Supporting Information

Air-induced Double Addition of P(O)-H to Bond to Alkynes: A Clean and Practical Method for the Preparation of 1,2-Bisphosphorylethanes

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General Comments

Unless otherwise noted, all reagents were purchased from commercial suppliers and used without further purification. All solvents were dried by standard methods. Diphenylphosphine oxide from Katayama Chemical Industries Company was purified by sublimation and stored in the glove box. $^1$H, $^{13}$C, and $^{31}$P NMR spectra were recorded on a JEOL (Tokyo, Japan) JNM-ECX400 FT NMR (400 MHz for $^1$H, 100 MHz for $^{13}$C, and 162 MHz for $^{31}$P ) using CDCl$_3$. The coupling constants $J$ are given in Hz. Melting points were determined on Opti Melt MPA100 apparatus (TOKYO INSTRUMENT, INC.).
Experimental procedures:

A typical procedure at 0.1 mmol scale: In a 1 mL closed vial were placed diphenylphosphine oxide (0.2 mmol), 1-octyne (0.1 mmol), dioxane (25μL), and air (1 mL) under nitrogen. The mixture was stirred at 100 °C for 22 h. The NMR yield of the product 1a was obtained in 80% based on $^{31}$P NMR spectroscopy. The product was isolated using a preparative GPC (Japan Analytical Industry Co., Ltd., LC-908) using chloroform as an eluent.

A typical procedure under solvent-free conditions at 1 mmol scale: In a 10 mL Schlenk flask were placed diphenylphosphine oxide (1.0 mmol), 1-octyne (1.5 mmol), and air (1 mL) under nitrogen. The mixture was stirred at 150 °C for 43 h. The mixture was washed with hexane (8 mL, 60 °C) to remove the remained starting materials to give NMR spectroscopically pure 1a.
1a \(^1\)

\[
\begin{align*}
\text{C}_6\text{H}_{13} & \quad \text{P(O)Ph}_2 \\
& \quad \text{P(O)Ph}_2 \\
\end{align*}
\]

\(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) 7.78–7.67 (m, 6H), 7.48–7.30 (m, 14H), 3.03–2.93 (m, 1H), 2.66–2.45 (m, 3H), 1.77–1.62 (m, 1H), 1.48–1.36 (m, 1H), 1.27–1.16 (m, 1H), 1.04–0.94 (m, 1H), 0.92–0.78 (m, 3H), 0.76–0.68 (m, 4H); \(^{31}\)P NMR (CDCl\(_3\), 162 MHz) \(\delta\) 37.9 (d, \(J = 48.6\) Hz), 30.9 (d, \(J = 48.6\) Hz).

1b

White solid; m.p. 146-148 °C; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) 7.71–7.62 (m, 6H), 7.45–7.28 (m, 14H), 3.09 (t, \(J = 6.6\) Hz, 2H), 2.95–2.85 (m, 1H), 2.61–2.40 (m, 2H), 1.79–1.64 (m, 1H), 1.50–1.29 (m, 2H), 1.27–1.13 (m, 2H), 1.09–0.98 (m, 1H); \(^{13}\)C NMR (CDCl\(_3\), 100 MHz) \(\delta\) 133.2 (d, \(J_{C-P} = 97.7\) Hz), 132.8 (d, \(J_{C-P} = 99.6\) Hz), 132.2, 132.0, 131.9, 131.8, 131.0 (d, \(J_{C-P} = 8.6\) Hz), 130.8 (d, \(J_{C-P} = 8.6\) Hz), 130.6 (d, \(J_{C-P} = 9.6\) Hz), 130.6 (d, \(J_{C-P} = 9.6\) Hz), 128.8 (d, \(J_{C-P} = 10.5\) Hz), 128.7 (d, \(J_{C-P} = 11.5\) Hz), 128.7 (d, \(J_{C-P} = 11.5\) Hz), 128.7 (d, \(J_{C-P} = 11.5\) Hz), 128.7 (d, \(J_{C-P} = 11.5\) Hz), 44.3, 32.5, 31.3 (dd, \(J_{C-P} = 69.0\) Hz, \(J = 3.8\) Hz), 27.5, 27.3 , \(J_{C-P} = 69.0\) Hz), 24.4 (d, \(J = 3.8\) Hz); \(^{31}\)P NMR (CDCl\(_3\), 162 MHz) \(\delta\) 37.5 (d, \(J = 44.9\) Hz), 30.9 (d, \(J = 44.9\) Hz).

1c

Colorless gel; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) 7.68–7.56 (m, 6H), 7.45–7.25 (m, 14H), 3.37 (br, 2H), 2.83–2.56 (m, 3H), 2.44–2.35 (m, 1H), 1.89–1.72 (m, 1H), 1.63–1.40 (m, 1H), 1.15 (br, 3H); \(^{13}\)C NMR (CDCl\(_3\), 100 MHz) \(\delta\) 133.1 (d, \(J_{C-P} = 100.6\) Hz), 132.1, 131.8, 131.6 (d, \(J_{C-P} = 99.6\) Hz), 130.9 (d, \(J_{C-P} = 8.6\) Hz), 130.9 (d, \(J_{C-P} = 8.6\) Hz), 130.9 (d, \(J_{C-P} = 8.6\) Hz), 130.8 (d, \(J_{C-P} = 8.6\) Hz), 128.8 (d, \(J_{C-P} = 10.5\) Hz), 128.8 (d, \(J_{C-P} = 10.5\) Hz), 128.8 (d, \(J_{C-P} = 10.5\) Hz), 128.8 (d, \(J_{C-P} = 11.5\) Hz), 128.8 (d, \(J_{C-P} = 11.5\) Hz), 128.8 (d, \(J_{C-P} = 11.5\) Hz), 128.8 (d, \(J_{C-P} = 11.5\) Hz), 60.6, 31.8 (dd, \(J_{C-P} = 68.0\) Hz, \(J = 3.8\) Hz), 31.7, 27.3 (d, \(J_{C-P} = 68.0\) Hz), 26.8, 22.9 (d, \(J = 3.8\) Hz); \(^{31}\)P NMR (CDCl\(_3\), 162 MHz) \(\delta\) 38.0 (d, \(J = 52.3\) Hz), 32.6 (d, \(J = 52.3\) Hz).

1d \(^1\)c

\(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) 7.76–7.62 (m, 6H), 7.53–7.33 (m, 14H), 2.88 (brs, 1H), 2.64–2.43 (m, 2H), 2.02–1.85 (m, 3H), 1.79–1.57 (m, 2H), 1.41–1.30 (m, 1H); \(^{31}\)P NMR (CDCl\(_3\), 162 MHz) \(\delta\) 37.2 (d, \(J = 44.9\) Hz), 31.1 (d, \(J = 44.9\) Hz).

1e \(^1\)c
$^{1}$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.71–7.60 (m, 6H), 7.44–7.28 (m, 14H), 3.84–3.74 (m, 2H), 3.06–2.91 (m, 1H), 2.61–2.43 (m, 2H), 2.13–2.00 (m, 1H), 1.97–1.85 (m, 1H), 1.00 (s, 9H); $^{31}$P NMR (CDCl$_3$, 162 MHz) $\delta$ 37.3 (d, $J = 44.9$ Hz), 31.0 (d, $J = 44.9$ Hz).

White solid; m.p. 155-156 °C; $^{1}$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.70–7.61 (m, 6H), 7.46–7.26 (m, 14H), 4.25 (dd, $J = 16.5, 5.5$ Hz, 2H), 3.37–3.25 (m, 1H), 2.66–2.47 (m, 2H), 1.75 (q, $J = 7.5$ Hz, 2H), 0.82 (t, $J = 7.8$ Hz, 3H); $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 173.4, 132.9 (d, $J_{C-P} = 99.6$ Hz), 132.1 (d, $J_{C-P} = 2.9$ Hz), 132.0 (d, $J_{C-P} = 2.9$ Hz), 131.9 (d, $J_{C-P} = 2.9$ Hz), 131.6 (d, $J_{C-P} = 97.7$ Hz), 131.0 (d, $J_{C-P} = 8.6$ Hz), 131.0 (d, $J_{C-P} = 9.6$ Hz), 130.6 (d, $J_{C-P} = 9.6$ Hz), 128.9 (d, $J_{C-P} = 11.5$ Hz), 128.7 (d, $J_{C-P} = 11.5$ Hz), 128.7 (d, $J_{C-P} = 11.5$ Hz), 128.6 (d, $J_{C-P} = 10.5$ Hz), 64.6, 32.1 (dd, $J_{C-P} = 68.0$ Hz, $J = 3.8$ Hz), 26.8, 25.3 (d, $J_{C-P} = 68.0$ Hz, 8.7); $^{31}$P NMR (CDCl$_3$, 162 MHz) $\delta$ 34.7 (d, $J = 41.1$ Hz), 31.1 (d, $J = 41.1$ Hz).

White solid; m.p. 117-118 °C; $^{1}$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.75–7.15 (m, 20H), 3.90–3.76 (m, 0.5H), 3.24–2.93 (m, 1.5H), 2.88–2.75 (m, 0.5H), 2.70–2.59 (m, 0.5H), 2.50–2.28 (m, 1H), 2.16–2.02 (m, 0.5H), 1.86–1.73 (m, 0.5H), 1.69–1.55 (m, 1H), 0.98 (d, $J = 5.9$ Hz, 1.5H), 0.79 (d, $J = 5.9$ Hz, 1.5H); $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 133.0 (d, $J_{C-P} = 101.6$ Hz), 132.1 (dd, $J_{C-P} = 2.9, 2.9$ Hz), 132.0 (dd, $J_{C-P} = 2.9, 2.9$ Hz), 131.8 (dd, $J_{C-P} = 2.9, 2.9$ Hz), 131.6 (dd, $J_{C-P} = 2.9, 2.9$ Hz), 131.6 (d, $J_{C-P} = 99.6$ Hz), 131.1-130.8 (m), 130.7 (dd, $J_{C-P} = 8.6, 9.6$ Hz), 130.3 (dd, $J_{C-P} = 9.6, 9.6$ Hz), 128.9-128.5 (m), 65.4 (d, $J_{C-P} = 2.9$ Hz), 63.2 (d, $J_{C-P} = 12.5$ Hz), 38.4 (d, $J_{C-P} = 193.5$ Hz), 29.6-26.9 (m), 23.7 (d, $J_{C-P} = 107.3$ Hz), $^{31}$P NMR (CDCl$_3$, 162 MHz) $\delta$ 39.4 (dd, $J = 48.6, 41.1$ Hz), 33.8 (dd, $J = 48.6, 41.1$ Hz).

White solid; m.p. 182-183 °C; $^{1}$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.95–7.90 (m, 2H), 7.71–7.66 (m, 2H), 7.63–7.28 (m, 2H), 7.45–7.30 (m, 7H), 7.21–7.11 (m, 7H), 4.87 (br, s, 1H), 3.42–3.33 (m, 1H), 2.98–
2.89 (m, 1H), 2.62–2.48 (m, 1H), 1.71–1.59 (m, 1H), 1.54–1.44 (m, 1H), 1.41–1.33 (m, 1H), 1.28–1.12 (m, 4H); $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 134.1 (d, $J_{C-P} = 94.9$ Hz), 133.5 (d, $J_{C-P} = 99.6$ Hz), 133.3 (d, $J_{C-P} = 99.6$ Hz), 131.9 (d, $J_{C-P} = 98.7$ Hz), 131.9 (br), 131.4 (br), 131.2 (d, $J_{C-P} = 8.6$ Hz), 130.6 (d, $J_{C-P} = 9.6$ Hz), 130.5 (d, $J_{C-P} = 9.6$ Hz), 130.2 (d, $J_{C-P} = 9.6$ Hz), 128.7 (d, $J_{C-P} = 12.5$ Hz), 128.6 (d, $J_{C-P} = 11.5$ Hz), 128.5 (d, $J_{C-P} = 11.5$ Hz), 128.4 (d, $J_{C-P} = 12.5$ Hz), 83.4 (dd, $J_{C-P} = 2.9$, 2.9 Hz), 40.3 (d, $J_{C-P} = 9.6$ Hz), 40.1, 39.7 (dd, $J_{C-P} = 66.1$ Hz, $J = 3.8$ Hz), 26.3 ($J_{C-P} = 68.0$ Hz), 23.6, 22.3; $^{31}$P NMR (CDCl$_3$, 162 MHz) $\delta$ 40.6 (d, $J = 22.4$ Hz), 30.7 (d, $J = 22.4$ Hz).

$^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 8.14–7.95 (m, 2H), 7.66–7.48 (m, 5H), 7.46–6.95 (m, 15H), 6.93–6.73 (m, 3H), 4.42–4.12 (m, 1H), 3.36–3.00 (m, 1H), 3.00–2.73 (m, 1H); $^{31}$P NMR (CDCl$_3$, 162 MHz) $\delta$ 36.0 (d, $J = 48.6$ Hz), 30.8 (d, $J = 48.6$ Hz).

$^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.98–7.91 (m, 2H), 7.51–7.44 (m, 5H), 7.39–7.34 (m, 1H), 7.30–7.24 (m, 4H), 7.22–7.28 (m, 3H), 7.16–7.08 (m, 3H), 7.02 (ddd, $J = 7.8$, 7.8, 2.7 Hz, 2H), 6.93 (dd, $J = 8.7$, 1.8 Hz, 2H), 6.28 (d, $J = 8.7$ Hz, 2H), 4.17 (dddd, $J = 12.4$, 12.4, 12.4, 1.4 Hz, 1H), 3.55 (s, 3H), 3.03 (dddd, $J = 11.9$, 11.4, 4.6, 4.6 Hz, 1H), 2.72 (dddd, $J = 15.1$, 15.4, 15.1, 1.4 Hz, 1H); $^{31}$P NMR (CDCl$_3$, 162 MHz) $\delta$ 35.8 (d, $J = 48.6$ Hz), 30.6 (d, $J = 48.6$ Hz).
**1H NMR (CDCl₃, 400 MHz)** δ 7.96–7.91 (m, 1H), 7.49–7.44 (m, 5H), 7.36 (dd, J = 7.3, 1.4 Hz, 1H), 7.31–7.26 (m, 4H), 7.24–7.16 (m, 4H), 7.10 (ddd, J = 7.8, 7.8, 3.2 Hz, 2H), 7.05 (ddd, J = 7.8, 7.8, 3.2 Hz, 2H), 6.78 (dd, J = 8.2, 1.8 Hz, 2H), 6.07 (d, J = 8.2 Hz, 2H), 4.10 (ddd, J = 12.8, 11.9, 6.9, 1.8 Hz, 1H), 3.01 (ddd, J = 11.9, 11.4, 4.6, 4.6 Hz, 1H), 2.71 (ddd, J = 15.1, 15.1, 11.4, 1.8 Hz, 1H); **31P NMR (CDCl₃, 162 MHz)** δ 35.8 (d, J = 48.6 Hz), 30.9 (d, J = 48.6 Hz).

**1m**

[Diagram of 1m]

**1H NMR (CDCl₃, 400 MHz)** δ 8.01–7.94 (m, 2H), 7.52–7.45 (m, 5H), 7.38 (ddd, J = 7.3, 7.3, 1.4 Hz, 1H), 7.32–7.25 (m, 4H), 7.24–7.17 (m, 3H), 7.16–7.08 (m, 5H), 7.00–6.95 (m, 4H), 4.30 (ddd, J = 12.1, 12.1, 6.9, 1.8 Hz, 1H), 3.06 (ddd, J = 11.9, 11.9, 4.1, 1.4 Hz, 1H), 2.76 (ddd, J = 15.3, 15.3, 11.4, 1.8 Hz, 1H); **31P NMR (CDCl₃, 162 MHz)** δ 35.3 (d, J = 44.9 Hz), 30.2 (d, J = 44.9 Hz).

**1n**

[Diagram of 1n]

**1H NMR (CDCl₃, 400 MHz)** δ 7.99–7.93 (m, 2H), 7.52–7.43 (m, 5H), 7.39 (ddd, J = 7.3, 7.3, 1.4 Hz, 1H), 7.34–7.26 (m, 6H), 7.24–7.18 (m, 3H), 7.14–7.08 (m, 5H), 6.97 (ddd, J = 7.8, 7.8, 3.2 Hz, 1H), 4.28 (ddd, J = 11.9, 11.9, 6.9, 1.4 Hz, 1H), 3.08 (ddd, J = 11.4, 11.4, 4.1, 1.4 Hz, 1H), 2.77 (ddd, J = 15.1, 15.1, 11.4, 1.8 Hz, 1H), 2.36 (s, 3H); **31P NMR (CDCl₃, 162 MHz)** δ 35.2 (d, J = 44.9 Hz), 30.2 (d, J = 44.9 Hz).

**1o**

[Diagram of 1o]

**1H NMR (CDCl₃, 400 MHz)** δ 7.98–7.93 (m, 2H), 7.53–7.44 (m, 5H), 7.38 (ddd, J = 7.3, 7.3, 1.4 Hz, 1H), 7.32–7.26 (m, 4H), 7.24–7.17 (m, 4H), 7.12 (ddd, J = 7.3, 7.3, 2.7 Hz, 2H), 7.04 (ddd, J = 7.8, 7.8, 3.2 Hz, 2H), 6.95 (dd, J = 8.7, 1.8 Hz, 2H), 6.69 (d, J = 8.2 Hz, 2H), 4.20 (ddd, J = 12.4, 12.4,
6.4, 1.8 Hz, 1H), 3.02 (dddd, J = 11.9, 11.9, 4.6, 4.6 Hz, 1H), 2.73 (dddd, J = 15.1, 15.1, 11.0, 1.8 Hz, 1H); ³¹P NMR (CDCl₃, 162 MHz) δ 35.4 (d, J = 48.6 Hz), 30.3 (d, J = 48.6 Hz).

1p

![Image of 1p](image)

¹H NMR (CDCl₃, 400 MHz) δ 8.08–7.98 (m, 2H), 7.74–7.67 (m, 1H), 7.63–7.53 (m, 5H), 7.50–7.33 (m, 5H), 7.32–7.18 (m, 4H), 7.16–7.07 (m, 4H), 7.01 (dd, J = 7.3, 7.3 Hz, 1H), 6.85 (dd, J = 7.8, 7.3 Hz, 1H), 6.78 (d, J = 7.8 Hz, 1H), 4.92 (ddd, J = 11.9, 11.9, 7.3 Hz, 1H), 3.22–3.08 (m, 1H); ³¹P NMR (CDCl₃, 162 MHz) δ 35.4 (d, J = 48.6 Hz), 30.3 (d, J = 48.6 Hz).

1q

![Image of 1q](image)

¹H NMR (CDCl₃, 400 MHz) δ 8.79–6.06 (br, 29H), 4.49 (br, 29H), 3.57 (br, 1H), 2.81 (m, 1H); ³¹P NMR (CDCl₃, 162 MHz) δ 35.2 (d, J = 41.1 Hz), 31.0 (d, J = 41.1 Hz).

1r

![Image of 1r](image)

Colorless oil; ¹H NMR (CDCl₃, 400 MHz) δ 7.68–7.48 (m, 6H), 7.35–7.30 (m, 2H), 6.90–6.80 (m, 6H), 6.78 (d, J = 7.3 Hz, 2H), 3.75–3.37 (m, 12H), 3.00–2.71 (m, 1H), 2.63–2.35 (m, 2H), 1.73–1.54 (m, 1H), 1.43–1.27 (m, 1H), 1.26–1.09 (m, 2H), 0.91–0.78 (m, 2H), 0.78–0.71 (m, 2H), 0.68 (t, J = 7.3, 7.3 Hz, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ 162.2, 162.1, 132.8 (d, J_C–P = 8.6 Hz), 132.7 (d, J_C–P = 9.6 Hz), 132.5 (d, J_C–P = 8.6 Hz), 132.4 (d, J_C–P = 9.6 Hz), 124.8 (d, J_C–P = 108.3 Hz), 123.2 (d, J_C–P = 101.6 Hz), 114.3 (d, J_C–P = 12.5 Hz), 114.1 (d, J_C–P = 11.5 Hz), 55.3 (d, J_C–P = 2.9 Hz), 31.2, 30.8 (d, J_C–P = 228.0 Hz), 29.3, 28.4, 27.8 (dd, J_C–P = 69.9 Hz, J = 3.8 Hz), 27.1, 22.4, 14.0; ³¹P NMR (CDCl₃, 162 MHz) δ 38.5 (d, J = 44.9 Hz), 31.2 (d, J = 44.9 Hz).

1s
White solid; m.p. 52-53 °C; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) 7.99–7.75 (m, 6H), 7.69–7.66 (m, 4H), 7.63–7.40 (m, 6H), 3.35–2.98 (m, 1H), 2.81–2.42 (m, 2H), 1.74–1.49 (m, 1H), 1.49–1.29 (m, 1H), 1.18–1.05 (m, 1H), 1.00–0.87 (m, 3H), 0.83–0.71 (m, 4H), 0.66 (t, \(J = 7.3, 7.3\) Hz, 3H); \(^{13}\)C NMR (CDCl\(_3\), 100 MHz) \(\delta\) 134.4 (d, \(J_{C-P} = 15.3\) Hz), 134.2 (d, \(J_{C-P} = 14.4\) Hz), 134.2 (d, \(J_{C-P} = 99.6\) Hz), 134.1 (d, \(J_{C-P} = 13.4\) Hz), 133.9 (d, \(J_{C-P} = 12.5\) Hz), 131.6, 131.4, 131.1 (d, \(J_{C-P} = 3.8\) Hz), 130.8 (d, \(J_{C-P} = 3.8\) Hz), 130.0 (d, \(J_{C-P} = 98.7\) Hz), 126.1-125.6 (m), 123.4 (q, \(J_{C-P} = 273.1\) Hz), 123.3 (q, \(J_{C-P} = 274.0\) Hz), 123.3 (q, \(J_{C-P} = 273.1\) Hz), 123.2 (q, \(J_{C-P} = 273.1\) Hz), 31.1, 30.9 (dd, \(J_{C-P} = 68.0\) Hz, \(J = 3.8\) Hz), 29.4 (d, \(J_{C-P} = 64.2\) Hz), 29.0, 28.7, 26.9, 22.3, 13.8; \(^{31}\)P NMR (CDCl\(_3\), 162 MHz) \(\delta\) 35.6 (d, \(J = 22.4\) Hz), 28.1 (d, \(J = 22.4\) Hz).

It

White solid; m.p. 190-192 °C; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) 7.31–7.23 (m, 8H), 7.21–7.12 (m, 12H), 2.67–2.59 (m, 8H), 2.23–2.09 (m, 2H), 1.85–1.46 (m, 29H), 1.26 (br, s, 22H), 0.89 (t, \(J = 6.9, 6.9\) Hz, 3H); \(^{13}\)C NMR (CDCl\(_3\), 100 MHz) \(\delta\) 141.8 (d, \(J_{C-P} = 3.8\) Hz), 141.7, 128.4 (d, \(J_{C-P} = 2.9\) Hz), 128.3, 125.9 (d, \(J_{C-P} = 3.8\) Hz), 125.9 (d, \(J_{C-P} = 2.9\) Hz), 35.4, 33.0, 32.9, 32.9, 32.8, 32.8, 32.7, 31.9, 31.1 (dd, \(J_{C-P} = 69.0\) Hz, \(J = 3.8\) Hz), 30.5, 30.0, 29.7, 29.4, 29.4, 28.0, 28.0, 26.9 (d, \(J_{C-P} = 63.2\) Hz), 26.5 (d, \(J_{C-P} = 63.2\) Hz), 25.7 (d, \(J_{C-P} = 62.3\) Hz), 22.7, 21.5 (d, \(J_{C-P} = 2.9\) Hz), 21.4 (d, \(J_{C-P} = 3.89\) Hz), 21.3 (d, \(J_{C-P} = 3.8\) Hz), 14.1; \(^{31}\)P NMR (CDCl\(_3\), 162 MHz) \(\delta\) 53.8 (d, \(J = 22.4\) Hz), 48.9 (d, \(J = 22.4\) Hz).
References


NMR Spectra

1a

$^1$H NMR

$^{31}$P NMR
1b

$^1$H NMR

$^{13}$C NMR
$^{31}$P NMR

$^{1}$H NMR
**13C NMR**

![13C NMR spectrum](image1)

**31P NMR**

![31P NMR spectrum](image2)
1H NMR

31P NMR
1e

$^1$H NMR

$^{31}$P NMR
If

$^1$H NMR

$^{13}$C NMR
$^{31}$P NMR

$^{1}$H NMR

S18
**13C NMR**

**31P NMR**
$^{31}$P NMR

$^{1}$H NMR
$^{13}$P NMR

$^1$H NMR
$^{13}$P NMR

$^1$H NMR
$^{13}$P NMR

$^1$H NMR
$^{13}$P NMR

$^1$H NMR
The document contains two sections labeled "13P NMR" and "1H NMR". Each section includes a spectrum of a compound with labeled peaks and annotations. The spectra are labeled with chemical structures and formulas, indicating the presence of phosphorus and hydrogen atoms. The pages appear to be part of a scientific or technical report, possibly related to chemical analysis or organic chemistry.
$^{13}$P NMR

$^1$H NMR
$^{13}$P NMR

$^1$H NMR
$^3$P NMR

$^1$H NMR
$^{31}$P NMR

Ir

$^1$H NMR
$^1$H NMR

$^{13}$C NMR
$^{31}$P NMR

$^1$H NMR
$^{13}$C NMR

$^{31}$P NMR