# Electronic Supplementary Information

**for**

# Synthesis of δ-phosphorothiolated alcohols by photoredox/copper catalyzed remote C($sp^3$)−H phosphorothiolation of N-alkoxypyridinium salts

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[†] *Z. Zheng and S. Shi contributed equally to this work.*

## Compound list

| | | | | | |
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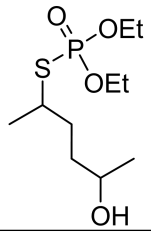
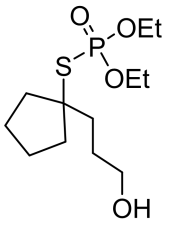
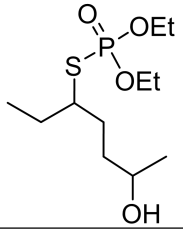
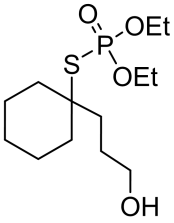
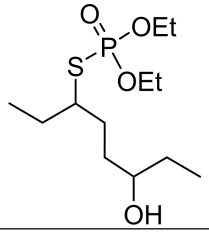
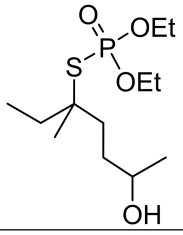
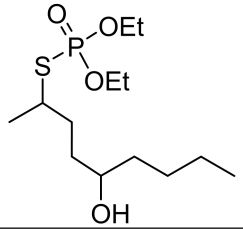
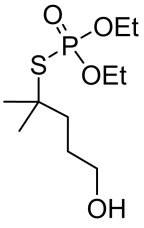
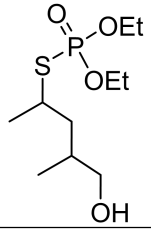
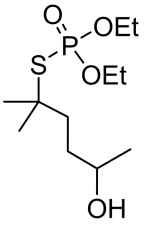
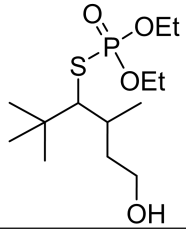
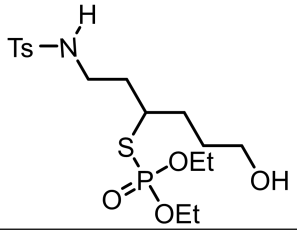
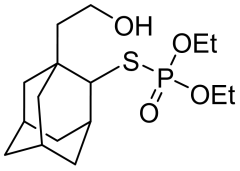
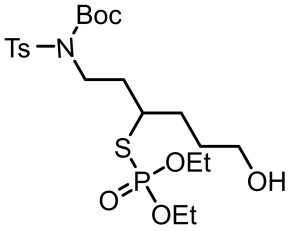
| **3o** |  | 44 | **3v** |  | 54 |
| **3p** |  | 45 | **3w** |  | 55 |
| **3q** |  | 47 | **3x** |  | 57 |
| **3r** |  | 48 | **3y** |  | 59 |
| **3s** |  | 50 | **3z** |  | 60 |
| **3t** |  | 51 | **3ac** |  | 62 |
| **3u** |  | 53 | **3ad** |  | 63 |

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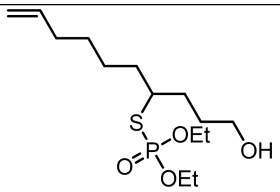
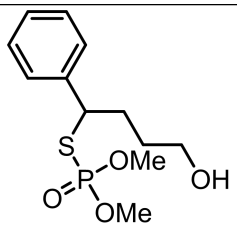
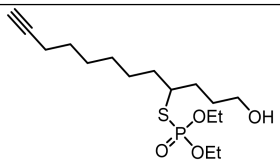
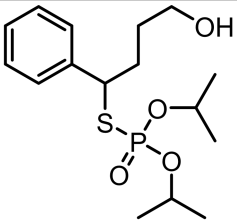
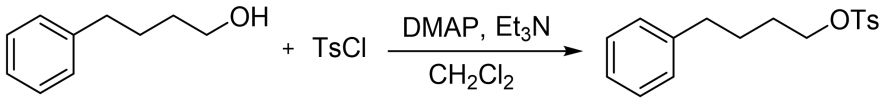
| **3ae** |  | 64 | **3ag** |  | 67 |
| **3af** |  | 65 | **3ah** |  | 68 |

## General Information:

$^{1}H$, $^{13}C$ and $^{31}P$ NMR spectra were recorded on a Bruker Av600 spectrometer using tetramethylsilane (TMS) in $CDCl_3$ as the internal standard for $^{1}H$, and $^{13}C$ NMR ($^{1}H$ NMR: TMS at 0.00 ppm, $CHCl_3$ at 7.26 ppm; $^{13}C$ NMR: $CDCl_3$ at 77.16 ppm) and 85% $H_3PO_4$ as external standard for $^{31}P$ NMR. Data are represented as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants in Hertz (Hz), integration. The products were purified by Column chromatography on silica gel 300 − 400 mesh. All products were firstly examined by Bruker AmaZon SL ESI-IT-MS (Bruker Daltonics Inc., Germany) in positive ion mode, then further characterized by HRMS (ESI-qTOF MS, Bruker micrOTOF-Q II) in positive ion mode too.

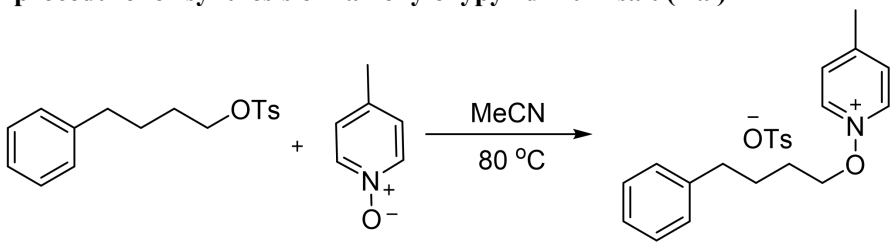
## 1. Synthesis of substrates.

**Experimental procedure for synthesis of alkyl tosylate.**



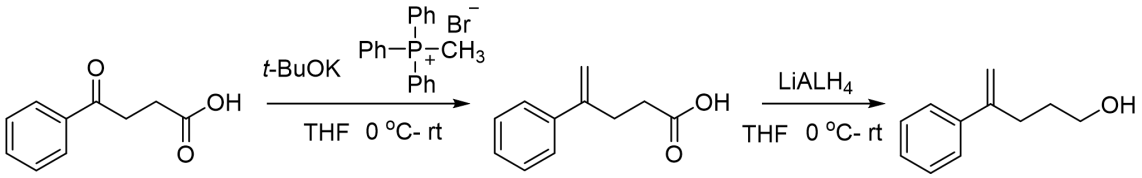

To a solution of TsCl (18.0 mmol) in $CH_2Cl_2$ was added appropriate 4-phenylbutan-1-ol (16.6 mmol). After added DMAP (101.0 mg) and $Et_3N$ (6.9 mL), the reaction mixture was stirred at room temperature for 12 h. The reaction mixture was monitored by TLC and 1 N HCl were added at the end of the reaction. The aqueous layer was then extracted with $CH_2Cl_2$ three times. The combined organic layer was dried over $MgSO_4$, filtered, and concentrated in vacuo. The residue was purified by silica gel column chromatography using a petroleum ether/AcOEt mixture [5:1 (v/v)] as the eluent to give alkyl tosylate.

**Experimental procedure for synthesis of nalkenyloxypyridinium salt ( 2a )**.




To a solution of 4-methylpyridine 1-oxide (2.0 mmol) in MeCN (1.0 mL) was added appropriate alkyl tosylate (2.2 mmol) in MeCN (1.0 mL). The reaction mixture was stirred at 80 °C for 24 h. The reaction mixture was monitored by TLC and evaporated under reduced pressure. The residue was recrystallized twice from DCM (2 mL) and $Et_2O$ (60 mL) solution at -20 °C to give a corresponding nalkenyloxypyridinium salt.

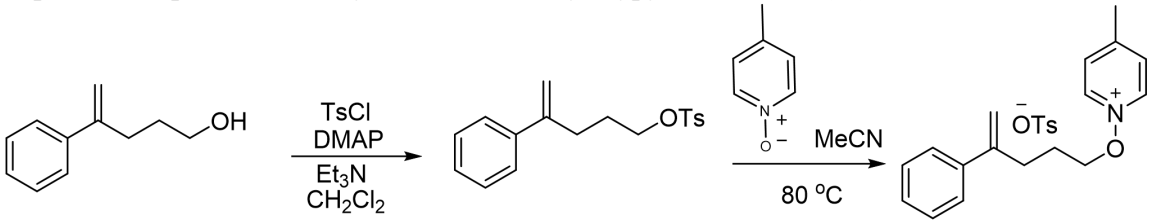
**Experimental procedure for the synthesis of terminal γ- or δ-hydroxyalkenes (4a).**




To a suspension of methyltriphenylphosphonium bromide (26.0 mmol) in dry THF (50 mL) was added potassium *tert*-butoxide (52.0 mmol) at 0 °C. The mixture was then stirred for 60 min. 4-Oxo-4-phenylbutanoic acid (20.0 mmol) was then added to the reaction mixture at 0 °C. The mixture was allowed to warm to room temperature, and then stirred for 16 h. After evaporation of THF, $CH_2Cl_2$ and 1 N NaOH were added. The aqueous layer was washed with $CH_2Cl_2$. 12 N HCl was then added until

the pH of the aqueous layer was 2.0. The aqueous layer was then extracted with $CH_2Cl_2$ twice. The combined organic layer was dried over $MgSO_4$, filtered, and concentrated in vacuo. The residue was purified by silica gel column chromatography using a petroleum ether/AcOEt mixture [from 10:1 to 5:1 (v/v)] as the eluent to give 4-phenylpent-4-enoic acid. 4-Phenylpent-4-enoic acid (10 mmol) was dissolved in dry THF (20 mL). Lithium aluminum hydride (20.0 mmol) was added portionwise at 0 °C. The reaction mixture was stirred for 30 min. The reaction was then quenched with 2 N NaOH and filtered through a pad of Celite. The organic layer was extracted with diethyl ether, dried over magnesium sulfate, and concentrated in vacuo. The residue was purified by silica gel column chromatography using a petroleum ether/AcOEt mixture [5:1 (v/v)] as the eluent to give 4-phenylpent-4-en-1-ol as a clear oil.

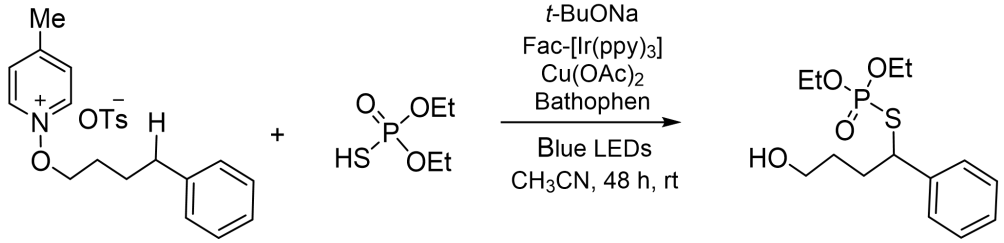
**Experimental procedure for synthesis of nalkenyloxypyridinium salt (4a).**




To a solution of TsCl (18.0 mmol) in $CH_2Cl_2$ was added appropriate 4- phenylpent-4-en-1-ol (16.6 mmol). After added DMAP (101.0 mg) and $Et_3N$ （6.9 mL）,the reaction mixture was stirred at room temperature for 12 h. The reaction mixture was monitored by TLC and 1 N HCl were added at the end of the reaction. The aqueous layer was then extracted with $CH_2Cl_2$ third. The combined organic layer was dried over $MgSO_4$, filtered, and concentrated in vacuo. The residue was purified by silica gel column chromatography using a petroleum ether/AcOEt mixture [5:1 (v/v)] as the eluent to give alkyl tosylate. To a solution of 4-methylpyridine 1-oxide (2.0 mmol) in MeCN (1.0 mL) was added appropriate alkyl tosylate (2.2 mmol) in MeCN (1.0 mL). The reaction mixture was stirred at 80 °C for 24 h. The reaction mixture was monitored by TLC and evaporated under reduced pressure. The residue was recrystallized twice from DCM (2 mL) and $Et_2O$ (60 mL) solution at -20 °C to give a corresponding nalkenyloxypyridinium salt.

# 2. Synthesis and characterization of products.

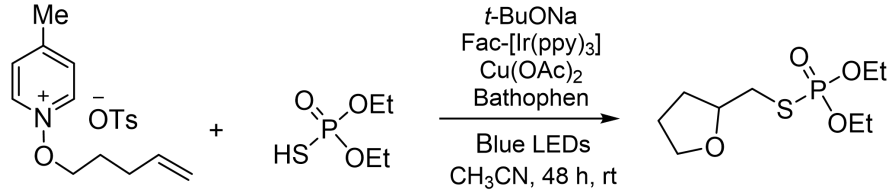
**Experimental procedure for synthesis of *O,O*-diethyl *S*-(4-hydroxy-1-phenylbutyl) phosphorothioate (3a).**




**1a** **2a** **3a**

An oven-dried Schlenk tube containing bathophenanthroline (6.6 mg, 0.02 mmol, 0.2 equiv), $Cu(OAc)_2$ (3.9 mg, 0.02 mmol, 0.2 equiv), fac-Ir(ppy)$_3$ (1.3 mg, 0.002 mmol, 0.02 equiv), **1a** (0.1 mmol, 1.0 equiv) was evacuated and purged with argon three times. Freshly distilled $CH_3CN$ (2.0 mL) and **2a** (1.0 mmol) were sequentially added to the system at room temperature. The resulting mixture was irradiated with 30 W blue LEDs strip for 48 h. After the removal of solvents under reduced pressure, the crude product was purified by column chromatography on silica gel with ethyl acetate/petroleum ether [1:1 (v/v)] as the eluent to give the pure product *O,O*-diethyl *S*-(4-hydroxy-1-phenylbutyl) phosphorothioate **3a**.

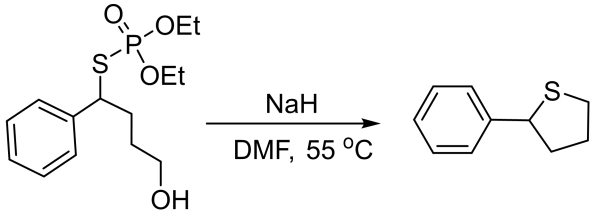
**Experimental procedure for synthesis of *O,O*-diethyl *S*-((tetrahydrofuran-2-yl)methyl) phosphorothioate (5a).**




**4a** **2a** **5a**

An oven-dried Schlenk tube containing bathophenanthroline (6.6 mg, 0.02 mmol, 0.2 equiv), $Cu(OAc)_2$ (3.9 mg, 0.02 mmol, 0.2 equiv), fac-$Ir(ppy)_3$ (1.3 mg, 0.002 mmol, 0.02 equiv), **4a** (0.1 mmol, 1.0 equiv) was evacuated and purged with argon three times. Freshly distilled $CH_3CN$ (2.0 mL) and **2a** (1.0 mmol) were sequentially added to the system at room temperature. The resulting mixture was irradiated with 30W blue LEDs strip for 48 h. After the removal of solvents under reduced pressure, the crude product was purified by column chromatography on silica gel with ethyl acetate/petroleum ether [1:1 (v/v)] as the eluent to give the pure product *O,O*-diethyl *S*-((tetrahydrofuran-2-yl)methyl) phosphorothioate **5a**.

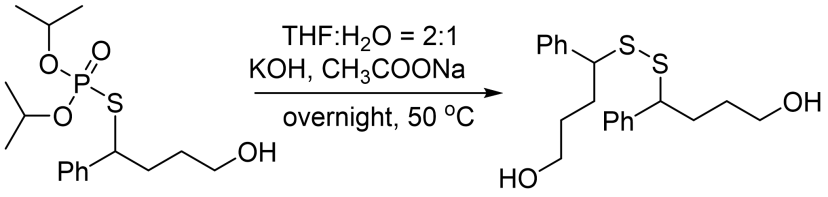
**Experimental procedure for synthesis of 2-phenyltetrahydrothiophene (6a).**




**3a** **6a**

To a solution of 3a (0.30 mmol, 95.4 mg) in DMF (6.0 mL) was added NaH (0.60 mmol, 24 mg). This solution was placed in a pre-heated oil bath that was set to 55 °C, and the reaction was stirred overnight. The reaction was diluted with Et2O (50mL), washed with a 1:1 H2O:brine solution (2 × 50mL), dried with MgSO4, concentrated in vacuo, and the residue was purified by silica gel chromatography (petroleum ether/AcOEt mixture [30:1 (v/v)]) to afford **6a** as a clear and colorless oil.

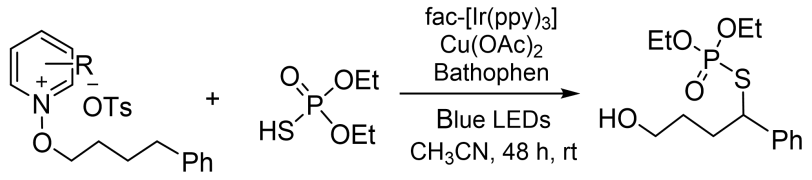
**Experimental procedure for synthesis of 4,4'-disulfanediylbis(4-phenylbutan-1-ol) (7a).**

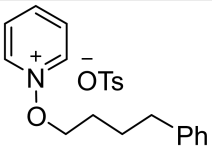
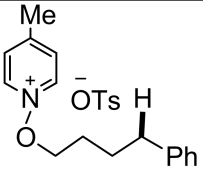
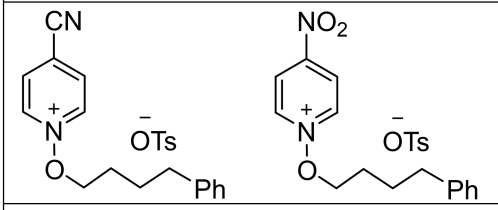
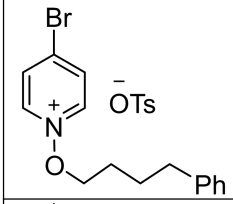
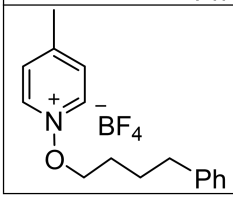



**3ac** **7a**

To a solution of **3ac** (0.30 mmol, 103.8 mg) in 2:1 THF:$H_2O$ solution (6.0 mL) was added KOH (0.60 mmol, 33.7 mg) and $CH_3COONa$ (0.60mmol, 49.2mg). This solution was placed in a pre-heated oil bath that was set to 50 °C, and the reaction was stirred overnight. The reaction mixture was monitored by TLC and 1 N HCl were added at the end of the reaction. The aqueous layer was then extracted with $CH_2Cl_2$ three times. The combined organic layer was dried over $MgSO_4$, filtered, and concentrated in vacuo. The residue was purified by silica gel column chromatography using a petroleum ether/AcOEt mixture [1:1 (v/v)] as the eluent to give**7a** as a clear and colorless oil.

**Table S1 Effects of pyridinium structures on the reaction efficiency**




| N-alkoxypyridiniumsalt | Yield (%) |
|---|---|
|  | 63 |
|  | 77 |
|  | trace |
|  | 59 |
|  | 66 |

**Luminescence quenching experiments**

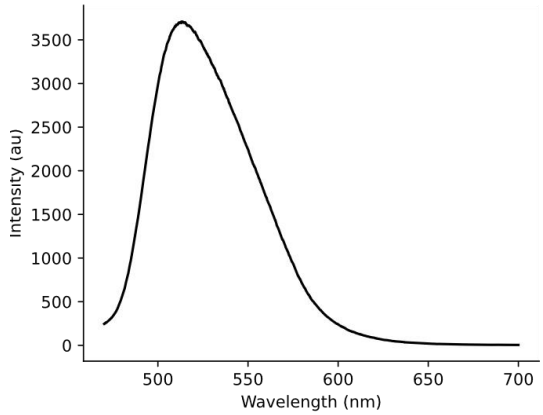



**Fig. S1** the emission spectra of a 5.0 × $10^{-3}$ M solution of *fac*-[*Ir(ppy)*$_3$] in degassed $CH_3CN$ excited at 516 nm;

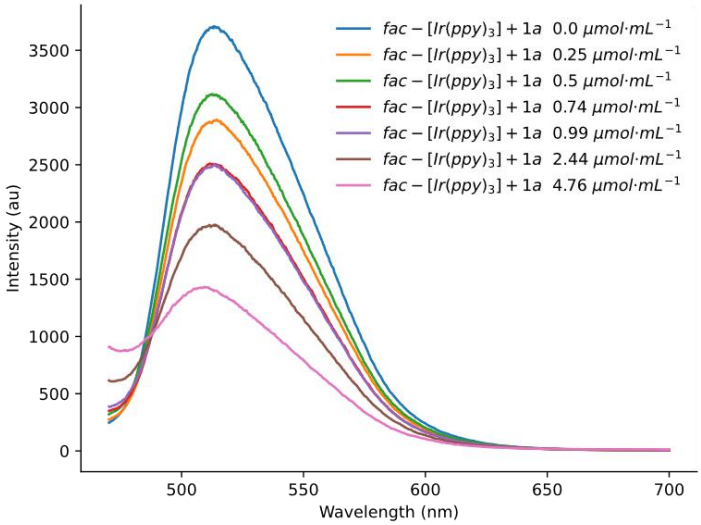



**Fig. S2** the emission spectra of a 5.0 × $10^{-3}$ M solution of *fac*-[*Ir(ppy)*$_3$] with various concentraions of **1a** in degassed $CH_3CN$ excited at 516 nm;

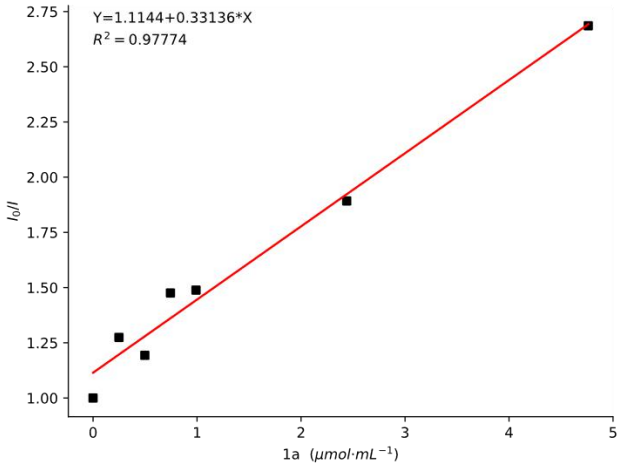



**Fig. S3** the Stern-Volmer plot of a 5.0 × $10^{-3}$ M solution of *fac*-[*Ir(ppy)*$_3$] with various concentraions of **1a** in degassed $CH_3CN$ excited at 516 nm;

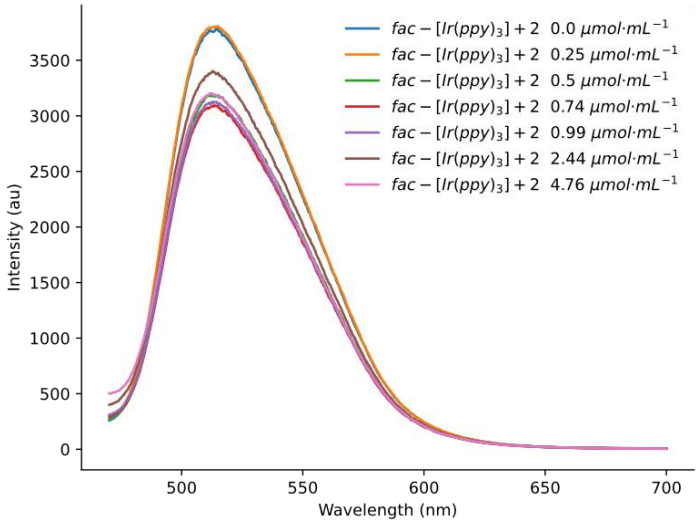



**Fig. S4** the emission spectra of a $5.0 \times 10^{-3}$ M solution of *fac*-[*Ir(ppy)*$_3$] with various concentraions of **2** in degassed $CH_3CN$ excited at 516 nm;

# 4. Cyclic Voltammetry Studies

The cyclic voltammograms were recorded in an electrolyte of $Et_4NPF_6$ (0.1 M) in the indicated solvent (6 mL) using a glassy carbon disk working electrode (diameter, 1 mm), a Pt wire auxiliary electrode a nd a SCE reference electrode. The scan rate was 100 mV/s.

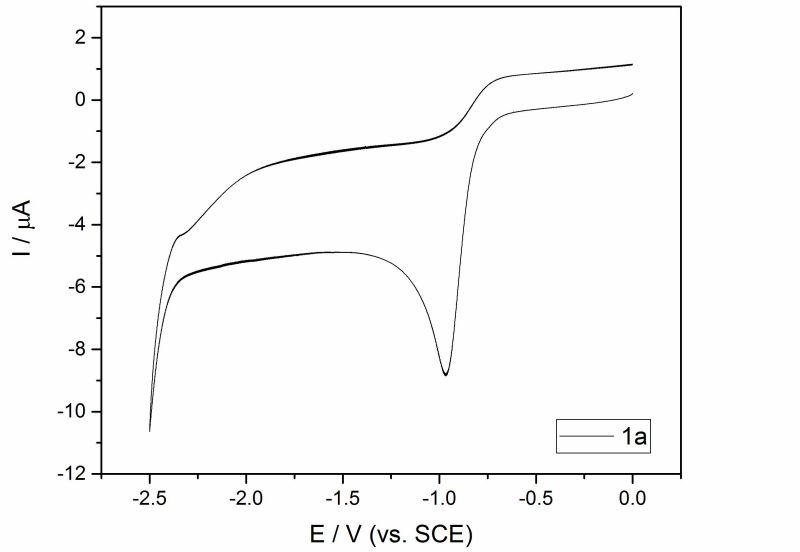
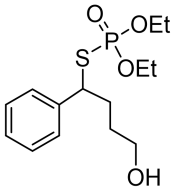



**Fig. S5** Cyclic voltammogram of **1a** (10 mM) in MeCN, $E_{p/2}$ = -0.88 V.

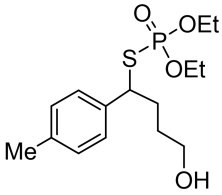
# Spectral data

***O,O*-diethyl *S*-(4-hydroxy-1-phenylbutyl) phosphorothioate (3a)**



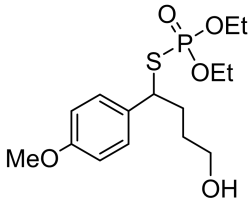
Yield: 23.3 mg, 73%. Brown oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.34-7.30 (m, 4H), 7.26-7.23 (m, 1H), 4.34-4.30 (m, 1H), 4.11-4.05 (m, 1H), 3.97-3.85 (m, 3H), 3.66-3.60 (m, 2H), 2.22-2.16 (m, 1H), 2.11-2.05(m, 1H), 1.62-1.49 (m, 2H), 1.23-1.19 (m, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 142.3 (d, $J$ = 4.3 Hz), (128.9 (s), 127.9 (s), 127.8 (s), 63.8 (d, $J$ = 5.9 Hz), 63.7 (d, $J$ = 5.9 Hz), 62.0 (s), 50.7 (d, $J$ = 3.2 Hz), 35.0 (d, $J$ = 7.5 Hz), 30.6 (s), 16.1 (dd, $J_1$= 7.6 Hz, $J_2$= 2.1 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 26.3. HRMS calcd for $C_{14}H_{23}NaO_4PS^+$ $[M+Na]^+$, 341.0947; found, 341.0945.

***O,O*-diethyl *S*-(4-hydroxy-1-(*p*-tolyl)butyl) phosphorothioate (3b)**



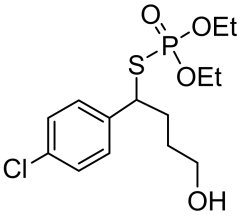
Yield: 23.2 mg, 70%. Brown oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.22 (d, $J$ = 8.04 Hz, 2H), 7.12 (d, $J$ = 7.92 Hz, 2H), 4.32-4.28 (m, 1H), 4.11-4.07 (m, 1H), 4.00-3.88 (m, 3H), 3.66-3.61 (m, 2H), 2.32 (s, 3H), 2.22-2.16 (m, 1H), 2.11-2.05 (m, 1H), 1.58-1.51 (m, 2H), 1.23 (q, $J$ = 7.34 Hz, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 139.1 (d, $J$ = 5.1 Hz) 137.6 (s), 129.5 (s), 127.6 (s), 63.7 (dd, $J_1$= 2.5 Hz, $J_2$= 5.7 Hz), 62.0 (s), 50.5 (d, $J$ = 3.2 Hz), 35.0 (d, $J$ = 6.8 Hz) 30.6 (s), 21.3 (s), 16.1 (d, $J$ = 7.5 Hz).$^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 26.4. HRMS calcd for $C_{15}H_{25}NaO_4PS^+$ $[M+Na]^+$, 355.1103; found, 355.1097.

***O,O*-diethyl *S*-(4-hydroxy-1-(4-methoxyphenyl)butyl) phosphorothioate (3c)**



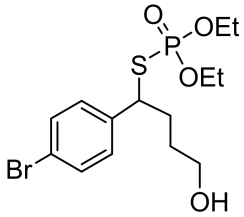
Yield: 21.6 mg, 62%. Brown oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.25 ( d, $J$ = 8.10 Hz, 2H), 6.84 (d, $J$ = 8.70 Hz, 2H), 4.33-4.29 (m, 1H), 4.11-4.07 (m, 1H), 3.99-3.90 (m, 1H), 3.79 (s, 3H), 3.66-3.61 (m, 2H), 2.21-2.16 (m, 1H), 2.09-2.04 (m, 1H), 1.56-1.51 (m, 2H), 1.26-1.21 (m, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 159.2 (s), 133.9 (s), 128.9 (s), 114.2 (s), 63.7 (t, $J$ = 5.6 Hz), 62.0 (s), 55.5 (s), 50.3 (d, $J$ = 3.2 Hz), 35.1 (d, $J$ = 7.2 Hz), 30.6 (s), 16.1 (dd, $J_1$= 4.2 Hz, $J_2$= 7.5 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 26.5. HRMS calcd for $C_{15}H_{25}NaO_5PS^+$ $[M+Na]^+$, 371.1053; found, 371.1049.

***S*-(1-(4-chlorophenyl)-4-hydroxybutyl) *O,O*-diethyl phosphorothioate (3d)**



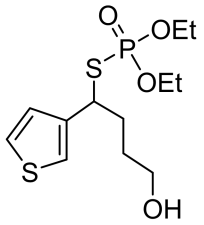
Yield: 26.4 mg, 75%. Brown oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.30-7.27 (m, 4H), 4.33-4.29 (m, 1H), 4.11-4.06 (m, 1H), 3.97-3.89 (m, 3H), 3.64 (t, $J$ = 5.64 Hz, 2H), 2.20-2.14 (m, 1H), 2.08-2.01 (m, 1H), 1.60-1.49 (m, 2H), 1.24 (t, $J$ = 7.62 Hz, 3H), 1.20 (t, $J$ = 7.08 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 141 (d, $J$ = 4.3 Hz), 133.6 (s), 129.2 (s) 129.0 (s), 63.9 (dd, $J_1$= 11.4 Hz, $J_2$= 6.1 Hz ), 63.8 (d, $J$ = 6.1 Hz), 62.0 (s), 50.0 (d, $J$ = 3.3 Hz), 34.8 (d, $J$ = 7.7 Hz), 30.5 (s), 16.1 (d, $J$ = 7.7 Hz) $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 24.4. HRMS calcd for $C_{14}H_{22}ClNaO_4PS^+$ $[M+Na]^+$, 375.0557; found, 375.0567.

***S*-(1-(4-bromophenyl)-4-hydroxybutyl) *O,O*-diethyl phosphorothioate (3e)**



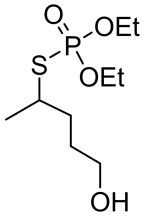
Yield: 28.6 mg, 72%. Brown oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.43 (d, $J$ = 8.30 Hz, 1H), 7.33-7.29 (m, 2H), 7.24-7.20 (m, 1H), 4.32-4.25 (m, 1H), 4.09-4.06 (m, 1H), 3.95-3.84 (m, 3H), 3.62-3.59 (m, 2H), 2.19-2.12 (m, 1H), 2.09-2.01 (m, 1H), 1.59-1.54 (m, 1H), 1.51-1.48 (m, 1H), 1.23-1.17 (m, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 142.3 (d, $J$ = 4.3 Hz), 132.0 (s), 128.9 (s), 127.8 (s), 127.8 (s), 63.7 (d, $J$ = 5.5 Hz), 62.1 (s), 50.7 (d, $J$ = 3.3 Hz), 35.0 (d, $J$ = 7.6 Hz), 30.6 (s), 16.1 (d, $J$ = 2.2 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 26.3. HRMS calcd for $C_{14}H_{22}BrNaO_4PS^+$ $[M+Na]^+$, 419.0052; found, 419.0050.

***O,O*-diethyl *S*-(4-hydroxy-1-(thiophen-2-yl)butyl) phosphorothioate (3f)**



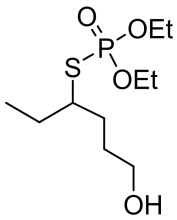
Yield: 16.2 mg, 50%. Brown oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.23 (d, $J$ = 5.04 Hz, 1H), 7.03 (d, $J$ = 3.30 Hz, 1H), 6.91 (dd, $J_1$= 3.57 Hz, $J_2$= 6.01 Hz, 1H), 4.73-4.60 (m, 1H), 4.16-4.12 (m, 1H), 4.06-3.97 (m, 3H), 3.68-3.65 (m, 2H), 2.27-2.23 (m, 1H), 2.19-2.13 (m, 1H), 1.67-1.62 (m, 2H), 1.30-1.25 (m, 6H).$^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 146.0 (d, $J$ = 5.6 Hz), 126.8 (s), 126.0 (s), 125.3 (s), 64.0 (t, $J$ = 6.7 Hz), 61.9 (s), 46.0 (d, $J$ = 3.1 Hz), 36.2 (d, $J$ = 6.30 Hz), 30.4 (s), 16.2 (t, $J$ = 7.3 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 25.6. HRMS calcd for $C_{12}H_{21}ClNaO_4PS_2^+$ $[M+Na]^+$, 347.0511; found, 347.0501.

***O,O*-diethyl *S*-(5-hydroxypentan-2-yl) phosphorothioate (3g)**



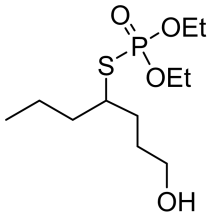
Yield: 16.6 mg, 65%. Brown oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.20-4.11 (m, 4H), 3.68 (t, $J$ = 6.06 Hz, 2H), 3.44-3.38 (m, 1H), 1.83-1.79 (m, 1H), 1.77-1.67 (m, 3H), 1.43 (d, $J$ = 6.84 Hz, 3H), 1.36 (t, $J$ = 7.08 Hz, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 63.8 (d, $J$ = 6.4 Hz), 62.4 (s), 42.8 (d, $J$ = 3.5 Hz), 35.3 (d, $J$ = 6.4 Hz), 30.0 (s), 23.5 (d, $J$ = 5.7 Hz), 16.3 (d, $J$ = 7.5 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 28.1. HRMS calcd for $C_9H_{21}NaO_4PS^+$ $[M+Na]^+$, 279.0790; found, 279.0794.

***O,O*-diethyl *S*-(6-hydroxyhexan-3-yl) phosphorothioate (3h)**



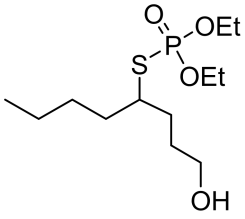
Yield: 18.4 mg, 68%. Brown oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.20-4.11 (m, 4H), 3.71 (t, $J$ = 6.12 Hz, 2H), 3.33-3.29 (m, 1H), 1.83-1.76 (m, 2H), 1.74-1.65 (m, 4H), 1.35 (t, $J$ = 7.05 Hz, 6H), 1.02 (t, $J$ = 7.32 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 63.8 (d, $J$ = 6.3 Hz), 62.4 (s), 49.8 (d, $J$ = 3.5 Hz), 32.8 (d, $J$ = 4.5 Hz), 29.7 (s), 29.3 (d, $J$ = 6.5 Hz), 16.3 (dd, $J_1$= 2.0 Hz, $J_2$= 7.6 Hz). 11.3 (s). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 27.4. HRMS calcd for $C_{10}H_{23}NaO_4PS^+$ $[M+Na]^+$, 293.0947; found, 293.0956.

***O,O*-diethyl *S*-(1-hydroxyheptan-4-yl) phosphorothioate (3i)**



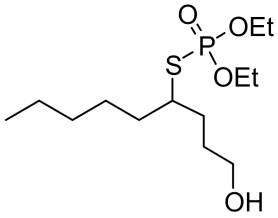
Yield: 19.3 mg, 68%. Brown oil. $^{1}$H NMR (400 MHz, $CDCl_3$) δ (ppm) 4.21-4.10 (m, 4H), 3.67 (t, $J$ = 6.18 Hz, 2H), 3.37-3.33 (m, 1H), 1.83-1.80 (m, 2H), 1.74-1.59 (m, 2H), 1.54-1.48 (m, 1H), 1.46-1.40 (m, 1H), 1.35 (t, $J$ = 7.05 Hz, 6H), 1.31-1.25 (m, 2H), 0.93 (t, $J$ = 7.32 Hz, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ (ppm) 63.8 (d, $J$ = 6.5 Hz), 62.4 (s), 48.0 (s), 38.4 (d, $J$ = 6.6 Hz), 33.3 (d, $J$ = 4.2 Hz) 29.6 (s), 20.2 (s), 16.3 (d, $J$ = 7.7 Hz). 14.0 (s). $^{31}$P NMR (162 MHz, $CDCl_3$) δ (ppm) 28.2. HRMS calcd for $C_{11}H_{25}NaO_4PS^+$ $[M+Na]^+$, 307.1103; found, 307.1104.

***O,O*-diethyl *S*-(1-hydroxyoctan-4-yl) phosphorothioate (3j)**



Yield: 20.5 mg, 69 %. Brown oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.19-4.10 (m, 4H), 3.67 (t, $J$ = 6.15 Hz, 2H), 3.36-3.31 (m, 1H), 1.82-1.79 (m, 2H), 1.75-1.60 (m, 4H), 1.47-1.40 (m, 2H), 1.35 (t, $J$ = 6.15 Hz, 6H), 1.32-1.28 (m, 2H), 0.90 (t, $J$ = 7.26 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 63.8 (d, $J$ = 6.4Hz), 62.4 (s), 48.3 (d, $J$ = 3.4Hz), 36.0 (d, $J$ = 6.5 Hz), 33.3 (d, $J$ = 4.4 Hz), 29.6 (s), 29.0 (s), 22.7 (s), 16.3 (d, $J$ = 7.7 Hz). 14.2 (s). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 27.8. HRMS calcd for $C_{12}H_{27}NaO_4PS^+$ $[M+Na]^+$, 321.1260; found, 321.1265.

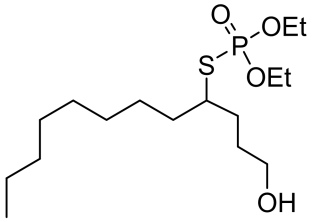
***O,O*-diethyl *S*-(1-hydroxynonan-4-yl) phosphorothioate (3k)**



Yield: 20.9 mg, 67%. Brown oil. $^{1}$H NMR (400 MHz, $CDCl_3$) δ (ppm) 4.23-4.10 (m, 4H), 3.67 (t, $J$ = 6.03 Hz, 2H), 3.37-3.32 (m, 1H), 1.83-1.79 (m, 2H), 1.76-1.59 (m, 6H), 1.49-1.41 (m, 2H), 1.35 (t, $J$ = 7.09 Hz, 6H), 1.31-1.25 (m, 2H), 0.89 (t, $J$ = 6.84 Hz, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ (ppm) 63.8 (d, $J$ = 6.4Hz), 62.4 (s), 48.3 (s), 36.0 (d, $J$ = 6.7 Hz), 33.3 (d, $J$ = 4.3 Hz), 31.8 (s), 29.6 (s), 26.6 (s), 22.8 (s), 16.3 (d, $J$ = 8.1 Hz). 14.2 (s). $^{31}$P NMR (162 MHz, $CDCl_3$) δ (ppm) 28.4. HRMS calcd for

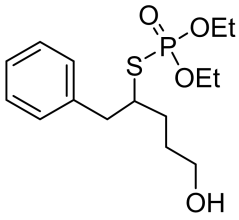
$C_{13}H_{29}NaO_4PS^+$ $[M+Na]^+$, 335.1416; found, 335.1419.

***O,O*-diethyl *S*-(1-hydroxydodecan-4-yl) phosphorothioate (3l)**



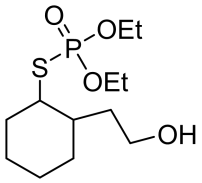
. Yield: 22.7 mg, 64%. Brown oil. $^1$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.20-4.10 (m, 4H), 3.67 (t, *J* = 6.15 Hz, 2H), 3.36-3.32 (m, 1H), 1.83-1.79 (m, 2H), 1.75-1.66 (m, 2H), 1.48-1.45 (m, 1H), 1.41-1.39 (m, 1H), 1.35 (t, *J* = 7.08 Hz, 6H), 1.30-1.24 (m, 10H), 0.88 (t, *J* = 7.00 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 63.8 (d, *J* = 6.5Hz), 62.4 (s), 48.3 (d, *J* = 3.9Hz), 36.3 (d, *J* = 6.6 Hz), 33.3 (d, *J* = 4.3 Hz), 32.1 (s), 29.7(s), 29.6 (s), 29.5(s), 29.4(s), 27.0 (s), 22.9 (s), 16.3 (dd, $J_1$= 7.7 Hz, $J_2$= 2.1 Hz). 14.3 (s). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 28.4. HRMS calcd for $C_{16}H_{36}O_4PS^+$ $[M+H]^+$, 355.2066; found, 355.2056.

***O,O*-diethyl *S*-(5-hydroxy-1-phenylpentan-2-yl) phosphorothioate (3m)**



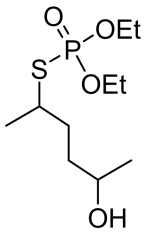
Yield: 22.6 mg, 68%. Brown oil. $^1$H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.32-7.28 (m, 2H), 7.23-7.21 (m, 3H), 4.15-4.11 (m, 1H), 4.05-4.00 (m, 1H), 3.94-3.87 (m, 2H), 3.65 (t, *J* = 5.85 Hz, 2H), 3.59-3.53 (m, 1H), 2.98 (d, *J* = 7.32 Hz, 2H), 1.85-1.83 (m, 1H), 1.79-1.74 (m, 3H), 1.31 (t, *J* = 7.05 Hz, 3H), 1.23 (t, *J* = 7.08 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 138.9 (s), 129.7 (s), 128.6 (s) 126.9 (s), 63.8 (dd, $J_1$= 6.2 Hz, $J_2$= 15.0 Hz), 62.3 (s), 49.5 (d, *J* = 3.3 Hz), 43.0 (d, *J* = 5.9 Hz), 32.7 (d, *J* = 5.0 Hz), 29.7 (s), 16.2 (dd, $J_1$= 7.4 Hz, $J_2$= 14.8 Hz) $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 26.8. HRMS calcd for $C_{15}H_{25}NaO_4PS^+$ $[M+Na]^+$, 355.1103; found, 355.1095.

***O,O*-diethyl *S*-(2-(2-hydroxyethyl)cyclohexyl) phosphorothioate (3n)**



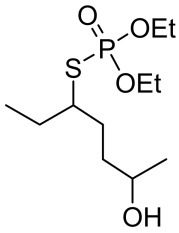
Yield: 18.4 mg, 62%. Brown oil. $^1$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.18-4.10 (m, 4H), 3.77-3.73 (m, 1H), 3.69-3.65 (m, 1H), 3.20-3.17 (m, 1H), 2.22-2.18 (m, 1H), 2.10-2.05 (m, 1H), 1.97-1.94 (m, 1H), 1.68-1.64 (m, 4H), 1.58-1.53 (m, 1H), 1.41-1.38 (m, 1H), 1.36-1.34 (m, 6H), 1.32-1.29 (m, 1H), 1.20-1.14 (m, 1H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 63.9 (t, *J* = 6.3 Hz), 60.5 (s), 51.1 (s), 39.7 (s), 36.6 (s), 31.3 (s), 29.9 (s), 26.0 (s), 24.7 (s), 16.3 (dd, $J_1$= 6.6 Hz, $J_2$= 6.6 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 28.6. HRMS calcd for $C_{12}H_{25}NaO_4PS^+$ $[M+Na]^+$, 319.1103; found, 319.1109.

***O,O*-diethyl *S*-(5-hydroxyhexan-2-yl) phosphorothioate (3o)**



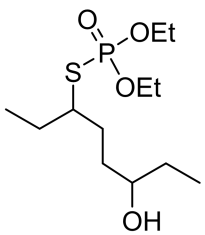
Yield: 18.1 mg, 67%. Brown oil. $^1$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.19-4.09 (m, 4H), 3.83-3.79 (m, 1H), 3.41-3.35 (m, 1H), 1.83-1.66 (m, 2H), 1.59-1.53 (m, 2H), 1.43-1.40 (m, 3H), 1.34 (t, *J* = 7.05 Hz, 6H). 1.19 (dd, $J_1$= 2.04, $J_2$= 6.18 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 67.6 (s), 63.8 (d, *J* = 6.9 Hz), 43.1 (d, *J* = 3.5 Hz), 36.2 (s), 35.0 (d, *J* = 6.4 Hz), 23.6 (s), 23.5 (d, *J* = 5.3 Hz), 16.3 (s). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 27.7 (s), 27.8 (s). HRMS calcd for $C_{10}H_{23}NaO_4PS^+$ $[M+Na]^+$, 293.0947; found, 293.0947.

***O,O*-diethyl *S*-(6-hydroxyheptan-3-yl) phosphorothioate (3p)**



Yield: 18.2 mg, 64%. Brown oil. $^1$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.18-4.06 (m, 4H), 3.80-3.76 (m, 1H), 3.27-3.22 (m, 1H), 1.82-1.70 (m, 3H), 1.68-1.60 (m, 1H), 1.57-1.51 (m, 2H), 1.32 (t, *J* = 7.08 Hz, 6H). 1.16 (dd, $J_1$= 2.49, $J_2$= 6.15 Hz, 3H). 1.00-0.97 (m, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 67.9 (s), 63.7 (d, *J* = 5.3 Hz), 42.9 (d, *J* = 3.6 Hz), 36.3 (s), 35.3 (d, *J* = 6.4 Hz), 23.8 (s), 23.4(S), 16.3 (s). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 27.8 (s), 27.7 (s). HRMS calcd for $C_{11}H_{25}NaO_4PS^+$ $[M+Na]^+$, 307.1103; found, 307.1106.

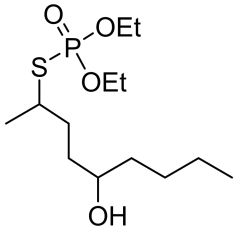
***O,O*-diethyl *S*-(6-hydroxyoctan-3-yl) phosphorothioate (3q)**



Yield: 19.6 mg, 66%. Brown oil. $^1$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.17-4.08 (m, 4H), 3.54-3.49 (m, 1H), 3.30-3.24 (m, 1H), 1.86-1.83 (m, 1H), 1.77-1.72 (m, 2H), 1.70-1.58 (m, 2H), 1.53-1.41 (m, 3H), 1.33 (t, *J* = 7.05 Hz, 6H). 1.01-0.98 (m, 3H), 0.92 (t, *J* = 7.41 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 73.1 (s),

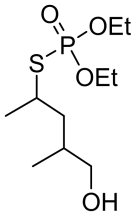
63.8 (d, $J$ = 8.1 Hz), 50.3 (d, $J$ = 3.4 Hz), 33.8 (S), 32.8 (d, $J$ = 4.5 Hz), 30.6 (S), 29.4 (d, $J$ = 6.6 Hz), 16.3 (s). 11.2 (S). 10.2 (S). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 28.4 (s), 28.3 (s). HRMS calcd for $C_{12}H_{27}NaO_4PS^+$ $[M+Na]^+$, 321.1260; found, 321.1256.

***O,O*-diethyl *S*-(5-hydroxynonan-2-yl) phosphorothioate (3r)**



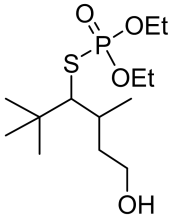
Yield: 19.0 mg, 61%. Brown oil. $^1$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.17-4.07 (m, 4H), 3.58-3.54 (m, 1H), 3.38-3.33 (m, 1H), 1.85-1.65 (m, 2H), 1.61-1.56 (m, 1H), 1.53-1.45 (m, 1H), 1.41-1.38 (m, 6H), 1.32 (t, $J$ = 7.05 Hz, 6H). 1.29-0.22 (m, 3H), 0.87 (t, $J$ = 7.08 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 71.6 (s), 63.6 (d, $J$ = 4.5 Hz), 43.3 (d, $J$ = 3.9 Hz), 37.5 (S), 35.2 (d, $J$ = 6.4 Hz), 34.6 (S), 28.1 (S), 27.3 (d, $J$ = 5.0 Hz), 22.9(S), 16.3 (s), 14.2 (S). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 26.9 (s), 26.3 (s). HRMS calcd for $C_{13}H_{29}NaO_4PS^+$ $[M+Na]^+$, 335.1416; found, 335.1411.

***O,O*-diethyl *S*-(5-hydroxy-4-methylpentan-2-yl) phosphorothioate (3s)**



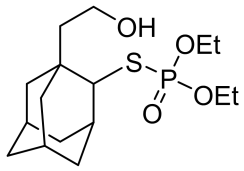
Yield: 15.9 mg, 59%. Brown oil. $^1$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.20-4.10 (m, 4H), 3.60-3.43 (m, 3H), 1.94-1.89 (m, 1H), 1.83-1.69 (m, 1H), 1.57-1.52 (m, 1H), 1.43 (t, $J$ = 6.11 Hz, 3H), 1.35 (t, $J$ = 7.08 Hz, 6H), 0.94 (dd, $J_1$= 6.78 Hz, $J_2$= 14.4 Hz). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 68.3 (s), 63.9 (d, $J$ = 6.7 Hz), 43.3 (d, $J$ = 5.6 Hz), 41.3 (d, $J$ = 3.8 Hz), 33.8 (s), 24.1 (d, $J$ = 5.1 Hz), 22.5 (s), 17.4 (s). 16.3 (d, J = 6.7 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 27.9 (s), 27.8 (s). HRMS calcd for $C_{10}H_{23}NaO_4PS^+$ $[M+Na]^+$, 293.0947; found, 293.0948.

***O,O*-diethyl *S*-(6-hydroxy-2,2,4-trimethylhexan-3-yl) phosphorothioate (3t)**



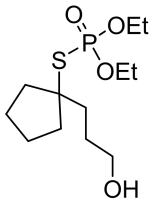
Yield: 15.3 mg, 49%. Brown oil. $^1$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.16-4.08 (m, 4H), 3.74-3.70 (m, 1H), 3.62-3.58 (m, 1H), 3.62-3.23 (m, 1H), 2.30-2.27 (m, 1H), 1.83-1.78 (m, 1H), 1.46-1.41 (m, 1H), 1.34-1.31 (m, 6H). 1.11-1.07 (m, 3H). 1.06 (s, 9H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 66.5 (s), 63.8 (d, $J$ = 2.6 Hz), 61.2 (s), 37.3 (s), 31.9 (s), 29.9 (s), 29.1 (s), 20.4 (s), 16.3 (s). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 28.1 (s), 28.0 (s). HRMS calcd for $C_{13}H_{29}NaO_4PS^+$ $[M+Na]^+$, 335.1416; found, 335.1408.

***O,O*-diethyl *S*-((1r,3s,5R,7S)-1-(2-hydroxyethyl)adamantan-2-yl) phosphorothioate (3u)**



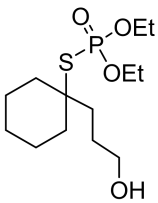
Yield: 23.7 mg, 68%. Brown oil. $^1$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.18-4.08 (m, 3H), 3.77-3.72 (m, 1H), 3.69-3.66 (m, 2H), 2.15-2.12 (m, 2H), 2.08-2.06 (m, 1H), 1.93-1.92 (m, 1H), 1.89-1.88 (m, 1H), 1.84-1.76 (m, 4H), 1.71-1.69 (m, 1H), 1.65-1.60 (m, 3H), 1.57-1.55 (m, 1H), 1.42-1.38 (m, 2H), 1.35-1.31 (m, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 64.0 (d, $J$ = 6.2 Hz), 6.39 (d, $J$ = 6.9 Hz), 58.9 (d, $J$ = 3.0 Hz), 58.2 (s), 43.6 (s) 42.3 (S), 39.2 (s), 38.3 (s), 37.1 (s), 36.7 (s), 36.2 (d, $J$ = 5.3 Hz), 31.8 (S), 28.1 (d, $J$ = 7.9 Hz), 16.3 (t, $J$ = 8.0 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 29.1 (s). HRMS calcd for $C_{16}H_{29}NaO_4PS^+$ $[M+Na]^+$, 371.1416; found, 371.1415.

***O,O*-diethyl *S*-(1-(3-hydroxypropyl)cyclopentyl) phosphorothioate (3v)**



Yield: 21.3 mg, 72%. Brown oil. $^1$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.17-4.09 (m, 4H), 3.67 (t, $J$ = 6.15 Hz, 2H), 2.09-2.06 (m, 2H), 1.98-1.95 (m, 2H), 1.85-1.80 (m, 4H), 1.72-1.69 (m, 4H), 1.33 (t, $J$ = 7.05 Hz, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 64.8 (d, $J$ = 4.1 Hz), 63.8 (d, $J$ = 7.2 Hz), 62.9 (s), 40.9 (d, $J$ = 8.0 Hz), 37.4 (s), 29.4 (s), 23.6 (s), 16.3 (d, $J$ = 6.8 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 25.3. HRMS calcd for $C_{12}H_{25}NaO_4PS^+$ $[M+Na]^+$, 319.1103; found, 319.1097.

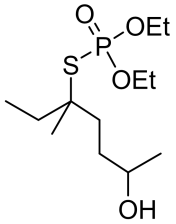
***O,O*-diethyl *S*-(1-(3-hydroxypropyl)cyclohexyl) phosphorothioate (3w)**



Yield: 23.3 mg, 75%. Brown oil. $^1$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.18-4.10 (m, 4H), 3.67 (t, $J$ = 6.06 Hz, 2H), 2.01-1.95 (m, 4H), 1.83-1.78 (m, 2H), 1.69-1.64 (m, 2H), 1.58-1.52 (m, 4H), 1.33 (t, $J$ = 7.11 Hz, 6H), 1.29-1.24 (m, 2H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 63.9 (d, $J$ = 6.7 Hz), 62.9 (s), 59.7 (d, $J$ = 4.0 Hz), 38.4 (d, $J$ = 7.7 Hz), 29.9 (s), 27.3 (s), 25.9 (s), 22.5 (s), 16.3 (d, $J$ = 6.6 Hz). $^{31}$P NMR (242 MHz,

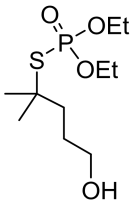
$CDCl_3$) δ (ppm) 25.3. HRMS calcd for $C_{12}H_{25}NaO_4PS^+$ $[M+Na]^+$, 333.1260; found, 333.1256.

***O,O*-diethyl *S*-(6-hydroxy-3-methylheptan-3-yl) phosphorothioate (3x)**



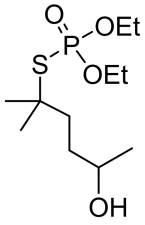
Yield: 22.1 mg, 74%. Brown oil. $^1$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.18-4.08 (m, 4H), 3.79-3.76 (m, 1H), 1.98-1.92 (m, 1H), 1.82-1.71 (m, 3H), 1.61-1.55 (m, 2H), 1.44 (d, *J* =15.94 Hz, 3H), 1.32 (t, *J* = 7.09 Hz, 6H), 1.18 (d, *J* = 6.23 Hz, 3H). 0.97-0.94 (m, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 67.9 (s), 63.8 (d, *J* = 1.8 Hz), 58.6 (d, *J* = 3.6 Hz), 37.0 (d, *J* = 5.4 Hz), 34.8 (d, *J* = 6.4 Hz), 34.2 (s), 27.5 (d, *J* = 5.6 Hz), 23.8 (s), 16.3 (s), 9.1 (s). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 25.1. HRMS calcd for $C_{12}H_{27}NaO_4PS^+$ $[M+Na]^+$, 321.1260; found, 321.1253.

***O,O*-diethyl *S*-(5-hydroxy-2-methylpentan-2-yl) phosphorothioate (3y)**



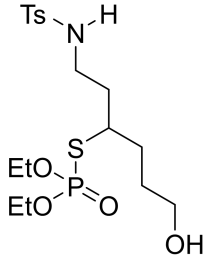
Yield: 20.5 mg, 76%. Brown oil. $^1$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.20-4.01 (m, 4H), 3.67 (t, *J* = 6.12 Hz, 2H), 1.87-1.84 (m, 2H), 1.77-1.72 (m, 2H), 1.51 (s, 6H), 1.34 (t, *J* = 7.08 Hz, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 63.9 (d, *J* = 6.8 Hz), 62.9 (s), 53.9 (d, *J* = 4.2 Hz), 40.1 (d, *J* = 5.1 Hz), 30.8 (d, *J* = 6.6 Hz), 28.5 (s), 16.3 (d, *J* = 7.5 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 28.2. HRMS calcd for $C_{10}H_{23}NaO_4PS^+$ $[M+Na]^+$, 293.0947; found, 293.0942.

***O,O*-diethyl *S*-(5-hydroxy-2-methylhexan-2-yl) phosphorothioate (3z)**



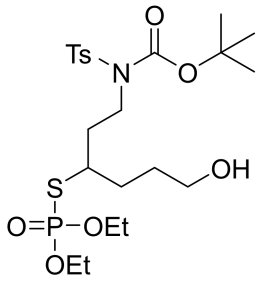
Yield: 20.7 mg, 73%. Brown oil. $^1$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.16-4.07 (m, 4H), 3.78-3.75 (m, 1H), 1.96-1.91 (m, 1H), 1.76-1.71 (m, 1H), 1.61-1.57 (m, 2H), 1.47 (d, *J* = 9.90 Hz, 6H), 1.31 (t, *J* = 7.05 Hz, 6H). 1.18 (d, *J* = 6.24 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 67.9 (s), 63.8 (t, *J* = 6.5 Hz), 53.9 (d, *J* = 4.3 Hz), 40.0 (d, *J* = 5.5 Hz), 34.7 (s), 30.8 (s), 23.8(s), 16.3 (d, *J* = 1.8 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 25.1. HRMS calcd for $C_{11}H_{25}NaO_4PS^+$ $[M+Na]^+$, 307.1103; found, 307.1099.

***O,O*-diethyl *S*-(6-hydroxy-1-((4-methylphenyl) sulfonamido) hexan-3-yl) phosphorothioate (3ac)**



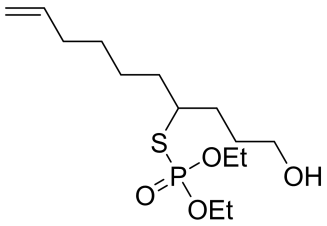
Yield: 20.2 mg, 46%. Brown oil. $^1$H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.75 (d, *J* = 8.28 Hz, 2H), 7.27 (d, *J* = 7.98 Hz 2H), 6.32 (dd, $J_1$= 5.10 Hz, $J_2$= 8.04 Hz, 1H) 4.16-3.98 (m, 4H), 3.62 (t, *J* = 5.76 Hz, 2H), 3.35-3.29 (m, 1H), 3.21-3.16 (m, 1H), 3.11-3.05 (m, 1H), 2.39 (S, 3H), 1.97-1.93 (m, 1H), 1.73-1.59 (m, 5H). 1.34 (t, *J* = 6.93 Hz, 3H), 1.27 (t, *J* = 6.96 Hz, 3H),$^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 143.1 (s), 137.8 (s), 129.7 (s), 127.4 (S), 64.3 (t, *J* = 7.1 Hz), 62.0 (S), 45.3 (d, *J* = 3.3 Hz), 40.3 (s), 37.3 (d, *J* = 3.7 Hz), 33.7 (d, *J* = 5.9 Hz), 29.7 (s), 21.6 (s), 16.1 (dd, $J_1$= 7.4 Hz, $J_2$= 3.4 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 28.59 (s). HRMS calcd for $C_{17}H_{31}NO_6PS_2^+$ $[M+H]^+$, 440.1325; found, 440.1324.

***tert*-butyl (3-((diethoxyphosphoryl)thio)-6-hydroxyhexyl) (tosyl) carbamate**



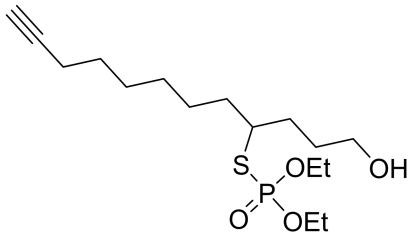
Yield: 32.9 mg, 61%. Brown oil. $^1$H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.76 (d, *J* = 7.76 Hz, 2H), 7.30 (d, *J* = 8.10 Hz 2H), 4.23-4.13 (m, 4H), 4.05-4.00 (m, 1H), 3.93-3.88 (m, 1H), 3.64 (t, *J* = 6.24 Hz, 2H), 3.42-3.35 (m, 1H), 2.43 (s, 3H), 2.20-2.16 (m, 1H), 2.14-2.08 (m, 1H). 1.90-1.83 (m, 2H), 1.78-1.73 (m, 2H), 1.35 (t, *J* = 7.08 Hz, 6H), 1.32 (s, 9H),$^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 150.9 (s), 144.4 (s), 137.4 (s), 129.4 (s), 127.9 (S), 84.6 (s), 63.9 (t, *J* = 6.9 Hz), 62.0 (S), 45.3 (d, *J* = 3.3 Hz), 45.0 (s), 36.4 (d, *J* = 5.7 Hz), 33.0 (d, *J* = 4.8 Hz), 29.5 (s), 28.0 (s), 21.7 (s), 16.2 (d, *J* = 7.0 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 26.62 (s). HRMS calcd for $C_{22}H_{39}NO_8PS_2^+$ $[M+H]^+$, 540.1849; found, 540.1854.

***O,O*-diethyl *S*-(1-hydroxydec-9-en-4-yl) phosphorothioate (3ae)**



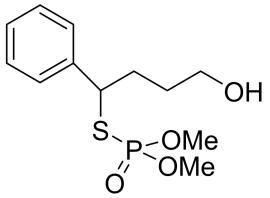
Yield: 21.1 mg, 65%. Brown oil.$^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 5.80-5.73 (m, 1H), 4.98-4.90 (m, 2H), 4.16-4.06 (m, 4H), 3.62 (t, *J* = 6.20 Hz, 2H), 3.32-3.28 (m, 1H), 2.03 (t, *J* = 6.96 Hz, 2H), 1.79-1.75 (m, 2H), 1.71-1.60 (m, 4H), 1.49-1.35 (m, 4H), 1.32 (t, *J* = 7.08 Hz, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 138.7 (s), 114.6 (s), 63.6 (d, *J* = 6.1 Hz), 62.1 (s), 48.2 (s), 36.0 (d, *J* = 6.1 Hz), 33.7 (s), 33.0 (d, *J* = 4.3 Hz), 29.5 (s), 28.7 (s), 26.2 (s), 16.1 (d, *J* = 7.3 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 28.21 (s). HRMS calcd for $C_{14}H_{29}NaO_4PS^+$ $[M+Na]^+$, 347.1416; found, 347.1406.

***O,O*-diethyl *S*-(1-hydroxydodec-11-yn-4-yl) phosphorothioate (3af)**



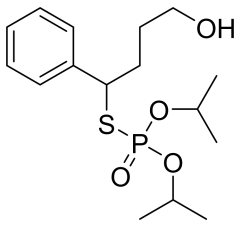
Yield: 21.0 mg, 60%. Brown oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.20-4.08 (m, 4H), 3.65 (t, *J* = 6.10 Hz, 2H), 3.36-3.29 (m, 1H), 2.19-2.15 (m, 2H), 1.92 (t, *J* = 2.58 Hz, 1H), 1.82-1.77 (m, 2H), 1.73-1.59 (m, 2H), 1.55-1.41 (m, 6H), 1.38-1.24 (m, 10H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 84.7 (s), 68.3 (s), 63.7 (d, *J* = 6.5 Hz), 62.2 (s), 48.2 (d, *J* = 3.5 Hz), 36.1 (d, *J* = 6.5 Hz), 33.1 (d, *J* = 4.4 Hz), 29.5 (s), 29.0 (s), 28.7 (s), 28.5 (s), 26.6 (s), 18.5 (s), 16.2 (d, *J* = 7.3 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 28.77 (s). HRMS calcd for $C_{16}H_{31}NaO_4PS^+$ $[M+Na]^+$, 373.1573; found, 373.1573.

***S*-(4-hydroxy-1-phenylbutyl) *O,O*-dimethyl phosphorothioate (3ag)**



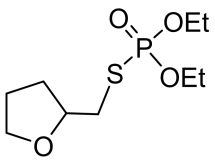
Yield: 18.6 mg, 64%. Brown oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.34-7.29 (m, 4H), 7.26-7.23 (m, 1H), 4.30-4.26 (m, 1H), 3.62-3.60 (m, 2H), 3.56 (dd, $J_1$ = 2.76 Hz, $J_2$ = 12.78 Hz, 6H), 2.19-2.14 (m, 1H), 2.10-2.03 (m, 1H), 1.61-1.56 (m, 1H), 1.53-1.49 (m, 1H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 142.0 (d, *J* = 3.7 Hz), 128.7 (s), 127.8 (s), 127.6 (s), 61.8 (s), 53.8 (dd, $J_1$ = 5.7 Hz, $J_2$ = 9.0 Hz), 50.7 (d, *J* = 3.3 Hz), 34.7 (d, *J* = 7.8 Hz), 30.5 (s). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 29.87 (s). HRMS calcd for $C_{12}H_{19}O_4PS^+$ $[M+H]^+$, 291.0814; found, 291.0813..

***S*-(4-hydroxy-1-phenylbutyl) *O,O*-diisopropyl phosphorothioate (3ah)**



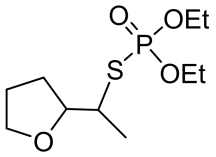
Yield: 26.6 mg, 77 %. Yellow oil. $^{1}$H NMR (600 MHz, CDCl3) δ (ppm) 7.34-7.29 (m, 4H), 7.25-7.22 (m, 1H), 4.70-4.64 (m, 1H), 4.49-4.43 (m, 1H), 4.39-4.35 (m, 1H), 3.66-3.59 (m, 2H), 2.27-2.21 (m, 1H), 2.12-2.07 (m, 1H), 1.55-1.50 (m, 2H), 1.33 ((d, J = 6.18 Hz, 3H), 1.22 (d, J = 6.21 Hz, 3H), 1.18 (t, J = 6.91 Hz, 6H). 3C NMR (150 MHz, CDCl3) δ (ppm) 142.2 ((d, J = 5.4 Hz), 128.8 (s), 127.8 (s), 127.7 (s), 73.0 ((d, J = 6.5 Hz), 72.8 ((d, J = 6.6 Hz), 61.8 (s), 50.5 ((d, J = 3.2 Hz), 35.0 ((d, J = 6.7 Hz), 30.4 (s), 24.0 ((d, J = 4.2 Hz), 23.7 ((d, J = 4.3 Hz), 23.6 ((d, J = 5.5 Hz), 23.5 ((d, J = 5.5 Hz). 31P NMR (242 MHz, CDCl3) δ (ppm) 23.96. HRMS Calcd for C16H28O4PS+ [M+H]+ 347.1440, found 347.1436

***O,O*-diethyl *S*-((tetrahydrofuran-2-yl)methyl) phosphorothioate (5a)**



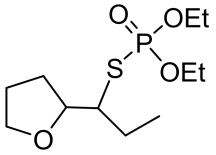
Yield: 20.0 mg, 79%. Yellow oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.20-4.11 (m, 4H), 4.10-4.05 (m, 1H), 3.89-3.86 (m, 1H), 3.77-3.74 (m, 1H), 3.01-2.90 (m, 2H), 2.08-2.02 (m, 1H), 1.95-1.85 (m, 2H), 1.67-1.61 (m, 1H). 1.34 (t, *J* = 7.08 Hz, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 78.2 (d, *J* = 5.6 Hz), 68.6 (s), 63.8 (d, *J* = 5.7 Hz), 35.7 (d, *J* = 3.4 Hz), 30.9 (s), 28.1 (s), 16.3 (d, *J* = 7.3 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 27.8. HRMS calcd for $C_9H_{20}O_4PS^{++}$ $[M+H]^+$, 255.0814; found, 255.0824.

***O,O*-diethyl *S*-(1-(tetrahydrofuran-2-yl)ethyl) phosphorothioate (5b)**



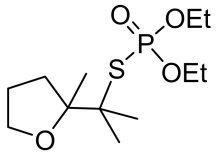
Yield: 21.9 mg, 82%. Yellow oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.18-4.09 (m, 4H), 3.94-3.91 (m, 1H), 3.87-3.83 (m, 1H), 3.78-3.73 (m, 1H), 3.62-3.56 (m, 1H), 2.02-1.94 (m, 1H), 1.93-1.83 (m, 2H). 1.76-1.68 (m, 1H), 1.44-1.40 (m, 3H), 1.32 (t, $J$ = 7.08 Hz, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 82.4 (d, $J$ = 7.4 Hz), 69.0 (s), 63.8 (d, $J$ = 6.3 Hz), 46.6 (d, $J$ = 3.4 Hz), 29.3 (s), 26.4 (s), 20.8 (d, $J$ = 3.3 Hz), 16.2 (d, $J$ = 3.6 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 28.1 (s), 27.5 (s). HRMS calcd for $C_{10}H_{22}O_4PS^+$ [M+H]$^+$, 269.0971; found, 269.0981.

***O,O*-diethyl *S*-(1-(tetrahydrofuran-2-yl)propyl) phosphorothioate (5c)**



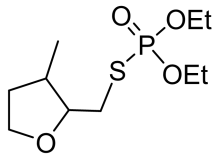
Yield: 22.5 mg, 80%. Yellow oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.14-4.05 (m, 4H), 4.03-3.91 (m, 1H), 3.84-3.79 (m, 1H), 3.73-3.67 (m, 1H), 3.27-3.20 (m, 1H), 1.99-1.91 (m, 1H), 1.89-1.86 (m, 1H). 1.84-1.78 (m, 2H), 1.77-1.71(m, 1H), 1.68-1.59 (m, 1H), 1.28 (t, $J$ = 7.05 Hz, 6H). 1.02-0.99 (m, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 81.0 (d, $J$ = 5.5 Hz), 68.9 (s), 63.6 (d, $J$ = 6.7 Hz), 54.2 (d, $J$ = 3.3 Hz), 29.5 (s), 27.4 (s), 26.3 (s), 16.1 (d, $J$ = 3.3 Hz), 11.8 (s). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 28.7 (s), 28.0 (s). HRMS calcd for $C_{11}H_{23}NaO_4PS^+$ [M+Na]$^+$, 305.0947; found, 305.0949.

***O,O*-diethyl *S*-(2-(5-methyltetrahydrofuran-2-yl)propan-2-yl) phosphorothioate (5d)**



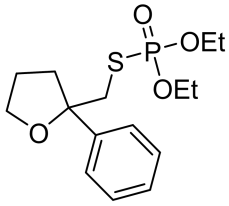
Yield: 25.1 mg, 85%. Yellow oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.25-4.06 (m, 6H), 2.04-1.86 (m, 4H), 1.51-1.50 (m, 6H). 1.28 (td, $J$ = 8.02 Hz, $J$ = 1.87 Hz, 6H). 1.21-1.19 (m, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 85.9 (d, $J$ = 7.8 Hz), 76.2 (s), 63.7 (d, $J$ = 6.6 Hz), 56.8 (d, $J$ = 4.3 Hz), 34.6 (s), 28.7 (s), 26.9 (d, $J$ = 4.4 Hz), 21.4 (s), 16.2 (d, $J$ = 4.0 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 24.6 (s), 24.5 (s). HRMS calcd for $C_{12}H_{25}NaO_4PS^+$ [M+Na]$^+$, 319.1103; found, 319.1102.

***O,O*-diethyl *S*-((4-methyltetrahydrofuran-2-yl)methyl) phosphorothioate (5e)**



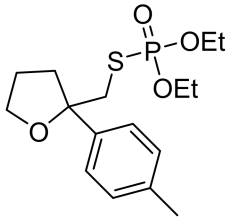
Yield: 20.6 mg, 77%. Yellow oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 4.21-4.08 (m, 5H), 4.01-3.89 (m, 1H), 3.39-3.28 (m, 1H), 3.06-2.90 (m, 2H). 2.37-2.19 (m, 1H). 1.89-1.63 (m, 1H). 1.34 (t, $J$ = 7.06 Hz, 6H), 1.25-1.20 (m, 1H), 1.03 (t, $J$ = 6.89 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 78.9 (s), 75.5 (s), 63.8 (s), 40.1 (s), 35.0 (s), 34.8 (s), 17.8 (s), 16.2 (s). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 28.2 (s), 27.7 (s). HRMS calcd for $C_{10}H_{21}NaO_4PS^+$ [M+Na]$^+$, 219.0790; found, 291.0791.

***O,O*-diethyl *S*-((2-phenyltetrahydrofuran-2-yl)methyl) phosphorothioate (5f)**



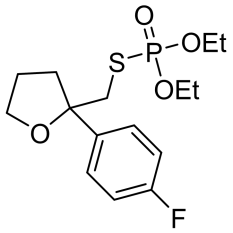
Yield: 26.7 mg, 81%. Yellow oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.58 (d, $J$ = 7.32 Hz, 2H), 7.35 (t, $J$ = 7.20 Hz, 2H), 7.27-7.24 (m, 1H), 4.37-4.34 (m, 1H), 4.15 (d, $J$ = 11.99 Hz, 1H), 3.93-3.87 (m, 2H), 3.82-3.76 (m, 2H), 3.69-3.67 (m, 2H), 2.62-2.58 (m, 1H), 2.43-2.38 (m, 1H), 1.87-1.84 (m, 1H). 1.58-1.55 (m, 1H), 1.18 (t, $J$ = 6.72 Hz, 3H), 1.12 (t, $J$ = 6.72 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 141.7 (s), 128.5 (s), 127.8 (s), 127.6 (s), 75.1 (d, $J$ = 5.3 Hz), 68.5 (s), 63.6 (t, $J$ = 6.5 Hz), 55.0 (d, $J$ = 3.9 Hz), 36.1 (s), 23.4 (s), 16.1 (dd, $J_1$= 7.7 Hz, $J_2$= 10.0 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 24.4. HRMS calcd for $C_{15}H_{23}NaO_4PS^+$ [M+Na]$^+$, 353.0947; found, 353.0940.

***O,O*-diethyl *S*-((2-(p-tolyl)tetrahydrofuran-2-yl)methyl) phosphorothioate (5g)**



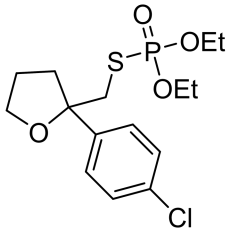
Yield: 28.2 mg, 82%. Yellow oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.45 (d, $J$ = 8.16 Hz, 2H), 7.16 (d, $J$ = 8.04 Hz, 2H), 4.32-4.30 (m, 1H), 4.14 (d, $J$ = 12.00 Hz, 1H), 3.96-3.87 (m, 2H), 3.86-3.70 (m, 1H), 3.76-3.65 (m, 3H), 2.61-2.58 (m, 1H), 2.38-2.36 (m, 1H), 2.33 (s, 3H), 1.87-1.84 (m, 1H). 1.60-1.55 (m, 1H), 1.17 (t, $J$ = 7.05 Hz, 3H), 1.13 (t, $J$ = 7.05 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 138.6 (d, $J$ = 3.7 Hz), 137.3 (s), 129.1 (s), 127.6 (s), 75.1 (d, $J$ = 6.1 Hz), 68.5 (s), 63.6 (t, $J$ = 6.5 Hz), 55.1 (d, $J$ = 3.9 Hz), 36.0 (d, $J$ = 6.3 Hz), 23.5 (s), 21.2 (s), 16.1 (dd, $J_1$= 3.2 Hz, $J_2$= 7.4 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 23.0. HRMS calcd for $C_{16}H_{25}NaO_4PS^+$ [M+Na]$^+$, 367.1103; found, 367.1100.

***O,O*-diethyl *S*-((2-(4-fluorophenyl)tetrahydrofuran-2-yl)methyl) phosphorothioate (5h)**



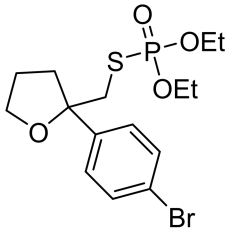
Yield: 20.8 mg, 60%. Yellow oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.55 (dd, $J_1$= 8.64 Hz, $J_2$= 5.28 Hz, 2H), 7.03 (t, $J$ = 8.64 Hz, 2H), 4.37-4.35 (m, 1H), 4.09 (d, $J$ = 12.12 Hz, 1H), 3.93-3.88 (m, 2H), 3.85-3.73 (m, 2H), 3.71-3.66 (m, 2H), 2.55-2.51 (m, 1H), 2.36-2.33 (m, 1H), 1.55-1.51 (m, 1H). 1.37-1.34 (m, 1H), 1.16 (dt, $J_1$= 7.08 Hz, $J_2$= 18.66 Hz, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 162.0 (d, $J$ = 247.4 Hz), 137.5 (s), 129.7 (t, $J$ = 8.2 Hz), 115.1 (t, $J$ = 21.2 Hz), 75.1 (d, $J$ = 4.7 Hz), 68.4 (s), 63.7 (dd, $J_1$= 2.2 Hz, $J_2$= 6.5 Hz), 54.4 (d, $J$ = 3.9 Hz), 36.5 (s), 23.3 (s), 16.1 (t, $J$ = 6.0 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 22.7. HRMS calcd for $C_{15}H_{22}FNaO_4PS^+$ $[M+Na]^+$, 371.0853; found, 371.0841.

***S*-((2-(4-chlorophenyl)tetrahydrofuran-2-yl)methyl) *O,O*-diethyl phosphorothioate (5i)**



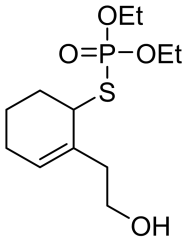
Yield: 27.3 mg, 75%. Yellow oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.52 (d, $J$ = 8.64 Hz, 2H), 7.32 (d, $J$ = 8.58 Hz, 2H), 4.39 (d, $J$ = 10.50 Hz, 1H), 4.08 (d, $J$ = 12.12 Hz, 1H), 3.95-3.89 (m, 2H), 3.85-3.80 (m, 1H), 3.79-3.74 (m, 1H), 3.72-3.68 (m, 2H), 2.52 (t, $J$ = 19.38 Hz, 1H), 2.37-2.34 (m, 1H). 1.82-1.78 (m, 1H), 1.57-1.52 (m, 1H), 1.20-1.15 (m, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 140.3 (s), 133.4 (s), 129.4 (s), 128.5 (s), 74.9 (d, $J$ = 4.6 Hz), 68.5 (s), 63.8 (dd, $J_1$= 1.6 Hz, $J_2$= 6.6 Hz), 54.4 (d, $J$ = 3.9 Hz), 36.4 (s), 23.4 (s), 16.1 (t, $J$ = 6.9 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 22.0. HRMS calcd for $C_{15}H_{22}ClNaO_4PS^+$ $[M+Na]^+$, 387.0557; found, 387.0562.

***S*-((2-(4-bromophenyl)tetrahydrofuran-2-yl)methyl) *O,O*-diethyl phosphorothioate (5j)**



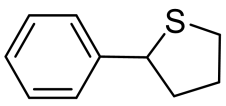
Yield: 32.6 mg, 80%. Yellow oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.57 (d, $J$ = 7.62 Hz, 1H), 7.46 (d, $J$ = 3.4 Hz, 1H), 7.34 (t, $J$ = 7.77 Hz, 1H), 7.26-7.24 (m, 1H), 4.35-4.34 (m, 1H), 4.14 (d, $J$ = 12.12 Hz, 1H), 3.95-3.85 (m, 2H), 3.83-3.73 (m, 2H), 3.71-3.65 (m, 2H), 2.60-2.48 (m, 1H), 2.39-2.32 (m, 1H), 1.88-1.82 (m, 1H). 1.59-1.53 (m, 1H), 1.12-1.10 (m, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 131.5 (s), 129.8 (s), 128.4 (s), 127.7 (s), 75.0 (d, $J$ = 5.1 Hz), 68.5 (s), 63.8 (d, $J$ = 6.6 Hz), 55.0 (d, $J$ = 4.0 Hz), 36.1 (s), 23.4 (s), 16.1 (d, $J$ = 7.6 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 22.9. HRMS calcd for $C_{15}H_{22}BrNaO_4PS^+$ $[M+Na]^+$, 431.0052; found, 431.0055.

***O,O*-diethyl *S*-(2-(2-hydroxyethyl)cyclohex-2-en-1-yl) phosphorothioate (5k)**



Yield: 20.6 mg, 70%. Brown oil. $^{1}$H NMR (600 MHz, $CDCl_3$) δ (ppm) 5.73 (s, 1H), 4.20-4.10 (m, 5H), 3.42-3.39 (d, $J$ = 13.08 Hz, 2H), 2.05-2.01 (m, 4H), 1.65-1.62 (m, 2H), 1.57-1.54 (m, 2H), 1.35 (t, $J$ = 7.11 Hz, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ (ppm) 133.4 (d, $J$ = 6.6 Hz) 126.6 (s), 63.6 (d, $J$ = 5.6 Hz), 38.7 (s), 38.6 (s), 27.2 (s), 25.5 (s), 22.8 (s), 22.2 (s), 16.3 (d, $J$ = 7.6 Hz). $^{31}$P NMR (242 MHz, $CDCl_3$) δ (ppm) 27.9. HRMS calcd for $C_{12}H_{23}NaO_4PS^+$ $[M+Na]^+$, 317.0947; found, 317.0941.

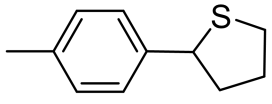
**2-phenyltetrahydrothiophene (6a)**



Yield: 13.1 mg, 80 %. Colorless oil. 1H NMR (600 MHz, CDCl3) δ (ppm 7.42 (d, J = 7.20 Hz, 2H), 7.30 (t, J = 7.45 Hz, 2H), 7.22 (t, J = 7.38 Hz, 1H), 4.54-4.50 (m, 1H), 3.16-3.13 (m, 1H), 3.04-2.99 (m, 1H), 2.42-2.36 (m, 1H), 2.31-2.25(m, 1H), 2.04-1.90 (m, 2H). 13C NMR (150 MHz, CDCl3) δ (ppm) 143.1 (s), 128.5 (s), 127.7 (s), 127.1 (s), 52.9 (s), 40.6 (s), 33.6 (s), 31.2 (s). HRMS Calcd for C10H13S+ [M+H]+ 165.0732, found 165.0739.

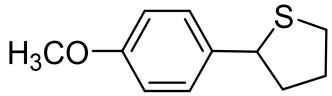
**2-(p-tolyl)tetrahydrothiophene (6b)**

Yield: 15.0 mg, 84 %. Colorless oil. 1H NMR (600 MHz, CDCl3) δ (ppm) 7.31 (d, J = 7.98 Hz, 2H),



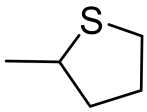
7.12 (d, J = 7.92 Hz, 2H), 4.51-4.49 (m, 1H), 3.18-3.14 (m, 1H), 3.03-3.00 (m, 1H), 2.39-2.35 (m, 1H), 2.33 (s, 3H), 2.29-2.26(m, 1H), 2.03-1.91 (m, 2H). 13C NMR (150 MHz, CDCl3) δ (ppm) 140.0 (s), 136.7 (s), 129.2 (s), 127.6 (s), 52.6 (s), 40.6 (s), 33.6 (s), 31.2 (s), 21.1 (s). HRMS Calcd for C11H15S+ [M+H]+ 179.0889, found 179.0887.

**2-(4-methoxyphenyl)tetrahydrothiophene (6c)**



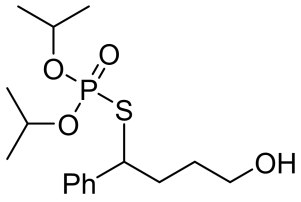
Yield: 15.1 mg, 78 %. Colorless oil. 1H NMR (600 MHz, CDCl3) δ (ppm) 7.33 (d, J = 8.72 Hz, 2H), 6.84 (d, J = 8.67 Hz, 2H), 4.48 (dd, J1 = 6.09 Hz, J2 = 8.73 Hz, 1H), 3.79 (s, 3H), 3.17-3.12 (m, 1H), 3.02-2.98 (m, 1H), 2.38-2.33 (m, 1H), 2.28-2.24(m, 1H), 2.01-1.95 (m, 1H), 1.94-1.88 (m, 1H). 13C NMR (150 MHz, CDCl3) δ (ppm) 158.7 (s), 135.0 (s), 128.7 (s), 113.9 (s), 55.4 (s), 52.4 (s), 40.7 (s), 33.5 (s), 31.1 (s). HRMS Calcd for C11H14NaOS+ [M+Na]+ 217.0658, found 217.0652.

**2-methyltetrahydrothiophene (6d)**



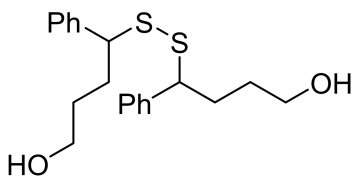
Yield: 7.1 mg, 70 %. Colorless oil. 1H NMR (600 MHz, CDCl3) δ (ppm) 3.33-3.30 (m, 1H), 2.83-2.78 (m, 1H), 2.72-2.68 (m, 1H), 1.97-1.92 (m, 2H), 1.80-1.73 (m, 1H), 1.42-1.37 (m, 1H), 1.17 (d, J = 6.66 Hz, 3H).13C NMR (150 MHz, CDCl3) δ (ppm) 43.1 (s), 40.6 (s), 32.6 (s), 30.1 (s), 22.6 (s).

***T*-(4-hydroxy-1-phenylbutyl) O,O-diisopropyl phosphorothioate (3ac)**



Yield: 26.6 mg, 77 %. Yellow oil. 1H NMR (600 MHz, CDCl3) δ (ppm) 7.34-7.29 (m, 4H), 7.25-7.22 (m, 1H), 4.70-4.64 (m, 1H), 4.49-4.43 (m, 1H), 4.39-4.35 (m, 1H), 3.66-3.59 (m, 2H), 2.27-2.21 (m, 1H), 2.12-2.07 (m, 1H), 1.55-1.50 (m, 2H), 1.33 ((d, J = 6.18 Hz, 3H), 1.22 (d, J = 6.21 Hz, 3H), 1.18 (t, J = 6.91 Hz, 6H). 3C NMR (150 MHz, CDCl3) δ (ppm) 142.2 ((d, J = 5.4 Hz), 128.8 (s), 127.8 (s), 127.7 (s), 73.0 ((d, J = 6.5 Hz), 72.8 ((d, J = 6.6 Hz), 61.8 (s), 50.5 ((d, J = 3.2 Hz), 35.0 ((d, J = 6.7 Hz), 30.4 (s), 24.0 ((d, J = 4.2 Hz), 23.7 ((d, J = 4.3 Hz), 23.6 ((d, J = 5.5 Hz), 23.5 ((d, J = 5.5 Hz). 31P NMR (242 MHz, CDCl3) δ (ppm) 23.96. HRMS Calcd for C16H28O4PS+ [M+H]+ 347.1440, found 347.1436.

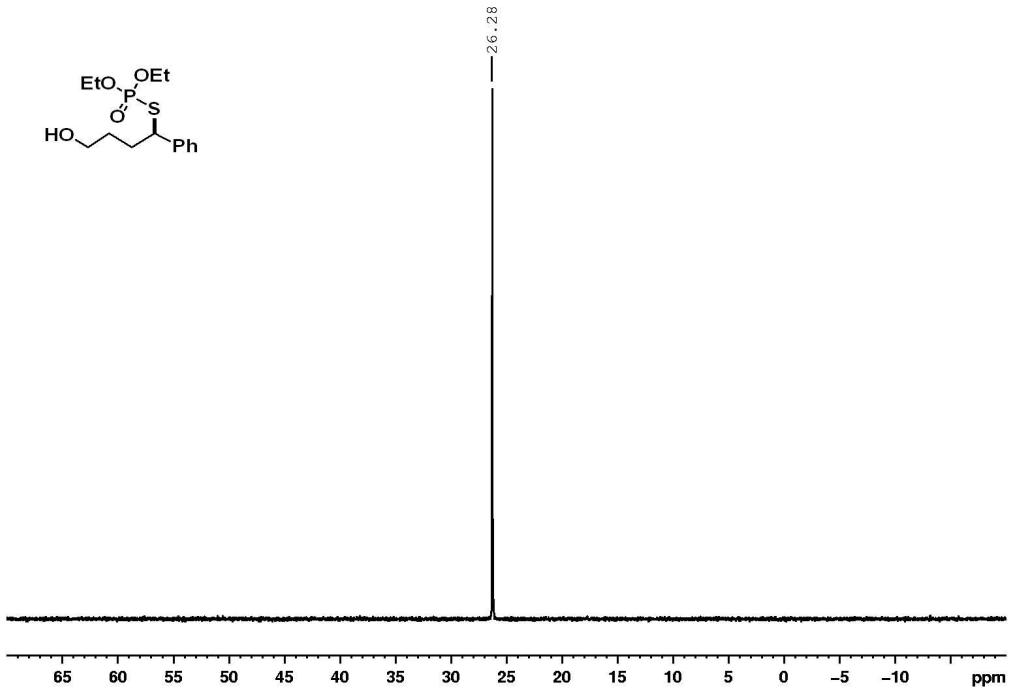
**4,4'-disulfanediylbis(4-phenylbutan-1-ol) (7a)**



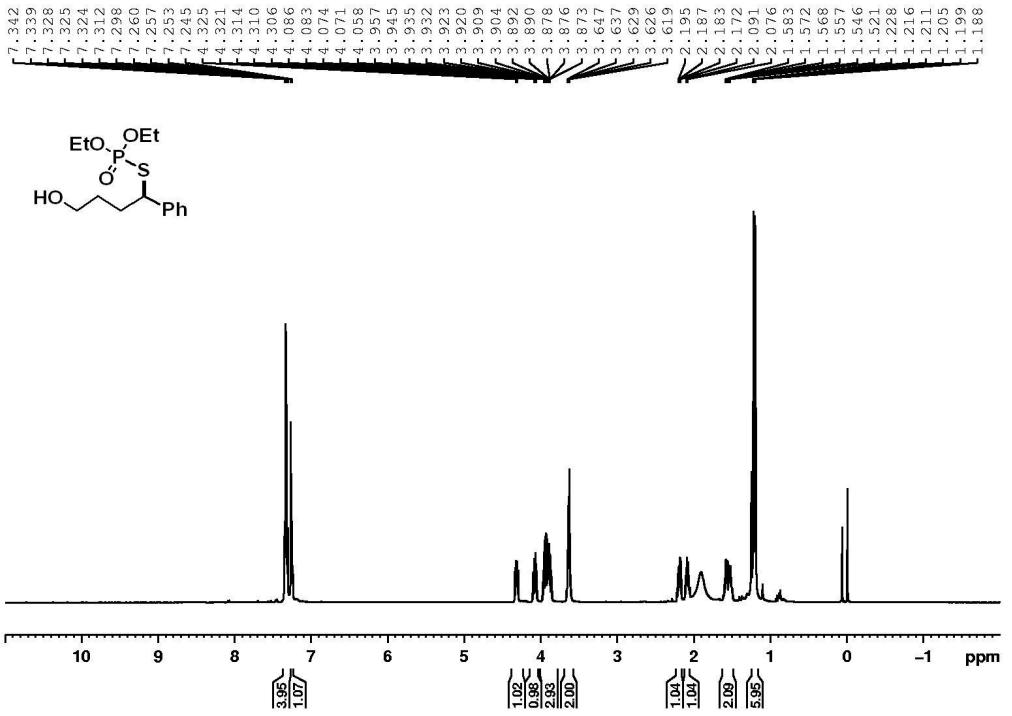
Yield: 29.0 mg, 80 %. Yellow oil.1H NMR (600 MHz, CDCl3) δ (ppm) 7.34-7.27 (m, 6H), 7.20-7.15 (m, 4H), 3.56-3.53 (m,4H) 3.53 (dd, J1 = 6.15 Hz, J2 = 9.21 Hz, 1H), 3.29 (dd, J1 = 6.64 Hz, J2 = 9.72 Hz, 1H), 2.12-2.06 (m, 1H), 2.02-1.97 (m, 1H), 1.89-1.80 (m, 2H), 1.45-1.38 (m, 4H). 13C NMR (150 MHz, CDCl3) δ (ppm) 141.4 (s), 141.2(s), 128.6 (s), 128.5 (s), 128.4 (s), 127.7 (s), 127.6 (s), 62.5 (s), 62.4 (s), 55.1 (s), 54.9 (s), 31.0 (s), 30.9 (s), 30.8 (s), 30.7 (s). HRMS Calcd for C20H27O2S2+ [M+H]+ 363.1447, found 363.1442.

## $^{31}$P NMR, $^{1}$H NMR and $^{13}$C NMR Spectra for All Compounds

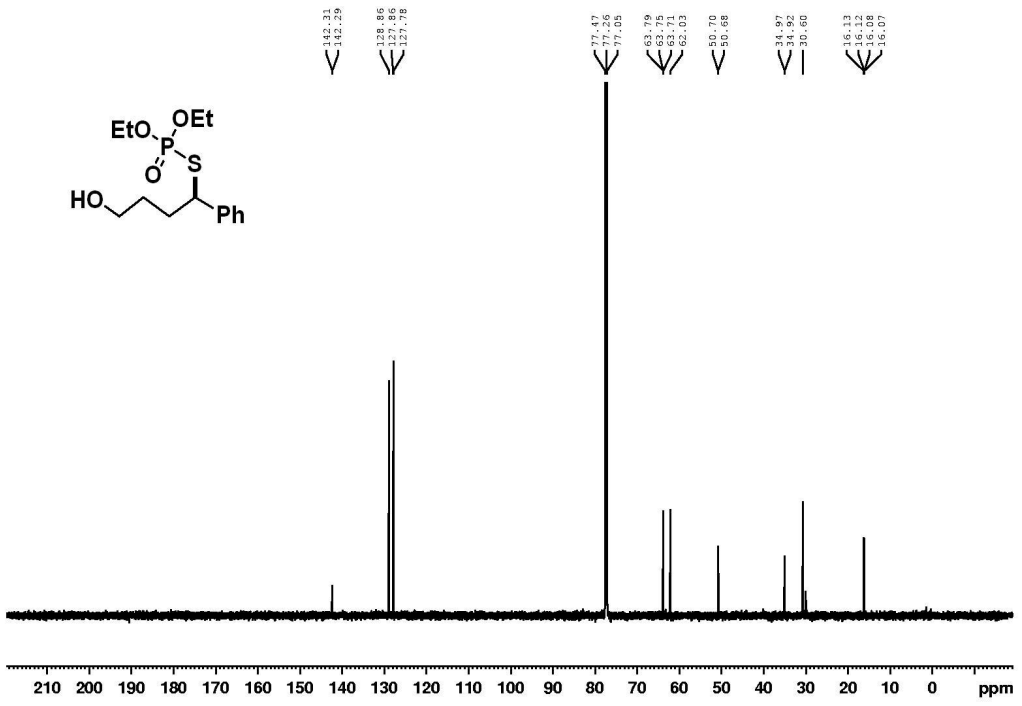
**3a**



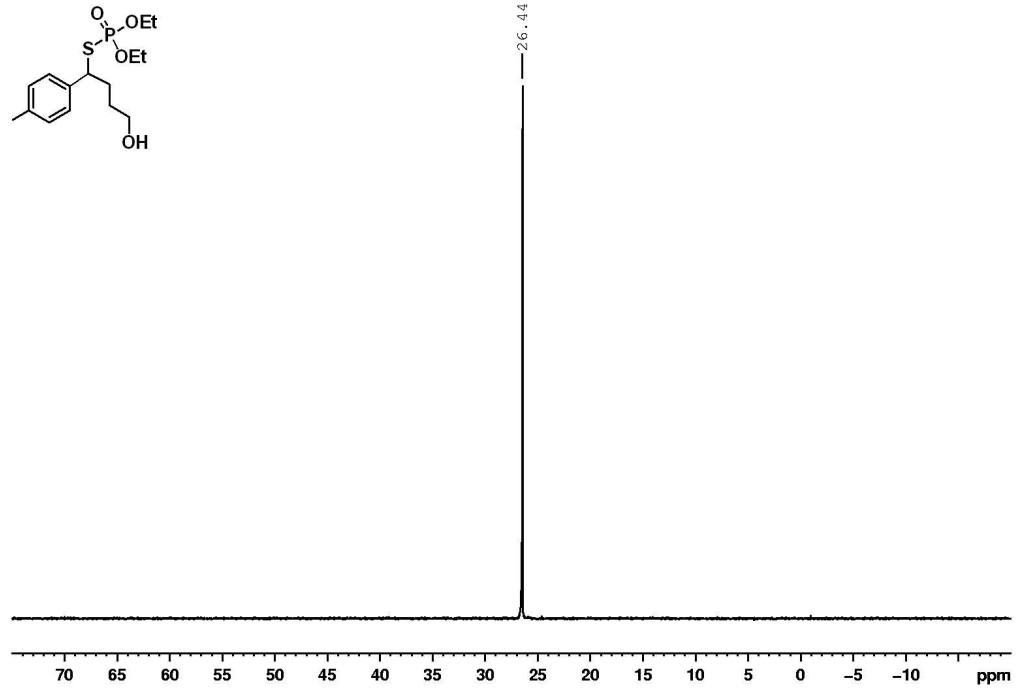

**3a**



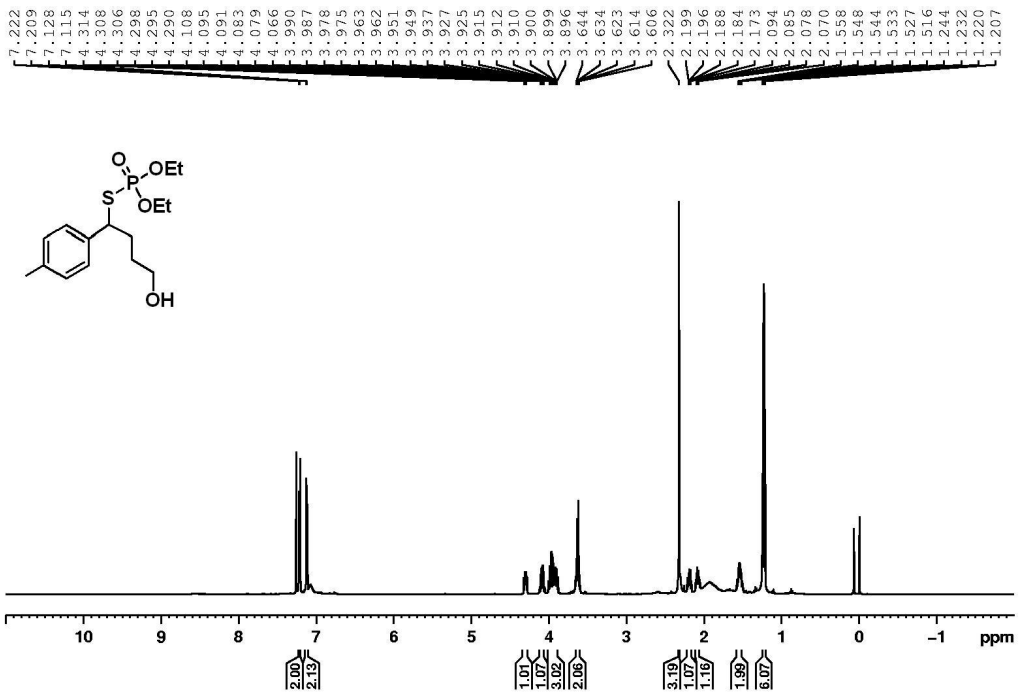
**3a**


142.31
142.29
128.86
127.86
127.78
77.47
77.26
77.05
63.79
63.75
63.71
62.03
50.70
50.68
34.97
34.92
30.60
16.13
16.12
16.08
16.07
EtO
OEt
P
O
S
HO
Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

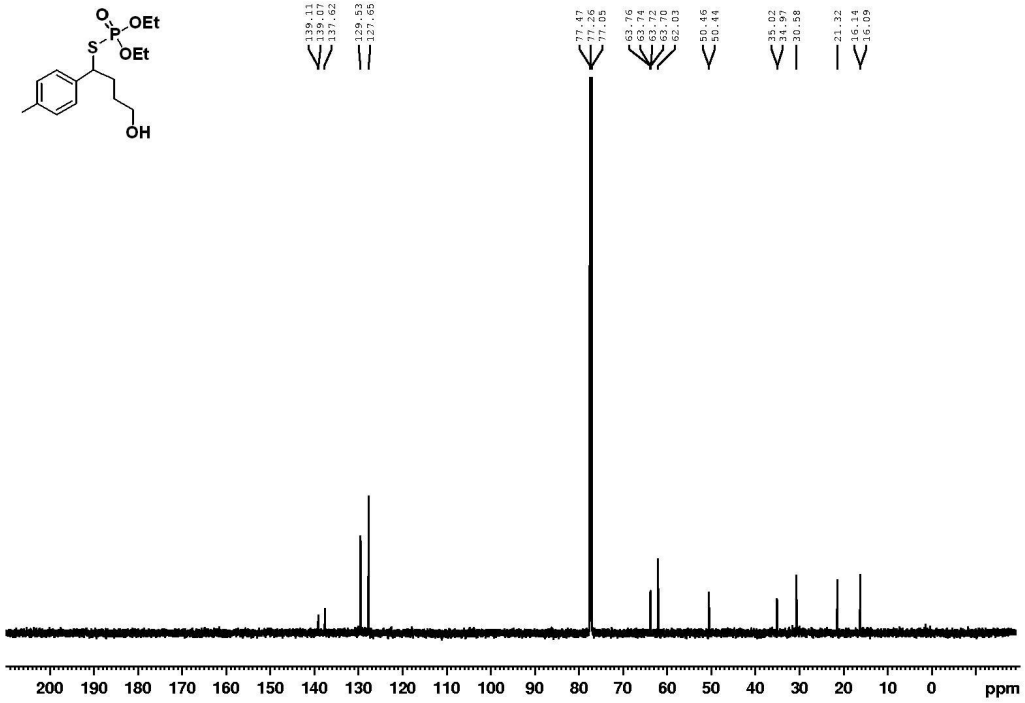
**3b**


O
OEt
P
S
OEt
OH
26.44
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

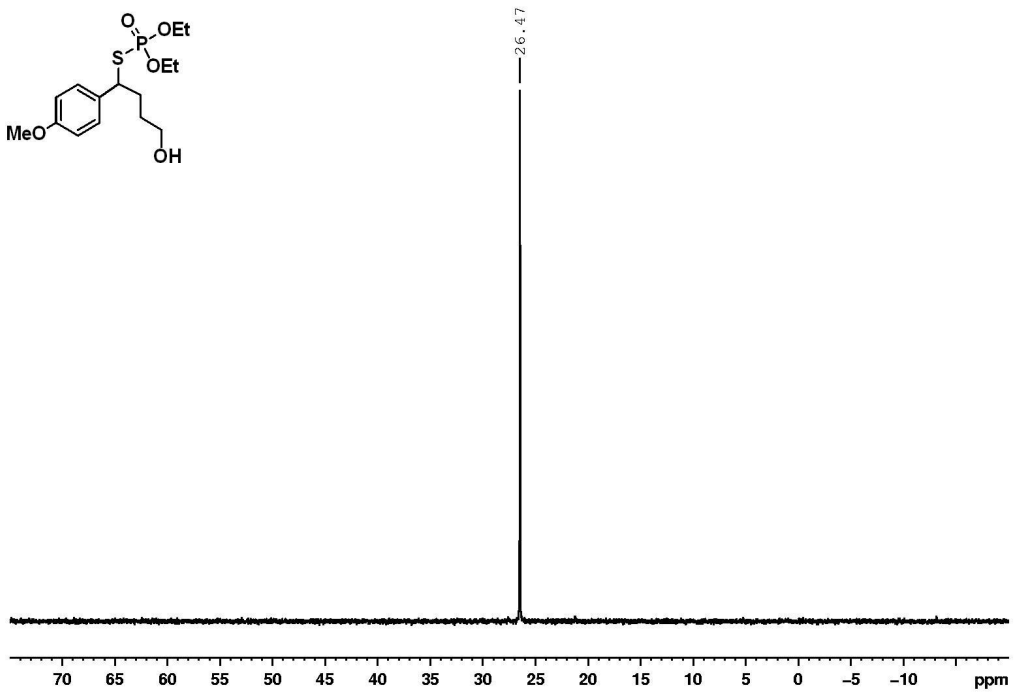
**3b**


7.222
7.209
7.128
7.115
4.314
4.308
4.306
4.298
4.295
4.290
4.108
4.095
4.091
4.083
4.079
4.066
3.990
3.987
3.978
3.975
3.963
3.962
3.951
3.949
3.937
3.927
3.925
3.915
3.912
3.910
3.900
3.899
3.896
3.644
3.634
3.623
3.614
3.606
2.322
2.199
2.196
2.188
2.184
2.173
2.094
2.085
2.078
2.070
1.558
1.548
1.544
1.533
1.527
1.516
1.244
1.232
1.220
1.207
OEt
OEt
OH
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
2.00
2.13
1.01
1.07
3.02
2.06
3.19
1.07
1.16
1.99
6.07

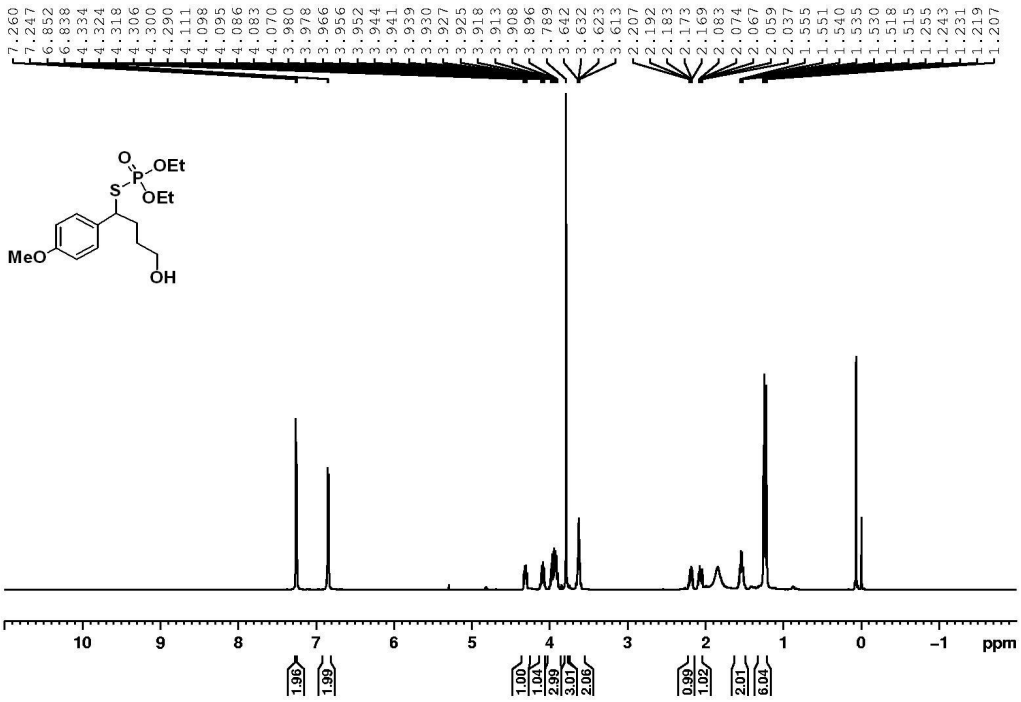
**3b**


OEt
OEt
OH
139.11
139.07
137.62
129.53
127.65
77.47
77.26
77.05
63.76
63.74
63.72
63.70
62.03
50.46
50.44
35.02
34.97
30.58
21.32
16.14
16.09
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

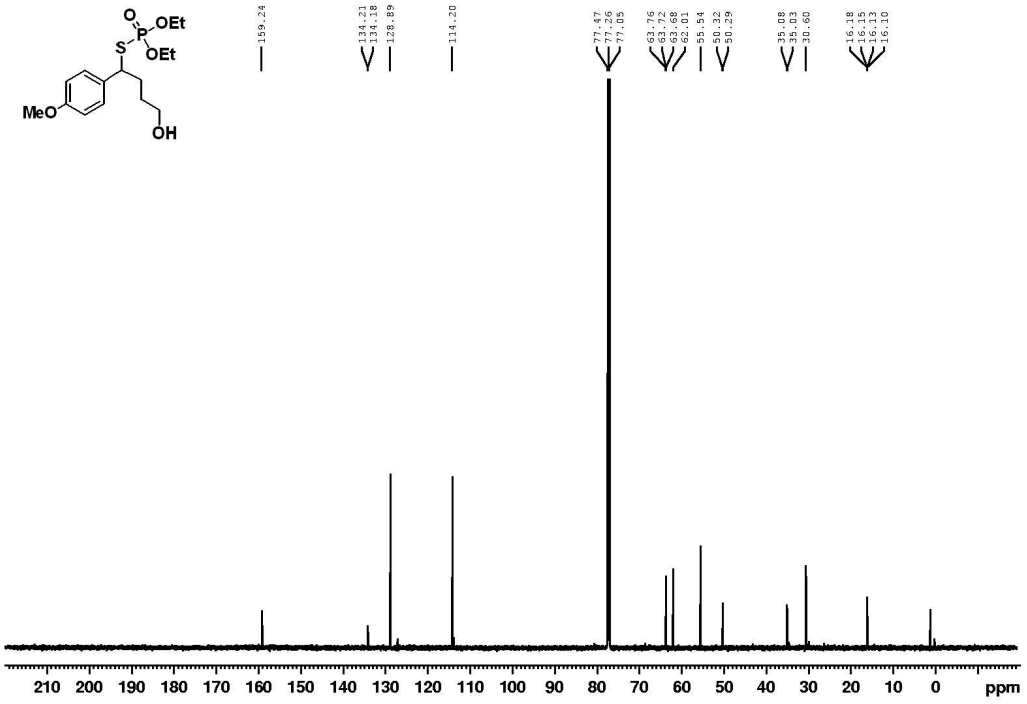
**3c**


O
OEt
P
S
OEt
MeO
OH
26.47
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

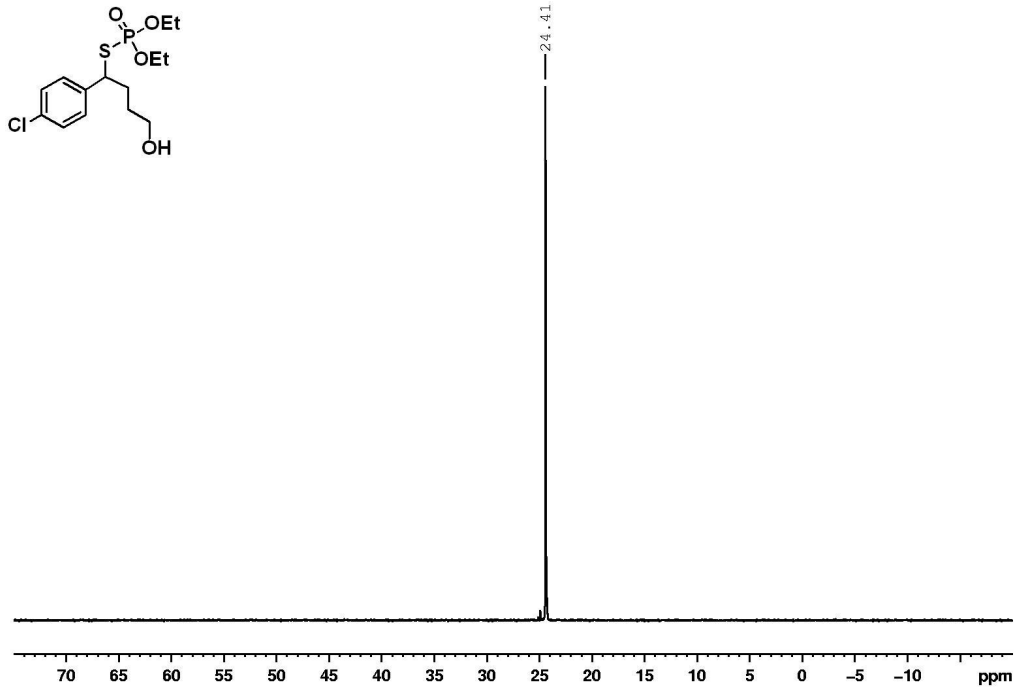
**3c**


7.260 7.247 6.852 6.838 4.334 4.324 4.318 4.306 4.300 4.290 4.111 4.098 4.095 4.086 4.083 4.070 3.980 3.978 3.966 3.956 3.952 3.944 3.941 3.939 3.930 3.927 3.925 3.918 3.913 3.908 3.896 3.789 3.642 3.632 3.623 3.613 2.207 2.192 2.183 2.173 2.169 2.083 2.074 2.067 2.059 2.037 1.555 1.551 1.540 1.535 1.530 1.518 1.515 1.255 1.243 1.231 1.219 1.207
O
OEt
P
S
OEt
MeO
OH
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
1.96 1.99 1.00 1.04 2.99 3.01 2.06 0.99 1.02 2.01 6.04

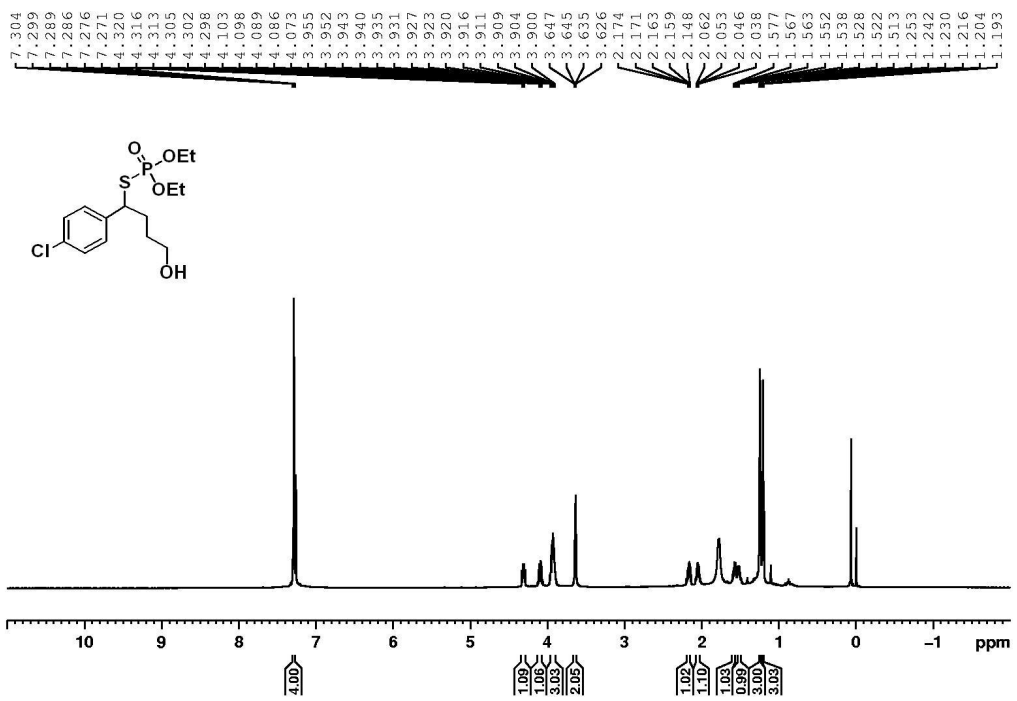
**3c**


159.24
134.21
134.18
128.89
114.20
77.47
77.26
77.05
63.76
63.72
63.68
62.01
55.54
50.32
50.29
35.08
35.03
30.60
16.18
16.15
16.13
16.10
OEt
OEt
MeO
OH
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

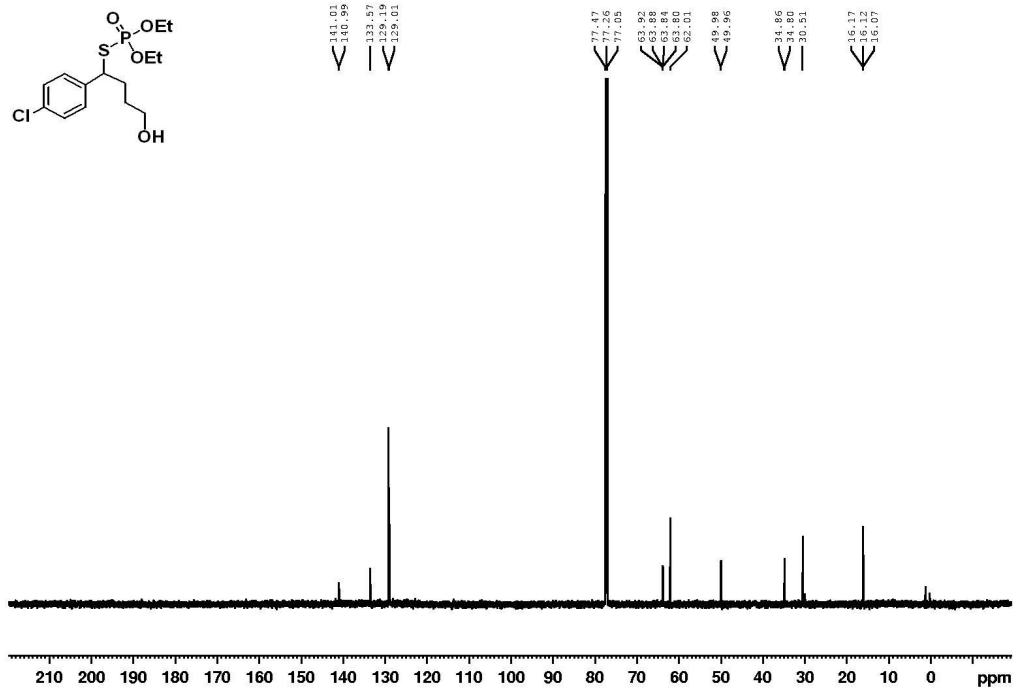
**3d**


24.41
OEt
OEt
Cl
OH
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

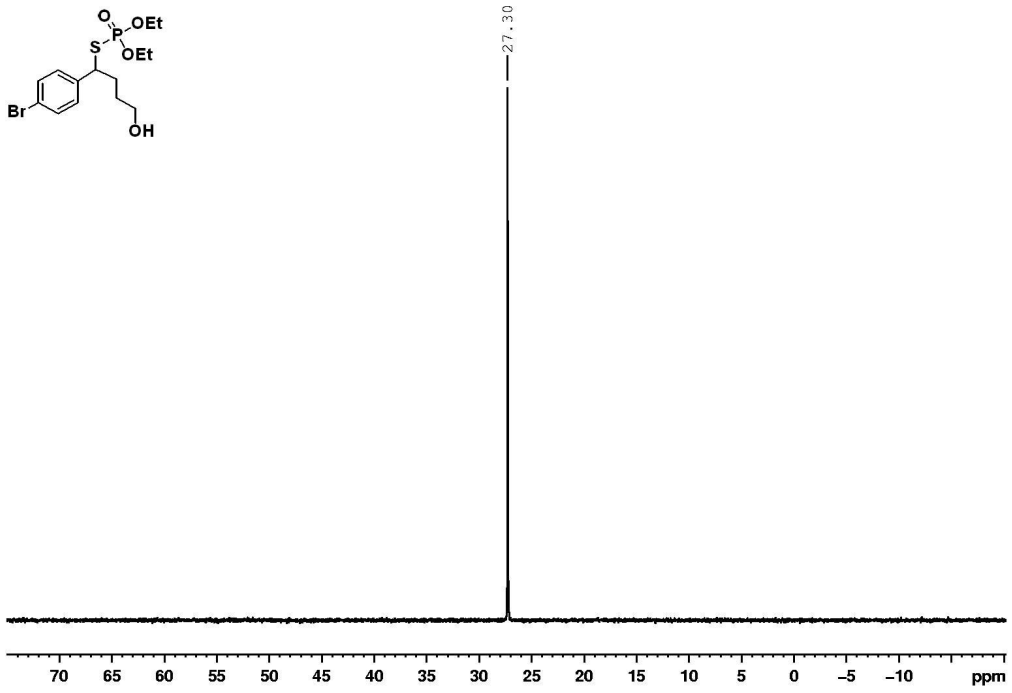
**3d**


7.304
7.299
7.289
7.286
7.276
7.271
4.320
4.316
4.313
4.305
4.302
4.298
4.103
4.098
4.089
4.086
4.073
3.955
3.952
3.943
3.940
3.935
3.931
3.927
3.923
3.920
3.916
3.911
3.909
3.904
3.900
3.647
3.645
3.635
3.626
2.174
2.171
2.163
2.159
2.148
2.062
2.053
2.046
2.038
1.577
1.567
1.563
1.552
1.538
1.528
1.522
1.513
1.253
1.242
1.230
1.216
1.204
1.193
O
OEt
P
S
OEt
Cl
OH
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
4.00
1.09
1.06
3.03
2.05
1.02
1.10
1.03
0.99
3.00
3.03

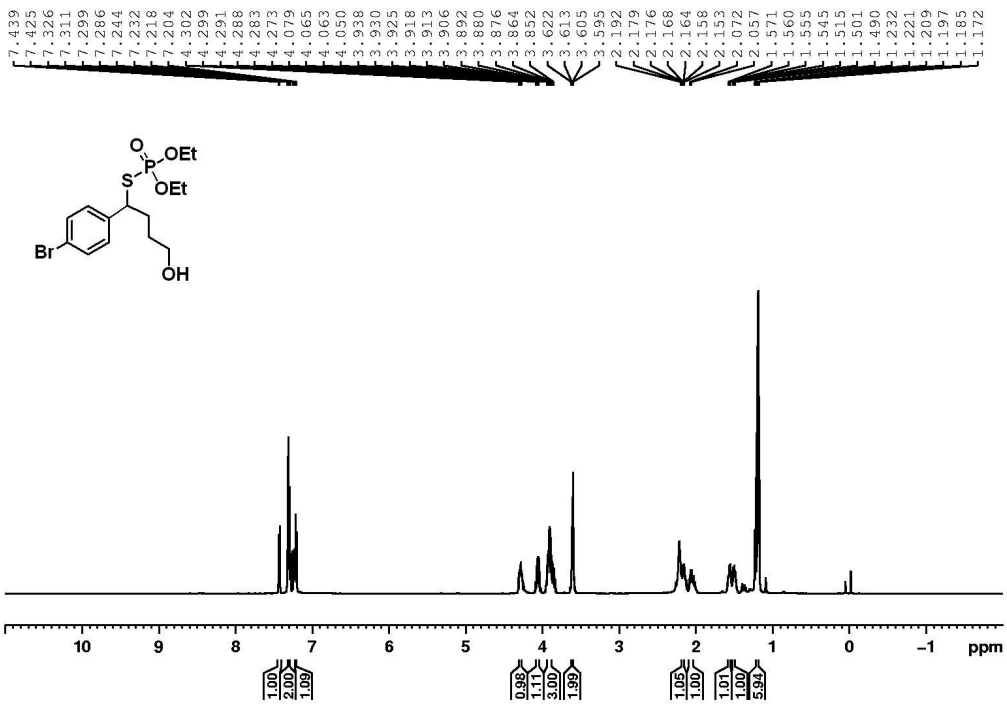
**3d**


O
OEt
P
S
OEt
Cl
OH
141.01
140.99
133.57
129.39
129.01
77.47
77.26
77.05
63.92
63.88
63.84
62.80
62.01
49.98
49.96
34.86
34.80
30.51
16.17
16.12
16.07
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

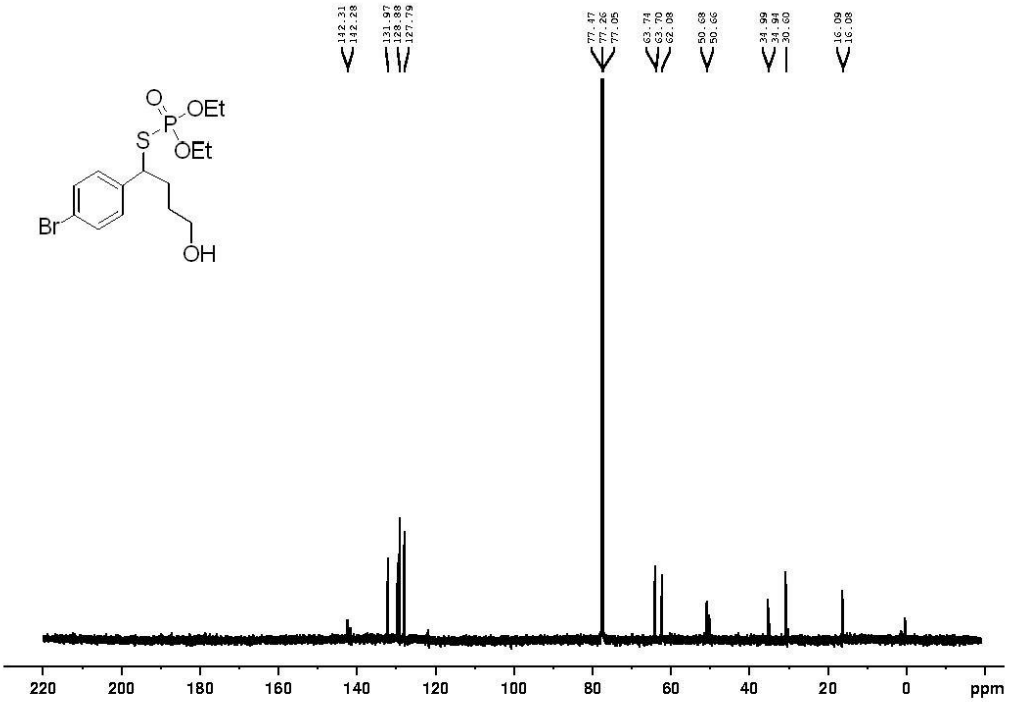
**3e**


27.30
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

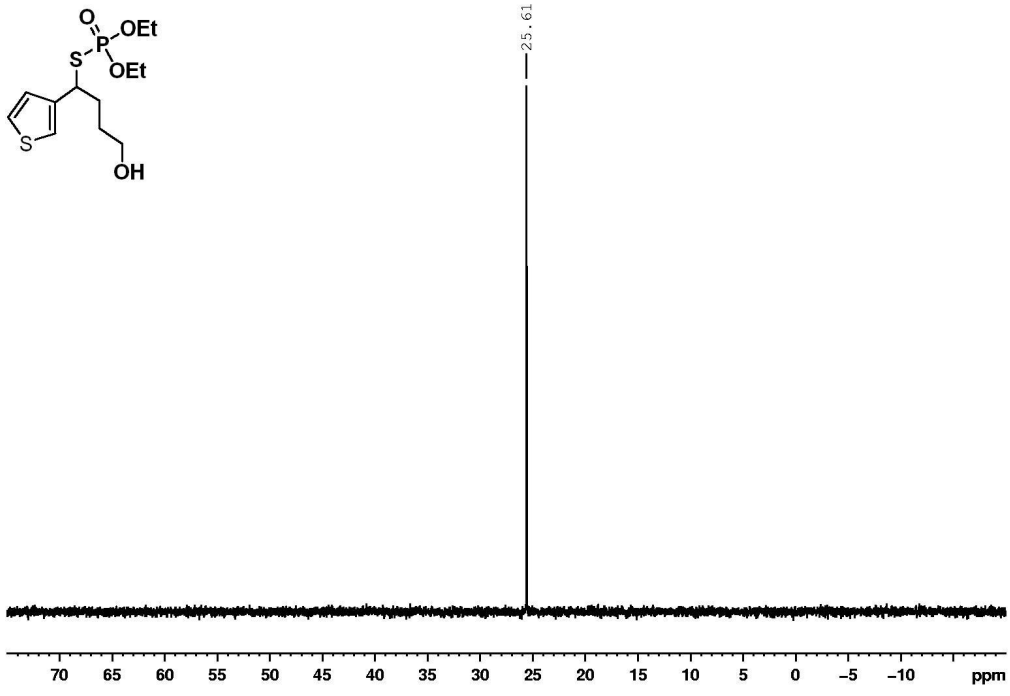
**3e**


7.439 7.425 7.326 7.311 7.299 7.286 7.244 7.232 7.218 7.204 4.302 4.299 4.291 4.288 4.283 4.273 4.079 4.065 4.063 4.050 3.938 3.930 3.925 3.918 3.913 3.906 3.892 3.880 3.876 3.864 3.852 3.622 3.613 3.605 3.595 2.192 2.179 2.176 2.168 2.164 2.158 2.153 2.072 2.057 1.571 1.560 1.555 1.545 1.515 1.501 1.490 1.232 1.221 1.209 1.197 1.185 1.172
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
1.00 2.00 1.09 0.98 1.11 3.00 1.99 1.05 1.00 1.01 1.00 5.94

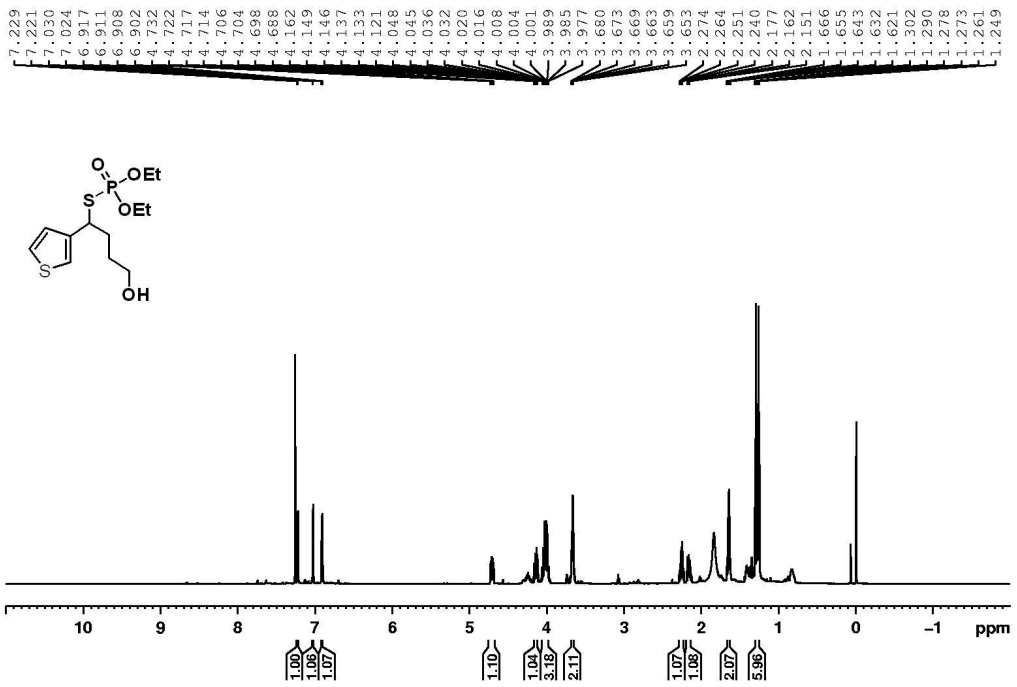
**3e**


142.31
142.28
131.97
128.88
127.79
77.47
77.26
77.05
63.74
63.70
62.08
50.68
50.66
34.99
34.94
30.60
16.09
16.08
OEt
OEt
S
P
O
Br
OH
220
200
180
160
140
120
100
80
60
40
20
0
ppm

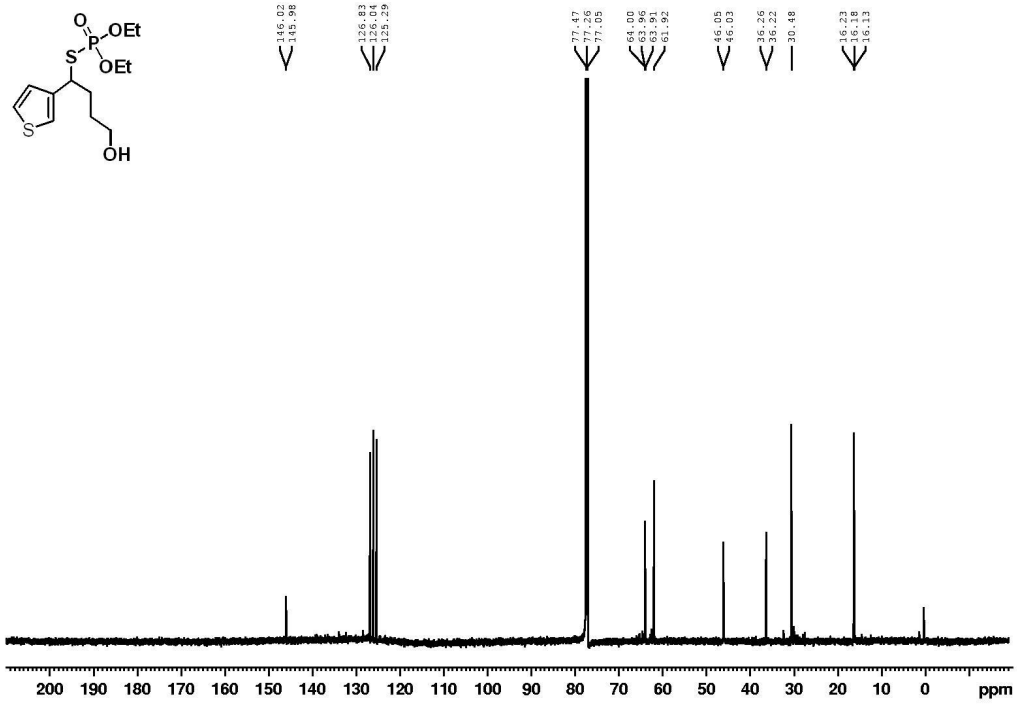
**3f**


25.61
OEt
OEt
S
P
O
S
OH
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
−5
−10
ppm

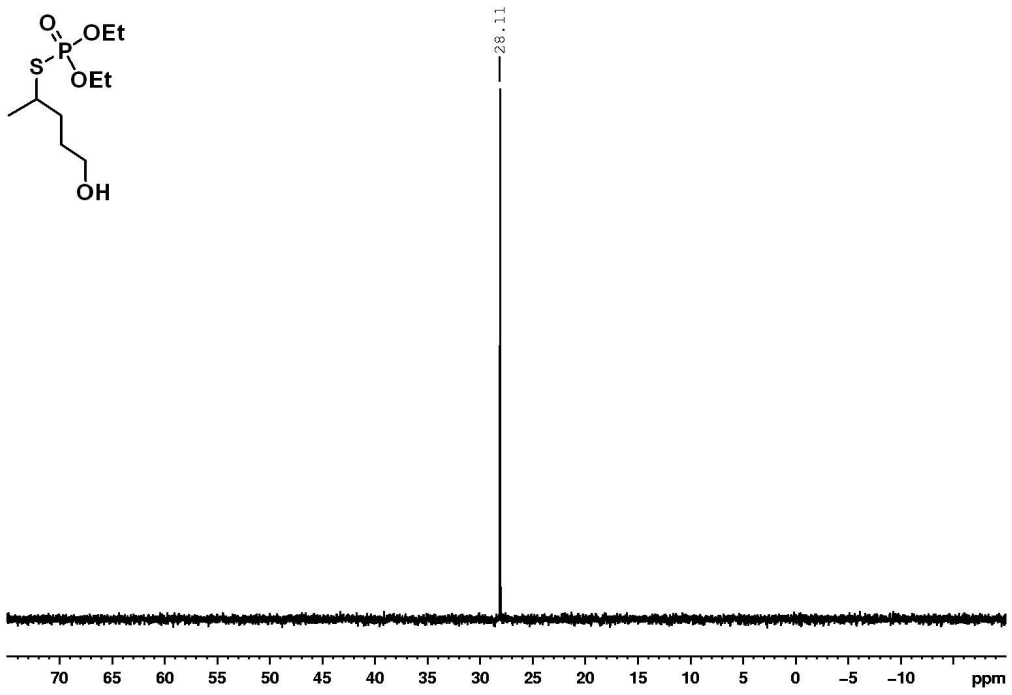
**3f**


7.229
7.221
7.030
7.024
6.917
6.911
6.908
6.902
4.732
4.722
4.717
4.714
4.706
4.704
4.698
4.688
4.162
4.149
4.146
4.137
4.133
4.121
4.048
4.045
4.036
4.032
4.020
4.016
4.008
4.004
4.001
3.989
3.985
3.977
3.680
3.673
3.669
3.663
3.659
3.653
2.274
2.264
2.251
2.240
2.177
2.162
2.151
1.666
1.655
1.643
1.632
1.621
1.302
1.290
1.278
1.273
1.261
1.249
OEt
OEt
OH
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
1.00
1.06
1.07
1.10
1.04
3.18
2.11
1.07
1.08
2.07
5.96

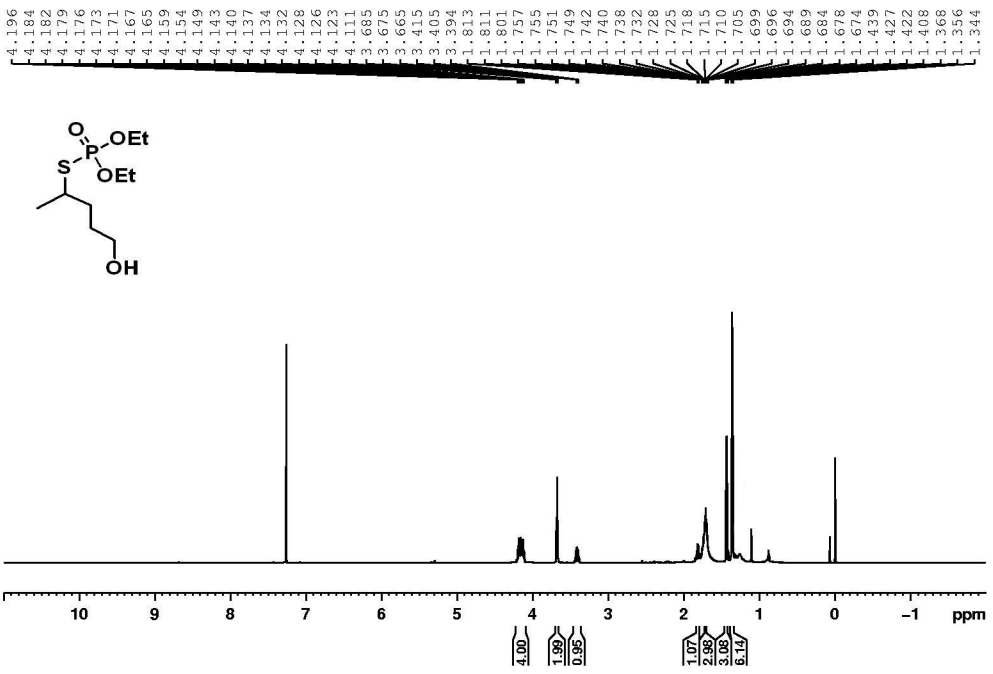
**3f**


146.02
145.98
126.83
126.04
125.29
77.47
77.26
77.05
64.00
63.96
63.91
61.92
46.05
46.03
36.26
36.22
30.48
16.23
16.18
16.13
OEt
OEt
OH
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

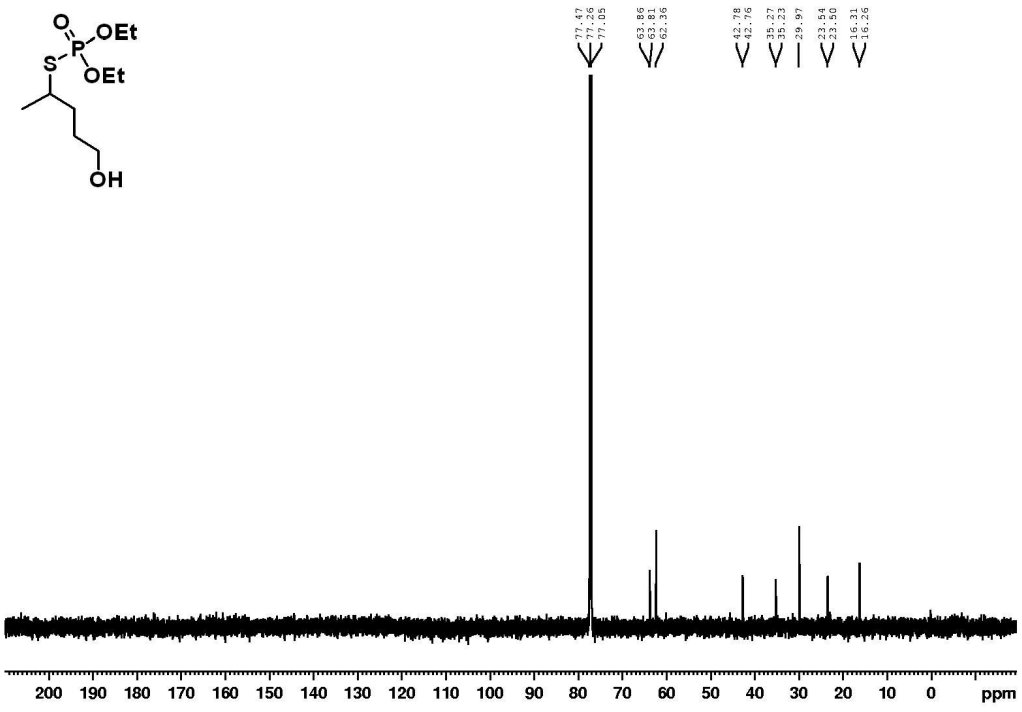
**3g**


28.11
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

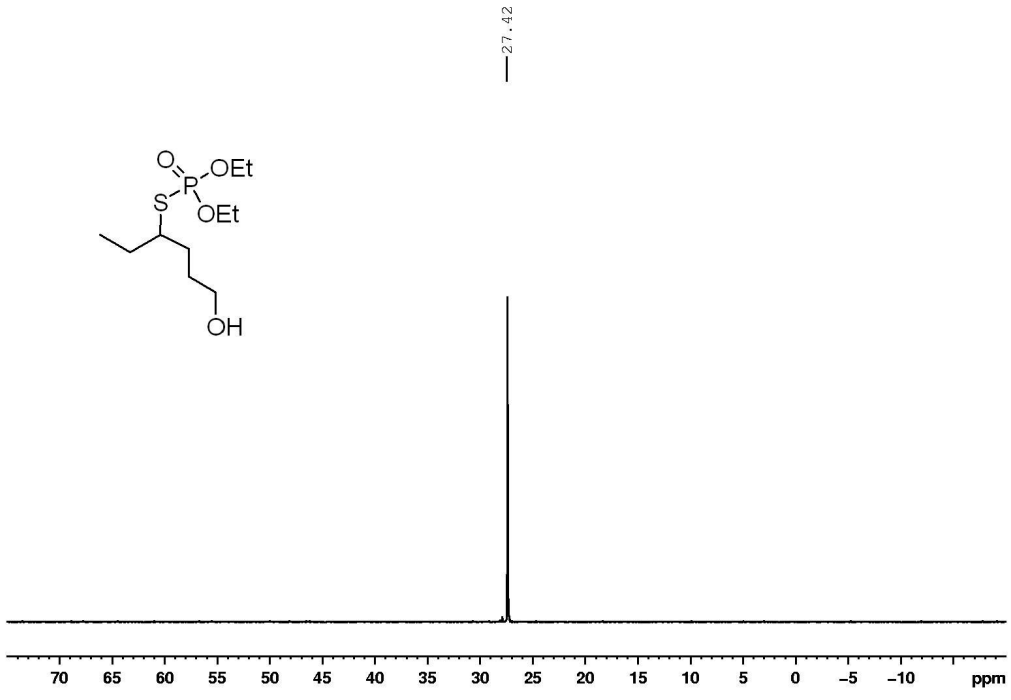
**3g**


4.196 4.184 4.182 4.179 4.176 4.173 4.171 4.167 4.165 4.159 4.154 4.149 4.143 4.140 4.137 4.134 4.132 4.128 4.126 4.123 4.111 3.685 3.675 3.665 3.415 3.405 3.394 1.813 1.811 1.801 1.757 1.755 1.751 1.749 1.742 1.740 1.738 1.732 1.728 1.725 1.718 1.715 1.710 1.705 1.699 1.696 1.694 1.689 1.684 1.678 1.674 1.439 1.427 1.422 1.408 1.368 1.356 1.344
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
4.00 1.99 0.95 1.07 2.98 3.08 6.14

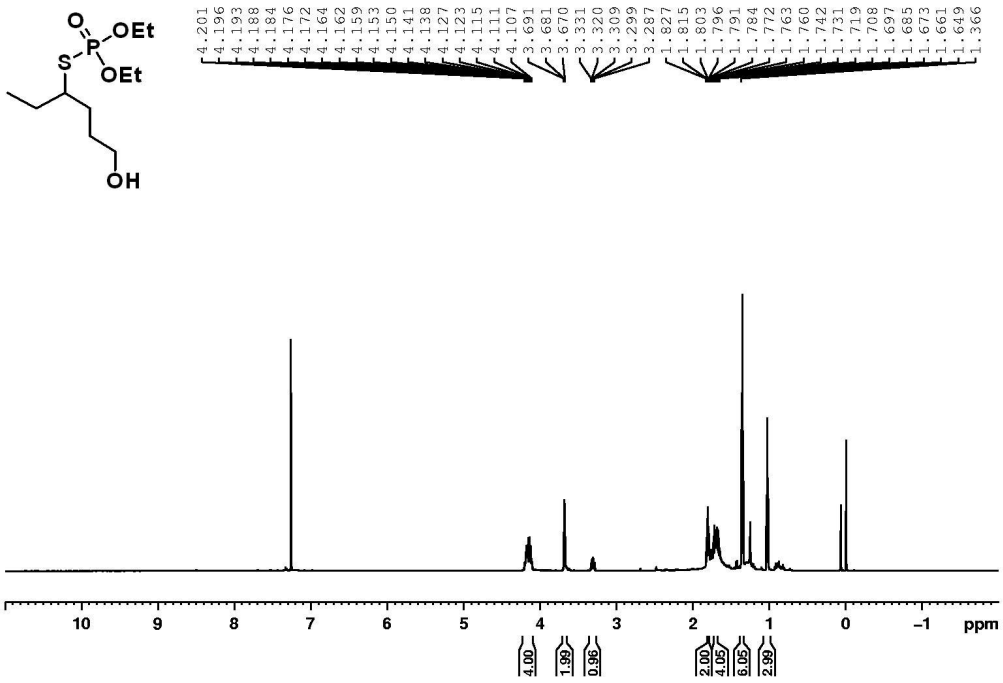
**3g**


O
OEt
P
S
OEt
OH
77.47
77.26
77.05
63.86
63.81
62.36
42.78
42.76
35.27
35.23
29.97
23.54
23.50
16.31
16.26
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

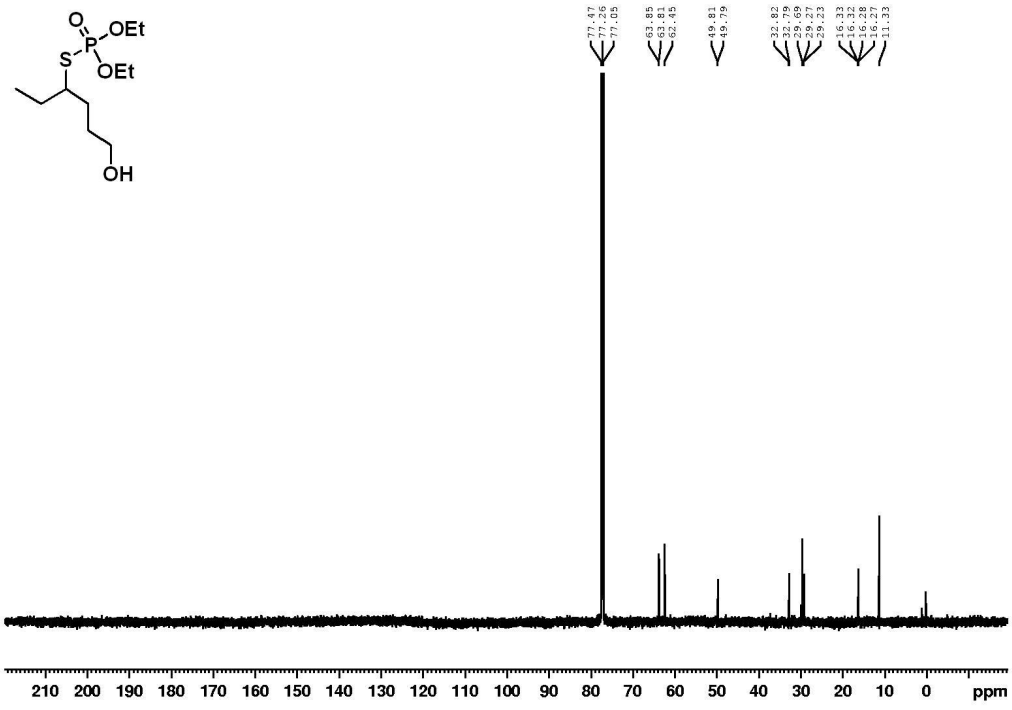
**3h**


27.42
O
OEt
P
S
OEt
OH
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

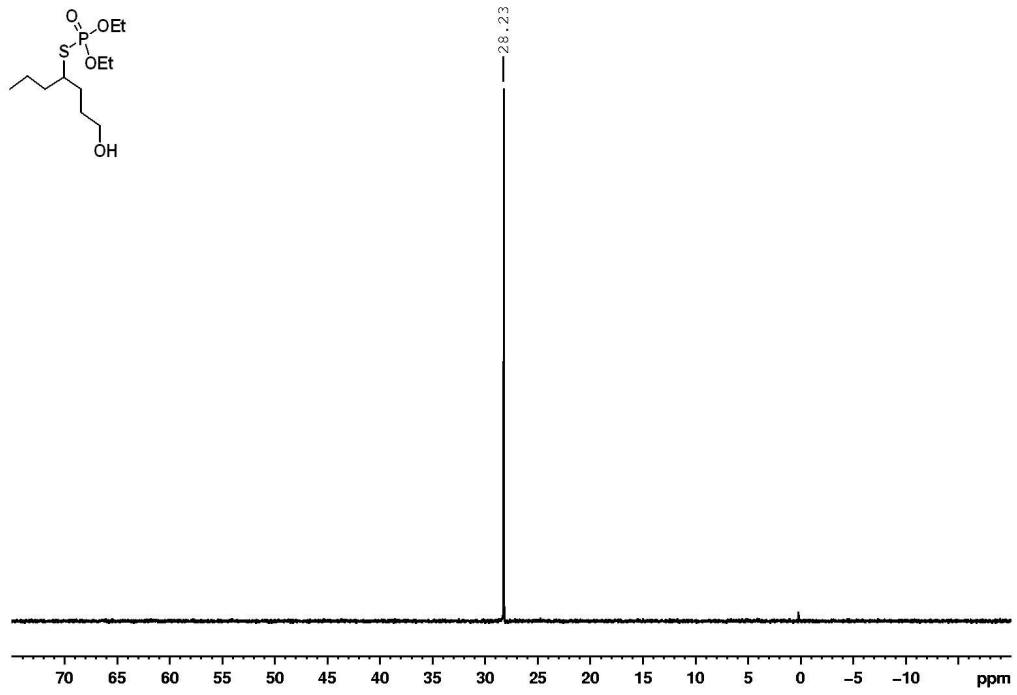
**3h**


4.201
4.196
4.193
4.188
4.184
4.176
4.172
4.164
4.162
4.159
4.153
4.150
4.141
4.138
4.127
4.123
4.115
4.111
4.107
3.691
3.681
3.670
3.331
3.320
3.309
3.299
3.287
1.827
1.815
1.803
1.796
1.791
1.784
1.772
1.763
1.760
1.742
1.731
1.719
1.708
1.697
1.685
1.673
1.661
1.649
1.366
OEt
OEt
OH
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
4.00
1.99
0.96
2.00
4.05
6.05
2.99

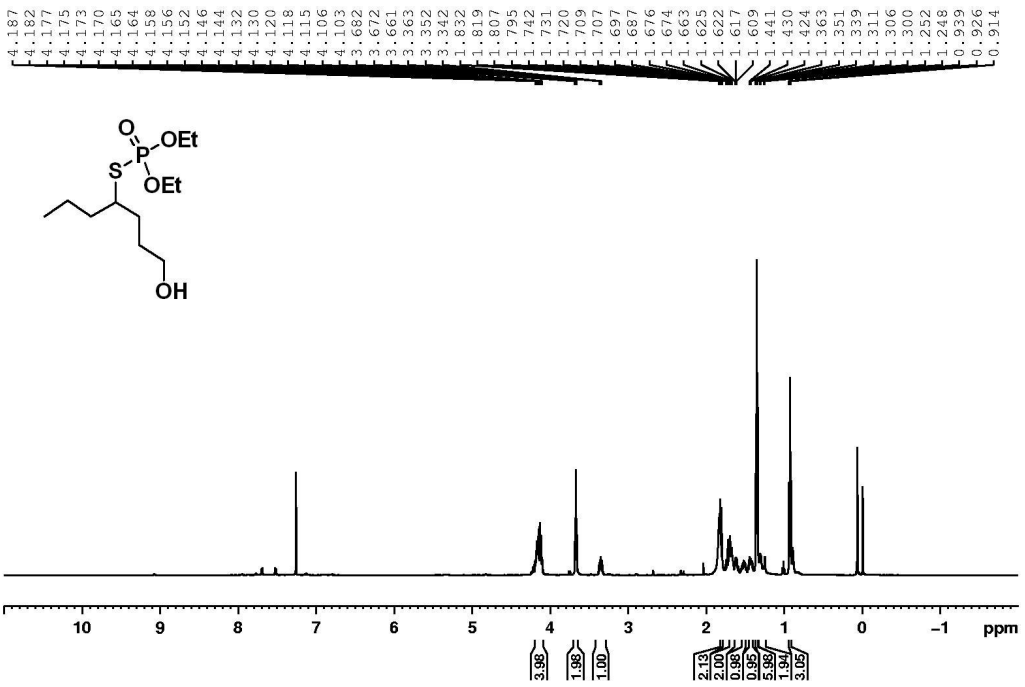
**3h**


77.47
77.26
77.05
63.85
63.81
62.45
49.81
49.79
33.82
33.79
29.69
29.27
29.23
16.33
16.32
16.28
16.27
11.33
OEt
OEt
OH
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

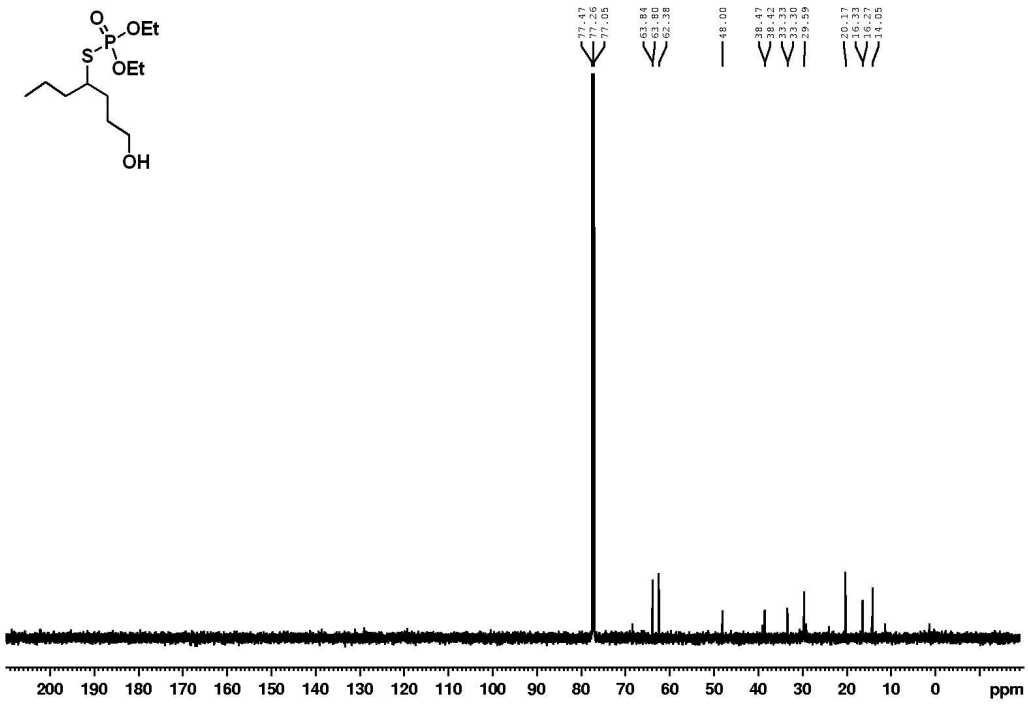
**3i**


28.23
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

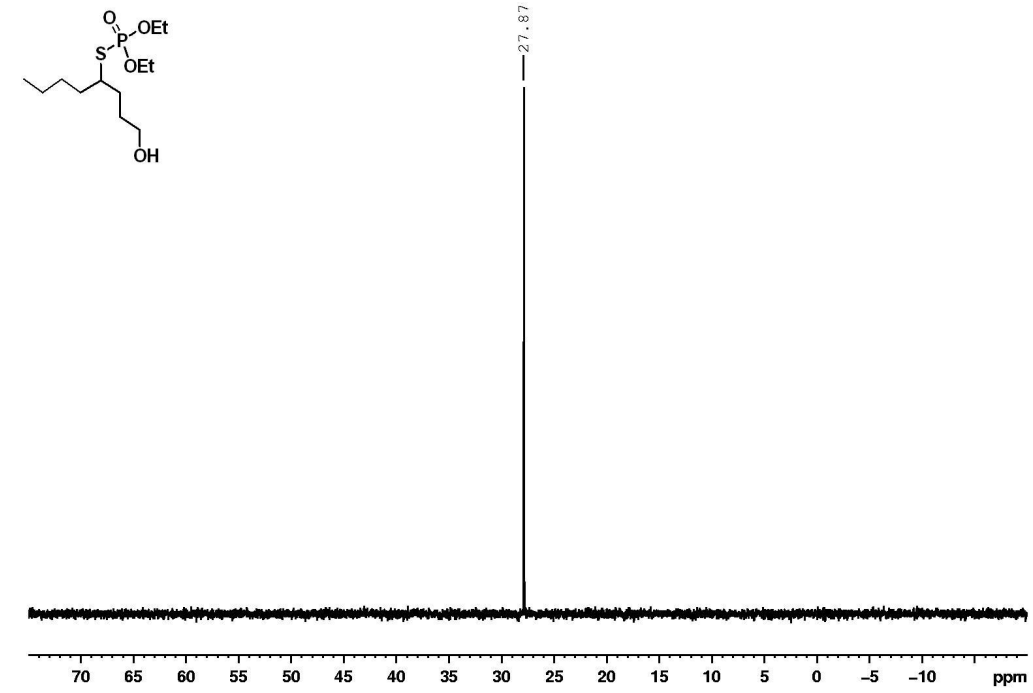
**3i**


4.187 4.182 4.177 4.175 4.173 4.170 4.165 4.164 4.158 4.156 4.152 4.146 4.144 4.132 4.130 4.120 4.118 4.115 4.106 4.103 3.682 3.672 3.661 3.363 3.352 3.342 1.832 1.819 1.807 1.795 1.742 1.731 1.720 1.709 1.707 1.697 1.687 1.676 1.674 1.663 1.625 1.622 1.617 1.609 1.441 1.430 1.424 1.363 1.351 1.339 1.311 1.306 1.300 1.252 1.248 0.939 0.926 0.914
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
3.98 1.98 1.00 2.13 2.00 0.98 0.95 5.99 1.94 3.05

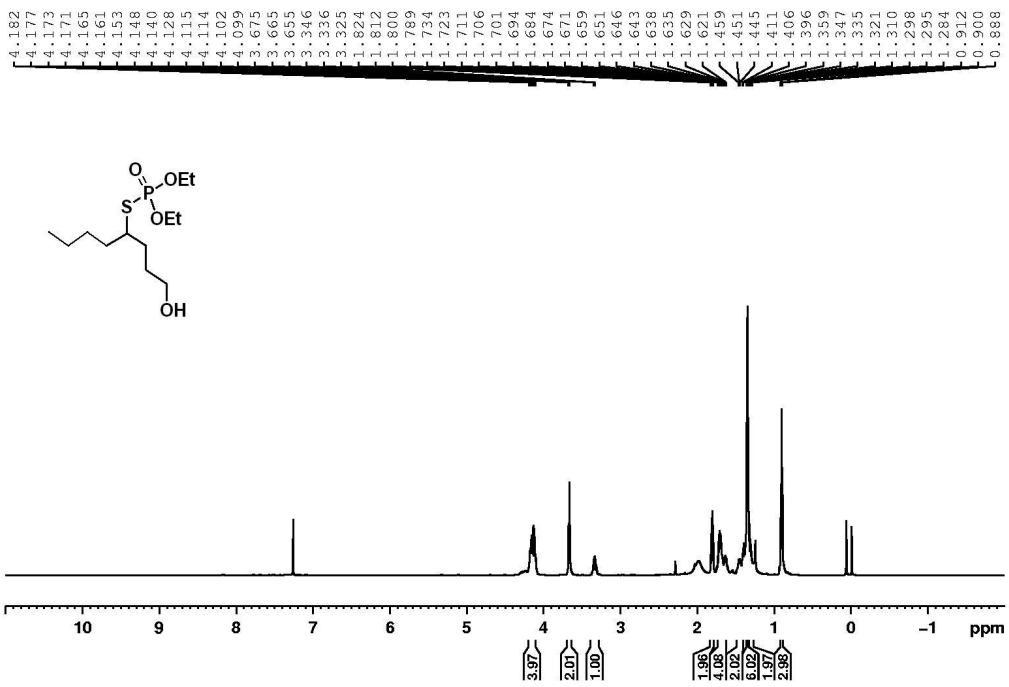
**3i**


O
OEt
S
P
OEt
OH
77.47
77.26
77.05
63.84
63.80
62.38
48.00
38.47
38.42
33.33
33.30
29.59
20.17
16.33
16.27
14.05
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

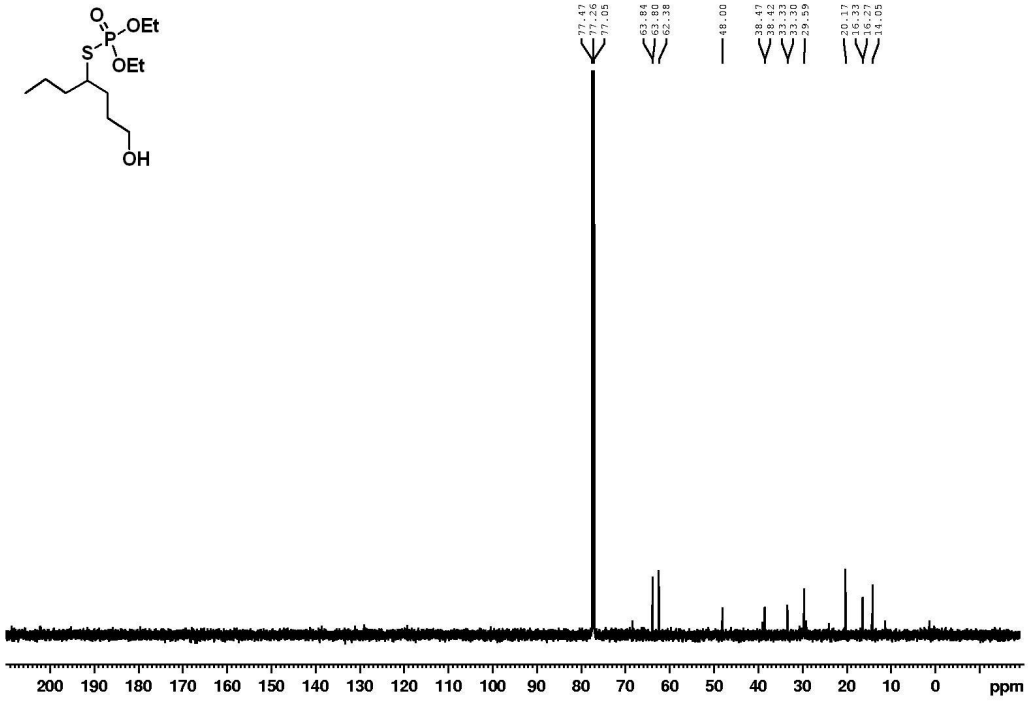
**3j**


O
OEt
S
P
OEt
OH
27.87
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

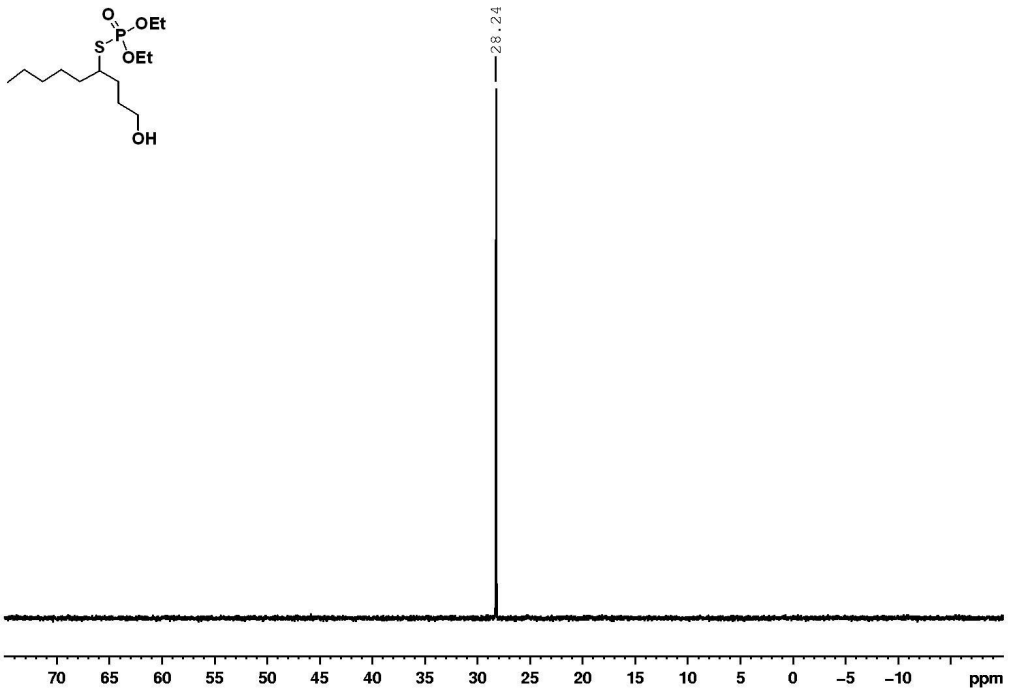
**3j**


4.182
4.177
4.173
4.171
4.165
4.161
4.153
4.148
4.140
4.128
4.115
4.114
4.102
4.099
3.675
3.665
3.655
3.346
3.336
3.325
1.824
1.812
1.800
1.789
1.734
1.723
1.711
1.706
1.701
1.694
1.684
1.674
1.671
1.659
1.651
1.646
1.643
1.638
1.635
1.629
1.621
1.459
1.451
1.445
1.411
1.406
1.396
1.359
1.347
1.335
1.321
1.310
1.298
1.295
1.284
0.912
0.900
0.888
OEt
OEt
OH
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
3.97
2.01
1.00
1.98
4.08
2.02
6.02
1.97
2.98

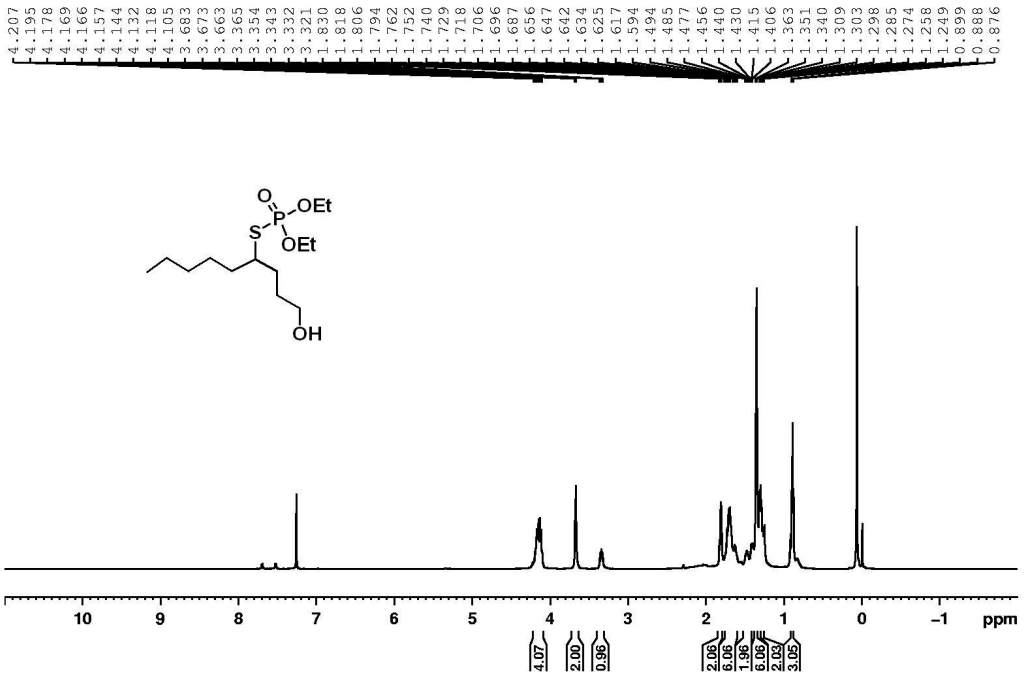
**3j**


OEt
OEt
OH
77.47
77.26
77.05
63.84
63.80
62.38
48.00
38.47
38.42
33.33
33.30
29.59
20.17
16.33
16.27
14.05
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

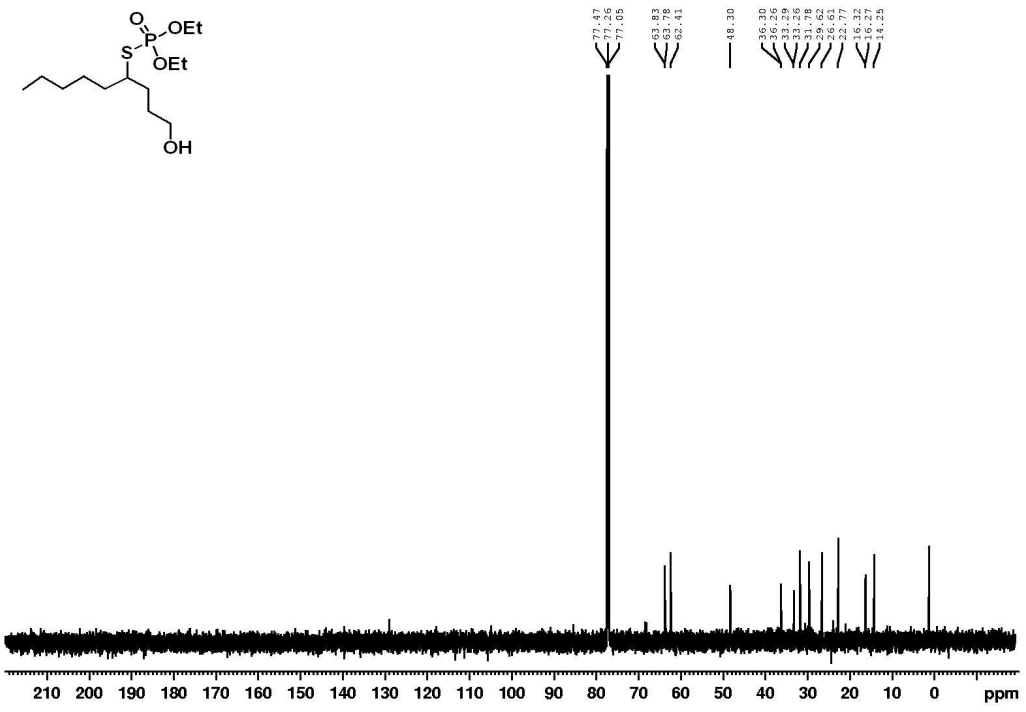
**3k**


28.24
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

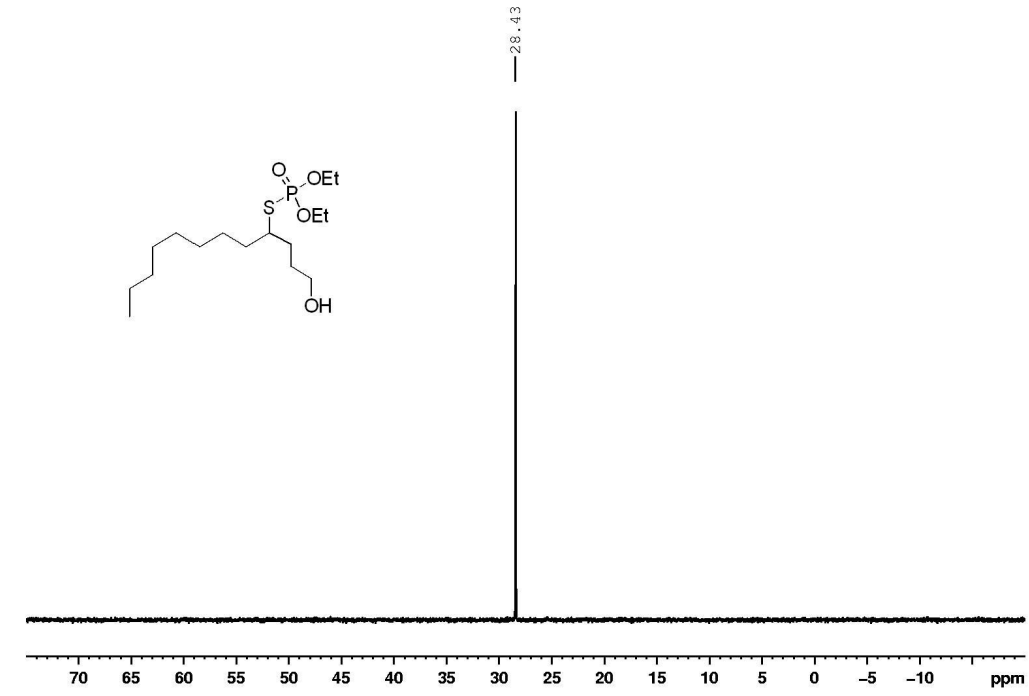
**3k**


4.207 4.195 4.178 4.169 4.166 4.157 4.144 4.132 4.118 4.105 3.683 3.673 3.663 3.365 3.354 3.343 3.332 3.321 1.830 1.818 1.806 1.794 1.762 1.752 1.740 1.729 1.718 1.706 1.696 1.687 1.656 1.647 1.642 1.634 1.625 1.617 1.594 1.494 1.485 1.477 1.456 1.440 1.430 1.415 1.406 1.363 1.351 1.340 1.309 1.303 1.298 1.285 1.274 1.258 1.249 0.899 0.888 0.876
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
4.07 2.00 0.96 2.08 6.08 1.96 6.06 2.03 3.05

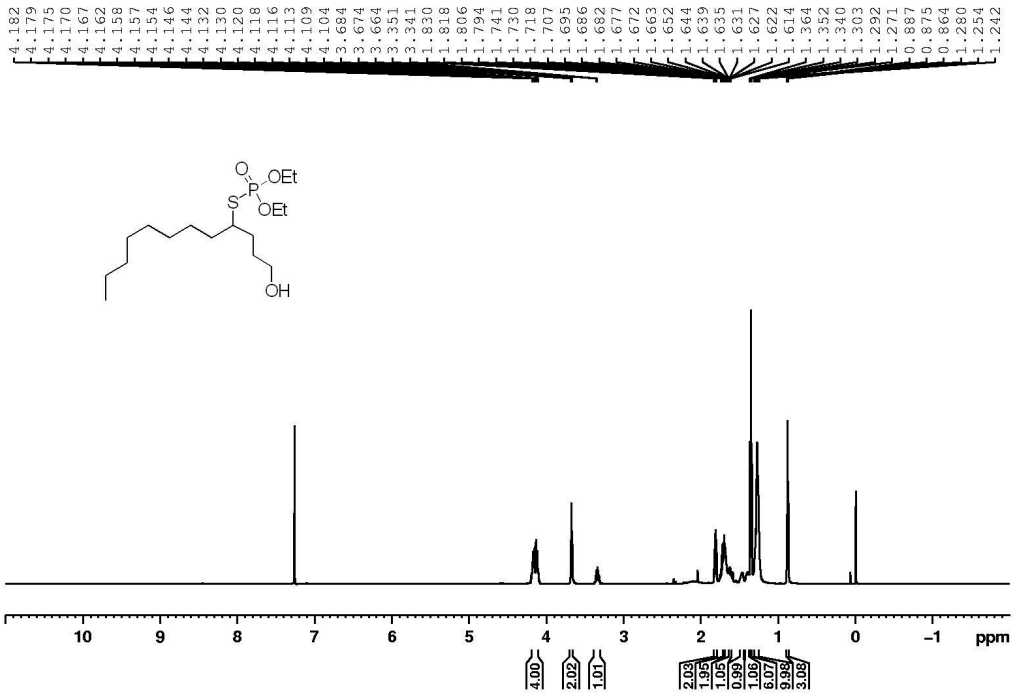
**3k**


OEt
OEt
S
P
O
OH
77.47
77.26
77.05
63.83
63.78
62.41
48.30
36.30
36.26
33.89
33.26
31.78
29.62
26.61
22.77
16.32
16.27
14.25
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

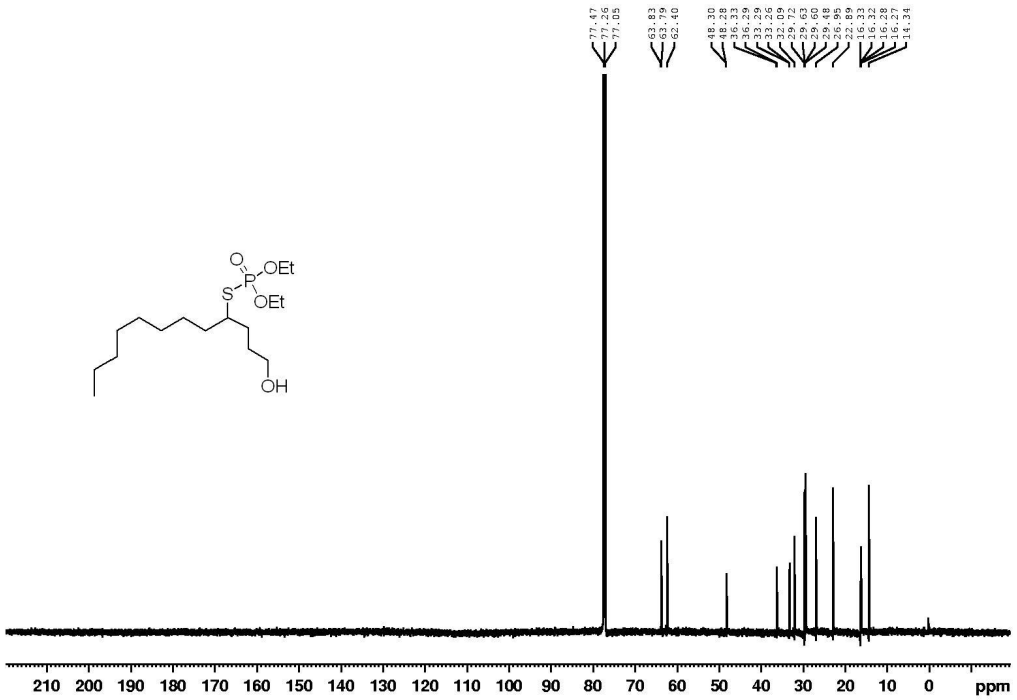
**3l**


28.43
OEt
OEt
S
P
O
OH
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

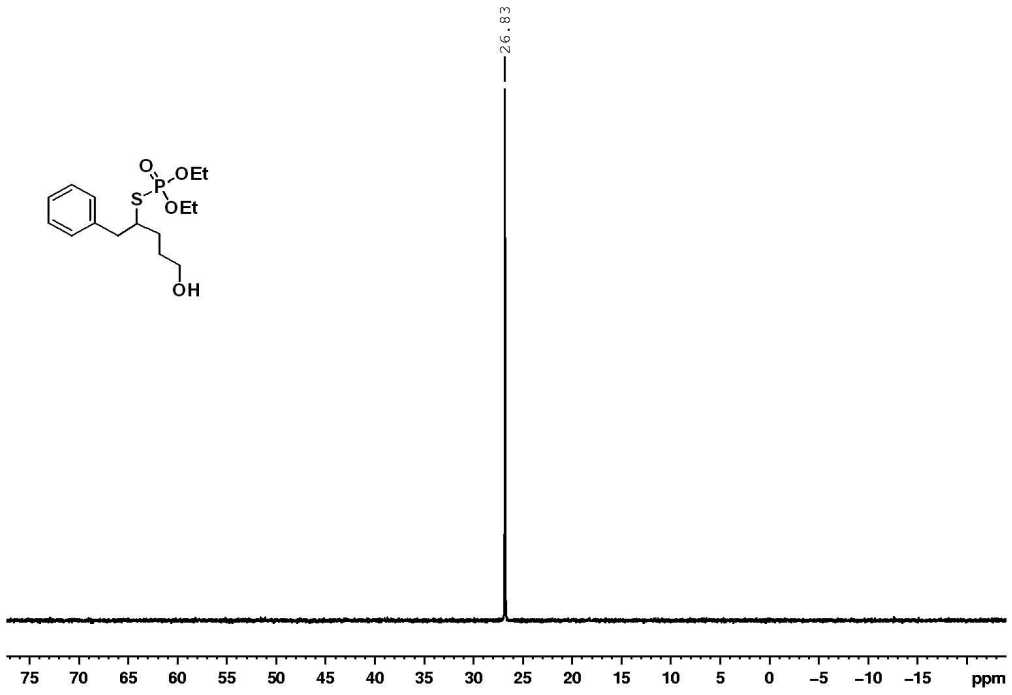
**3l**


4.182
4.179
4.175
4.170
4.167
4.162
4.158
4.157
4.154
4.146
4.144
4.132
4.130
4.120
4.118
4.116
4.113
4.109
4.104
3.684
3.674
3.664
3.351
3.341
1.830
1.818
1.806
1.794
1.741
1.730
1.718
1.707
1.695
1.686
1.682
1.677
1.672
1.663
1.652
1.644
1.639
1.635
1.631
1.627
1.622
1.614
1.364
1.352
1.340
1.303
1.292
1.271
0.887
0.875
0.864
1.280
1.254
1.242
OEt
OEt
S
P
O
OH
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
4.00
2.02
1.01
2.03
1.95
1.05
0.99
1.06
6.07
9.98
3.08

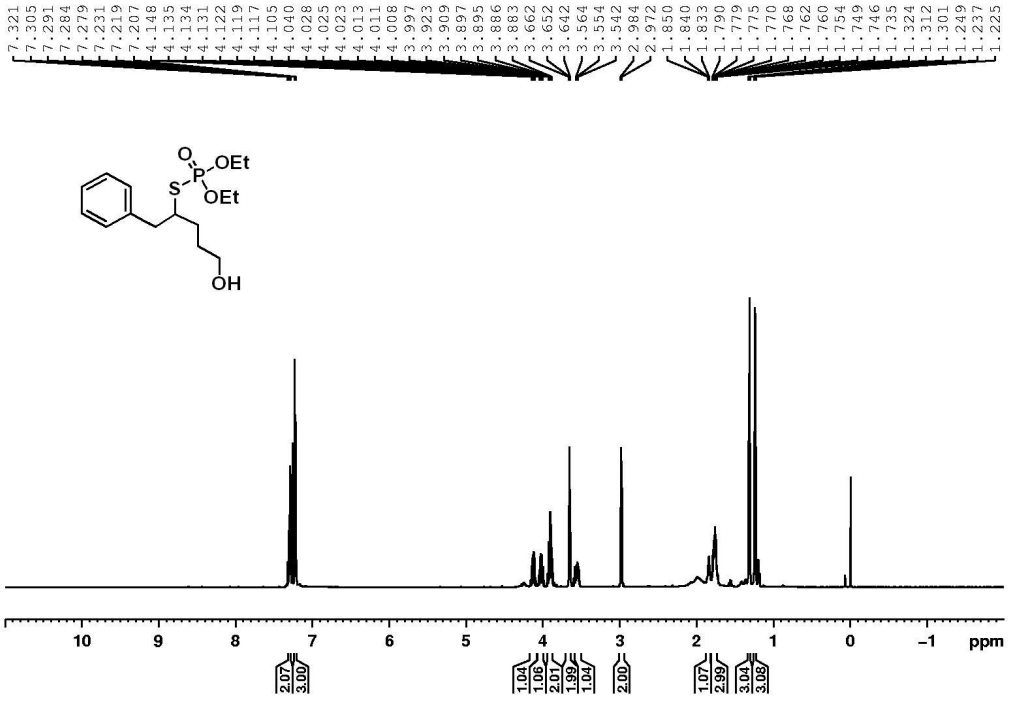
**3l**


77.47
77.26
77.05
63.83
63.49
62.40
48.30
48.28
36.33
36.29
33.29
33.26
32.09
29.72
29.63
29.60
29.48
26.95
22.89
16.33
16.22
16.28
16.27
14.34
OEt
OEt
S
P
O
OH
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

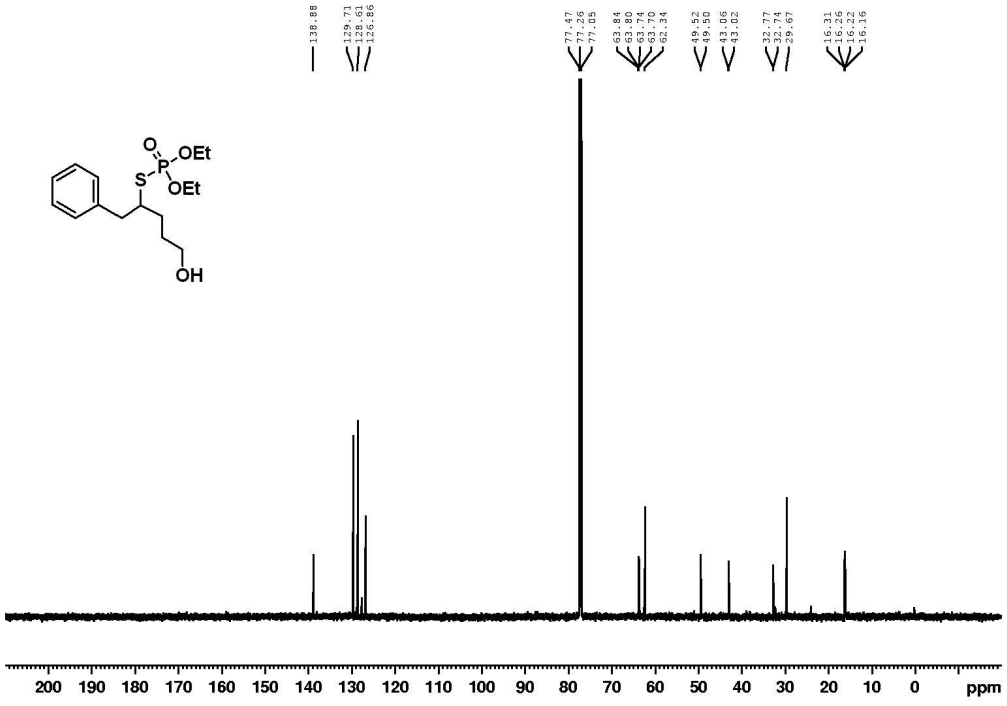
**3m**


26.83
O
OEt
S
P
OEt
OH
75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 −15 ppm

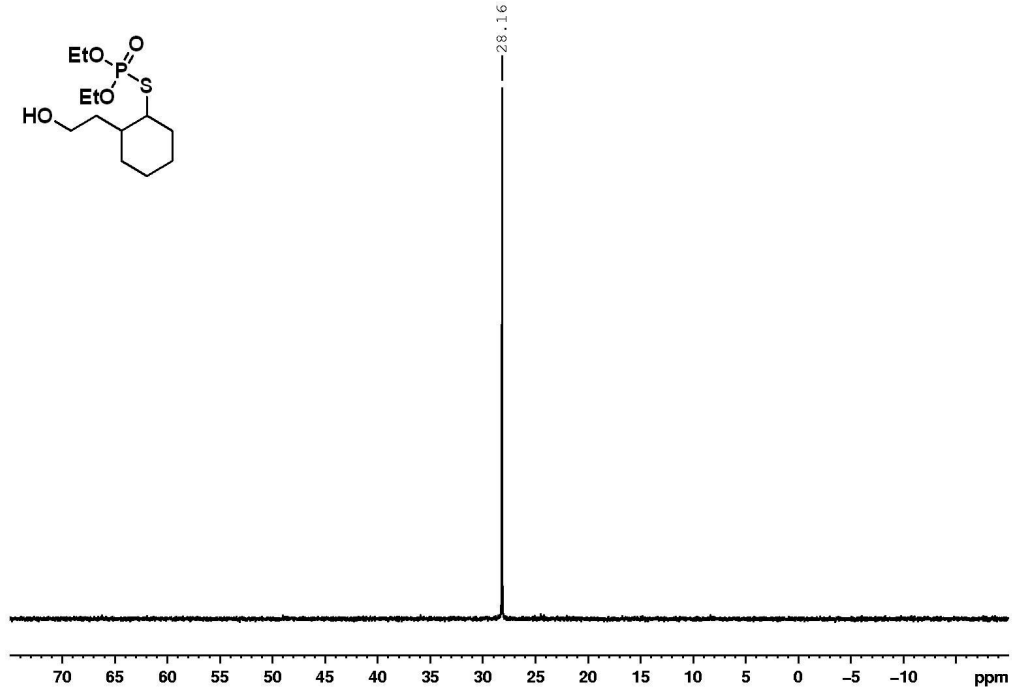
**3m**


7.321 7.305 7.291 7.284 7.279 7.231 7.219 7.207 4.148 4.135 4.134 4.131 4.122 4.119 4.117 4.105 4.040 4.028 4.025 4.023 4.013 4.011 4.008 3.997 3.923 3.909 3.897 3.895 3.886 3.883 3.662 3.652 3.642 3.564 3.554 3.542 2.984 2.972 1.850 1.840 1.833 1.790 1.779 1.775 1.770 1.768 1.762 1.760 1.754 1.749 1.746 1.735 1.324 1.312 1.301 1.249 1.237 1.225
O
OEt
S
P
OEt
OH
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
2.07 3.00 1.04 1.06 2.01 1.99 1.04 2.00 1.07 2.99 3.04 3.08

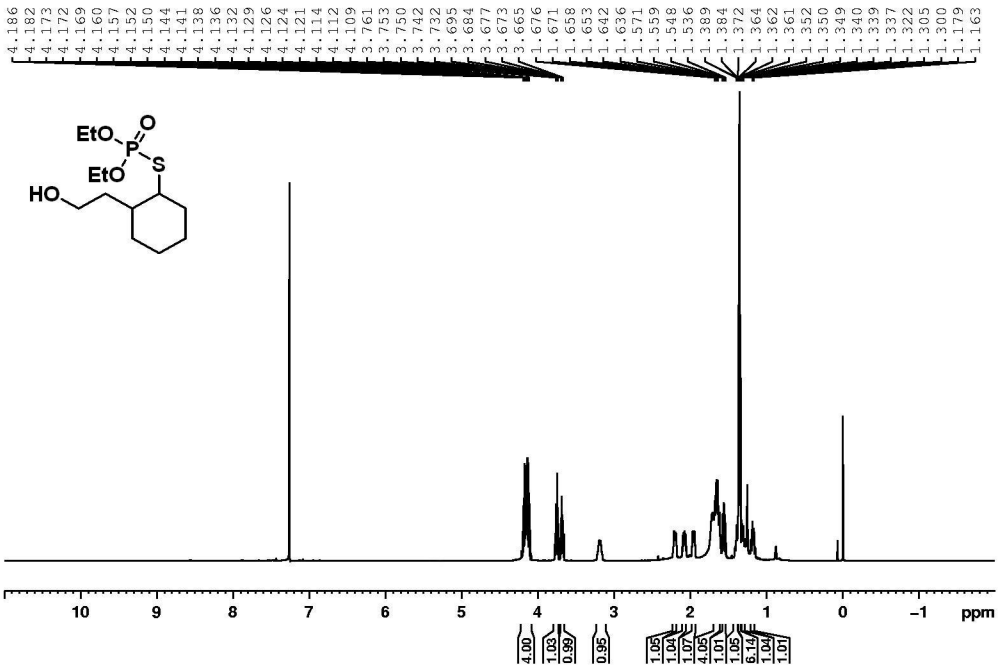
**3m**


138.88
129.71
128.61
126.86
77.47
77.26
77.05
63.84
63.80
63.74
63.70
62.34
49.52
49.50
43.06
43.02
32.77
32.74
29.67
16.31
16.26
16.22
16.16
O
OEt
S
OEt
OH
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

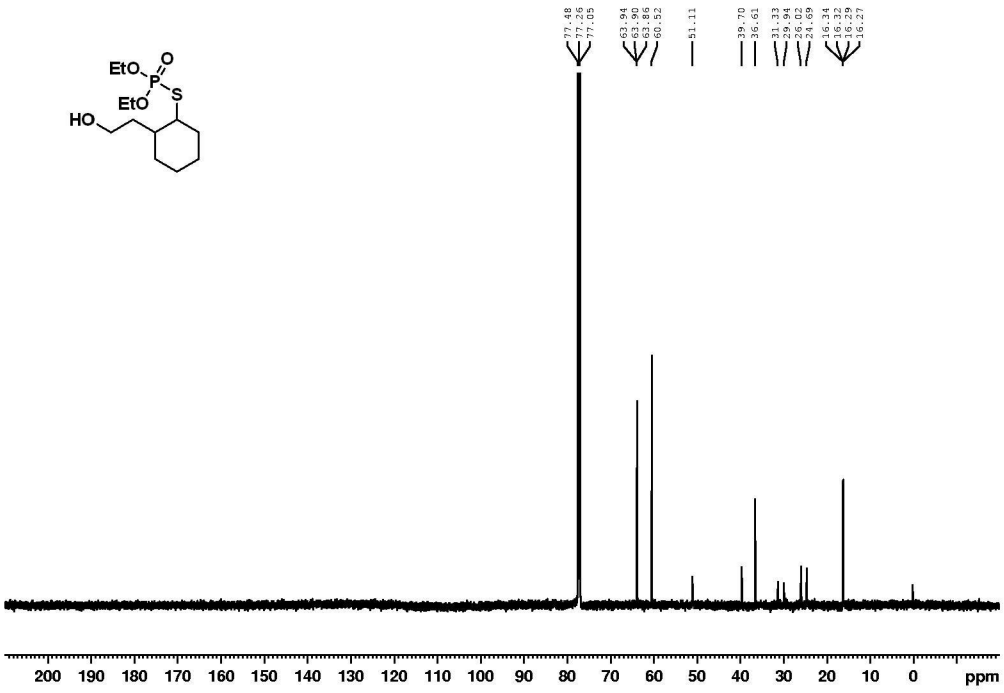
**3n**


28.16
EtO
O
P
S
EtO
HO
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 –5 –10 ppm

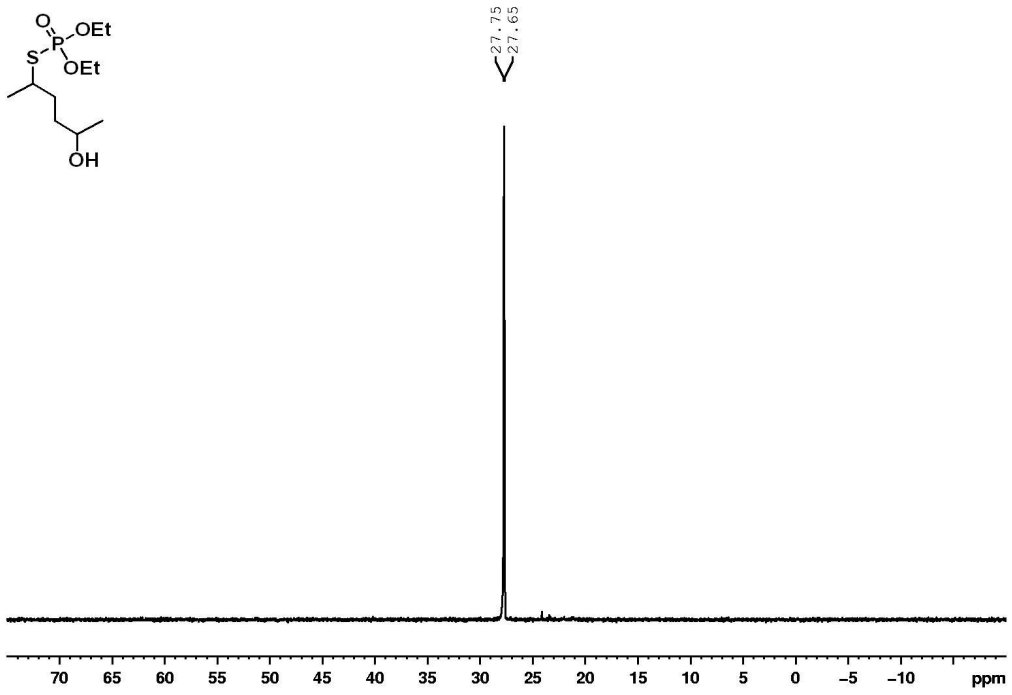
**3n**


4.186
4.182
4.173
4.172
4.169
4.160
4.157
4.152
4.150
4.144
4.141
4.138
4.136
4.132
4.129
4.126
4.124
4.121
4.114
4.112
4.109
3.761
3.753
3.750
3.742
3.732
3.695
3.684
3.677
3.673
3.665
1.676
1.671
1.658
1.653
1.642
1.636
1.571
1.559
1.548
1.536
1.389
1.384
1.372
1.364
1.362
1.361
1.352
1.350
1.349
1.340
1.339
1.337
1.322
1.305
1.300
1.179
1.163
EtO
O
P
S
EtO
HO
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
4.00
1.03
0.99
0.95
1.05
1.04
1.07
4.05
1.01
1.05
6.14
1.04
1.01

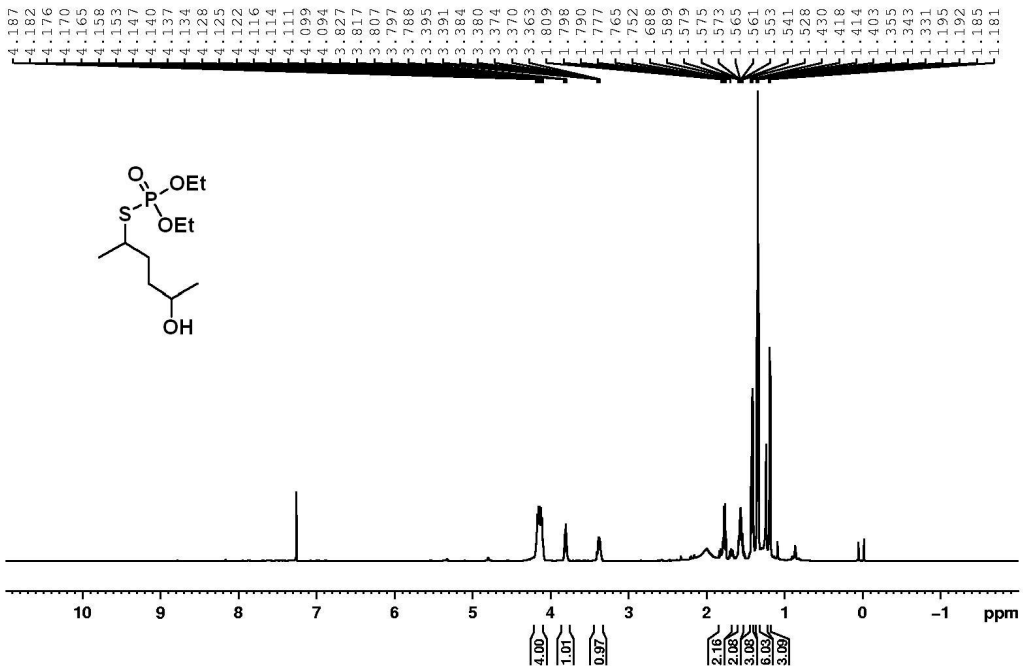
**3n**


77.48
77.26
77.05
63.94
63.90
63.86
60.52
51.11
39.70
36.61
31.33
29.94
29.62
24.69
16.34
16.32
16.29
16.27
EtO
O
P
S
EtO
HO
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

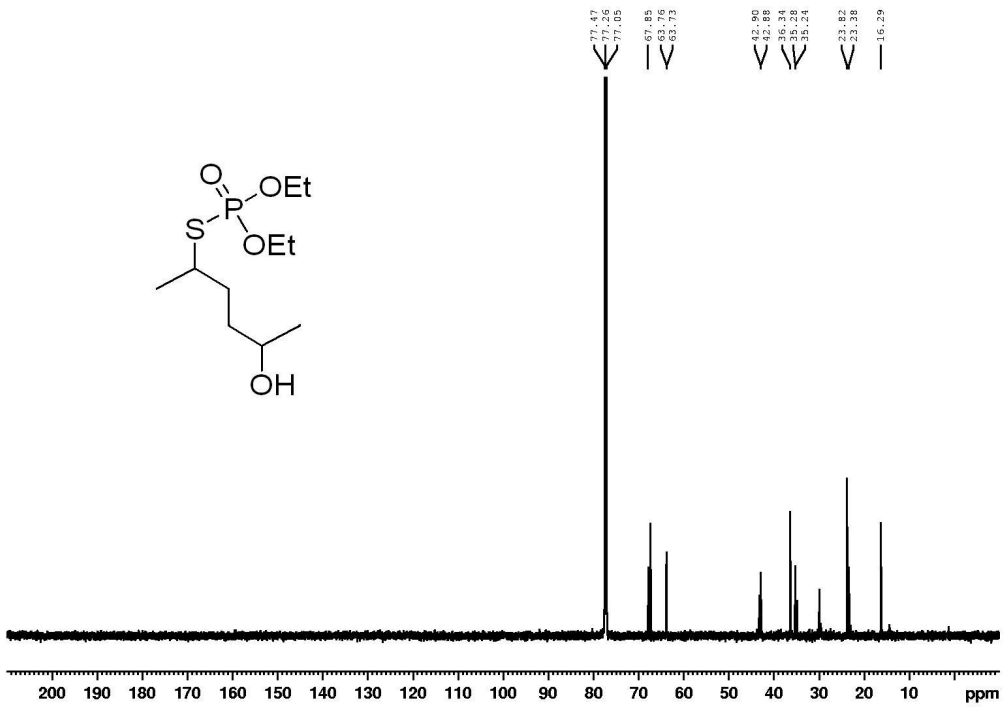
**3o**


27.75
27.65
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

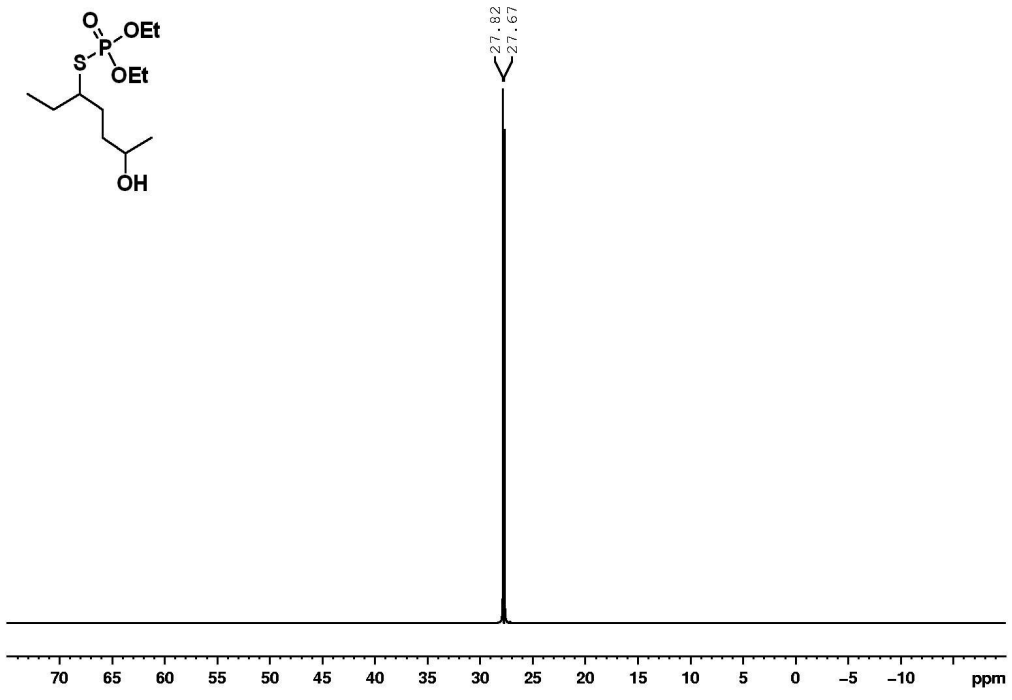
**3o**


10 9 8 7 6 5 4 3 2 1 0 −1 ppm
4.00
1.01
0.97
2.16
2.08
3.08
6.03
3.09

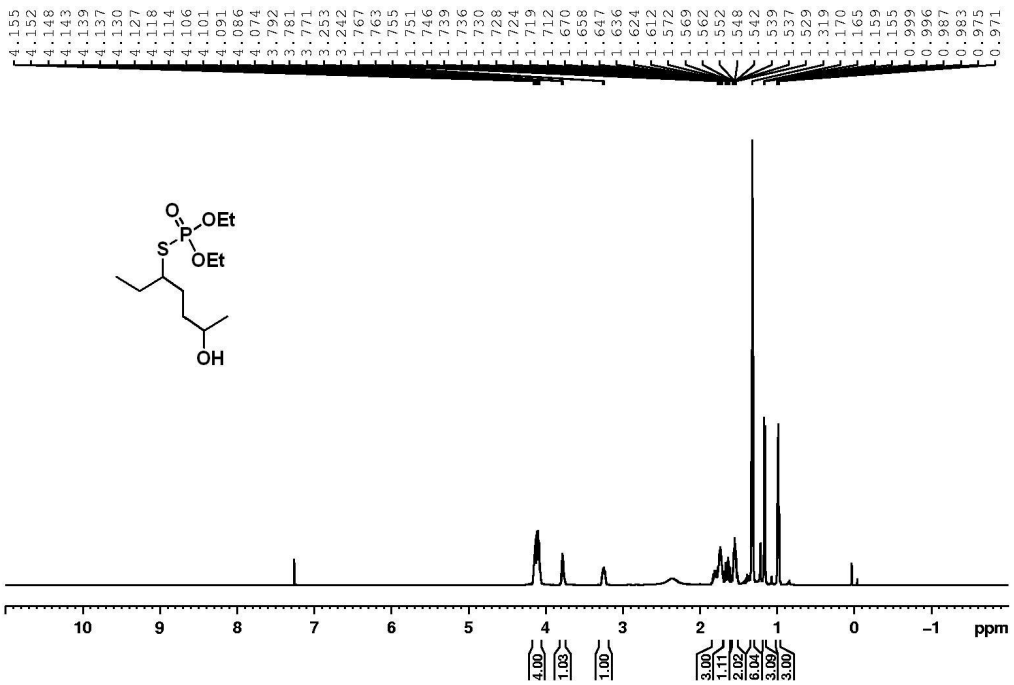
**3o**


77.47
77.26
77.05
67.85
63.76
63.73
42.90
42.88
36.34
35.28
35.24
23.82
23.38
16.29
O
OEt
P
S
OEt
OH
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

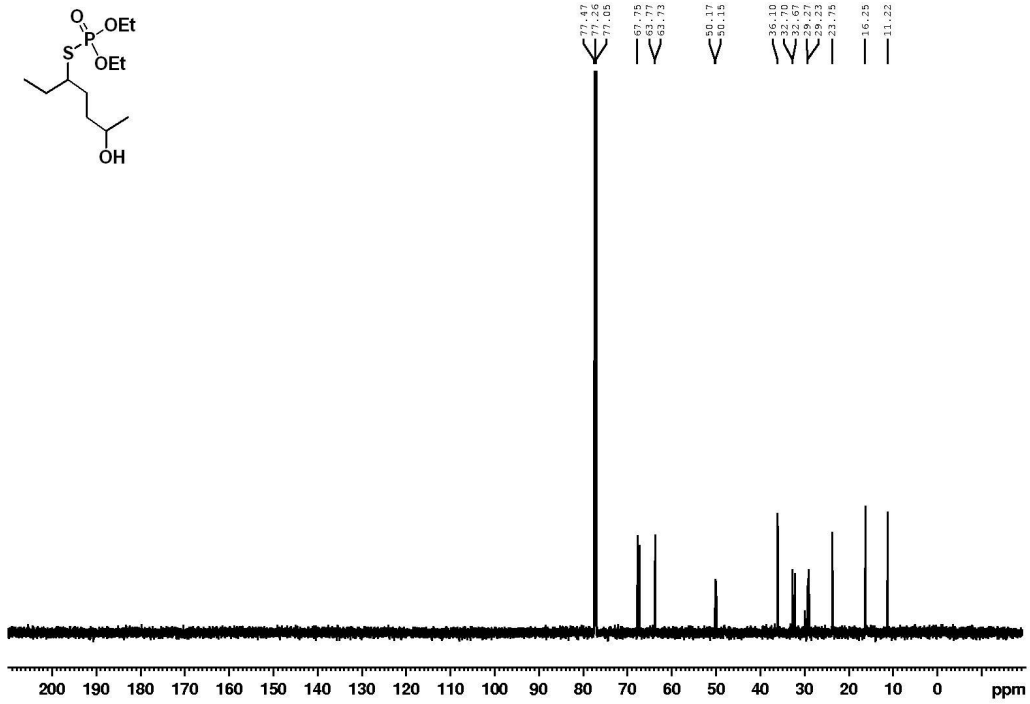
**3p**


27.82
27.67
O
OEt
P
S
OEt
OH
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

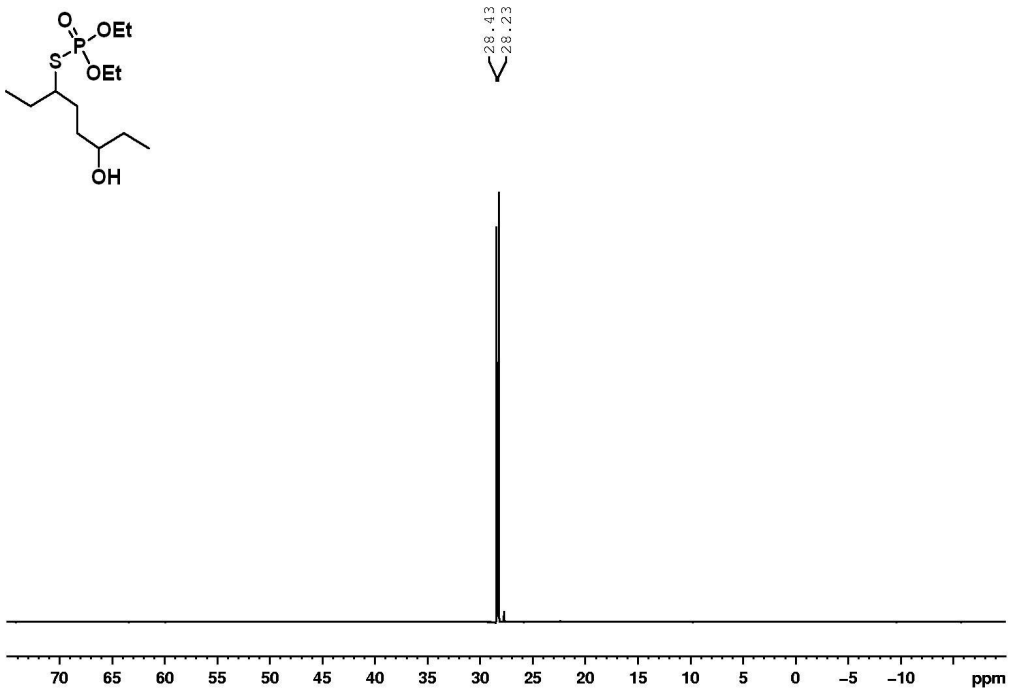
**3p**


4.155
4.152
4.148
4.143
4.139
4.137
4.130
4.127
4.118
4.114
4.106
4.101
4.091
4.086
4.074
3.792
3.781
3.771
3.253
3.242
1.767
1.763
1.755
1.751
1.746
1.739
1.736
1.730
1.728
1.724
1.719
1.712
1.670
1.658
1.647
1.636
1.624
1.612
1.572
1.569
1.562
1.552
1.548
1.542
1.539
1.537
1.529
1.319
1.170
1.165
1.159
1.155
0.999
0.996
0.987
0.983
0.975
0.971
OEt
OEt
OH
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
4.00
1.03
1.00
3.00
1.11
2.02
6.04
3.09
3.00

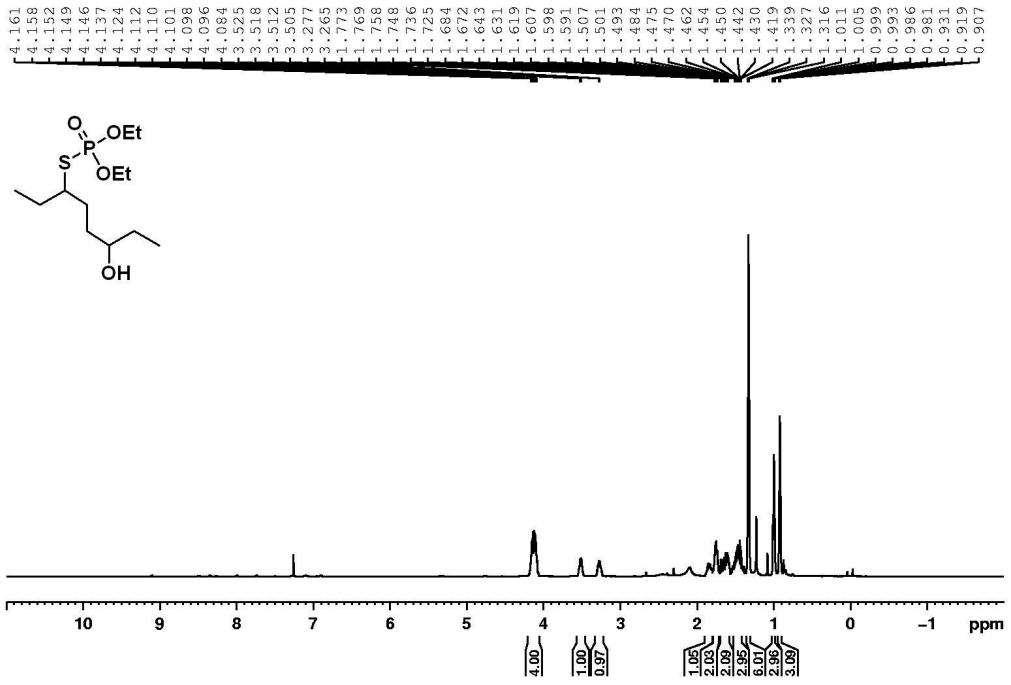
**3p**


OEt
OEt
OH
77.47
77.26
77.05
67.75
63.77
63.73
50.17
50.15
36.10
32.70
32.67
29.27
29.23
23.75
16.25
11.22
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

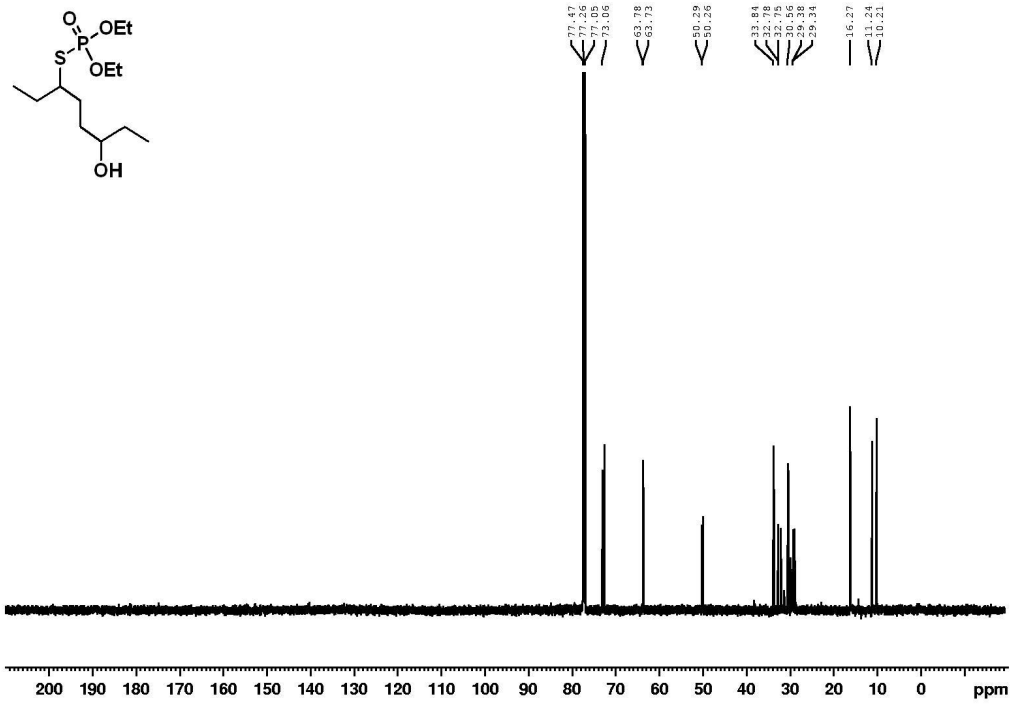
**3q**


28.43
28.23
OEt
OEt
OH
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

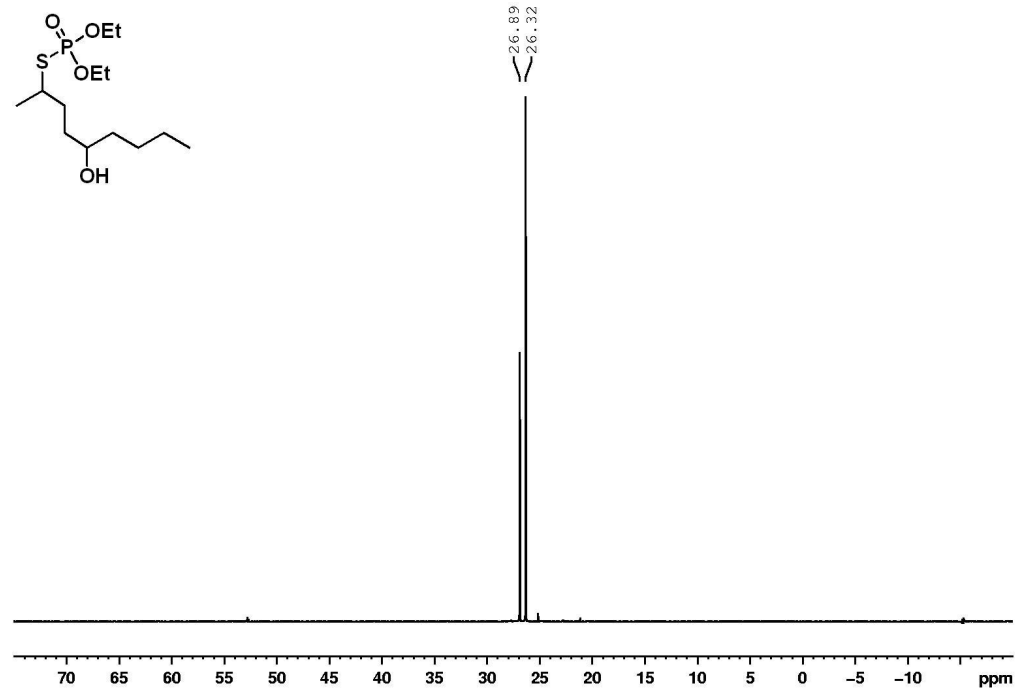
**3q**


4.161 4.158 4.152 4.149 4.146 4.137 4.124 4.112 4.110 4.101 4.098 4.096 4.084 3.525 3.518 3.512 3.505 3.277 3.265 1.773 1.769 1.758 1.748 1.736 1.725 1.684 1.672 1.643 1.631 1.619 1.607 1.598 1.591 1.507 1.501 1.493 1.484 1.475 1.470 1.462 1.454 1.450 1.442 1.430 1.419 1.339 1.327 1.316 1.011 1.005 0.999 0.993 0.986 0.981 0.931 0.919 0.907
OEt
OEt
OH
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
4.00
1.00
0.97
1.05
2.03
2.09
2.95
5.01
2.96
3.09

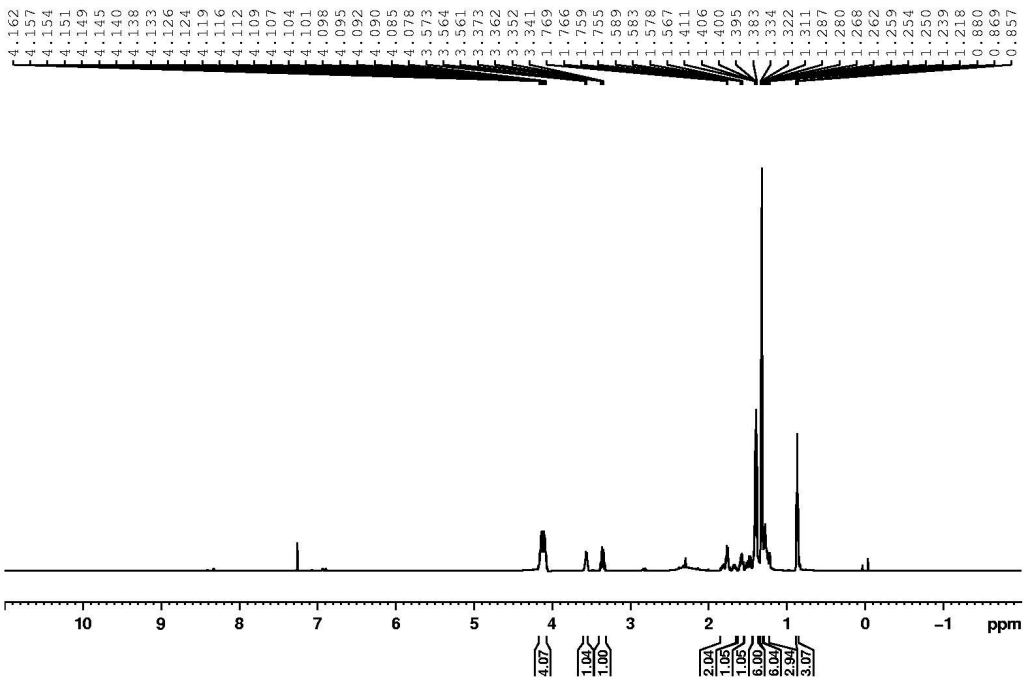
**3q**


77.47
77.26
77.05
73.06
63.78
63.73
50.29
50.26
33.84
32.78
32.75
30.46
29.38
29.34
16.27
11.24
10.21
OEt
OEt
OH
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

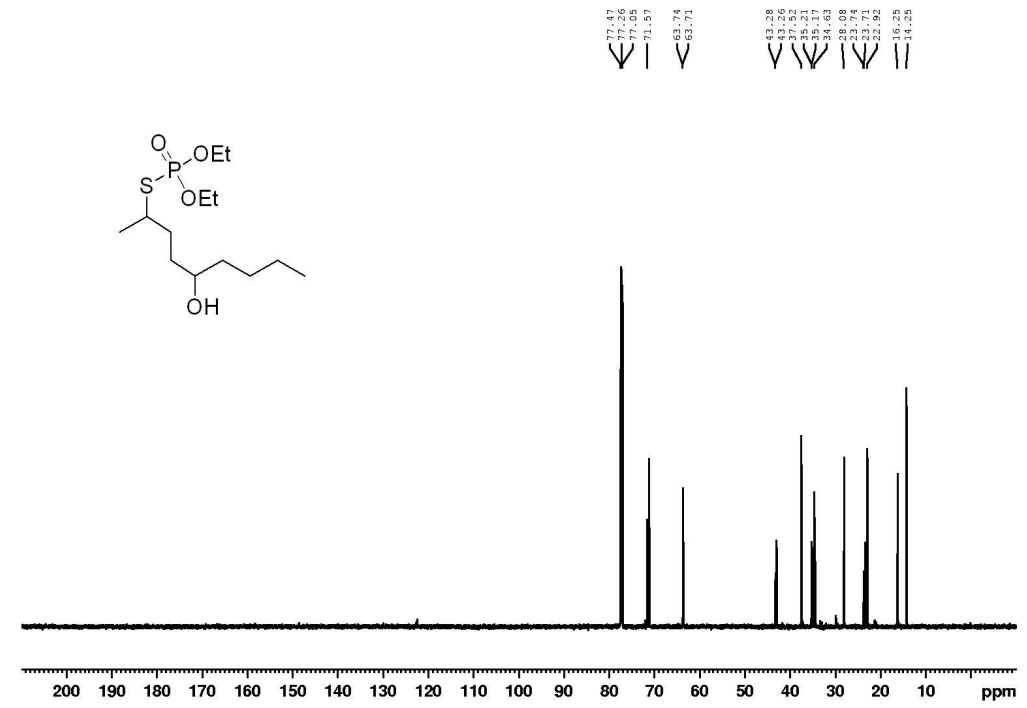
**3r**


26.89
26.32
OEt
OEt
OH
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

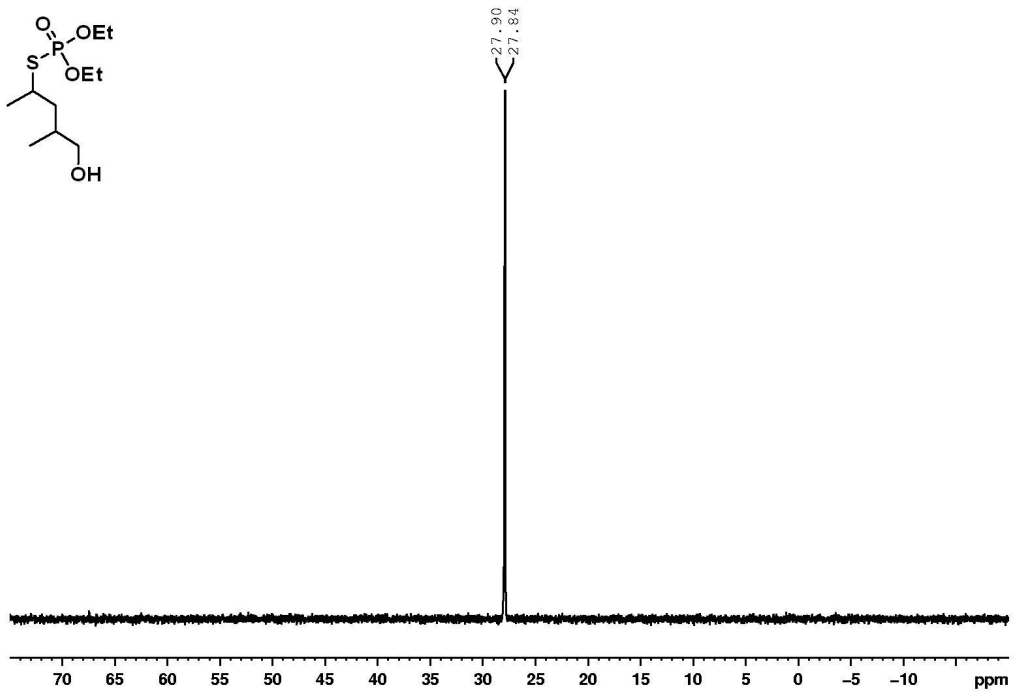
**3r**


4.162
4.157
4.154
4.151
4.149
4.145
4.140
4.138
4.133
4.126
4.124
4.119
4.116
4.112
4.109
4.107
4.104
4.101
4.098
4.095
4.092
4.090
4.085
4.078
3.573
3.564
3.561
3.373
3.362
3.352
3.341
1.769
1.766
1.759
1.755
1.589
1.583
1.578
1.567
1.411
1.406
1.400
1.395
1.383
1.334
1.322
1.311
1.287
1.280
1.268
1.262
1.259
1.254
1.250
1.239
1.218
0.880
0.869
0.857
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
4.07
1.04
1.00
2.04
1.05
1.05
6.00
6.04
2.94
3.07

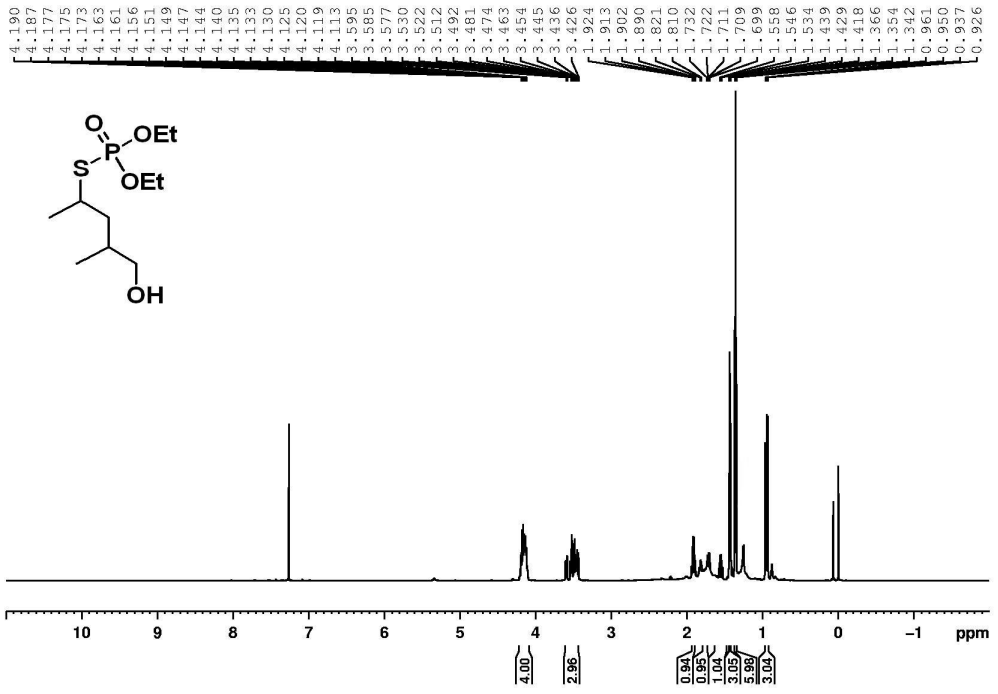
**3r**


77.47
77.26
77.05
71.57
63.74
63.71
43.28
43.26
37.52
35.21
35.17
34.63
28.08
23.74
23.71
22.92
16.25
14.25
O
OEt
P
S
OEt
OH
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

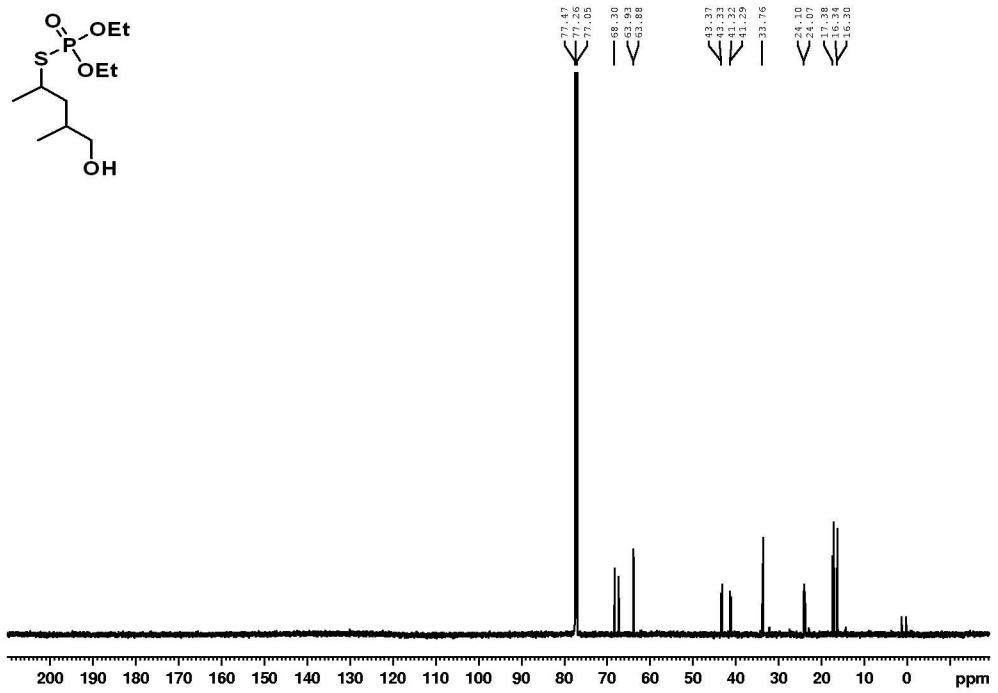
**3s**


27.90
27.84
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
−5
−10
ppm

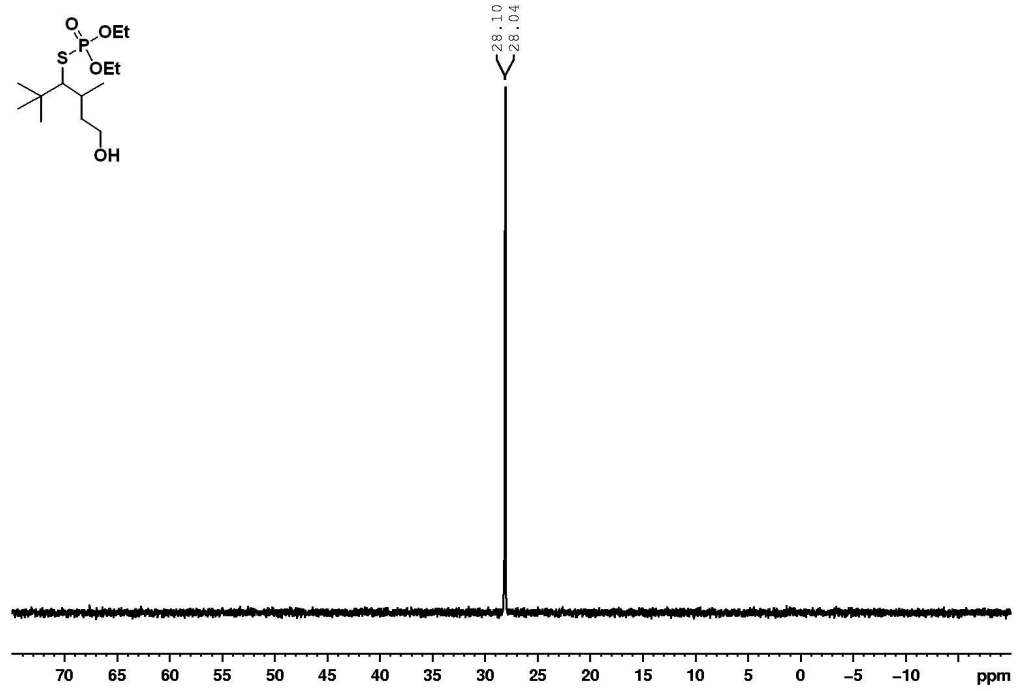
**3s**


10
9
8
7
6
5
4
3
2
1
0
−1
ppm
4.00
2.96
0.94
0.95
1.04
3.05
5.98
3.04

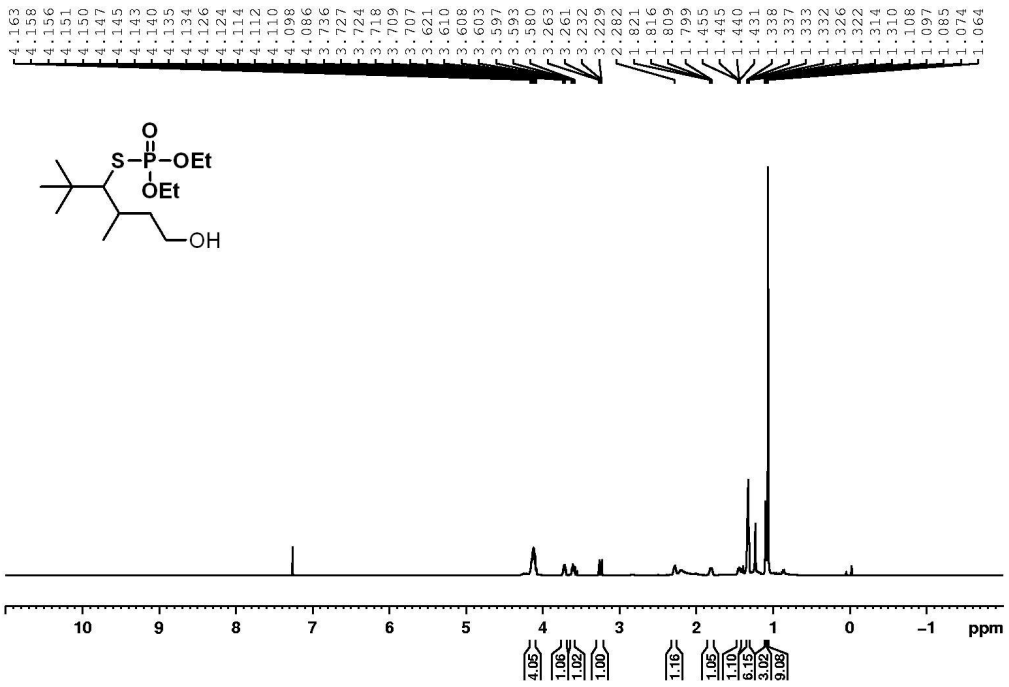
**3r**


O
OEt
P
S
OEt
OH
77.47
77.26
77.05
68.30
63.93
63.88
43.37
43.33
41.32
41.29
33.76
24.10
24.07
17.38
16.34
16.30
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

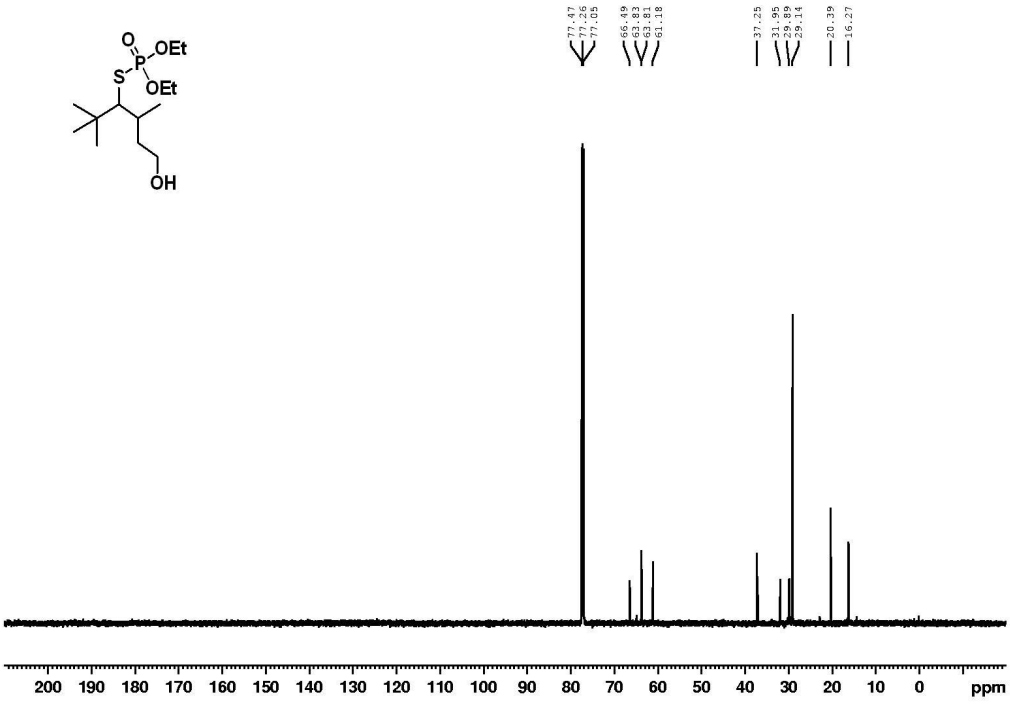
**3t**


O
OEt
P
S
OEt
OH
28.10
28.04
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

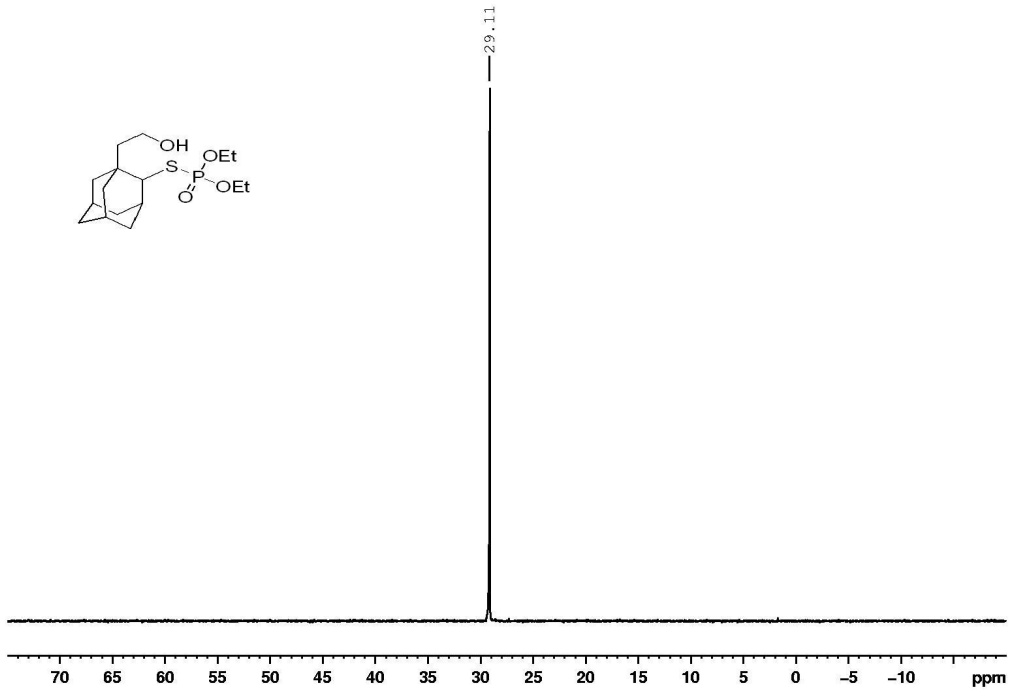
**3t**


4.163
4.158
4.156
4.151
4.150
4.147
4.145
4.143
4.140
4.135
4.134
4.126
4.124
4.114
4.112
4.110
4.098
4.086
3.736
3.727
3.724
3.718
3.709
3.707
3.621
3.610
3.608
3.603
3.597
3.593
3.580
3.263
3.261
3.232
3.229
2.282
1.821
1.816
1.809
1.799
1.455
1.445
1.440
1.431
1.338
1.337
1.333
1.332
1.326
1.322
1.314
1.310
1.108
1.097
1.085
1.074
1.064
O
S–P–OEt
OEt
OH
10 9 8 7 6 5 4 3 2 1 0 –1 ppm
4.05
1.06
1.02
1.00
1.16
1.05
1.10
6.15
3.02
9.08

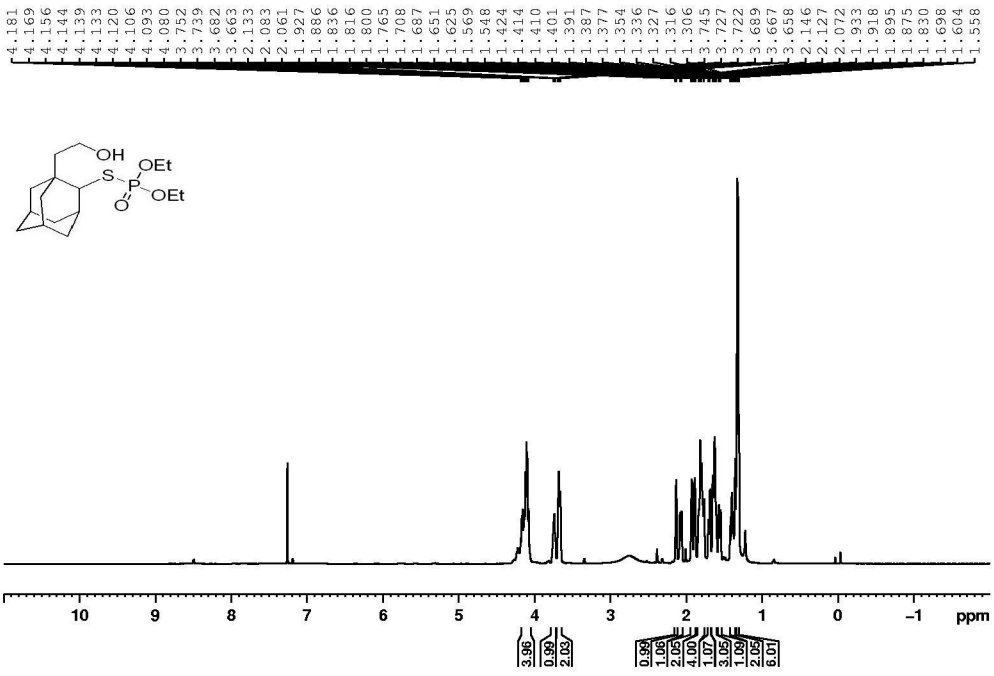
**3t**


O
OEt
S
P
OEt
OH
77.47
77.26
77.05
66.49
63.83
63.81
61.18
37.25
31.95
29.89
29.14
20.39
16.27
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

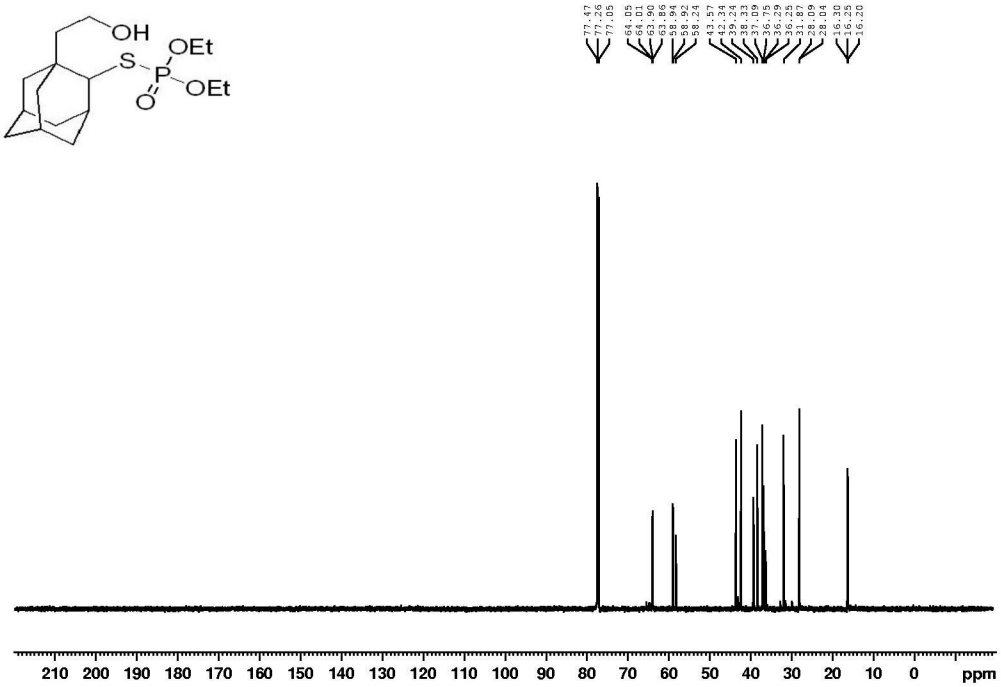
**3u**


29.11
OH
S
P
OEt
OEt
O
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

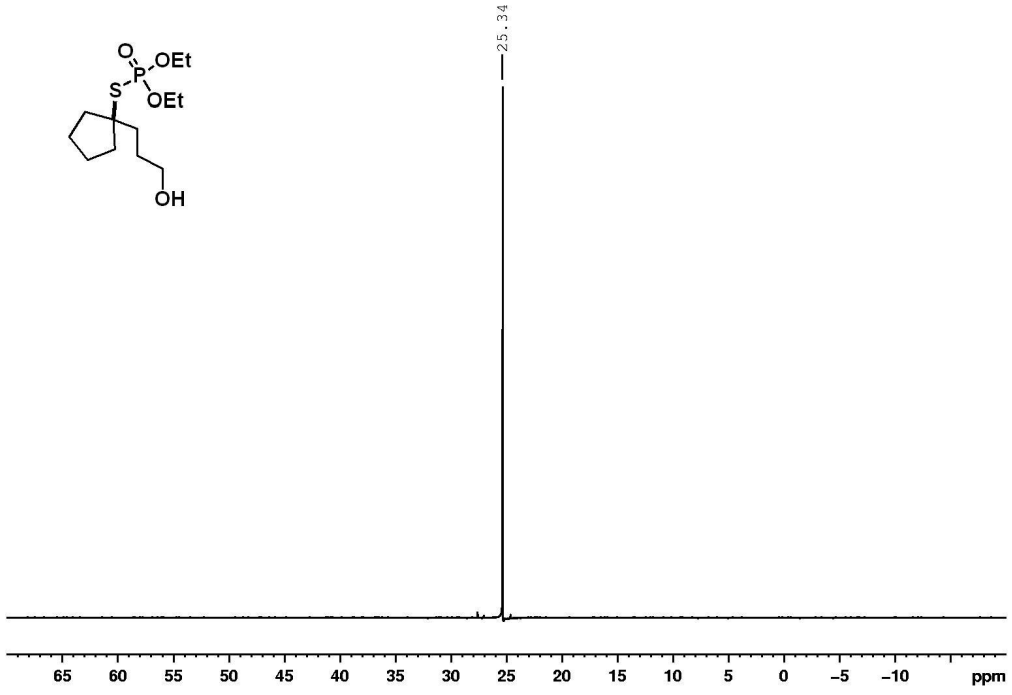
**3u**


4.181 4.169 4.156 4.144 4.139 4.133 4.120 4.106 4.093 4.080 3.752 3.739 3.682 3.663 2.133 2.083 2.061 1.927 1.886 1.836 1.816 1.800 1.765 1.708 1.687 1.651 1.625 1.569 1.548 1.424 1.414 1.410 1.401 1.391 1.387 1.377 1.354 1.336 1.327 1.316 1.306 3.745 3.727 3.722 3.689 3.667 3.658 2.146 2.127 2.072 1.933 1.918 1.895 1.875 1.830 1.698 1.604 1.558
OH
S
P
OEt
OEt
O
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
3.96 0.99 2.03
0.98 1.06 2.06 4.00 1.07 3.05 1.09 2.05 6.01

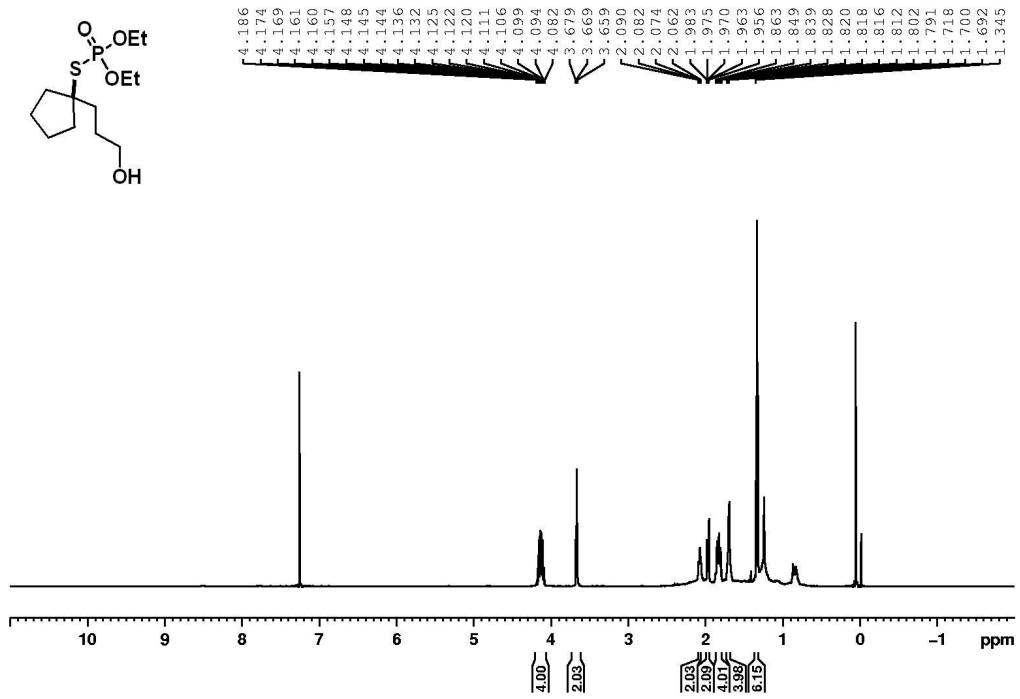
**3u**


OH
OEt
S
P
OEt
O
77.47
77.26
77.05
64.05
64.01
63.90
63.86
58.94
58.82
58.24
43.57
42.34
39.24
38.33
37.09
36.75
36.29
36.25
31.87
28.09
28.04
16.30
16.25
16.20
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

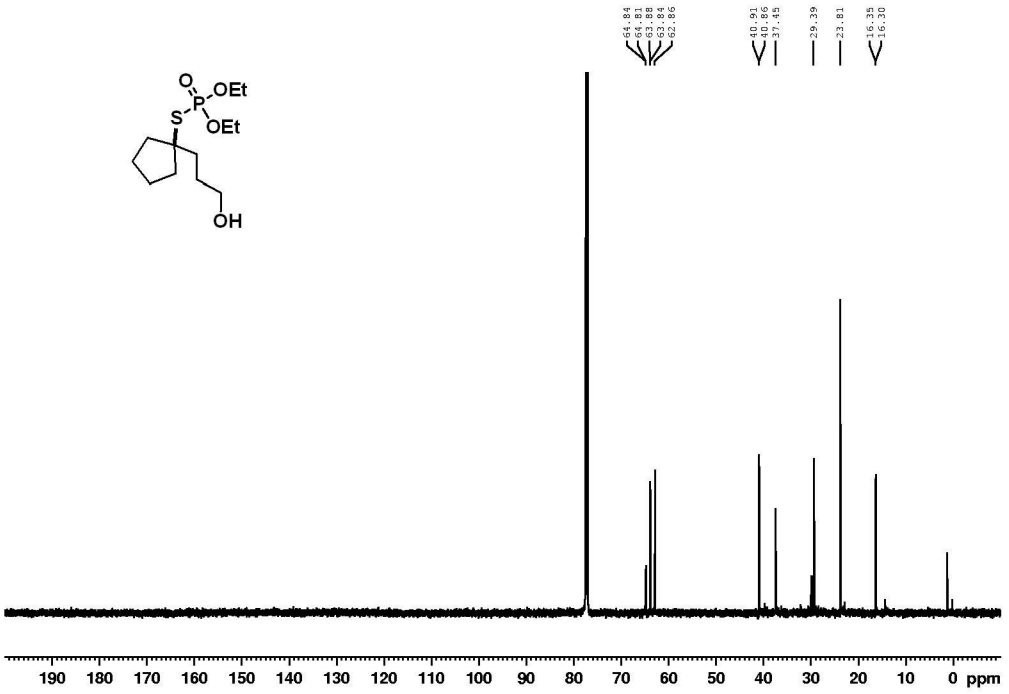
**3v**


O
P
OEt
S
OEt
OH
25.34
65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

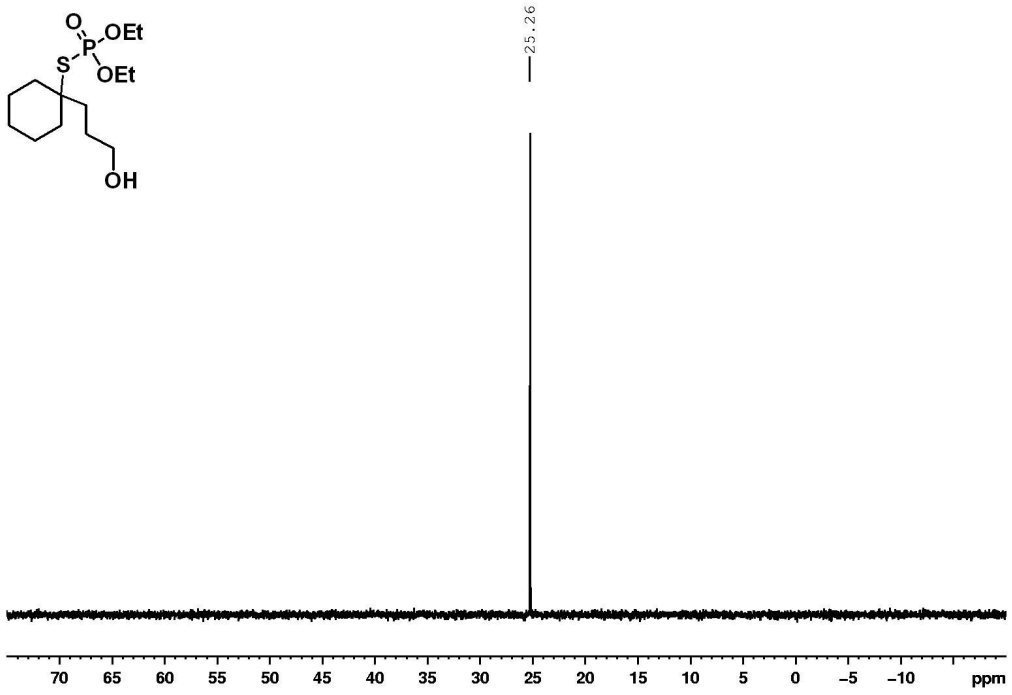
**3v**


4.186
4.174
4.169
4.161
4.160
4.157
4.148
4.145
4.144
4.136
4.132
4.125
4.122
4.120
4.111
4.106
4.099
4.094
4.082
3.679
3.669
3.659
2.090
2.082
2.074
2.062
1.983
1.975
1.970
1.963
1.956
1.863
1.849
1.839
1.828
1.820
1.818
1.816
1.812
1.802
1.791
1.718
1.700
1.692
1.345
OEt
OEt
OH
10
9
8
7
6
5
4
3
2
1
0
–1
ppm
4.00
2.03
2.03
2.09
4.01
3.98
6.15

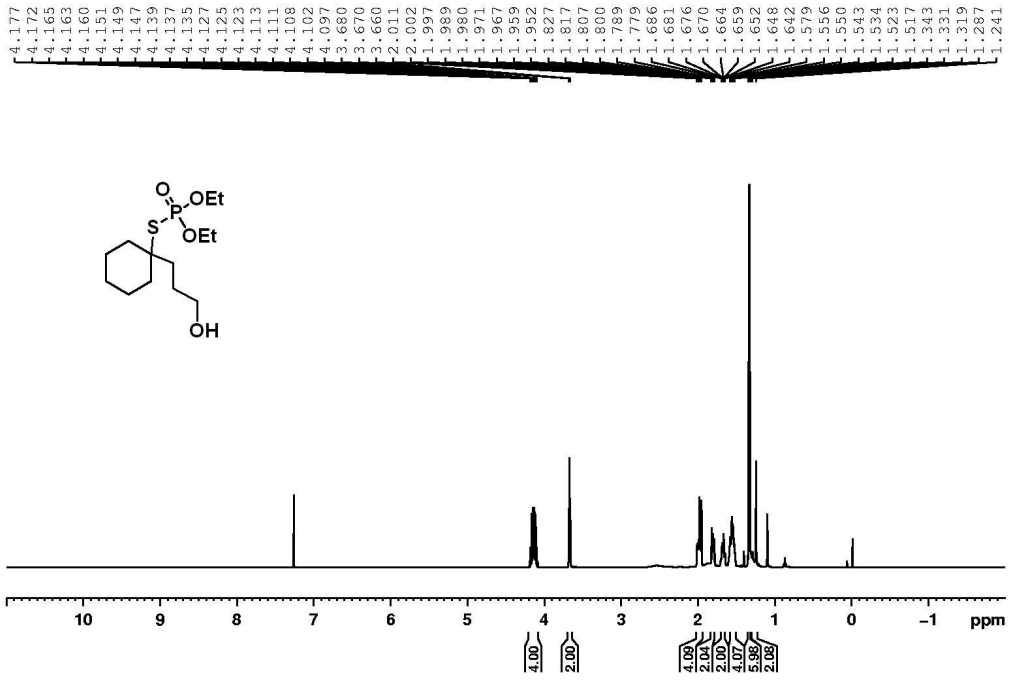
**3v**


64.84
64.81
63.88
63.84
62.86
40.91
40.86
37.45
29.39
23.81
16.35
16.30
OEt
OEt
OH
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

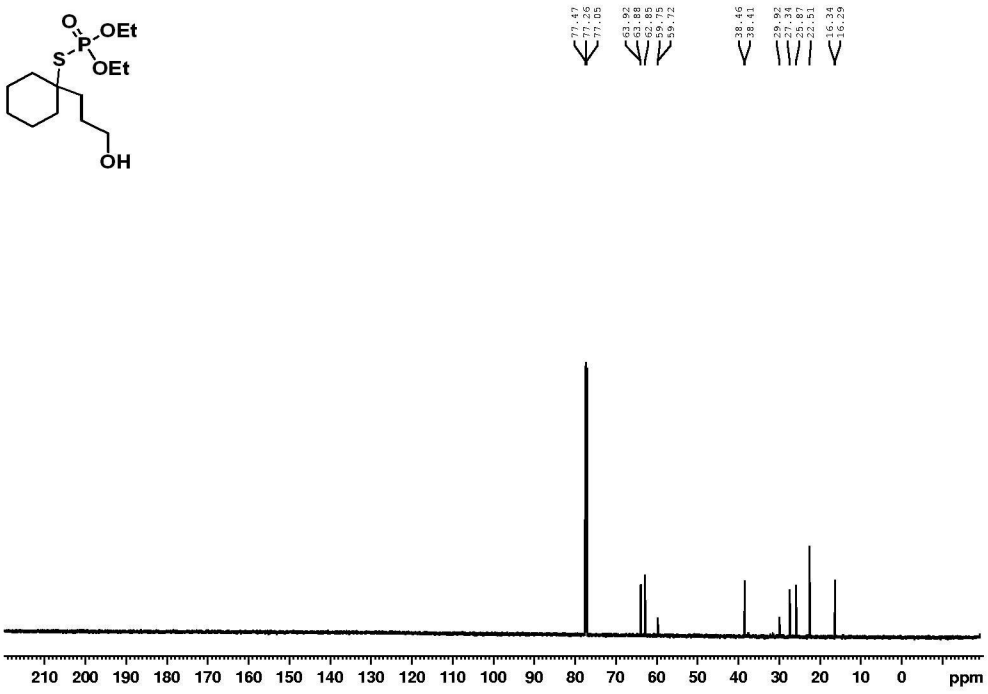
**3w**


O
OEt
P
S
OEt
OH
25.26
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

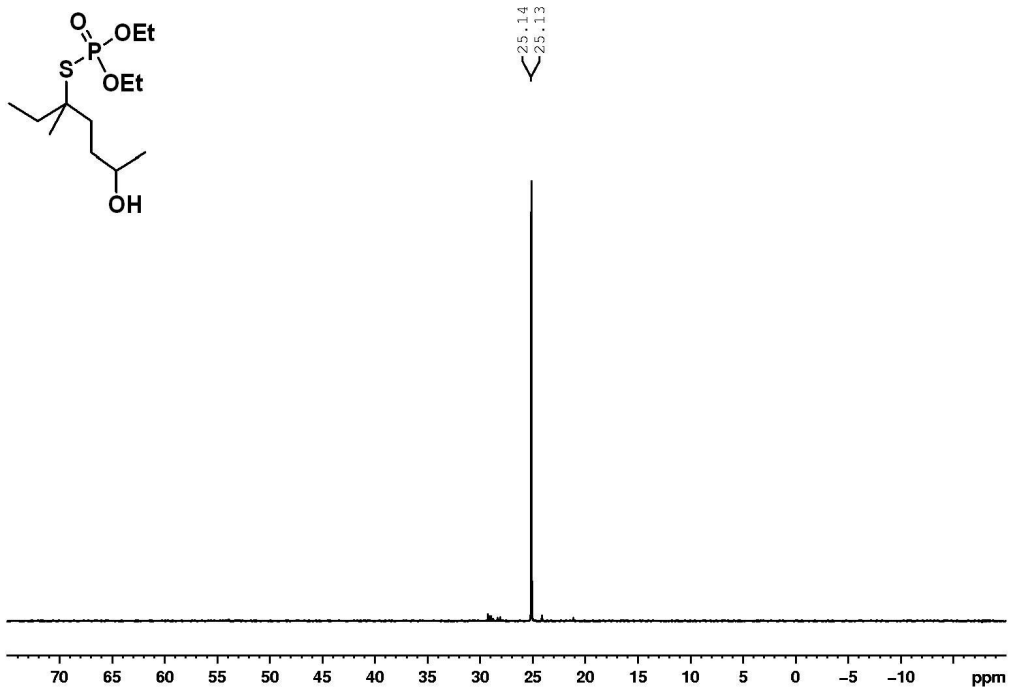
**3w**


4.177 4.172 4.165 4.163 4.160 4.151 4.149 4.147 4.139 4.137 4.135 4.127 4.125 4.123 4.113 4.111 4.108 4.102 4.097 3.680 3.670 3.660 2.011 2.002 1.997 1.989 1.980 1.971 1.967 1.959 1.952 1.827 1.817 1.807 1.800 1.789 1.779 1.686 1.681 1.676 1.670 1.664 1.659 1.652 1.648 1.642 1.579 1.556 1.550 1.543 1.534 1.523 1.517 1.343 1.331 1.319 1.287 1.241
O
OEt
P
S
OEt
OH
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
4.00 2.00 4.09 2.04 2.00 4.07 5.96 2.09

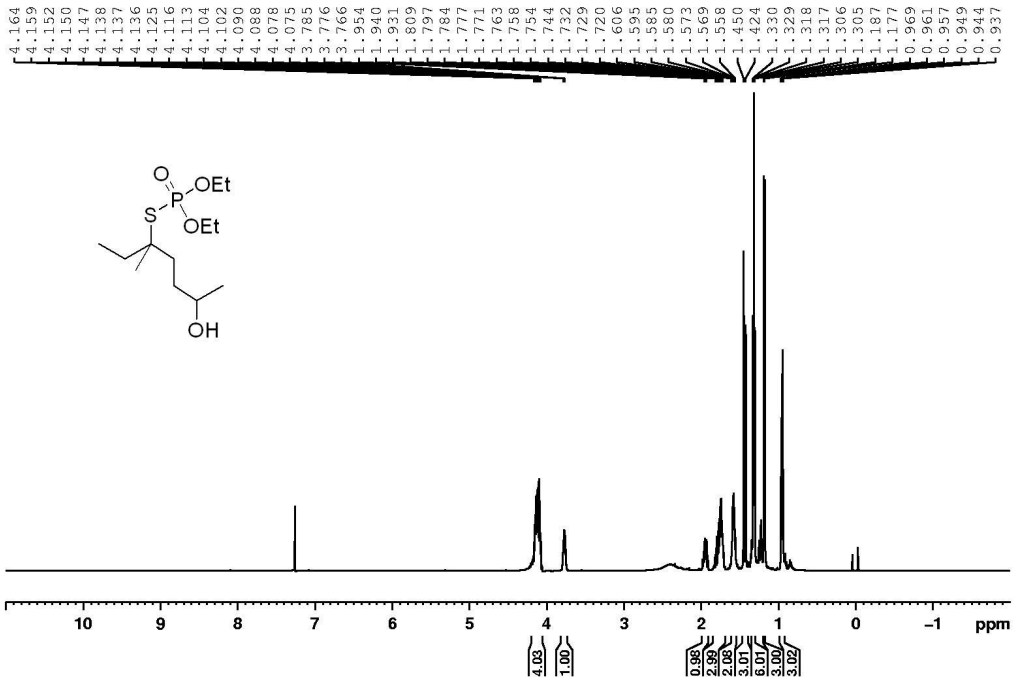
**3w**


O
OEt
P
S
OEt
OH
77.47
77.36
77.05
63.92
63.88
62.85
59.75
59.72
38.46
38.41
29.92
27.34
25.87
22.51
16.34
16.29
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

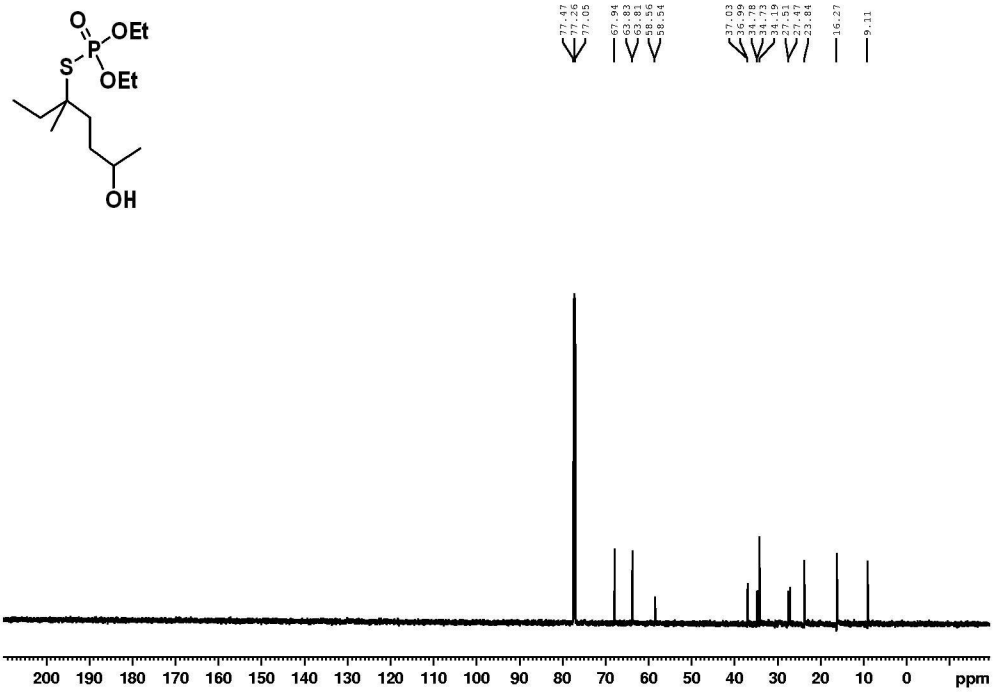
**3x**


O
OEt
P
S
OEt
OH
25.14
25.13
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

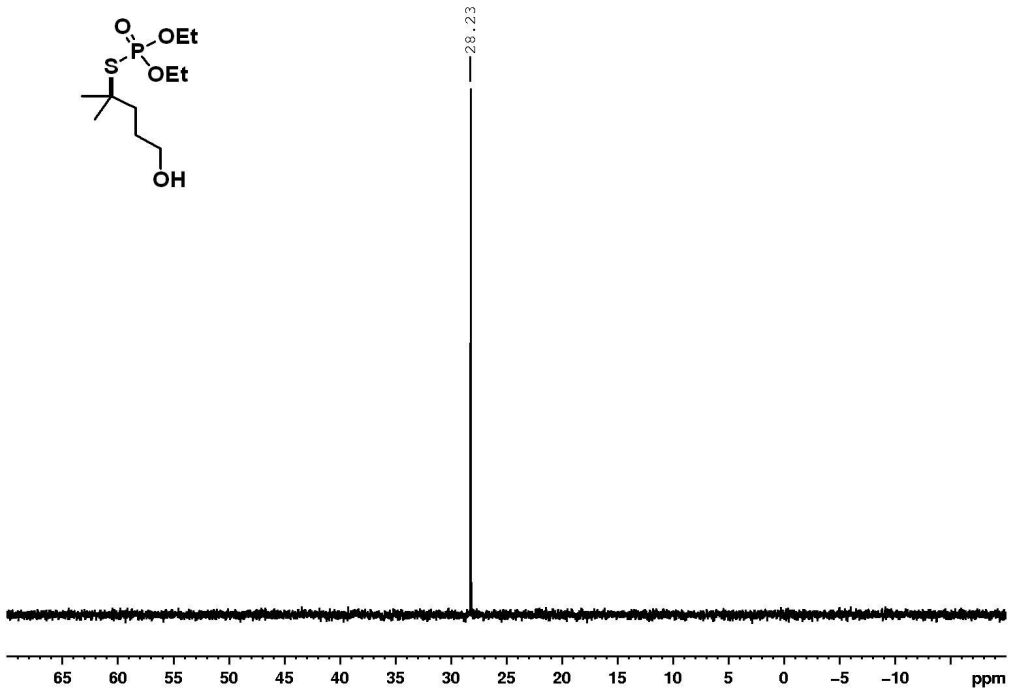
**3x**


4.164
4.159
4.152
4.150
4.147
4.138
4.137
4.136
4.125
4.116
4.113
4.104
4.102
4.090
4.088
4.078
4.075
3.785
3.776
3.766
1.954
1.940
1.931
1.809
1.797
1.784
1.777
1.771
1.763
1.758
1.754
1.744
1.732
1.729
1.720
1.606
1.595
1.585
1.580
1.573
1.569
1.558
1.450
1.424
1.330
1.329
1.318
1.317
1.306
1.305
1.187
1.177
0.969
0.961
0.957
0.949
0.944
0.937
OEt
OEt
OH
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
4.03
1.00
0.98
2.99
2.08
3.01
6.01
3.00
3.02

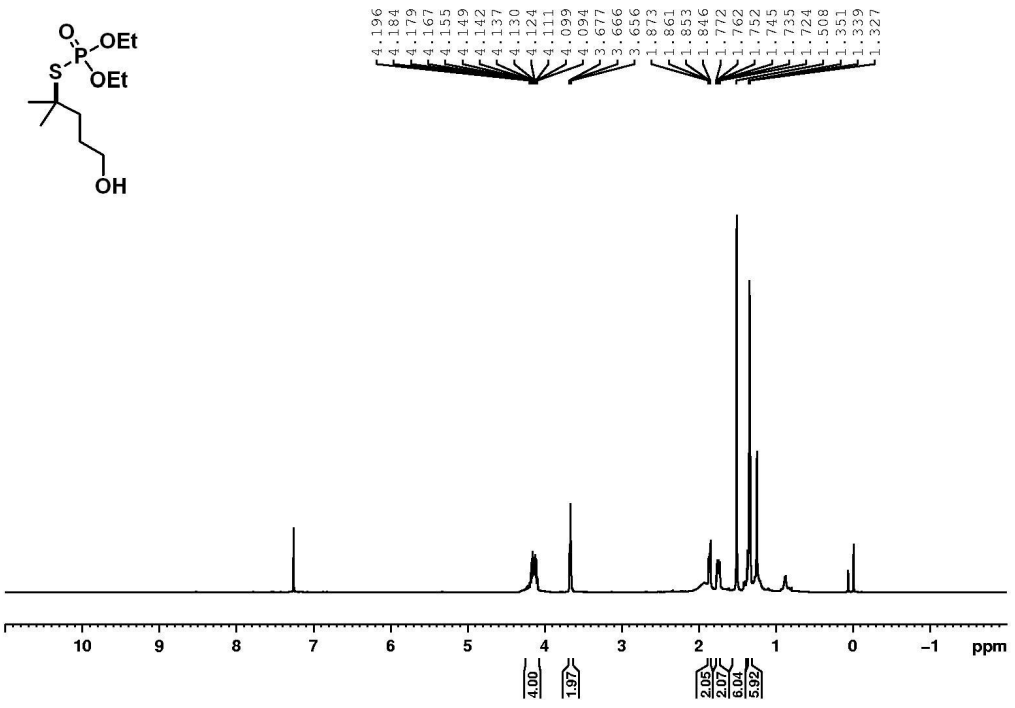
**3x**


OEt
OEt
OH
77.47
77.26
77.05
67.94
63.83
63.81
58.56
58.54
37.03
36.99
34.78
34.73
34.19
27.51
27.47
23.84
16.27
9.11
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

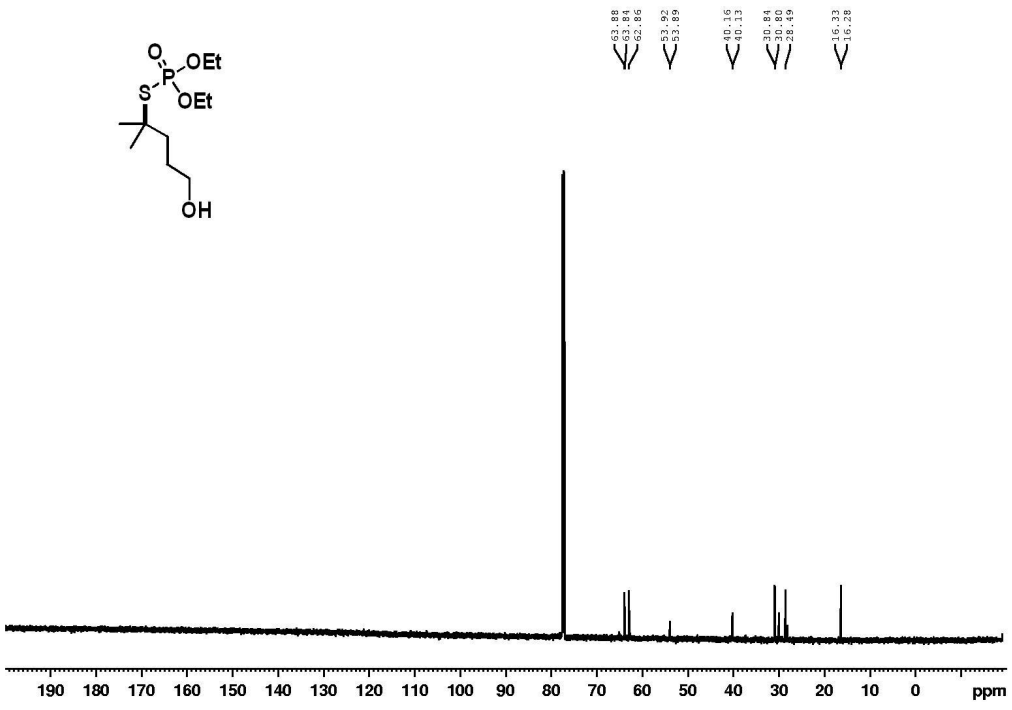
**3y**


O
OEt
P
S
OEt
OH
28.23
65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

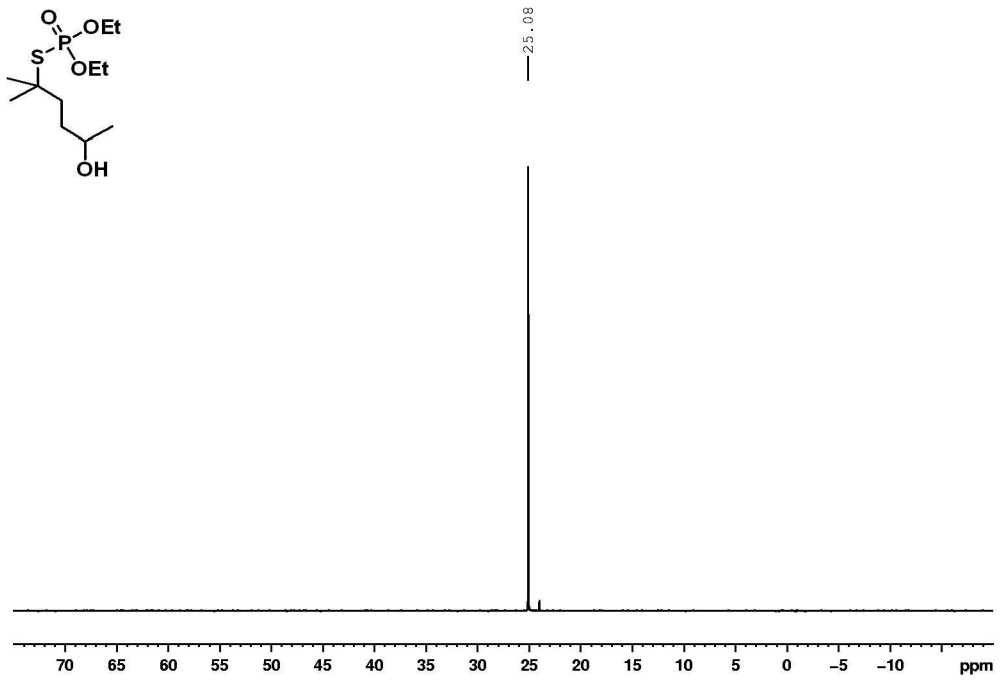
**3y**


O
OEt
P
S
OEt
OH
4.196
4.184
4.179
4.167
4.155
4.149
4.142
4.137
4.130
4.124
4.111
4.099
4.094
3.677
3.666
3.656
1.873
1.861
1.853
1.846
1.772
1.762
1.752
1.745
1.735
1.724
1.508
1.351
1.339
1.327
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
4.00
1.97
2.05
2.07
6.04
5.92

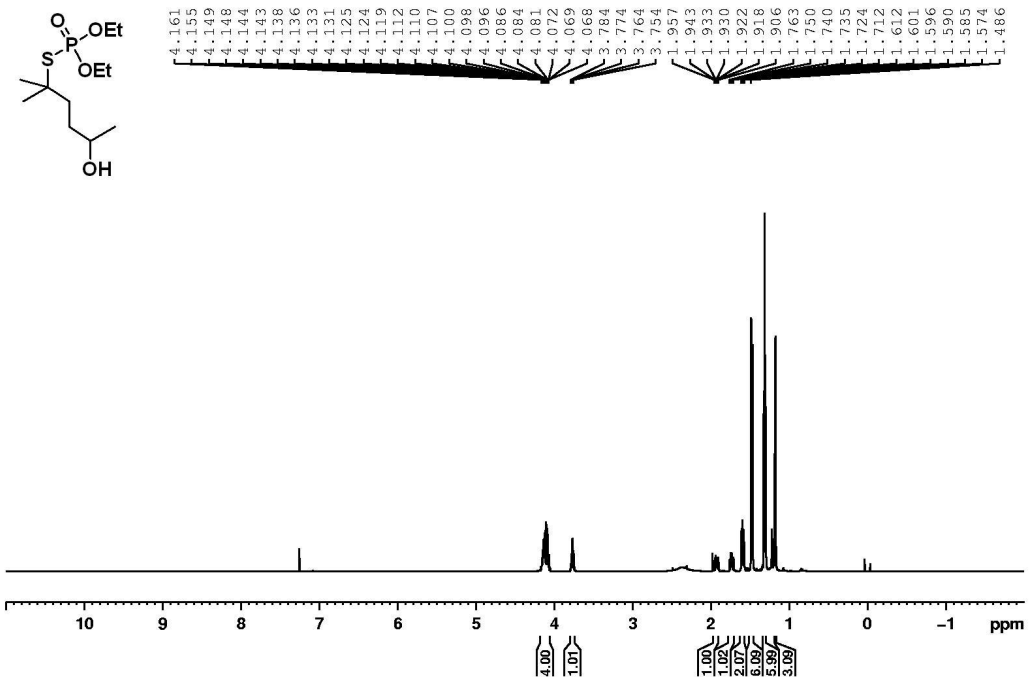
**3y**


63.88
63.84
62.86
53.92
53.89
40.16
40.13
30.84
30.80
28.49
16.33
16.28
OEt
OEt
OH
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

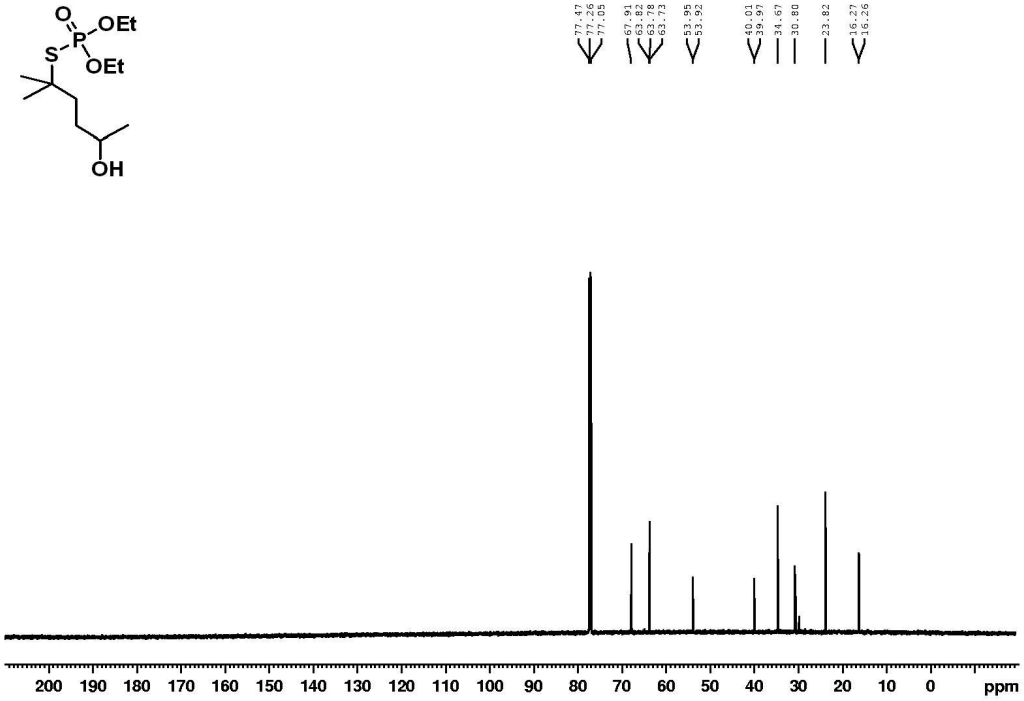
**3z**


25.08
OEt
OEt
OH
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

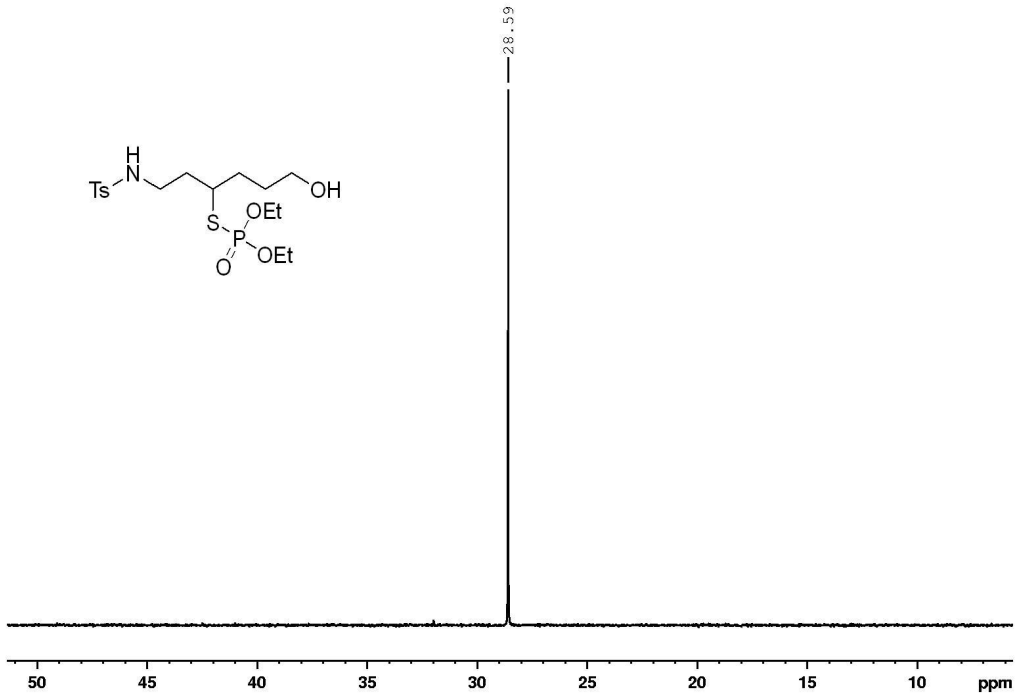
**3z**


4.161
4.155
4.149
4.148
4.144
4.143
4.138
4.136
4.133
4.131
4.125
4.124
4.119
4.112
4.110
4.107
4.100
4.098
4.096
4.086
4.084
4.081
4.072
4.069
4.068
3.784
3.774
3.764
3.754
1.957
1.943
1.933
1.930
1.922
1.918
1.906
1.763
1.750
1.740
1.735
1.724
1.712
1.612
1.601
1.596
1.590
1.585
1.574
1.486
OEt
OEt
S
P
O
OH
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
4.00
1.01
1.00
1.02
2.07
6.09
5.89
3.09

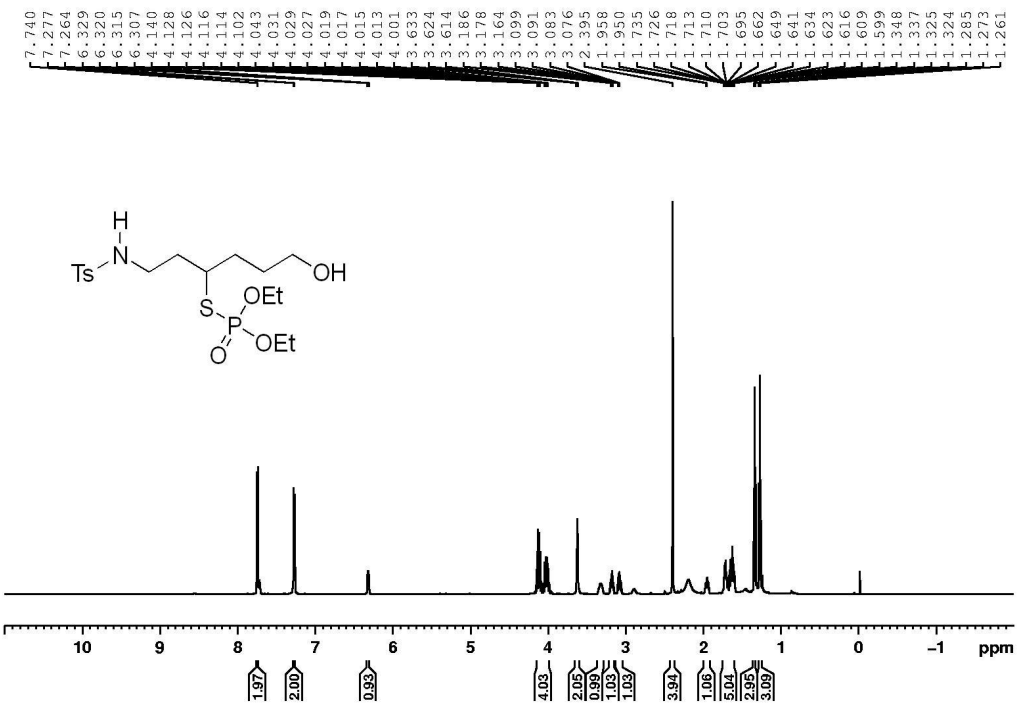
**3z**


77.47
77.26
77.05
67.91
63.82
63.78
63.73
53.95
53.92
40.01
39.97
34.67
30.80
23.82
16.27
16.26
OEt
OEt
S
P
O
OH
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

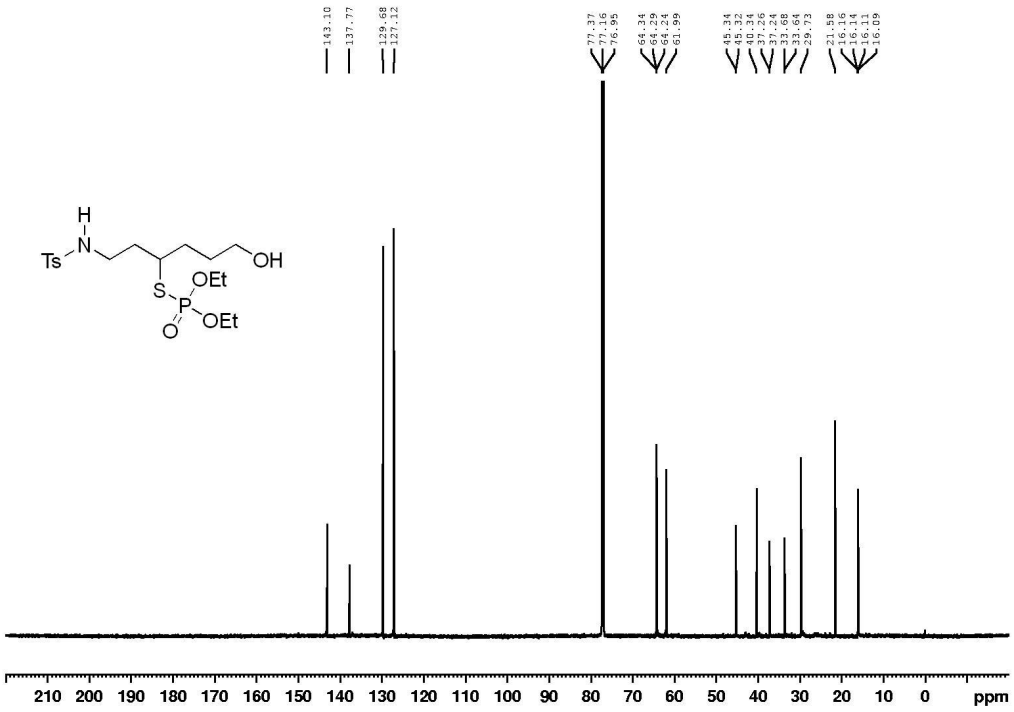
**3ac**


28.59
H
N
Ts
OH
S
OEt
P
O
OEt
50
45
40
35
30
25
20
15
10
ppm

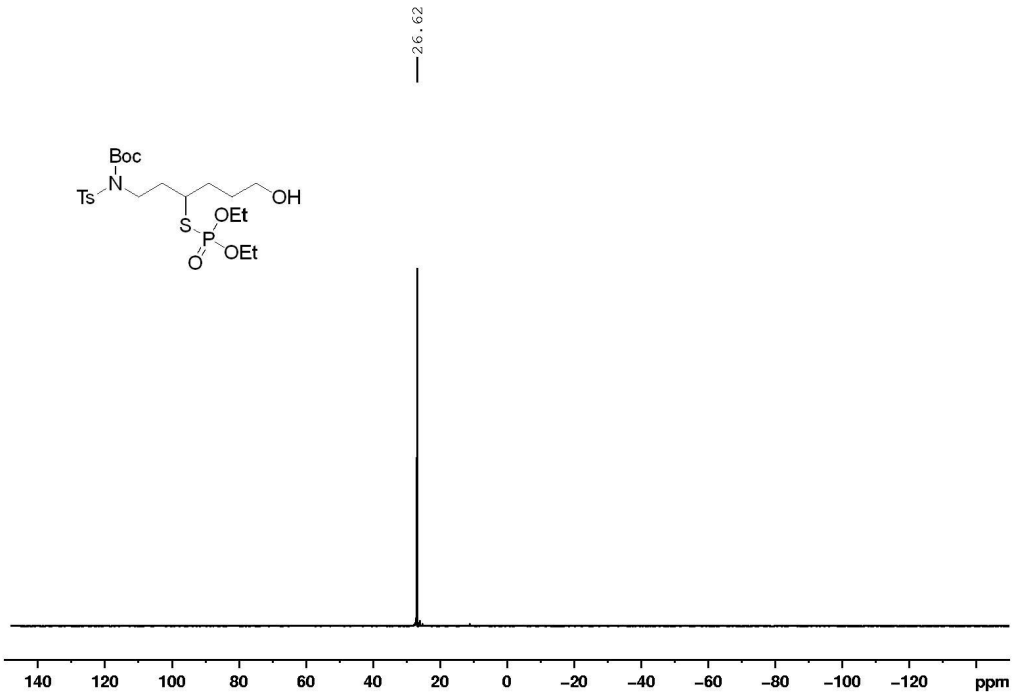
**3ac**


7.740
7.277
7.264
6.329
6.320
6.315
6.307
4.140
4.128
4.126
4.116
4.114
4.102
4.043
4.031
4.029
4.027
4.019
4.017
4.015
4.013
4.001
3.633
3.624
3.614
3.186
3.178
3.164
3.099
3.091
3.083
3.076
2.395
1.958
1.950
1.735
1.726
1.718
1.713
1.710
1.703
1.695
1.662
1.649
1.641
1.634
1.623
1.616
1.609
1.599
1.348
1.337
1.325
1.324
1.285
1.273
1.261
H
N
Ts
OH
S
OEt
P
O
OEt
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
1.97
2.00
0.93
4.03
2.05
0.99
1.03
1.03
3.94
1.06
5.04
2.95
3.09

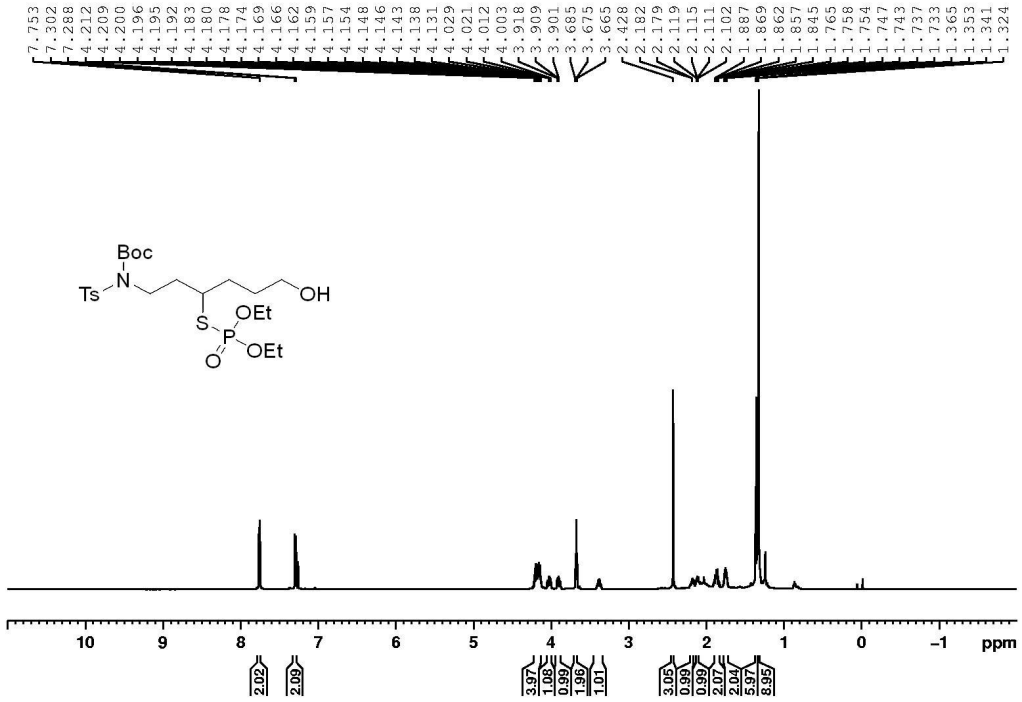
**3ac**


143.10
137.77
129.68
127.32
77.37
77.16
76.95
64.34
64.29
64.24
61.99
45.34
45.22
40.34
37.26
37.24
33.68
33.64
29.73
21.58
16.16
16.14
16.11
16.09
H
N
Ts
OH
S
OEt
P
O
OEt
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

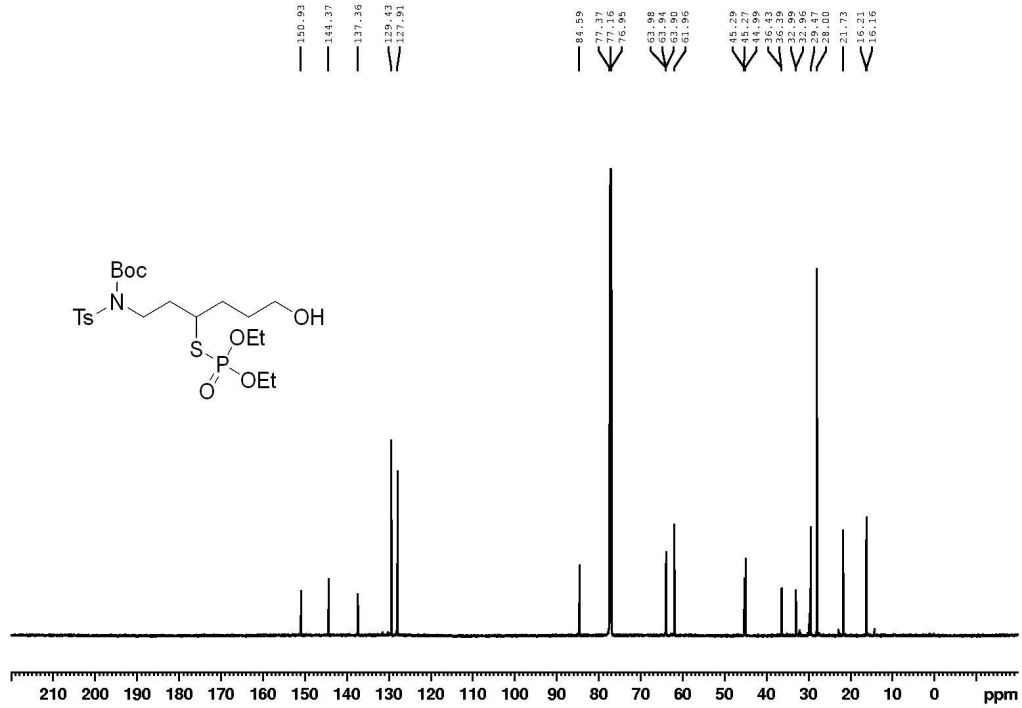
**3ad**


26.62
Boc
N
Ts
OH
S
OEt
P
O
OEt
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 ppm

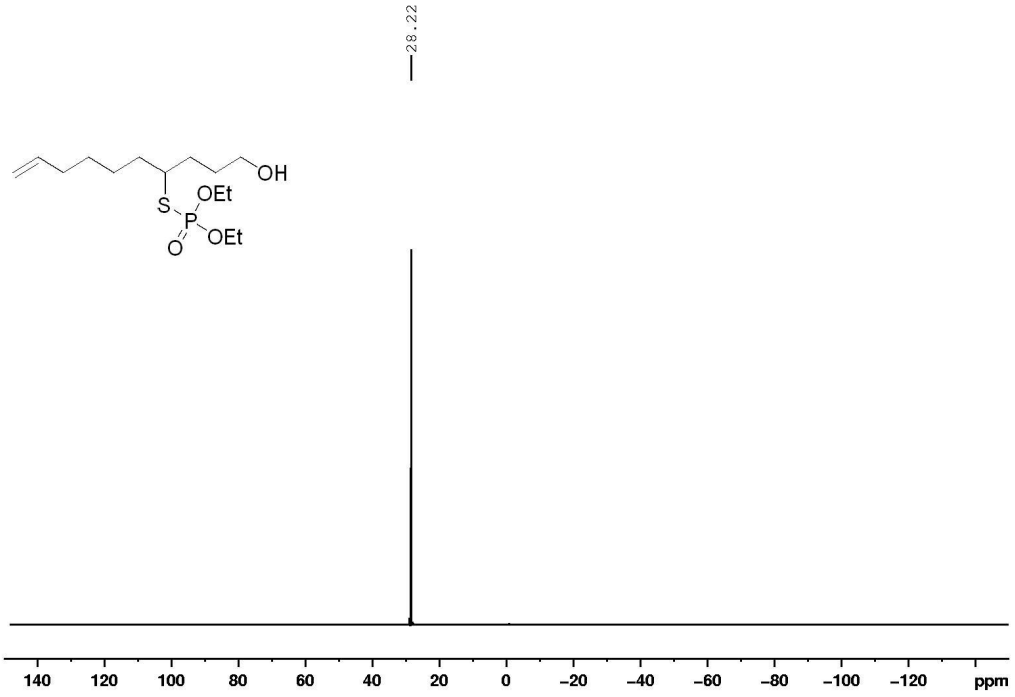
**3ad**


7.753
7.302
7.288
4.212
4.209
4.200
4.196
4.195
4.192
4.183
4.180
4.178
4.174
4.169
4.166
4.162
4.159
4.157
4.154
4.148
4.146
4.143
4.138
4.131
4.029
4.021
4.012
4.003
3.918
3.909
3.901
3.685
3.675
3.665
2.428
2.182
2.179
2.119
2.115
2.111
2.102
1.887
1.869
1.862
1.857
1.845
1.765
1.758
1.754
1.747
1.743
1.737
1.733
1.365
1.353
1.341
1.324
Boc
N
Ts
OH
S
OEt
P
O
OEt
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
2.02
2.09
3.97
1.08
0.99
1.96
1.01
3.05
0.99
0.99
2.01
2.04
5.97
8.95

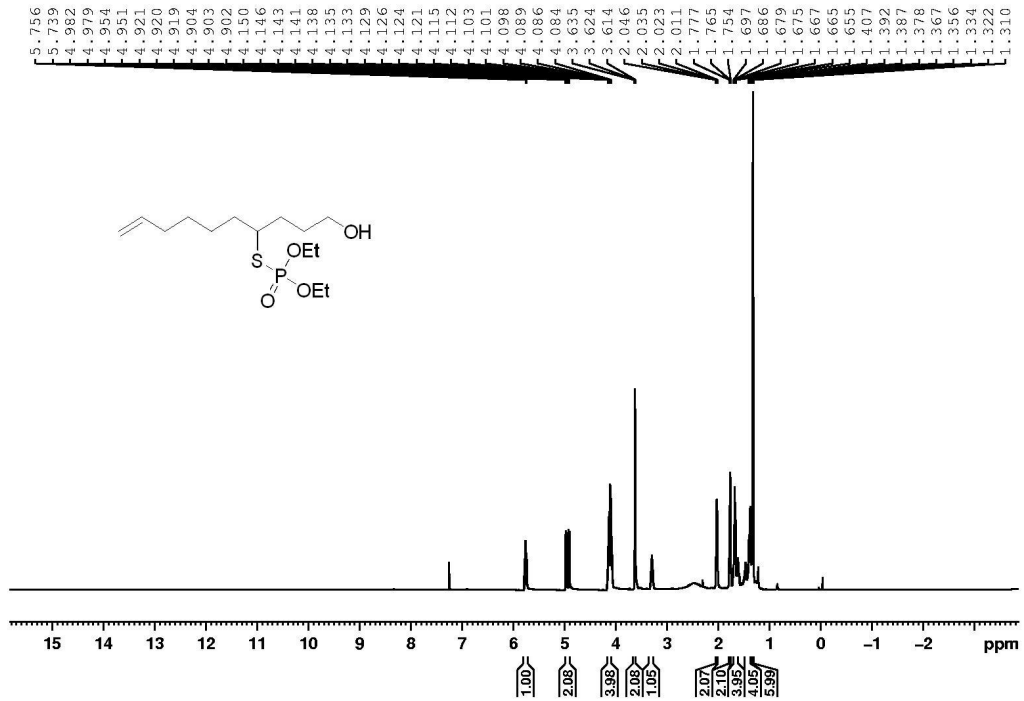
**3ad**


150.93
144.37
137.36
129.43
127.91
84.59
77.37
77.16
76.95
63.98
63.84
63.90
61.96
45.39
45.27
44.99
36.43
36.39
32.99
32.66
29.47
28.00
21.73
16.21
16.16
Boc
N
Ts
OH
S
OEt
P
O
OEt
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

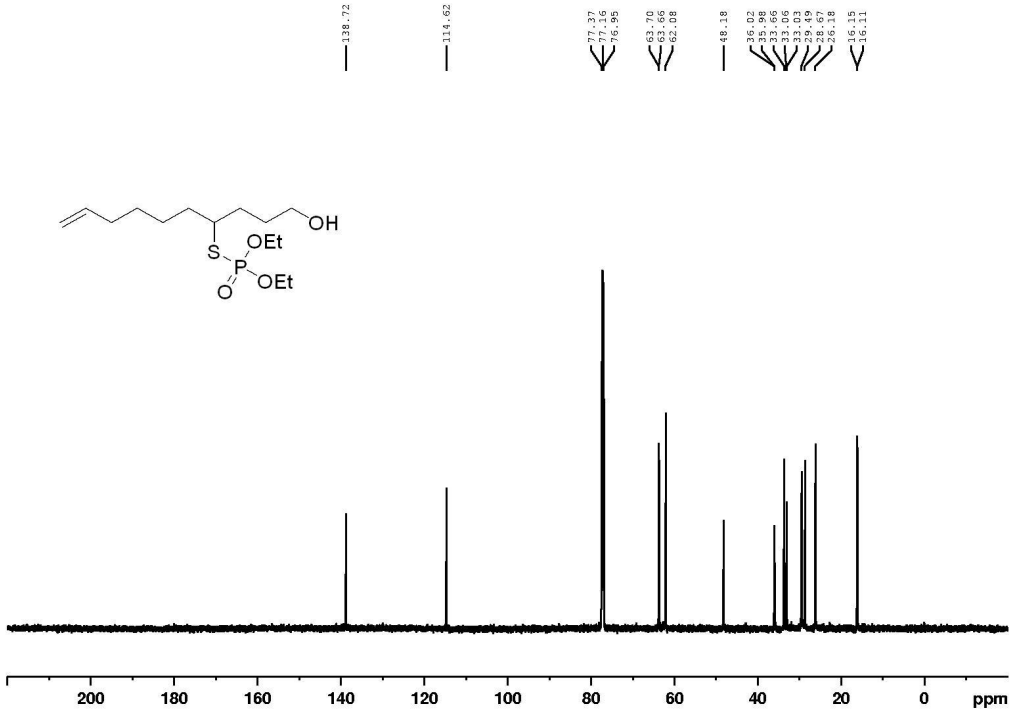
**3ae**


28.22
OH
S
OEt
P
O
OEt
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
ppm

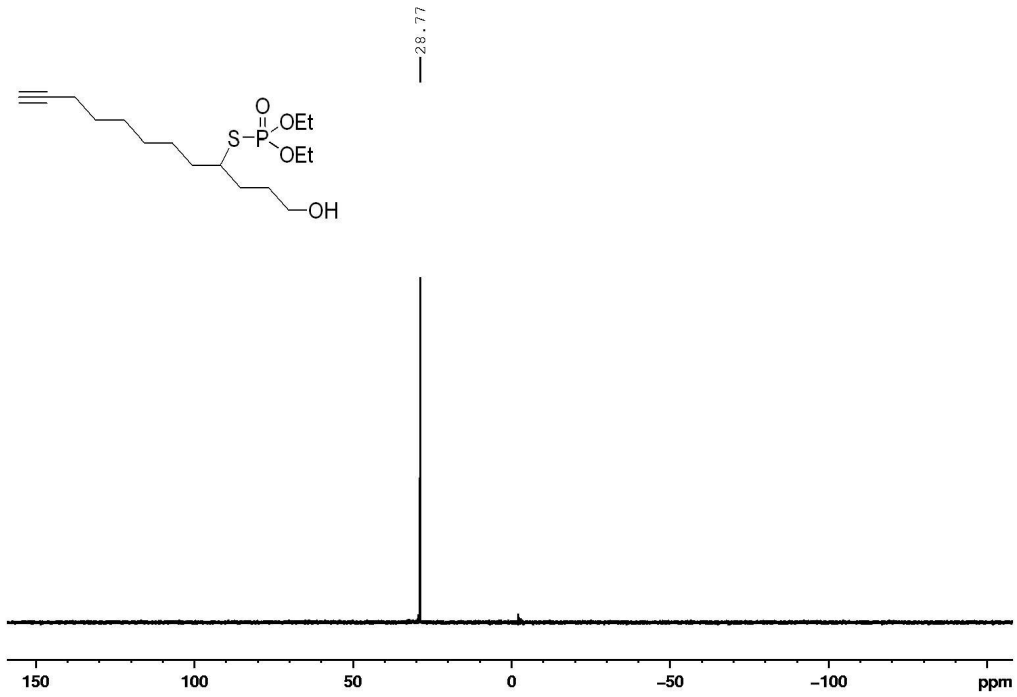
**3ae**


5.756
5.739
4.982
4.979
4.954
4.951
4.921
4.920
4.919
4.904
4.903
4.902
4.150
4.146
4.143
4.141
4.138
4.135
4.133
4.129
4.126
4.124
4.121
4.115
4.112
4.103
4.101
4.098
4.089
4.086
4.084
3.635
3.624
3.614
2.046
2.035
2.023
2.011
1.777
1.765
1.754
1.697
1.686
1.679
1.675
1.667
1.665
1.655
1.407
1.392
1.387
1.378
1.367
1.356
1.334
1.322
1.310
OH
S
OEt
P
O
OEt
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
ppm
1.00
2.08
3.98
2.08
1.05
2.07
2.10
3.95
4.05
5.99

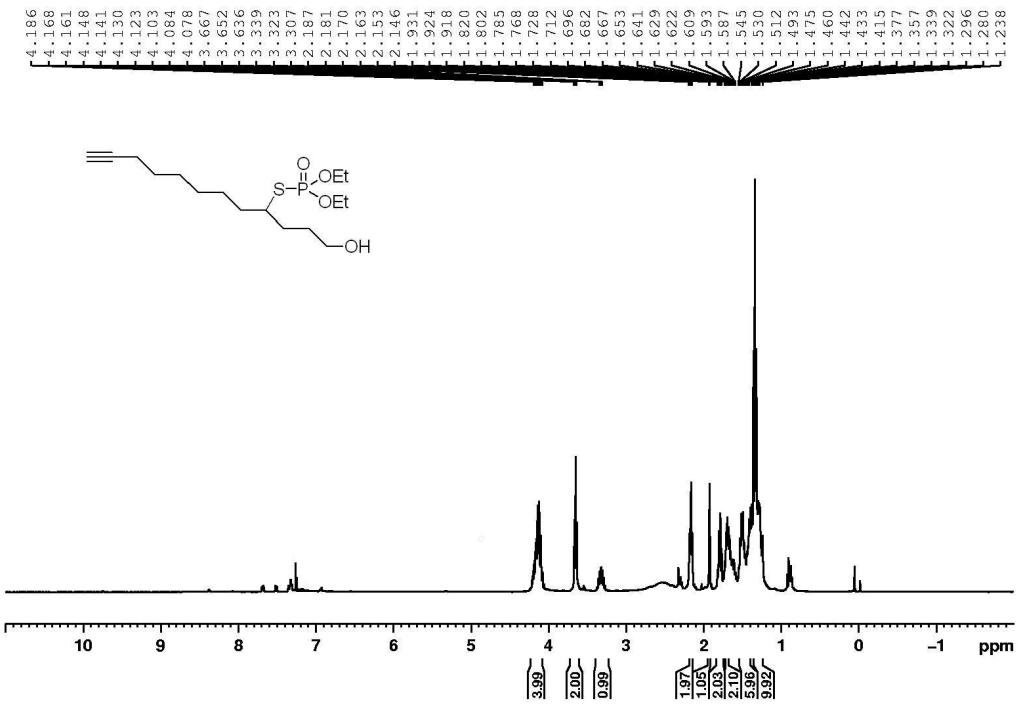
**3ae**


138.72
114.62
77.37
77.16
76.95
63.70
63.66
62.08
48.18
36.02
35.98
33.66
33.06
31.23
29.49
28.67
26.18
16.15
16.11
OH
S
OEt
P
OEt
O
200
180
160
140
120
100
80
60
40
20
0
ppm

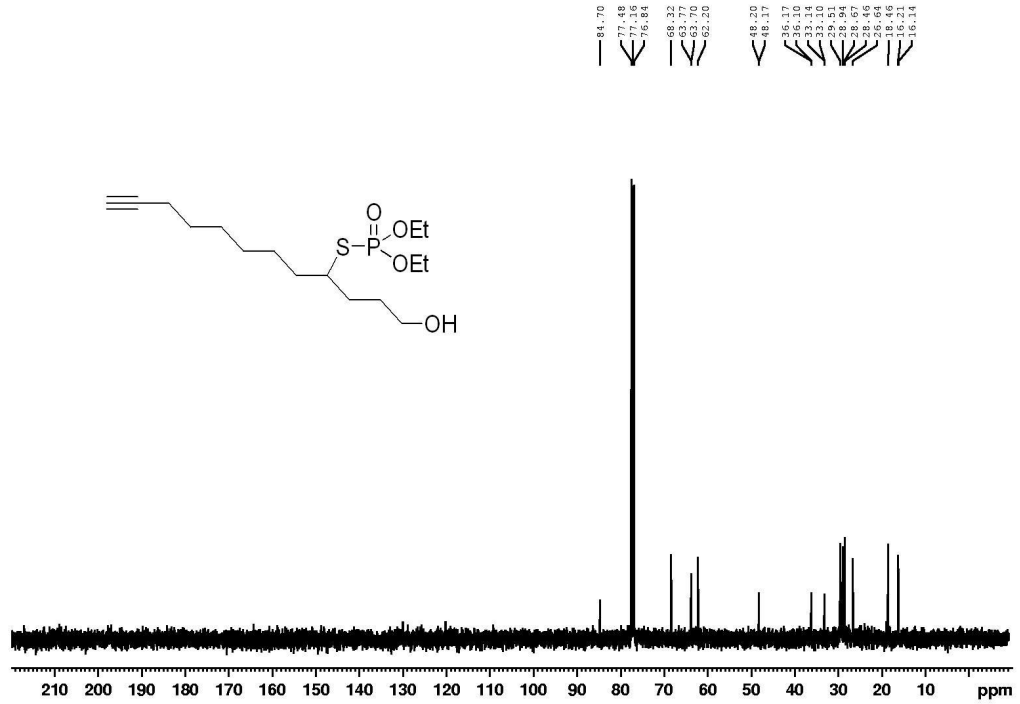
**3af**


28.77
O
S
P
OEt
OEt
OH
150
100
50
0
-50
-100
ppm

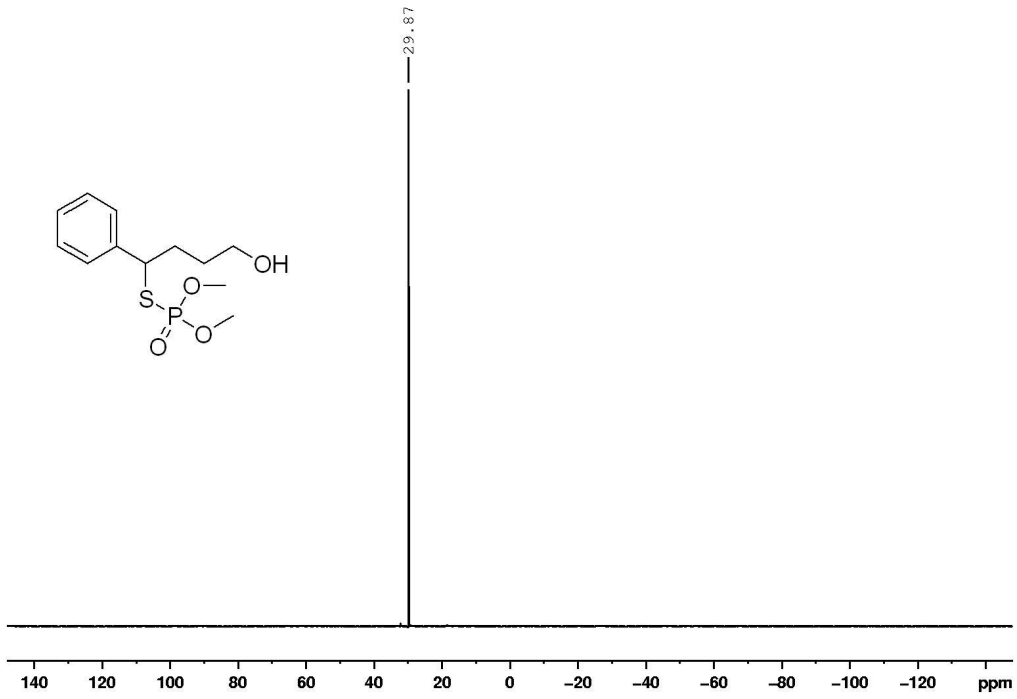
**3af**


4.186
4.168
4.161
4.148
4.141
4.130
4.123
4.103
4.084
4.078
3.667
3.652
3.636
3.339
3.323
3.307
2.187
2.181
2.170
2.163
2.153
2.146
1.931
1.924
1.918
1.820
1.802
1.785
1.768
1.728
1.712
1.696
1.682
1.667
1.653
1.641
1.629
1.622
1.609
1.593
1.587
1.545
1.530
1.512
1.493
1.475
1.460
1.442
1.433
1.415
1.377
1.357
1.339
1.322
1.296
1.280
1.238
O
OEt
S
P
OEt
OH
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
3.99
2.00
0.99
1.97
1.05
2.03
2.10
5.96
9.92

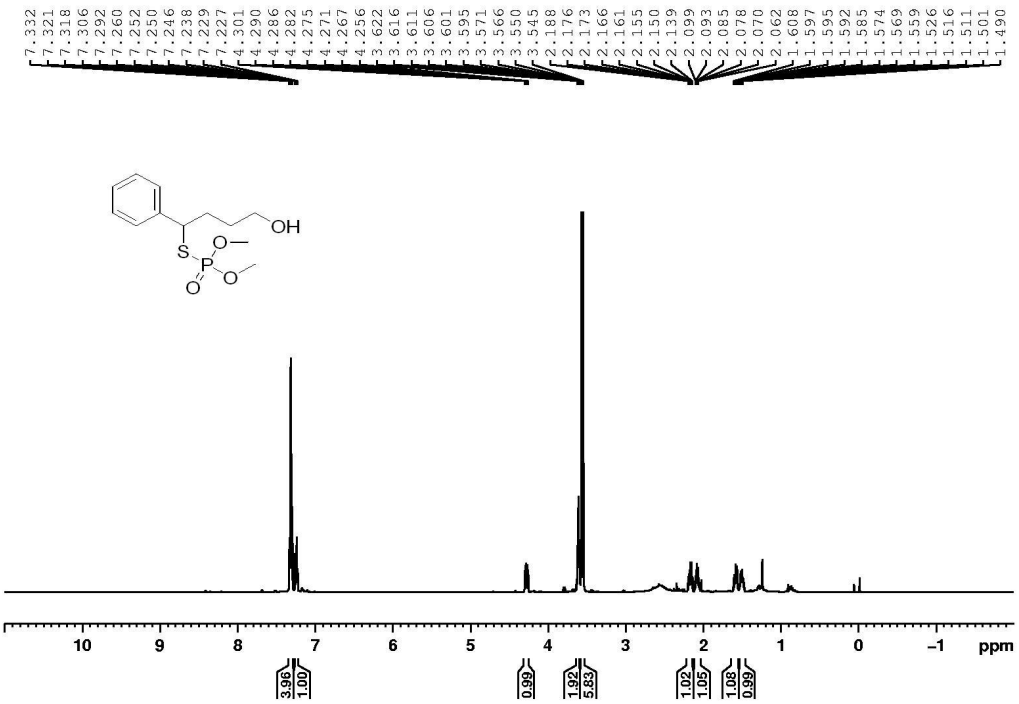
**3af**


84.70
77.48
77.16
76.84
68.32
63.77
63.70
62.20
48.20
48.17
36.17
36.10
33.14
33.10
29.51
28.94
28.67
28.46
26.64
18.46
16.21
16.14
O
OEt
S
P
OEt
OH
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

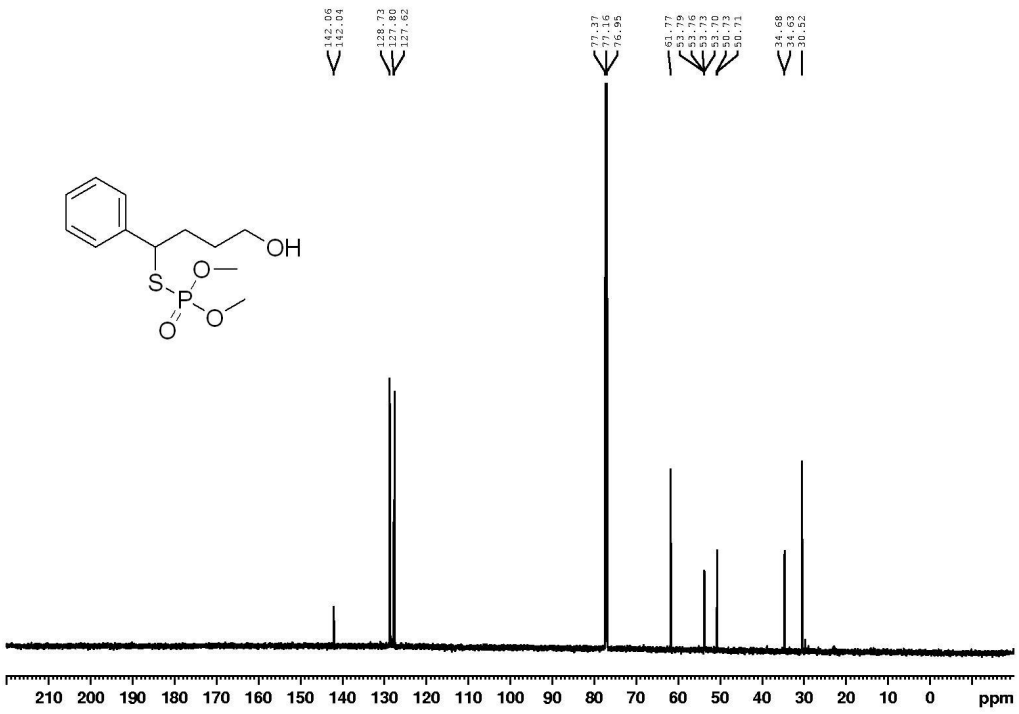
**3ag**


29.87
OH
S
O
P
O
O
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
ppm

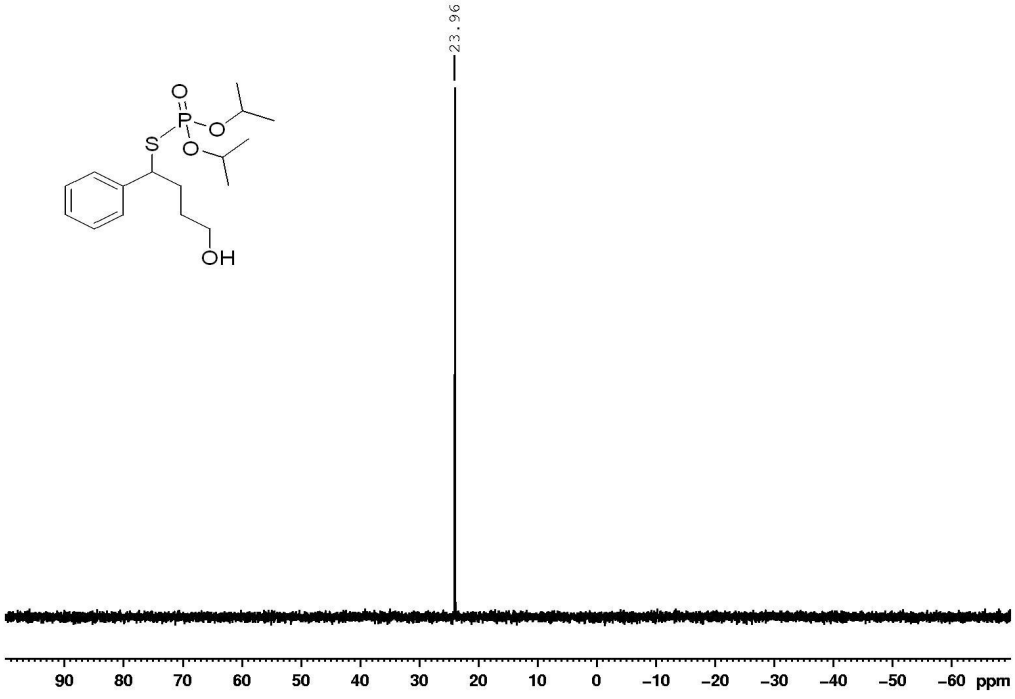
**3ag**


7.332
7.321
7.318
7.306
7.292
7.260
7.252
7.250
7.246
7.238
7.229
7.227
4.301
4.290
4.286
4.282
4.275
4.271
4.267
4.256
3.622
3.616
3.611
3.606
3.601
3.595
3.571
3.566
3.550
3.545
2.188
2.176
2.173
2.166
2.161
2.155
2.150
2.139
2.099
2.093
2.085
2.078
2.070
2.062
1.608
1.597
1.595
1.592
1.585
1.574
1.569
1.559
1.526
1.516
1.511
1.501
1.490
OH
S
O
P
O
O
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
3.96
1.00
0.99
1.92
5.83
1.02
1.05
1.08
0.99

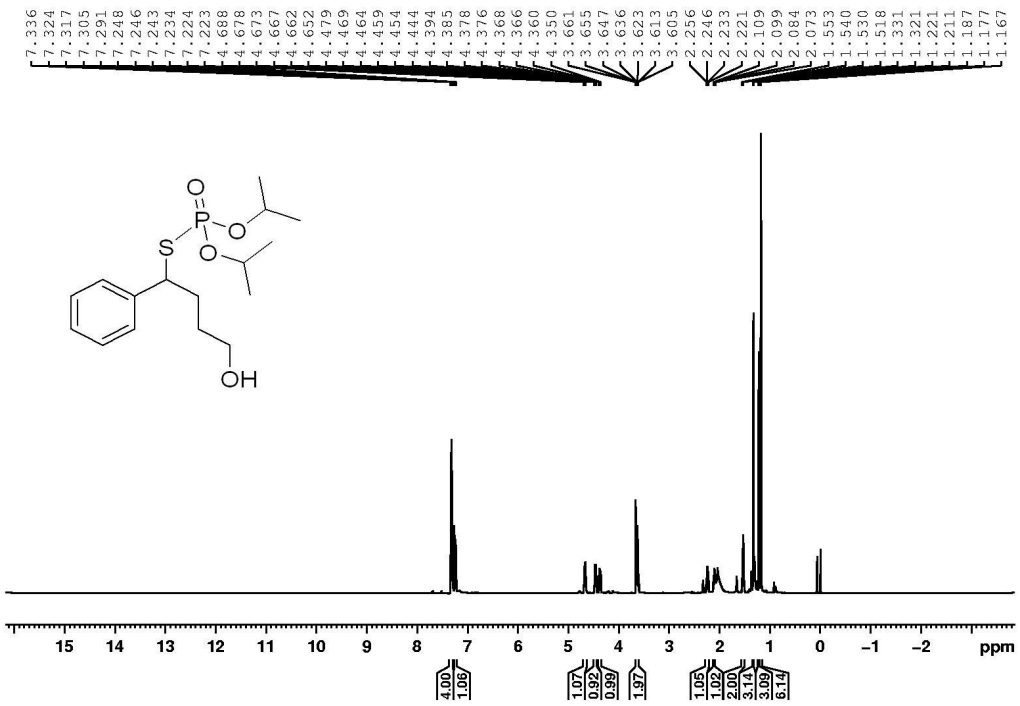
**3ag**


142.06
142.04
128.73
127.80
127.62
77.37
77.16
76.95
61.77
53.79
53.76
53.73
53.70
50.73
50.71
34.68
34.63
30.52
OH
S
O
P
O
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

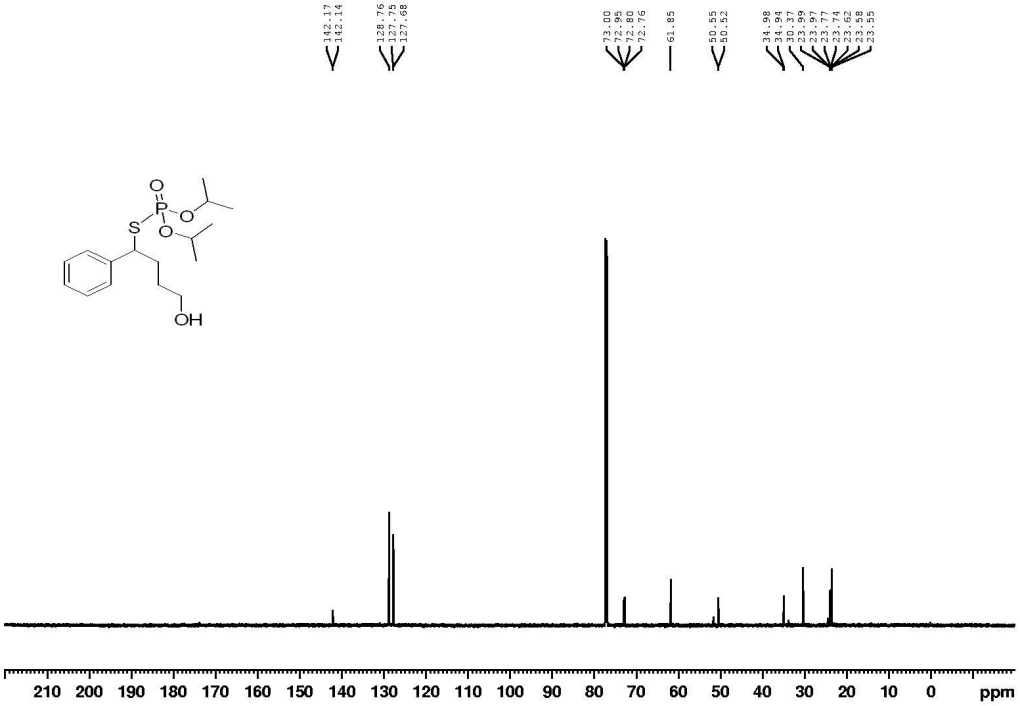
**3ah**


23.96
O
P
S
O
O
OH
90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40 −50 −60 ppm

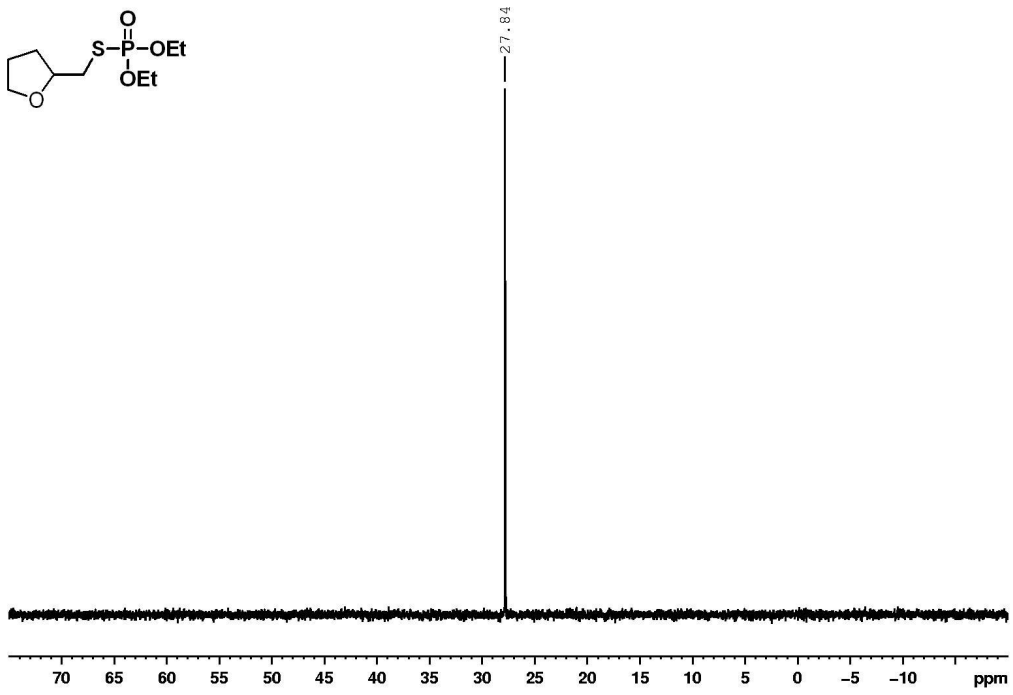
**3ah**


7.336
7.324
7.317
7.305
7.291
7.248
7.246
7.243
7.234
7.224
7.223
4.688
4.678
4.673
4.667
4.662
4.652
4.479
4.469
4.464
4.459
4.454
4.444
4.394
4.385
4.378
4.376
4.368
4.366
4.360
4.350
3.661
3.655
3.647
3.636
3.623
3.613
3.605
2.256
2.246
2.233
2.221
2.109
2.099
2.084
2.073
1.553
1.540
1.530
1.518
1.331
1.321
1.221
1.211
1.187
1.177
1.167
O
P
S
O
O
OH
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 ppm
4.00
1.06
1.07
0.92
0.99
1.97
1.05
1.02
2.00
3.14
3.09
6.14

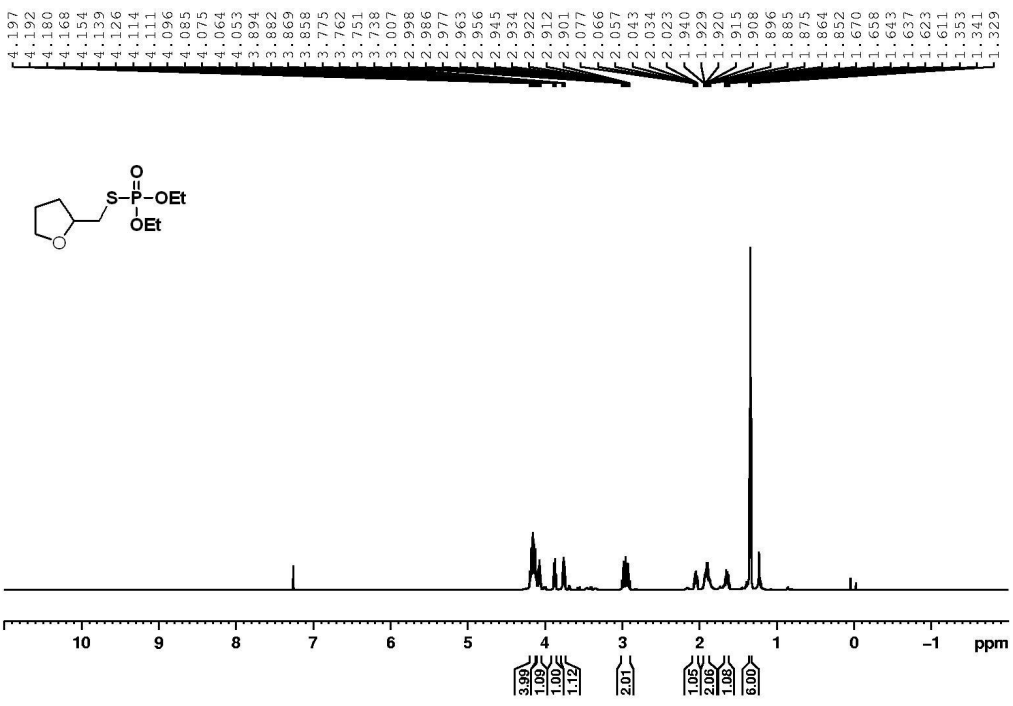
**3ah**


142.17
142.14
128.76
127.75
127.68
73.00
72.85
72.80
72.76
61.85
50.55
50.52
34.98
34.94
30.37
23.99
23.97
23.77
23.74
23.62
23.58
23.55
O
P
S
O
O
OH
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

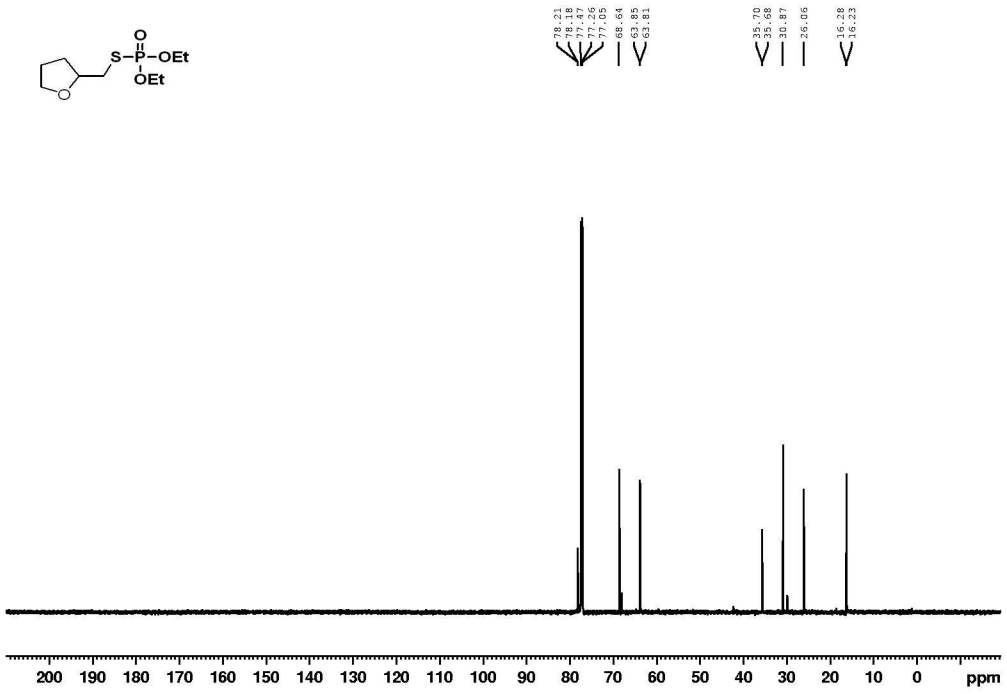
**5a**


27.84
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

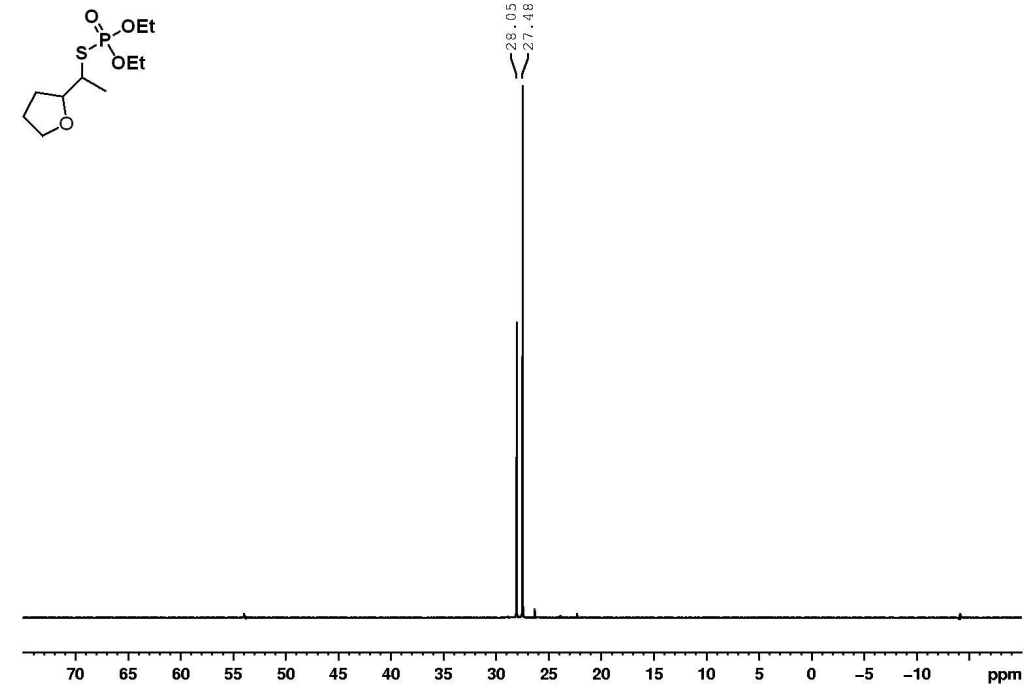
**5a**


4.197
4.192
4.180
4.168
4.154
4.139
4.126
4.114
4.111
4.096
4.085
4.075
4.064
4.053
3.894
3.882
3.869
3.858
3.775
3.762
3.751
3.738
3.007
2.998
2.986
2.977
2.963
2.956
2.945
2.934
2.922
2.912
2.901
2.077
2.066
2.057
2.043
2.034
2.023
1.940
1.929
1.920
1.915
1.908
1.896
1.885
1.875
1.864
1.852
1.670
1.658
1.643
1.637
1.623
1.611
1.353
1.341
1.329
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
3.99
1.09
1.00
1.12
2.01
1.05
2.06
1.08
6.00

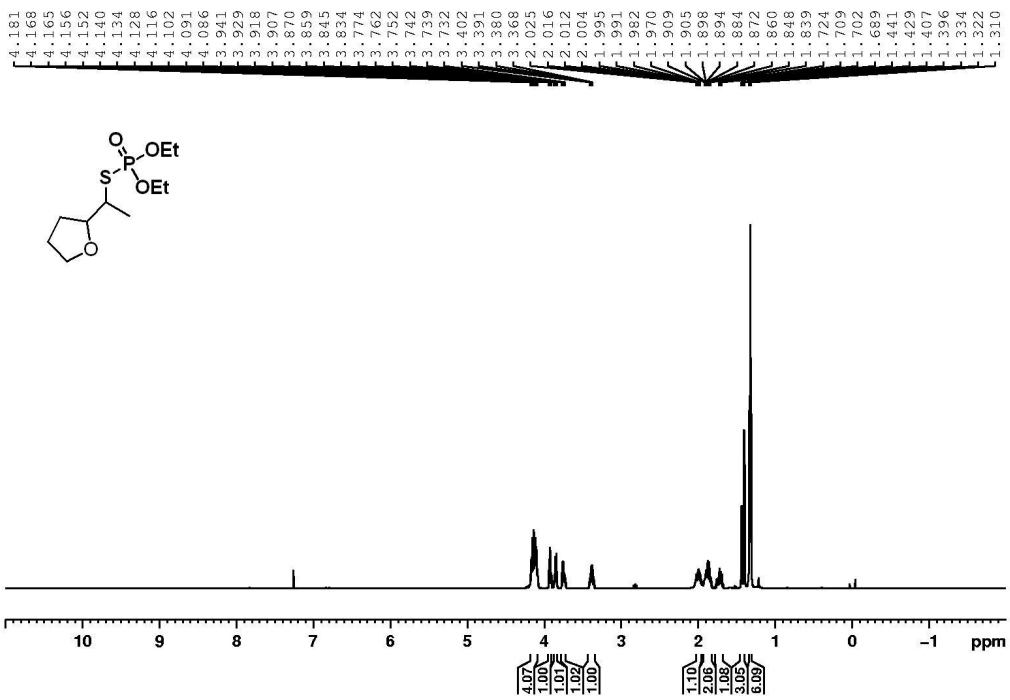
**5a**


O
S–P–OEt
OEt
78.21
78.18
77.47
77.26
77.05
68.64
63.85
63.81
35.70
35.68
30.87
26.06
16.28
16.23
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

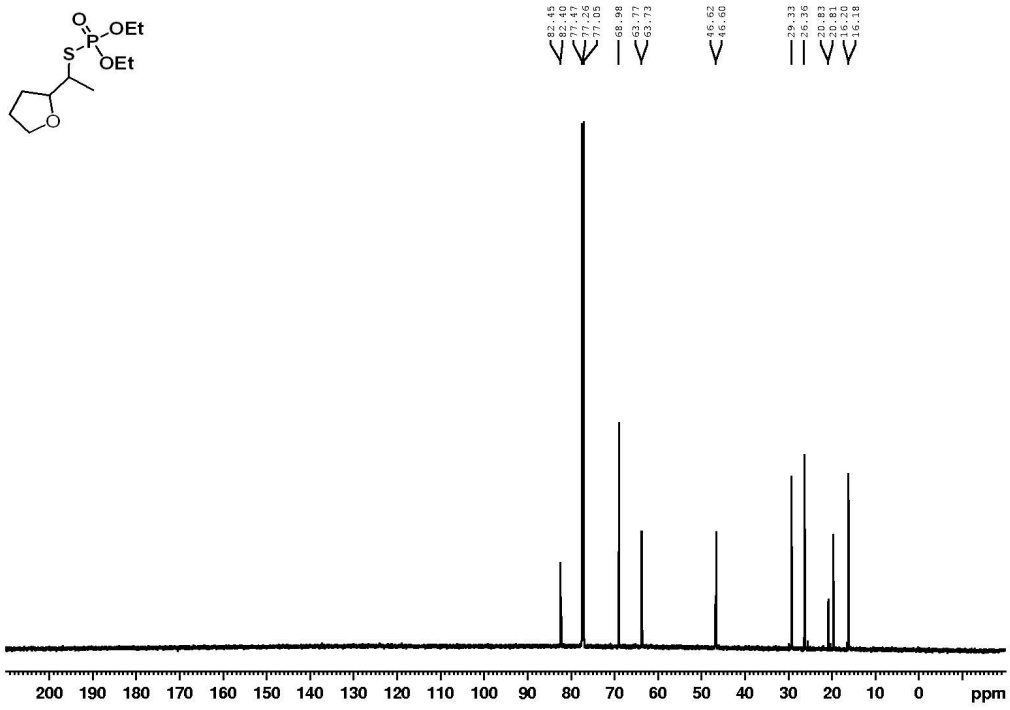
**5b**


O
OEt
S
P
OEt
28.05
27.48
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 –5 –10 ppm

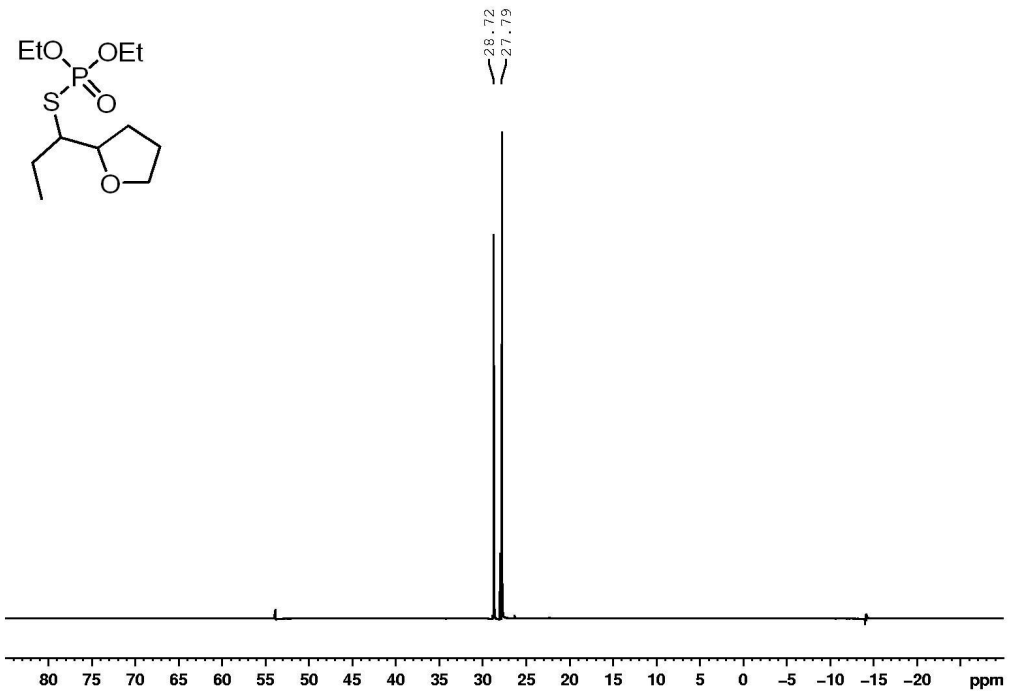
**5b**


4.181
4.168
4.165
4.156
4.152
4.140
4.134
4.128
4.116
4.102
4.091
4.086
3.941
3.929
3.918
3.907
3.870
3.859
3.845
3.834
3.774
3.762
3.752
3.742
3.739
3.732
3.402
3.391
3.380
3.368
2.025
2.016
2.012
2.004
1.995
1.991
1.982
1.970
1.909
1.905
1.898
1.894
1.884
1.872
1.860
1.848
1.839
1.724
1.709
1.702
1.689
1.441
1.429
1.407
1.396
1.334
1.322
1.310
OEt
OEt
O
P
S
O
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
4.07 1.00 1.01 1.02 1.00
1.10 2.06 1.08 3.05 6.09

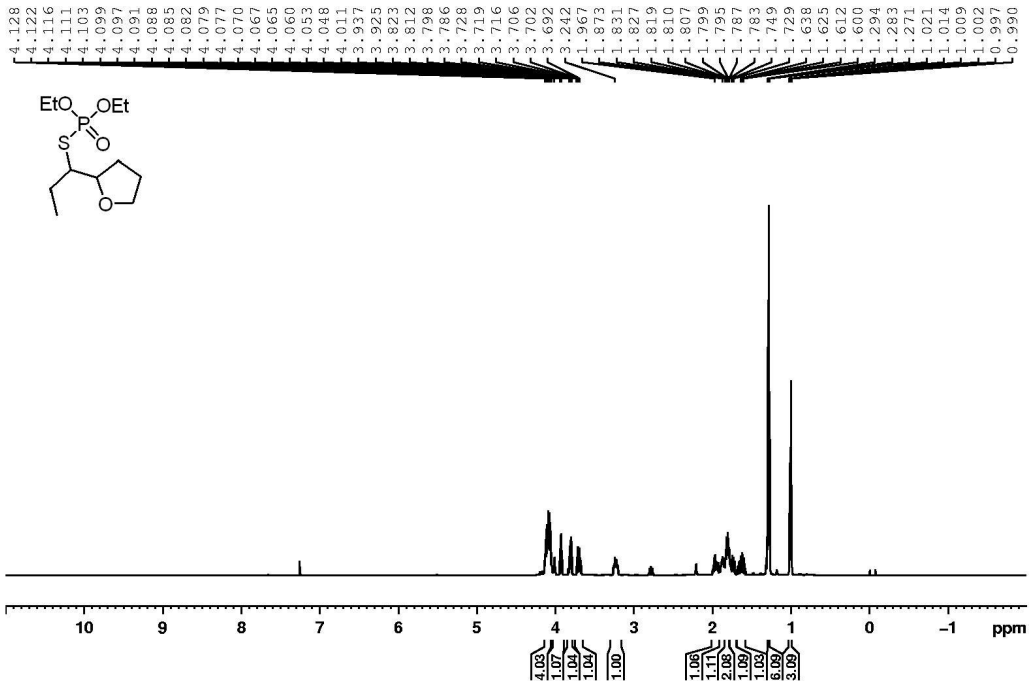
**5b**


OEt
OEt
O
P
S
O
82.45
82.40
82.17
77.26
77.05
68.98
63.77
63.73
46.62
46.60
29.33
26.36
20.83
20.81
16.20
16.18
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

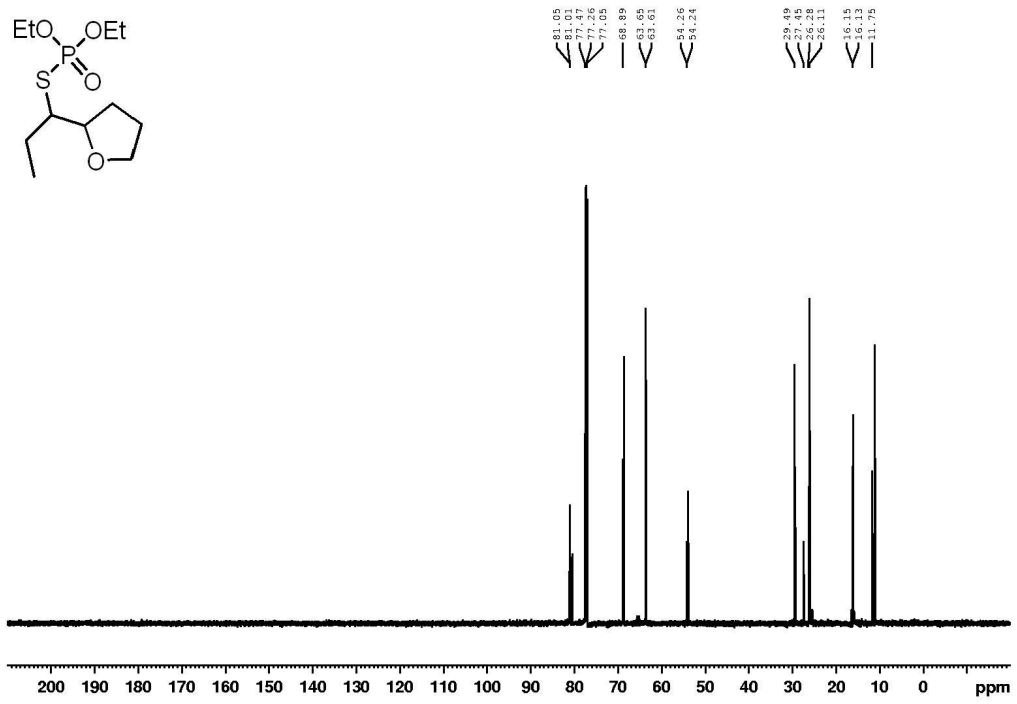
**5c**


28.72
27.79
80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 −15 −20 ppm

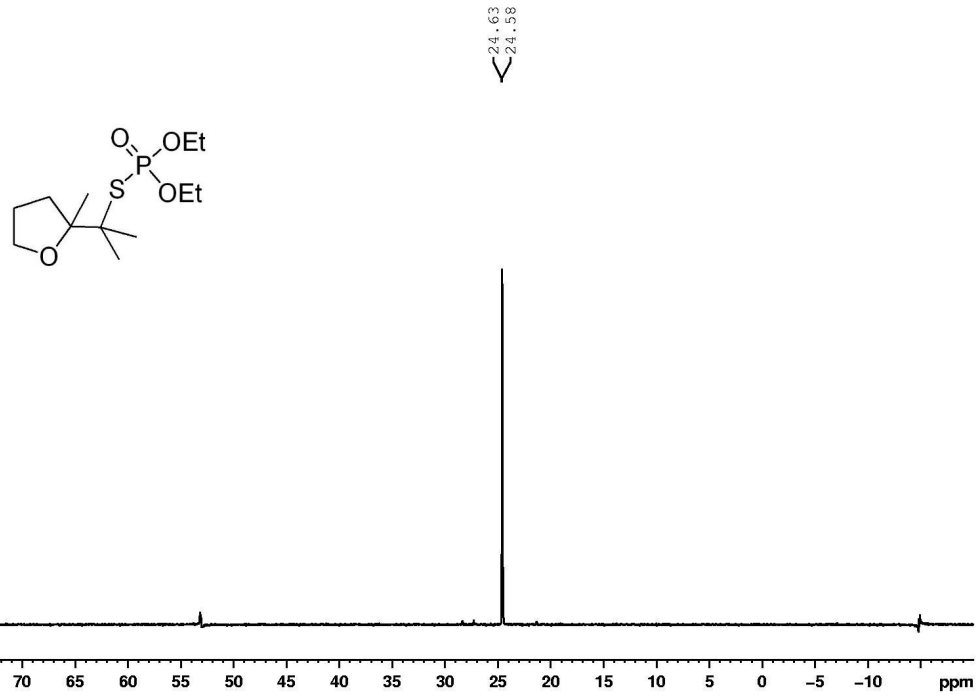
**5c**


10 9 8 7 6 5 4 3 2 1 0 −1 ppm
4.03
1.07
1.04
1.04
1.00
1.06
1.11
2.08
1.09
1.03
6.09
3.00

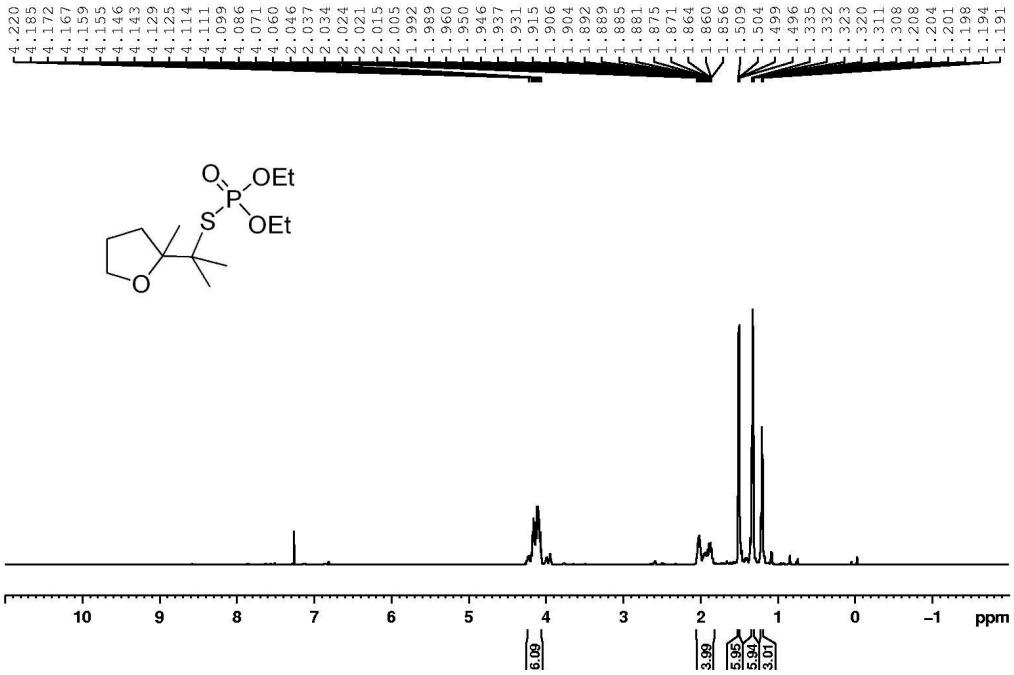
**5c**


EtO
OEt
P
S
O
O
81.05
81.01
77.37
77.26
77.05
68.89
63.65
63.61
54.26
54.24
29.49
27.45
26.28
26.11
16.15
16.13
11.76
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

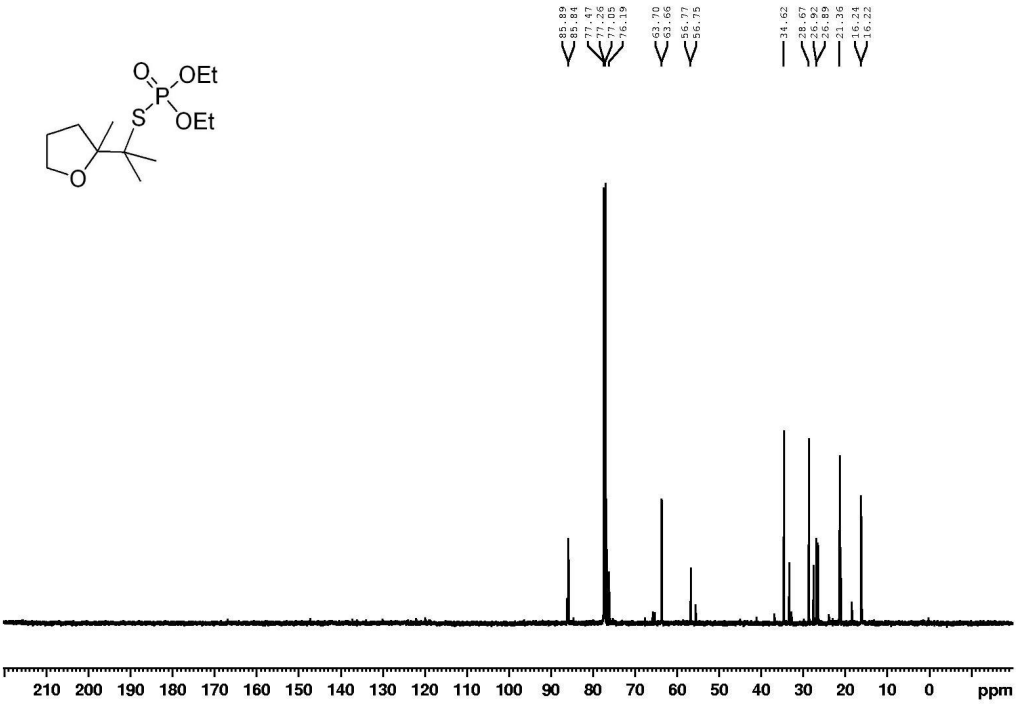
**5d**


24.63
24.58
O
P
OEt
S
OEt
O
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

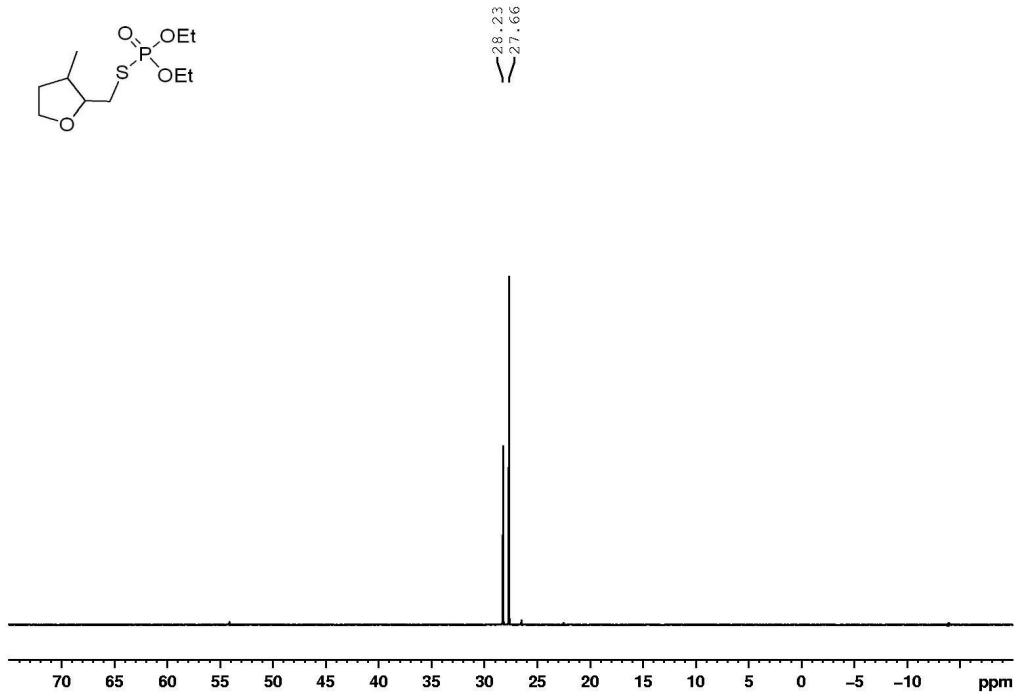
**5d**


4.220
4.185
4.172
4.167
4.159
4.155
4.146
4.143
4.129
4.125
4.114
4.111
4.099
4.086
4.071
4.060
2.046
2.037
2.034
2.024
2.021
2.015
2.005
1.992
1.989
1.960
1.950
1.946
1.937
1.931
1.915
1.906
1.904
1.892
1.889
1.885
1.881
1.875
1.871
1.864
1.860
1.856
1.509
1.504
1.499
1.496
1.335
1.332
1.323
1.320
1.311
1.308
1.208
1.204
1.201
1.198
1.194
1.191
O
OEt
P
S
OEt
O
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
5.09
3.99
5.95
5.94
3.01

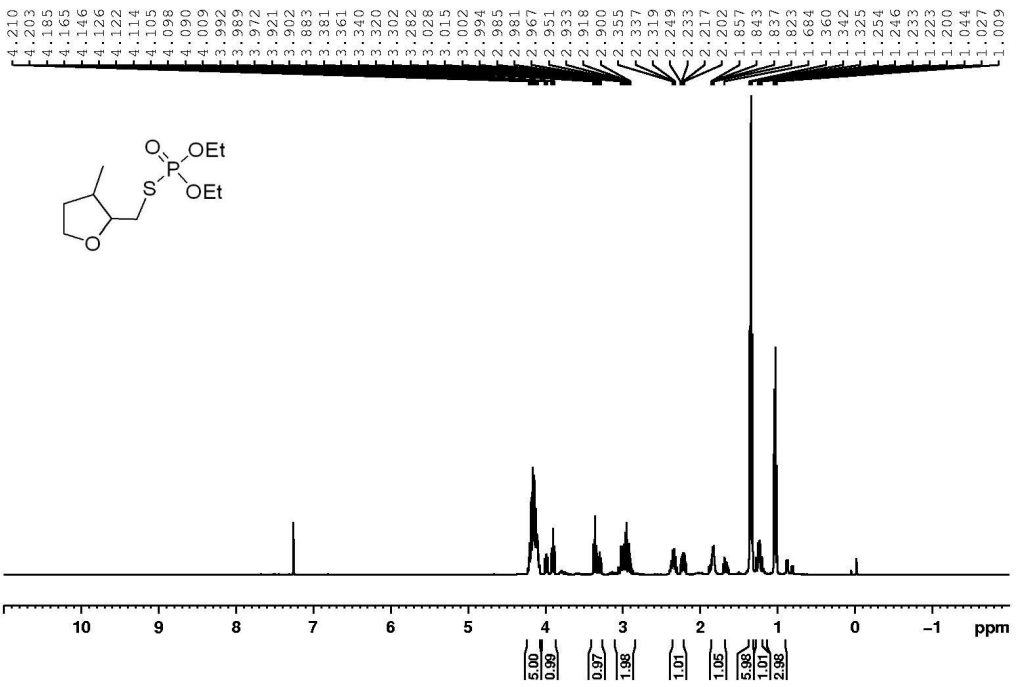
**5d**


85.89
85.84
77.47
77.26
77.05
76.19
63.70
63.66
56.77
56.75
34.62
28.67
26.68
26.89
21.36
16.24
16.22
O
OEt
P
S
OEt
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

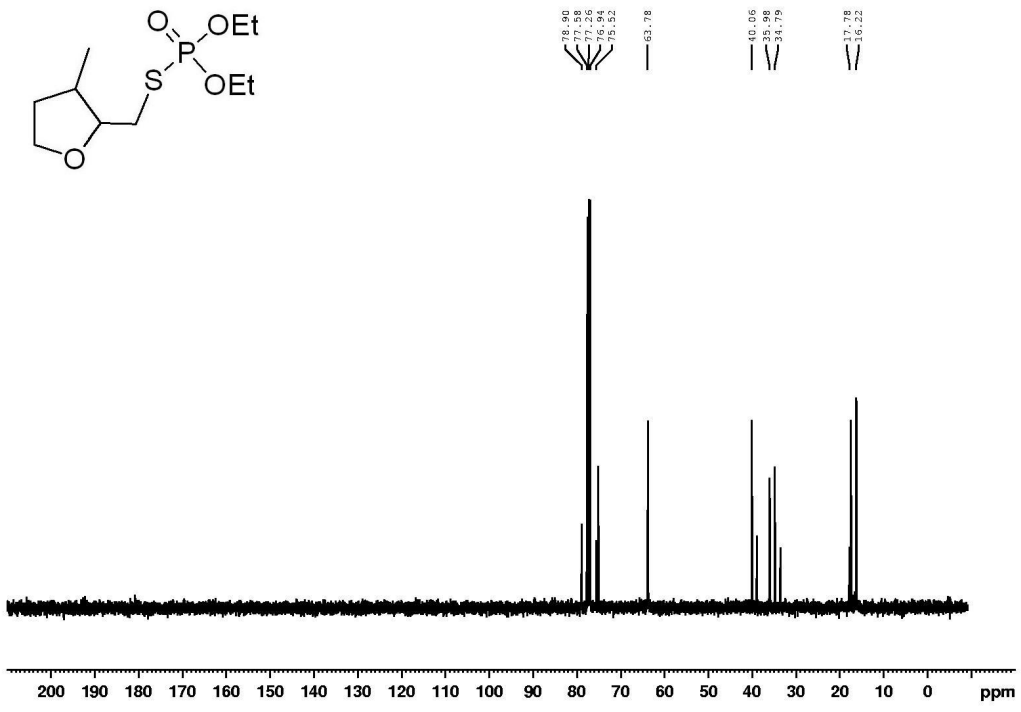
**5e**


28.23
27.66
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

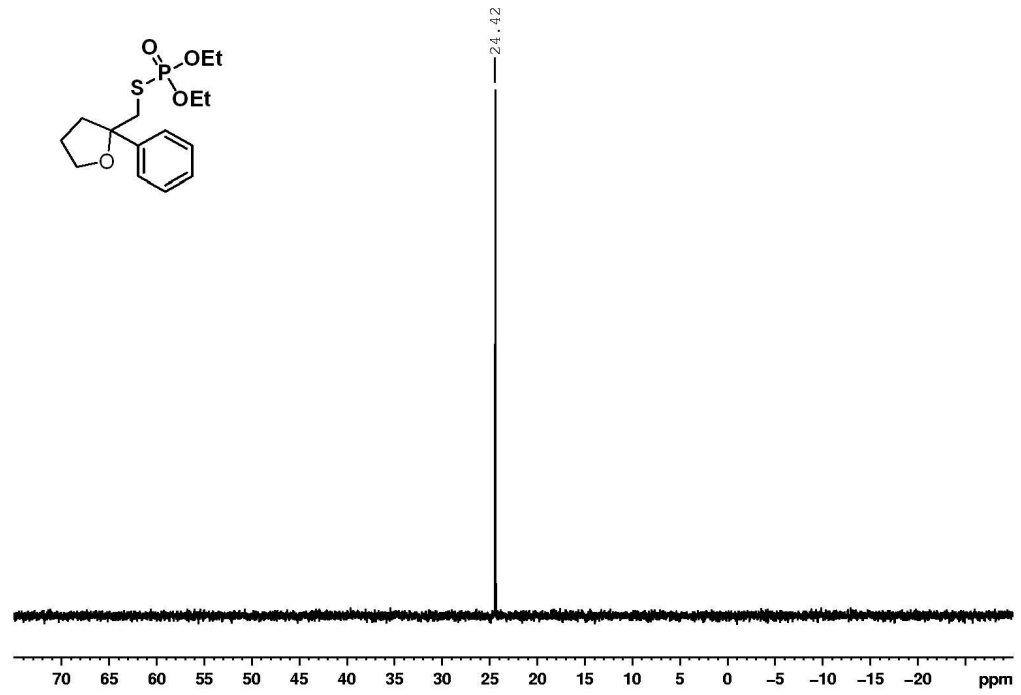
**5e**

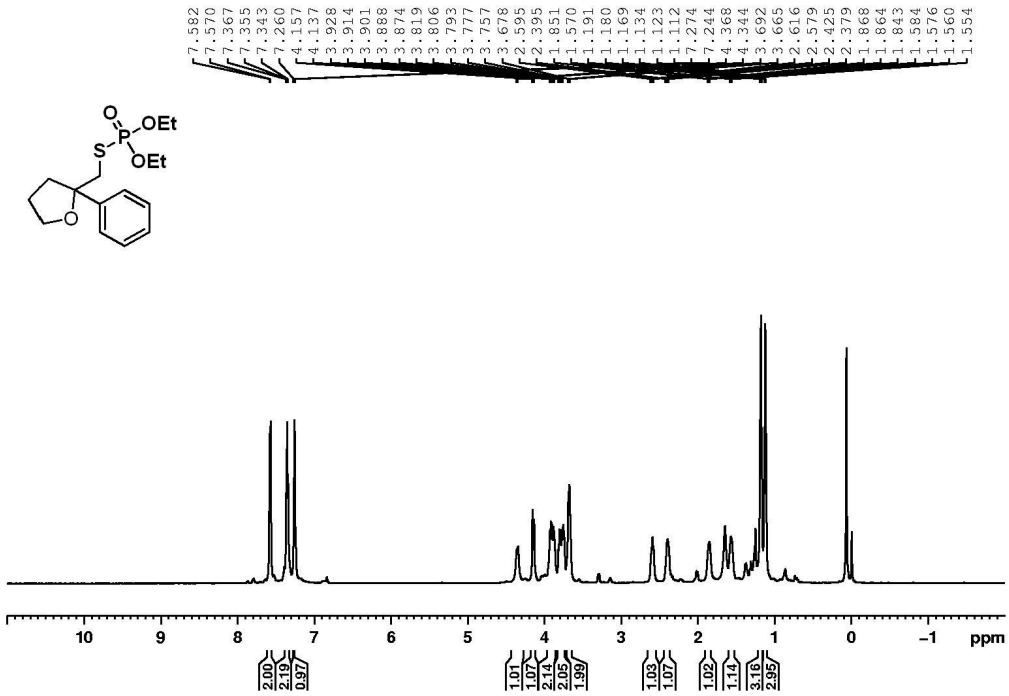

4.210 4.203 4.185 4.165 4.146 4.126 4.122 4.114 4.105 4.098 4.090 4.009 3.992 3.989 3.972 3.921 3.902 3.883 3.381 3.361 3.340 3.320 3.302 3.282 3.028 3.015 3.002 2.994 2.985 2.981 2.967 2.951 2.933 2.918 2.900 2.355 2.337 2.319 2.249 2.233 2.217 2.202 1.857 1.843 1.837 1.823 1.684 1.360 1.342 1.325 1.254 1.246 1.233 1.223 1.200 1.044 1.027 1.009
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
5.00 0.99 0.97 1.38 1.01 1.05 5.98 1.01 2.98

**5e**

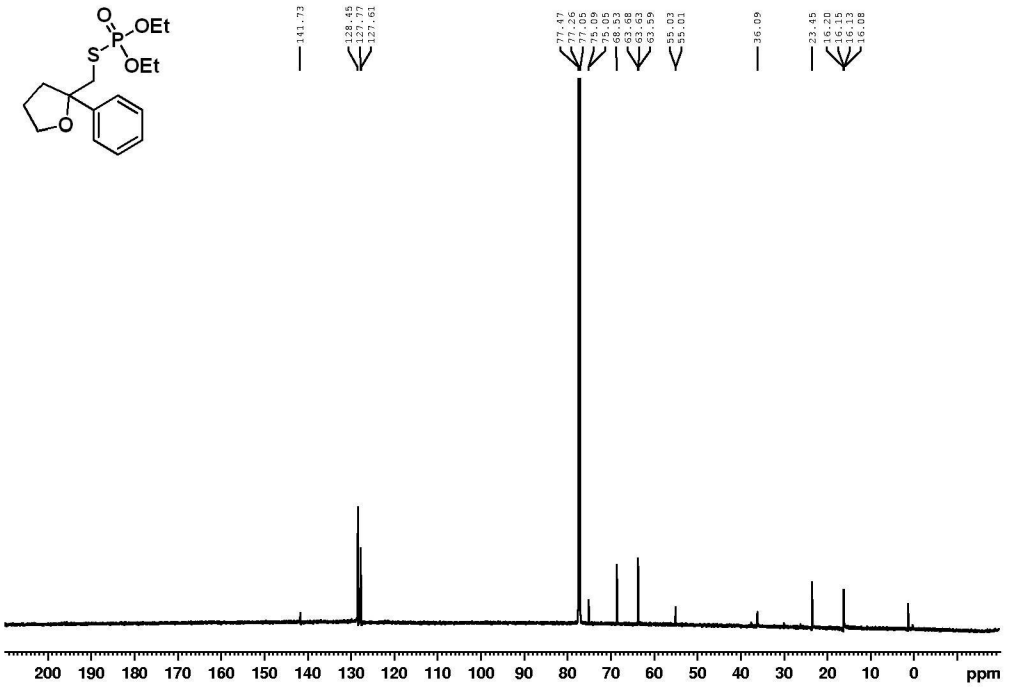

O
OEt
P
S
OEt
O
78.90
77.58
77.26
76.94
75.52
63.78
40.06
35.98
34.79
17.78
16.22
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

**5f**

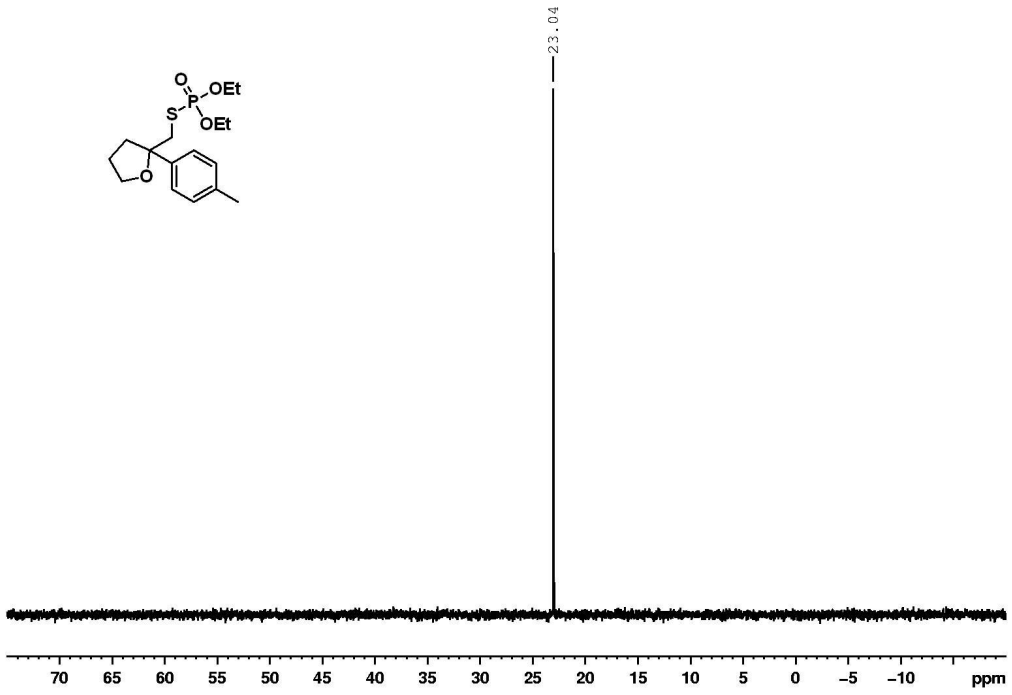

O
OEt
P
S
OEt
O
24.42
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 −15 −20 ppm


7.582
7.570
7.367
7.355
7.343
7.260
4.157
4.137
3.928
3.914
3.901
3.888
3.874
3.819
3.806
3.793
3.777
3.757
3.678
2.595
2.395
1.851
1.570
1.191
1.180
1.169
1.134
1.123
1.112
7.274
7.244
4.368
4.344
3.692
3.665
2.616
2.579
2.425
2.379
1.868
1.864
1.843
1.584
1.576
1.560
1.554
OEt
OEt
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
2.00 2.19 0.97 1.01 1.07 2.14 2.05 1.99 1.03 1.07 1.02 1.14 3.16 2.95

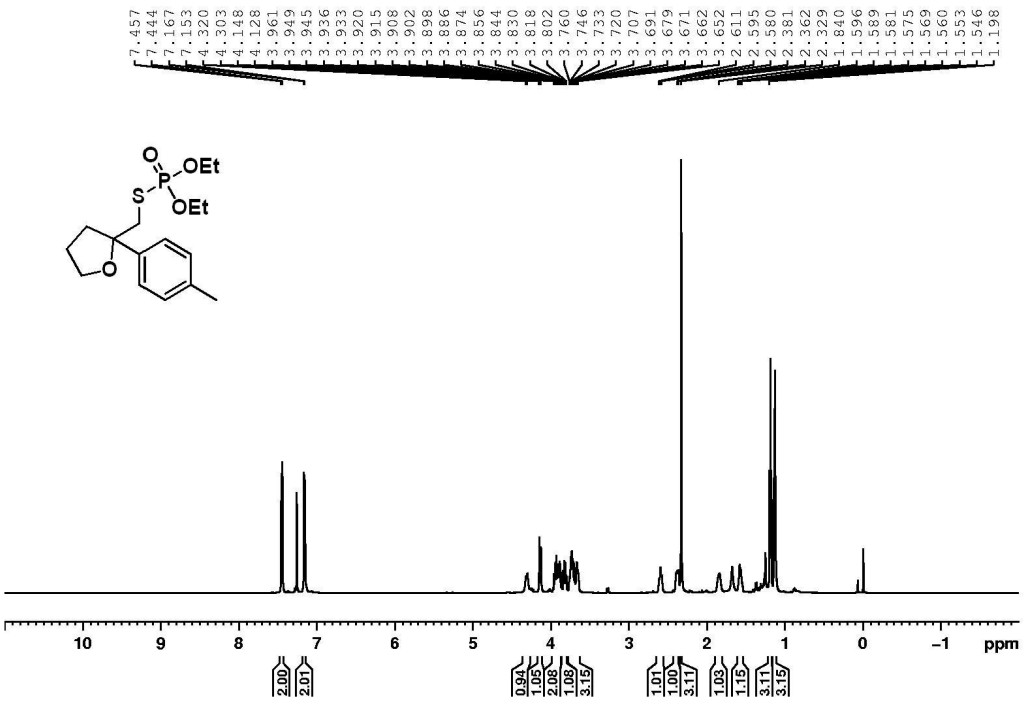
**5f**


OEt
OEt
141.73
128.45
127.77
127.61
77.47
77.26
77.05
75.09
75.05
68.53
63.68
63.63
63.59
55.03
55.01
36.09
23.45
16.20
16.15
16.13
16.08
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

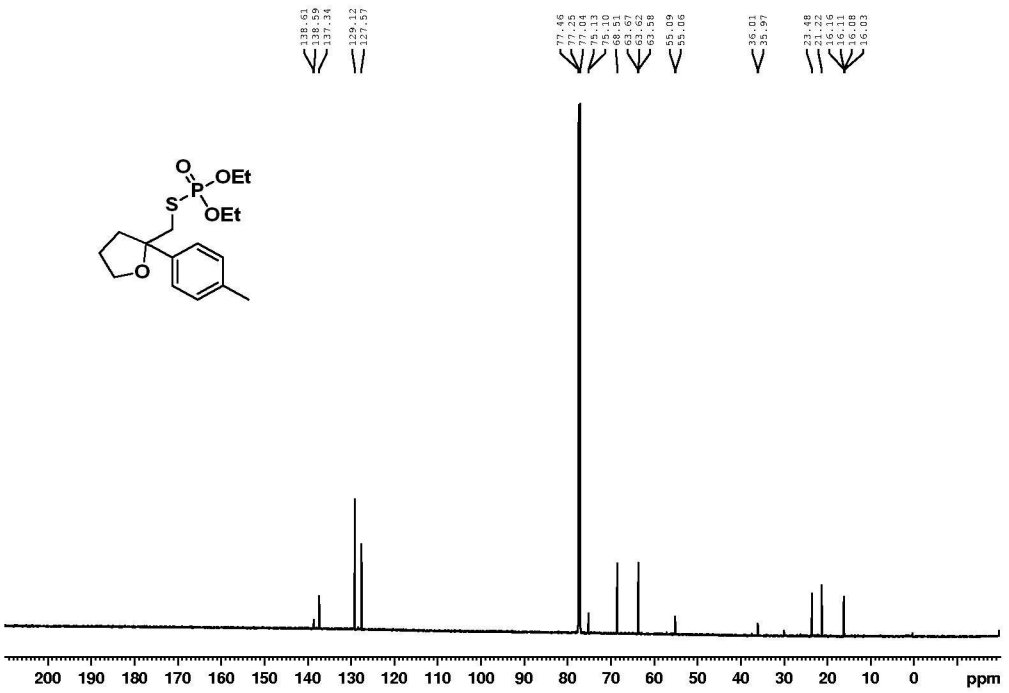
**5g**


23.04
OEt
OEt
S
P
O
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

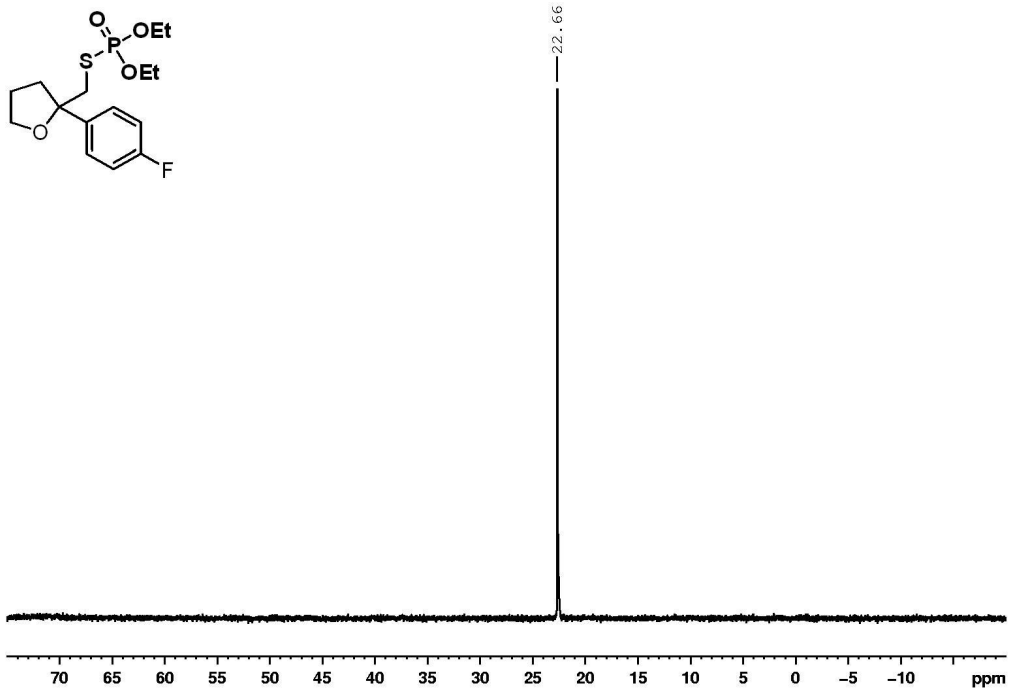
**5g**


7.457 7.444 7.167 7.153 4.320 4.303 4.148 4.128 3.961 3.949 3.945 3.936 3.933 3.920 3.915 3.908 3.902 3.898 3.886 3.874 3.856 3.844 3.830 3.818 3.802 3.760 3.746 3.733 3.720 3.707 3.691 3.679 3.671 3.662 3.652 2.611 2.595 2.580 2.381 2.362 2.329 1.840 1.596 1.589 1.581 1.575 1.569 1.560 1.553 1.546 1.198
OEt
OEt
S
P
O
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
2.00 2.01 0.94 1.05 2.08 1.08 3.15 1.01 1.00 3.11 1.03 1.15 3.11 3.15

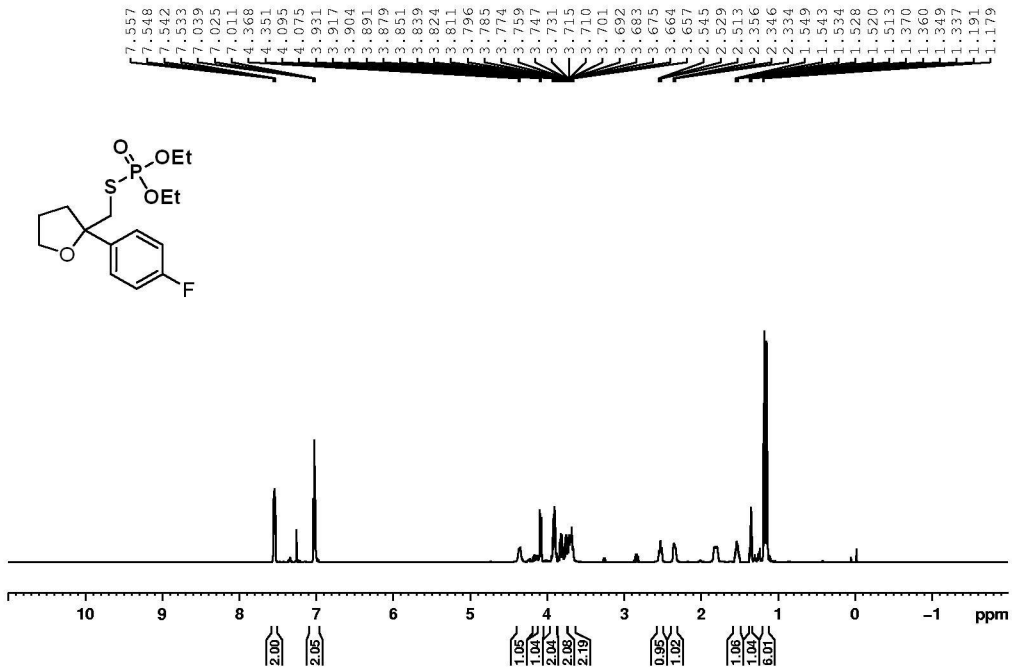
**5g**


138.61
138.59
137.34
129.12
127.57
77.46
77.25
77.04
75.13
75.10
65.51
63.67
63.62
63.58
55.09
55.06
36.01
35.97
23.48
21.22
16.16
16.11
16.08
16.03
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

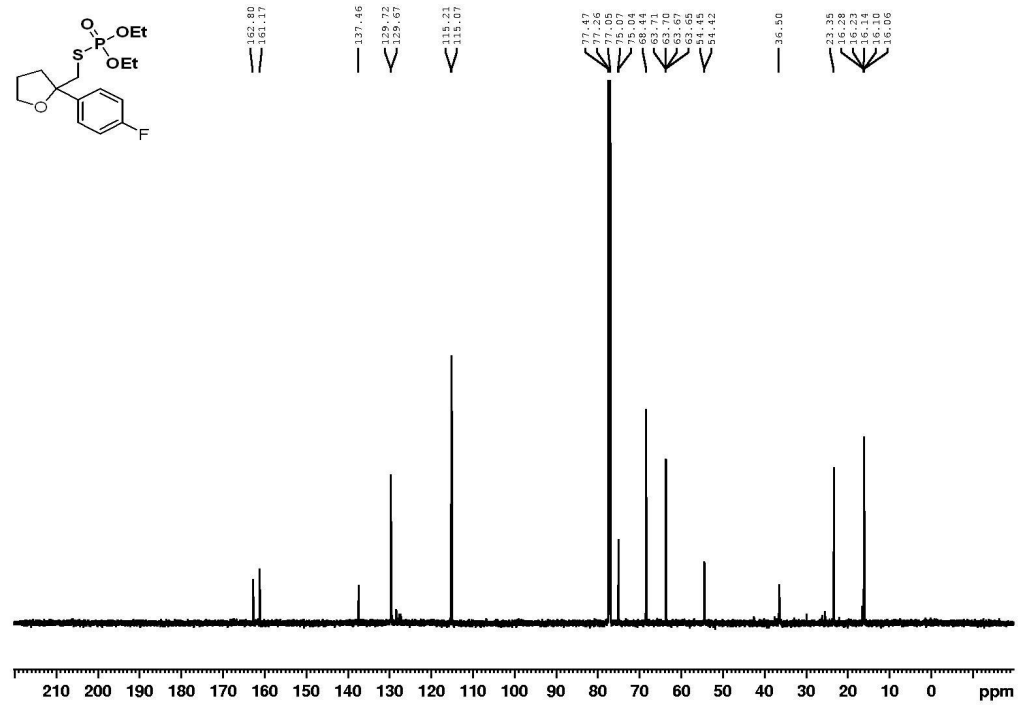
**5h**


22.66
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 ppm

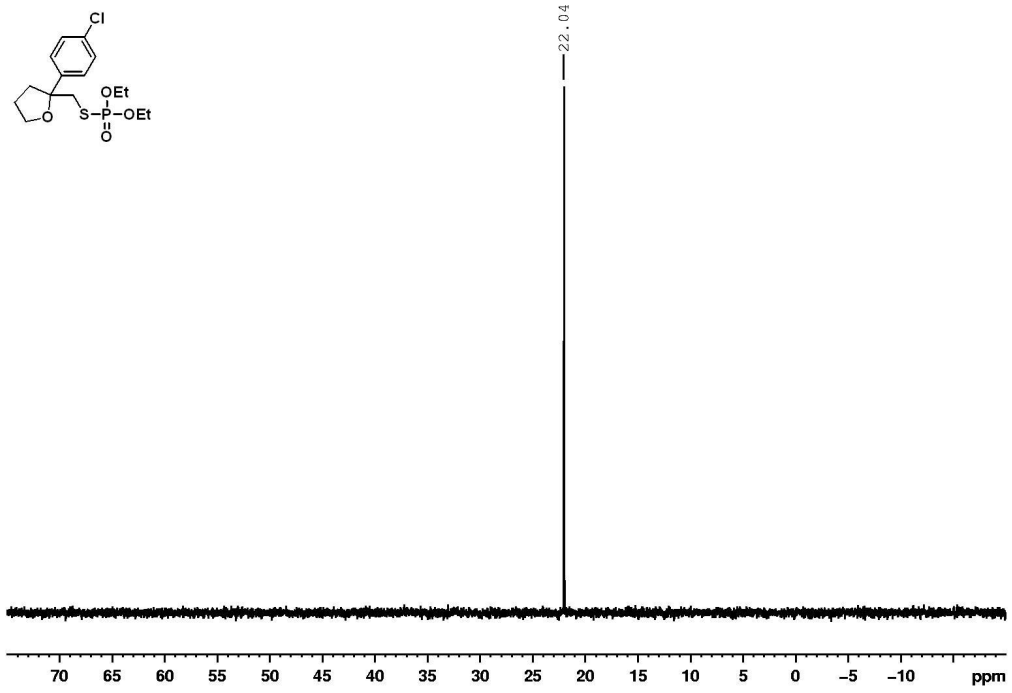
**5h**


7.557
7.548
7.542
7.533
7.039
7.025
7.011
4.368
4.351
4.095
4.075
3.931
3.917
3.904
3.891
3.879
3.851
3.839
3.824
3.811
3.796
3.785
3.774
3.759
3.747
3.731
3.715
3.710
3.701
3.692
3.683
3.675
3.664
3.657
2.545
2.529
2.513
2.356
2.346
2.334
1.549
1.543
1.534
1.528
1.520
1.513
1.370
1.360
1.349
1.337
1.191
1.179
OEt
OEt
S
O
P
O
F
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
2.00
2.05
1.05
1.04
2.04
2.08
2.19
0.95
1.02
1.06
1.04
6.01

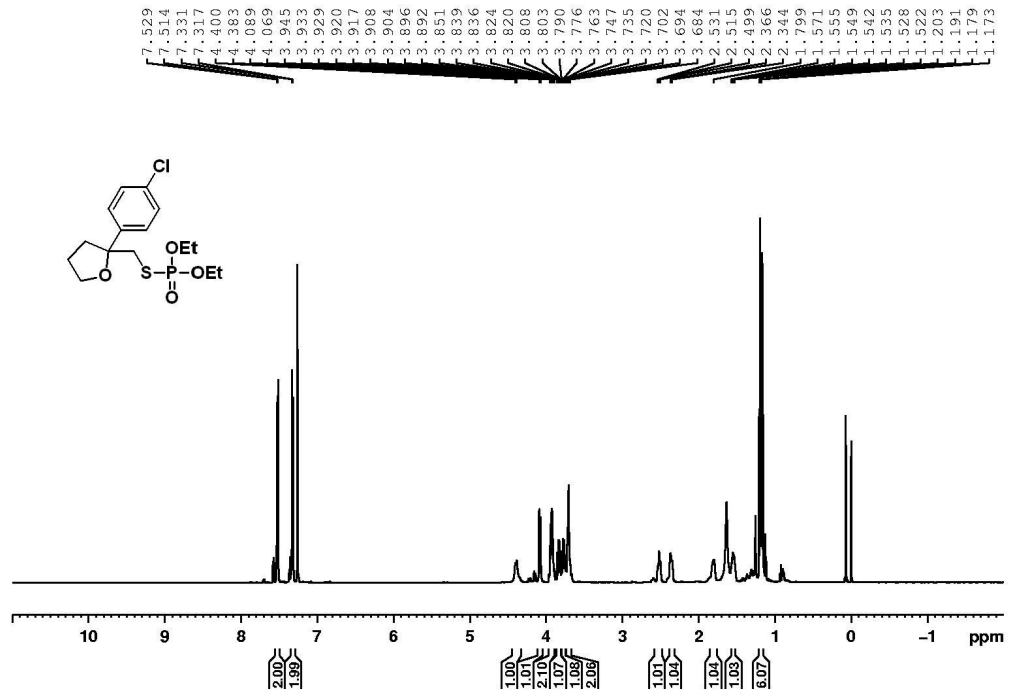
**5h**


162.80
161.17
137.46
129.72
129.67
115.21
115.07
77.47
77.26
77.05
75.07
75.04
68.44
63.71
63.70
63.67
63.65
54.45
54.42
36.50
23.35
16.28
16.23
16.14
16.10
16.06
OEt
OEt
S
O
P
O
F
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

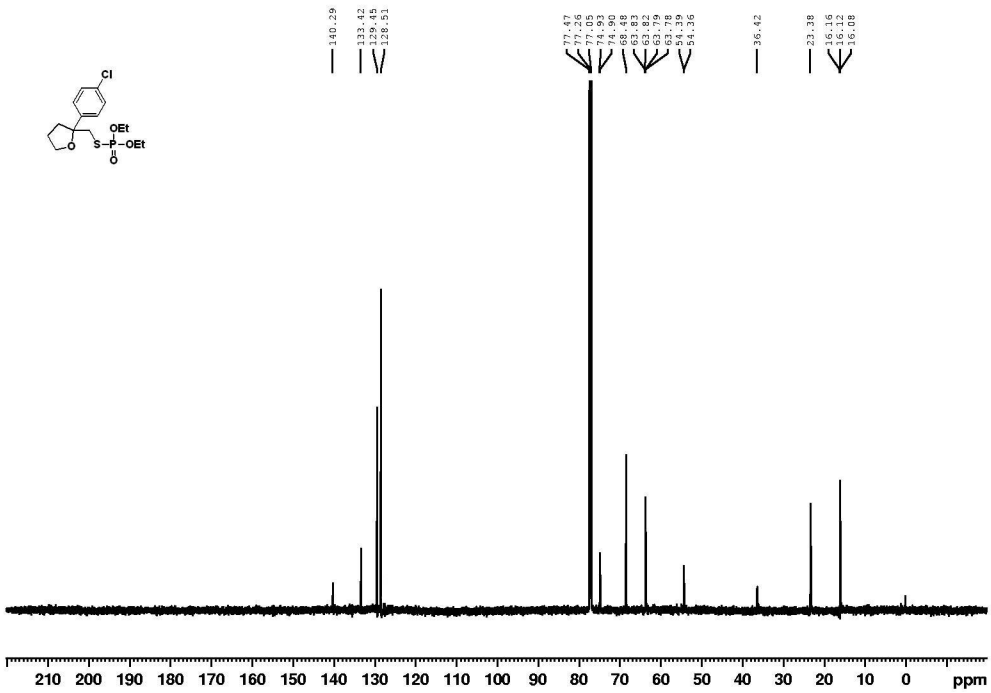
**5i**


Cl
OEt
S–P–OEt
O
22.04
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 –5 –10 ppm

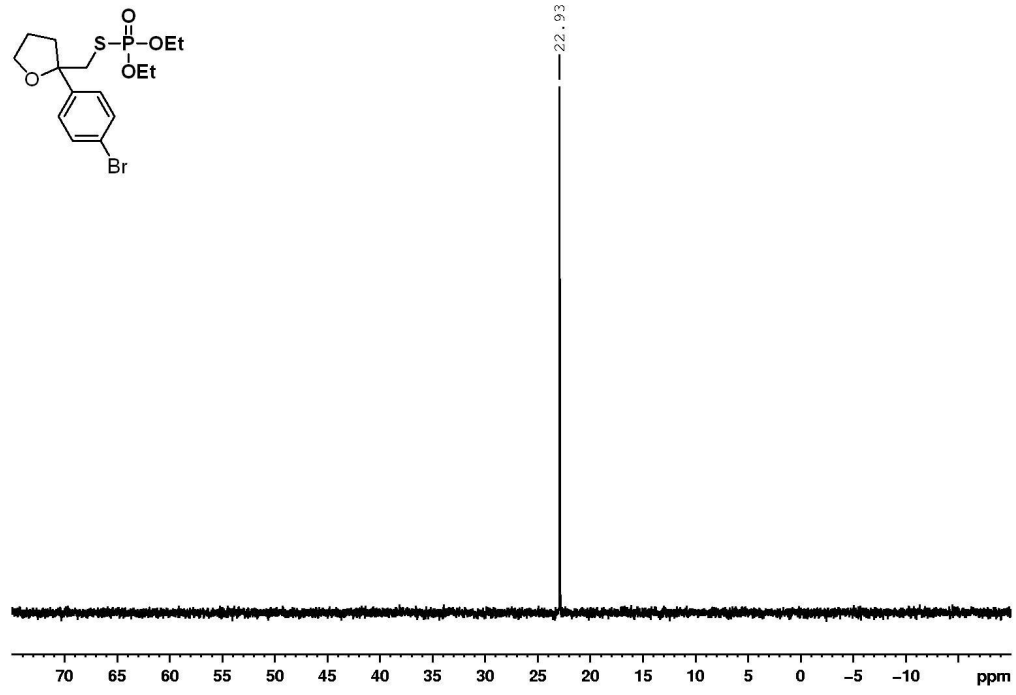
**5i**


7.529
7.514
7.331
7.317
4.400
4.383
4.089
4.069
3.945
3.933
3.929
3.920
3.917
3.908
3.904
3.896
3.892
3.851
3.839
3.836
3.824
3.820
3.808
3.803
3.790
3.776
3.763
3.747
3.735
3.720
3.702
3.694
3.684
2.531
2.515
2.499
2.366
2.344
1.799
1.571
1.555
1.549
1.542
1.535
1.528
1.522
1.203
1.191
1.179
1.173
Cl
OEt
S–P–OEt
O
10 9 8 7 6 5 4 3 2 1 0 –1 ppm
2.00
1.99
1.00
1.01
2.10
1.07
1.08
2.09
1.01
1.04
1.04
1.03
6.07

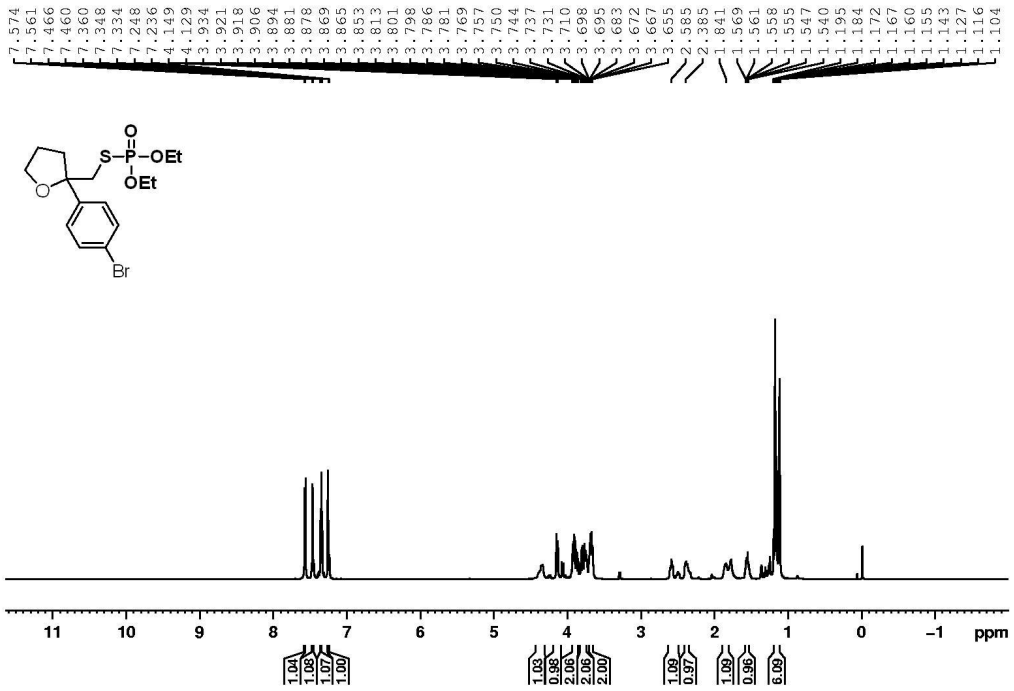
**5i**


140.29
133.42
129.45
128.51
77.47
77.26
77.05
74.93
74.90
68.48
63.83
63.82
63.79
63.78
54.39
54.36
36.42
23.38
16.16
16.12
16.08
Cl
OEt
S–P–OEt
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

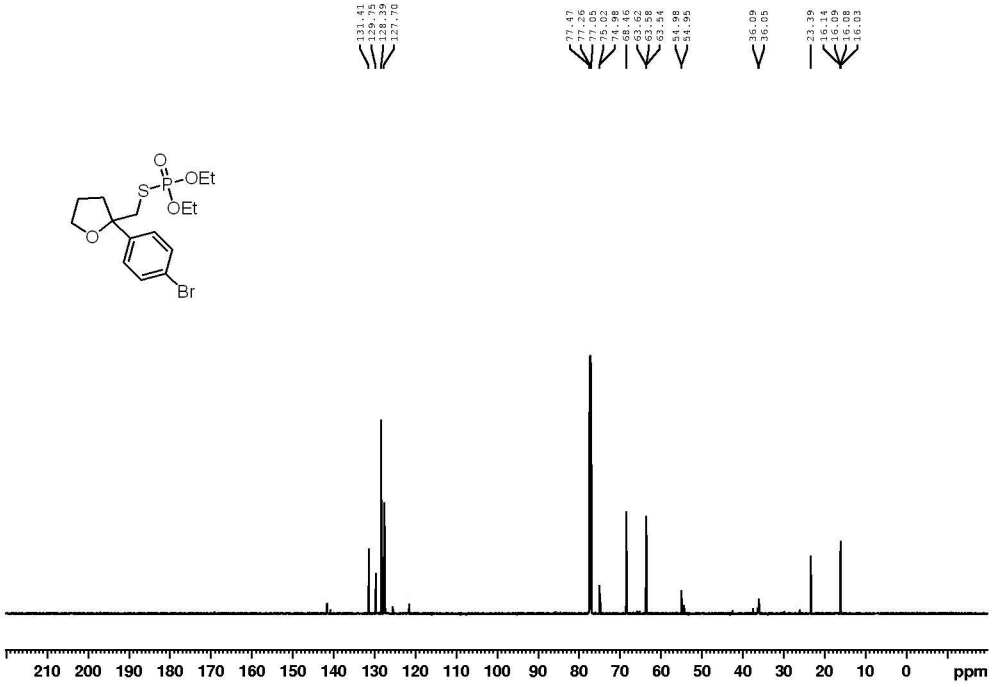
**5j**


O
S–P–OEt
OEt
O
Br
22.93
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 –5 –10 ppm

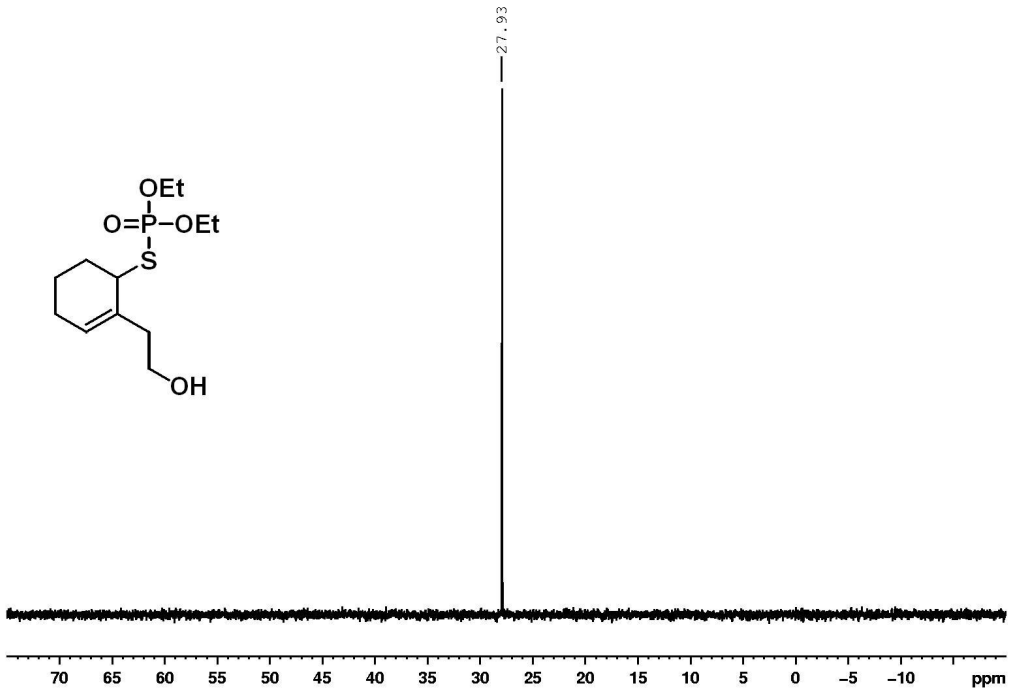
**5j**


7.574
7.561
7.466
7.460
7.360
7.348
7.334
7.248
7.236
4.149
4.129
3.934
3.921
3.918
3.906
3.894
3.881
3.878
3.869
3.865
3.853
3.813
3.801
3.798
3.786
3.781
3.769
3.757
3.750
3.744
3.737
3.731
3.710
3.698
3.695
3.683
3.672
3.667
3.655
2.585
2.385
1.841
1.569
1.561
1.558
1.555
1.547
1.540
1.195
1.184
1.172
1.167
1.160
1.155
1.143
1.127
1.116
1.104
O
S
P
OEt
OEt
O
Br
11
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
1.04
1.08
1.07
1.00
1.03
0.98
2.06
2.06
2.00
1.09
0.97
1.09
0.96
6.09

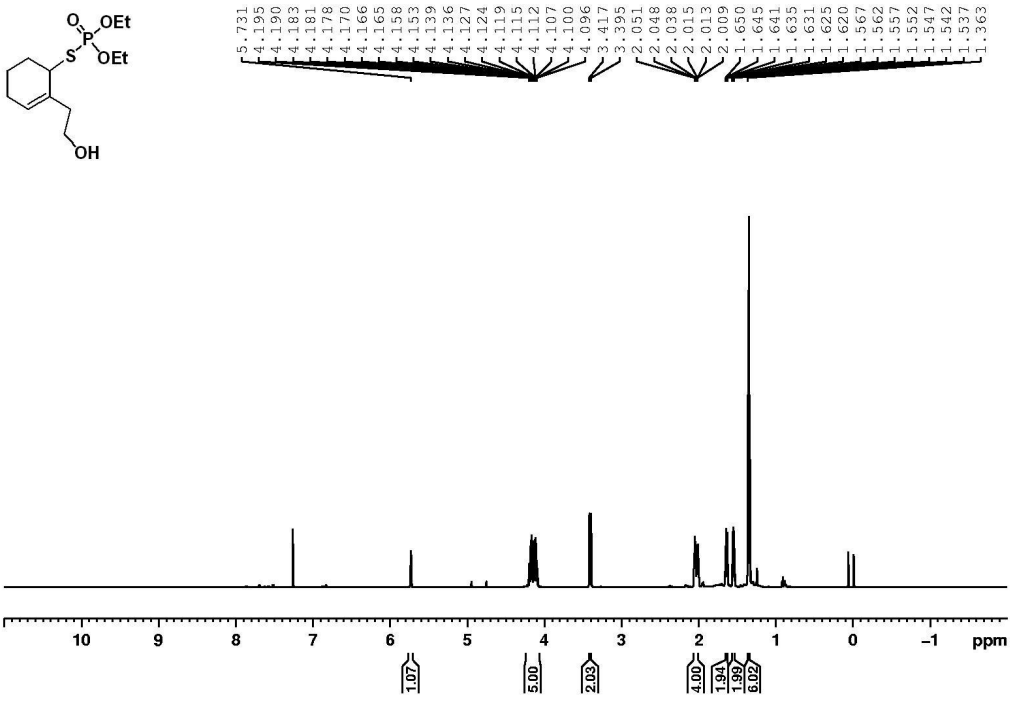
**5j**


131.41
129.35
128.39
127.70
77.47
77.26
77.05
75.02
74.98
68.46
63.62
63.58
63.54
54.98
54.95
36.09
36.05
23.39
16.14
16.09
16.08
16.03
O
S
P
OEt
OEt
O
Br
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

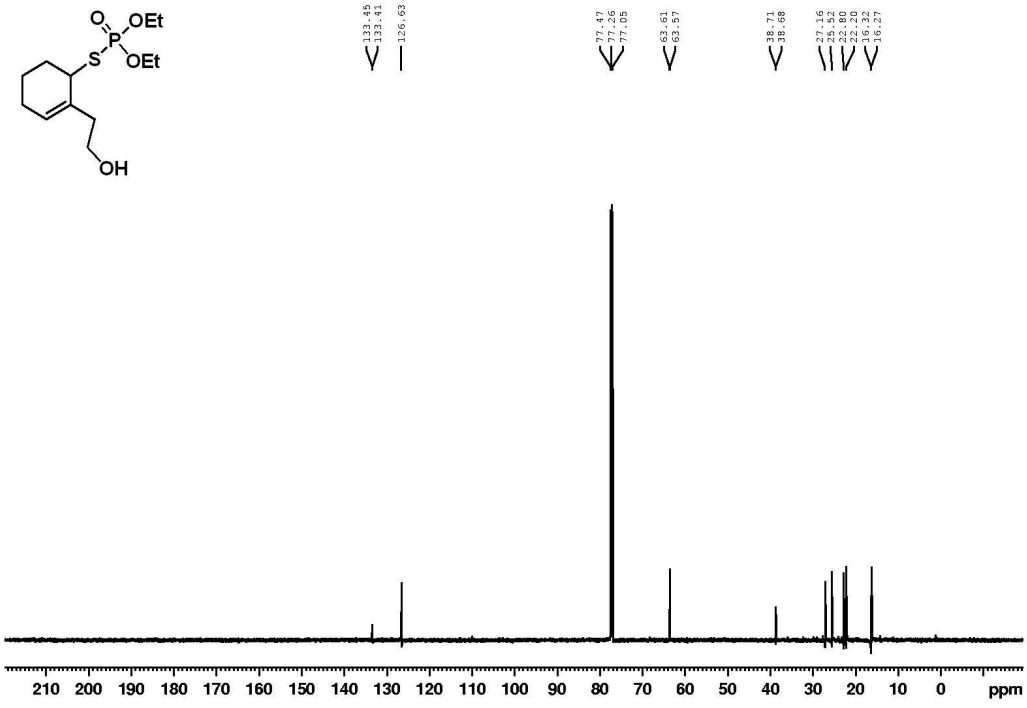
**5k**


27.93
OEt
O=P–OEt
S
OH
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 –5 –10 ppm

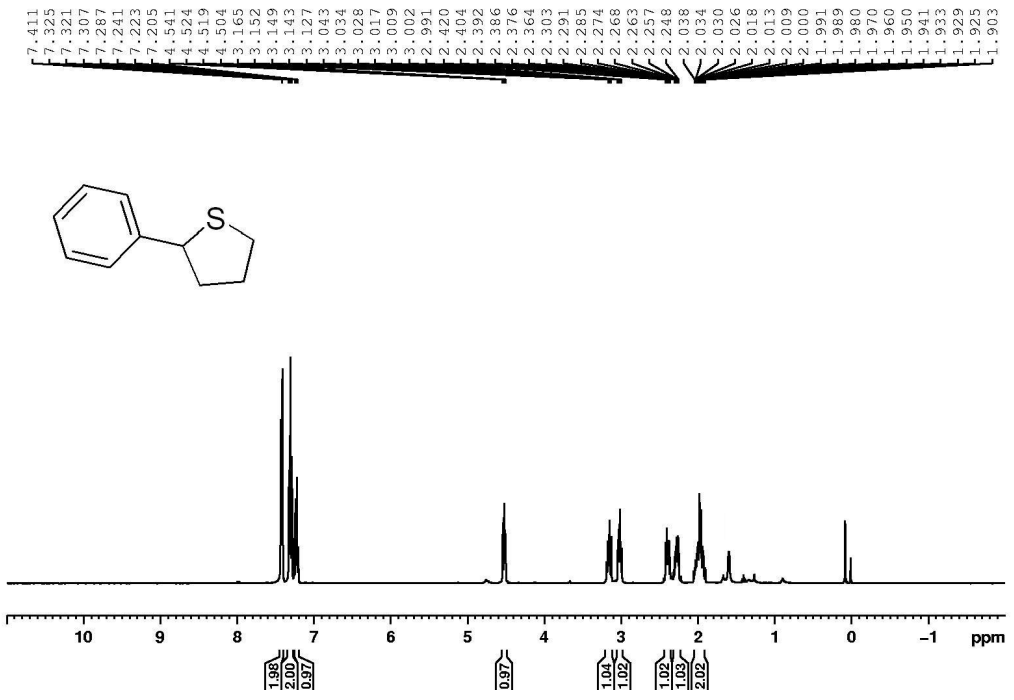
**5k**


OEt
OEt
S
OH
5.731 4.195 4.190 4.183 4.181 4.178 4.170 4.166 4.165 4.158 4.153 4.139 4.136 4.127 4.124 4.119 4.115 4.112 4.107 4.100 4.096 3.417 3.395 2.051 2.048 2.038 2.015 2.013 2.009 1.650 1.645 1.641 1.635 1.631 1.625 1.620 1.567 1.562 1.557 1.552 1.547 1.542 1.537 1.363
10 9 8 7 6 5 4 3 2 1 0 –1 ppm
1.07 5.00 2.03 4.00 1.94 1.99 6.02

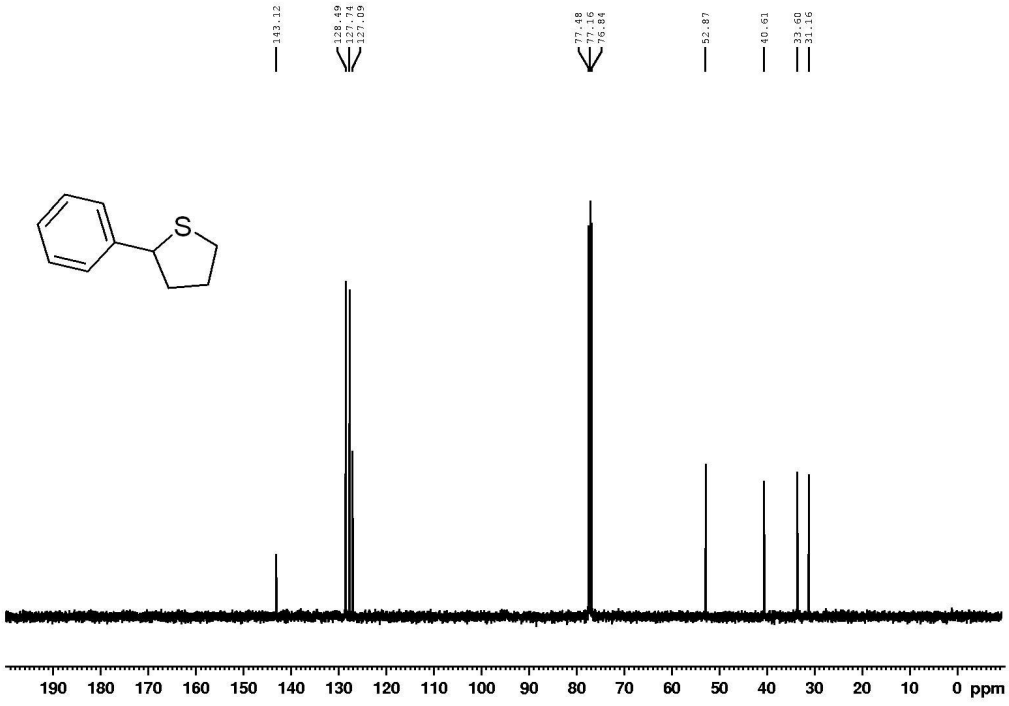
**5k**


OEt
OEt
S
O
P
OH
133.45
133.41
126.63
77.47
77.26
77.05
63.61
63.57
38.71
38.68
27.16
25.52
22.80
22.20
16.32
16.27
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

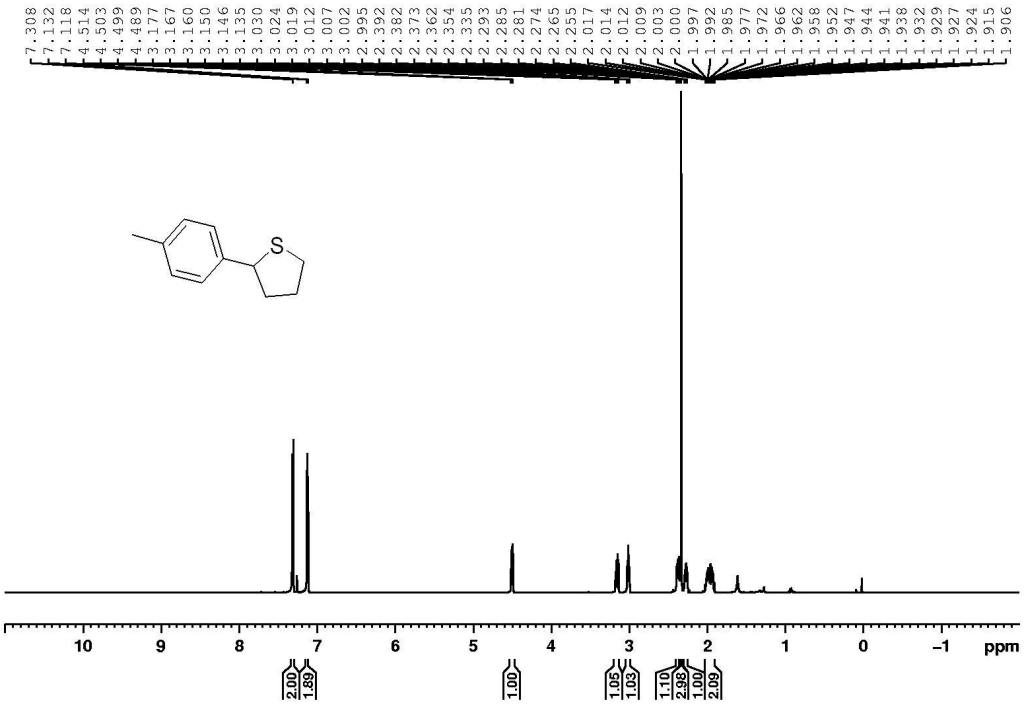
**6a**


7.411
7.325
7.321
7.307
7.287
7.241
7.223
7.205
4.541
4.524
4.519
4.504
3.165
3.152
3.149
3.143
3.127
3.043
3.034
3.028
3.017
3.009
3.002
2.991
2.420
2.404
2.392
2.386
2.376
2.364
2.303
2.291
2.285
2.274
2.268
2.263
2.257
2.248
2.038
2.034
2.030
2.026
2.018
2.013
2.009
2.000
1.991
1.989
1.980
1.970
1.960
1.950
1.941
1.933
1.929
1.925
1.903
S
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
1.98
2.00
0.97
0.97
1.04
1.02
1.02
1.03
2.02

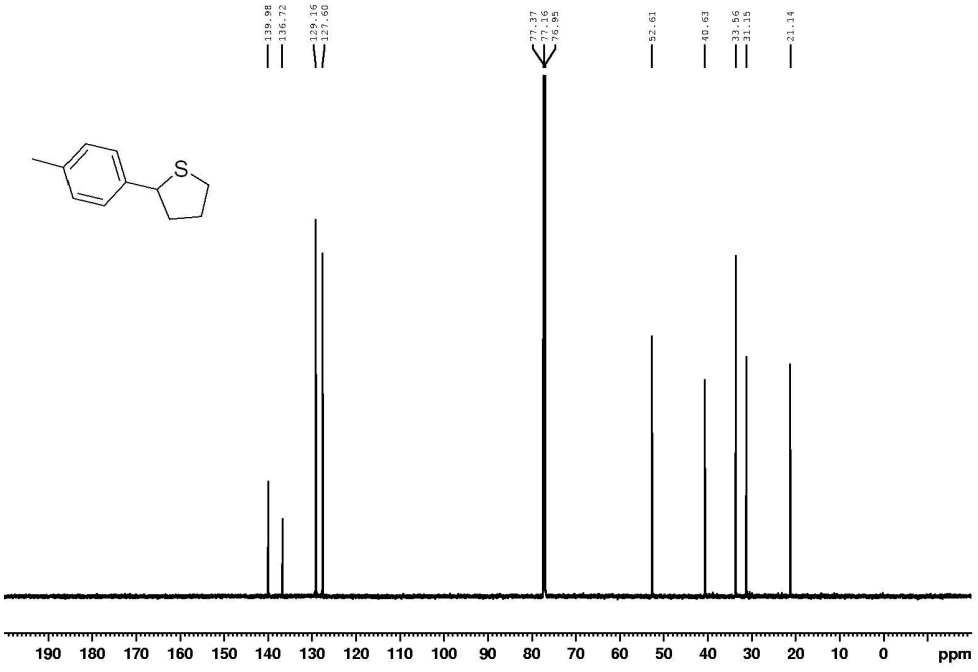
**6a**


143.12
128.49
127.74
127.09
77.48
77.16
76.84
52.87
40.61
33.60
31.16
S
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

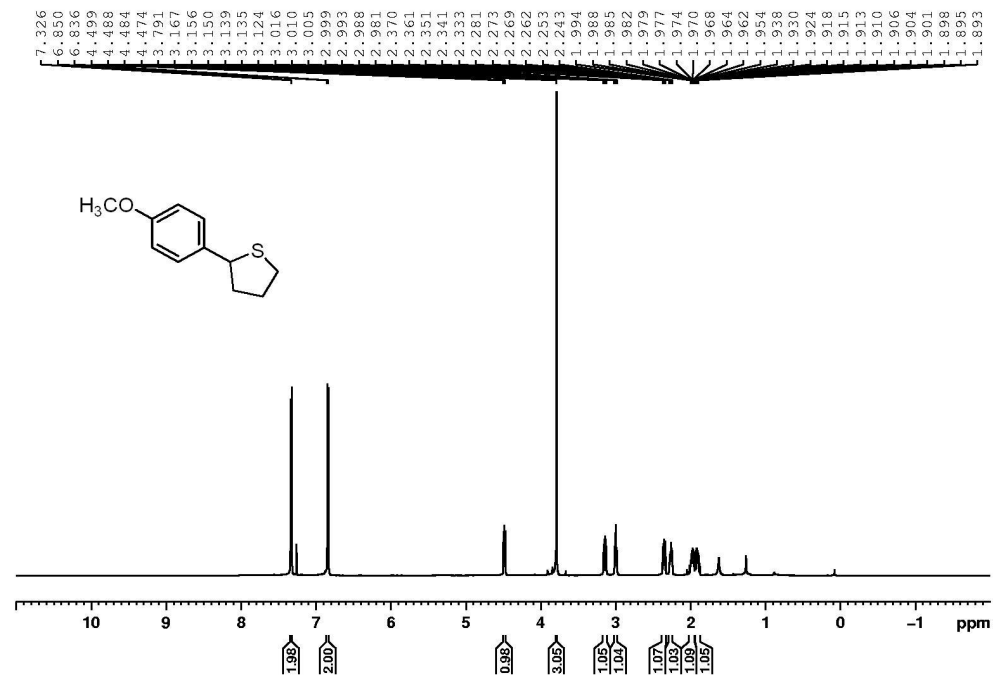
**6b**


7.308
7.132
7.118
4.514
4.503
4.499
4.489
3.177
3.167
3.160
3.150
3.146
3.135
3.030
3.024
3.019
3.012
3.007
3.002
2.995
2.392
2.382
2.373
2.362
2.354
2.335
2.293
2.285
2.281
2.274
2.265
2.255
2.017
2.014
2.012
2.009
2.003
2.000
1.997
1.992
1.985
1.977
1.972
1.966
1.962
1.958
1.952
1.947
1.944
1.941
1.938
1.932
1.929
1.927
1.924
1.915
1.906
S
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
2.00
1.89
1.00
1.05
1.03
1.10
2.98
1.00
2.09

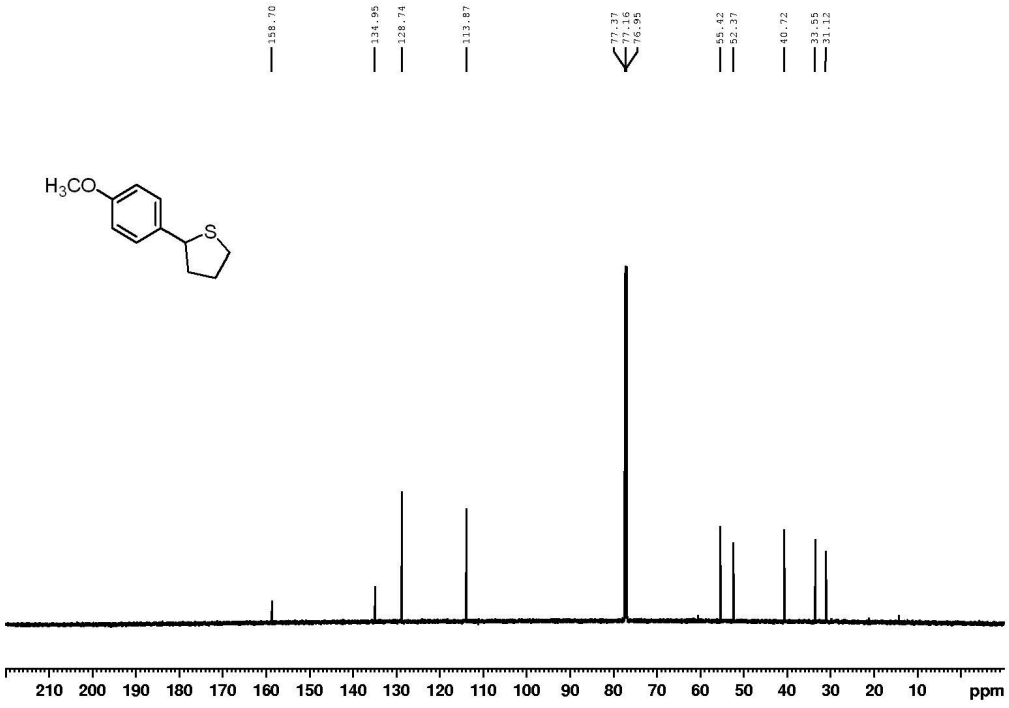
**6b**


139.98
136.72
129.16
127.60
77.37
77.16
76.95
52.61
40.63
33.56
31.15
21.14
S
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

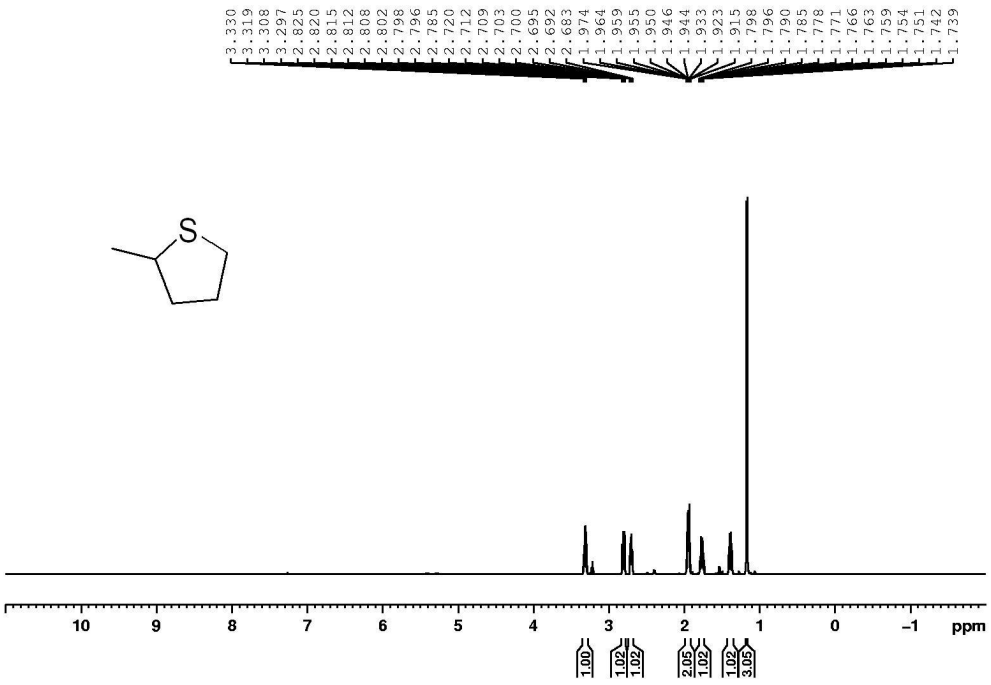
**6c**


7.326
6.850
6.836
4.499
4.488
4.484
4.474
3.791
3.167
3.156
3.150
3.139
3.135
3.124
3.016
3.010
3.005
2.999
2.993
2.988
2.981
2.370
2.361
2.351
2.341
2.333
2.281
2.273
2.269
2.262
2.253
2.243
1.994
1.988
1.985
1.982
1.979
1.977
1.974
1.970
1.968
1.964
1.962
1.954
1.938
1.930
1.924
1.918
1.915
1.913
1.910
1.906
1.904
1.901
1.898
1.895
1.893
H3CO
S
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
1.98
2.00
0.98
3.05
1.05
1.04
1.07
1.03
1.09
1.05

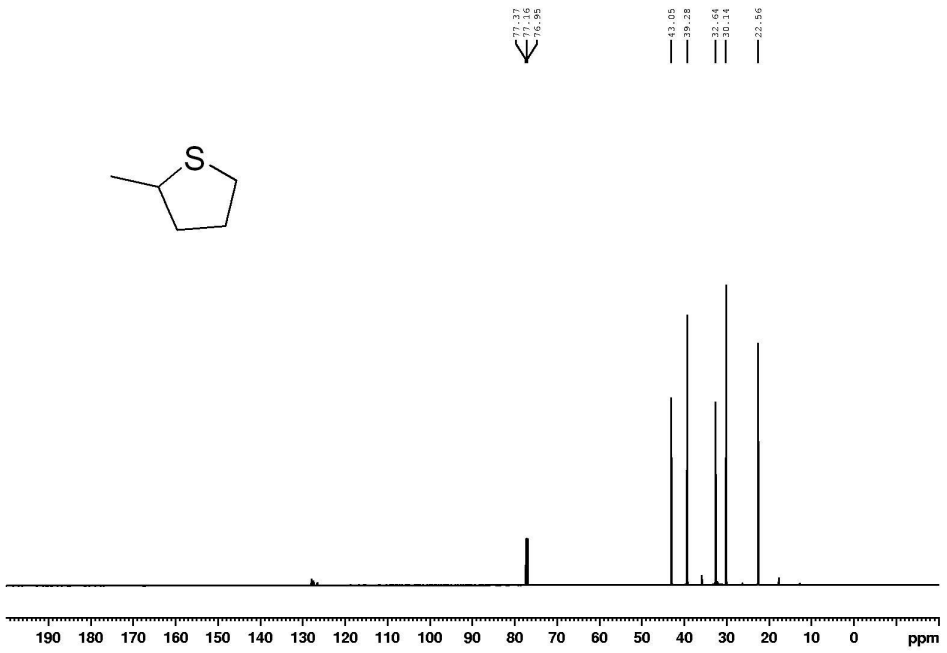
**6c**


158.70
134.95
128.74
113.87
77.37
77.16
76.95
55.42
52.37
40.72
33.55
31.12
H3CO
S
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

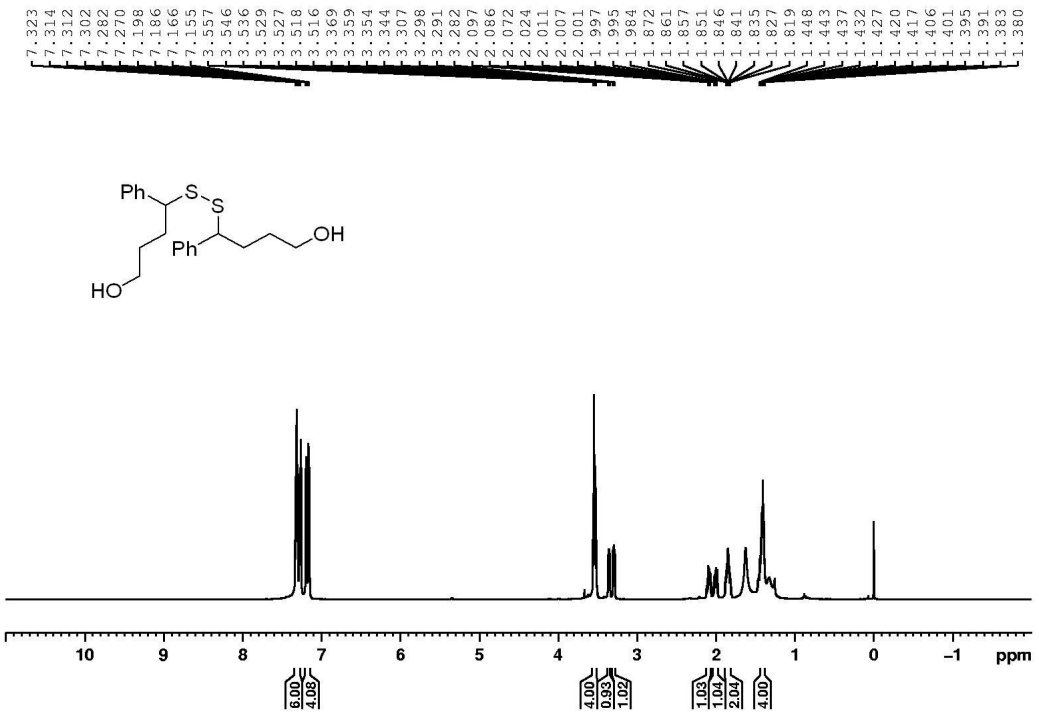
**6d**


3.330
3.319
3.308
3.297
2.825
2.820
2.815
2.812
2.808
2.802
2.798
2.796
2.785
2.720
2.712
2.709
2.703
2.700
2.695
2.692
2.683
1.974
1.964
1.959
1.955
1.950
1.946
1.944
1.933
1.923
1.915
1.798
1.796
1.790
1.785
1.778
1.771
1.766
1.763
1.759
1.754
1.751
1.742
1.739
S
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
1.00
1.02
1.02
2.05
1.02
1.02
3.05

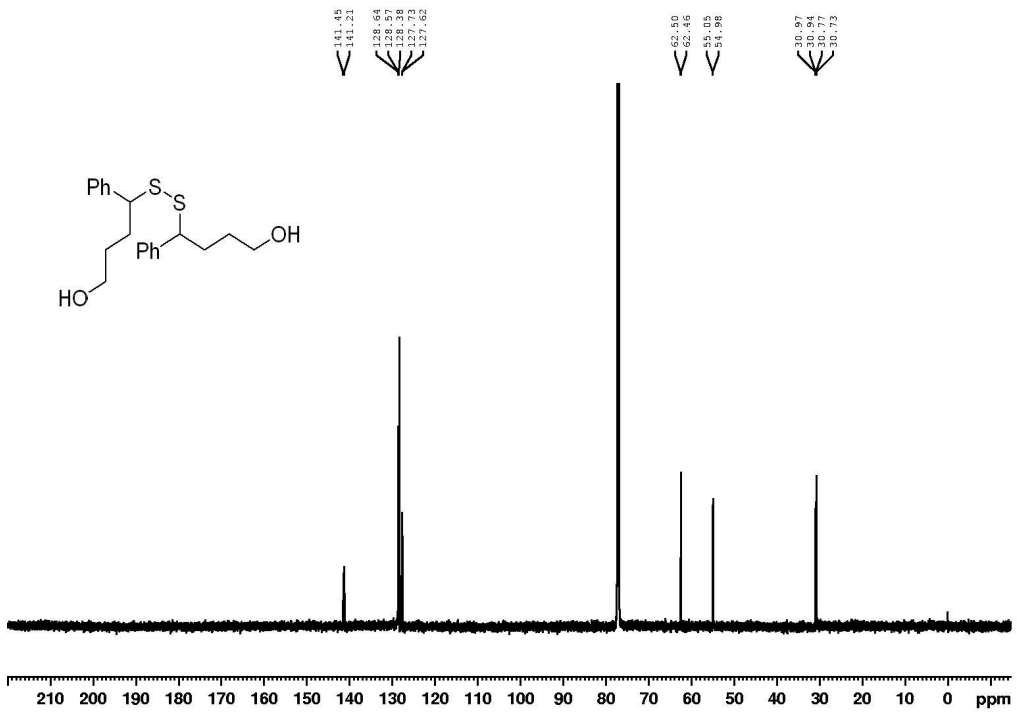
**6d**


77.37
77.16
76.95
43.05
39.28
35.64
30.14
22.56
S
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

**7a**


7.323
7.314
7.312
7.302
7.282
7.270
7.198
7.186
7.166
7.155
3.557
3.546
3.536
3.529
3.527
3.518
3.516
3.369
3.359
3.354
3.344
3.307
3.298
3.291
3.282
2.097
2.086
2.072
2.024
2.011
2.007
2.001
1.997
1.995
1.984
1.872
1.861
1.857
1.851
1.846
1.841
1.835
1.827
1.819
1.448
1.443
1.437
1.432
1.427
1.420
1.417
1.406
1.401
1.395
1.391
1.383
1.380
Ph
S
S
Ph
OH
HO
10 9 8 7 6 5 4 3 2 1 0 –1 ppm
6.00
4.08
4.00
0.93
1.02
1.03
1.04
2.04
4.00

**7a**


141.45
141.21
128.64
128.57
128.38
127.73
127.62
62.50
62.46
55.05
54.98
30.97
30.94
30.77
30.73
Ph
S
S
OH
Ph
HO
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm