Supporting Information

**Ru**(II)-catalyzed **P**(III)-assisted C8-alkylation of naphthphosphines

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1. General Information

Unless otherwise noted, all reactions were carried out in a flamedried, sealed Schlenk reaction tube under an atmosphere of argon. Unless otherwise noted, materials were purchased from Alfa-Aesar and J&K Scientific Ltd. and used as received. All the solvents were purchased from commercial suppliers and purified by standard procedures as specified in Purification of Laboratory Chemicals, 4th Ed (W. L. F. Armarego, D. D. Perrin, Butterworth-Heinemann: 1997). Analytical thin layer chromatography (TLC) was performed on silica gel plates with F-254 indicator and compounds were visualized by irradiation with UV light. Flash column chromatography was carried out using silica gel (200-300 mesh) at increased pressure. The $^1$H, $^{13}$C, $^{31}$P and $^{19}$F NMR spectroscopic data were recorded on Bruker Mercury Plus 400 MHz NMR spectrometers. Chemical shifts (δ) for $^1$H and $^{13}$C are referenced to internal solvent resonances and reported relative to SiMe₄. Chemical shifts for $^{31}$P are reported relative to an external 85% H₃PO₄ standard. $^1$H NMR coupling constants were reported in Hz, and multiplicity was indicated as follows: s (singlet); d (doublet); t (triplet); q (quartet); m (multiplet); dd (doublet of doublets). The diffraction data of crystals were collected on a Rigaku XtaLAB Synergy CCD diffractometer with graphite monochromated Cu-Kα radiation ($\lambda = 1.54056$ Å) at 293 or 100 K. Absorption corrections were applied by SADABS. All the structures were solved by direct methods and refined by full-matrix least-squares method on $F^2$ using SHELXTL-2014. All non-hydrogen atoms were refined with anisotropic displacement parameters. The hydrogen atoms of the ligand were generated geometrically. High resolution mass spectra (HRMS) were recorded on the Thermo Scientific Exactive Plus equipped with ESI ionization source.
2. Experimental Procedures

2.1 Optimization of the reaction conditions

Table 1 Reaction conditions

<table>
<thead>
<tr>
<th>Entry</th>
<th>Change from standard conditions</th>
<th>Yield</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>none</td>
<td>87%\textsuperscript{b}</td>
</tr>
<tr>
<td>2</td>
<td>Add NaOAc 2 eq.</td>
<td>68%</td>
</tr>
<tr>
<td>3</td>
<td>MesCO\textsubscript{2}H instead of 1-AdCO\textsubscript{2}H</td>
<td>48%</td>
</tr>
<tr>
<td>4</td>
<td>toluene instead of EtOH</td>
<td>11%</td>
</tr>
<tr>
<td>5</td>
<td>1,4-dioxane instead of EtOH</td>
<td>5%</td>
</tr>
<tr>
<td>6</td>
<td>without 1-Ad-CO\textsubscript{2}H</td>
<td>0%</td>
</tr>
<tr>
<td>7</td>
<td>without [RuCl\textsubscript{2}(\textit{p}-cymene)]\textsubscript{2}</td>
<td>0%</td>
</tr>
<tr>
<td>8</td>
<td>80°C instead of 100 °C</td>
<td>64%</td>
</tr>
</tbody>
</table>

\textsuperscript{a}Reaction conditions: 1a (0.1 mmol), 2a (0.4 mmol), [RuCl\textsubscript{2}(\textit{p}-cymene)]\textsubscript{2} (5 mol\%) and 1-Ad-CO\textsubscript{2}H (30 mol\%) in 1 mL EtOH at 100 °C under argon, 12 h. Yields determined by \textsuperscript{31}P NMR using triphenylphosphine as an internal standard. \textsuperscript{b}Isolated yield.

2.2 General procedure for Ru(II)-catalyzed direct alkylation

Scheme 1
To a 25 mL Schlenk tube was added 1-naphthalenylidiphenylphosphane 1 (0.2 mmol, 1.0 equiv), alkene 2 (0.8 mmol, 4.0 equiv), [RuCl\textsubscript{2}(\textit{p}-cymene)]\textsubscript{2} (6.1 mg 5.0 mol\%), 1-Ad-COOH (10.8 mg, 30 mol\%). The tube was purged with Ar three times, followed by addition of EtOH (1 mL). The mixture was stirred at 100 °C for 12 h. The solution was then cooled to room temperature and the solvent was removed under
vacuum directly. The crude product was purified by prepared column chromatography affording the alkylation product 3.

2.3 General procedures for Synthetic Applications

a) Gram-scale reaction

Scheme 2
To a 50 mL Schlenk tube was added 1-naphthalenylidiphenylphosphane 1a (3 mmol, 1.0 equiv), Ethyl Acrylate 2a (12 mmol, 4.0 equiv), [RuCl₂(p-cymene)]₂ (91.8 mg, 5.0 mol%), 1-Ad-COOH (162 mg, 30 mol%). The tube was purged with Ar three times, followed by addition of EtOH (5 mL). The mixture was stirred at 100 °C for 24 h. The solution was then cooled to room temperature and the solvent was removed under vacuum directly. The crude product was purified by column chromatography on silica gel (Rₐ = 0.4, EA/hexane = 1/25) affording the alkylation product 3aa (white solid, 1.05 g, 85% yield).

Scheme 3
To a 50 mL Schlenk tube was added 1-naphthalenylidiphenylphosphane 1a (3 mmol, 1.0 equiv), Ethyl Acrylate 2a (12 mmol, 4.0 equiv), [RuCl₂(p-cymene)]₂ (91.8 mg, 5.0 mol%), 1-Ad-COOH (162 mg, 30 mol%). The tube was purged with Ar three times, followed by addition of EtOH (5 mL). The mixture was stirred at 100 °C for 24 h. The solution was then cooled to room temperature and the solvent was removed under vacuum directly. The crude product was purified by column chromatography on silica gel (Rₐ = 0.4, EA/hexane = 1/25) affording the alkylation product 3aa (white solid, 1.05 g, 85% yield).

Scheme 4
To a 50 mL Schlenk tube was added 1-naphthalenylidiphenylphosphane 1a (3 mmol, 1.0 equiv), Ethyl Acrylate 2a (12 mmol, 4.0 equiv), [RuCl$_2$(p-cymene)]$_2$ (91.8 mg, 5.0 mol%), 1-Ad-COOH (162 mg, 30 mol%). The tube was purged with Ar three times, followed by addition of EtOH (5 mL). The mixture was stirred at 100 °C for 24 h. The solution was then cooled to room temperature and the solvent was removed under vacuum directly. The crude product was purified by column chromatography on silica gel ($R_f = 0.4$, EA/hexane = 1/25) affording the alkylation product 3aa (white solid, 1.05 g, 85% yield).

### 2.4 General procedures for Phosphine-Promoted Pd-catalyzed Suzuki-Miyaura Coupling

**Scheme 5**

First, catalyst stock solutions were prepared by dissolving Pd(OAc)$_2$ (2.3 mg) and 3aa (20.6 mg) in DCM (10 mL) under nitrogen atmosphere at room temperature with continuous stirring for 5 min. Add 0.1 mL of the stock solution to a 25 mL Schlenk tube and remove the solvent under vacuum. Aryl bromide 6 (1 mmol, 1.0 equiv), n-butylboronic acid 7a (1.3 mmol, 1.3 equiv), K$_2$CO$_3$ (276.0 mg, 2 equiv) were added to a Schlenk tube. The tube was then purged 3 times with Ar followed by addition of toluene and water (10:1, 2 mL). The mixture was stirred at 100 °C for 16 h. The solution was then cooled to room temperature and the solvent was removed under vacuum directly.

### 2.5 General procedures for mechanistic experiments

a) Synthesis of ruthenium(II) Int A

**Scheme 6**

To a 50 mL Schlenk flask was added 1-naphthalenylidiphenylphosphane 1a (2 mmol, 1.0 equiv), [RuCl$_2$(p-cymene)]$_2$ (1 mmol, 50 mol%), NaOAc (4 mmol, 2.0 equiv). The tube was purged with Ar three times, followed by addition of 10 mL DCM. The mixture was stirred at room temperature for 24 h. The solvent was then removed under vacuum directly. The crude product was purified by neutral alumina
(Al₂O₃) column chromatography (R_f = 0.6, EA/hexane = 5/1) affording the product **Int A** (0.81g, 68% yield) as red powder. \(^1\)H NMR (400 MHz, CDCl₃) δ 8.32-8.30 (m, 1H), 8.13-8.07 (m, 2H), 7.87-7.84 (m, 1H), 7.67-7.63 (m, 1H), 7.49-7.46 (m, 1H), 7.45-7.40 (m, 4H), 7.37-7.29 (m, 4H), 7.21-7.16 (m, 2H), 5.82 (t, \(J = 6.9\) Hz, 2H), 4.56 (d, \(J = 6.2\) Hz, 1H), 4.46 (d, \(J = 6.1\) Hz, 1H), 2.45-2.38 (m, 1H), 1.93 (s, 3H), 1.26 (s, 2H), 1.05 (d, \(J = 6.9\) Hz, 3H), 0.74 (d, \(J = 7.0\) Hz, 3H).

**b) Synthesis of ruthenium(II) Int B**

\[
\begin{align*}
&\text{PCy}_2H \\
&\text{1o} \\
&\xrightarrow{[\text{RuCl}_2(\mu-\text{cymene})]_2, \text{NaOAc, DCM, rt}} \\
&\text{Int B}
\end{align*}
\]

**Scheme 7**

To a 50 mL Schlenk flask was added dicyclohexyl(naphthalen-1-yl)phosphane 1o (1 mmol, 1.0 equiv), [RuCl₂(μ-cymene)]₂ (0.5 mmol, 50 mol%), NaOAc (2 mmol, 2.0 equiv). The tube was purged with Ar three times, followed by addition of 10 mL DCM. The mixture was stirred at room temperature for 24 h. The solvent was then removed under vacuum directly. The crude product was purified by neutral alumina (Al₂O₃) column chromatography (R_f = 0.4, EA/hexane = 5/1) affording the product **Int B** (0.45g, 74% yield) as red powder.

\(^1\)H NMR (400 MHz, CDCl₃) δ 170.8 (d, \(J = 12.5\) Hz), 149.4 (d, \(J = 31.1\) Hz), 135.4 (d, \(J = 42.9\) Hz), 133.1 (d, \(J = 13.9\) Hz), 130.2 (d, \(J = 2.4\) Hz), 126.9 (d, \(J = 11.0\) Hz), 123.6 (d, \(J = 7.1\) Hz), 121.2, 110.6, 96.6 (d, \(J = 5.8\) Hz), 95.3, 90.3 (d, \(J = 3.2\) Hz), 89.6, 83.8, 39.3 (d, \(J = 24.0\) Hz), 35.3 (d, \(J = 23.8\) Hz), 30.5, 29.7, 28.9 (d, \(J = 2.8\) Hz), 27.7, 27.6 (d, \(J = 2.6\) Hz), 27.5, 27.2 (d, \(J = 9.0\) Hz), 27.0 (d, \(J = 7.6\) Hz), 26.9 (d, \(J = 8.0\) Hz), 26.2, 23.0, 22.2, 18.0. \(^{31}\)P NMR (162 MHz, CDCl₃) δ 71.52.

c) Cyclometalated complex **Int A** as substrate

\[
\begin{align*}
&\text{Ph}_2\text{P---Ru-Cl} \\
&\text{Int A} \\
&\xrightarrow{2a \text{ 4 eq., } 1a \text{ 1 eq.}} \\
&\text{3aa 47%}
\end{align*}
\]

**Scheme 8**

To a 25 mL Schlenk tube was added Int A (0.1 mmol, 1.0 equiv), naphthalen-1-ylidiphenylphosphane 1a, ethyl acrylate 2a (0.4 mmol, 4.0 equiv), 1-Ad-COOH (54 mg, 3.0 equiv). The tube was purged with Ar three times, followed by addition of EtOH (1 mL). The mixture was stirred at 100 °C for 12 h. The
solution was then cooled to room temperature and the solvent was removed under vacuum directly. The crude product was purified by prepared column chromatography ($R_f = 0.4$, EA/hexane = 1/25) affording the alkylation product 3aa (47% yield).

d) Cyclometalated complex Int B as substrate

![Scheme 9]

To a 25 mL Schlenk tube was added Int B (0.1 mmol, 1.0 equiv), dicyclohexyl(naphthalen-1-yl)phosphane 1o, Ethyl Acrylate 2a (0.4 mmol, 4.0 equiv), 1-Ad-COOH (54 mg, 3.0 equiv). The tube was purged with Ar three times, followed by addition of EtOH (1 mL). The mixture was stirred at 100 °C for 12 h. The solution was then cooled to room temperature and the solvent was removed under vacuum directly. Failed to generate the alkylated product 3oa.

e) Intermolecular H/D exchange

![Scheme 10]

To a 25 mL Schlenk tube was added (4-methoxynaphthalen-1-yl)diphenylphosphane 1e (0.2 mmol, 1.0 equiv), [RuCl$_2$(p-cymene)]$_2$ (5.0 mol%), 1-Ad-COOH (10.8 mg, 30 mol%), CD$_3$OD (10.0 equiv). The tube was purged with Ar three times, followed by addition of EtOH (1.0 mL). The mixture was stirred at 100 °C for 12 h. The solution was then cooled to room temperature and the solvent was removed under vacuum directly. The crude product was purified by prepared column chromatography ($R_f = 0.7$, EA/hexane = 1/50) affording the mixture products, which was analyzed by $^1$H NMR spectroscopy.
f) Parallel experiments KIE measurement

Kinetic isotopic effect was determined by performing two independent experiments of naphthalen-1-yldiphenylphosphane 1a or deuterated naphthalen-1-yldiphenylphosphane 1a-d8 and Ethyl Acrylate 2a as a coupling partner. Yields determined by 1H NMR using CH$_2$Br$_2$ as the internal standard were recorded at predetermined time intervals for 2 h to evaluate the yield of the reaction. The yield of alkylated product using starting material 1a was 37%, The yield of alkylated product using starting material 1a-d8 was 33%. The kH/kD ratio of 1.1 was obtained.

3. Reference


4. Characterization Data

**ethyl 3-(8-(diphenylphosphaneyl)naphthalen-1-yl)propanoate (3aa):** The representative procedure 2.1 was followed, using 1-naphthalenyldiphenylphosphane 1a (62.4 mg, 0.2 mmol) and Ethyl Acrylate 2a (0.8 mmol, 4.0 equiv). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3aa (71.7 mg, 87%) as a white solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.83 (dd, \(J = 8.0, 1.6\) Hz, 1H), 7.78-7.71 (m, 1H), 7.41-7.35 (m, 2H), 7.32-7.28 (m, 7H), 7.25-7.19 (m, 5H), 4.11 (q, \(J = 7.2\) Hz, 2H), 3.82-3.73 (m, 2H), 2.85-2.78 (m, 2H), 1.21 (t, \(J = 7.2\) Hz, 3H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 173.1, 138.6 (d, \(J = 1.9\) Hz), 137.9 (d, \(J = 12.8\) Hz), 136.3, 135.6 (d, \(J = 5.5\) Hz), 134.9 (d, \(J = 20.3\) Hz), 134.0 (d, \(J = 20.3\) Hz), 133.7 (d, \(J = 28.6\) Hz), 131.4, 128.9 (d, \(J = 1.8\) Hz), 128.6, 128.6 (d, \(J = 10.6\) Hz), 125.4, 124.7, 60.1, 37.6 (d, \(J = 15.6\) Hz), 32.9 (d, \(J = 28.2\) Hz), 14.2. \(^{31}\)P NMR (162 MHz, CDCl\(_3\)) \(\delta\) -2.50. HRMS (ESI+) exact mass calculated for [M+H]\(^+\) (C\(_{27}\)H\(_{26}\)O\(_2\)P): 413.1665, found: 413.1665.

**ethyl 3-(8-(diphenylphosphaneyl)-7-methylnaphthalen-1-yl)propanoate (3ba):** The representative procedure 2.1 was followed, using (2-methylnaphthalen-1-yl)diphenylphosphane 1b (85.2 mg, 0.2 mmol) and Ethyl acrylate 2a (80.1 mg, 0.8 mmol). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3ba (31.5 mg, 37%) as a white solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.79 (d, \(J = 8.0\) Hz, 1H), 7.72-7.68 (m, 1H), 7.43-7.33 (m, 6H), 7.32-7.26 (m, 5H), 7.25-7.22 (m, 1H), 7.19 (d, \(J = 8.0\) Hz, 1H), 3.94 (q, \(J = 7.2\) Hz, 2H), 3.60-3.47 (m, 2H), 2.86-2.76 (m, 2H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 173.0, 146.6 (d, \(J = 3.1\) Hz), 139.5 (d, \(J = 32.1\) Hz), 138.7 (d, \(J = 3.4\) Hz), 137.1 (d, \(J = 16.6\) Hz), 133.3, 133.2, 131.9 (d, \(J = 1.8\) Hz), 131.5 (d, \(J = 18.1\) Hz), 130.0, 129.7 (d, \(J = 1.5\) Hz), 128.4, 128.3 (d, \(J = 5.0\) Hz), 128.2, 127.7 (d, \(J = 1.6\) Hz), 127.4, 124.8, 60.0, 37.5 (d, \(J = 14.2\) Hz), 32.9 (d, \(J = 29.1\) Hz), 24.9 (d, \(J = 1.9\) Hz), 14.1. \(^{31}\)P NMR (162 MHz, CDCl\(_3\)) \(\delta\) -5.95. HRMS (ESI+) exact mass calculated for [M+H]\(^+\) (C\(_{28}\)H\(_{28}\)O\(_2\)P): 427.1821, found: 427.1821.
ethyl 3-(8-(diphenylphosphaneyl)-5-methylnaphthalen-1-yl)propanoate (3da): The representative procedure 2.1 was followed, using (4-methylnaphthalen-1-yl)diphenylphosphane 1d (85.2 mg, 0.2 mmol) and Ethyl acrylate 2a (80.1 mg, 0.8 mmol). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3da (49.1 mg, 58%) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.99 (dt, $J = 8.0, 1.6$ Hz, 1H), 7.48-7.37 (m, 2H), 7.34-7.27 (m, 6H), 7.27-7.18 (m, 6H), 7.20-7.08 (m, 2H), 4.11 (q, $J = 7.2$ Hz, 2H), 3.83-3.72 (m, 2H), 2.86-2.77 (m, 2H), 2.69 (s, 3H), 1.22 (t, $J = 7.2$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 173.1, 139.2 (d, $J = 2.1$ Hz), 138.2 (d, $J = 13.1$ Hz), 136.9, 136.2, 135.1 (d, $J = 20.4$ Hz), 134.6 (d, $J = 5.5$ Hz), 133.9 (d, $J = 20.2$ Hz), 131.4 (d, $J = 27.6$ Hz), 130.0, 128.5, 128.5 (d, $J = 6.8$ Hz), 126.2, 125.3, 124.2, 60.2, 37.6 (d, $J = 16.0$ Hz), 33.1 (d, $J = 29.1$ Hz), 20.8, 14.3. $^{31}$P NMR (162 MHz, CDCl$_3$) δ -2.19. HRMS (ESI+) exact mass calculated for [M+H]$^+$ (C$_{28}$H$_{28}$O$_3$P): 427.1821, found: 427.1820.

![Image of ethyl 3-(8-(diphenylphosphaneyl)-5-methylnaphthalen-1-yl)propanoate (3da)](image_url)

ethyl 3-(8-(diphenylphosphaneyl)-5-methoxynaphthalen-1-yl)propanoate (3ea): The representative procedure 2.1 was followed, using (4-methoxynaphthalen-1-yl)diphenylphosphane 1e (88.4 mg, 0.2 mmol) and Ethyl acrylate 2a (80.1 mg, 0.8 mmol). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3ea (61.9 mg, 70 %) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.38-8.27 (m, 1H), 7.42-7.37 (m, 2H), 7.35-7.27 (m, 6H), 7.26-7.21 (m, 4H), 7.15 (dd, $J = 8.0, 4.8$ Hz, 1H), 6.70 (d, $J = 8.4$ Hz, 1H), 4.11 (q, $J = 7.2$ Hz, 2H), 3.97 (s, 3H), 3.83-3.72 (m, 2H), 2.87-2.75 (m, 2H), 1.21 (t, $J = 7.2$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 173.1, 156.9, 138.5 (d, $J = 13.4$ Hz), 138.4, 137.1, 135.8, 133.8 (d, $J = 20.2$ Hz), 130.8, 128.5 (d, $J = 8.8$ Hz), 128.4, 127.6 (d, $J = 6.0$ Hz), 124.8, 124.1, 123.8, 121.7, 103.4, 60.1, 55.5, 37.6 (d, $J = 16.0$ Hz), 32.9 (d, $J = 29.0$ Hz), 29.7, 14.3. $^{31}$P NMR (162 MHz, CDCl$_3$) δ -3.47. HRMS (ESI+) exact mass calculated for [M+H]$^+$ (C$_{28}$H$_{28}$O$_3$P): 443.1771, found: 443.1771.

![Image of ethyl 3-(8-(diphenylphosphaneyl)-5-methoxynaphthalen-1-yl)propanoate (3ea)](image_url)

ethy 3-(8-(diphenylphosphaneyl)-5-fluoronaphthalen-1-yl)propanoate (3fa): The representative procedure 2.1 was followed, using (4-fluoronaphthalen-1-yl)diphenylphosphane 1f (66.0 mg, 0.2 mmol) and Ethyl acrylate 2a (80.1 mg, 0.8 mmol). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3fa (38.7 mg, 45 %) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.12 (d, $J = 8.0$ Hz, 1H), 7.50-7.41 (m, 2H), 7.35-7.29 (m, 6H), 7.25-7.13 (m, 5H), 7.02-6.97 (m, 1H), 4.12 (q, $J = 7.2$ Hz, 2H), 3.83-3.72 (m, 2H), 2.86-2.76 (m, 2H), 1.22 (t, $J = 7.2$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 172.9,
161.4, 158.8, 138.8, 138.8 (d, \( J = 4.9 \) Hz), 137.7 (d, \( J = 13.0 \) Hz), 136.4 (dd), 136.2 (d, \( J = 8.9 \) Hz), 133.9 (d, \( J = 20.4 \) Hz), 131.2, 129.3 (dd, \( J = 28.7, 4.6 \) Hz), 128.7 (d, \( J = 14.3 \) Hz), 128.7, 125.8 (d, \( J = 2.2 \) Hz), 125.7 (d, \( J = 5.9 \) Hz), 120.4 (d, \( J = 9.5 \) Hz), 108.6 (d, \( J = 19.6 \) Hz), 60.2, 37.5 (d, \( J = 15.7 \) Hz), 32.8 (d, \( J = 28.7 \) Hz), 14.3.\(^{31}\)P NMR (162 MHz, CDCl\(_3\)) \( \delta -3.49. \) \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \( \delta -118.36. \) HRMS (ESI+) exact mass calculated for [M+H]+ (C\(_{27}\)H\(_{25}\)FO\(_2\)P): 431.1571, found: 431.1571.

\[ \text{methyl 4-}(\text{diphenylphosphaneyl})-5-(\text{3-ethoxy-3-oxopropyl})-1-naphthoate (3ga): } \]

The representative procedure 2.1 was followed, using methyl 4-(diphenylphosphaneyl)-1-naphthoate 1g (74.0 mg, 0.2 mmol) and Ethyl acrylate 2a (80.1 mg, 0.8 mmol). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3ga (56.4 mg, 60 %) as a white solid.\(^{1}\)H NMR (400 MHz, CDCl\(_3\)) \( \delta 8.64 (dt, \( J = 8.8, 1.6 \) Hz, 1H), 7.78 (d, \( J = 7.6 \) Hz, 1H), 7.51-7.46 (m, 1H), 7.41-7.38 (m, 1H), 7.37-7.28 (m, 6H), 7.24-7.16 (m, 5H), 4.10 (q, \( J = 7.2 \) Hz, 2H), 3.98 (s, 3H), 3.83-3.74 (m, 2H), 2.81-2.72 (m, 2H), 1.21 (t, \( J = 7.2 \) Hz, 3H).\(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \( \delta 172.9, 168.7, 140.0, 139.7, 138.9 (d, \( J = 2.1 \) Hz), 137.2 (d, \( J = 12.6 \) Hz), 135.2 (d, \( J = 18.7 \) Hz), 134.5, 134.1 (d, \( J = 20.5 \) Hz), 132.6 (d, \( J = 4.6 \) Hz), 130.5, 130.3, 129.0, 128.7 (d, \( J = 7.0 \) Hz), 127.2, 126.8, 125.5, 60.2, 52.4, 37.4 (d, \( J = 14.9 \) Hz), 33.2 (d, \( J = 29.0 \) Hz), 29.7, 14.2.\(^{31}\)P NMR (162 MHz, CDCl\(_3\)) \( \delta -0.54. \) HRMS (ESI+) exact mass calculated for [M+H]+ (C\(_{29}\)H\(_{28}\)O\(_4\)P): 471.1720, found: 471.1720.

\[ \text{ethyl 2-}(\text{5-}(\text{diphenylphosphaneyl})-4-(\text{3-ethoxy-3-oxopropyl})\text{naphthalen-1-yl})-2\text{-methylpropanoate (3ha): } \]

The representative procedure 2.1 was followed, using ethyl 2-(4-(diphenylphosphaneyl)naphthalen-1-yl)-2-methylpropanoate 1h (85.2 mg, 0.2 mmol) and Ethyl acrylate 2a (80.1 mg, 0.8 mmol). Purification by prepared column chromatography (EA/hexane = 1/10) yielded 3ha (56.8 mg, 54 %) as a white solid.\(^{1}\)H NMR (400 MHz, CDCl\(_3\)) \( \delta 7.88 (dd, \( J = 8.0, 1.6 \) Hz, 1H), 7.46 (d, \( J = 8.0 \) Hz, 1H), 7.36-7.27 (m, 7H), 7.26-7.18 (m, 6H), 4.14-4.06 (m, 2H), 3.82-3.73 (m, 2H), 2.88-2.79 (m, 1H), 1.75 (s, 6H), 1.21 (t, \( J = 7.2 \) Hz, 3H), 1.03 (t, \( J = 7.2 \) Hz, 3H).\(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \( \delta 178.9, 173.1, 140.6 (d, \( J = 1.8 \) Hz), 138.1 (d, \( J = 13.3 \) Hz), 137.9, 136.2 (d, \( J = 20.5 \) Hz), 135.8, 134.6 (d, \( J = 29.0 \) Hz), 133.9 (d, \( J = 20.3 \) Hz), 133.0 (d, \( J = 5.5 \) Hz), 129.5, 128.6, 128.5 (d, \( J = 10.2 \) Hz), 126.7, 124.3, 122.8, 60.9, 60.2, 46.5, 37.3 (d, \( J = 15.8 \) Hz), 33.1 (d, \( J = 30.0 \) Hz), 27.9, 14.2, 13.9.\(^{31}\)P NMR (162
ethyl 3-(8-(diphenylphosphaneyl)-4-(2-methyl-1-morpholo-1-oxopropan-2-yl)naphthalen-1-yl)propanoate (3ia): The representative procedure 2.1 was followed, using 2-(5-(diphenylphosphaneyl)naphthalen-1-yl)-2-methyl-1-morpholinopropan-1-one 1i (93.4 mg, 0.2 mmol) and Ethyl acrylate 2a (80.1 mg, 0.8 mmol). Purification by prepared column chromatography (EA/hexane = 1/5) yielded 3ia (59.1 mg, 52 %) as a white solid. ^1H NMR (400 MHz, CDCl$_3$) δ 8.09 (dd, J = 8.4, 1.6 Hz, 1H), 7.46-7.41 (m, 1H), 7.36-7.23 (m, 11H), 7.19-7.09 (m, 2H), 4.10 (q, J = 7.1 Hz, 2H), 4.02-3.85 (m, 2H), 3.74-3.53 (m, 2H), 3.38-3.20 (m, 2H), 3.01-2.64 (m, 5H), 2.23-2.08 (m, 1H), 1.74 (s, 6H), 1.21 (t, J = 7.1 Hz, 3H). ^13C NMR (101 MHz, CDCl$_3$) δ 176.1, 172.9, 142.2 (d, J = 1.6 Hz), 138.1 (d, J = 1.6 Hz), 137.8 (d, J = 12.9 Hz), 137.5 (d, J = 13.5 Hz), 136.0, 135.9, 135.7, 135.2, 134.9, 134.2, 134.0 (d, J = 6.6 Hz), 133.7, 132.7 (d, J = 5.0 Hz), 130.1, 128.8, 128.6, 128.6 (d, J = 15.5 Hz), 127.2, 124.5, 121.3, 66.8, 65.3, 60.2, 46.7, 43.2, 37.2 (d, J = 15.4 Hz), 33.2 (d, J = 30.4 Hz), 30.6, 26.8, 14.2. ^31P NMR (162 MHz, CDCl$_3$) δ -1.06. HRMS (ESI+) exact mass calculated for [M+H]$^+$ (C$_{33}$H$_{36}$O$_4$P): 527.2346, found: 527.2347.

ethyl 3-(6-(diphenylphosphaneyl)-1,2-dihydroacenaphthylen-5-yl)propanoate (3ja): The representative procedure 2.1 was followed, using (1,2-dihydroacenaphthylen-5-yl)diphenylphospane 1j (67.6 mg, 0.2 mmol) and Ethyl acrylate 2a (80.1 mg, 0.8 mmol). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3ja (52.6 mg, 60 %) as a white solid. ^1H NMR (400 MHz, CDCl$_3$) δ 7.34-7.28 (m, 7H), 7.27-7.21 (m, 5H), 7.15-7.11 (m, 2H), 4.12 (q, J = 7.2 Hz, 2H), 3.72-3.63 (m, 2H), 3.35 (s, 4H), 2.82-2.73 (m, 2H), 1.23 (t, J = 7.2 Hz, 3H). ^13C NMR (101 MHz, CDCl$_3$) δ 173.2, 148.9, 145.6 (d, J = 2.0 Hz), 140.6 (d, J = 6.9 Hz), 138.1 (d, J = 12.4 Hz), 137.4, 134.7 (d, J = 2.0 Hz), 133.9 (d, J = 20.0 Hz), 133.5 (d, J = 22.3 Hz), 131.4, 128.8 (d, J = 25.4 Hz), 128.5, 128.5, 119.7, 119.3, 60.1, 37.7 (d, J = 14.5 Hz), 31.6 (d, J = 24.5 Hz), 29.9 (d, J = 34.2 Hz), 14.3. ^31P NMR (162 MHz, CDCl$_3$) δ -6.61. HRMS (ESI+) exact mass calculated for [M+H]$^+$ (C$_{29}$H$_{32}$O$_2$P): 439.1821, found: 439.1823.
ethyl 3-(10-(diphenylphosphaneyl)phenanthren-1-yl)propanoate (3ka): The representative procedure 2.1 was followed, using (4b,5-dihydrophenanthren-9-yl)diphenylphosphane 1k (72.8 mg, 0.2 mmol) and Ethyl acrylate 2a (80.1 mg, 0.8 mmol). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3ka (73.9 mg, 80 %) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.72 (d, $J = 8.4$ Hz, 1H), 8.66 (d, $J = 8.4$ Hz, 1H), 7.66-7.61 (m, 1H), 7.59-7.54 (m, 1H), 7.53-7.44 (m, 4H), 7.40-7.26 (m, 10H), 4.11 (q, $J = 7.2$ Hz, 2H), 3.89-3.72 (m, 2H), 2.90-2.73 (m, 2H), 1.22 (t, $J = 7.2$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 173.0, 139.4 (d, $J = 2.0$ Hz), 138.2, 137.6 (d, $J = 12.7$ Hz), 134.1 (d, $J = 20.4$ Hz), 132.8 (d, $J = 19.2$ Hz), 132.4 (d, $J = 29.6$ Hz), 132.0 (d, $J = 5.2$ Hz), 131.2, 130.8, 130.4, 128.8, 128.6 (d, $J = 6.9$ Hz), 128.5, 127.6, 126.6, 126.1, 122.8, 122.2 (d, $J = 1.6$ Hz), 60.2, 37.7 (d, $J = 14.9$ Hz), 33.1 (d, $J = 28.7$ Hz), 14.2. $^{31}$P NMR (162 MHz, CDCl$_3$) δ -0.79. HRMS (ESI+) exact mass calculated for [M+H]$^+$ (C$_{31}$H$_{28}$O$_2$P): 463.1821, found: 463.1822.

ethyl 3-(10-(diphenylphosphaneyl)pyren-1-yl)propanoate (3la): The representative procedure 2.1 was followed, using (3a1,8-dihydropyren-4-yl)diphenylphosphane 1l (77.6 mg, 0.2 mmol) and Ethyl acrylate 2a (80.1 mg, 0.8 mmol). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3la (68.1 mg, 70 %) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.23-8.04 (m, 3H), 8.05-7.92 (m, 4H), 7.79-7.70 (m, 1H), 7.37-7.27 (m, 6H), 7.29-7.20 (m, 4H), 4.15-4.04 (m, 4H), 3.04-2.85 (m, 2H), 1.17 (t, $J = 7.2$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 173.22, 138.28 (d, $J = 13.1$ Hz), 137.69 (d, $J = 2.0$ Hz), 134.69 (d, $J = 21.2$ Hz), 134.48, 133.97 (d, $J = 20.2$ Hz), 132.40, 131.36 (d, $J = 28.3$ Hz), 131.23, 131.02, 130.55, 128.71, 128.66 (d, $J = 7.1$ Hz), 128.09, 127.82, 126.75 (d, $J = 6.1$ Hz), 126.14, 125.26, 125.09, 124.81, 124.03 (d, $J = 2.0$ Hz), 60.28, 36.96 (d, $J = 16.2$ Hz), 33.92 (d, $J = 31.3$ Hz), 14.29. $^{31}$P NMR (162 MHz, CDCl$_3$) δ -1.82. HRMS (ESI+) exact mass calculated for [M+H]$^+$ (C$_{33}$H$_{28}$O$_2$P): 487.1821, found: 487.1821.

ethyl 3-(4-(diphenylphosphaneyl)quinolin-5-yl)propanoate (3ma): The representative procedure 2.1 was followed, using 5-(diphenylphosphaneyl)quinoline 1m (62.6 mg, 0.2 mmol) and Ethyl acrylate 2a
(80.1 mg, 0.8 mmol). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3ma (63.6 mg, 77%) as a white solid. 1H NMR (400 MHz, CDCl3) δ 8.68 (d, J = 4.4 Hz, 1H), 8.09-8.00 (m, 1H), 7.64-7.58 (m, 1H), 7.42-7.31 (m, 7H), 7.25-7.18 (m, 4H), 6.96 (t, J = 4.4 Hz, 1H), 4.12 (q, J = 7.2 Hz, 2H), 3.75-3.67 (m, 2H), 2.78-2.70 (m, 2H), 1.23 (t, J = 7.2 Hz, 3H). 13C NMR (101 MHz, CDCl3) δ 172.7, 149.7 (d, J = 3.5 Hz), 148.6, 145.8 (d, J = 34.6 Hz), 138.8 (d, J = 2.3 Hz), 135.8 (d, J = 11.2 Hz), 134.2 (d, J = 20.7 Hz), 130.2 (d, J = 2.0 Hz), 130.0, 129.6 (d, J = 16.3 Hz), 129.4, 128.9 (d, J = 7.2 Hz), 128.8, 128.3, 60.3, 37.3 (d, J = 13.4 Hz), 32.2 (d, J = 25.9 Hz), 14.2. 31P NMR (162 MHz, CDCl3) δ -3.25. HRMS (ESI+) exact mass calculated for [M+H]+ (C26H25NO2P): 414.1617, found: 414.1618.

methyl 3-(8-(diphenyldiphenylene)naphthalen-1-yl)propanoate (3ab): The representative procedure 2.1 was followed, using 1-naphthalenyldiphenylphosphine 1a (62.4 mg, 0.2 mmol) and Methyl acrylate 2b (0.8 mmol, 4.0 equiv). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3ab (63.7 mg, 80%) as a white solid. 1H NMR (400 MHz, CDCl3) δ 7.83 (dd, J = 8.0, 1.6 Hz, 1H), 7.77-7.74 (m, 1H), 7.41-7.34 (m, 2H), 7.33-7.26 (m, 7H), 7.24-7.19 (m, 5H), 3.80-3.73 (m, 2H), 3.64 (s, 3H), 2.85-2.78 (m, 2H). 13C NMR (101 MHz, CDCl3) δ 173.5, 138.6 (d, J = 1.9 Hz), 137.9 (d, J = 12.7 Hz), 136.3, 135.6 (d, J = 5.6 Hz), 134.9 (d, J = 20.5 Hz), 134.0 (d, J = 20.2 Hz), 133.8, 133.5, 131.4, 130.2, 129.0 (d, J = 1.9 Hz), 128.6, 128.6 (d, J = 11.2 Hz), 125.4, 124.8, 51.4, 37.3 (d, J = 15.6 Hz), 32.9 (d, J = 28.1 Hz). 31P NMR (162 MHz, CDCl3) δ -2.56. HRMS (ESI+) exact mass calculated for [M+H]+ (C26H23O2P): 399.1508, found: 399.1508.

butyl 3-(8-(diphenyldiphenylene)naphthalen-1-yl)propanoate (3ac): The representative procedure 2.1 was followed, using 1-naphthalenyldiphenylphosphine 1a (62.4 mg, 0.2 mmol) and Butyl Acrylate 2c (0.8 mmol, 4.0 equiv). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3ac (61.6 mg, 70%) as a white solid. 1H NMR (400 MHz, CDCl3) 1H NMR (400 MHz, Chloroform-d) δ 7.83 (dd, J = 8, 1.6 Hz, 1H), 7.78-7.72 (m, 1H), 7.42-7.34 (m, 2H), 7.34-7.26 (m, 7H), 7.25-7.18 (m, 5H), 4.06 (t, J = 6.8 Hz, 2H), 3.83-3.72 (m, 2H), 2.88-2.77 (m, 2H), 1.59-1.51 (m, 2H), 1.37-1.28 (m, 2H), 0.89 (t, J = 7.2 Hz, 3H). 13C NMR (101 MHz, CDCl3) δ 173.2, 138.7 (d, J = 1.9 Hz), 137.9 (d, J = 12.9 Hz), 136.2, 135.6 (d, J = 5.5 Hz), 134.9 (d, J = 20.2 Hz), 134.0 (d, J = 20.3 Hz), 133.7 (d, J = 28.7 Hz), 131.4, 130.2, 128.9 (d, J = 1.8 Hz), 128.6, 128.6 (d, J = 10.6 Hz), 64.4, 37.6 (d, J = 15.7 Hz), 32.9 (d, J = 28.3 Hz), 31.4, 28.6, 25.6, 22.5, 14.0. 31P NMR (162 MHz, CDCl3) δ -2.43. HRMS (ESI+) exact mass calculated for [M+H]+ (C34H30OP): 441.1978, found: 441.1979.
hexyl 3-(8-(diphenylphosphaneyl)naphthalen-1-yl)propanoate (3ad): The representative procedure 2.1 was followed, using 1-naphthalenylidiphenylphosphane 1a (62.4 mg, 0.2 mmol) and Hexyl acrylate 2d (0.8 mmol, 4.0 equiv). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3ad (77.7 mg, 83%) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.84 (dd, $J = 8.0$, 1.6 Hz, 1H), 7.79-7.71 (m, 1H), 7.42-7.34 (m, 2H), 7.34-7.25 (m, 7H), 7.25-7.19 (m, 5H), 4.05 (t, $J = 6.8$ Hz, 2H), 3.85-3.72 (m, 2H), 2.88-2.76 (m, 2H), 1.60-1.52 (m, 2H), 1.31-1.23 (m, 6H), 0.85 (t, $J = 6.8$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 173.22, 138.73 (d, $J = 2$ Hz), 137.98 (d, $J = 13.1$Hz), 136.28, 135.63 (d, $J = 5.1$ Hz), 134.96 (d, $J = 20.2$ Hz), 134.04 (d, $J = 23.2$ Hz), 133.77 (d, $J = 29.2$ Hz), 131.43, 130.28, 128.97 (d, $J = 2$ Hz), 128.64, 128.63 (d, $J = 11.1$ Hz), 64.46, 37.64 (d, $J = 16.2$ Hz), 33.00 (d, $J = 28.3$ Hz), 31.50, 28.67, 25.65, 22.56, 14.06. $^{31}$P NMR (162 MHz, CDCl$_3$) δ -2.44. HRMS (ESI+) exact mass calculated for [M+H]$^+$ (C$_{31}$H$_{34}$O$_2$P): 469.2291, found: 469.2291.

isobutyl 3-(8-(diphenylphosphaneyl)naphthalen-1-yl)propanoate (3ae): The representative procedure 2.1 was followed, using 1-naphthalenylidiphenylphosphane 1a (62.4 mg, 0.2 mmol) and Isobutyl acrylat 2e (0.8 mmol, 4.0 equiv). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3ae (63.9 mg, 75%) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.83 (dd, $J = 8.0$, 1.6 Hz, 1H), 7.78-7.73 (m, 1H), 7.40-7.34 (m, 2H), 7.33-7.26 (m, 7H), 7.24-7.19 (m, 5H), 3.84 (d, $J = 6.4$ Hz, 2H), 3.82-3.74 (m, 2H), 2.87-2.78 (m, 2H), 1.93-1.83 (m, 1H), 0.89 (s, 3H), 0.87 (s, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 173.12, 138.73 (d, $J = 1.9$ Hz), 137.98 (d, $J = 13.0$ Hz), 136.28, 135.63 (d, $J = 5.4$ Hz), 134.96 (d, $J = 20.3$ Hz), 134.04 (d, $J = 20.3$ Hz), 133.77 (d, $J = 29.2$ Hz), 131.43, 130.28, 128.97 (d, $J = 2$ Hz), 128.64, 128.63 (d, $J = 11.1$ Hz), 64.46, 37.64 (d, $J = 16.2$ Hz), 33.00 (d, $J = 28.3$ Hz), 31.50, 28.67, 25.65, 22.56, 14.06. $^{31}$P NMR (162 MHz, CDCl$_3$) δ -2.46. HRMS (ESI+) exact mass calculated for [M+H]$^+$ (C$_{29}$H$_{30}$O$_2$P): 441.1978, found: 441.1978.

tert-butyl 3-(8-(diphenylphosphaneyl)naphthalen-1-yl)propanoate (3af): The representative procedure 2.1 was followed, using 1-naphthalenylidiphenylphosphane 1a (62.4 mg, 0.2 mmol) and tert-Butyl acrylate 2f (0.8 mmol, 4.0 equiv). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3af (44 mg, 50%) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.83 (dd, $J = 8.0$, 1.6 Hz,
1H), 7.78-7.73 (m, 1H), 7.40-7.35 (m, 2H), 7.34-7.26 (m, 7H), 7.25-7.19 (m, 5H), 3.78-3.70 (m, 2H), 2.77-2.69 (m, 2H), 1.41 (s, 9H). 13C NMR (101 MHz, CDCl3) δ 172.5, 138.9 (d, J = 2.1 Hz), 138.1 (d, J = 13.4 Hz), 136.2, 135.5 (d, J = 5.5 Hz), 135.0 (d, J = 20.2 Hz), 134.0 (d, J = 20.4 Hz), 133.8 (d, J = 29.0 Hz), 131.3, 130.1, 128.8 (d, J = 1.8 Hz), 128.6, 128.5, 125.4, 124.7, 79.9, 38.7 (d, J = 15.2 Hz), 33.0 (d, J = 28.4 Hz), 28.1. 31P NMR (162 MHz, CDCl3) δ -2.43. HRMS (ESI+) exact mass calculated for [M+H]+ (C29H30O2P): 441.1978, found: 441.1978.

**benzyl 3-(8-(diphenylphosphaneyl)naphthalen-1-yl)propanoate (3ag):** The representative procedure 2.1 was followed, using 1-naphthalenyldiphenylphosphane 1a (62.4 mg, 0.2 mmol) and Benzylacrylate 2g (0.8 mmol, 4.0 equiv). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3ag (42.7 mg, 45%) as a white solid. 1H NMR (400 MHz, CDCl3) δ 7.84 (dd, J = 8.0, 1.6 Hz, 1H), 7.78-7.74 (m, 1H), 7.39-7.34 (m, 2H), 7.23-7.16 (m, 5H), 5.12 (s, 2H), 3.83-3.76 (m, 2H), 2.92-2.86 (m, 2H). 13C NMR (101 MHz, CDCl3) δ 172.9, 138.5 (d, J = 1.9 Hz), 137.8 (d, J = 12.7 Hz), 136.2, 136.2, 135.6 (d, J = 5.5 Hz), 134.8 (d, J = 20.2 Hz), 134.0 (d, J = 20.2 Hz), 133.7 (d, J = 28.6 Hz), 131.4, 130.3, 128.6 (d, J = 10.5 Hz), 128.5 (d, J = 16.6 Hz), 128.1, 128.0, 125.4, 124.7, 66.0, 37.6 (d, J = 16.0 Hz), 32.9 (d, J = 28.1 Hz). 31P NMR (162 MHz, CDCl3) δ -2.50. HRMS (ESI+) exact mass calculated for [M+H]+ (C32H28O2P): 475.1821, found: 475.1821.

**2-ethylhexyl 3-(8-(diphenylphosphaneyl)naphthalen-1-yl)propanoate (3ah):** The representative procedure 2.1 was followed, using 1-naphthalenyldiphenylphosphane 1a (62.4 mg, 0.2 mmol) and 2-Ethylhexyl acrylate 2h (0.8 mmol, 4.0 equiv). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3ag (38.6 mg, 40%) as a white solid. 1H NMR (400 MHz, CDCl3) δ 7.85 (dd, J = 8.0, 1.6 Hz, 1H), 7.79-7.74 (m, 1H), 7.42-7.36 (m, 2H), 7.35-7.27 (m, 7H), 7.26-7.19 (m, 5H), 4.02-3.96 (m, 2H), 3.83-3.76 (m, 2H), 2.88-2.81 (m, 2H), 1.57-1.51 (m, 1H), 1.33-1.24 (m, 8H), 0.86 (t, J = 7.2 Hz, 6H). 13C NMR (101 MHz, CDCl3) δ 173.3, 138.7 (d, J = 2.1 Hz), 137.9 (d, J = 13.0 Hz), 136.2, 135.6 (d, J = 5.6 Hz), 134.8 (d, J = 20.3 Hz), 134.0 (d, J = 20.4 Hz), 133.6, 131.4, 130.2, 128.9 (d, J = 1.8 Hz), 128.6, 128.6 (d, J = 10.9 Hz), 125.4, 124.7, 66.6, 38.7, 37.6 (d, J = 15.7 Hz), 33.0 (d, J = 28.2 Hz), 30.3, 28.9, 23.7, 22.9, 14.1, 11.0. 31P NMR (162 MHz, CDCl3) δ -2.37. HRMS (ESI+) exact mass calculated for [M+H]+ (C33H38O2P): 497.2604, found: 497.2604.
dodecyl 3-(8-(diphenylphosphaneyl)naphthalen-1-yl)propanoate (3ai): The representative procedure 2.1 was followed, using 1-naphthalenylidiphenylphosphane 1a (62.4 mg, 0.2 mmol) and Dodecyl acrylate 2i (0.8 mmol, 4.0 equiv). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3ai (73.9 mg, 67%) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.84 (dd, $J = 8.0, 1.6$ Hz, 1H), 7.78-7.74 (m, 1H), 7.42-7.34 (m, 2H), 7.34-7.25 (m, 7H), 7.25-7.19 (m, 5H), 4.05 (t, $J = 6.8$ Hz, 2H), 3.83-3.72 (m, 2H), 2.88-2.77 (m, 2H), 1.60-1.54 (m, 2H), 1.24 (s, 16H), 0.87 (t, $J = 6.8$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 173.2, 138.7 (d, $J = 12.7$ Hz), 137.9 (d, $J = 12.9$ Hz), 136.2, 135.6 (d, $J = 5.5$ Hz), 134.9 (d, $J = 20.1$ Hz), 134.0 (d, $J = 20.2$ Hz), 133.7 (d, $J = 28.6$ Hz), 131.4, 130.2, 128.9 (d, $J = 1.8$ Hz), 128.6, 128.6 (d, $J = 10.7$ Hz), 125.4, 124.7, 64.4, 37.6 (d, $J = 15.7$ Hz), 32.9 (d, $J = 28.0$ Hz), 31.9, 29.6, 29.6, 29.5, 29.3, 29.3, 28.7, 25.9, 22.7. $^{31}$P NMR (162 MHz, CDCl$_3$) $\delta$ -2.46.

2-methoxyethyl 3-(8-(diphenylphosphaneyl)naphthalen-1-yl)propanoate (3aj): The representative procedure 2.1 was followed, using 1-naphthalenylidiphenylphosphane 1a (62.4 mg, 0.2 mmol) and 2-Methoxyethyl acrylate 2j (0.8 mmol, 4.0 equiv). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3aj (68.2 mg, 77%) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.84 (dd, $J = 8.0, 1.6$ Hz, 1H), 7.79-7.74 (m, 1H), 7.41-7.35 (m, 2H), 7.34-7.27 (m, 7H), 7.25-7.18 (m, 5H), 4.25-4.19 (m, 2H), 3.82-3.74 (m, 2H), 3.59-3.53 (m, 2H), 3.35 (s, 3H), 2.91-2.84 (m, 2H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 173.0, 138.5, 137.9 (d, $J = 12.7$ Hz), 136.2, 135.6, 134.9 (d, $J = 20.1$ Hz), 134.0 (d, $J = 20.2$ Hz), 133.7 (d, $J = 28.8$ Hz), 131.4, 130.3, 129.0, 128.6, 128.6 (d, $J = 11.2$ Hz), 125.4, 124.7, 70.5, 63.3, 59.0, 37.4 (d, $J = 16.1$ Hz), 32.8 (d, $J = 28.0$ Hz). $^{31}$P NMR (162 MHz, CDCl$_3$) $\delta$ -2.46. HRMS (ESI+) exact mass calculated for [M+H]$^+$ (C$_{37}$H$_{46}$O$_2$P): 553.3230, found: 553.3230.

N-(tert-butyl)-3-(8-(diphenylphosphaneyl)naphthalen-1-yl)propanamide (3ak): The representative procedure 2.1 was followed, using 1-naphthalenylidiphenylphosphane 1a (62.4 mg, 0.2 mmol) and N-tert-Butylacrylamide 2k (0.8 mmol, 4.0 equiv). Purification by prepared column chromatography (EA/hexane = 1/2) yielded 3ak (29.0 mg, 33%) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.87 (dd,
$J = 8.0, 1.6$ Hz, $1H), 7.81-7.76$ (m, $1H$), $7.41$ (d, $J = 4.0$ Hz, $2H$), $7.37-7.27$ (m, $7H$), $7.23-7.17$ (m, $5H$), $6.10$ (s, $1H$), $3.74-3.65$ (m, $2H$), $2.60-2.52$ (m, $2H$), $1.33$ (s, $9H$).

$^{13}C$ NMR (101 MHz, CDCl$_3$) δ 172.2, 138.7 (d, $J = 2.2$ Hz), 137.3 (d, $J = 9.7$ Hz), 136.0, 135.6 (d, $J = 5.5$ Hz), 134.4 (d, $J = 19.0$ Hz), 133.8 (d, $J = 20.0$ Hz), 132.6 (d, $J = 25.3$ Hz), 131.7, 130.8, 128.9 (d, $J = 16.5$ Hz), 128.7, 125.7, 124.7, 50.9, 41.9 (d, $J = 12.3$ Hz), 35.2 (d, $J = 25.7$ Hz), 28.9. $^{31}P$ NMR (162 MHz, CDCl$_3$) δ -1.41.

HRMS (ESI+) exact mass calculated for [M+H]$^+$ (C$_{29}$H$_{31}$NOP): 440.2138, found: 440.2138.

3-(8-(diphenylphosphanylidene)naphthalen-1-yl)-N,N-dimethylpropanamide (3a1): The representative procedure 2.1 was followed, using 1-naphthalenyldiphenylphosphane 1a (62.4 mg, 0.2 mmol) and N,N-Dimethylacrylamide 2k (0.8 mmol, 4.0 equiv). Purification by prepared column chromatography (EA/hexane = 1/2) yielded 3a1 (35.4 mg, 43%) as a white solid. $^1H$ NMR (400 MHz, CDCl$_3$) δ 7.85 (dd, $J = 8.0, 1.6$ Hz, 1H), 7.79-7.74 (m, 1H), 7.43-7.38 (m, 2H), 7.34-7.27 (m, 7H), 7.26-7.20 (m, 5H), 3.90-3.82 (m, 2H), 2.91 (s, 3H), 2.74-2.66 (m, 5H). $^{13}C$ NMR (101 MHz, CDCl$_3$) δ 172.3, 139.7 (d, $J = 1.8$ Hz), 138.0 (d, $J = 13.1$ Hz), 136.4, 135.6 (d, $J = 5.6$ Hz), 135.0 (d, $J = 20.3$ Hz), 134.0 (d, $J = 20.2$ Hz), 133.7 (d, $J = 28.6$ Hz), 131.5, 130.6, 128.7 (d, $J = 1.8$ Hz), 128.6, 128.5, 125.5, 124.6, 37.0, 36.9 (d, $J = 13.9$ Hz), 35.3, 33.5 (d, $J = 28.4$ Hz). $^{31}P$ NMR (162 MHz, CDCl$_3$) δ -3.27. HRMS (ESI+) exact mass calculated for [M+H]$^+$ (C$_{27}$H$_{27}$NOP): 412.1825, found: 412.1825.

3-(8-(diphenylphosphanylidene)naphthalen-1-yl)-1-morpholinopropan-1-one (3am): The representative procedure 2.1 was followed, using 1-naphthalenyldiphenylphosphane 1a (62.4 mg, 0.2 mmol) and 4-Acryloylmorpholine 2m (0.8 mmol, 4.0 equiv). Purification by prepared column chromatography (EA/hexane = 1/2) yielded 3am (36.3 mg, 40%) as a white solid. $^1H$ NMR (400 MHz, CDCl$_3$) δ 7.86 (dd, $J = 8.0, 1.6$ Hz, 1H), 7.81-7.74 (m, 1H), 7.43-7.37 (m, 2H), 7.36-7.27 (m, 7H), 7.25-7.15 (m, 5H), 3.93-3.83 (m, 2H), 3.58 (s, 4H), 3.32 (t, $J = 4.8$ Hz, 2H), 3.13 (t, $J = 4.8$ Hz, 2H), 2.77-2.66 (m, 2H). $^{13}C$ NMR (101 MHz, CDCl$_3$) δ 171.1, 139.2 (d, $J = 1.9$ Hz), 137.8 (d, $J = 12.6$ Hz), 136.4, 135.6 (d, $J = 5.5$ Hz), 134.9 (d, $J = 20.1$ Hz), 133.9 (d, $J = 20.1$ Hz), 133.5 (d, $J = 28.1$ Hz), 131.5, 130.8, 129.0 (d, $J = 1.9$ Hz), 128.7, 128.6 (d, $J = 10.3$ Hz), 125.6, 124.7, 66.6 (d, $J = 45.0$ Hz), 45.7, 41.8, 36.3 (d, $J = 14.3$ Hz), 33.6 (d, $J = 28.1$ Hz). $^{31}P$ NMR (162 MHz, CDCl$_3$) δ -3.15. HRMS (ESI+) exact mass calculated for [M+H]$^+$ (C$_{29}$H$_{29}$NO$_2$P): 454.1930, found: 454.1931.
(1R,2R,5R)-2-isopropyl-5-methylcyclohexyl 3-(8-(diphenylphosphaneyl)naphthalen-1-yl)propanoate (3n): The representative procedure 2.1 was followed, using 1-naphthalenylidiphenylphosphane 1a (62.4 mg, 0.2 mmol) and (1S,2S,5S)-2-isopropyl-5-methylcyclohexyl acrylate 2n (0.8 mmol, 4.0 equiv). Purification by prepared column chromatography (EA/hexane = 1/25) yielded 3an (58.5 mg, 56%) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.84 (dd, $J$ = 8.0, 1.6 Hz, 1H), 7.79-7.73 (m, 1H), 7.41-7.35 (m, 2H), 7.33-7.26 (m, 7H), 7.25-7.18 (m, 5H), 4.74-4.67 (m, 1H), 3.85-3.68 (m, 2H), 2.93-2.74 (m, 2H), 1.99-1.94 (m, 1H), 1.87-1.80 (m, 1H), 1.68-1.61 (m, 1H), 1.51-1.44 (m, 1H), 1.36-1.26 (m, 2H), 1.08-1.00 (m, 1H), 0.93-0.86 (m, 5H), 0.78 (d, $J$ = 6.8 Hz, 3H), 0.70 (d, $J$ = 6.8 Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 172.7, 138.7 (d, $J$ = 2.1 Hz), 138.2 (d, $J$ = 13.5 Hz), 137.9 (d, $J$ = 12.7 Hz), 136.1, 135.6 (d, $J$ = 5.4 Hz), 134.9 (d, $J$ = 20.0 Hz), 134.1 (d, $J$ = 6.9 Hz), 133.9 (d, $J$ = 7.0 Hz), 133.6, 131.4, 128.9 (d, $J$ = 1.8 Hz), 128.6, 128.6, 128.5, 128.5, 128.5, 125.4, 124.7, 73.8, 47.0, 41.0, 37.8 (d, $J$ = 16.5 Hz), 34.3, 33.0 (d, $J$ = 28.0 Hz), 31.4, 26.0, 23.3, 22.0, 20.7, 16.2.$^{31}$P NMR (162 MHz, CDCl$_3$) $\delta$ -2.13. HRMS (ESI+) exact mass calculated for [M+H]$^+$ (C$_{35}$H$_{40}$O$_2$P): 523.2766, found: 523.2769.

3-(8-(diphenylphosphaneyl)naphthalen-1-yl)propanoic acid (4aa): Purification by prepared column chromatography (EA/hexane = 1/2) yielded 4aa (167.5 mg, 87%) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.84 (dd, $J$ = 8.0, 1.6 Hz, 1H), 7.80-7.72 (m, 1H), 7.44-7.32 (m, 2H), 7.33-7.26 (m, 7H), 7.25-7.18 (m, 5H), 3.85-3.68 (m, 2H), 2.93-2.74 (m, 2H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 178.37, 138.7 (d, $J$ = 2.1 Hz), 138.2 (d, $J$ = 13.5 Hz), 137.9 (d, $J$ = 12.7 Hz), 136.1, 135.6 (d, $J$ = 5.4 Hz), 134.9 (d, $J$ = 20.0 Hz), 134.1 (d, $J$ = 6.9 Hz), 133.9 (d, $J$ = 7.0 Hz), 133.6, 131.4, 128.9 (d, $J$ = 1.8 Hz), 128.6, 128.6, 128.5, 128.5, 128.5, 125.4, 124.7, 73.8, 47.0, 41.0, 37.8 (d, $J$ = 16.5 Hz), 34.3, 33.0 (d, $J$ = 28.0 Hz), 31.4, 26.0, 23.3, 22.0, 20.7, 16.2.$^{31}$P NMR (162 MHz, CDCl$_3$) $\delta$ -2.13. HRMS (ESI+) exact mass calculated for [M+H]$^+$ (C$_{25}$H$_{22}$O$_2$P): 385.1357, found: 385.1359.

3-(8-(diphenylphosphaneyl)naphthalen-1-yl)propan-1-ol (5aa): Purification by prepared column chromatography (EA/hexane = 1/15) yielded 3aa (183.6 mg, 99%) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.83 (dd, $J$ = 8.0, 1.6 Hz, 1H), 7.76-7.70 (m, 1H), 7.40-7.27 (m, 8H), 7.26-7.17 (m, 6H), 3.71
(t, J = 6.0 Hz, 2H), 3.57-3.48 (m, 2H), 2.14-2.00 (m, 3H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 140.03 (d, \(J = 2.2\) Hz), 137.64 (d, \(J = 10.8\) Hz), 136.17, 135.56 (d, \(J = 5.6\) Hz), 134.90 (d, \(J = 19.4\) Hz), 133.90 (d, \(J = 19.9\) Hz), 133.17 (d, \(J = 26.3\) Hz), 131.49, 130.09, 128.71, 128.60 (d, \(J = 6.9\) Hz), 128.49 (d, \(J = 1.8\) Hz), 125.44, 124.59, 62.66, 36.36 (d, \(J = 9.3\) Hz). \(^{31}\)P NMR (162 MHz, CDCl\(_3\)) \(\delta\) -1.73.


**butylbenzene (8a):** The representative procedure \(\text{2.3}\) was followed, using bromobenzene \(6a\) (155.9 mg, 1.0 mmol) and n-Butylboronic acid \(7a\) (132.5 mg, 1.3 mmol). Purification by prepared column chromatography (EA/hexane = 1/100) yielded \(8a\) (128.6mg, 96 %) as a colorless oil. \(^{1}\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.31-7.21 (m, 2H), 7.19-7.12 (m, 3H), 2.63-2.57 (m, 2H), 1.64-1.55 (m, 2H), 1.40-1.30 (m, 2H), 0.92 (t, \(J = 6.8\) Hz 3H). HRMS (ESI+) exact mass calculated for [M+H]\(^{+}\) (C\(_{12}\)H\(_{17}\)O): 177.1274, found: 177.1274.

**1-(4-butylphenyl)ethan-1-one (8b):** The representative procedure \(\text{2.3}\) was followed, using 1-(4-bromophenyl)ethan-1-one \(6b\) (198.0 mg, 1.0 mmol) and n-Butylboronic acid \(7a\) (132.5 mg, 1.3 mmol). Purification by prepared column chromatography (EA/hexane = 1/50) yielded \(8b\) (174.4 mg, 98 %) as a white solid. \(^{1}\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.86 (d, \(J = 7.2\) Hz, 2H), 7.24 (d, \(J = 8.0\) Hz, 2H), 2.64 (t, \(J = 8.0\) Hz, 2H), 2.55 (s, 3H), 1.64-1.55 (m, 2H), 1.41-1.27 (m, 2H), 0.92 (t, \(J = 7.6\) Hz, 3H). HRMS (ESI+) exact mass calculated for [M+H]\(^{+}\) (C\(_{12}\)H\(_{17}\)O): 177.1274, found: 177.1274.

**1-butyl-4-nitrobenzene (8c):** The representative procedure \(\text{2.3}\) was followed, using 1-bromo-4-nitrobenzene \(6c\) (201.0 mg, 1.0 mmol) and n-Butylboronic acid \(7a\) (132.5 mg, 1.3 mmol). Purification by prepared column chromatography (EA/hexane = 1/50) yielded \(8c\) (152.2 mg, 85 %) as a white solid. \(^{1}\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.12 (d, \(J = 8.8\) Hz, 2H), 7.32 (d, \(J = 8.8\) Hz, 2H), 2.75-2.67 (m, 2H), 1.67-1.58 (m, 2H), 1.43-1.30 (m, 2H), 0.94 (t, \(J = 7.2\) Hz, 3H). HRMS (ESI+) exact mass calculated for [M+Na]\(^{+}\) (C\(_{10}\)H\(_{13}\)NO\(_2\)Na): 202.0838 found: 202.0840.
9-butylanthracene (8d): The representative procedure 2.3 was followed, using 9-bromoanthracene 6d (256.0 mg, 1.0 mmol) and n-Butylboronic acid 7a (132.5 mg, 1.3 mmol). Purification by prepared column chromatography (EA/hexane = 1/100) yielded 8d (231.7.0 mg, 99 %) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.38-8.20 (m, 3H), 7.99-7.91 (m, 2H), 7.53-7.38 (m, 4H), 3.65-3.52 (m, 2H), 1.89-1.76 (m, 2H), 1.64-1.53 (m, 2H), 1.03 (t, $J$ = 7.2 Hz, 3H). HRMS (ESI+) exact mass calculated for [M+H]$^+$ (C$_{18}$H$_{19}$): 235.1481, found: 235.1482.

9-butylphenanthrene (8e): The representative procedure 2.3 was followed, using 9-bromophenanthrene 6e (256.0 mg, 1.0 mmol) and n-Butylboronic acid 7a (132.5 mg, 1.3 mmol). Purification by prepared column chromatography (EA/hexane = 1/100) yielded 8e (231.7.0 mg, 99 %) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.91-8.84 (m, 1H), 8.82-8.77 (m, 1H), 8.33-8.24 (m, 1H), 8.01-7.94 (m, 1H), 7.83-7.77 (m, 2H), 7.77-7.68 (m, 3H), 3.31-3.21 (m, 2H), 2.02-1.93 (m, 2H), 1.73-1.63 (m, 2H), 1.19 (t, $J$ = 7.2 Hz, 3H). HRMS (ESI+) exact mass calculated for [M+H]$^+$ (C$_{18}$H$_{19}$): 235.1481, found: 235.1482.

1-butylpyrene (8f): The representative procedure 2.3 was followed, using 1-bromopyrene 6f (280.0 mg, 1.0 mmol) and n-Butylboronic acid 7a (132.5 mg, 1.3 mmol). Purification by prepared column chromatography (EA/hexane = 1/100) yielded 8f (255.6 mg, 99 %) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.51-8.41 (m, 1H), 8.51-8.41 (m, 1H), 8.37-8.29 (m, 2H), 8.29-8.22 (m, 2H), 8.21-8.12 (m, 3H), 8.05-7.99 (m, 1H), 3.57-3.44 (m, 2H), 2.12-2.00 (m, 2H), 1.80-1.68 (m, 2H), 1.32-1.21 (m, 3H). HRMS (ESI+) exact mass calculated for [M+H]$^+$ (C$_{20}$H$_{19}$): 259.1481, found: 235.1483.

4-butylquinoline (8g): The representative procedure 2.3 was followed, using 4-bromoquinoline 6g (206.9 mg, 1.0 mmol) and n-Butylboronic acid 7a (132.5 mg, 1.3 mmol). Purification by prepared column chromatography (EA/hexane = 1/5) yielded 8g (111.0 mg, 60 %) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.78 (d, $J$ = 4.4 Hz, 1H), 8.11 (d, $J$ = 8.8 Hz, 1H), 8.00 (d, $J$ = 7.6Hz, 1H), 7.71-7.64 (m, 1H),
7.55-7.47 (m, 1H), 7.25-7.08 (m, 1H), 3.07-2.95 (m, 2H), 1.77-1.65 (m, 2H), 1.51-1.36 (m, 2H), 0.96 (t, \(J = 7.2\) Hz, 3H). HRMS (ESI+) exact mass calculated for [M+H]+ (C_{13}H_{16}N): 186.1277, found: 186.1277.

5. Crystallographic Data

X-ray data for 3aa

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\[aR_1 = \frac{\sum |F_o| - |F_c|}{\sum |F_o|}, \quad b wR_2 = \frac{\sum w(F_o^2 - F_c^2)^2}{\sum w(F_o^4)}^{1/2}\]
6. NMR Spectra

$^1$H NMR, CDCl$_3$, 400 MHz

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$^{31}$P NMR, CDCl$_3$, 162 MHz

$^1$H NMR, CDCl$_3$, 400 MHz
$^{13}$C NMR, CDCl$_3$, 101 MHz

$^{31}$P NMR, CDCl$_3$, 162 MHz
$^{13}$C NMR, CDCl$_3$, 101 MHz

$^1$H NMR, CDCl$_3$, 400 MHz
$^3$P NMR, CDCl$_3$, 162 MHz

$^1$H NMR, CDCl$_3$, 400 MHz
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$^{31}$P NMR, CDCl$_3$, 162 MHz
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$^{31}$P NMR, CDCl$_3$, 162 MHz

$^1$H NMR, CDCl$_3$, 400 MHz
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$^{31}$P NMR, CDCl$_3$, 162 MHz
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31P NMR, CDCl₃, 162 MHz

3H NMR, CDCl₃, 400 MHz
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$^{31}$P NMR, CDCl$_3$, 162 MHz
$^1$H NMR, CDCl$_3$, 400 MHz

$^{13}$C NMR, CDCl$_3$, 101 MHz
$^{31}$P NMR, CDCl$_3$, 162 MHz

$^1$H NMR, CDCl$_3$, 400 MHz
$^{13}$C NMR, CDCl$_3$, 101 MHz

$^{31}$P NMR, CDCl$_3$, 162 MHz
\[\text{PPh}_2 \quad \text{CO}_2\text{t}-\text{Bu}\]

3ae

$^1$H NMR, CDCl$_3$, 400 MHz

\[\text{PPh}_2 \quad \text{CO}_2\text{t}-\text{Bu}\]

3ae

$^{13}$C NMR, CDCl$_3$, 101 MHz
$^{31}$P NMR, CDCl$_3$, 162 MHz

$^1$H NMR, CDCl$_3$, 400 MHz
$^1\text{H NMR, CDCl}_3, 400 \text{ MHz}$

$^{13}\text{C NMR, CDCl}_3, 101 \text{ MHz}$
$^{31}$P NMR, CDCl$_3$, 162 MHz

$^1$H NMR, CDCl$_3$, 400 MHz
3ah

$^{13}$C NMR, CDCl$_3$, 101 MHz

$^{31}$P NMR, CDCl$_3$, 162 MHz
$\text{PPh}_2\overset{\text{O}}{\text{(CH}_2\text{)}_{11}\text{CH}_3}$

3ai

$^1\text{H NMR, CDCl}_3$, 400 MHz

$\text{PPh}_2\overset{\text{O}}{\text{(CH}_2\text{)}_{11}\text{CH}_3}$

3ai

$^{13}\text{C NMR, CDCl}_3$, 101 MHz
3ai
$^{31}$P NMR, CDCl$_3$, 162 MHz

3aj
$^1$H NMR, CDCl$_3$, 400 MHz
$^{13}$C NMR, CDCl$_3$, 101 MHz

$^{31}$P NMR, CDCl$_3$, 162 MHz
$^{1}$H NMR, CDCl$_3$, 400 MHz

$^{13}$C NMR, CDCl$_3$, 101 MHz
$^{31}$P NMR, CDCl$_3$, 162 MHz

$^1$H NMR, CDCl$_3$, 400 MHz
$^{13}$C NMR, CDCl$_3$, 101 MHz

$^{31}$P NMR, CDCl$_3$, 162 MHz
$^{31}$P NMR, CDCl$_3$, 162 MHz

$^{1}$H NMR, CDCl$_3$, 400 MHz
$^{13}$C NMR, CDCl$_3$, 101 MHz

$^{31}$P NMR, CDCl$_3$, 162 MHz
5aa

$^1$H NMR, CDCl$_3$, 400 MHz

5aa

$^{13}$C NMR, CDCl$_3$, 101 MHz
$^{31}$P NMR, CDCl$_3$, 162 MHz

$^1$H NMR, CDCl$_3$, 400 MHz
**Int B**

$^1$H NMR, CDCl$_3$, 400 MHz

$^{13}$C NMR, CDCl$_3$, 101 MHz
$^{31}\text{P NMR, CDCl}_3, 162\text{ MHz}$

$^1\text{H NMR, CDCl}_3, 400\text{ MHz}$
8b

$^1$H NMR, CDCl$_3$, 400 MHz

8c

$^1$H NMR, CDCl$_3$, 400 MHz
8d
$^1$H NMR, CDCl$_3$, 400 MHz

8e
$^1$H NMR, CDCl$_3$, 400 MHz
n-Bu

\( ^1H \) NMR, CDCl\(_3\), 400 MHz

n-Bu

\( ^1H \) NMR, CDCl\(_3\), 400 MHz