Supporting Information

for

Highly regio- and stereoselective phosphinylphosphination of terminal alkynes with tetraphenyldiphosphine monoxide under radical conditions

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1. Characterization Data

\((E)\text{-}1\text{-}(\text{diphenylphosphinyl})\text{-}2\text{-}(\text{diphenylthiophosphinyl})\text{-}\text{oct-1-ene}\ (3a)\)

\[
\begin{array}{c}
\text{Ph}_2\text{P} \\
\text{O} \\
\text{S} \\
\text{PPh}_2
\end{array}
\]

Colorless oil; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 7.78-7.72 (m, 4H), 7.70-7.65 (m, 4H), 7.54-7.48 (m, 4H), 7.46-7.41 (m, 8H), 7.19 (dd, \(J_{\text{H-P}} = 27.9, 24.3\) Hz, 1H), 2.90-2.82 (m, 2H), 1.09-0.86 (m, 8H), 0.72 (t, \(J = 7.3\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) 158.1 (d, \(J_{\text{C-P}} = 58.5\) Hz), 135.1 (dd, \(J_{\text{C-P}} = 88.2, 7.6\) Hz), 133.7 (d, \(J = 104.9\) Hz), 132.3 (d, \(J = 10.5\) Hz), 131.5 (d, \(J = 115.4\) Hz), 131.7, 130.9 (d, \(J = 9.5\) Hz), 128.8 (dd, \(J = 11.4, 8.6\) Hz), 31.3 (dd, \(J = 10.0, 5.8\) Hz), 31.2, 31.1, 29.5, 22.4, 14.03; \(^{31}\)P NMR (162 MHz, CDCl\(_3\)): \(\delta\) 49.6 (d, \(J_{\text{P-P}} = 56.4\) Hz), 20.26 (d, \(J_{\text{P-P}} = 56.4\) Hz); IR (KBr, cm\(^{-1}\)): 3055, 2954, 2928, 1436, 1202, 744, 719, 693, 640; HRMS (ESI+) \(m/z\) calcd for C\(_{32}\)H\(_{34}\)NaOP\(_2\)S [M+Na]+: 551.1703, found: 551.1696.

\((E)\text{-}1\text{-}(\text{diphenylphosphinyl})\text{-}2\text{-}(\text{diphenylthiophosphinyl})\text{-}\text{dodec-1-ene}\ (3b)\)

\[
\begin{array}{c}
\text{Ph}_2\text{P} \\
\text{O} \\
\text{S} \\
\text{PPh}_2
\end{array}
\]

Colorless oil; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 7.79-7.71 (m, 4H), 7.70-7.65 (m, 4H), 7.54-7.48 (m, 4H), 7.46-7.41 (m, 8H), 7.20 (dd, \(J_{\text{H-P}} = 27.9, 24.0\) Hz, 1H), 2.90-2.81 (m, 2H), 1.33-0.90 (m, 16H), 0.86 (t, \(J = 7.3\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) 158.1 (d, \(J_{\text{C-P}} = 58.5\) Hz), 135.1 (dd, \(J_{\text{C-P}} = 88.2, 7.6\) Hz), 133.7 (d, \(J = 104.9\) Hz), 132.3 (d, \(J = 10.5\) Hz), 131.5 (d, \(J = 115.4\) Hz), 131.7, 130.9 (d, \(J = 9.5\) Hz), 128.8 (dd, \(J = 11.4, 8.6\) Hz), 31.3 (dd, \(J = 9.5, 6.7\) Hz), 31.1, 29.9, 29.6, 29.44, 29.36, 29.0, 22.8, 14.2; \(^{31}\)P NMR (162 MHz, CDCl\(_3\)): \(\delta\) 48.9 (d, \(J_{\text{P-P}} = 58.6\) Hz), 19.6 (d, \(J_{\text{P-P}} = 58.6\) Hz); IR (KBr, cm\(^{-1}\)): 3054, 2924, 2852, 1436, 1202.
1117, 1102, 744, 693, 640, 545, 527, 498; HRMS (ESI+) m/z calcd for C\textsubscript{36}H\textsubscript{42}NaOP\textsubscript{2}S [M+Na]\textsuperscript{+}: 607.2329, found: 607.2328.

\textit{(E)- 1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-5-methylhex-1-ene (3c)}

\begin{figure}
\centering
\includegraphics[width=0.5\textwidth]{3c.png}
\end{figure}

Colorless oil; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}): \(\delta 7.77-7.72\) (m, 4H), 7.70-7.66 (m, 4H), 7.54-7.49 (m, 4H), 7.47-7.42 (m, 8H), 7.27 (dd, \(J_{\text{H-P}} = 27.9, 23.8\) Hz, 1H), 2.90-2.81 (m, 2H), 1.28-1.17 (m, 1H), 0.94-0.87 (m, 2H), 0.56 (d, \(J = 6.9\) Hz, 6H); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}): \(\delta 158.3\) (d, \(J_{\text{C-P}} = 57.5\) Hz), 135.3 (dd, \(J_{\text{C-P}} = 88.2, 7.7\) Hz), 133.8 (d, \(J = 105.4\) Hz), 132.4 (d, \(J = 9.6\) Hz), 131.5 (d, \(J = 115.0\) Hz), 131.7, 130.9 (d, \(J = 10.5\) Hz), 128.8 (dd, \(J = 12.5, 6.7\) Hz), 39.6, 29.5 (dd, \(J = 9.6, 7.7\) Hz), 28.6, 22.0; \textsuperscript{31}P NMR (162 MHz, CDCl\textsubscript{3}): \(\delta 49.6\) (d, \(J_{\text{P-P}} = 56.4\) Hz), 20.0 (d, \(J_{\text{P-P}} = 56.4\) Hz); IR (KBr, cm\textsuperscript{-1}): 2955, 1436, 1198, 1102, 745, 719, 693; HRMS (ESI+) m/z calcd for C\textsubscript{31}H\textsubscript{32}NaOP\textsubscript{2}S [M+Na]\textsuperscript{+}: 537.1547, found: 537.1547.

\textit{(E)-1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-5-chloropent-1-ene (3d)}

\begin{figure}
\centering
\includegraphics[width=0.5\textwidth]{3d.png}
\end{figure}

Colorless oil; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}): \(\delta 7.77-7.71\) (m, 4H), 7.69-7.34 (m, 4H), 7.56-7.52 (m, 4H), 7.48-7.44 (m, 8H), 7.27 (dd, \(J_{\text{H-P}} = 28.0, 24.0\) Hz, 1H), 3.28 (t, \(J = 6.3\) Hz, 2H), 3.03-2.94 (m, 2H), 1.63-1.56 (m, 2H); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}): \(\delta 156.7\) (d, \(J_{\text{C-P}} = 59.1\) Hz), 136.0 (dd, \(J_{\text{C-P}} = 87.3, 6.7\) Hz), 133.1 (d, \(J = 104.9\) Hz), 132.3 (d, \(J = 9.5\) Hz), 130.9 (d, \(J = 9.5\) Hz), 130.7 (d, \(J = 83.9\) Hz), 129.0 (dd, \(J = 12.4, 3.8\) Hz), 44.73, 33.7, 28.9 (dd, \(J = 10.0, 6.7\) Hz); \textsuperscript{31}P NMR (162 MHz, CDCl\textsubscript{3}): \(\delta 48.9\) (d, \(J_{\text{P-P}} = 54.1\) Hz), 20.4 (d, \(J_{\text{P-P}} = 54.1\) Hz); IR (KBr,
Colorless oil; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.77-7.70 (m, 4H), 7.67-7.62 (m, 4H), 7.58-7.52 (m, 4H), 7.50-7.44 (m, 4H), 7.22 (dd, $J_{\text{H-P}} = 27.1$, 24.0 Hz, 1H), 3.02-2.94 (m, 2H), 2.18 (t, $J = 7.2$ Hz, 2H), 1.55-1.47 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 155.8 (d, $J_{\text{C-P}} = 60.1$ Hz), 136.3 (dd, $J_{\text{C-P}} = 86.3$, 7.6 Hz), 133.1 (d, $J = 105.9$ Hz), 132.4 (d, $J = 2.9$ Hz), 132.3 (d, $J = 10.5$ Hz), 130.9 (d, $J = 9.5$ Hz), 130.6 (d, $J = 83.9$ Hz), 129.1 (d, $J = 12.4$ Hz), 119.2, 29.9 (dd, $J = 10.0$, 6.7 Hz), 27.0, 17.2; $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 48.6 (d, $J_{\text{P-P}} = 55.2$ Hz), 20.0 (d, $J_{\text{P-P}} = 55.2$ Hz); IR (KBr, cm$^{-1}$): 3054, 2937, 2245, 1436, 1197, 1102, 745, 720, 693, 642; HRMS (ESI+) $m/z$ calcd for C$_{30}$H$_{27}$NNaOP$_2$S [M+Na]$^+$: 534.1186, found: 534.1186.
20.0 (d, \( J_{P-P} = 58.2 \) Hz); IR (KBr, cm\(^{-1}\)) : 3054, 2937, 1732, 1436, 1196, 1101, 744, 719, 693; HRMS (ESI+) \( m/z \) calcd for C\(_{31}\)H\(_{30}\)NaO\(_3\)P\(_2\)S \([\text{M+Na}]^+\) : 567.1289, found: 567.1289.

\((E)-1\)-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-3-phenylprop-1-ene (3i)

\[
\text{Ph}_2\text{P} = \text{Ph}_2\text{S} \\
\text{O} \\
\begin{array}{c}
\end{array}
\]

White solide; mp. 124-125 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) : \( \delta \) 7.63-7.56 (m, 8H), 7.52-7.48 (m, 2H), 7.44-7.38 (m, 6H), 7.31-7.27 (m, 4H), 7.04 (dd, \( J_{H-P} = 25.8, 23.1 \) Hz, 1H), 6.99 (d, \( J = 5.9 \) Hz, 2H), 6.87-6.80 (m, 3H), 4.89 (d, \( J = 19.0 \) Hz, 2H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) : \( \delta \) 156.4 (d, \( J_{C-P} = 59.1 \) Hz), 136.6, 136.0 (dd, \( J_{C-P} = 87.7, 6.7 \) Hz), 133.3 (d, \( J = 104.9 \) Hz), 132.2 (d, \( J = 10.5 \) Hz), 132.1 (d, \( J = 2.9 \) Hz), 131.8 (d, \( J = 2.9 \) Hz), 130.9 (d, \( J = 9.5 \) Hz), 130.7 (d, \( J = 83.0 \) Hz), 129.6, 128.9 (d, \( J = 12.4 \) Hz), 128.5 (d, \( J = 12.4 \) Hz), 127.8, 126.1, 35.3 (dd, \( J = 10.5, 6.7 \) Hz); \(^{31}\)P NMR (162 MHz, CDCl\(_3\)) : \( \delta \) 49.8 (d, \( J_{P-P} = 54.2 \) Hz), 19.4 (d, \( J_{P-P} = 54.2 \) Hz); IR (KBr, cm\(^{-1}\)) : 3054, 2923, 1587, 1436, 1206, 1109, 741, 718, 692, 528; HRMS (ESI+) \( m/z \) calcd for C\(_{33}\)H\(_{28}\)NaOP\(_2\)S \([\text{M+Na}]^+\) : 557.1234, found: 557.1228.

\((E)-1\)-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-(2-cyclohexyl)ethene (3j)

\[
\text{Ph}_2\text{P} = \text{Ph}_2\text{S} \\
\text{O} \\
\begin{array}{c}
\end{array}
\]

Colorless oil; \(^1\)H NMR (400 MHz, CDCl\(_3\)) : \( \delta \) 7.83-7.75 (m, 4H), 7.63-7.56 (m, 4H), 7.53-7.48 (m, 4H), 7.47-7.39 (m, 8H), 6.82 (dd, \( J_{H-P} = 30.4, 21.3 \) Hz, 1H), 3.02-2.91 (m, 1H), 2.28-2.18 (m, 2H), 1.65-1.46 (m, 3H), 1.39-1.30 (m, 2H), 1.28-1.18 (m, 1H), 1.03-0.92 (m, 2H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) : \( \delta \) 161.3 (d, \( J_{C-P} = 56.3 \) Hz), 134.6 (dd, \( J_{C-P} = 87.7, 8.6 \) Hz), 134.1 (d, \( J = 105.9 \) Hz), 132.3 (d, \( J = 10.5 \) Hz), 132.9 (dd, \( J = 13.4, 1.9 \) Hz), 131.1 (d, \( J = 83.0 \) Hz),
130.8 (d, $J = 9.5$ Hz), 128.7 (dd, $J = 12.4, 6.7$ Hz), 43.1 (dd, $J = 11.4, 5.7$ Hz), 32.6, 29.4, 25.2;

$^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 51.1 (d, $J_{P-P} = 60.6$ Hz), 18.2 (d, $J_{P-P} = 60.6$ Hz); IR (KBr, cm$^{-1}$): 3055, 2927, 2852, 2226, 1436, 1200, 1117, 1102, 734, 720, 694, 645; HRMS (ESI+) $m/z$ calcd for C$_{32}$H$_{32}$NaOP$_2$S [M+Na]$^+$: 549.1547, found: 549.1547.

(E)-1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-2-(4-florophenyl)ethene (3k)

![structure](image)

Colorless oil; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.78-7.73 (m, 4H), 7.56 (dd, $J_{H-P} = 24.9, 20.1$ Hz, 1H), 7.53-7.44 (m, 5H), 7.40-7.35 (m, 6H), 7.28-7.24 (m, 5H), 6.90 (td, $J = 6.9, 1.4$ Hz, 2H), 6.51 (t, $J = 8.7$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 162.7 (d, $J_{C-F} = 248.9$ Hz), 156.2 (d, $J_{C-P} = 60.1$ Hz), 138.3 (d, $J_{C-P} = 88.7, 9.5$ Hz), 132.5 (d, $J_{C-P} = 105.9$ Hz), 132.6 (d, $J_{C-P} = 10.5$ Hz), 132.1 (d, $J = 2.9$ Hz), 130.0 (t, $J = 4.8$ Hz), 131.8 (d, $J = 1.9$ Hz), 130.9, 130.7 (d, $J = 9.5$ Hz), 129.5 (d, $J_{C-P} = 84.9$ Hz), 128.57, 128.58 (d, $J = 21.9$ Hz), 128.0 (d, $J = 12.4$ Hz), 127.6 (d, $J = 12.4$ Hz), 114.4 (d, $J_{C-F} = 21.0$ Hz); $^{19}$F NMR (377 MHz, CDCl$_3$): $\delta$ -112.43; $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 47.8 (d, $J_{P-P} = 50.7$ Hz), 18.4 (d, $J_{P-P} = 50.7$ Hz); IR (KBr, cm$^{-1}$): 3057, 1600, 1503, 1436, 1229, 1187, 1099, 838, 747, 726, 692, 639, 548, 528, 503; HRMS (ESI+) $m/z$ calcd for C$_{32}$H$_{25}$FNaOP$_2$S [M+Na]$^+$: 561.0983, found: 561.0982.

(E)-1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-2-(4-methoxyphenyl)ethene (3l)

![structure](image)
Colorless oil; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.77-7.72 (m, 4H), 7.58 (dd, $J_{H-P} = 25.4$, 20.1 Hz, 1H), 7.56-7.51 (m, 4H), 7.50-7.45 (m, 2H), 7.41-7.36 (m, 6H), 7.32-7.27 (m, 4H), 6.85 (dd, $J = 8.7$, 1.8 Hz, 2H), 6.39 (d, $J = 8.2$ Hz, 2H), 3.65 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 159.4, 152.6 (d, $J_{C-P} = 59.4$ Hz), 138.9 (dd, $J_{C-P} = 73.3$, 11.0 Hz), 132.9 (d, $J = 105.9$ Hz), 132.7 (d, $J = 10.5$ Hz), 131.63 (d, $J = 2.9$ Hz), 131.57 (d, $J = 3.8$ Hz), 132.0 (d, $J = 2.9$ Hz), 131.9, 131.7 (dd, $J = 8.6$, 2.9 Hz), 131.4, 131.3, 130.2 (d, $J = 83.4$ Hz), 128.6 (d, $J = 12.5$ Hz), 128.4 (d, $J = 13.4$ Hz), 124.8, 112.7, 55.2; $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 47.5 (d, $J_{P-P} = 52.0$ Hz), 17.90 (d, $J_{P-P} = 52.0$ Hz); IR (KBr, cm$^{-1}$): 3053, 2924, 1605, 1505, 1436, 1250, 1180, 1099, 748, 719, 693, 640; HRMS (ESI+) $m/z$ calcd for C$_{33}$H$_{28}$NaO$_2$P$_2$S [M+Na]$^+$: 573.1183, found: 573.1183.

$(E)$-1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-2-(4-tert-butylphenyl)ethene

(3m)

White solid; mp. > 250 °C; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.74-7.68 (m, 4H), 7.64 (dd, $J_{H-P} = 25.7$, 19.7 Hz, 1H), 7.53-7.44 (m, 6H), 7.39-7.32 (m, 6H), 7.28-7.24 (m, 4H), 6.83 (d, $J = 8.2$ Hz, 2H), 6.78 (dd, $J = 8.7$, 1.4 Hz, 2H), 1.41 (s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 156.8 (d, $J_{C-P} = 70.9$ Hz), 151.3, 138.2 (dd, $J_{C-P} = 89.1$, 8.6 Hz), 133.0 (d, $J = 106.4$ Hz), 132.7 (d, $J = 10.54$ Hz), 132.0 (d, $J = 2.9$ Hz), 131.5 (d, $J = 1.9$ Hz), 130.8 (d, $J = 9.6$ Hz), 130.1 (d, $J = 84.35$ Hz), 129.9 (d, $J = 3.8$ Hz), 128.5 (dd, $J = 12.5$, 7.7 Hz), 124.2, 34.5, 31.2; $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 48.5 (d, $J_{P-P} = 52.0$ Hz), 18.9 (d, $J_{P-P} = 52.0$ Hz); IR (KBr) 3051, 2924, 1605, 1436, 1250, 1180, 1099, 748, 719, 693, 640; HRMS (ESI+) $m/z$ calcd for C$_{36}$H$_{34}$NaOP$_2$S [M+Na]$^+$: 599.1703, found: 599.1702.
(E)-1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-2-(4-octylphenyl)ethene (3n)

Colorless oil; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.74-7.68 (m, 4H), 7.66 (dd, $J_{\text{H-P}}$ = 25.4, 20.2 Hz, 1H), 7.55-7.49 (m, 4H), 7.46-7.43 (m, 2H), 7.38-7.33 (m, 6H), 7.29-7.24 (m, 4H), 6.76 (dd, $J$ = 7.8, 1.4 Hz, 2H), 6.63 (d, $J$ = 7.8 Hz, 2H), 2.36 (t, $J$ = 7.6 Hz, 2H), 1.46-1.36 (m, 2H), 1.31-1.21 (m, 10H), 0.88 (t, $J$ = 6.6 Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 156.9 (d, $J_{\text{C-P}}$ = 58.5 Hz), 143.4, 138.0 (dd, $J_{\text{C-P}}$ = 89.1, 9.6 Hz), 133.0 (d, $J_{\text{C-P}}$ = 106.4 Hz), 132.7 (d, $J$ = 9.6 Hz), 131.9 (d, $J$ = 1.9 Hz), 131.5 (d, $J$ = 2.9 Hz), 130.8 (d, $J$ = 10.5 Hz), 130.6 (d, $J$ = 8.6 Hz), 129.99 (d, $J$ = 84.4 Hz), 129.95 (d, $J$ = 3.8 Hz), 128.4 (dd, $J$ = 12.5, 6.7 Hz), 127.3, 35.6, 32.0, 31.3, 29.5, 29.4, 29.3, 22.8, 14.2; $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 48.4 (d, $J_{\text{P-P}}$ = 52.0 Hz), 18.9 (d, $J_{\text{P-P}}$ = 52.0 Hz); IR (KBr, cm$^{-1}$): 2925, 2854, 1437, 1186, 1106, 720, 693; HRMS (ESI+) $m/z$ calcd for C$_{40}$H$_{42}$NaOP$_2$S [M+Na]$^+$: 655.2329, found: 655.2329.

(3o)

White solid; mp. 89-90 °C; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.80-7.75 (m, 4H), 7.68 (dd, $J_{\text{H-P}}$ = 30.2, 19.2 Hz, 1H), 7.56-7.52 (m, 4H), 7.45-7.47 (m, 2H), 7.30-7.39 (m, 8H), 7.06 (d, $J$ = 8.2 Hz, 2H), 6.93 (dd, $J$ = 8.2, 1.4 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 152.3 (d, $J_{\text{C-P}}$ = 59.1 Hz), 141.0 (dd, $J_{\text{C-P}}$ = 72.5, 9.5 Hz), 132.8 (d, $J$ = 10.5 Hz), 132.7 (d, $J$ = 4.8 Hz), 132.4, 132.3 (d, $J$ = 5.7 Hz), 132.1, 131.8, 131.7 (d, $J$ = 78.2 Hz), 131.6 (d, $J$ = 8.6 Hz), 131.5 (d, $J$ = 10.5 Hz), 131.3 (d, $J$ = 2.9 Hz), 130.8 (d, $J$ = 2.9 Hz), 130.2 (d, $J$ = 84.9 Hz),
128.9, 128.6 (d, $J = 12.4$ Hz), 128.5 (d, $J = 12.4$ Hz), 128.4 (d, $J = 13.4$ Hz), 127.6, 127.0, 126.8 (d, $J = 68.6$ Hz), 125.8; $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 48.4 (d, $J_{P-P} = 52.0$ Hz), 18.9 (d, $J_{P-P} = 52.0$ Hz); IR (KBr, cm$^{-1}$): 3053, 1482, 1436, 1185, 1099, 719, 692, 642, 545, 509; HRMS (ESI+) $m/z$ calcd for C$_{40}$H$_{42}$NaOP$_2$S [M+Na]$^+$: 655.2329, found: 655.2329.
2. Copies of NMR spectra

![NMR spectra diagram]
abundance
0 0.1 0.2 0.3 0.4 0.5 0.6 0.7
X : parts per Million : Carbon13

Ph$_2$P$_2$O

nHex

S$^3$PPh$_2$

3a
Ph₂P-OⁿDec

S-PPh₂

3b

 abuncl1.0 2.0 3.0

X : parts per Million : Proton

8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0...

1.016

0.997

0.981

0.962

0.944

0.925

0.910

0.878

0.860

0.842

16.18

8.20

4.08

4.00

3.99

3.24

1.94

1.11

1.01

1.00

0.99

0.98

0.97

0.96

0.95

0.94

0.93

0.92

0.91

0.90

0.89

0.88

0.87

0.86

0.85
abundance
0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8
X : parts per Million : Carbon13
170.0 160.0 150.0 140.0 130.0 120.0 110.0 ...
128.827
128.740
77.486
77.160
76.844
31.970
31.280
31.107
29.919
29.555
29.440
29.364
29.009
22.782
14.236
abundance
0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0 13.0 14.0
X : parts per Million : Phosphorus31
100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0 -30.0
49.741
49.366
20.197
19.848
abundance
0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0
X : parts per Million : Proton
8.0 7.0 6.0 5.0 4.0 3.0 ... 8
3.009
2.986
2.964
2.944
1.632
1.615
1.603
1.594
1.575
1.557
1.535
8.404.34
4.074.03
2.00
1.96
2.04
1.17
abundance
0 0.1 0.2 0.3 0.4 0.5 0.6
X : parts per Million : Carbon13
170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 ... 29
132.295
132.189
130.906
130.810
130.082
129.191
129.066
119.246
77.486
77.160
76.844
29.814
26.969
17.197
abundance

X : parts per Million : Proton

8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0

3g
$\text{Ph}_2\text{P} - \sigma - \text{Ph}_2\text{P}$

3k
\text{X : parts per Million : Phosphorus31}
**Abundance**

| Parts Per Million (ppm) | Peak Intensity |
|-------------------------|----------------|
| 7.425                   | 6.0            |
| 7.417                   | 4.0            |
| 7.402                   | 2.0            |
| 7.395                   | 1.0            |
| 7.388                   | 1.0            |
| 7.281                   | 1.0            |
| 7.273                   | 1.0            |
| 7.260                   | 1.0            |
| 7.047                   | 4.0            |
| 6.474                   | 2.0            |
| 8.06                    | 2.0            |
| 7.04                    | 2.0            |
| 4.00                    | 2.0            |
| 4.00                    | 2.0            |
| 2.09                    | 2.0            |
| 2.00                    | 2.0            |
| 2.03                    | 2.0            |
| 0.97                    |                |

**Chemical Structure**

![Chemical Structure Image]

**30**

X: parts per Million; Proton
