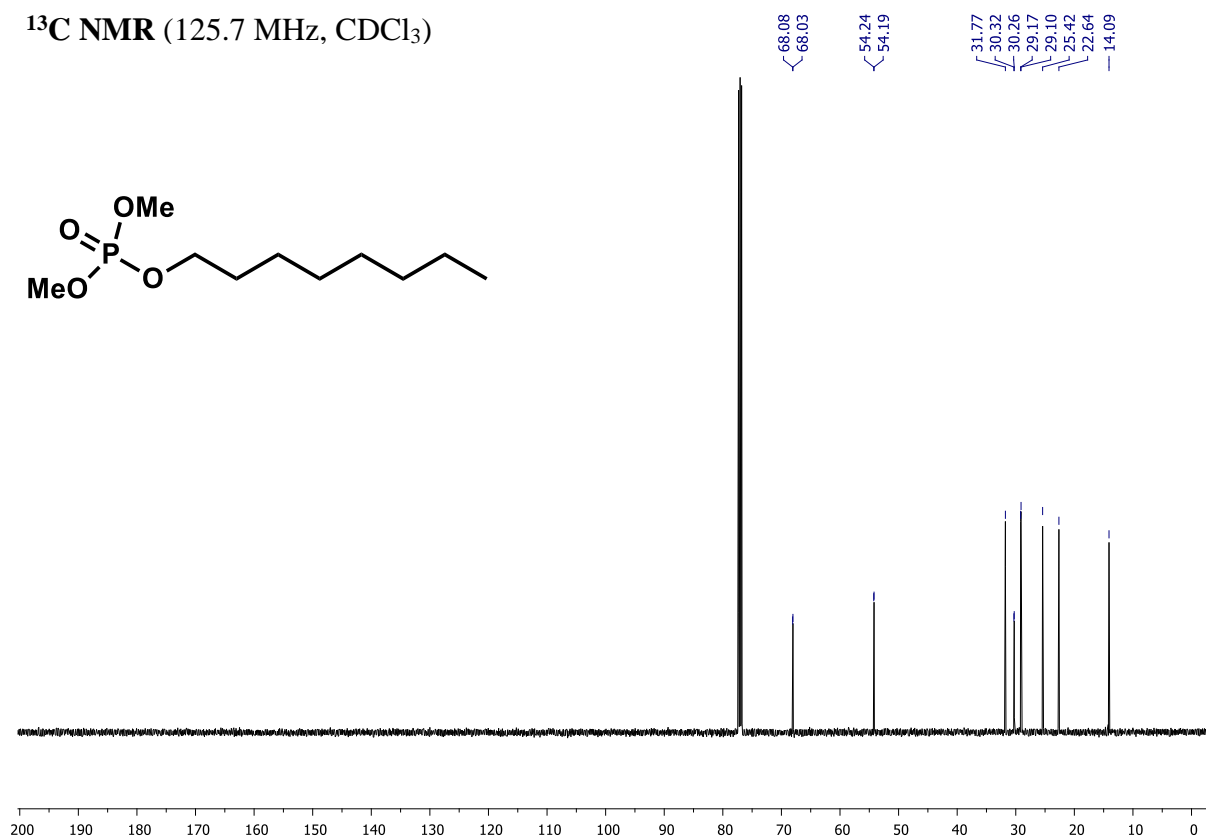
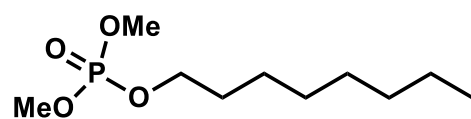


Dimethyl octyl phosphate (3d)

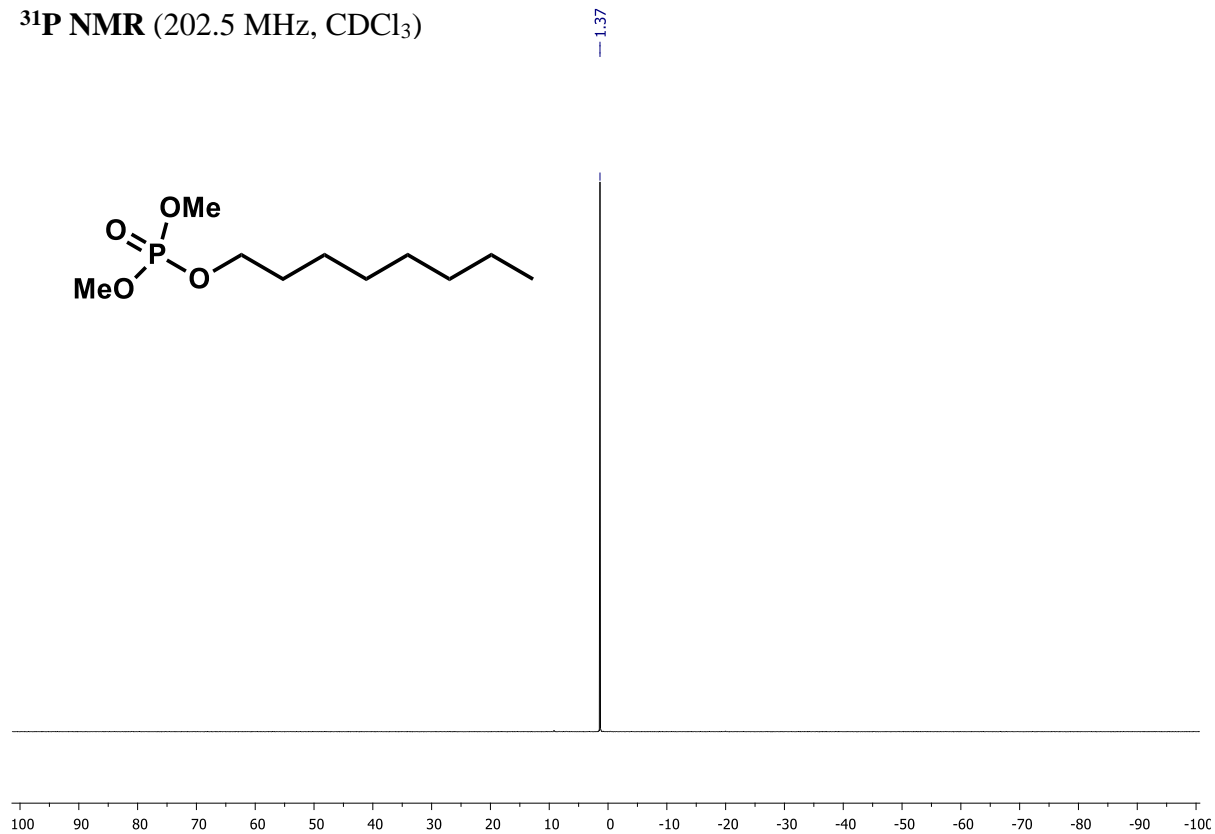
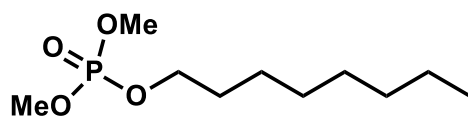
^1H NMR (500 MHz, CDCl_3)



^{13}C NMR (125.7 MHz, CDCl_3)

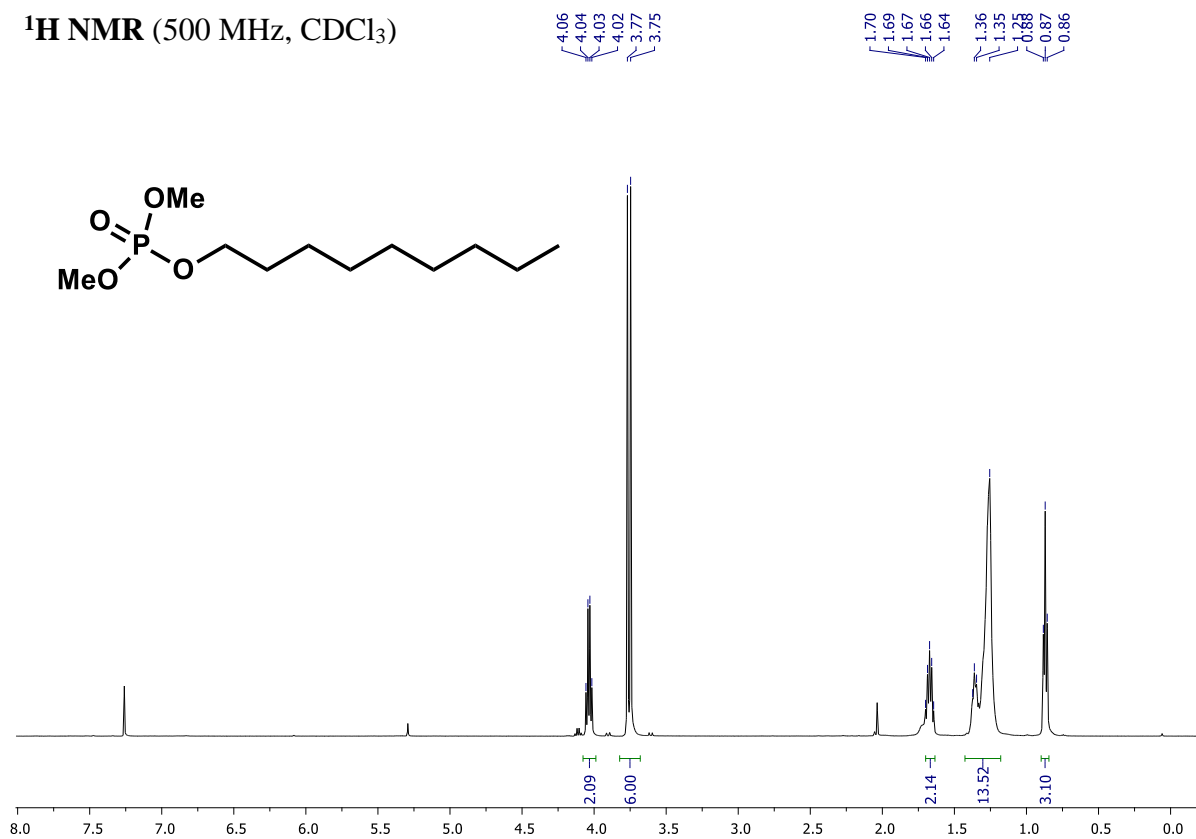


^{31}P NMR (202.5 MHz, CDCl_3)

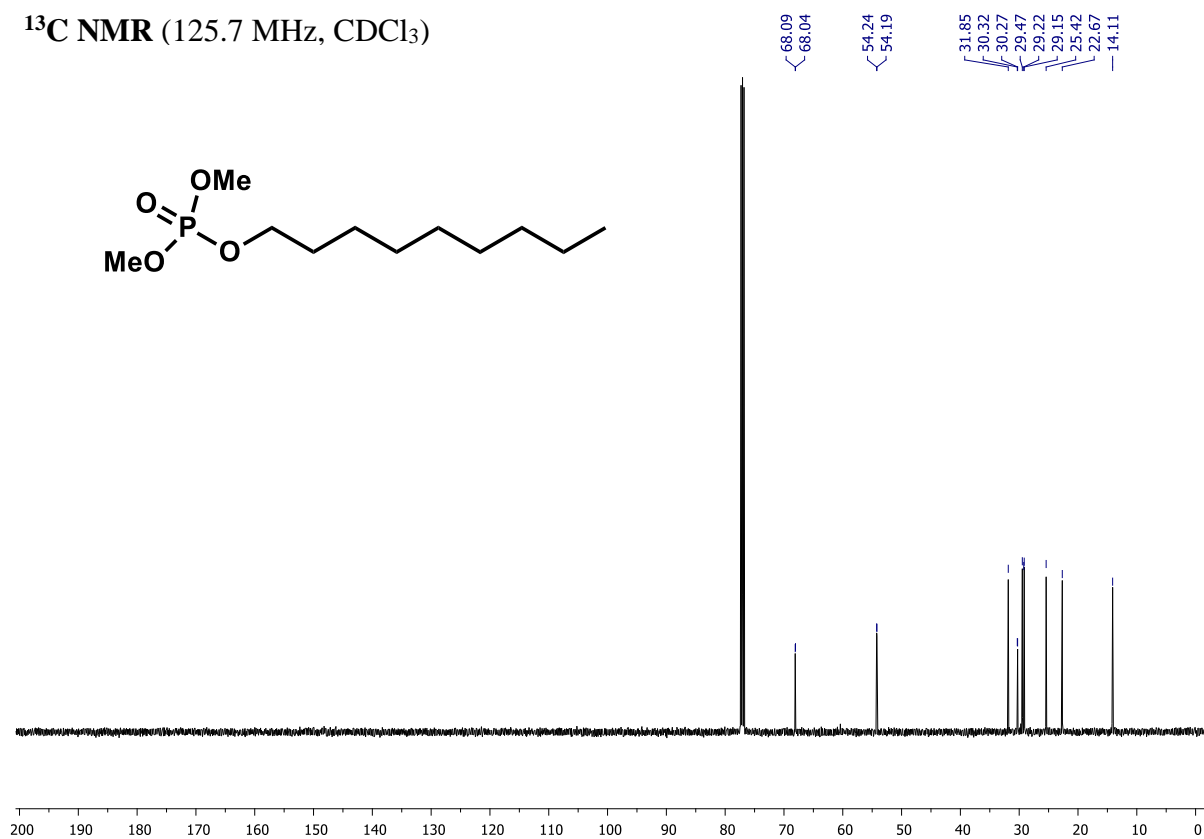


Dimethyl nonyl phosphate (3e)

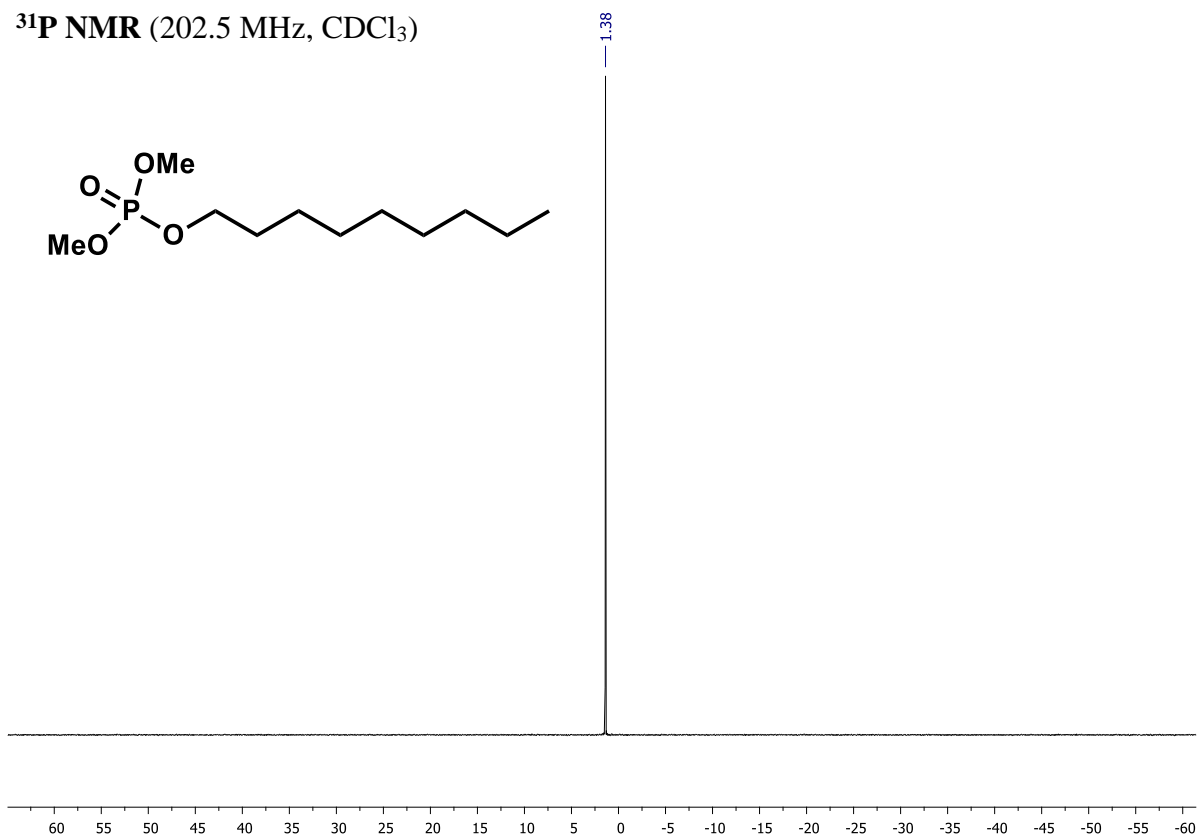
^1H NMR (500 MHz, CDCl_3)



^{13}C NMR (125.7 MHz, CDCl_3)

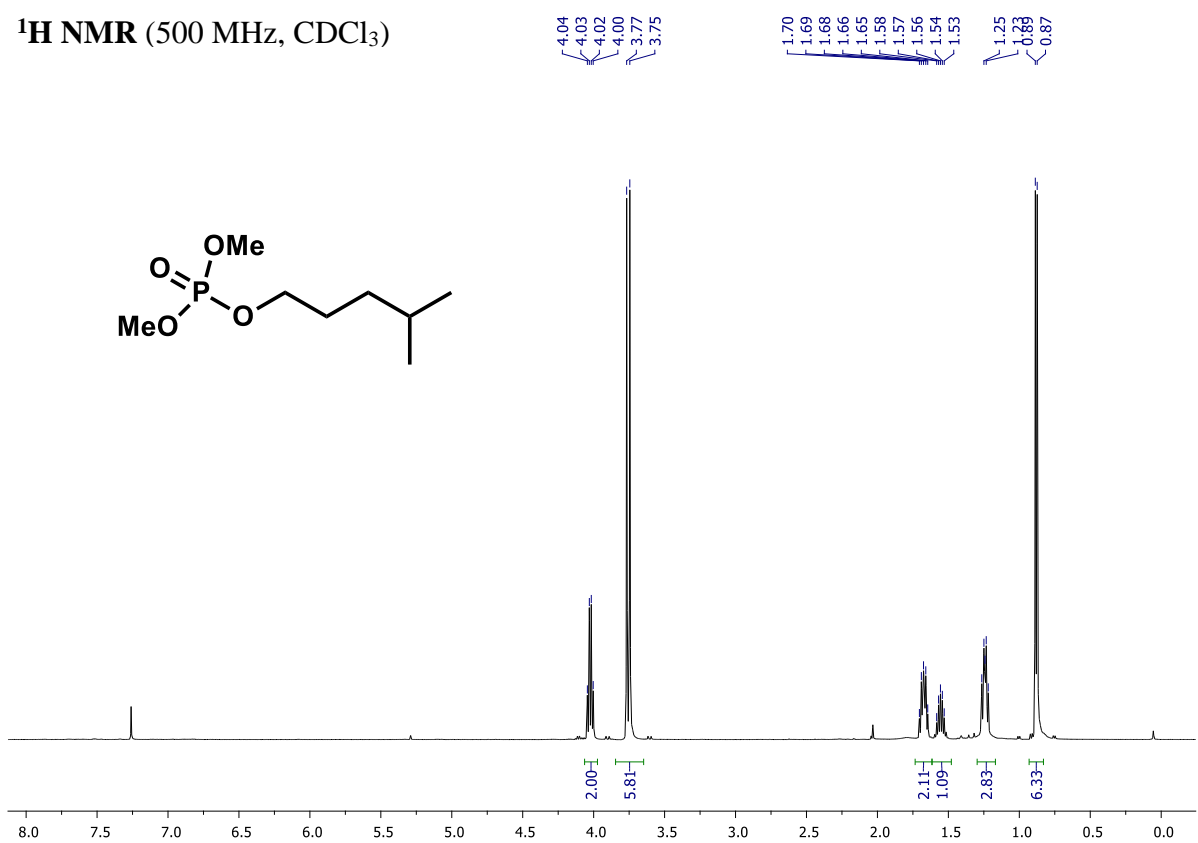


^{31}P NMR (202.5 MHz, CDCl_3)

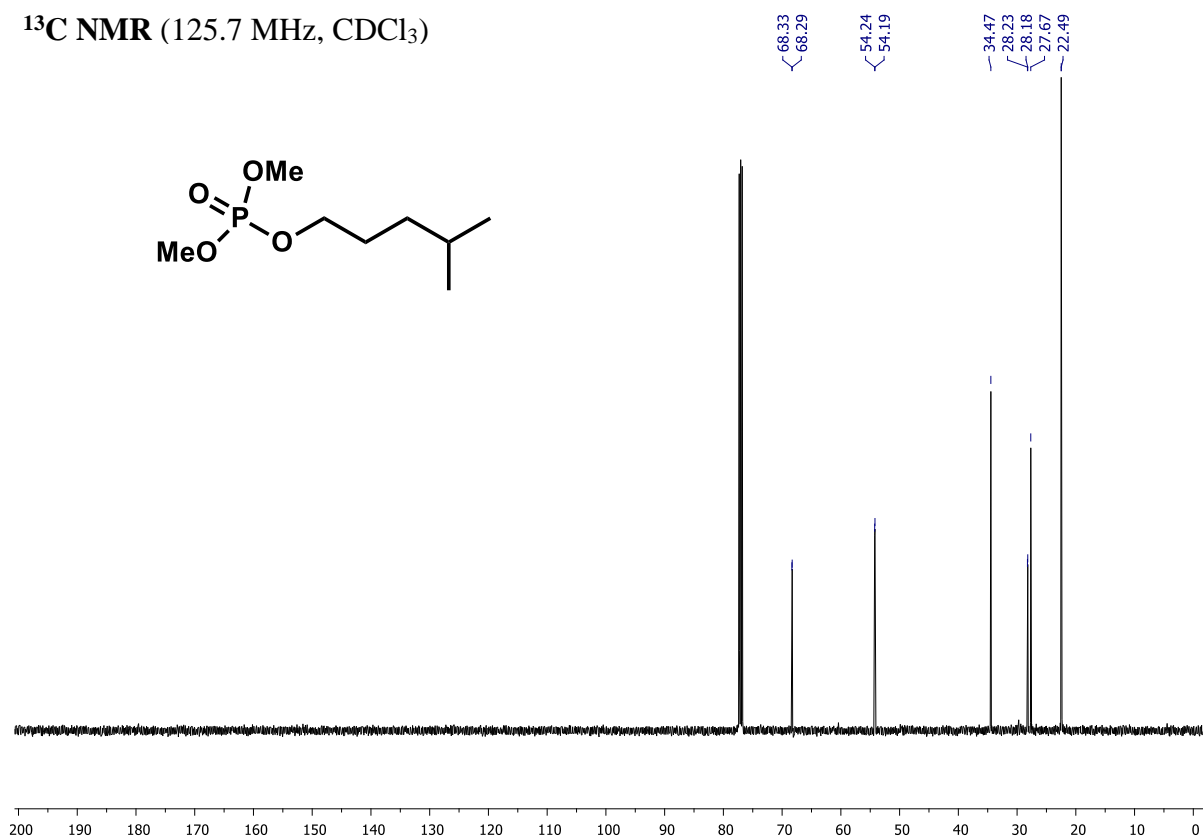


Dimethyl hexyl phosphate (3f)

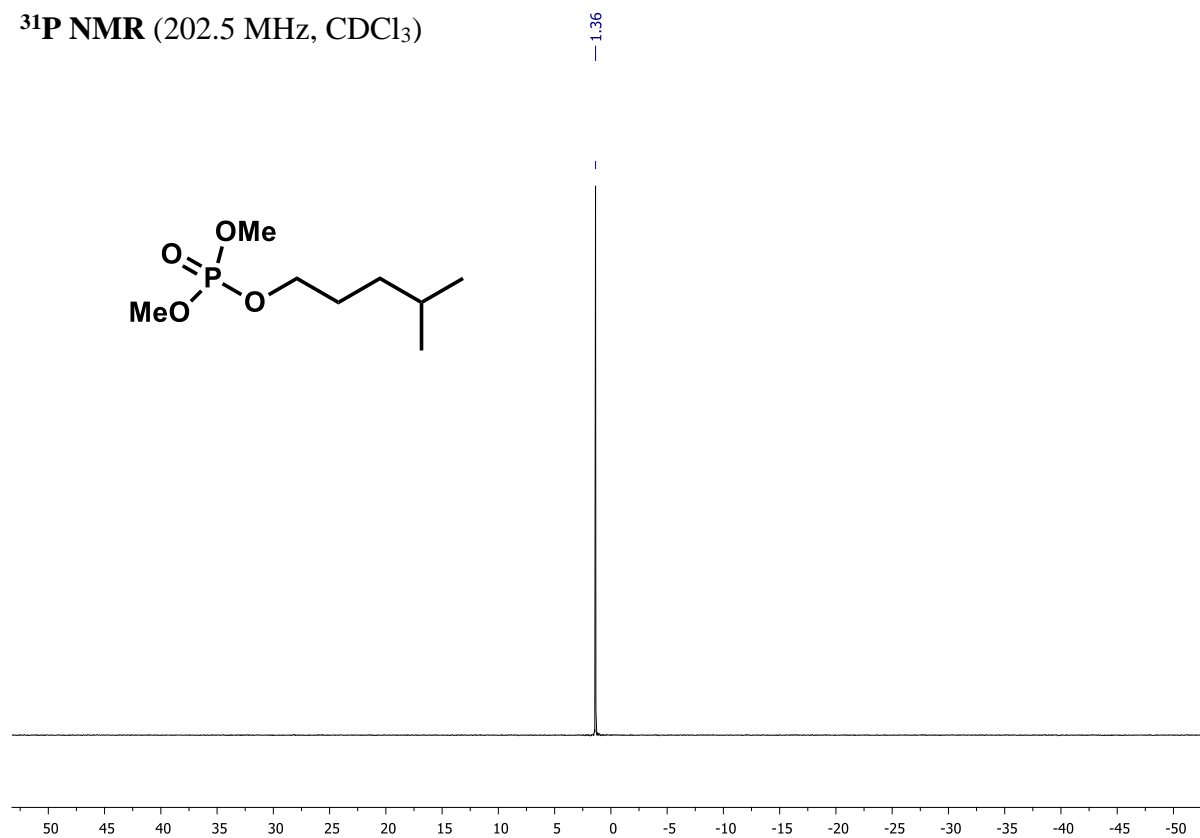
^1H NMR (500 MHz, CDCl_3)



^{13}C NMR (125.7 MHz, CDCl_3)



^{31}P NMR (202.5 MHz, CDCl_3)



Dimethyl (4-phenylbutyl) phosphate (3g)

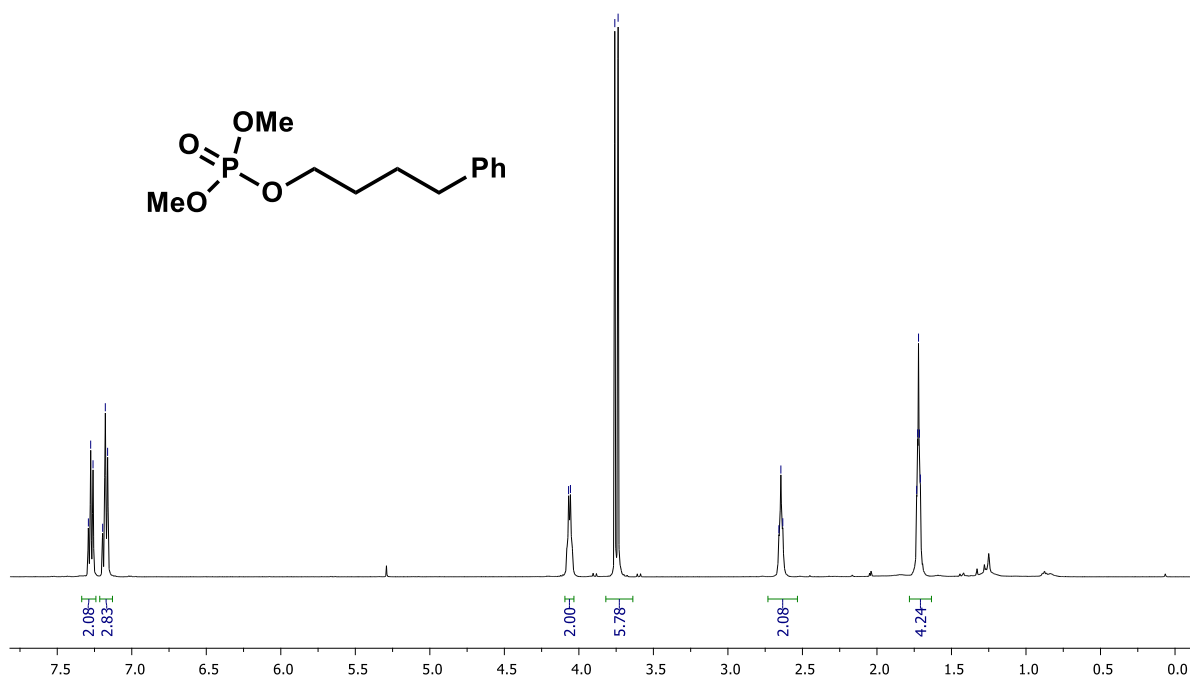
7.29
7.28
7.26
7.20
7.18
7.16

4.07
4.06
3.76
3.74

2.66
2.64
2.63

1.73
1.72
1.71

¹H NMR (500 MHz, CDCl₃)



141.92

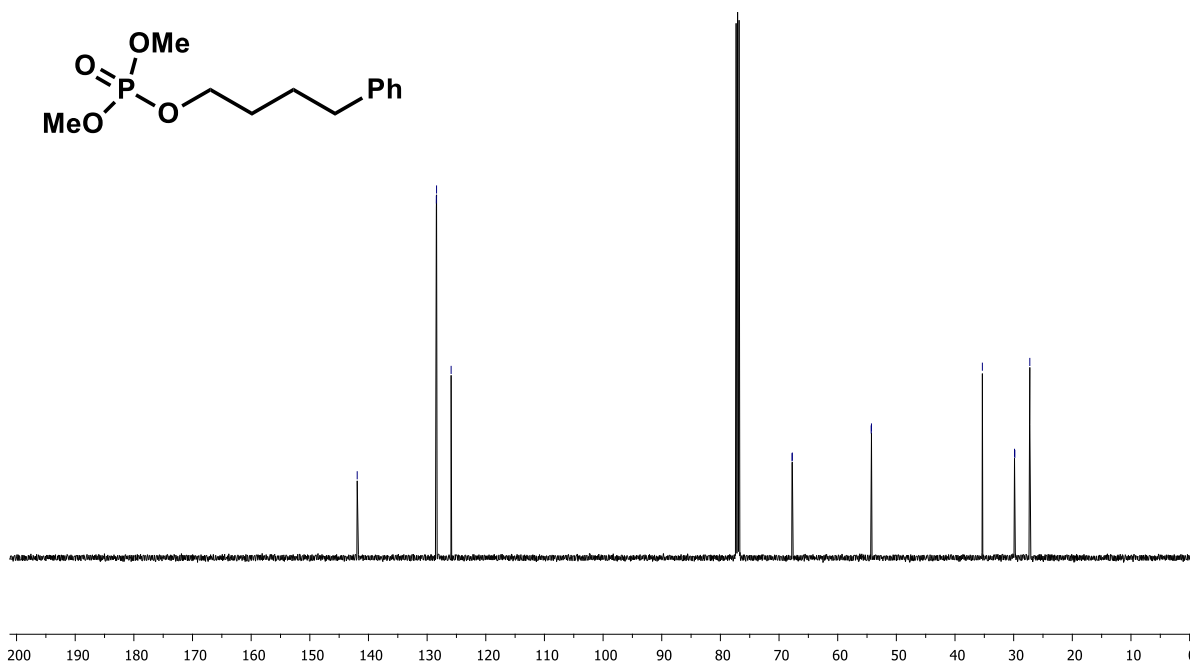
128.43
128.39
125.91

67.78
67.74

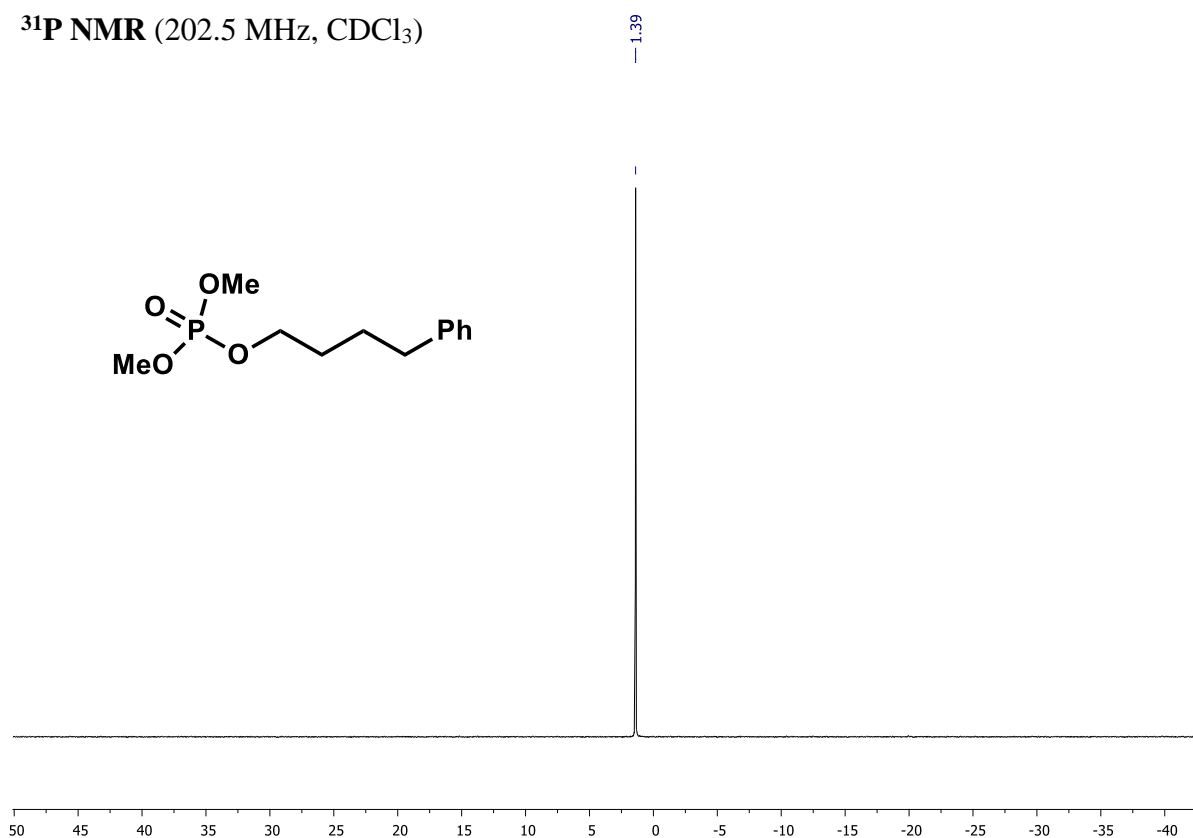
54.29
54.24

35.33
29.86
29.80
27.25

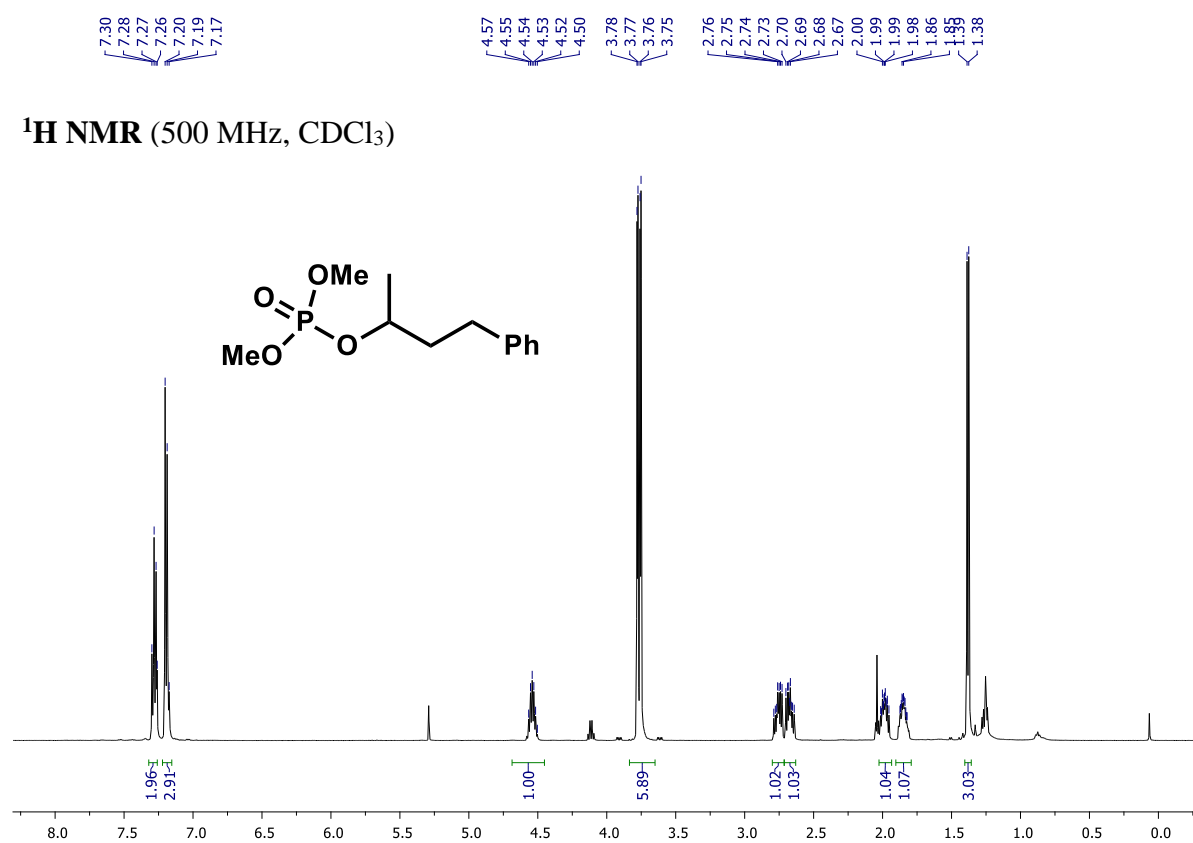
¹³C NMR (125.7 MHz, CDCl₃)

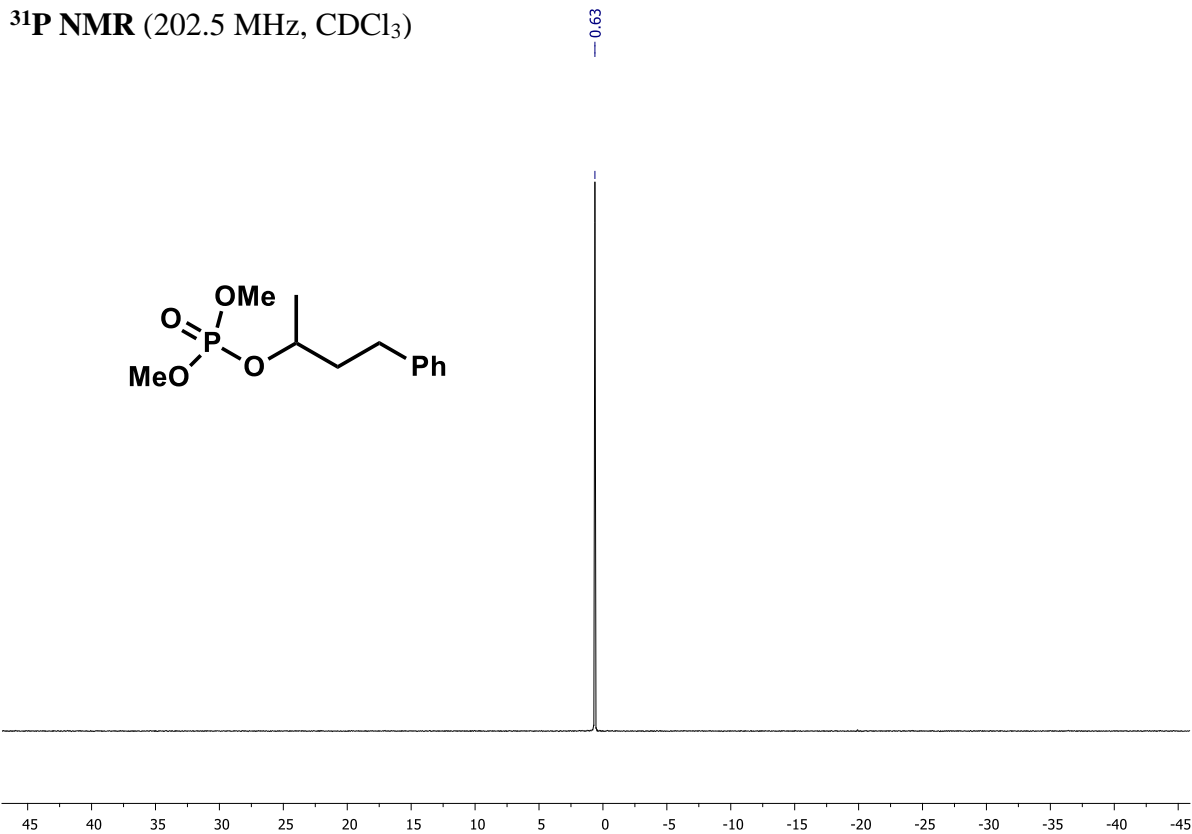
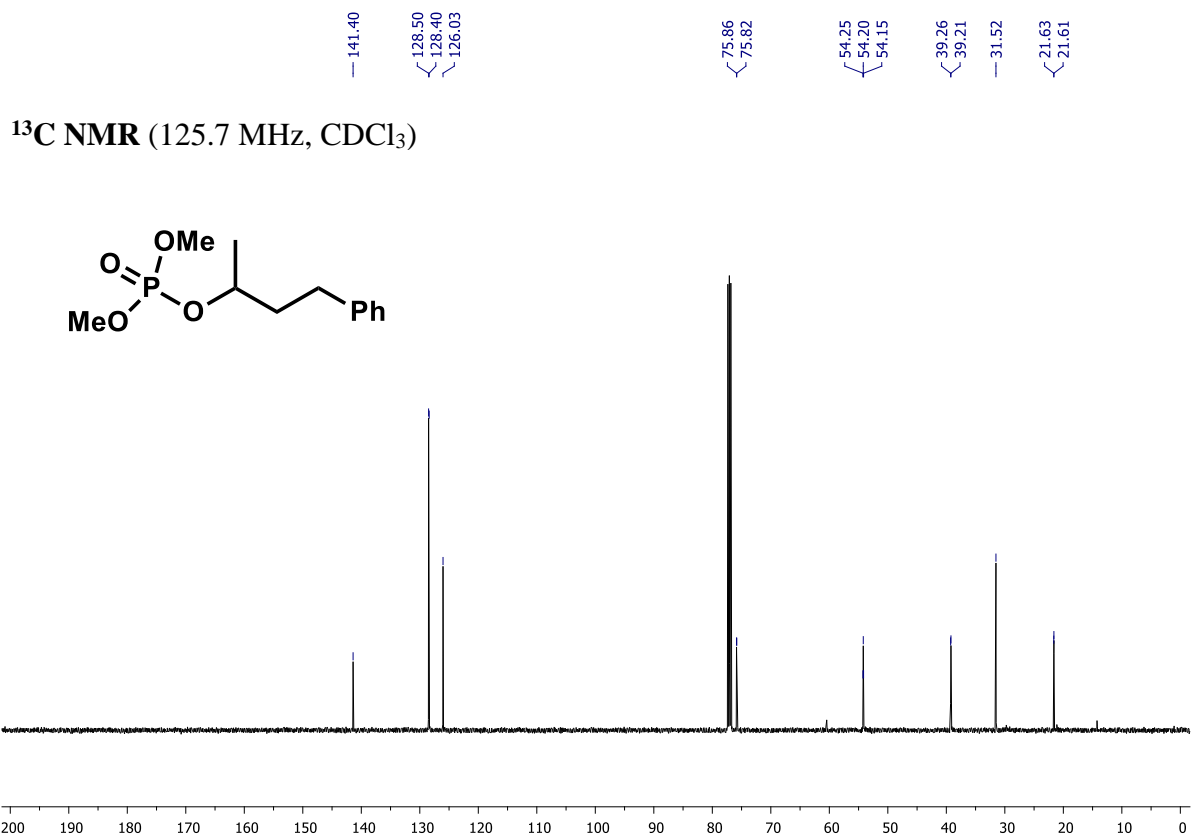


^{31}P NMR (202.5 MHz, CDCl_3)

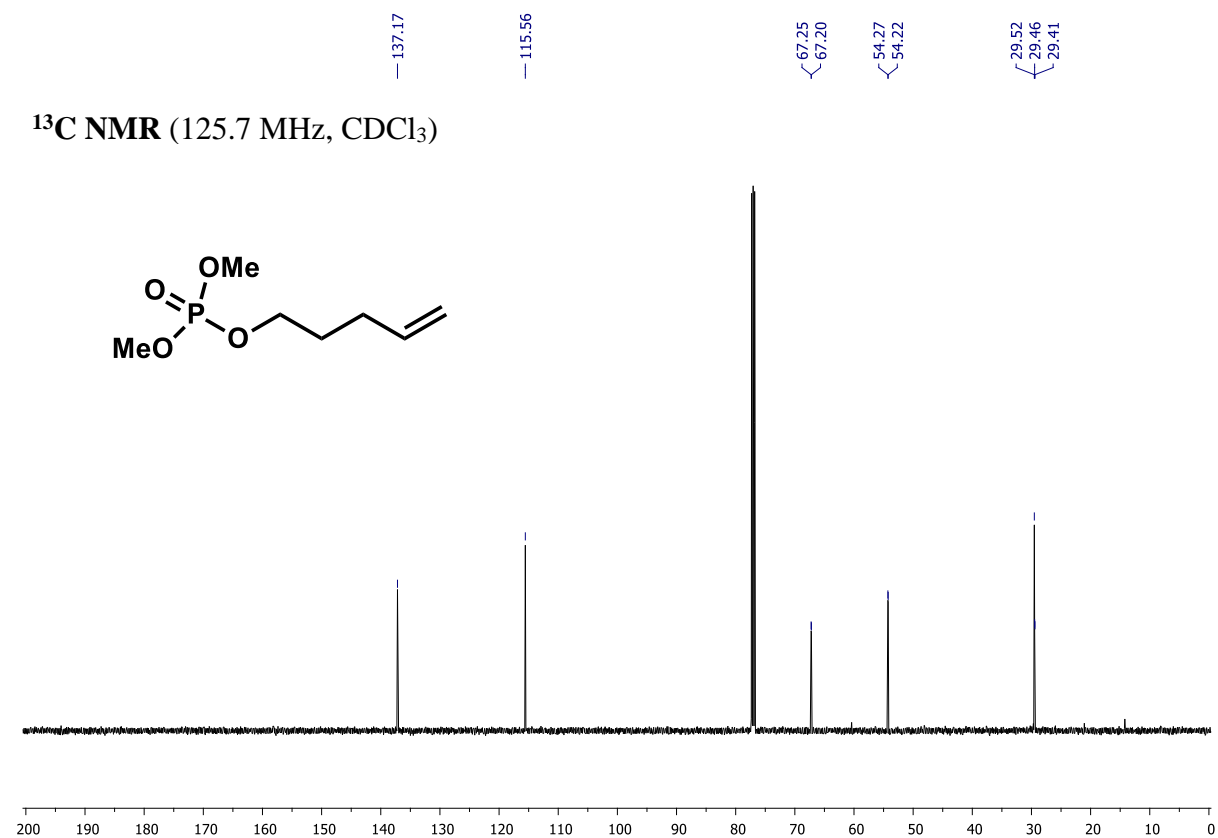
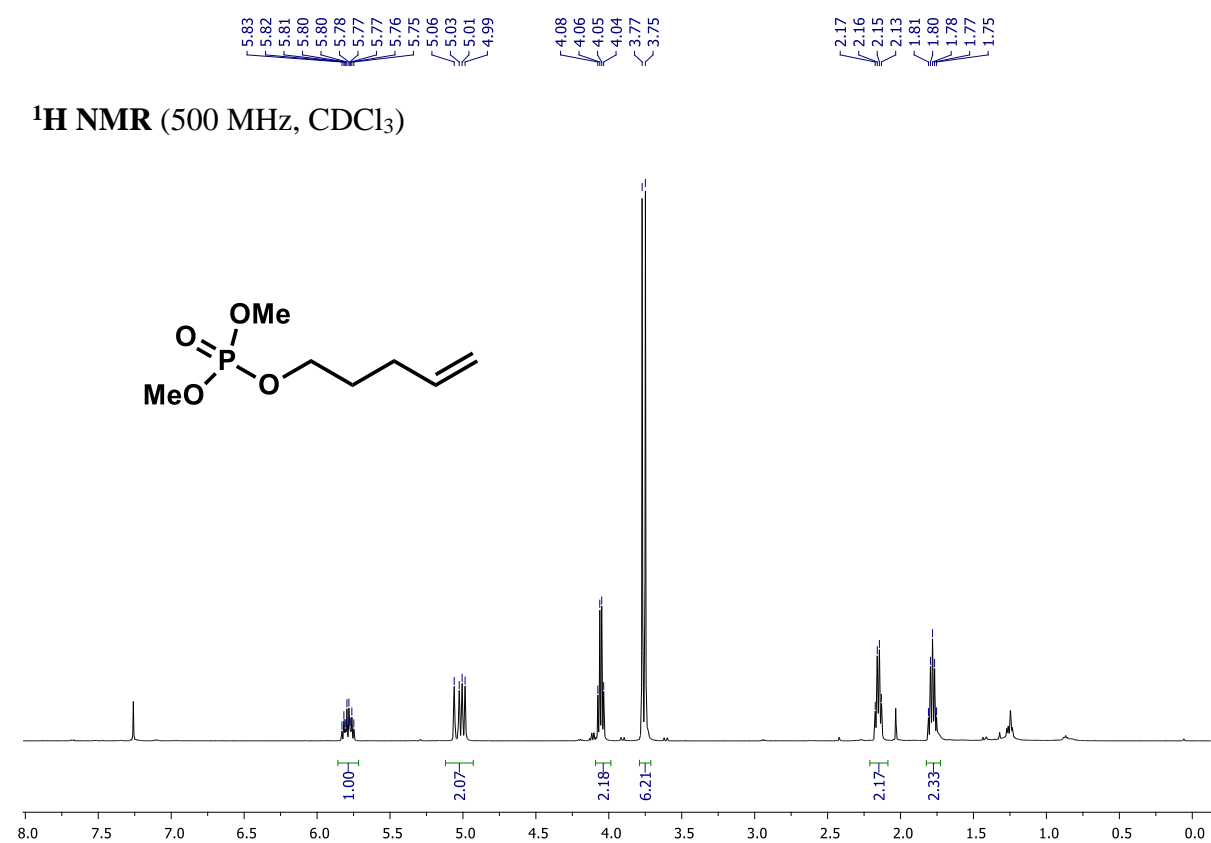


Dimethyl (4-phenylbutan-2-yl) phosphate (3h)

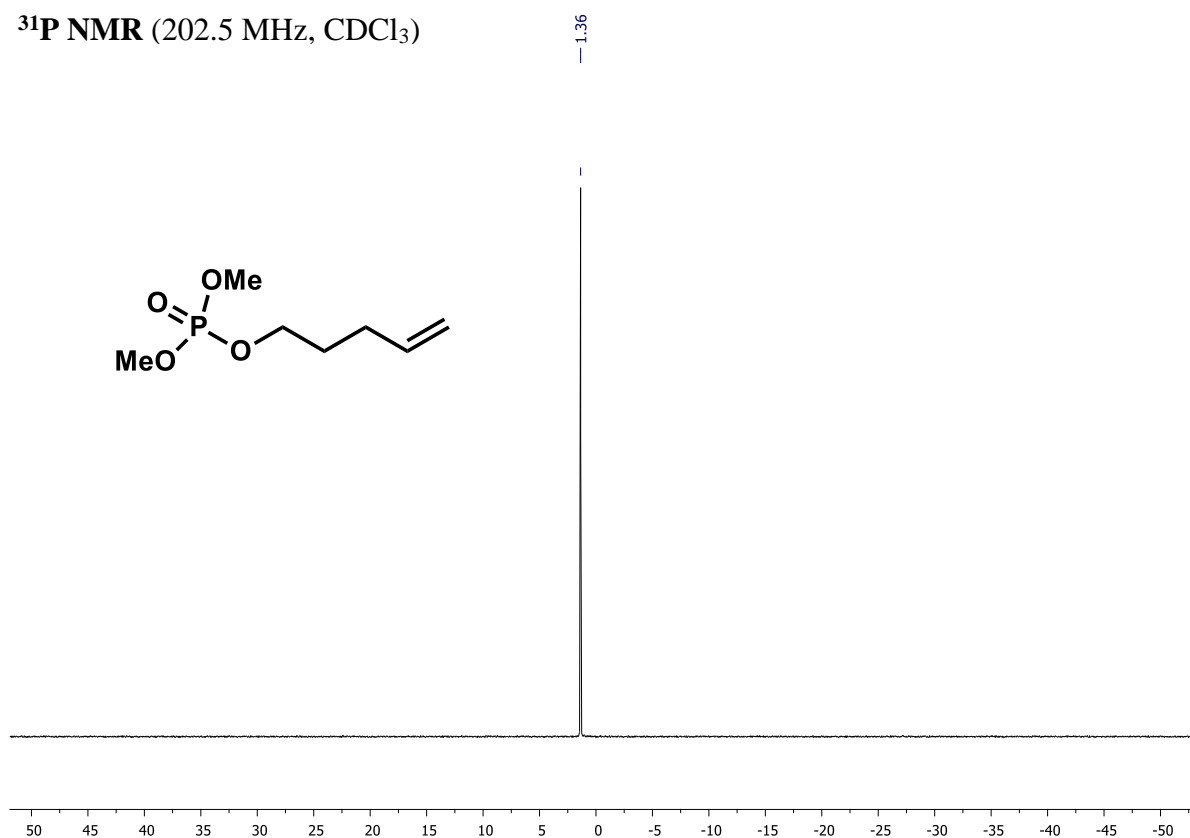




Dimethyl pent-4-en-1-yl phosphate (3i)

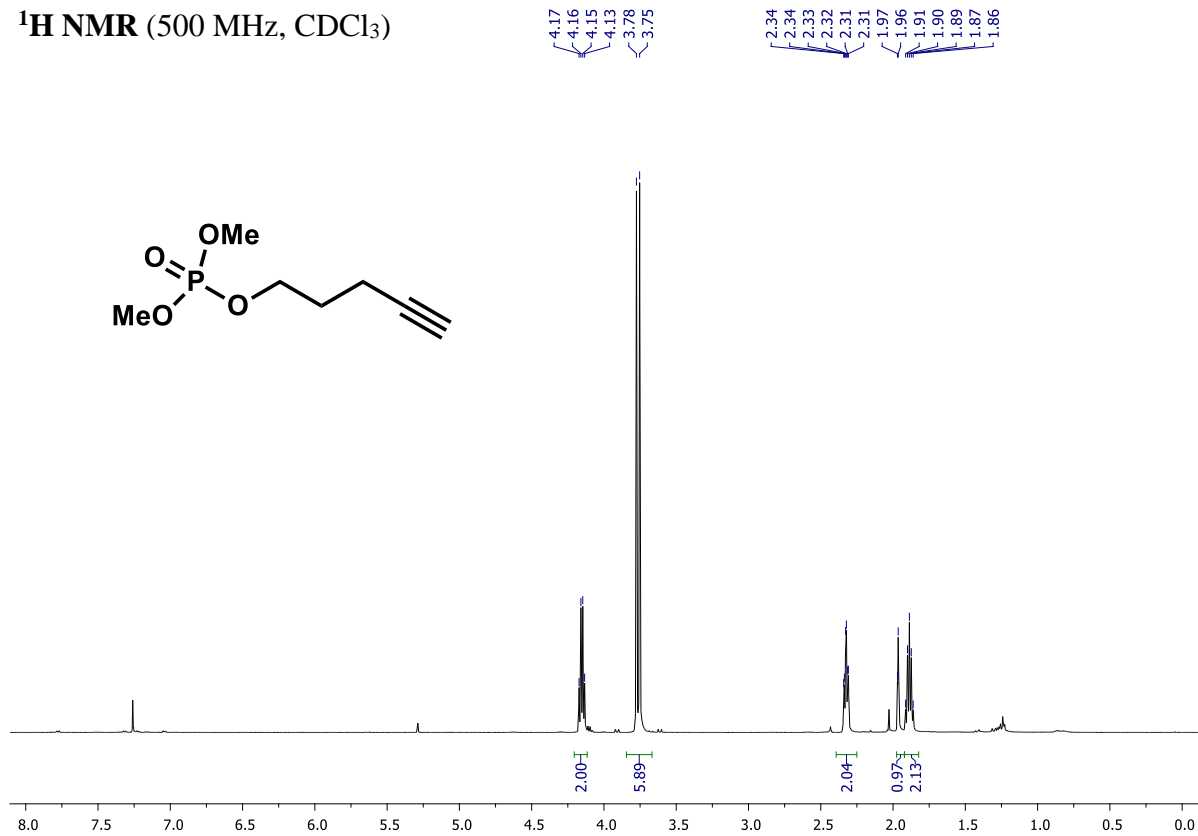


^{31}P NMR (202.5 MHz, CDCl_3)

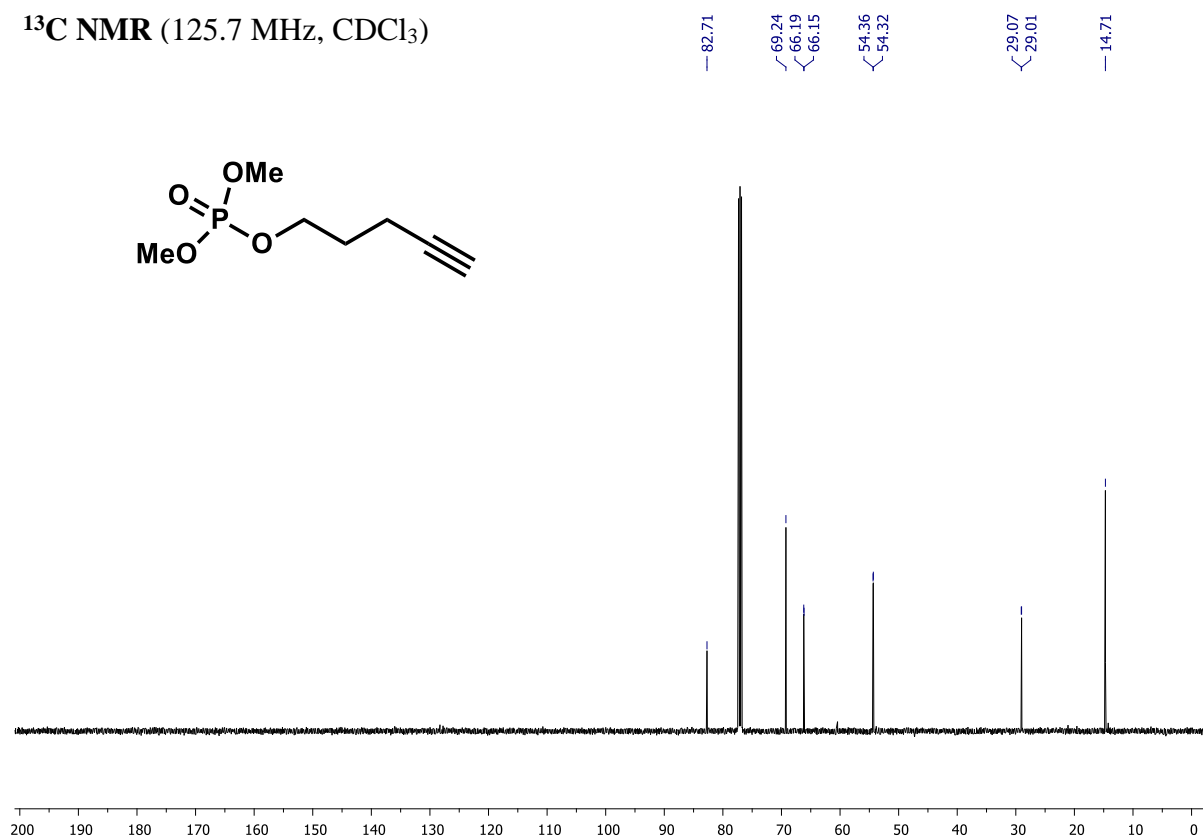


Dimethyl pent-4-yn-1-yl phosphate (3j)

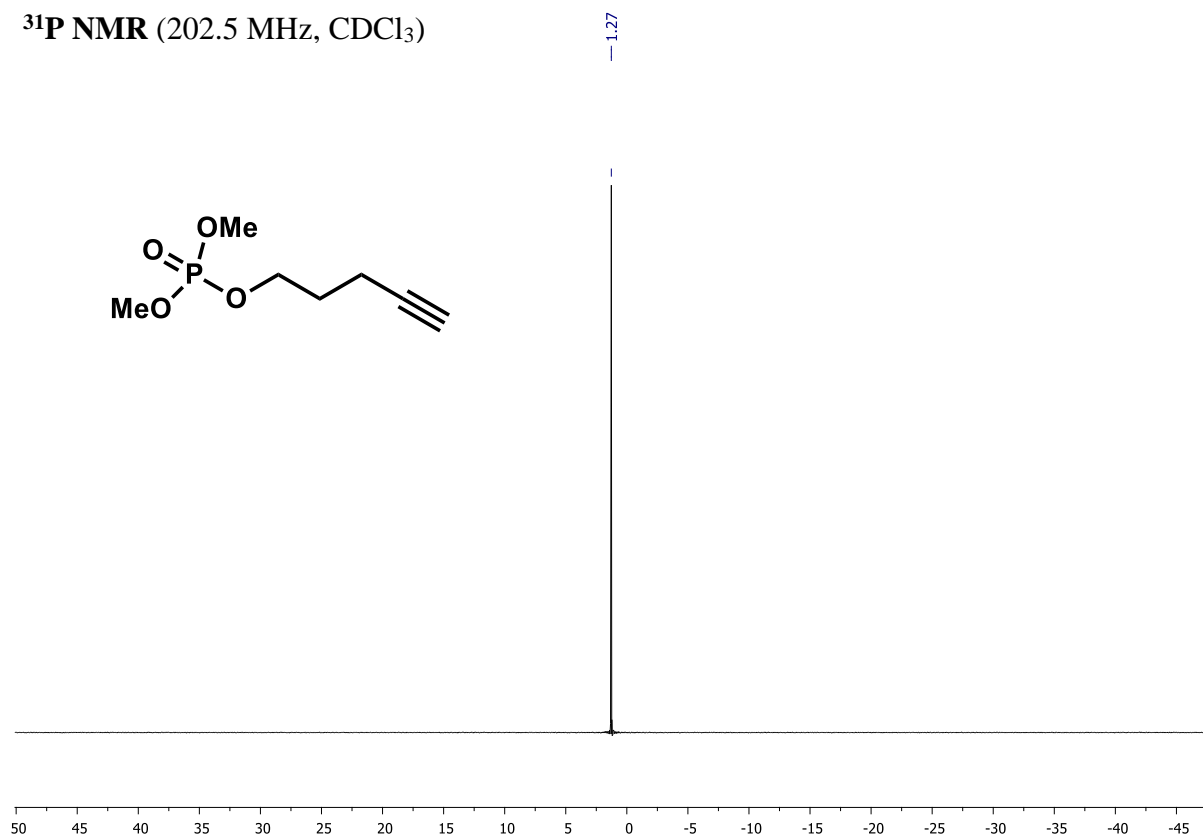
^1H NMR (500 MHz, CDCl_3)



^{13}C NMR (125.7 MHz, CDCl_3)

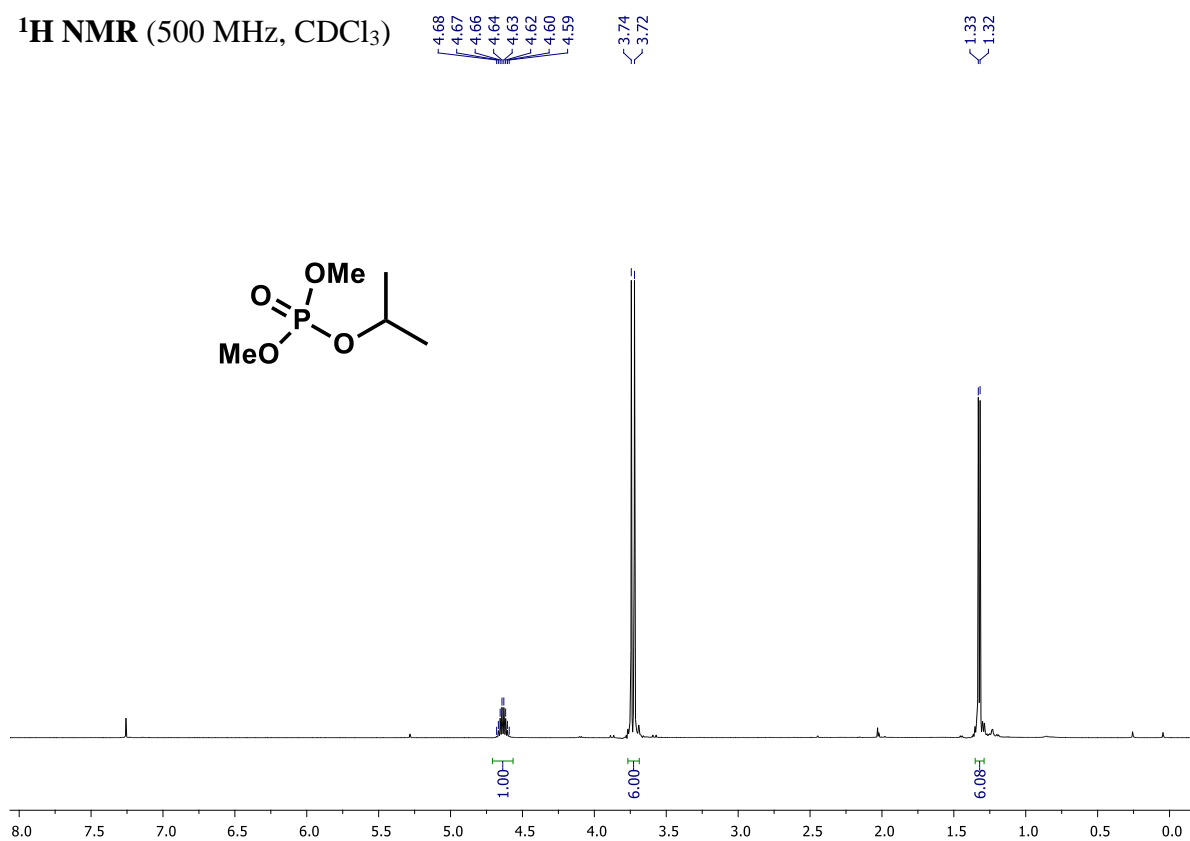


^{31}P NMR (202.5 MHz, CDCl_3)

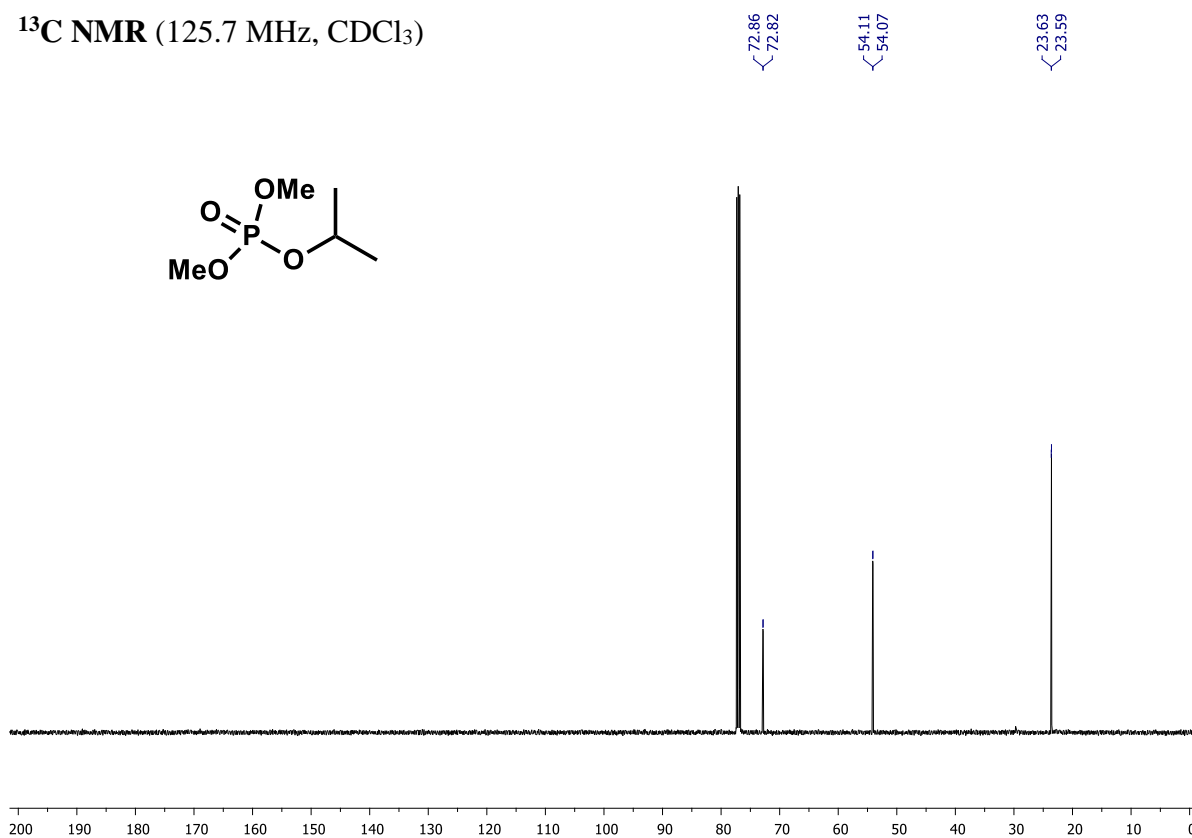


Isopropyl dimethyl phosphate (3k)

^1H NMR (500 MHz, CDCl_3)

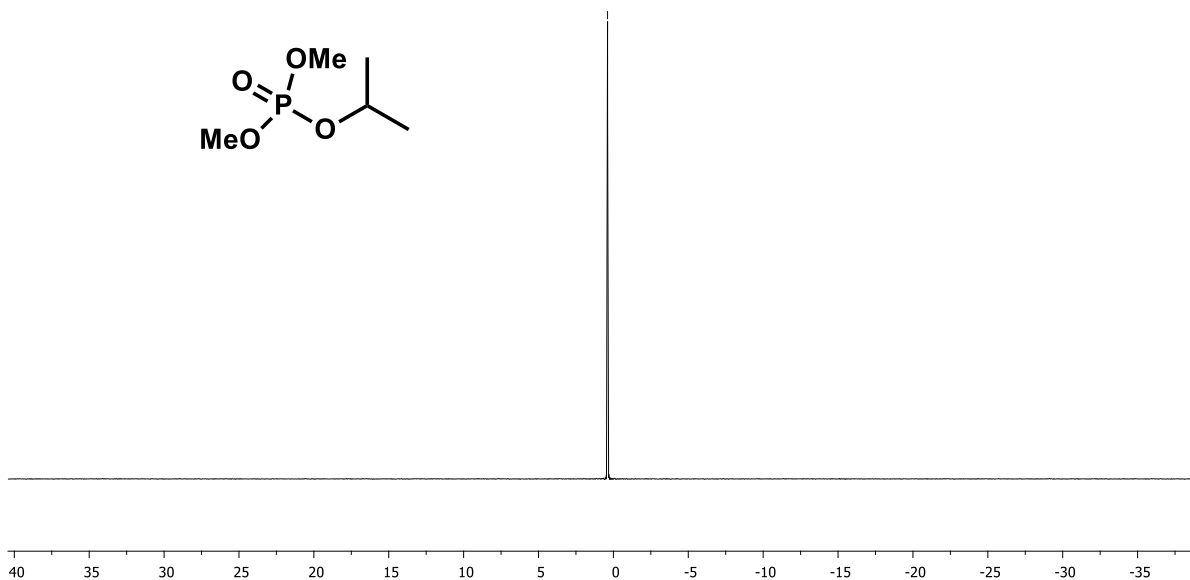
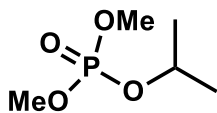


^{13}C NMR (125.7 MHz, CDCl_3)



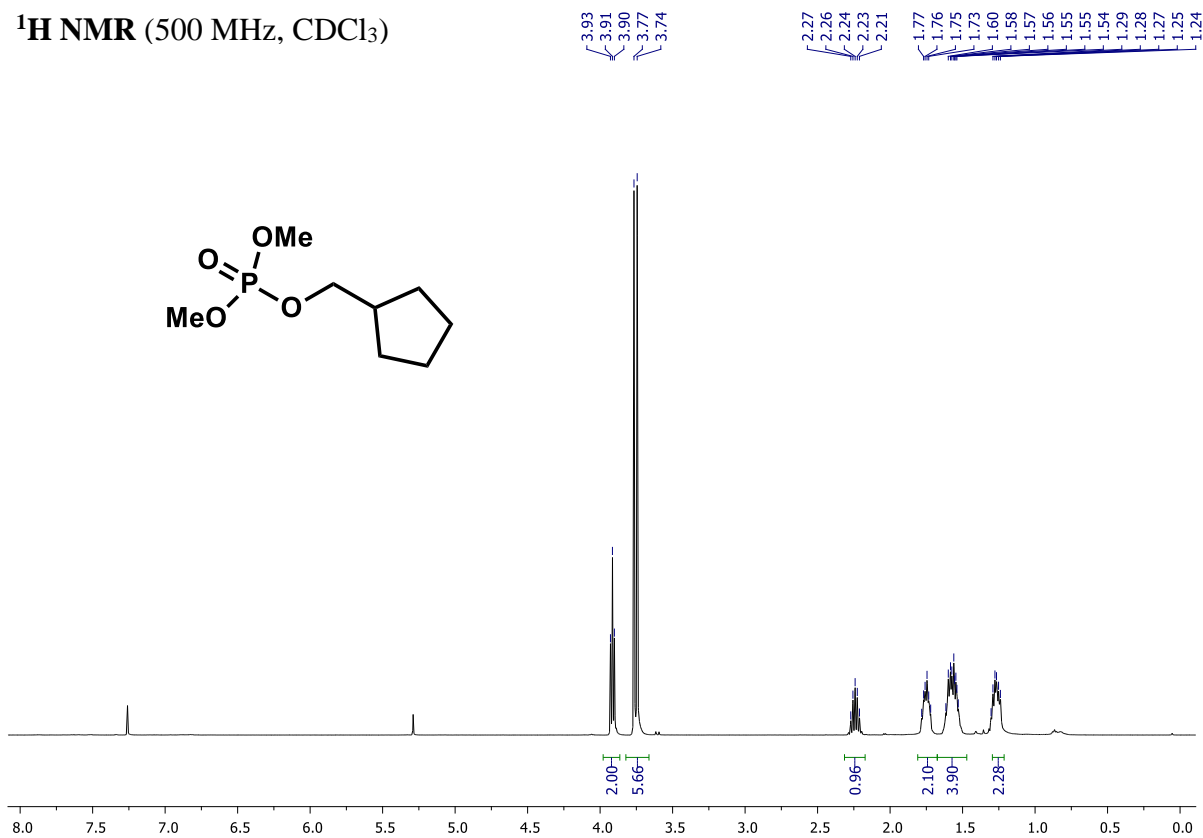
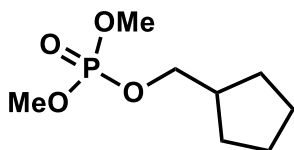
^{31}P NMR (202.5 MHz, CDCl_3)

— 0.39

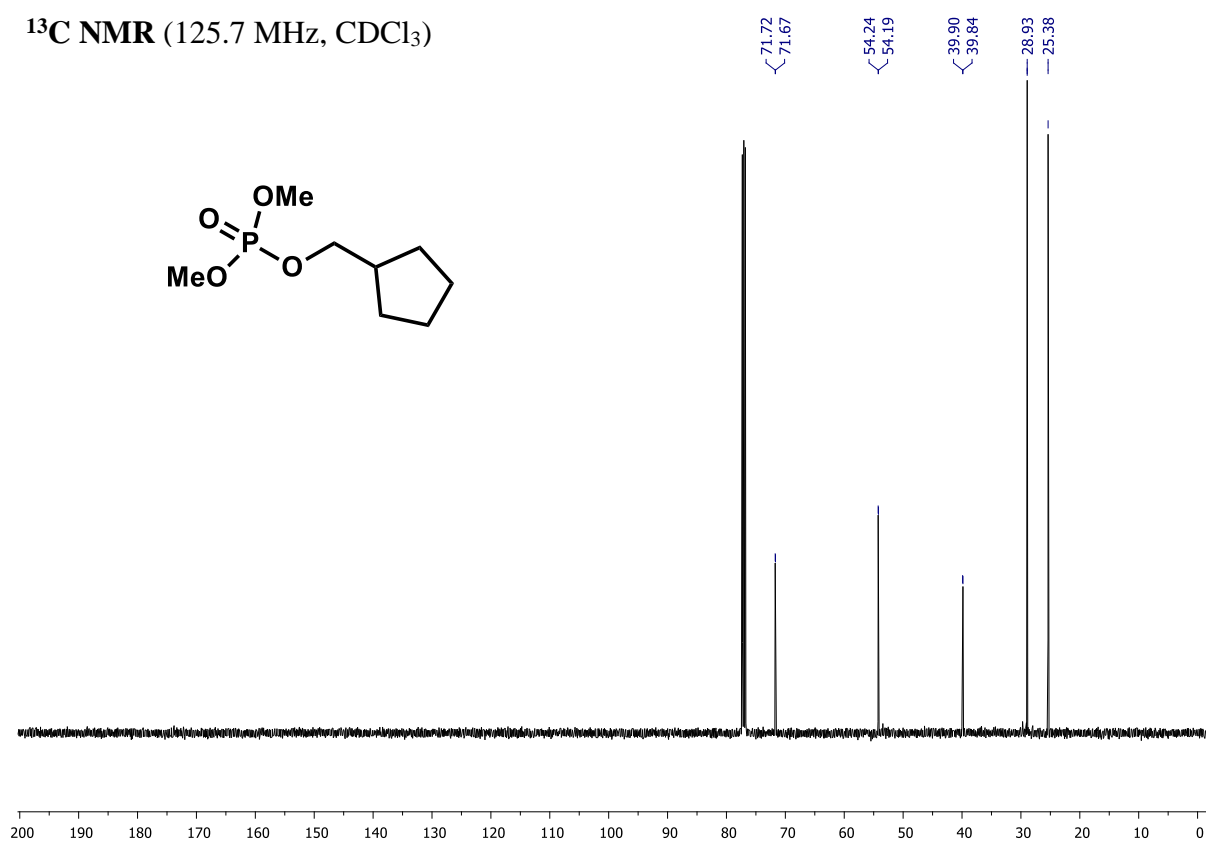


Cyclopentylmethyl dimethyl phosphate (3l)

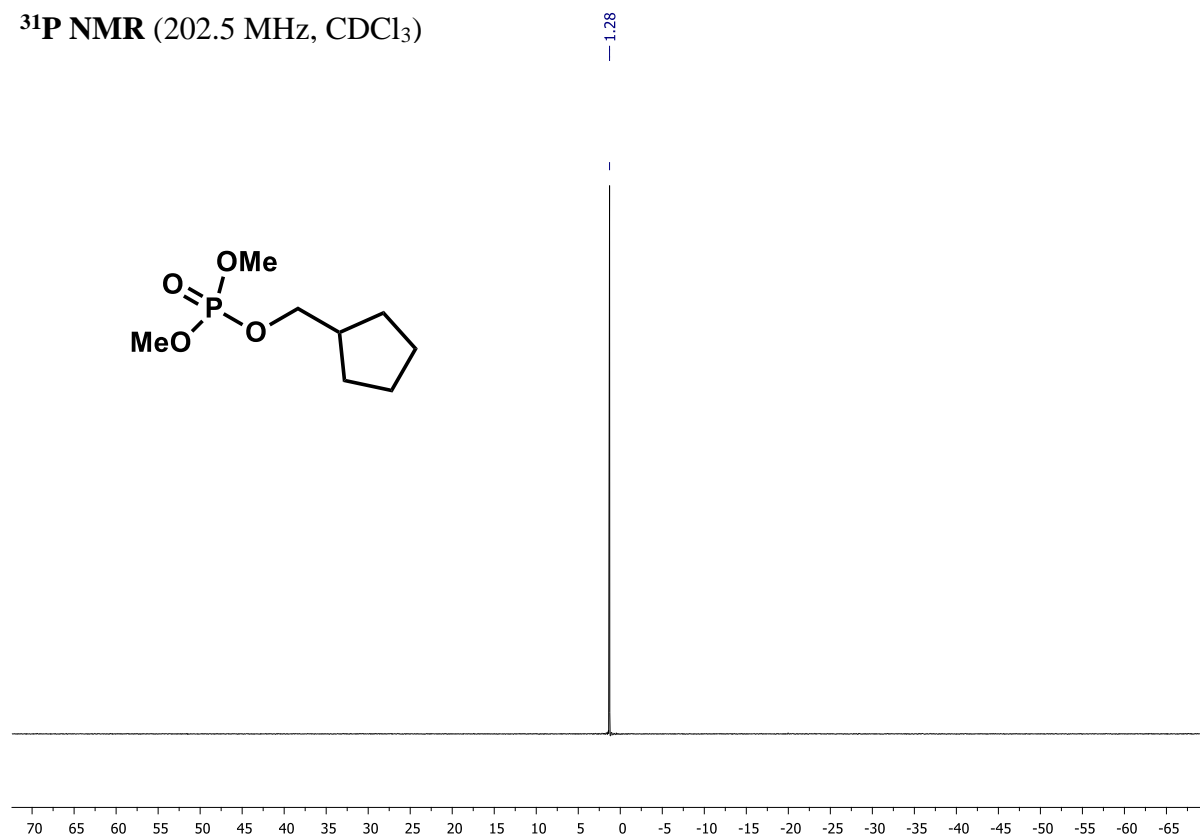
^1H NMR (500 MHz, CDCl_3)



^{13}C NMR (125.7 MHz, CDCl_3)

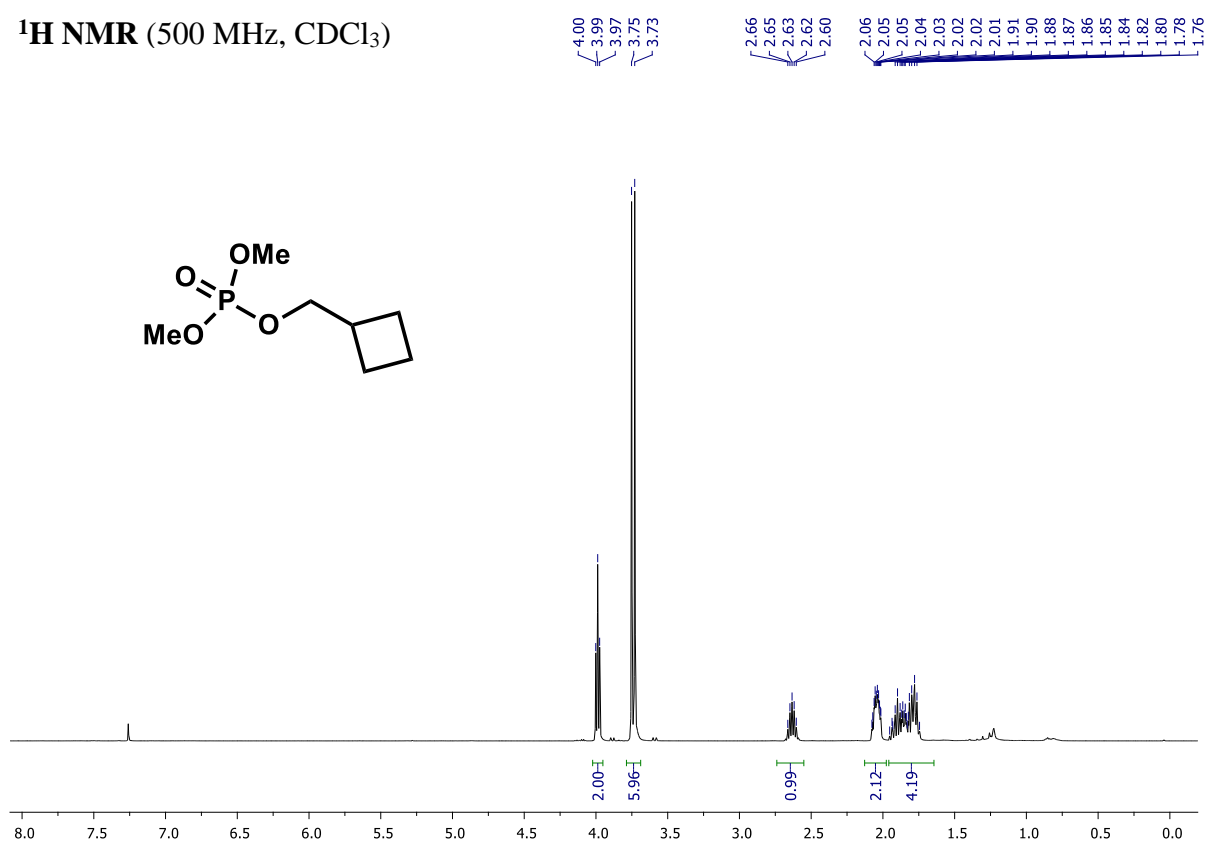


^{31}P NMR (202.5 MHz, CDCl_3)

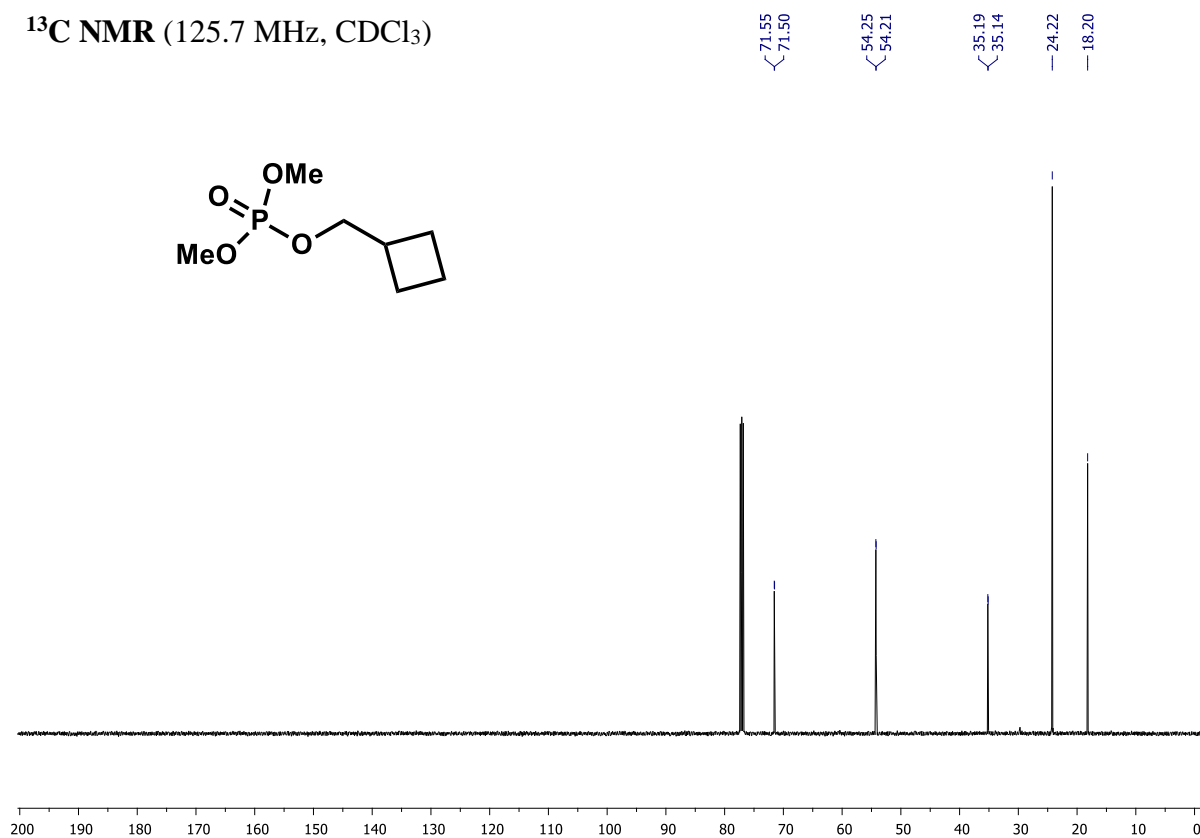


Cyclobutylmethyl dimethyl phosphate (3m)

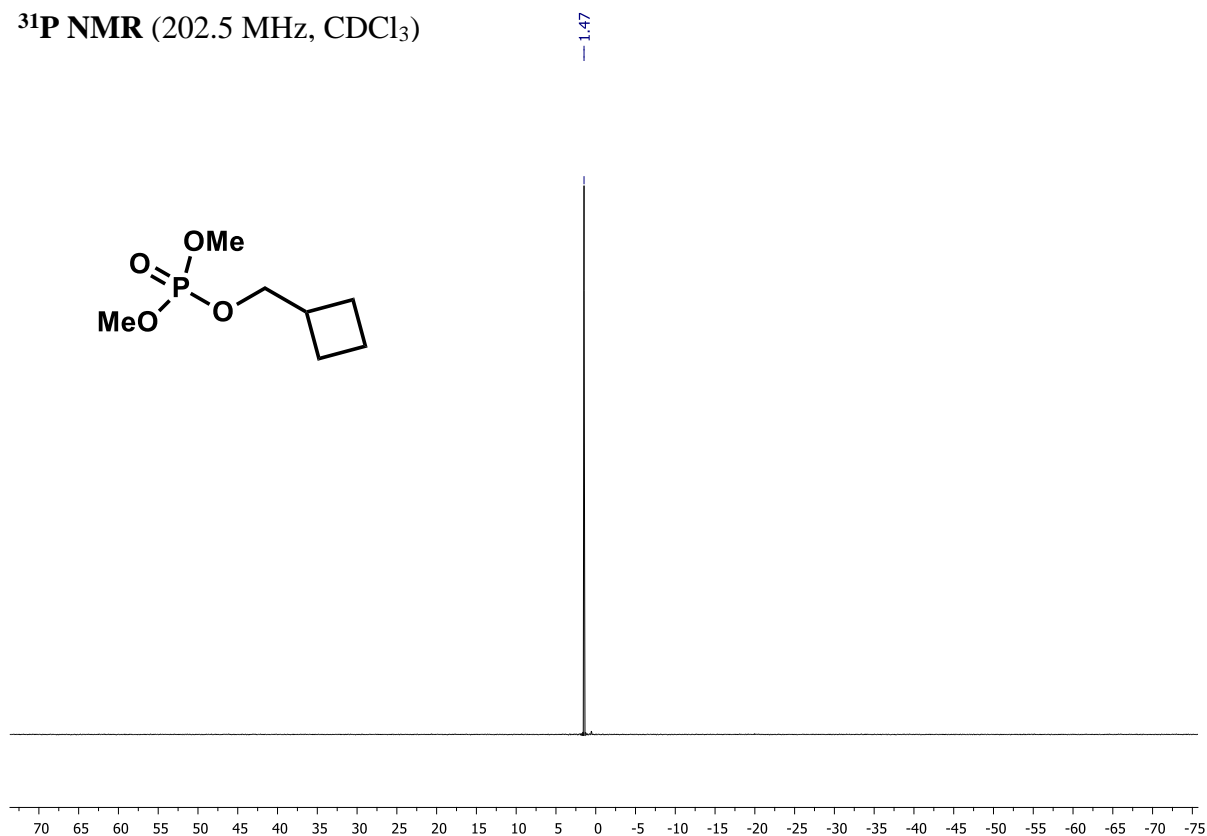
^1H NMR (500 MHz, CDCl_3)



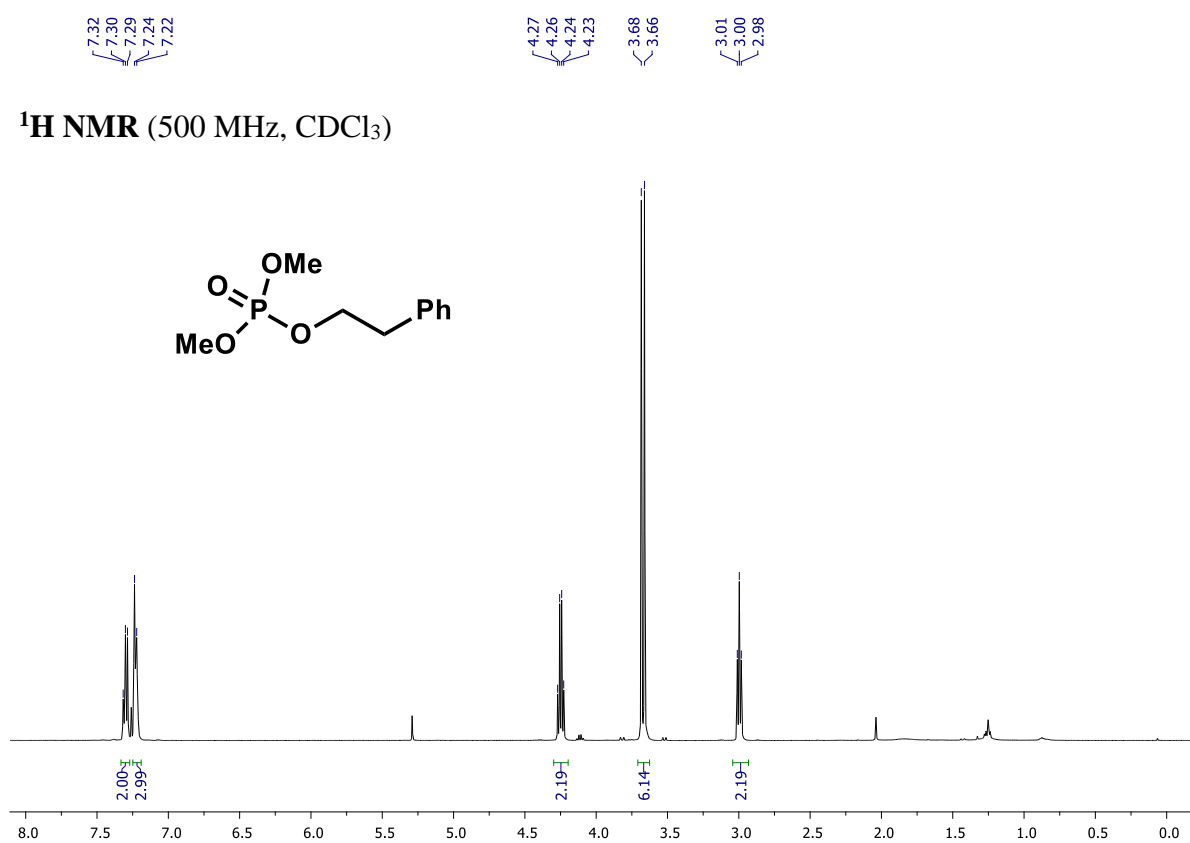
^{13}C NMR (125.7 MHz, CDCl_3)

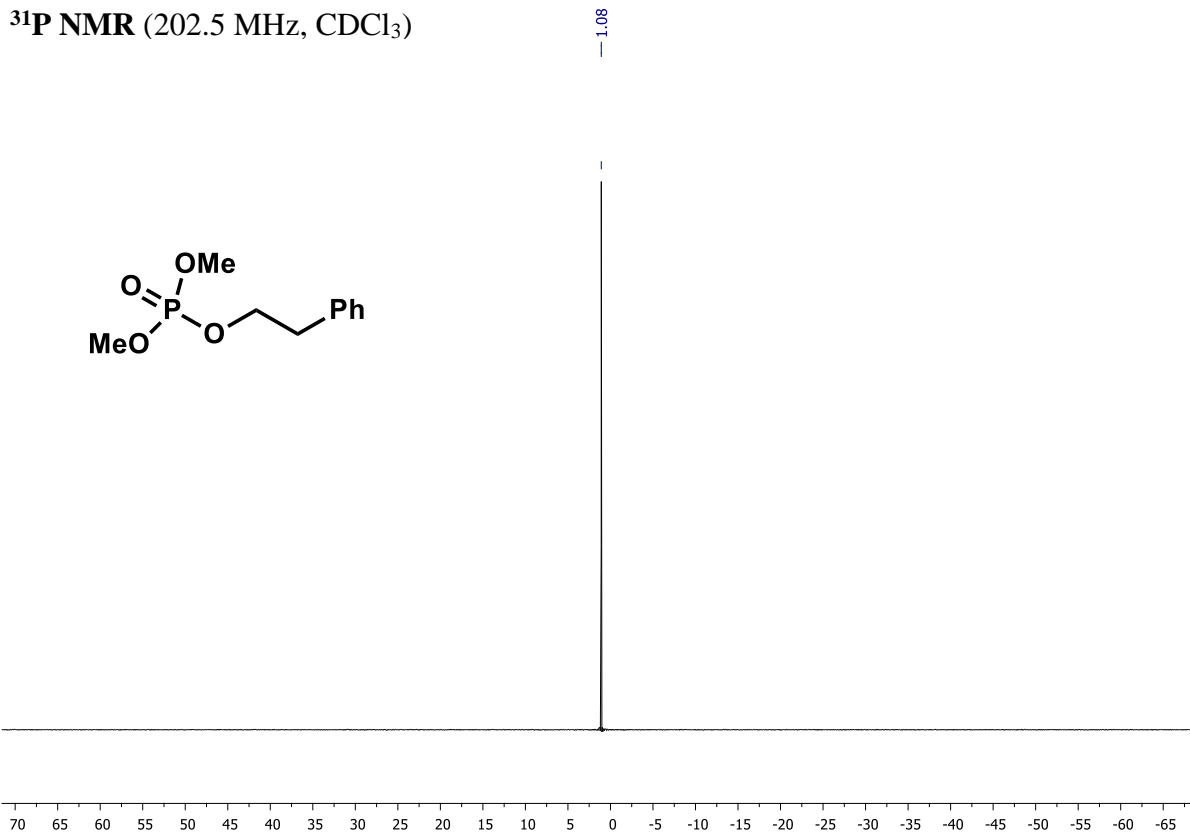
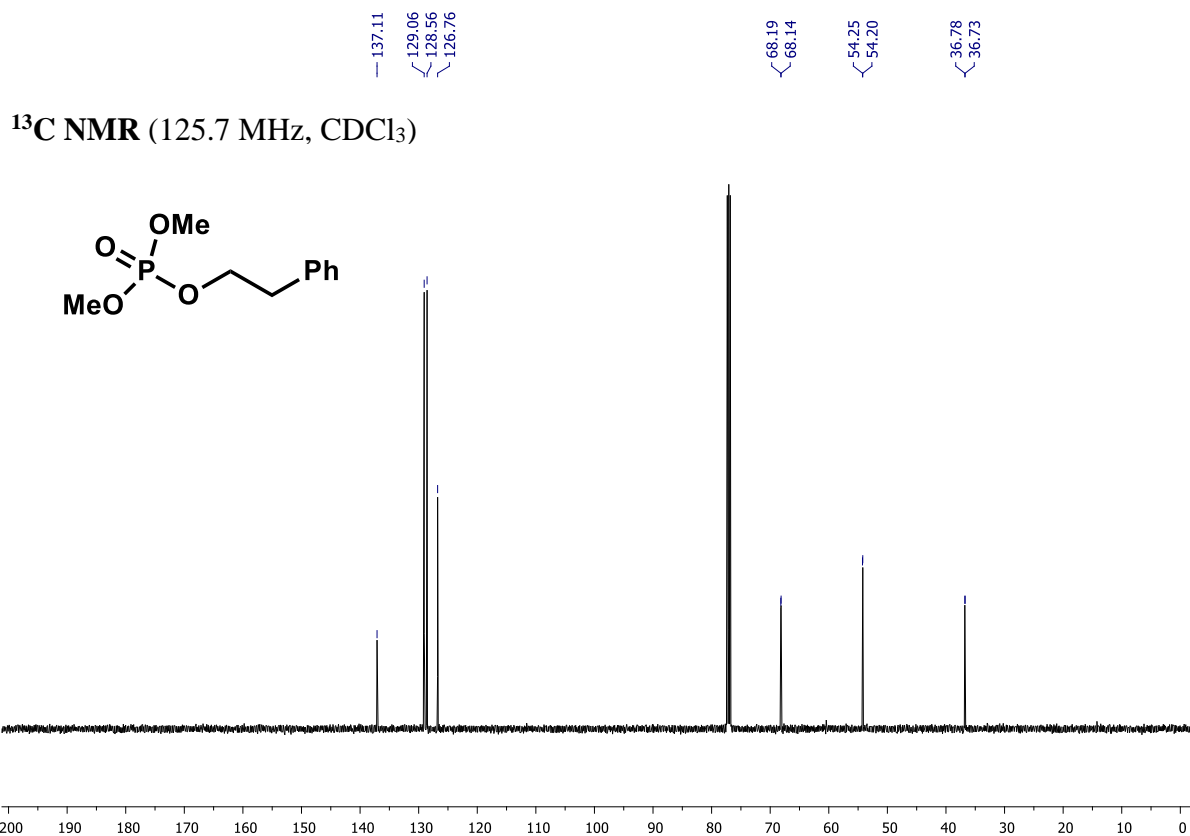


^{31}P NMR (202.5 MHz, CDCl_3)

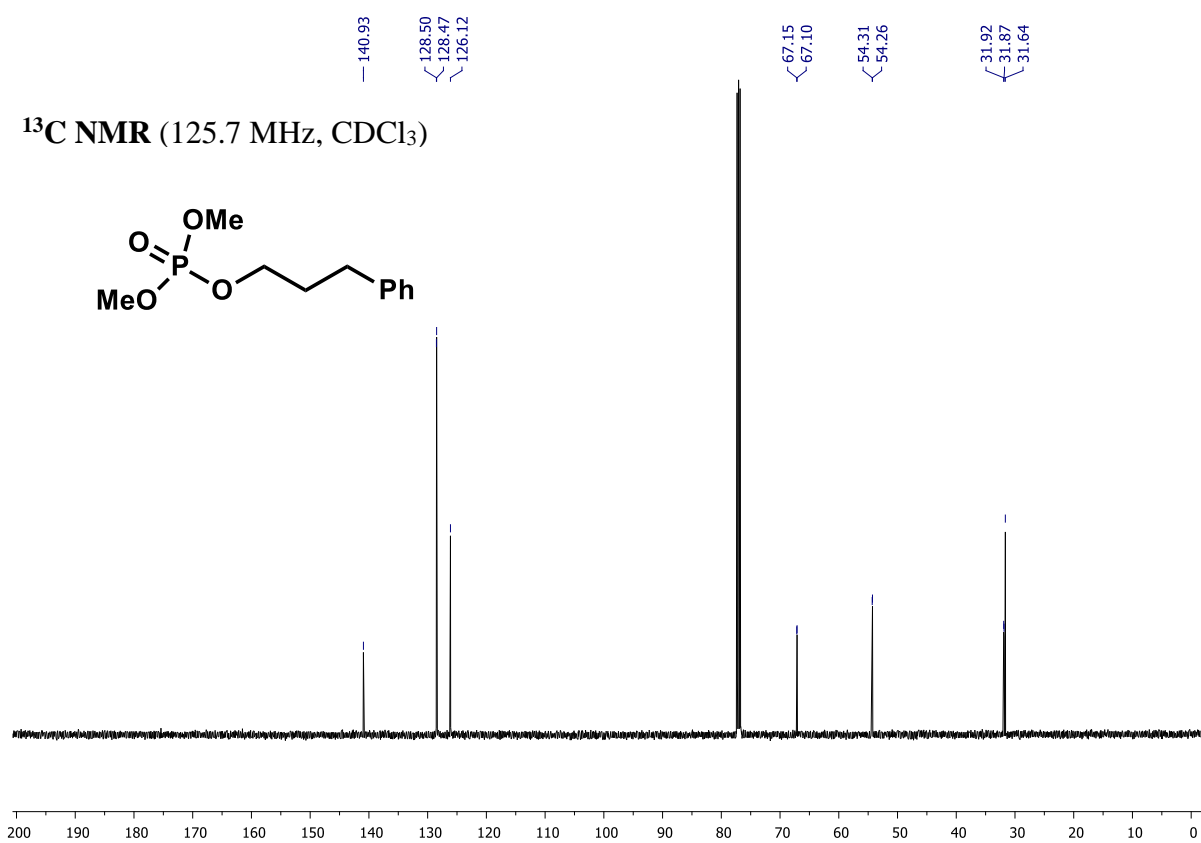
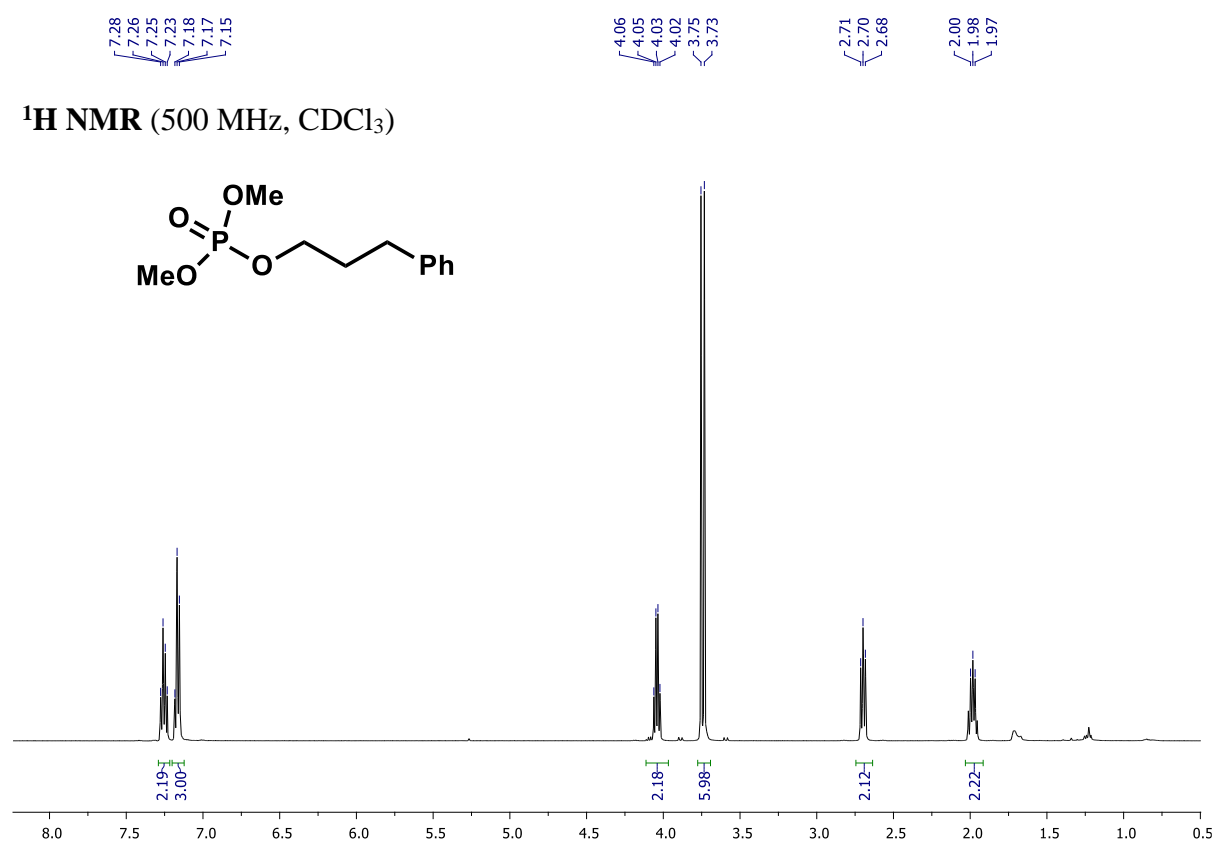


Dimethyl (3-phenylethyl) phosphate (3n)

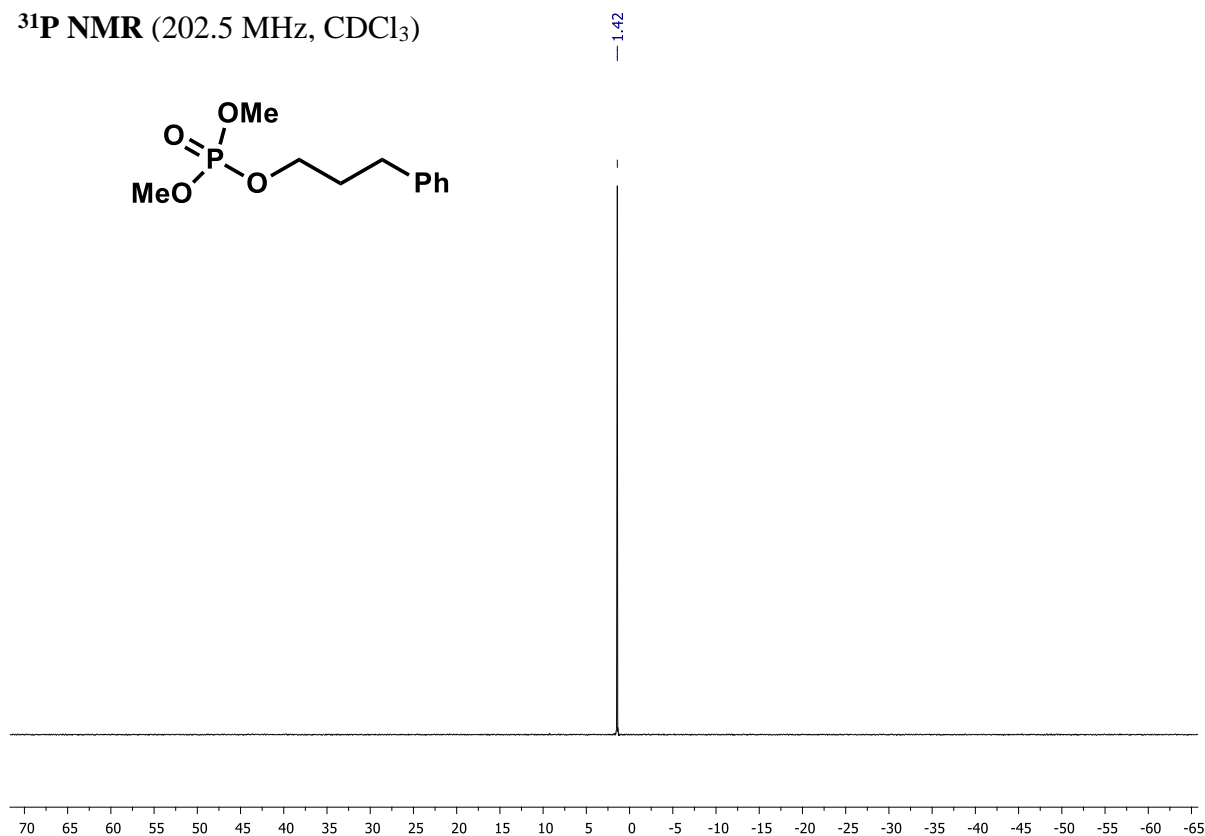
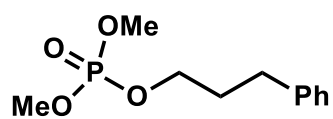




Dimethyl (3-phenylpropyl) phosphate (3o)

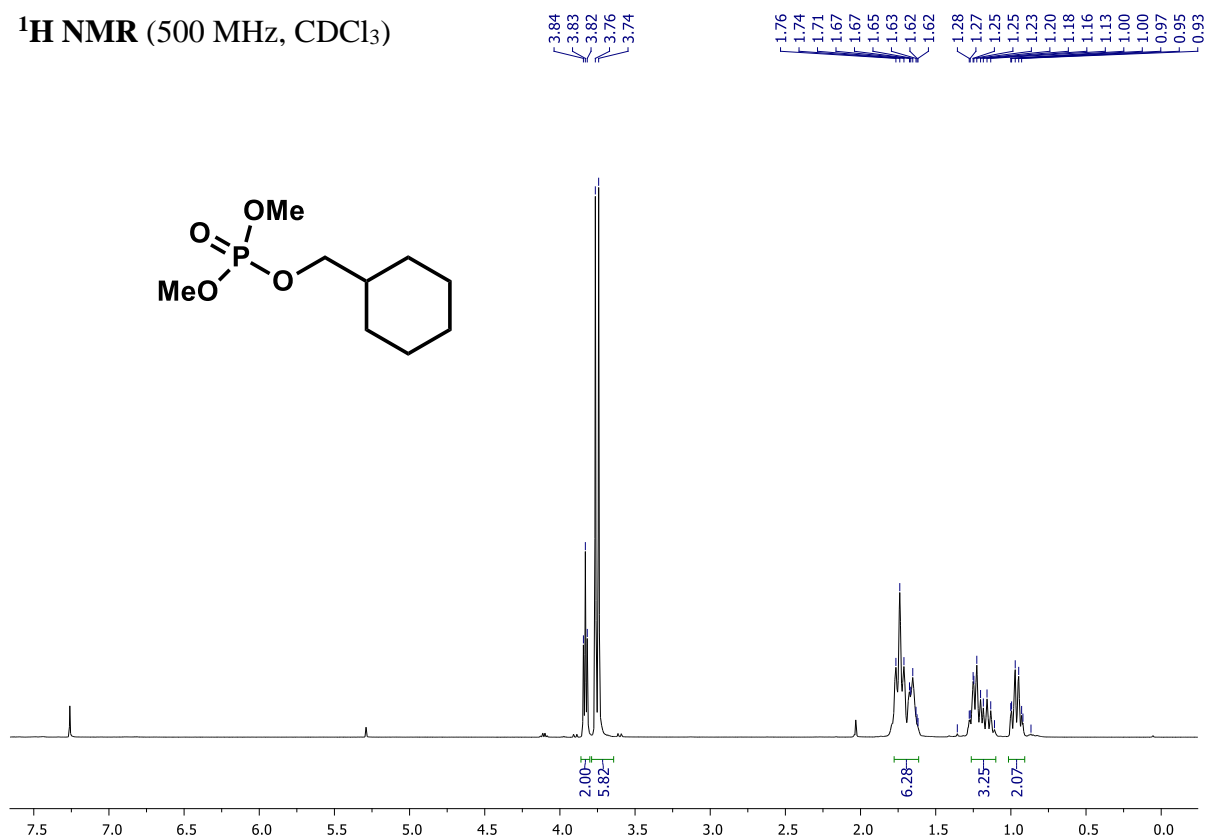
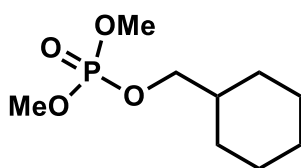


^{31}P NMR (202.5 MHz, CDCl_3)

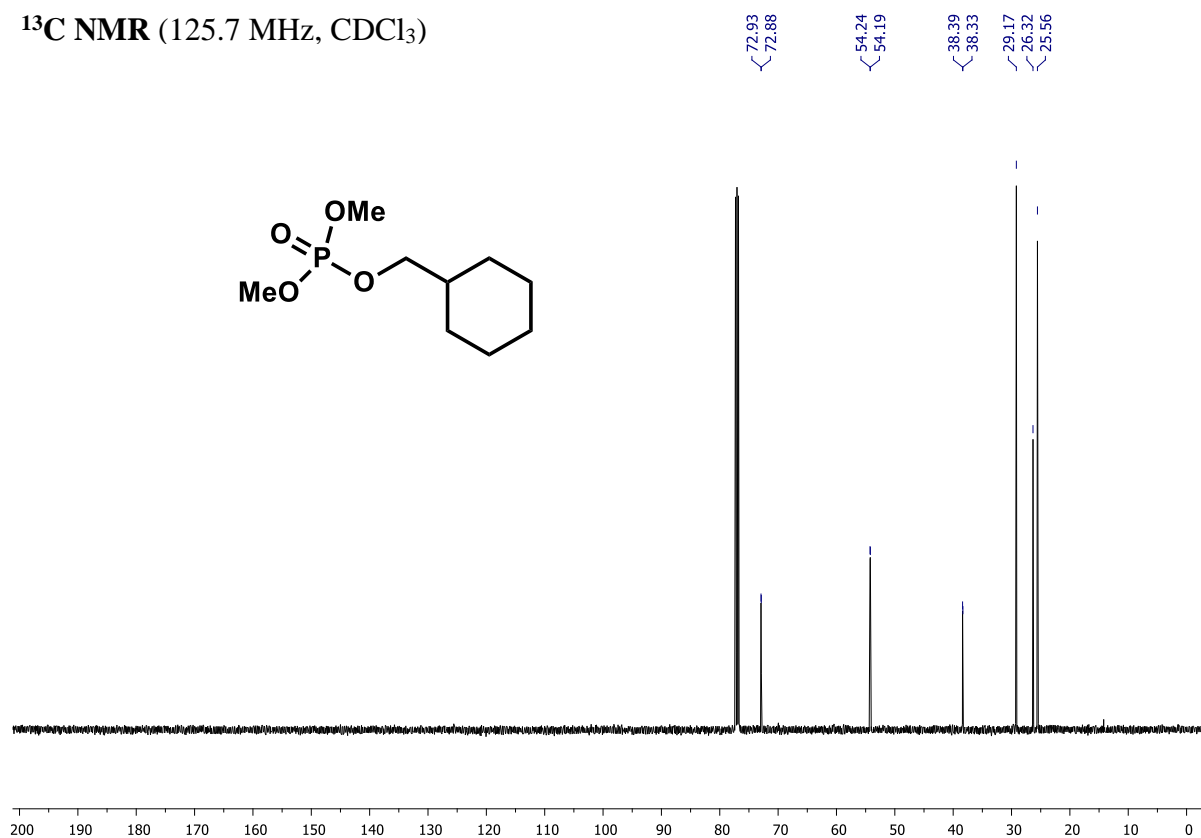


Cyclohexylmethyl dimethyl phosphate (3p)

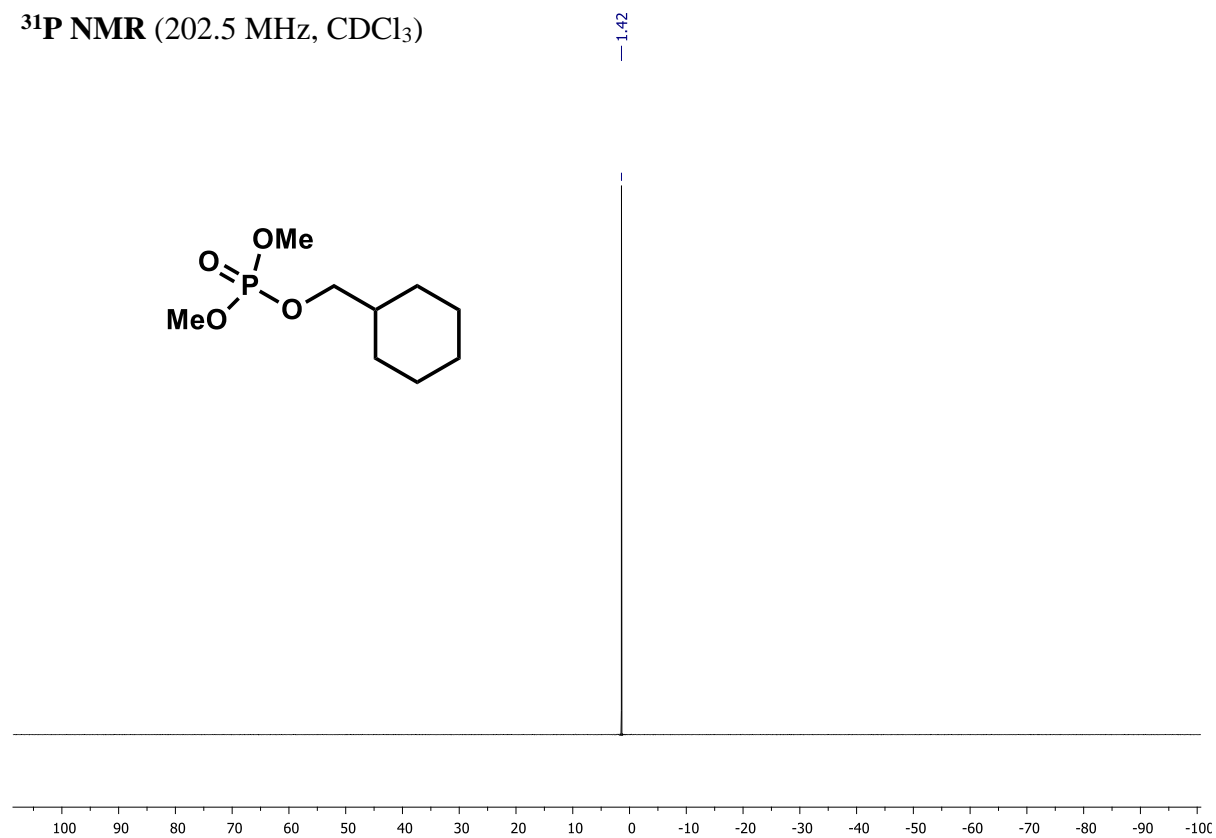
^1H NMR (500 MHz, CDCl_3)



^{13}C NMR (125.7 MHz, CDCl_3)

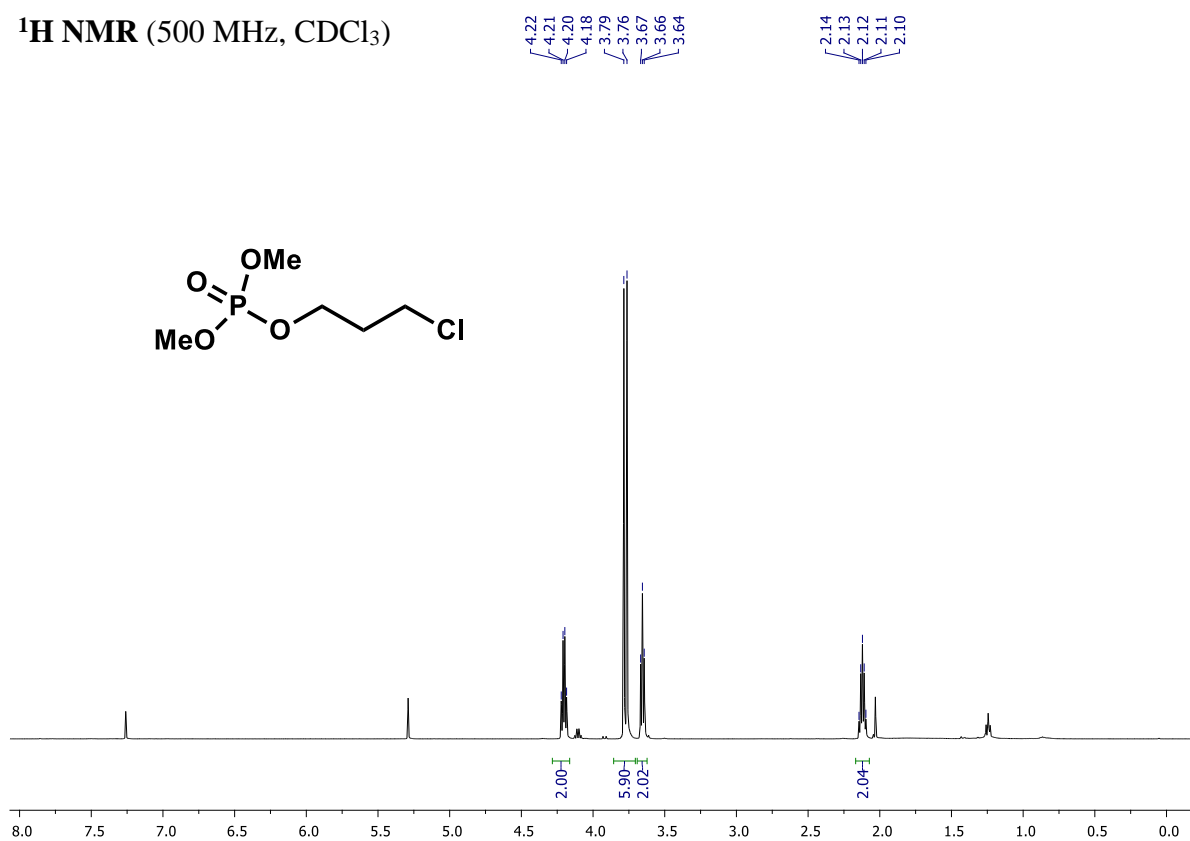


^{31}P NMR (202.5 MHz, CDCl_3)

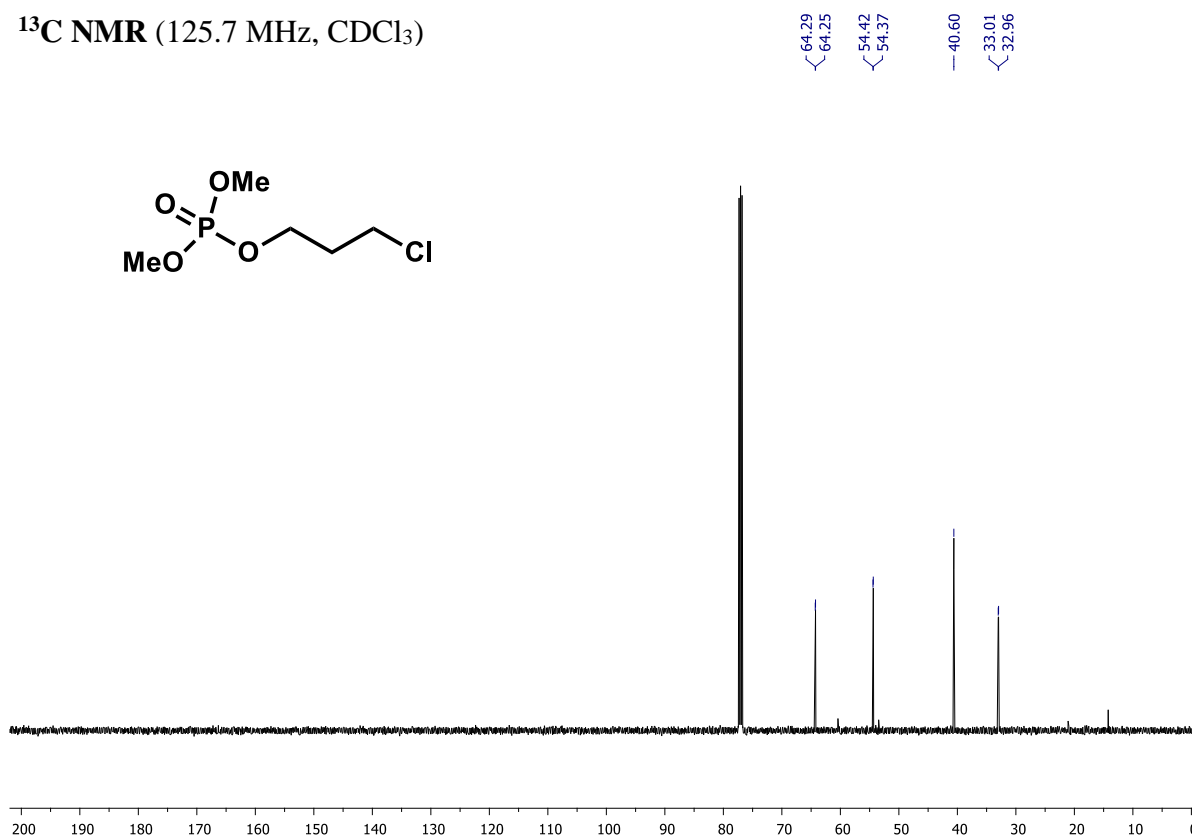


3-chloropropyl dimethyl phosphate (3q)

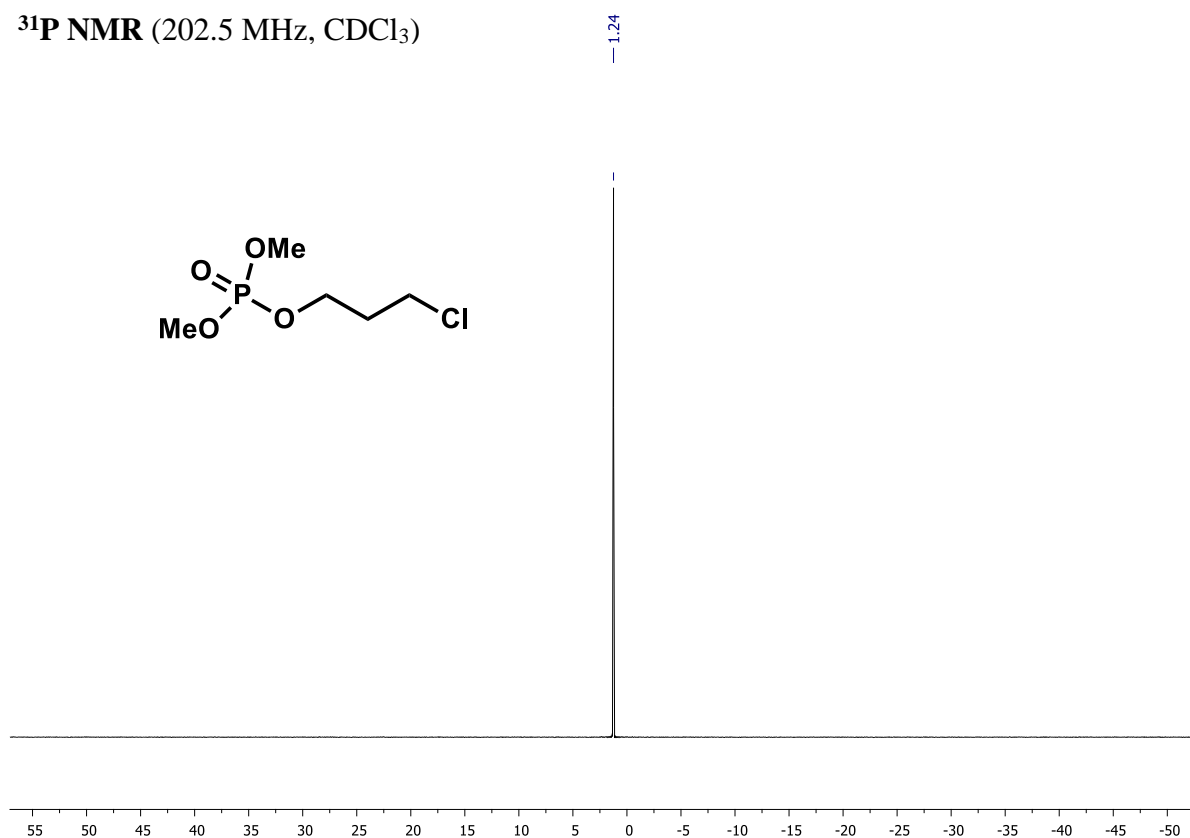
^1H NMR (500 MHz, CDCl_3)



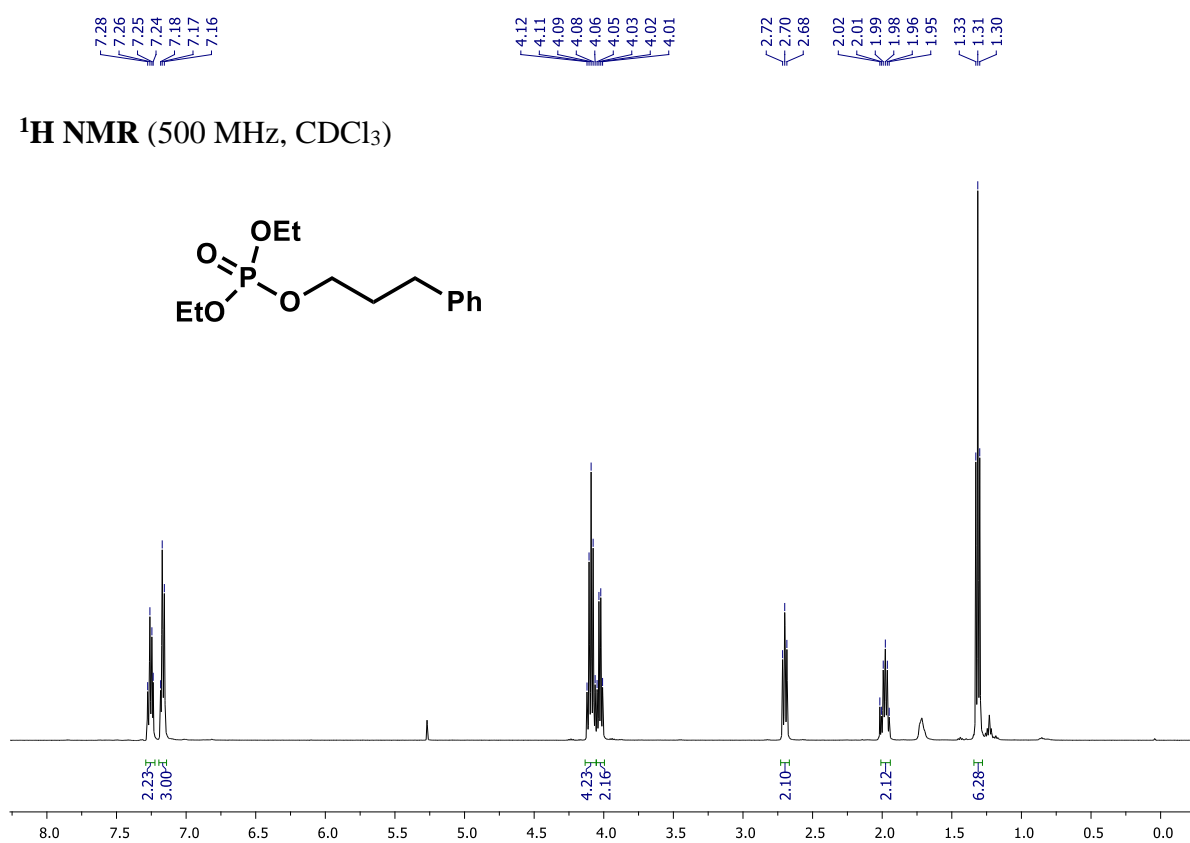
^{13}C NMR (125.7 MHz, CDCl_3)

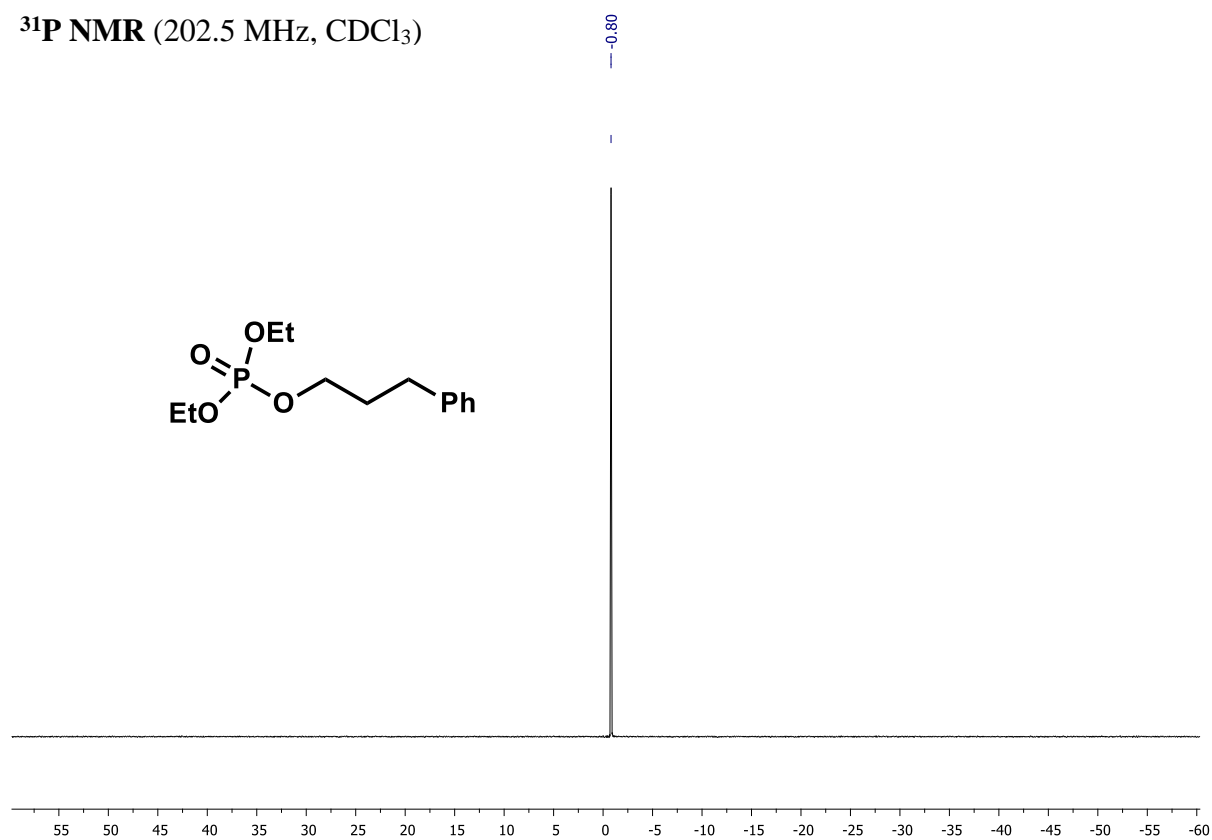
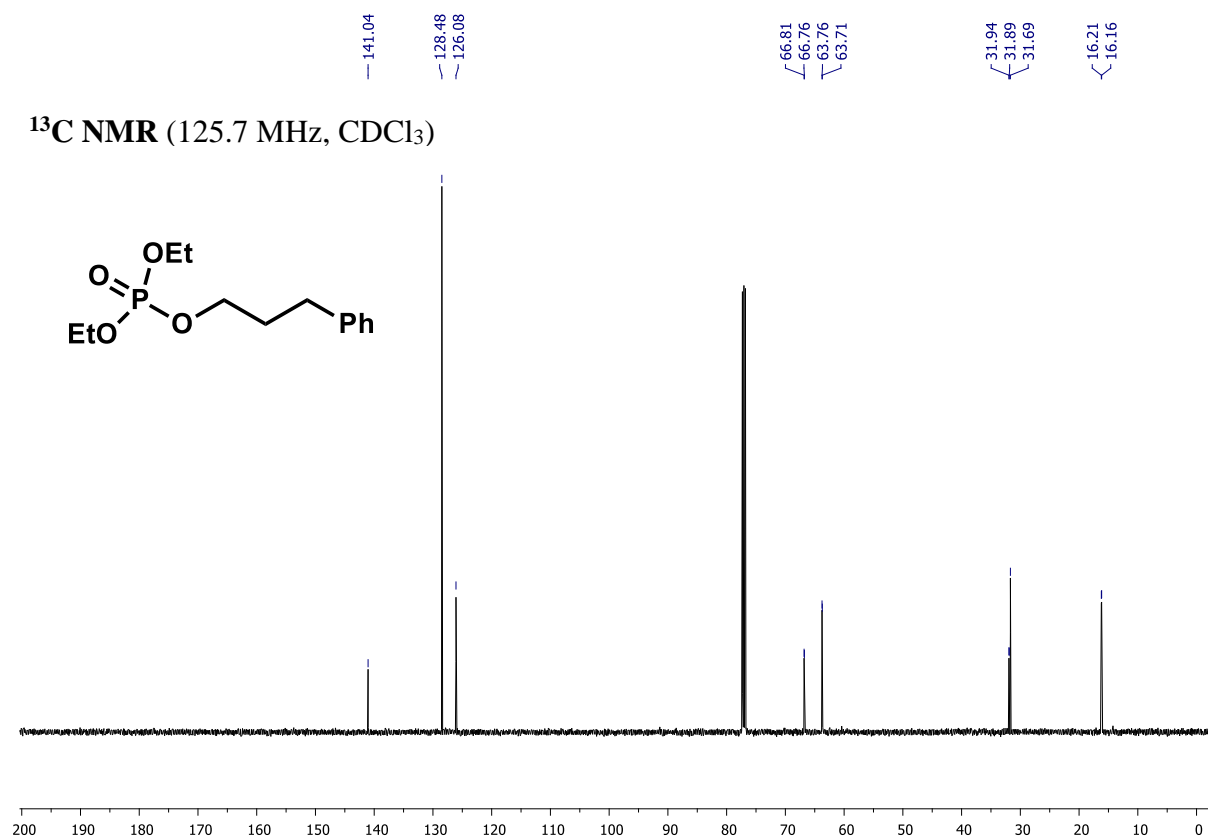


^{31}P NMR (202.5 MHz, CDCl_3)



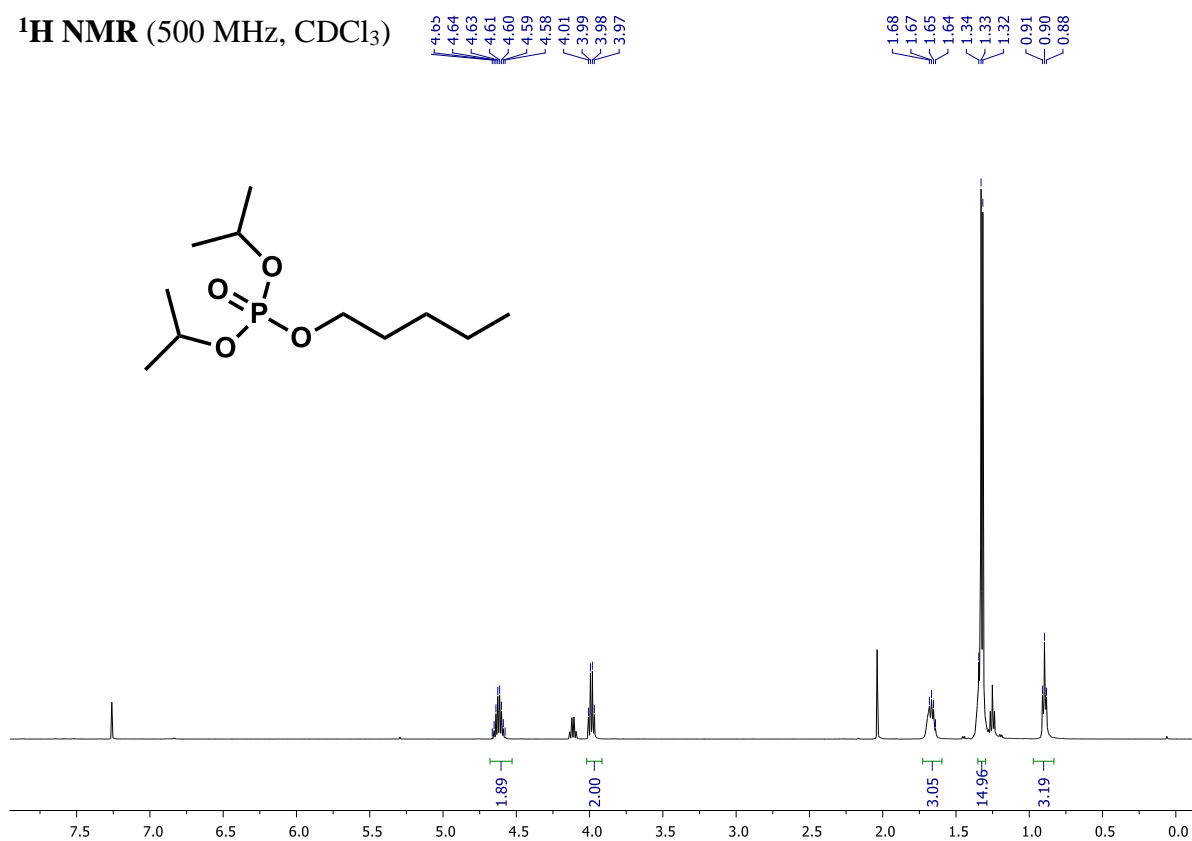
Diethyl (3-phenylpropyl) phosphate (3r)



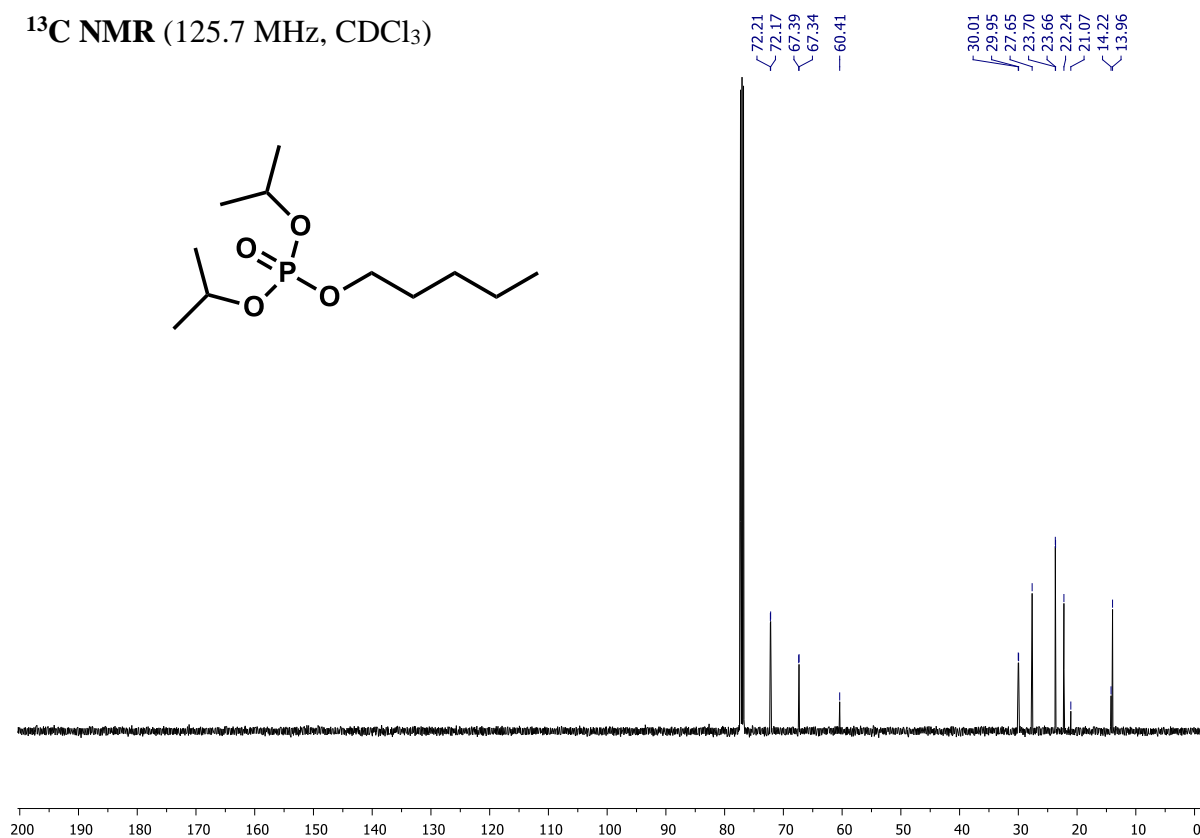


Diisopropyl pentyl phosphate (3s)

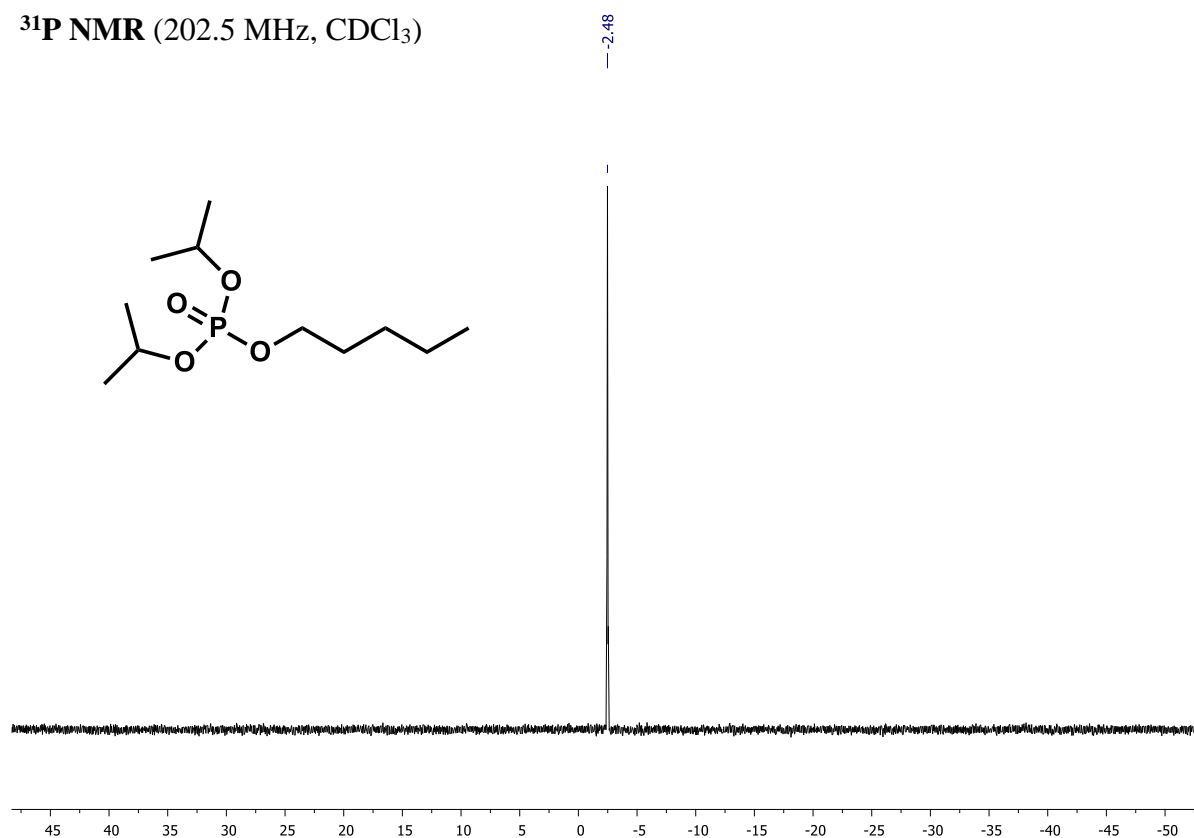
^1H NMR (500 MHz, CDCl_3)



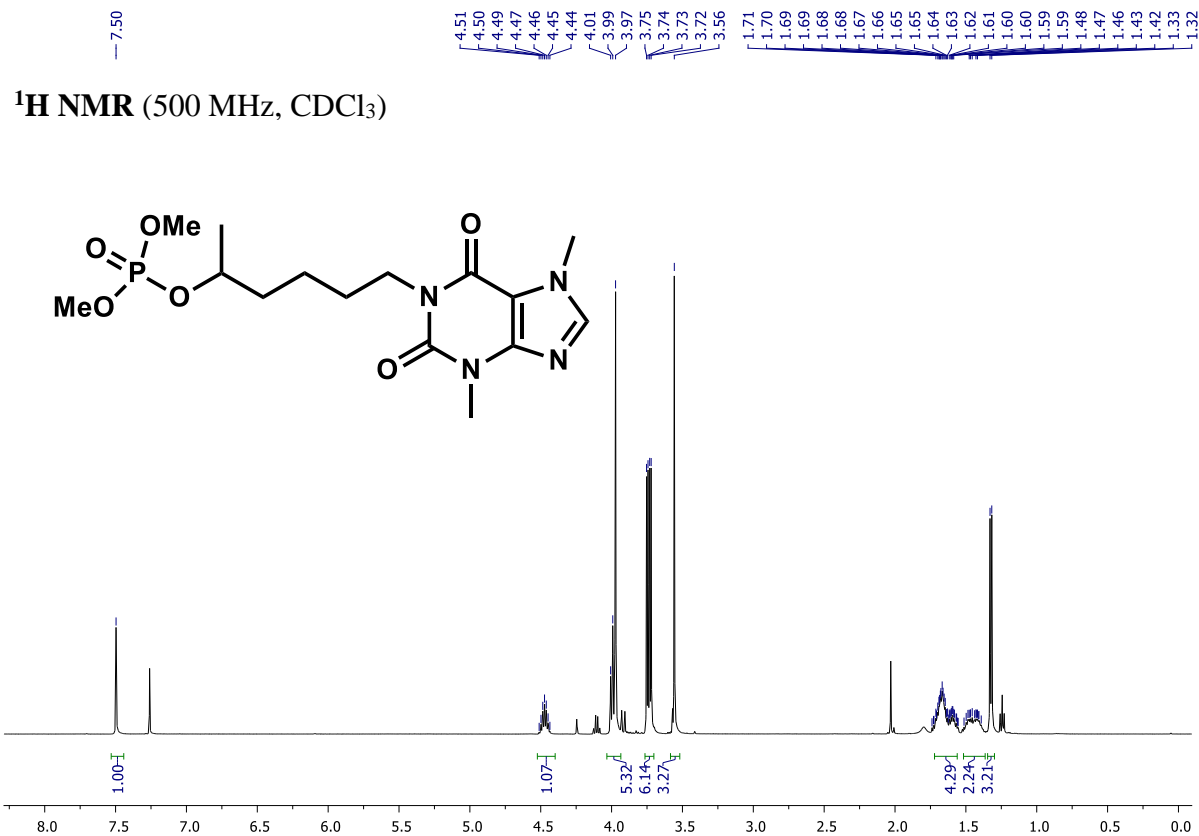
^{13}C NMR (125.7 MHz, CDCl_3)



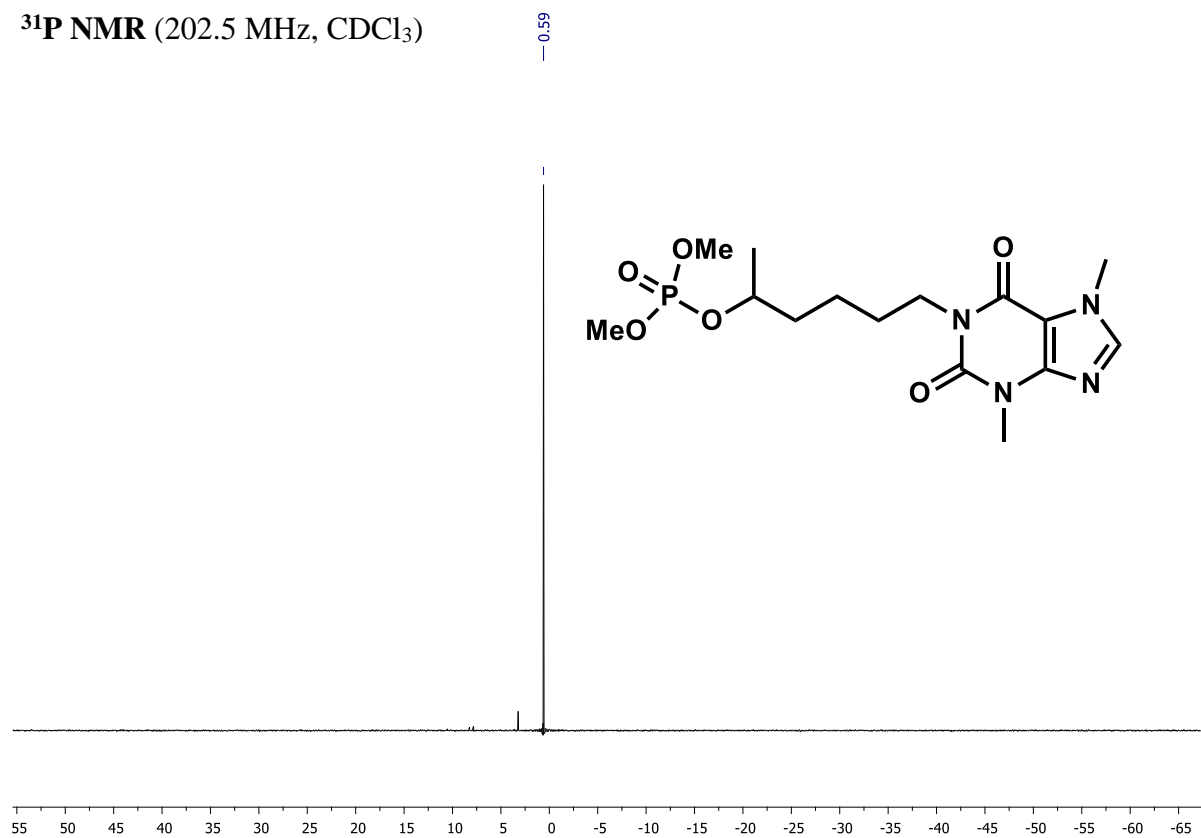
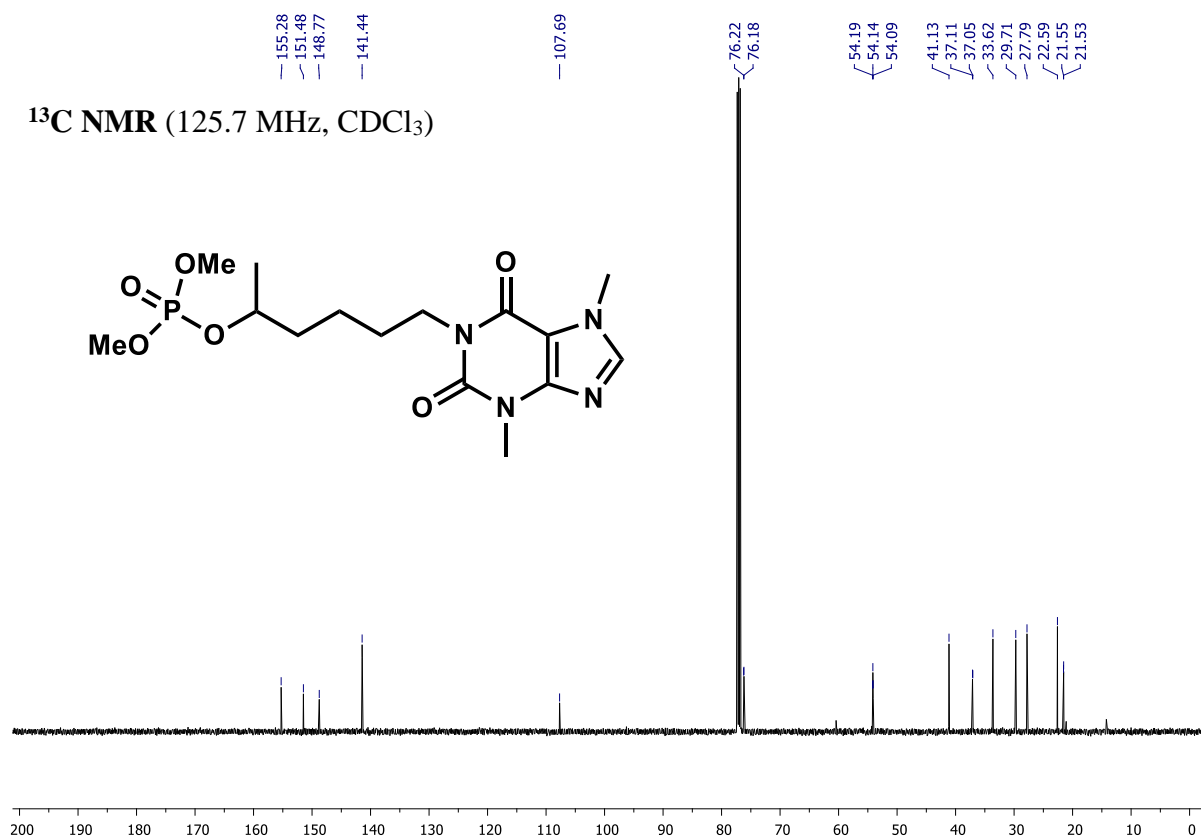
^{31}P NMR (202.5 MHz, CDCl_3)



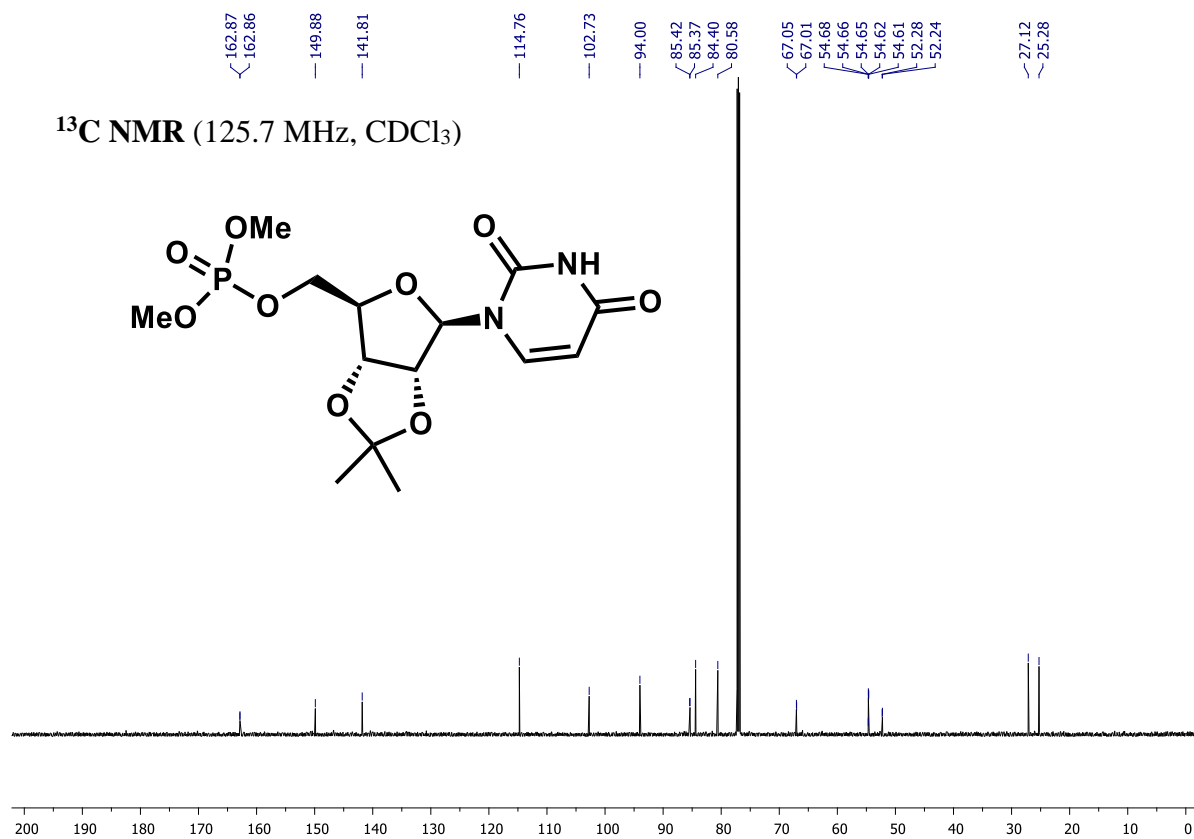
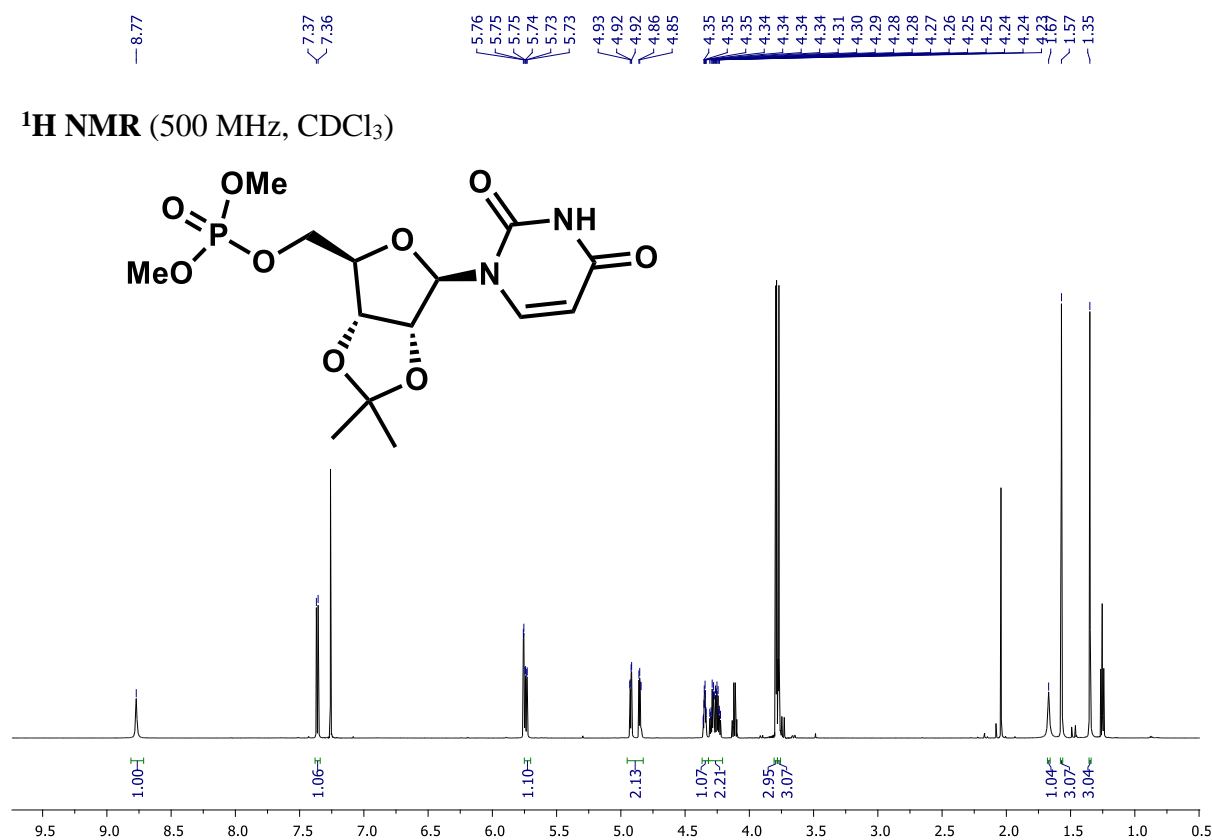
5-(3,7-dimethyl-2,6-dioxo-2,3,6,7-tetrahydro-1H-purin-1-yl)hexan-2-yl dimethyl phosphate (3t)



^1H NMR (500 MHz, CDCl_3)

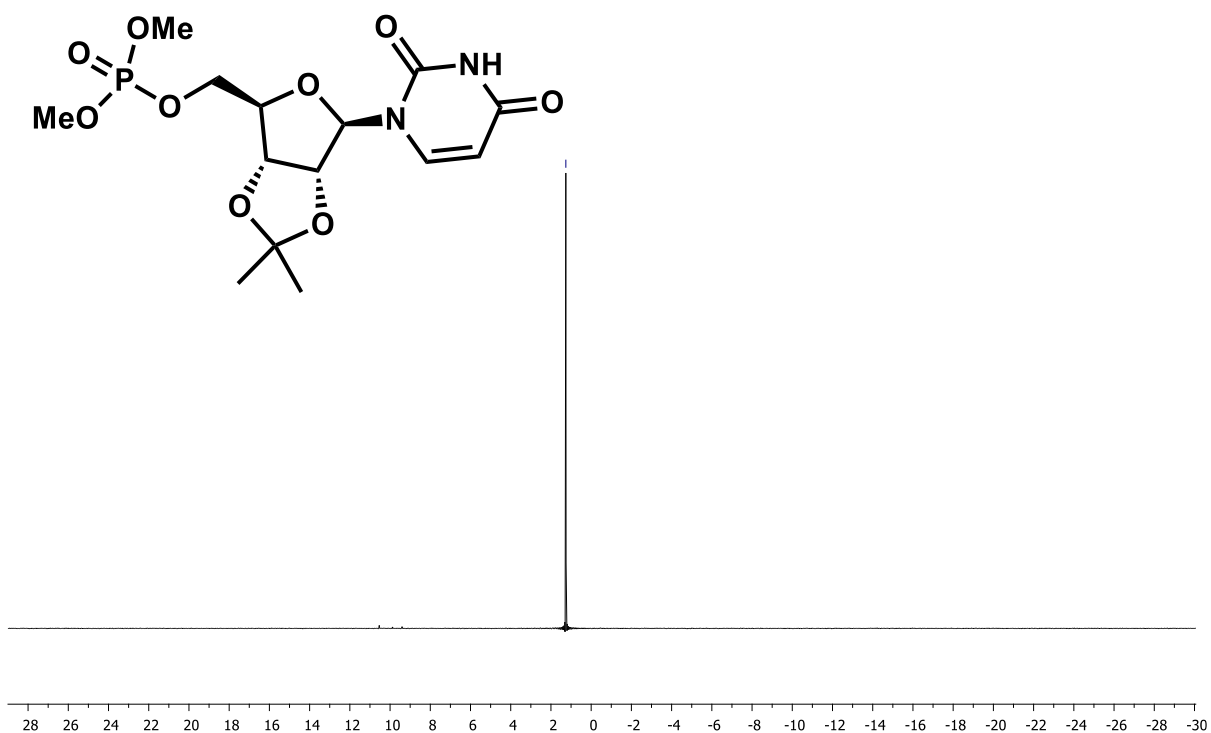


(6-(2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)-2,2-dimethyltetrahydrofuro[3,4-d][1,3]dioxol-4-yl)methyl dimethyl phosphate (3u)



³¹P NMR (202.5 MHz, CDCl₃)

— 1.26



Dimethyl pentyl phosphate (3a)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

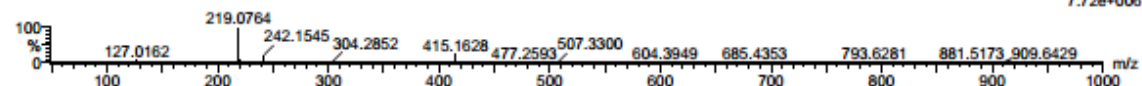
105 formula(e) evaluated with 2 results within limits (up to 10 closest results for each mass)

Elements Used:

C: 0-20 H: 0-100 O: 0-6 Na: 0-2 P: 0-1

AI_170T2_infusion 105 (2.070) Cm (103:108)

1: TOF MS ES+
7.72e+006



Minimum:				-1.5					
Maximum:		20.0	5.0	50.0					
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula	
219.0764	219.0762	0.2	0.9	5.5	2741.0	8.816	0.01	C12 H13 O Na2	
	219.0762	0.2	0.9	-0.5	2732.1	0.000	99.99	C7 H17 O4 Na P	

Dimethyl hexyl phosphate (3b)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

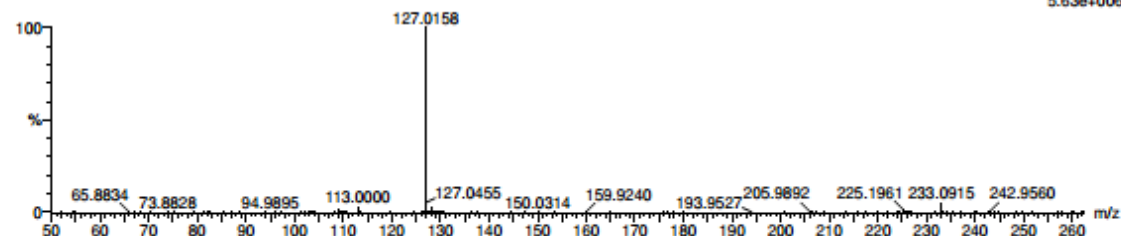
61 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-30 H: 0-100 O: 0-4 Na: 0-1 P: 0-1

1: TOF MS ES+

AI_360 241 (2.251) Cm (241:243-(222:234+264:280))
5.63e+006



Minimum:				-1.5					
Maximum:		2.0	5.0	50.0					
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula	
233.0915	233.0919	-0.4	-1.7	-0.5	82.9	n/a	n/a	C8 H19 O4 Na P	

Dimethyl heptyl phosphate (3c)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

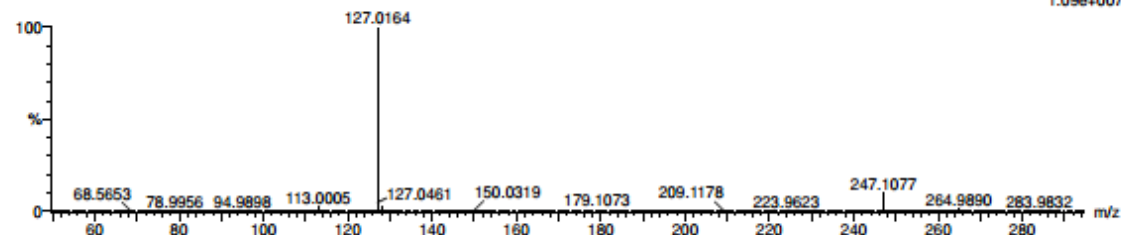
65 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-30 H: 0-100 O: 0-4 Na: 0-1 P: 0-1

1: TOF MS ES+

AI_352 259 (2.418) Cm (258:263-(247:252+289:295))
1.09e+007



Minimum:

Maximum: 2.0 5.0 -1.5

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
247.1077	247.1075	0.2	0.8	-0.5	661.4	n/a	n/a	C9 H21 O4 Na P

Dimethyl octyl phosphate (3d)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

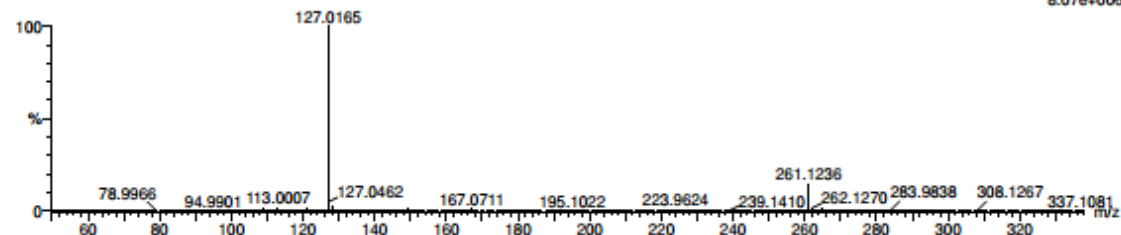
67 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-30 H: 0-100 O: 0-4 P: 0-1 Na: 0-1

1: TOF MS ES+

AI_348 278 (2.593) Cm (277:281-(267:272+303:308))
8.07e+006



Minimum:

Maximum: 2.0 5.0 -1.5

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
261.1236	261.1232	0.4	1.5	-0.5	914.5	n/a	n/a	C10 H23 O4 P Na

Dimethyl nonyl phosphate (3e)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

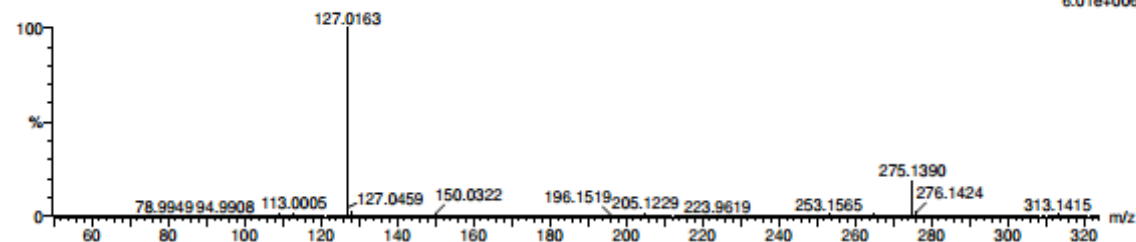
73 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-30 H: 0-100 O: 0-4 P: 0-1 Na: 0-1

1: TOF MS ES+

Al_347 296 (2.760) Cm (295:300-(287:291+325:331))
6.01e+006



Minimum: -1.5
Maximum: 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
275.1390	275.1388	0.2	0.7	-0.5	847.7	n/a	n/a	C11 H25 O4 P Na

Dimethyl hexyl phosphate (3f)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

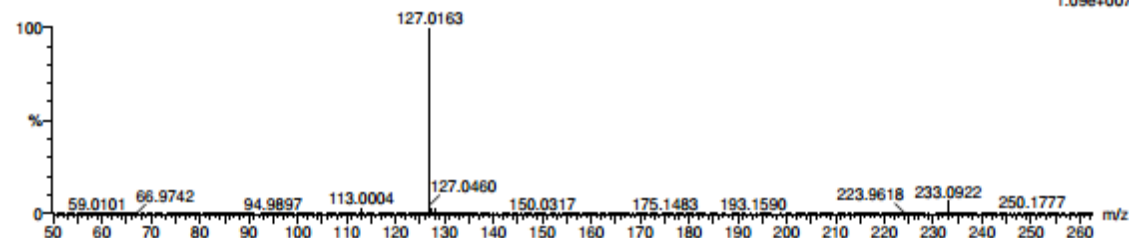
95 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-25 H: 0-100 O: 0-8 P: 0-1 Na: 0-1

1: TOF MS ES+

Al_366 235 (2.198) Cm (235:240-(226:232+261:270))
1.09e+007



Minimum: -1.5
Maximum: 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
233.0922	233.0919	0.3	1.3	-0.5	476.7	n/a	n/a	C8 H19 O4 P Na

Dimethyl (4-phenylbutyl) phosphate (3g)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

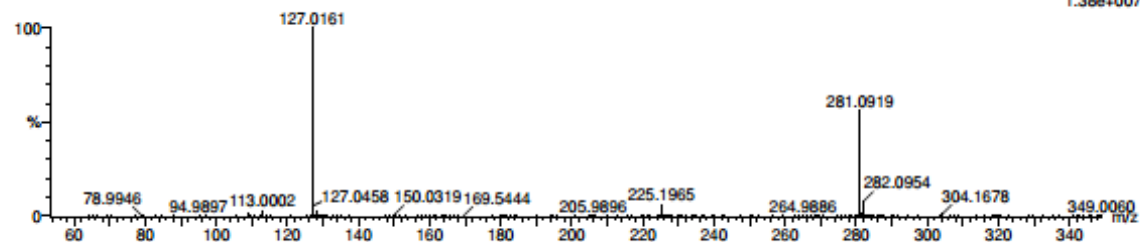
85 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-15 H: 0-100 O: 0-4 Na: 0-1 P: 0-3

1: TOF MS ES+

AI_380 247 (2.304) Cm (246:254-(221:240+283:311))
1.38e+007



Minimum: -1.5
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
281.0919	281.0919	0.0	0.0	3.5	539.6	n/a	n/a	C12 H19 O4 Na P

Dimethyl (4-phenylbutan-2-yl) phosphate (3h)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

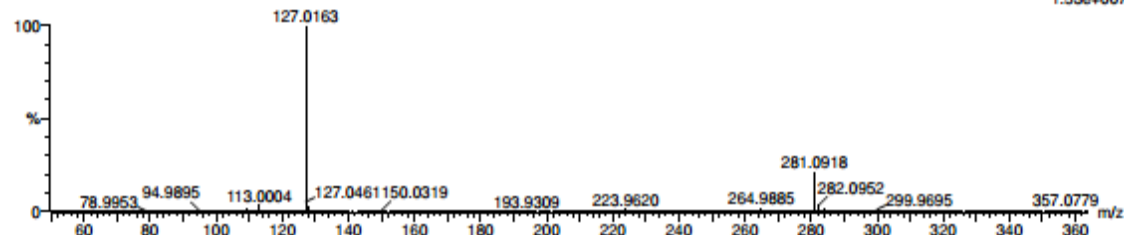
30 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-15 H: 0-100 O: 0-4 Na: 0-1 P: 0-1

1: TOF MS ES+

AI_388 245 (2.286) Cm (243:248-(234:239+273:279))
1.53e+007



Minimum: -1.5
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
281.0918	281.0919	-0.1	-0.4	3.5	1015.5	n/a	n/a	C12 H19 O4 Na P

Dimethyl pent-4-en-1-yl phosphate (3i)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

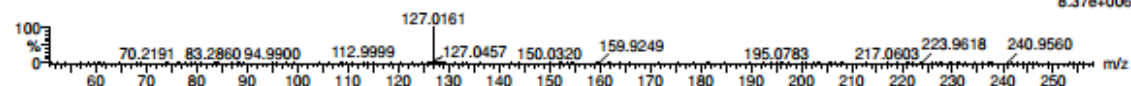
99 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)

Elements Used:

C: 0-30 H: 0-100 O: 0-10 P: 0-1 Na: 0-1

AI_362 201 (1.882) Cm (199:204-(182:196+228:241))

1: TOF MS ES+
8.37e+006



Minimum: -1.5
Maximum: 20.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
217.0603	217.0606	-0.3	-1.4	0.5	131.3	n/a	n/a	C7 H15 O4 P Na

Dimethyl pent-4-yn-1-yl phosphate (3j)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

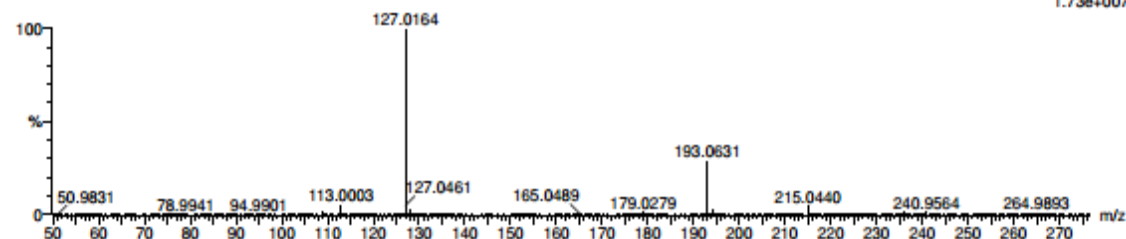
73 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-10 H: 0-100 O: 0-4 Na: 0-1 P: 0-3

1: TOF MS ES+

AI_379 176 (1.645) Cm (175:180-(162:169+225:236))
1.73e+007



Minimum: -1.5
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
193.0631	193.0630	0.1	0.5	1.5	803.7	n/a	n/a	C7 H14 O4 P

Isopropyl dimethyl phosphate (3k)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

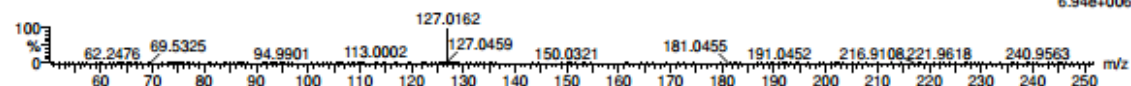
68 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)

Elements Used:

C: 0-20 H: 0-100 O: 0-6 P: 0-1 Na: 0-1

AI_423 166 (1.557) Cm (166:168-(153:159+186:192))

1: TOF MS ES+
6.94e+006



Minimum: -1.5
Maximum: 20.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
191.0452	191.0449	0.3	1.6	-0.5	77.7	n/a	n/a	C5 H13 O4 P Na

Cyclopentylmethyl dimethyl phosphate (3l)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

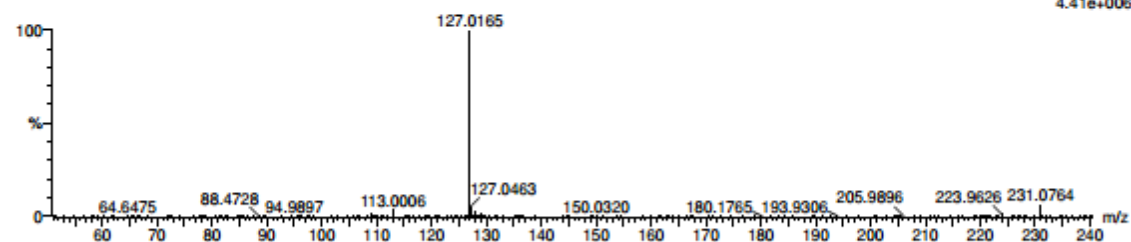
97 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-25 H: 0-100 O: 0-8 P: 0-1 Na: 0-1

1: TOF MS ES+

AI_365 219 (2.049) Cm (218:219-(209:215+238:246))
4.41e+006



Minimum: -1.5
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
231.0764	231.0762	0.2	0.9	0.5	141.5	n/a	n/a	C8 H17 O4 P Na

Cyclobutylmethyl dimethyl phosphate (3m)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

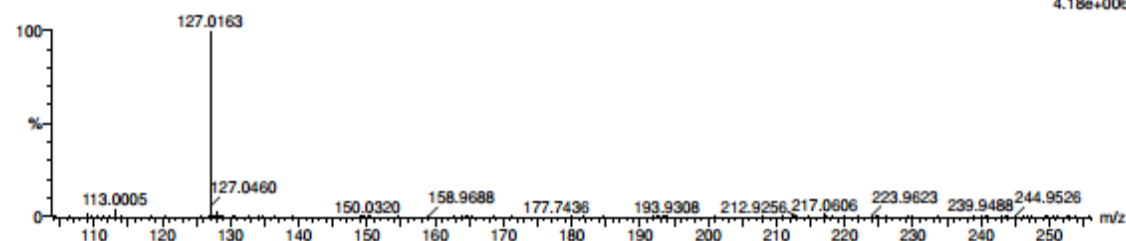
56 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-30 H: 0-100 O: 0-4 P: 0-1 Na: 0-1

1: TOF MS ES+

AI_385 206 (1.926) Cm (206:208-(181:188+248:261))
4.18e+006



Minimum: -1.5
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
217.0606	217.0606	0.0	0.0	0.5	82.1	n/a	n/a	C7 H15 O4 P Na

Dimethyl (3-phenylethyl) phosphate (3n)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

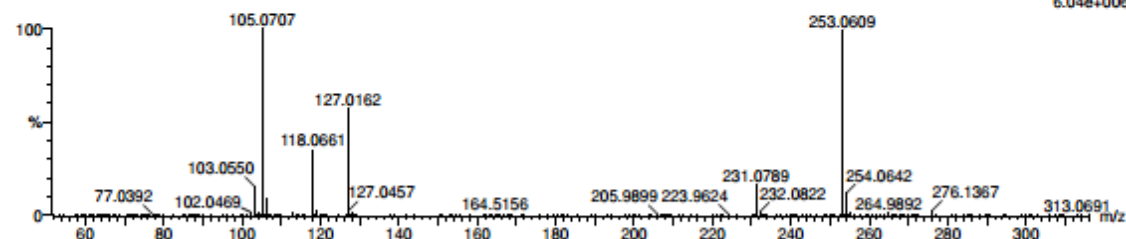
43 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-15 H: 0-100 O: 0-4 P: 0-1 Na: 0-1

1: TOF MS ES+

AI_381 216 (2.014) Cm (215:219-(204:210+243:262))
6.04e+006



Minimum: -1.5
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
253.0609	253.0606	0.3	1.2	3.5	391.9	n/a	n/a	C10 H15 O4 P Na

Dimethyl (3-phenylpropyl) phosphate (3o)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

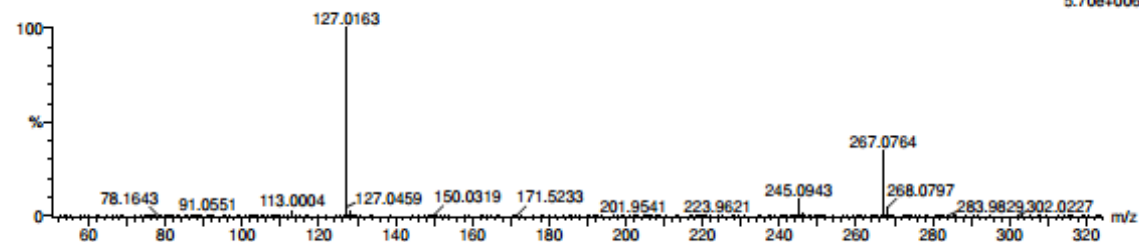
70 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-30 H: 0-100 O: 0-4 Na: 0-1 P: 0-1

1: TOF MS ES+

AI_344 232 (2.163) Cm (231:236-(214:226+253:264))
5.70e+006



Minimum: -1.5
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
267.0764	267.0762	0.2	0.7	3.5	313.7	n/a	n/a	C11 H17 O4 Na P

Cyclohexylmethyl dimethyl phosphate (3p)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

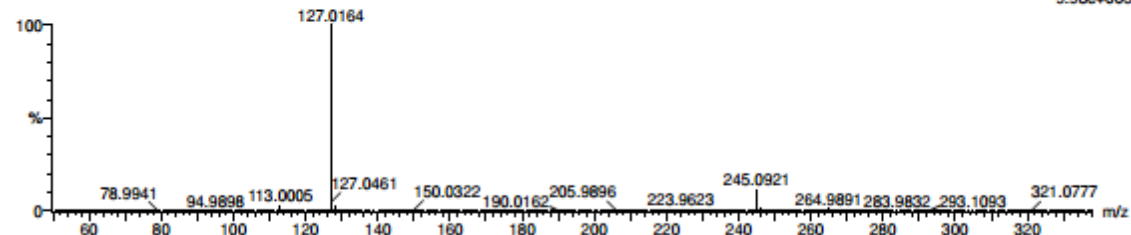
63 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-30 H: 0-100 O: 0-4 Na: 0-1 P: 0-1

1: TOF MS ES+

AI_351 238 (2.224) Cm (237:242-(226:232+263:267))
9.98e+006



Minimum: -1.5
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
245.0921	245.0919	0.2	0.8	0.5	726.8	n/a	n/a	C9 H19 O4 Na P

3-chloropropyl dimethyl phosphate (3q)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

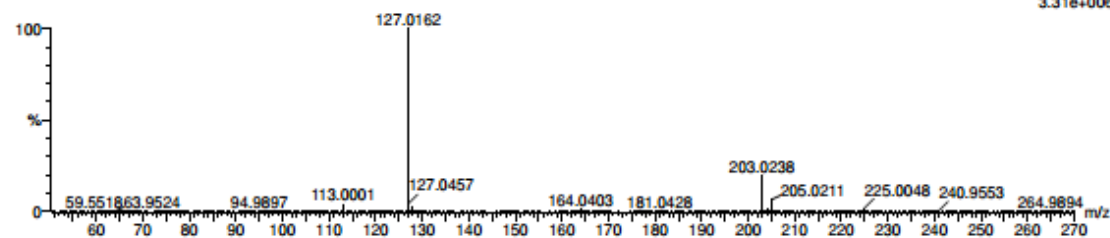
82 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-25 H: 0-100 O: 0-8 ³⁵Cl: 0-1 P: 0-1

1: TOF MS ES+

Al_372 177 (1.654) Cm (176:179-(168:173+189:194))
3.31e+006



Minimum: -1.5
Maximum: 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
203.0238	203.0240	-0.2	-1.0	-0.5	442.0	n/a	n/a	C5 H13 O4 ³⁵ Cl P

Diethyl (3-phenylpropyl) phosphate (3r)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

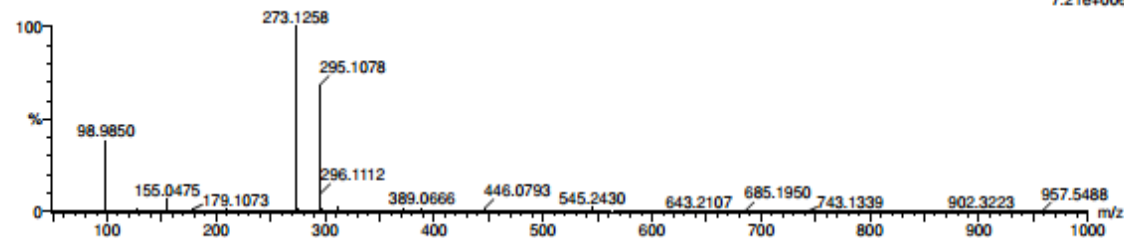
76 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-30 H: 0-100 O: 0-4 Na: 0-1 P: 0-1

1: TOF MS ES+

Al_353 259 (2.418) Cm (258:263-(246:252+282:288))
7.21e+006



Minimum: -1.5
Maximum: 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
295.1078	295.1075	0.3	1.0	3.5	1009.0	n/a	n/a	C13 H21 O4 Na P

Diisopropyl pentyl phosphate (3s)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

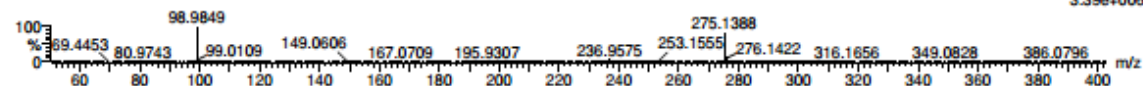
134 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)

Elements Used:

C: 0-30 H: 0-100 O: 0-10 P: 0-1 Na: 0-1

AI_359 277 (2.585) Cm (276:280-(261:269+300:306))

1: TOF MS ES+
3.39e+006



Minimum: -1.5
Maximum: 20.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
275.1388	275.1388	0.0	0.0	-0.5	784.7	n/a	n/a	C11 H25 O4 P Na

5-(3,7-dimethyl-2,6-dioxo-2,3,6,7-tetrahydro-1H-purin-1-yl)hexan-2-yl dimethyl phosphate (3t)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

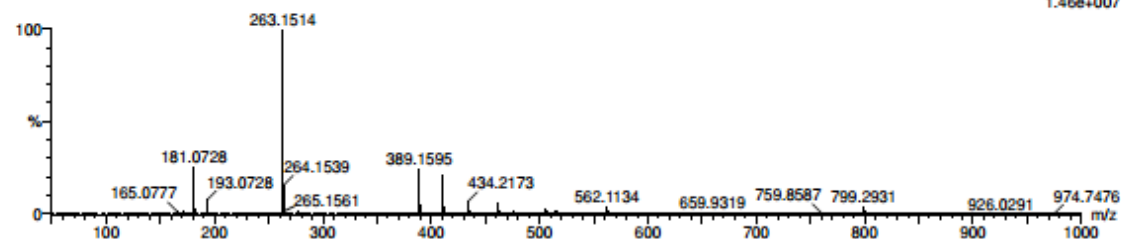
219 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-20 H: 0-100 N: 0-4 O: 0-6 P: 0-1 Na: 0-1

1: TOF MS ES+

AI_418 187 (1.750) Cm (186:190-(175:182+207:216))
1.46e+007



Minimum: -1.5
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
411.1414	411.1409	0.5	1.2	5.5	455.5	0.087	91.68	C15 H25 N4 O6 P Na
	411.1433	-1.9	-4.6	8.5	457.9	2.487	8.32	C17 H24 N4 O6 P

(6-(2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)-2,2-dimethyltetrahydrofuro[3,4-d][1,3]dioxol-4-yl)methyl dimethyl phosphate (3u)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

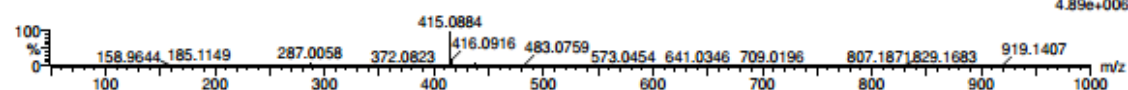
305 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)

Elements Used:

C: 0-20 H: 0-100 N: 0-3 O: 0-9 P: 0-1 Na: 0-1

AI_529_INFUSION 42 (0.846) Cm (36:44)

1: TOF MS ES+
4.89e+006



Minimum: -1.5
Maximum: 20.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
415.0884	415.0882	0.2	0.5	5.5	2216.1	n/a	n/a	C14 H21 N2 O9 P Na