Supporting Information

Direct C-OH/P(O)-H Dehydration Coupling Forming Phosphine Oxides

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Table of Contents

1) General information
2) Procedures for the base-promoted dehydration coupling of P(O) – H compounds with phenylethanol
3) Characterization and analytical data of products 3
4) References
5) Copies of 1H NMR, 13C NMR and 31P NMR spectra
1. General information

All reactions were carried out in dry Schlenk tubes under N₂ atmosphere. Dry solvents were obtained by purification according to standard methods. Reagents were used as received unless otherwise noted. Column chromatography was performed using Silica Gel 60 (particle size 37–54 μm). The pure products were obtained by means of column chromatography. ¹H NMR, ¹³C NMR and ³¹P NMR data were acquired on a Bruker-400 spectrometer (400 MHz for ¹H, 100 MHz for ¹³C, and 162 MHz for ³¹P NMR spectroscopy). Chemical shifts for ¹H NMR are referred to internal Me₄Si (0 ppm) and reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz) and integration.

2. Procedures for the base-promoted dehydration coupling of P(O)-H compounds with phenylethanol

1) A Typical procedure.

\[
\begin{align*}
\text{Ph} & \quad + \quad \text{Ph} \\
\text{OH} & \quad + \quad \text{P(O)} \\
1a & \quad + \quad 2a \\
\text{t-BuONa, DMAc} & \quad 130^\circ \text{C}, 24 \text{ h} \\
\text{Ph} & \quad \text{Ph} \\
3a & \quad \text{P(O)}
\end{align*}
\]

Under the Nitrogen atmosphere, 0.5 mmol phenylethanol 1a, 0.2 mmol diphenylphosphine oxide 2a, 0.2 mmol sodium tert-butoxide and 1 mL DMAc were charged into a 25 mL Schlenk tube, and the mixture was heated at 130 °C for 24 h. After being cooled to room temperature, the mixture was added to 10 mL CH₂Cl₂ and washed by 10 mL water for three times. After removal of the volatiles in vacuum, the residues were passed through a short silica chromatography (particle size 37–54 μm, ethyl acetate/petroleum ether as eluent) to afford analytically pure 3a in 80% isolated yield.

2) 10 mmol scale experiment

Under the Nitrogen atmosphere, 25 mmol phenylethanol 1a (3.0541 g), 10 mmol diphenylphosphine oxide 2a (2.0220 g), 10 mmol sodium tert-butoxide and 40 mL DMAc were charged into a 100 mL Schlenk tube, and the mixture was heated at 130 °C for 24 h. After being cooled to room temperature, the mixture was added to 200 mL CH₂Cl₂ and washed by 100 mL water for three times. After removal of the volatiles in vacuum, the residues were passed through a silica chromatography (particle size 37–54 μm, ethyl acetate/petroleum ether as eluent) to afford analytically pure 3a in 85% isolated yield (2.6041 g). When the experiment was conducted in the absence of solvent DMAc, 3a was obtained in 76% isolated yield according to a similar procedure (2.3281 g).
3. Characterization and analytical data of products 3

phenethyldiphenylphosphine oxide (3a). Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/2) afforded 3a (49.0 mg, 80% yield) as a white solid; \( ^1H \) NMR (400 MHz, CDCl\(_3\)): \( \delta \) 7.79–7.74 (m, 4H), 7.56–7.44 (m, 6H), 7.28–7.24 (m, 2H), 7.19–7.15 (m, 3H), 2.98–2.88 (m, 2H), 2.63–2.54 (m, 2H); \( ^{13}C \) NMR (100 MHz, CDCl\(_3\)): \( \delta \) 141.2 (d, \( J_{C,P} = 15.3 \) Hz), 132.7 (d, \( J_{C,P} = 97.8 \) Hz), 131.9 (d, \( J_{C,P} = 2.6 \) Hz), 130.8 (d, \( J_{C,P} = 9.2 \) Hz), 128.8, 128.7 (d, \( J_{C,P} = 6.5 \) Hz), 128.1, 126.4, 31.9 (d, \( J_{C,P} = 69.6 \) Hz), 27.5 (d, \( J_{C,P} = 3.0 \) Hz); \( ^{31}P \) NMR (162 MHz, CDCl\(_3\)): \( \delta \) 31.69. This compound is known.

(4-methylphenethyl)diphenylphosphine oxide (3b). Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/2) afforded 3b (46.8 mg, 73% yield) as a white solid; \( ^1H \) NMR (400 MHz, CDCl\(_3\)): \( \delta \) 7.81–7.71 (m, 4H), 7.55–7.43 (m, 6H), 7.05 (m, 4H), 2.92–2.86 (m, 2H), 2.60–2.53 (m, 2H), 2.29 (s, 3H); \( ^{13}C \) NMR (100 MHz, CDCl\(_3\)): \( \delta \) 138.1 (d, \( J_{C,P} = 15.3 \) Hz), 135.9, 132.8 (d, \( J_{C,P} = 97.6 \) Hz), 131.8 (d, \( J_{C,P} = 2.6 \) Hz), 130.8 (d, \( J_{C,P} = 9.3 \) Hz), 129.3, 128.7 (d, \( J_{C,P} = 11.5 \) Hz), 128.0, 32.0 (d, \( J_{C,P} = 69.3 \) Hz), 27.1 (d, \( J_{C,P} = 3.1 \) Hz), 21.0; \( ^{31}P \) NMR (162 MHz, CDCl\(_3\)): \( \delta \) 31.66. This compound is known.

(4-methoxyphenethyl)diphenylphosphine oxide (3c). Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/2) afforded 3c (47.1 mg, 70% yield) as a white solid; \( ^1H \) NMR (400 MHz, CDCl\(_3\)): \( \delta \) 7.82–7.71 (m, 4H), 7.55–7.48 (m, 6H), 7.09–7.07 (m, 2H), 6.81–6.79 (m, 2H), 3.77 (s, 3H), 2.91–2.85 (m, 2H), 2.59–2.52 (m, 2H); \( ^{13}C \) NMR (100 MHz, CDCl\(_3\)): \( \delta \) 158.1, 133.2 (d, \( J_{C,P} = 15.6 \) Hz), 132.8 (d, \( J_{C,P} = 97.5 \) Hz), 131.8 (d, \( J_{C,P} = 2.6 \) Hz), 130.8 (d, \( J_{C,P} = 9.3 \) Hz), 129.0, 128.7 (d, \( J_{C,P} = 11.5 \) Hz), 114.0, 55.3, 32.1 (d, \( J_{C,P} = 69.2 \) Hz), 26.7 (d, \( J_{C,P} = 3.1 \) Hz); \( ^{31}P \) NMR (162 MHz, CDCl\(_3\)): \( \delta \) 31.62. This compound is known.
(4-fluorophenethyl)diphenylphosphine oxide (3d). Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (2/1) afforded 3d (38.3 mg, 59% yield) as a white solid; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.72–7.64 (m, 4H), 7.48–7.36 (m, 6H), 7.06–7.00 (m, 2H), 6.84 (m, 2H), 2.83 (m, 2H), 2.47 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 161.5 (d, $J_{C-P}$ = 244.4 Hz), 136.7 (dd, $J_{C-P}$ = 15.0 Hz, $J_{C-F}$ = 3.1 Hz), 132.7 (d, $J_{C-P}$ = 98.3 Hz), 131.9 (d, $J_{C-P}$ = 2.6 Hz), 130.8 (d, $J_{C-P}$ = 9.3 Hz), 129.5 (d, $J_{C-P}$ = 7.9 Hz), 128.8 (d, $J_{C-P}$ = 11.6 Hz), 115.4 (d, $J_{C-P}$ = 21.3 Hz), 32.0 (d, $J_{C-P}$ = 69.8 Hz), 26.8 (d, $J_{C-P}$ = 2.9 Hz); $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 31.43. This compound is known.\(^2\)

(4-chlorophenethyl)diphenylphosphine oxide (3e). Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/2) afforded 3e (60.7 mg, 89% yield) as a white solid; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.80–7.70 (m, 4H), 7.53–7.48 (m, 6H), 7.21–7.20 (m, 2H), 7.09–7.07 (m, 2H), 2.94–2.88 (m, 2H), 2.58–2.52 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 139.6 (d, $J_{C-P}$ = 15.0 Hz), 132.6 (d, $J_{C-P}$ = 98.4 Hz), 132.1, 131.9 (d, $J_{C-P}$ = 2.6 Hz), 130.8 (d, $J_{C-P}$ = 9.3 Hz), 129.5, 128.8 (d, $J_{C-P}$ = 11.6 Hz), 128.7, 31.8 (d, $J_{C-P}$ = 69.5 Hz), 27.0 (d, $J_{C-P}$ = 2.9 Hz); $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 31.30. This compound is known.\(^2\)

(3-bromophenethyl)diphenylphosphine oxide (3f). Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/2) afforded 3f (67.8 mg, 88% yield) as a white solid; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.78–7.74 (m, 4H), 7.55–7.46 (m, 6H), 7.29–7.27 (m, 2H), 7.13–7.08 (m, 2H), 2.94–2.88 (m, 2H), 2.58–2.52 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 143.4 (d, $J_{C-P}$ = 14.9 Hz), 132.6 (d, $J_{C-P}$ = 98.1 Hz), 132.1, 131.9 (d, $J_{C-P}$ = 2.7 Hz), 131.1, 130.8 (d, $J_{C-P}$ = 9.3 Hz), 130.2, 129.5, 128.8 (d, $J_{C-P}$ = 11.6 Hz), 126.9, 122.6, 31.6 (d, $J_{C-P}$ = 69.5 Hz), 27.3 (d, $J_{C-P}$ = 2.9 Hz); $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 31.20. This compound is known.

(2-(naphthalen-1-yl)ethyl)diphenylphosphine oxide (3g). Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/2) afforded 3g (64.9 mg, 91% yield) as a pale yellow solid; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.89–7.80 (m, 6H), 7.73–7.71 (m, 1H), 7.58–7.48 (m, 8H), 7.39–7.32 (m, 2H), 3.45–3.39 (m, 2H), 2.75–2.68 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 137.3 (d, $J_{C-P}$ = 14.6 Hz), 133.9, 132.8 (d, $J_{C-P}$ = 97.7 Hz), 131.9 (d, $J_{C-P}$ = 2.7 Hz), 131.3, 130.8 (d, $J_{C-P}$ = 9.3 Hz), 128.9, 128.7 (d, $J_{C-P}$ = 11.5 Hz), 127.2, 126.2,
125.8, 125.7, 125.6, 123.3, 31.2 (d, $J_{C,P} = 69.1$ Hz), 24.8 (d, $J_{C,P} = 2.8$ Hz); $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 31.69. This compound is known.$^1$

![3h](image)

**diphenyl(2-(thiophen-2-yl)ethyl)phosphine oxide (3h).** Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/2) afforded 3h (48.7 mg, 78% yield) as a white solid; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.79–7.74 (m, 4H), 7.54–7.49 (m, 6H), 7.11 (s, 1H), 6.88 (s, 1H), 6.79 (s, 1H), 3.18–3.13 (m, 2H), 2.89–2.63 (m, 2H);$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 143.9 (d, $J_{C,P} = 17.5$ Hz), 132.5 (d, $J = 99.3$ Hz), 132.0 (d, $J_{C,P} = 2.7$ Hz), 130.8 (d, $J_{C,P} = 9.3$ Hz), 128.8 (d, $J_{C,P} = 11.6$ Hz), 126.9, 124.6, 123.6, 32.2 (d, $J_{C,P} = 69.2$ Hz), 22.2 (d, $J_{C,P} = 2.1$ Hz);$^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 31.07. This compound is known.$^1$

![3i](image)

**diphenyl(2-(pyridin-2-yl)ethyl)phosphine oxide (3i).** Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/1) afforded 3i (40.0 mg, 65% yield) as a white solid; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.49–8.48 (m, 1H), 7.81–7.78 (m, 4H), 7.55–7.44 (m, 7H), 7.14–7.12 (m, 1H), 7.10–7.07 (m, 1H), 3.15–3.09 (m, 2H), 2.82–2.78 (m, 2H);$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 160.2 (d, $J_{C,P} = 14.4$ Hz), 149.3, 136.5, 132.8 (d, $J_{C,P} = 98.2$ Hz), 131.8 (d, $J = 2.7$ Hz), 130.8 (d, $J_{C,P} = 9.4$ Hz), 128.7 (d, $J_{C,P} = 11.7$ Hz), 123.11, 121.51, 29.7 (d, $J_{C,P} = 2.7$ Hz), 29.2 (d, $J_{C,P} = 71.2$ Hz);$^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 32.39. This compound is known.$^2$

![3j](image)

**diphenyl(2-phenylpropyl)phosphine oxide (3j).** Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/2) afforded 3j (48.7 mg, 76% yield) as a white solid; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.77–7.72 (m, 2H), 7.68–7.63 (m, 2H), 7.49–7.35 (m, 6H), 7.21–7.17 (m, 2H), 7.13–7.09 (m, 3H), 3.38–3.27 (m, 1H), 2.63–2.54 (m, 2H), 1.38–1.37 (m, 3H);$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 147.0 (d, $J_{C,P} = 10.2$ Hz), 134.4 (d, $J_{C,P} = 97.2$ Hz), 132.8 (d, $J_{C,P} = 97.6$ Hz), 131.6 (d, $J_{C,P} = 2.7$ Hz), 131.5 (d, $J_{C,P} = 2.7$ Hz), 130.8 (d, $J_{C,P} = 9.1$ Hz), 130.5 (d, $J_{C,P} = 9.2$ Hz), 128.6 (d, $J_{C,P} = 23.9$ Hz), 128.55 (d, $J_{C,P} = 1.6$ Hz), 128.5, 126.6, 126.4, 38.4 (d, $J_{C,P} = 69.0$ Hz), 34.3 (d, $J_{C,P} = 3.1$ Hz), 23.5 (d, $J_{C,P} = 5.2$ Hz);$^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 30.30. This compound is known.$^2$
**3k**

diphenyl(1-phenylpropan-2-yl)phosphine oxide (3k). Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/2) afforded 3k (36.5 mg, 57% yield) as a white solid; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 7.91–7.81 (m, 4H), 7.55–7.45 (m, 6H), 7.28–7.24 (m, 2H), 7.20–7.17 (m, 1H), 7.12–7.10 (m, 2H), 3.09–3.02 (m, 1H), 2.87–2.59 (m, 2H), 1.09–1.04 (m, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) 139.6 (d, \(J_{C-P} = 14.7\) Hz), 132.2 (d, \(J_{C-P} = 94.1\) Hz), 132.2 (d, \(J_{C-P} = 96.2\) Hz), 131.7 (d, \(J_{C-P} = 2.8\) Hz), 131.6 (d, \(J_{C-P} = 2.6\) Hz), 131.1, 131.0 (d, \(J_{C-P} = 8.6\) Hz), 129.0, 128.70, 128.7 (d, \(J_{C-P} = 23.5\) Hz), 128.5, 126.4, 35.0, 34.5 (d, \(J_{C-P} = 70.6\) Hz), 11.7 (d, \(J_{C-P} = 2.5\) Hz); \(^{31}\)P NMR (162 MHz, CDCl\(_3\)): \(\delta\) 36.16. This compound is known.\(^4\)

**3l**

(2,3-dihydro-1H-inden-2-yl)diphenylphosphine oxide(3l). Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/1) afforded 3l (45.2 mg, 71% yield) as a white solid; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 7.84–7.80 (m, 4H), 7.54–7.46 (m, 6H), 7.12 (s, 4H), 3.46–3.10 (m, 3H), 3.06–3.01 (m, 2H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) 141.7 (d, \(J_{C-P} = 9.9\) Hz), 132.8 (d, \(J_{C-P} = 96.8\) Hz), 131.8 (d, \(J_{C-P} = 2.6\) Hz), 131.0 (d, \(J_{C-P} = 8.9\) Hz), 128.7 (d, \(J_{C-P} = 11.3\) Hz), 126.7, 124.3, 37.8 (d, \(J_{C-P} = 75.7\) Hz), 33.1; \(^{31}\)P NMR (162 MHz, CDCl\(_3\)): \(\delta\) 33.38. Melting point: 170.8-176.3 °C. HRMS: Cal. for C\(_{21}\)H\(_{19}\)OP 318.1174. Found 318.1182. IR: 3021, 1435, 1183, 1118, 746, 722, 695 cm\(^{-1}\).

**3m**

(1,3-diphenylpropan-2-yl)diphenylphosphine oxide(3m). Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/1) afforded 3m (36.5 mg, 46% yield) as a white solid; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 7.79–7.74 (m, 4H), 7.43–7.34 (m, 6H), 7.05–7.04 (m, 6H), 6.81–6.79 (m, 4H), 3.10–3.02 (m, 2H), 2.93–2.79 (m, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) 139.7 (d, \(J_{C-P} = 9.1\) Hz), 132.6 (d, \(J_{C-P} = 93.9\) Hz), 131.4 (d, \(J_{C-P} = 2.6\) Hz), 130.8 (d, \(J_{C-P} = 8.7\) Hz), 129.0, 128.5 (d, \(J_{C-P} = 11.3\) Hz), 128.2, 126.1, 42.2 (d, \(J_{C-P} = 68.9\) Hz), 33.8; \(^{31}\)P NMR (162 MHz, CDCl\(_3\)): \(\delta\) 34.59. This compound is known.\(^7\)
(1,2-diphenylethyl)diphenylphosphine oxide (3n). Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/1) afforded 3n (24.7 mg, 32% yield) as a white solid; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.01–7.97 (m, 2H), 7.59 (s, 3H), 7.46–7.42 (m, 2H), 7.33–7.30 (m, 1H), 7.24–7.17 (m, 4H), 7.12–7.07 (m, 6H), 6.83–6.81 (m, 2H), 3.68–3.63 (m, 1H), 3.37–3.24 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 139.4 (d, $J_{C,P}$ = 14.1 Hz), 135.4 (d, $J_{C,P}$ = 5.3 Hz), 132.1 (d, $J_{C,P}$ = 98.7 Hz), 132.0 (d, $J_{C,P}$ = 94.1 Hz), 131.6 (d, $J_{C,P}$ = 59.1 Hz), 131.7 (d, $J_{C,P}$ = 59.1 Hz), 131.2 (d, $J_{C,P}$ = 94.1 Hz), 131.1 (d, $J_{C,P}$ = 40.1 Hz), 130.0 (d, $J_{C,P}$ = 5.9 Hz), 128.9 (d, $J_{C,P}$ = 11.2 Hz), 128.4 (d, $J_{C,P}$ = 53.9 Hz), 128.0 (d, $J_{C,P}$ = 11.6 Hz), 127.0 (d, $J_{C,P}$ = 2.4 Hz), 126.2, 49.2 (d, $J_{C,P}$ = 66.1 Hz), 36.0. $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 32.86. This compound is known.

ethylidiphenylphosphine oxide (3o). Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/1) afforded 3o (16.1 mg, 35% yield) as a white solid; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.76–7.72 (m, 4H), 7.54–7.45 (m, 6H), 2.33–2.24 (m, 2H), 1.26–1.24 (m, 1H), 1.20–1.16 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 132.9 (d, $J_{C,P}$ = 97.5 Hz), 131.7 (d, $J_{C,P}$ = 2.8 Hz), 130.9 (d, $J_{C,P}$ = 9.1 Hz), 128.6 (d, $J_{C,P}$ = 11.5 Hz), 22.7 (d, $J_{C,P}$ = 72.5 Hz), 5.6 (d, $J_{C,P}$ = 5.1 Hz); $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 34.06. This compound is known.

heptyldiphenylphosphine oxide (3p). Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/1) afforded 3p (22.8 mg, 38% yield) as a white solid; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.68–7.63 (m, 4H), 7.45–7.36 (m, 6H), 2.21–2.14 (m, 2H), 1.59–1.49 (m, 2H), 1.32–1.27 (m, 2H), 1.18–1.15 (m, 6H), 0.78–0.75 (m, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 133.2 (d, $J_{C,P}$ = 97.2 Hz), 131.6 (d, $J_{C,P}$ = 2.6 Hz), 130.8 (d, $J_{C,P}$ = 9.2 Hz), 128.6 (d, $J_{C,P}$ = 9.4 Hz), 31.6, 31.0 (d, $J_{C,P}$ = 14.6 Hz), 29.7 (d, $J_{C,P}$ = 76.9 Hz), 28.7, 22.6, 21.4 (d, $J_{C,P}$ = 3.8 Hz), 14.0; $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 32.66. This compound is known.
**phenethyldip-tolylphosphine oxide(3q).** Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/2) afforded 3q (50.8 mg, 76% yield) as a whitesolid; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.59–7.54 (m, 4H), 7.21–7.17 (m, 6H), 7.12–7.08 (m, 3H), 2.86–2.80 (m, 2H), 2.50–2.43 (m, 2H), 2.32 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$142.2 (d, $J_{C,P}$ = 2.6 Hz), 141.4 (d, $J_{C,P}$ = 15.2 Hz), 130.8 (d, $J_{C,P}$ = 9.6 Hz), 129.6 (d, $J_{C,P}$ = 100.2 Hz), 129.5 (d, $J_{C,P}$ = 12.0 Hz), 128.6, 128.1, 126.3, 32.1 (d, $J_{C,P}$ = 70.1 Hz), 27.6 (d, $J_{C,P}$ = 2.9 Hz), 21.6.; $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$32.01. This compound is known.$^1$

![3q](image)

**bis(4-(dimethylamino)phenyl)(phenethyl)phosphine oxide(3r).** Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/1) afforded 3r (65.9 mg, 84% yield) as a white solid; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.59–7.54 (m, 4H), 7.27–7.24 (m, 2H), 7.18–7.15 (m, 3H), 6.73–6.70 (m, 4H), 3.00 (s, 12H), 2.93–2.87 (m, 2H), 2.50–2.43 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 152.2 (d, $J_{C,P}$ = 2.3 Hz), 142.1 (d, $J_{C,P}$ = 15.5 Hz), 132.2 (d, $J_{C,P}$ = 10.4 Hz), 128.3 (d, $J_{C,P}$ = 39.0 Hz), 126.0, 118.9, 117.8, 111.5 (d, $J_{C,P}$ = 12.3 Hz), 40.0, 32.6 (d, $J_{C,P}$ = 70.8 Hz), 29.0 (d, $J_{C,P}$ = 2.7 Hz); $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 32.84. Melting point: 177.5-179.1 $^\circ$C. HRMS: Cal. for C$_{24}$H$_{29}$N$_2$OP 392.2018. Found 392.2025. IR: 2943, 2861, 1452, 1400, 1165, 1118, 817, 740 cm$^{-1}$.

![3r](image)

**dimesityl(phenethyl)phosphine oxide(3s).** Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/2) afforded 3s (55.5 mg, 71% yield) as a white solid; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.19–7.16 (m, 2H), 7.11–7.04 (m, 3H), 6.75 (s, 4H), 2.79–2.73 (m, 2H), 2.64–2.58 (m, 2H), 2.30 (s, 12H), 2.19 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 142.0 (d, $J_{C,P}$ = 16.8 Hz), 141.2 (d, $J_{C,P}$ = 10.0 Hz), 140.7 (d, $J_{C,P}$ = 2.6 Hz), 131.1 (d, $J_{C,P}$ = 11.0 Hz), 130.0 (d, $J_{C,P}$ = 94.1 Hz), 128.4 (d, $J_{C,P}$ = 28.2 Hz), 126.1, 38.2 (d, $J_{C,P}$ = 64.7 Hz), 28.8 (d, $J_{C,P}$ = 2.5 Hz), 22.9 (d, $J_{C,P}$ = 4.1 Hz), 20.9; $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 40.39. Melting point: 219.8-223.5 $^\circ$C. HRMS: Cal. for C$_{26}$H$_{31}$OP 390.2113. Found 390.2116. IR: 2918, 1400, 1200, 1169, 764, 735, 699, 646 cm$^{-1}$.

![3s](image)
di(naphthalen-2-yl)(phenethyl)phosphine oxide (3t). Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/2) afforded 3t (56.1 mg, 69% yield) as a white solid; \( ^1 \)H NMR (400 MHz, CDCl\(_3\)): \( \delta 8.37\text{–}8.34 \) (m, 2H), 7.81\text{–}7.79 (m, 4H), 7.74\text{–}7.72 (m, 2H), 7.64\text{–}7.59 (m, 2H), 7.47\text{–}7.40 (m, 2H), 7.08\text{–}7.02 (m, 3H), 2.93\text{–}2.87 (m, 2H), 2.70\text{–}2.63 (m, 2H); \( ^{13} \)C NMR (100 MHz, CDCl\(_3\)): \( \delta 141.2 \) (d, \( J_{C,P} = 15.2 \) Hz), 134.7 (d, \( J_{C,P} = 2.3 \) Hz), 132.9 (d, \( J_{C,P} = 8.5 \) Hz), 132.7 (d, \( J_{C,P} = 12.5 \) Hz), 129.9 (d, \( J_{C,P} = 98.2 \) Hz), 128.9, 128.8, 128.7, 128.3, 128.2, 127.9, 127.1, 126.4, 125.6 (d, \( J_{C,P} = 10.5 \) Hz), 31.8 (d, \( J_{C,P} = 69.5 \) Hz), 27.7 (d, \( J_{C,P} = 3.0 \) Hz); \( ^{31} \)P NMR (162 MHz, CDCl\(_3\)): \( \delta 31.99 \). Melting point: 233.4\text{–}236.1 °C. HRMS: Cal. for C\(_{28}\)H\(_{23}\)OP 406.1487. Found 406.1482. IR: 3094, 1452, 1440, 1167, 1094, 700, 653 cm\(^{-1}\).

dicyclohexyl(phenethyl)phosphine oxide (3u). Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (2/1) afforded 3u (38.2 mg, 60% yield) as a white solid; \( ^1 \)H NMR (400 MHz, CDCl\(_3\)): \( \delta 7.32\text{–}7.28 \) (m, 2H), 7.23\text{–}7.19 (m, 3H), 1.98\text{–}1.93 (m, 5H), 1.85\text{–}1.75 (m, 9H), 1.42\text{–}1.39 (m, 4H), 1.28\text{–}1.24 (m, 6H); \( ^{13} \)C NMR (100 MHz, CDCl\(_3\)): \( \delta 141.8 \) (d, \( J_{C,P} = 12.5 \) Hz), 128.6, 128.0, 126.3, 36.4 (d, \( J_{C,P} = 63.8 \) Hz), 28.1 (d, \( J_{C,P} = 3.6 \) Hz), 26.6 (dd, \( J_{C,P} = 12.1, 2.9 \) Hz), 26.0, 25.7 (d, \( J_{C,P} = 3.1 \) Hz), 25.6 (d, \( J_{C,P} = 58.4 \) Hz); \( ^{31} \)P NMR (162 MHz, CDCl\(_3\)): \( \delta 50.24 \). This compound is known.\(^6\)

dibutyl(phenethyl)phosphine oxide (3v). Purification by column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (2/1) afforded 3v (34.6 mg, 65% yield) as a colorless oil; \( ^1 \)H NMR (400 MHz, CDCl\(_3\)): \( \delta 7.32\text{–}7.28 \) (m, 2H), 7.24\text{–}7.20 (m, 3H), 2.05\text{–}1.99 (m, 2H), 1.75\text{–}1.68 (m, 4H), 1.60\text{–}1.50 (m, 4H), 1.46\text{–}1.37 (m, 4H), 0.946\text{–}0.910 (m, 6H); \( ^{13} \)C NMR (100 MHz, CDCl\(_3\)): \( \delta 141.2 \) (d, \( J_{C,P} = 13.2 \) Hz), 128.7, 128.1, 126.4, 29.7 (d, \( J_{C,P} = 62.6 \) Hz), 27.8 (d, \( J_{C,P} = 64.5 \) Hz), 27.7 (d, \( J_{C,P} = 3.1 \) Hz), 24.3 (d, \( J_{C,P} = 14.3 \) Hz), 23.8 (d, \( J_{C,P} = 3.7 \) Hz), 13.6; \( ^{31} \)P NMR (162 MHz, CDCl\(_3\)): \( \delta 48.17 \). This compound is known.\(^1\)
4. References:


5. Copies of $^1$H NMR, $^{31}$P NMR and $^{13}$C NMR spectroscopies

[Image of NMR spectra]

$3a$