Supporting Information

A Unique Copper-Catalyzed Cross-Hydrogen (H₂) Removal Coupling to Stereoselective Synthesis of 3-Phosphoindoles

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1. General information.

$^1$H and $^{13}$C NMR spectra were recorded on a Bruker advance III 400 spectrometer in CDCl$_3$ with TMS as internal standard. $^{31}$P NMR spectra and $^{19}$F NMR were recorded on the same instrument. Mass spectra were measured using Bruker microTOF-Q II. The starting materials were purchased from Aldrich, Acros Organics, J&K Chemicals or TCI and used without further purification. Solvents were dried and purified according to the procedure from “Purification of Laboratory Chemicals book”. Column chromatography was carried out on silica gel (particle size 200-400 mesh ASTM).

2. Screening results and typical procedure.

2.1 Reaction Conditions Screening

In an initial study, we chose the N-methylindole-2-ethyl formate (1a) and Ph$_2$P(O)H (5a) as model substrates. We extensively screened catalysts, solvents, and temperatures in an argon atmosphere and summarized in Table S1. Gratifyingly, we found that some copper salts such as CuCl, CuBr, CuBr·Me$_2$S, CuCl$_2$, Cu(acac)$_2$, and Cu(OAc)$_2$ serve to helpfully prompt the reaction (Table 1, entries 1-6). Among the CuCl gave the best results and the desired product of N-methyl-3-phosphoindole (2a) was obtained in 64% yield in CH$_3$CN at 50 °C under the Ar atmosphere (Table 1, entry 1). Encouraged by this result, we further optimized the reaction conditions. Solvents screening showed that the CH$_3$CN is still the best choice (Table 1, entries 7-10). If the reaction was carried out under the oxygen, the yield of N-methyl-3-phosphoindole (2a) has not obvious change (Table 1, entry 11). Subsequently, we focused our attention on a variety of nitrogen and phosphine ligands in order to understand their potential in this transformation. Extensive evaluation of ligands leads us to discover that phosphine ligands were effective in this regard (Table 1, entries 12-18). To our delight, the least expensive and most stable PPh$_3$ is the best choice. The yield of N-methyl-3-phosphoindole was further improved to 97% (Table 1, entry 18). However, when the reaction was carried out under O$_2$ or air, the lower yield of N-methyl-3-phosphoindole was observed (Table 1, entries 19-20). If the reaction time has been prolonged, the good yield would be afforded under the N$_2$ atmosphere. The addition of different bases to this transformation proved unhelpful. Furthermore, the control experiment demonstrated that the use of only PPh$_3$ failed to prompt the reaction.
Table 1. Reaction Conditions Screening.\textsuperscript{a}

<table>
<thead>
<tr>
<th>Entry</th>
<th>Cu [mol%]</th>
<th>Ligand [mol%]</th>
<th>Solvent</th>
<th>Yield [%][\textsuperscript{b}]</th>
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<td>CH\textsubscript{3}CN</td>
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<tr>
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\textsuperscript{a} All reactions were carried out in the presence of 0.3 mmol of HP(O)Ph\textsubscript{2} in 3.0 mL CH\textsubscript{3}CN at 50 °C for 24 h under argon. \textsuperscript{b} Isolated yield. \textsuperscript{c} Reaction was carried out under the O\textsubscript{2}. \textsuperscript{d} Reaction was carried out under the N\textsubscript{2} for 36 h. \textsuperscript{e} Reaction was carried out under the air.

2.2 General Procedures for CuCl-Catalyzed Direct Dehydrogenative Phosphorylation for Synthesis of 3-Phosphoindoles (2):

In a Schlenk tube, 1 (0.600 mmol), HP(O)Ph\textsubscript{2} (0.300 mmol), CuCl (0.015 mmol), and PPh\textsubscript{3} (0.018 mmol) were added and charged with Ar three times. Then, anhydrous CH\textsubscript{3}CN (3.0 mL) were added. The mixture was allowed to stir at 50 °C for 24 hours (monitored by TLC). After substrate was consumed, the reaction was cooled to room temperature and concentrated in vacuo. the resulting residue diluted with EtOAc (5.0 ml) and washed with water (5.0 ml). The aqueous phase was extracted with EtOAc (2×10.0 mL). The combination of organic phase was dried with anhydrous Na\textsubscript{2}SO\textsubscript{4}.
and concentrated in vacuo, and the resulting residue was purified by column chromatography using hexanes/EtOAc (10/1 to 1/1) as the eluent.

2.3 General Procedures for CuCl-Catalyzed Direct Dehydrogenative Phosphorylation for Synthesis of 3-Phosphoindoles (4):

In a Schlenk tube, 3 (0.600 mmol), optically pure H-Phosphinate (0.300 mmol), CuCl (0.015 mmol), and PPh₃ (0.018 mmol) were added and charged with Ar three times. Then, anhydrous CH₃CN (3.0 mL) were added. The mixture was allowed to stir at 80°C for 24 hours (monitored by TLC). After substrate was consumed, the reaction was cooled to room temperature and concentrated in vacuo. the resulting residue diluted with EtOAc (5.0 ml) and washed with water (5.0 ml). The aqueous phase was extracted with EtOAc (2×10.0 mL). The combination of organic phase was dried with anhydrous Na₂SO₄ and concentrated in vacuo, and the resulting residue was purified by column chromatography using hexanes/EtOAc (10/1 to 1/1) as the eluent.

2.4 Gram-scale synthesis of 2a.

![Chemical structure](image)

2.5 A Typical Procedure for the Preparation of Optically Pure H-Phosphinates:[82]

The mixture of (L)-(−)-menthol (100 g, 641 mmol) and pyridine (51.3 mL, 641 mmol) in Et₂O (200 mL) was added dropwise with stirring to a PhPCl₂(87.2 mL, 641 mmol) solution in Et₂O (400 mL) at 0°C and then stirred at room temperature overnight. Water (12 mL, 667 mmol) was added, and the reaction mixture was washed with water and extracted with hexane. The hexane layer was dried over magnesium sulfate, filtered, and concentrated. Recrystallization of the mixture in hexane (twice) at -30°C gave pure H-Phosphinates a white crystal (Rₚ/Sₚ>99/1).

2.6 General Procedures for the determination of the dr values. [S1]

In a Schlenk tube, N-methylindole-2-ethyl formate (0.600 mmol), the racemic Menthoxyl-phenylphosphinate (0.300 mmol), CuCl (0.015 mmol), and PPh₃ (0.018 mmol) were added and charged with Ar three times. Then, anhydrous CH₃CN (3.0 mL) were added. The mixture was allowed to stir at 80°C for 24 hours (monitored by TLC). After substrate was consumed, the reaction was cooled to room temperature and concentrated in vacuo. The resulting residue diluted with EtOAc (5.0 ml) and washed with water (5.0 ml). The aqueous phase was extracted with EtOAc (2×10.0 mL). The combination of organic phase was dried with anhydrous Na₂SO₄ and
concentrated in vacuo. Then the determination of the diastereoselectivity by $^{31}$PNMR, or after by flash column chromatography and determined dr values by $^{31}$PNMR (Figure S1 and Figure S2).

**Figure S1.** The dr value of racemic H-Phosphinate according to the $^{31}$PNMR.

**Figure S2.** The dr value of the optically pure H-Phosphinate according to the $^{31}$PNMR.
3. Preliminary mechanistic studies.

3.1 Radicals Trapping Experiments:

a) Procedures for using BHT: In a Schlenk tube, \(N\)-methylindole-2-ethyl formate (0.600 mmol), HP(O)Ph\(_2\) (0.300 mmol), CuCl (0.015 mmol), BHT (0.600 mmol) and PPh\(_3\) (0.018 mmol) were added and charged with Ar three times. Then, anhydrous CH\(_3\)CN (3.0 mL) were added. The mixture was allowed to stir at 50°C for 24 hours (monitored by TLC). After substrate was consumed, the reaction was cooled to room temperature and concentrated in vacuo. the resulting residue diluted with EtOAc (5.0 ml) and washed with water (5.0 ml). The aqueous phase was extracted with EtOAc (2×10.0 mL). The combination of organic phase was dried with anhydrous Na\(_2\)SO\(_4\) and concentrated in vacuo, and the resulting residue was purified by column chromatography to give 2a in 94% yield.

b) Procedures for using cyclohexa-1, 4-diene: In a Schlenk tube, \(N\)-methylindole-2-ethyl formate (0.600 mmol), HP(O)Ph\(_2\) (0.300 mmol), CuCl (0.015 mmol), cyclohexa-1, 4-diene (0.600 mmol) and PPh\(_3\) (0.018 mmol) were added and charged with Ar three times. Then, anhydrous CH\(_3\)CN (3.0 mL) were added. The mixture was allowed to stir at 50°C for 24 hours (monitored by TLC). After substrate was consumed, the reaction was cooled to room temperature and concentrated in vacuo. the resulting residue diluted with EtOAc (5.0 ml) and washed with water (5.0 ml). The aqueous phase was extracted with EtOAc (2×10.0 mL). The combination of organic phase was dried with anhydrous Na\(_2\)SO\(_4\) and concentrated in vacuo, and the resulting residue was purified by column chromatography to give 2a in 89% yield.

3.2 Competing kinetic isotope effect (KIE) experiment: Intermolecular KIE experiment: \(1a-d_1\) were synthesized deuterium substrates according the literature procedure.\(^{[S3]}\)

In a Schlenk tube, \(1a-d_3\) (0.300 mmol), \(1a-d_1\) (0.300 mmol), HP(O)Ph\(_2\) (0.300 mmol), CuCl (0.015 mmol), and PPh\(_3\) (0.018 mmol) were added and charged with Ar three times. Then, anhydrous CH\(_3\)CN (3.0 mL) were added. The mixture was allowed to stir at 50°C for 10 hours (monitored by TLC). After substrate was consumed, the reaction was cooled to room temperature and concentrated in vacuo. the resulting residue diluted with EtOAc (5.0 ml) and washed with water (5.0 ml). The aqueous phase was extracted with EtOAc (2×10.0 mL). The combination of organic phase was dried with anhydrous Na\(_2\)SO\(_4\) and concentrated in vacuo, and the resulting residue was purified by column chromatography to give \(2a-d_3\) and 2a. The products were under 1H-NMR analysis. (Figure S3).
Figure S3. H NMR spectra of the mixture of compound 2a and 2a-$d_3$.

Figure S4. H NMR spectra of 2a.
3.3 CuH and Stryker’s Reagent-Catalyzed Direct Dehydrogenative Coupling Reaction:

a) Procedures for using CuH: In a Schlenk tube, N-methylindole-2-ethyl formate (0.600 mmol), HP(O)Ph₂ (0.300 mmol), CuH (0.015 mmol), BHT (0.600 mmol) and PPh₃ (0.018 mmol) were added and charged with Ar three times. Then, anhydrous CH₃CN (3.0 mL) were added. The mixture was allowed to stir at 50°C for 24 hours (monitored by TLC). After substrate was consumed, the reaction was cooled to room temperature and concentrated in vacuo. the resulting residue dilute with EtOAc (5.0 ml) and washed with water (5.0 ml). The aqueous phase was extracted with EtOAc (2×10.0 mL). The combination of organic phase was dried with anhydrous Na₂SO₄ and concentrated in vacuo, and the resulting residue was purified by column chromatography to give 2a in 76 % yield.

b) Procedures for using Stryker’s Reagent: In a Schlenk tube, N-methylindole-2-ethyl formate (0.600 mmol), HP(O)Ph₂ (0.300 mmol), [CuH(PPh₃)]₆ (0.003 mmol), BHT (0.600 mmol) and PPh₃ (0.018 mmol) were added and charged with Ar three times. Then, anhydrous CH₃CN (3.0 mL) were added. The mixture was allowed to stir at 50°C for 24 hours (monitored by TLC). After substrate was consumed, the reaction was cooled to room temperature and concentrated in vacuo. the resulting residue dilute with EtOAc (5.0 ml) and washed with water (5.0 ml). The aqueous phase was extracted with EtOAc (2×10.0 mL). The combination of organic phase was dried with anhydrous Na₂SO₄ and concentrated in vacuo, and the resulting residue was purified by column chromatography to give 2a in 78 % yield.

Reference:
3.4 Hydrogen detection
1. a) Hydrogen detector:
   
   HEWLETT5890 PACKADR SERIES II GAS CHROMATOGRAPH
   
   b) Carrier gas:  Ar

![Figure S5](image1)

**Figure S5.** The standard spectrogram of H₂.

![Figure S6](image2)

**Figure S6.** The detected spectrogram of H₂.

a) Procedures for the detected H₂ by Gas Chromatograph:

By using the high purity of Ar as carrier gas, we first injected the pure H₂ into the Hewlett5890 Packadr Series II Gas Chromatograph and detected standard spectrogram of H₂ (Figure S5). Then 15 minutes later, we injected the sample gas of reaction system into the Gas Chromatograph and observed the peak of H₂ (Figure S6). This result showed that the H₂ has been released from the reaction.
b) Procedures for the detected H$_2$ by Portable H$_2$ Detector:

In a 50 mL round-bottom flask, N-methylindole-2-ethyl formate (10.0 mmol), H-Phosphinate (5.0 mmol), CuCl 5.0 mmol%, and 6.0 mmol% were added and charged with Ar three times. Then, anhydrous CH$_3$CN (20.0 mL) were added. The mixture was allowed to stir at 50°C for 24 hours and then the reaction was cooled to room temperature. After we injected the syringe needle, which linked with Portable H$_2$ Detector, into the reaction system and H$_2$ Detector began to alarm and the instantaneous H$_2$ concentration was detected with 223 ppm. It worth to note that the highest concentration reached 476 ppm.
3. Mixing Stryker's reagent with HP(O)Ph$_2$
   a) Hydrogen detector: TAYASAF-MG01 portable H$_2$ Detector

   ![Image of Portable H$_2$ Detector]

   **Figure S8.** the observation of H$_2$ evolution

   b) Procedures for the detected H$_2$ by Portable H$_2$ Detector:

   In a 10 mL round-bottom flask, H-Phosphinate (0.5 mmol), Stryker's reagent (0.5 mmol) were added and charged with Ar three times. Then, anhydrous CH$_3$CN (5.0 mL) were added. The mixture was allowed to stir at 50°C for 12 hours and then the reaction was cooled to room temperature. After we injected the syringe needle, which linked with Portable H$_2$ Detector, into the reaction system and H$_2$ Detector began to alarm and the instantaneous H$_2$ concentration was detected with 113 ppm.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (97% yield) as a white solid. Mp: 148-150 °C. R_f (ethyl acetate: petroleum ether, 2:1): 0.29. ¹H NMR (400 MHz, CDCl₃): δ 7.84 - 7.76 (m, 4H), 7.53 - 7.48 (m, 2H), 7.46 - 7.39 (m, 5H), 7.33 - 7.27 (m, 1H), 7.10 (d, J = 8.4 Hz, 1H), 7.00 (t, J = 7.6 Hz, 1H), 3.96 (s, 3H), 3.76 (q, J = 7.2 Hz, 2H), 1.05 (t, J = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 161.6, 138.1 (d, J_C-P = 11.0 Hz), 135.5 (d, J_C-P = 16.0 Hz), 134.6 (d, J_C-P = 109.0 Hz), 131.5, 131.4 (d, J_C-P = 3.0 Hz), 128.5 (d, J_C-P = 9.0 Hz), 128.3 (d, J_C-P = 13.0 Hz), 124.6, 122.7, 121.9, 110.3, 107.2 (d, J_C-P = 120.0 Hz), 61.9, 31.8, 13.4. ³¹P NMR (162 MHz, CDCl₃): δ 21.66. HRMS Calcd for C₂₄H₂₂N₃O₃P [M + Na]⁺ 426.1230, found 426.1224.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (93% yield) as a white solid. Mp: 226-228 °C. R_f (ethyl acetate: petroleum ether, 2:1): 0.13. ¹H NMR (400 MHz, d₆-DMSO): δ 12.85 (brs, 1H), 7.72 - 7.66 (m, 4H), 7.59 - 7.56 (m, 3H), 7.51 - 7.49 (m, 4H), 7.31 - 7.26 (m, 2H), 6.98 (t, J = 6.8 Hz , 1H), 3.81 (q, J = 6.8 Hz , 2H), 0.89 (t, J = 6.8 Hz, 3H). ¹³C NMR (100 MHz, d₆-DMSO): δ 160.2, 136.7 (d, J_C-P = 12.0 Hz), 135.2 (d, J_C-P = 108.0 Hz), 132.3 (d, J_C-P = 15.0 Hz), 131.5, 131.0 (d, J_C-P = 10.0 Hz), 129.9 (d, J_C-P = 8.0 Hz), 128.5 (d, J_C-P = 12.0 Hz), 124.9, 122.5, 121.5, 113.1, 107.4 (d, J_C-P = 118.0 Hz), 61.1, 13.6. ³¹P NMR (162 MHz, d₆-DMSO): δ 21.13. HRMS Calcd for C₂₃H₂₀N₃O₃P [M + Na]⁺ 412.1073, found 412.1065.
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (78% yield) as a white solid. Mp: 155-157 °C.  

Rf (ethyl acetate: petroleum ether, 2:1): 0.24.  

$^1$H NMR (400 MHz, CDCl₃): $\delta$ 7.82 - 7.76 (m, 4H), 7.53 - 7.49 (m, 2H), 7.47 - 7.41 (m, 4H), 7.34 - 7.32 (m, 2H), 7.19 (d, $J = 8.4$ Hz, 1H), 7.04 - 7.00 (m, 1H), 5.24 (s, 2H), 4.21 (q, $J = 7.2$ Hz, 2H), 3.73 (q, $J = 7.2$ Hz, 2H), 1.25 (t, $J = 7.2$ Hz, 3H), 0.97 (t, $J = 7.2$ Hz, 3H).  

$^{13}$C NMR (100 MHz, CDCl₃): $\delta$ 167.8, 161.2, 138.2 (d, $J_{C-P} = 10.0$ Hz), 134.8 (d, $J_{C-P} = 110.0$ Hz), 134.4, 131.5 (d, $J_{C-P} = 10.0$ Hz), 128.9 (d, $J_{C-P} = 8.0$ Hz), 128.3 (d, $J_{C-P} = 12.0$ Hz), 125.3, 123.4, 122.3, 109.7, 109.6 (d, $J_{C-P} = 118.0$ Hz), 61.8, 61.6, 46.5, 14.0, 13.4.  

$^{31}$P NMR (162 MHz, CDCl₃): $\delta$ 22.64. HRMS Calcd for C$_{27}$H$_{26}$NO$_5$P [M + Na]$^+$ 498.1441, found 498.1450.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (80% yield) as a yellow oil.  

Rf (ethyl acetate: petroleum ether, 2:1): 0.42.  

$^1$H NMR (400 MHz, CDCl₃): $\delta$ 7.83 - 7.76 (m, 4H), 7.55 - 7.50 (m, 2H), 7.47 - 7.42 (m, 4H), 7.36 (d, $J = 8.4$ Hz, 1H), 7.31 - 7.23 (m, 4H), 7.15 (d, $J = 8.0$ Hz, 1H), 7.08 (d, $J = 6.8$ Hz, 1H), 7.00 (t, $J = 7.2$ Hz, 1H), 5.64 (s, 2H), 3.65 (q, $J = 7.2$ Hz, 2H), 0.93 (t, $J = 7.2$ Hz, 3H).  

$^{13}$C NMR (100 MHz, CDCl₃): $\delta$ 161.7, 137.9 (d, $J_{C-P} = 11.0$ Hz), 136.6, 135.7 (d, $J_{C-P} = 16.0$ Hz), 134.6 (d, $J_{C-P} = 109.0$ Hz), 131.7, 131.5 (d, $J_{C-P} = 4.0$ Hz), 128.9, 1128.7 (d, $J_{C-P} = 5.0$ Hz), 128.4, 128.3, 127.7, 126.5, 124.9, 123.0, 122.1, 110.9, 108.0 (d, $J_{C-P} = 109.0$ Hz), 61.8, 48.5, 13.4.  

$^{31}$P NMR (162 MHz, CDCl₃): $\delta$ 21.44. HRMS Calcd for C$_{30}$H$_{26}$NNaO$_5$P [M + Na]$^+$ 502.1546, found 502.1548.
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (88% yield) as a white solid. Mp: 215-217 °C. \( R_f \) (ethyl acetate: petroleum ether, 2:1): 0.10. \(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \) 7.70 - 7.64 (m, 4H), 7.53 - 7.46 (m, 3H), 7.45 - 7.40 (m, 4H), 7.22 - 7.17 (m, 1H), 6.93 - 6.84 (m, 2H), 4.77 (s, 3H), 3.36 (s, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \( \delta \) 162.0, 138.4 (d, \( J_{CP} = 12.0 \) Hz), 135.4 (d, \( J_{CP} = 111.0 \) Hz), 134.4 (d, \( J_{CP} = 16.0 \) Hz), 133.2 (d, \( J_{CP} = 3.0 \) Hz), 132.7, 132.6, 131.2 (d, \( J_{CP} = 9.0 \) Hz), 129.9, 129.8, 126.5, 123.4, 123.1, 114.0, 108.0 (d, \( J_{CP} = 123.0 \) Hz), 52.5. \(^{31}\)P NMR (162 MHz, CDCl\(_3\)): \( \delta \) 26.10. HRMS Calcd for C\(_{22}\)H\(_{18}\)NNaO\(_3\)P [M + Na]\(^+\) 398.0917, found 398.0902.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (91% yield) as a white solid. Mp: 171-173 °C. \( R_f \) (ethyl acetate: petroleum ether, 2:1): 0.19. \(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \) 7.83 - 7.75 (m, 4H), 7.52 - 7.47 (m, 2H), 7.44 - 7.39 (m, 5H), 7.31 (t, \( J = 7.2 \) Hz, 1H), 7.17 (d, \( J = 8.4 \) Hz, 1H), 7.00 (t, \( J = 7.6 \) Hz, 1H), 3.96 (s, 3H), 3.27 (s, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \( \delta \) 162.0, 138.4 (d, \( J_{CP} = 10.0 \) Hz), 134.9 134.8 (d, \( J_{CP} = 109.0 \) Hz), 131.4 (d, \( J_{CP} = 2.0 \) Hz), 131.3 (d, \( J_{CP} = 10.0 \) Hz), 128.7 (d, \( J_{CP} = 10.0 \) Hz), 128.5, 128.4, 124.9, 123.0, 122.0, 110.4, 108.1 (d, \( J_{CP} = 119.0 \) Hz), 51.8, 31.9. \(^{31}\)P NMR (162 MHz, CDCl\(_3\)): \( \delta \) 21.05. HRMS Calcd for C\(_{23}\)H\(_{20}\)NNaO\(_3\)P [M + Na]\(^+\) 412.1073, found 412.1079.
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (90% yield) as a yellow oil. \( R_f \) (ethyl acetate: petroleum ether, 2:1): 0.35. \(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \) 7.80 - 7.74 (m, 4H), 7.52 - 7.47 (m, 2H), 7.44 - 7.40 (m, 4H), 7.12 (d, \( J = 8.4 \text{ Hz}, 1H \)), 6.99 (d, \( J = 7.2 \text{ Hz}, 1H \)), 6.87 (t, \( J = 7.6 \text{ Hz}, 1H \)), 4.13 (s, 3H), 3.76 (q, \( J = 7.2 \text{ Hz}, 2H \)), 2.77 (s, 3H), 1.09 (t, \( J = 7.2 \text{ Hz}, 3H \)). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \( \delta \) 162.2, 137.6 (d, \( J_{\text{C-P}} = 16.0 \text{ Hz} \)), 137.0 (d, \( J_{\text{C-P}} = 11.0 \text{ Hz} \)), 134.3 (d, \( J_{\text{C-P}} = 109.0 \text{ Hz} \)), 132.0, 131.9, 131.8, 131.6, 131.4 (d, \( J_{\text{C-P}} = 2.0 \text{ Hz} \)), 129.3 (d, \( J_{\text{C-P}} = 8.0 \text{ Hz} \)), 128.5, 128.3 (d, \( J_{\text{C-P}} = 6.0 \text{ Hz} \)), 128.2, 127.3, 121.9, 121.8, 120.7, 106.0 (d, \( J_{\text{C-P}} = 121.0 \text{ Hz} \)), 61.9, 34.9, 20.5, 13.5. \(^{31}\)P NMR (162 MHz, CDCl\(_3\)): \( \delta \) 21.57. HRMS Calcd for \( \text{C}_{25}\text{H}_{24}\text{NNaO}_3\text{P} \) [M + Na]\(^+\) 440.1410, found 440.1398.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (90% yield) as a white solid. Mp: 163-165 °C. \( R_f \) (ethyl acetate: petroleum ether, 2:1): 0.27. \(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \) 7.81 - 7.76 (m, 4H), 7.51 - 7.48 (m, 2H), 7.45 - 7.40 (m, 4H), 7.19 (s, 1H), 6.90 (d, \( J = 8.4 \text{ Hz}, 1H \)), 6.82 (t, \( J = 8.4 \text{ Hz}, 1H \)), 3.94 (s, 3H), 3.76 (q, \( J = 7.2 \text{ Hz}, 2H \)), 2.43 (s, 3H), 1.03 (t, \( J = 7.2 \text{ Hz}, 3H \)). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \( \delta \) 161.6, 138.7 (d, \( J_{\text{C-P}} = 11.0 \text{ Hz} \)), 135.1 (d, \( J_{\text{C-P}} = 109.0 \text{ Hz} \)), 134.9, 134.8 (d, \( J_{\text{C-P}} = 12.0 \text{ Hz} \)), 131.4 (d, \( J_{\text{C-P}} = 10.0 \text{ Hz} \)), 131.3 (d, \( J_{\text{C-P}} = 3.0 \text{ Hz} \)), 128.2 (d, \( J_{\text{C-P}} = 12.0 \text{ Hz} \)), 126.5 (d, \( J_{\text{C-P}} = 9.0 \text{ Hz} \)), 123.9, 122.3, 110.0, 107.9 (d, \( J_{\text{C-P}} = 121.0 \text{ Hz} \)), 61.5, 31.7, 21.7, 13.4. \(^{31}\)P NMR (162 MHz, CDCl\(_3\)): \( \delta \) 21.60. HRMS Calcd for \( \text{C}_{25}\text{H}_{24}\text{NNaO}_3\text{P} \) [M + Na]\(^+\) 440.1386, found 440.1382.
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2:1) give the product (89% yield) as a yellow oil. \( R_f \) (ethyl acetate: petroleum ether, 2:1): 0.32. \(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \) 7.76 - 7.70 (m, 4H), 7.49 - 7.45 (m, 2H), 7.42 - 7.38 (m, 4H), 7.27 - 7.23 (m, 2H), 6.96 - 6.93 (m, 1H), 3.76 (s, 3H), 3.47 (q, \( J = 7.2 \text{ Hz} \), 2H), 2.73 (s, 3H), 1.11 (t, \( J = 7.2 \text{ Hz} \), 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \( \delta \) 162.4, 138.2 (d, \( J_{C-P} = 10.0 \text{ Hz} \)), 138.0, 135.0 (d, \( J_{C-P} = 108.0 \text{ Hz} \)), 133.7, 132.0 (d, \( J_{C-P} = 10.0 \text{ Hz} \)), 131.3 (d, \( J_{C-P} = 2.0 \text{ Hz} \)), 128.1, 128.0 (d, \( J_{C-P} = 6.0 \text{ Hz} \)), 124.5, 124.3, 107.6, 104.9 (d, \( J_{C-P} = 120.0 \text{ Hz} \)), 61.9, 31.5, 22.6, 13.6. \(^{31}\)P NMR (162 MHz, CDCl\(_3\)): \( \delta \) 22.36. HRMS Calcd for C\(_{25}\)H\(_{24}\)NNaO\(_3\)P \([M + Na]^{+}\) 440.1386, found 440.1398.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2:1) give the product (91% yield) as a white solid. Mp: 77-79 °C. \( R_f \) (ethyl acetate: petroleum ether, 2:1): 0.33. \(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \) 7.79 - 7.76 (m, 4H), 7.52 - 7.48 (m, 2H), 7.45 - 7.40 (m, 4H), 7.30 - 7.27 (m, 1H), 7.15 - 7.11 (m, 1H), 6.97 (s, 1H), 3.93 (s, 3H), 3.72 (q, \( J = 7.2 \text{ Hz} \), 2H), 2.22 (t, \( J = 7.2 \text{ Hz} \), 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \( \delta \) 161.5, 136.7 (d, \( J_{C-P} = 11.0 \text{ Hz} \)), 135.0 (d, \( J_{C-P} = 17.0 \text{ Hz} \)), 134.8 (d, \( J_{C-P} = 109.0 \text{ Hz} \)), 131.5 (d, \( J_{C-P} = 10.0 \text{ Hz} \)), 131.3 (d, \( J_{C-P} = 3.0 \text{ Hz} \)), 129.0 (d, \( J_{C-P} = 9.0 \text{ Hz} \)), 128.2 (d, \( J_{C-P} = 12.0 \text{ Hz} \)), 128.1, 126.5, 109.9, 106.7 (d, \( J_{C-P} = 120.0 \text{ Hz} \)), 61.5, 31.9, 21.3, 13.4. \(^{31}\)P NMR (162 MHz, CDCl\(_3\)): \( \delta \) 22.48. HRMS Calcd for C\(_{25}\)H\(_{24}\)NNaO\(_3\)P \([M + Na]^{+}\) 440.1386, found 440.1398.
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (96% yield) as a white solid. Mp: 145-147 °C. Rf (ethyl acetate: petroleum ether, 2: 1): 0.26. \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 7.84 - 7.78 (m, 4H), 7.51 - 7.42 (m, 6H), 7.28 (d, \(J = 9.6\) Hz, 1H), 6.93 (dd, \(J = 9.2\) Hz, 2.4Hz, 1H), 6.38 (d, \(J = 2.0\) Hz, 1H), 3.95 (s, 3H), 3.76 (q, \(J = 7.2\) Hz, 2H), 3.42 (s, 3H), 1.02 (t, \(J = 7.2\) Hz, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) 161.3, 155.2, 135.1 (d, \(J_{C-P} = 108.0\) Hz), 134.9 (d, \(J_{C-P} = 16.0\) Hz), 133.5 (d, \(J_{C-P} = 11.0\) Hz), 131.5, 131.4, 131.2 (d, \(J_{C-P} = 3.0\) Hz), 129.1 (d, \(J_{C-P} = 9.0\) Hz), 128.3, 128.2, 116.4, 111.1, 106.7 (d, \(J_{C-P} = 120.0\) Hz), 102.5, 61.4, 55.1, 32.0, 13.3. \(^{31}\)P NMR (162 MHz, CDCl\(_3\)): \(\delta\) 21.25. HRMS Calcd for C\(_{25}\)H\(_{24}\)N\(_2\)NaO\(_3\)P [M + Na]+ 456.1335, found 456.1338.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (87% yield) as a white solid. Mp: 152-154 °C. Rf (ethyl acetate: petroleum ether, 2:1): 0.25. \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 7.81 - 7.76 (m, 4H), 7.57 - 7.52 (m, 2H), 7.49 - 7.43 (m, 4H), 7.37 - 7.33 (m, 1H), 7.07 (td, \(J = 8.8\) Hz, 2.4Hz, 1H), 6.70 (dd, \(J = 10.0\) Hz, 6.4 Hz, 1H), 3.97 (s, 3H), 3.78 (q, \(J = 7.2\) Hz, 2H), 1.03 (t, \(J = 7.2\) Hz, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) 161.2, 158.6 (d, \(J_{C-P} = 237.0\) Hz), 136.6 (d, \(J_{C-P} = 16.0\) Hz), 134.8 (d, \(J_{C-P} = 11.0\) Hz), 133.9 (d, \(J_{C-P} = 109.0\) Hz), 131.8 (d, \(J_{C-P} = 3.0\) Hz), 131.6, 131.5, 129.1 (d, \(J_{C-P} = 11.0\) Hz), 129.0, 128.5, 128.4, 113.8 (d, \(J_{C-P} = 27.0\) Hz), 111.4 (d, \(J_{C-P} = 10.0\) Hz), 107.6 (d, \(J_{C-P} = 25.0\) Hz), 106.8 (d, \(J_{C-P} = 122.0\) Hz), 61.9, 32.2, 13.5. \(^{31}\)P NMR (162 MHz, CDCl\(_3\)): \(\delta\) 22.78. \(^{19}\)F NMR (376 MHz, CDCl\(_3\)): \(\delta\) -119.88. HRMS Calcd for C\(_{24}\)H\(_{21}\)FNNaO\(_3\)P [M + Na]+ 444.1135, found 444.1128.
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (90% yield) as a white solid. Mp: 149-151 °C. RF (ethyl acetate: petroleum ether, 2:1): 0.25. 1H NMR (400 MHz, CDCl3): δ 7.81 - 7.75 (m, 4H), 7.55 - 7.51 (m, 2H), 7.47 - 7.43 (m, 4H), 7.33 - 7.22 (m, 2H), 7.19 (s, 1H), 3.94 (s, 3H), 3.75 (q, J = 7.2 Hz, 2H), 1.03 (t, J = 7.2 Hz, 3H). 13C NMR (100 MHz, CDCl3): δ 161.4, 136.8 (d, J_C-P = 11.0 Hz), 136.1 (d, J_C-P = 10.0 Hz), 133.9 (d, J_C-P = 2.0 Hz), 131.8 (d, J_C-P = 10.0 Hz), 129.9 (d, J_C-P = 9.0 Hz), 128.7, 128.6, 128.1, 125.5, 122.6, 111.7, 107.5 (d, J_C-P = 119.0 Hz), 62.2, 32.4, 13.7. 31P NMR (162 MHz, CDCl3): δ 22.03. HRMS Calcd for C24H21ClNO3P [M + Na]+ 460.0840, found 460.0828.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (81% yield) as a white solid. Mp: 162-164 °C. RF (ethyl acetate: petroleum ether, 2:1): 0.22. 1H NMR (400 MHz, CDCl3): δ 7.78 - 7.74 (m, 4H), 7.56 - 7.52 (m, 2H), 7.48 - 7.43 (m, 4H), 7.40 - 7.36 (m, 1H), 7.31 - 7.25 (m, 2H), 3.94 (s, 3H), 3.76 (q, J = 7.2 Hz, 2H), 1.03 (t, J = 7.2 Hz, 3H). 13C NMR (100 MHz, CDCl3): δ 161.1, 136.8 (d, J_C-P = 10.0 Hz), 136.1 (d, J_C-P = 6.0 Hz), 134.2 (d, J_C-P = 109.0 Hz), 131.7 (d, J_C-P = 2.0 Hz), 131.6, 131.5, 130.2 (d, J_C-P = 8.0 Hz), 128.4, 128.3, 127.8, 125.5, 115.5, 111.7, 107.3 (d, J_C-P = 119.0 Hz), 61.9, 32.1, 13.4. 31P NMR (162 MHz, CDCl3): δ 21.99. HRMS Calcd for C24H21BrNaO3P [M + Na]+ 504.0355, found 504.0325.
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2:1) give the product (81% yield) as a white solid. Mp: 158-160 °C. Rf (ethyl acetate: petroleum ether, 2:1): 0.35. 1H NMR (400 MHz, CDCl3): δ 7.81 - 7.75 (m, 4H), 7.57 - 7.53 (m, 2H), 7.51 - 7.44 (m, 7H), 7.31 - 7.27 (m, 1H), 4.01 (s, 3H), 3.82 (q, J = 7.2 Hz, 2H), 1.04 (t, J = 7.2 Hz, 3H). 13C NMR (100 MHz, CDCl3): δ 161.1, 139.4 (d, J_{C-P} = 10.0 Hz), 137.1 (d, J_{C-P} = 15.0 Hz), 134.2 (d, J_{C-P} = 110.0 Hz), 132.0, 131.8 (d, J_{C-P} = 3.0 Hz), 131.6, 131.5, 131.4, 131.3, 128.6, 128.5 (d, J_{C-P} = 5.0 Hz), 128.4, 127.9 (d, J_{C-P} = 9.0 Hz), 124.4 (d, J_{C-P} = 270.0 Hz), 124.3 (q, J_{C-P} = 32.0 Hz), 121.2 (d, J_{C-P} = 4.0 Hz), 110.9, 109.2 (d, J_{C-P} = 118.0 Hz), 62.1, 32.2, 13.4. 31P NMR (162 MHz, CDCl3): δ 21.35. HRMS Calcd for C_{25}H_{21}F_{3}NO_{3}P [M + Na]^+ 494.1103, found 494.1104.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2:1) give the product (61% yield) as a yellow oil. Rf (ethyl acetate: petroleum ether, 2:1): 0.18. 1H NMR (400 MHz, DMSO): δ 12.55 (brs, 1H), 9.03 (brs, 1H), 7.70 - 7.64 (m, 4H), 7.61 - 7.47 (m, 6H), 7.38 (d, J = 7.2 Hz, 1H), 7.03 (d, J = 2.0 Hz, 1H), 6.85 (dd, J = 8.8 Hz, 2.4 Hz, 1H), 3.77 (q, J = 7.2 Hz, 2H), 0.88 (t, J = 7.2 Hz, 3H). 13C NMR (100 MHz, DMSO): δ 160.1, 152.2, 135.9, 134.8, 134.8, 131.5 131.3, 131.2, 131.1, 130.9, 130.8, 128.3, 128.2, 116.4, 113.4, 105.8, 60.7, 13.5. 31P NMR (162 MHz, DMSO): δ 22.06. HRMS Calcd for C_{23}H_{20}NNO_{3}P [M + Na]^+ 428.1022, found 428.1020.
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (89% yield) as a white solid. Mp: 187-189 °C. Rf (ethyl acetate: petroleum ether, 2:1): 0.34. **1H NMR** (400 MHz, CDCl₃): δ 7.78 - 7.73 (m, 4H), 7.54 - 7.49 (m, 2H), 7.46 - 7.43 (m, 4H), 7.23 (s, 1H), 6.94 (s, 1H), 4.08 (s, 3H), 3.72 (q, J = 7.2 Hz, 2H), 2.71 (s, 3H), 1.06 (t, J = 7.2 Hz, 3H). **13C NMR** (100 MHz, CDCl₃): δ 161.7, 138.0 (d, J_C-P = 12.0 Hz), 135.9 (d, J_C-P = 146.0 Hz), 131.4 (d, J_C-P = 2.0 Hz), 130.7, 130.6, 128.3, 128.2, 127.9, 124.7, 123.1, 122.3, 110.3, 106.6 (d, J_C-P = 157.0 Hz), 61.8, 60.5 (d, J_C-P = 6.0 Hz), 16.4 (d, J_C-P = 6.0 Hz), 13.6. **31P NMR** (162 MHz, CDCl₃): δ 21.98. **HRMS** Calcd for C₂₅H₂₃ClINaO₃P [M + Na]+: 474.0996, found 474.0988.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (69% yield) as a yellow oil. Rf (ethyl acetate: petroleum ether, 2:1): 0.33. **1H NMR** (400 MHz, CDCl₃): δ 8.25 (d, J = 8.0 Hz, 1H), 7.88 - 7.82 (m, 2H), 7.48 - 7.36 (m, 5H), 7.30 - 7.25 (m, 1H), 4.30 - 4.22 (m, 1H), 4.19 - 4.05 (m, 1H), 3.94 (s, 3H), 1.37 (t, J = 7.2 Hz, 3H), 1.21 (t, J = 7.2 Hz, 3H). **13C NMR** (100 MHz, CDCl₃): δ 161.7, 138.0 (d, J_C-P = 12.0 Hz), 135.6 (d, J_C-P = 20.0 Hz), 133.9 (d, J_C-P = 146.0 Hz), 131.4 (d, J_C-P = 2.0 Hz), 130.7, 130.6, 128.3, 128.2, 127.9, 124.7, 123.1, 122.3, 110.3, 106.6 (d, J_C-P = 157.0 Hz), 61.8, 60.5 (d, J_C-P = 6.0 Hz), 16.4 (d, J_C-P = 6.0 Hz), 13.6. **31P NMR** (162 MHz, CDCl₃): δ 29.06. **HRMS** Calcd for C₂₀H₂₂NNaO₄P [M + Na]+: 394.1179, found 394.1180.
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (22% yield) as a yellow oil. \textbf{R}_f (ethyl acetate: petroleum ether, 4:1): 0.35. \textbf{H NMR (400 MHz, DMSO)}: δ 12.62 (brs, 1H), 10.56 (brs, 1H), 7.82 (brs, 1H), 7.69 - 7.64 (m, 2H), 7.61 - 7.53 (m, 9H), 7.21 - 7.17 (m, 1H), 6.87 - 6.82 (m, 1H), 6.37 (d, \( J = 8.4 \) Hz, 1H). \textbf{13C NMR (100 MHz, DMSO)}: δ 161.6, 139.6 (d, \( J_{C-P} = 16.0 \) Hz), 135.9 (d, \( J_{C-P} = 12.0 \) Hz), 133.9 (d, \( J_{C-P} = 2.0 \) Hz), 132.8, 131.8, 131.7, 129.5, 129.4, 129.3, 129.2, 124.4, 121.9, 121.3, 113.8, 101.7 (d, \( J_{C-P} = 119.0 \) Hz). \textbf{31P NMR (162 MHz, DMSO)}: δ 26.10. \textbf{HRMS} Calcd for C_{21}H_{18}N_{2}O_{2}P [M + H]^+ 361.1100, found 361.1105.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (95% yield) as a white solid. Mp: 191-193 °C. \textbf{R}_f (ethyl acetate: petroleum ether, 2:1): 0.35. \textbf{H NMR (400 MHz, CDCl₃)}: δ 7.78 - 7.72 (m, 4H), 7.51 - 7.48 (m, 2H), 7.43 - 7.36 (m, 5H), 7.28 - 7.24 (m, 1H), 6.96 (t, \( J = 7.6 \) Hz, 1H), 6.79 (t, \( J = 8.4 \) Hz, 1H), 3.75 (s, 3H), 2.61 (s, 3H). \textbf{13C NMR (100 MHz, CDCl₃)}: δ 198.1, 145.3 (d, \( J_{C-P} = 17.0 \) Hz), 137.5 (d, \( J_{C-P} = 11.0 \) Hz), 133.9 (d, \( J_{C-P} = 109.0 \) Hz), 131.7 (d, \( J_{C-P} = 2.0 \) Hz), 131.6 (d, \( J_{C-P} = 11.0 \) Hz), 128.4 (d, \( J_{C-P} = 13.0 \) Hz), 127.6 (d, \( J_{C-P} = 10.0 \) Hz), 123.9, 121.8, 110.4, 104.2 (d, \( J_{C-P} = 120.0 \) Hz), 33.5, 31.3. \textbf{31P NMR (162 MHz, CDCl₃)}: δ 21.36. \textbf{HRMS} Calcd for C_{23}H_{20}NaO_{2}P [M + Na]^+ 396.1124, found 396.1123.
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2:1) give the product (96% yield) as a yellow oil. $R_f$ (ethyl acetate: petroleum ether, 2:1): 0.28. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 10.83 (brs, 1H), 7.77 - 7.71 (m, 4H), 7.60 - 7.55 (m, 2H), 7.50 - 7.45 (m, 5H), 7.42 - 7.37 (m, 1H), 7.03 - 6.98 (m, 1H), 6.80 (d, $J = 8.4$ Hz, 1H), 4.19 (s, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 186.8, 140.0 (d, $J_{C-P} = 15.0$ Hz), 139.4 (d, $J_{C-P} = 12.0$ Hz), 133.8 (d, $J_{C-P} = 108.0$ Hz), 132.2 (d, $J_{C-P} = 3.0$ Hz), 132.1, 132.0, 131.8, 131.7, 129.0, 128.8, 128.7, 127.9, 127.8, 126.6, 122.8, 122.4, 114.8 (d, $J_{C-P} = 115.0$ Hz), 111.0, 32.8. $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 23.19. HRMS Calcd for C$_{22}$H$_{18}$NNaO$_2$P [M + Na]$^+$ 382.0967, found 382.0957.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2:1) give the product (93% yield) as a white solid. Mp: 201-203 °C. $R_f$ (ethyl acetate: petroleum ether, 2:1): 0.35. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.85 - 7.74 (m, 4H), 7.68 - 7.61 (m, 1H), 7.58 - 7.54 (m, 2H), 7.50 - 7.44 (m, 4H), 7.41 - 7.38 (m, 2H), 7.18 - 7.12 (m, 1H), 3.88 (d, $J = 6.0$ Hz, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 138.2 (d, $J_{C-P} = 11.0$ Hz), 132.6, 132.3 (d, $J_{C-P} = 3.0$ Hz), 128.5 (d, $J_{C-P} = 12.0$ Hz), 126.4, 122.9, 122.8, 110.9 (d, $J_{C-P} = 1.0$ Hz), 110.4, 76.7 (d, $J_{C-P} = 3.0$ Hz), 31.8. $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 19.71. HRMS Calcd for C$_{22}$H$_{17}$N$_2$NaOP [M + Na]$^+$ 379.0971, found 379.0958.
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2:1) give the product (86% yield) as a yellow oil. 

\[ R_f \] (ethyl acetate: petroleum ether, 4:1): 0.38. 

**1H NMR (400 MHz, CDCl\(_3\))**:

\[ \delta \] 11.43 (brs, 1H), 11.29 (brs, 1H), 7.73 - 7.67 (m, 4H), 7.62 - 7.55 (m, 3H), 7.48 - 7.44 (m, 4H), 7.26 - 7.21 (m, 1H), 6.92 - 6.87 (m, 1H), 6.57 (d, \( J = 8.0 \) Hz, 1H), 3.07 (d, \( J = 4.0 \) Hz, 3H).

**13C NMR (100 MHz, CDCl\(_3\))**:

\[ \delta \] 160.0, 139.2 (d, \( J_{C-P} = 15.0 \) Hz), 133.3, 132.3 (d, \( J_{C-P} = 3.0 \) Hz), 132.1, 132.0, 131.9, 131.8, 129.5 (d, \( J_{C-P} = 12.0 \) Hz), 128.8, 128.7, 124.3, 121.7, 121.6, 112.8, 101.1 (d, \( J_{C-P} = 121.0 \) Hz), 26.9.

**31P NMR (162 MHz, CDCl\(_3\))**:

\[ \delta \] 28.39.

**HRMS** Calcd for C\(_{22}\)H\(_{20}\)N\(_2\)O\(_2\)P \([M + H]^+\] 375.1257, found 375.1263.

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Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2:1) give the product (78% yield) as a white solid. Mp: 173-175 °C. 

\[ R_f \] (ethyl acetate: petroleum ether, 2:1): 0.50. 

**1H NMR (400 MHz, d\(_6\)-DMSO)**:

\[ \delta \] 12.31 (brs, 1H), 8.33 (s, 1H), 7.65 - 7.59 (m, 4H), 7.54 - 7.48 (m, 3H), 7.43 - 7.38 (m, 2H), 7.34 - 7.30 (m, 4H), 7.20 - 7.10 (m, 4H), 6.82 (t, \( J = 7.6 \) Hz, 1H), 6.52 (d, \( J = 8.4 \) Hz, 1H). 

**13C NMR (100 MHz, d\(_6\)-DMSO)**:

\[ \delta \] 147.1 (d, \( J_{C-P} = 18.0 \) Hz), 137.0 (d, \( J_{C-P} = 12.0 \) Hz), 135.5 (d, \( J_{C-P} = 106.0 \) Hz), 132.0, 131.9, 131, 131.5, 130.2, 130.0 (d, \( J_{C-P} = 5.0 \) Hz), 128.8, 128.6 (d, \( J_{C-P} = 12.0 \) Hz), 128.0, 122.5, 120.7 (d, \( J_{C-P} = 3.0 \) Hz), 101.0 (d, \( J_{C-P} = 124.0 \) Hz), 79.6.

**31P NMR (162 MHz, d\(_6\)-DMSO)**:

\[ \delta \] 16.87. 

**HRMS** Calcd for C\(_{26}\)H\(_{21}\)NOP \([M + H]^+\] 394.1355, found 394.1362.
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (73% yield) as a yellow oil. \( R_f \) (ethyl acetate: petroleum ether, 2:1): 0.39. \(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \) 8.50 (d, \( J = 4.4 \) Hz, 1H), 7.71 - 7.65 (m, 4H), 7.52 (d, \( J = 7.6 \) Hz, 1H), 7.42 (d, \( J = 8.4 \) Hz, 1H), 7.38 - 7.31 (m, 3H), 7.30 - 7.22 (m, 5H), 7.05 - 7.01 (m, 1H), 6.99 - 6.97 (m, 2H), 3.70 (s, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \( \delta \) 149.8, 148.8, 145.8 (d, \( J_{C-P} = 18.0 \) Hz), 137.9 (d, \( J_{C-P} = 11.0 \) Hz), 135.7, 134.5 (d, \( J_{C-P} = 107.0 \) Hz), 131.7, 131.6, 131.0 (d, \( J_{C-P} = 3.0 \) Hz), 129.0 (d, \( J_{C-P} = 9.0 \) Hz), 128.2 (d, \( J_{C-P} = 2.0 \) Hz), 128.1, 123.1, 121.9, 110.0, 103.4 (d, \( J_{C-P} = 125.0 \) Hz), 31.3. \(^{31}\)P NMR (162 MHz, CDCl\(_3\)): \( \delta \) 18.72. HRMS Calcd for C\(_{25}\)H\(_{22}\)N\(_2\)OP [M + H]\(^+\) 409.1464, found 409.1471.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (77% yield) as a yellow oil. \( R_f \) (ethyl acetate: petroleum ether, 2:1): 0.45. \(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \) 7.88 - 7.82 (m, 4H), 7.51 - 7.39 (m, 7H), 7.36 - 7.26 (m, 2H), 7.03 (t, \( J = 8.0 \) Hz, 1H), 3.97 (s, 3H), 3.58 - 3.42 (m, 3H), 1.61 - 1.48 (m, 1H), 0.95 (d, \( J = 4.0 \) Hz, 1H), 0.82 (t, \( J = 8.0 \) Hz, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \( \delta \) 156.5, 138.3 (d, \( J_{C-P} = 11.0 \) Hz), 134.7 (d, \( J_{C-P} = 108.0 \) Hz), 132.8 (d, \( J_{C-P} = 16.0 \) Hz), 131.7, 131.6 (d, \( J_{C-P} = 3.0 \) Hz), 131.5, 131.2 (d, \( J_{C-P} = 3.0 \) Hz), 131.2 (d, \( J_{C-P} = 2.0 \) Hz), 129.1 (d, \( J_{C-P} = 9.0 \) Hz), 128.3 (d, \( J_{C-P} = 5.0 \) Hz), 128.2 (d, \( J_{C-P} = 5.0 \) Hz), 124.2, 122.8, 121.7, 110.2, 106.7 (d, \( J_{C-P} = 122.0 \) Hz), 73.3, 69.9, 32.7, 31.7, 19.1, 18.7. \(^{31}\)P NMR (162 MHz, CDCl\(_3\)): \( \delta \) 20.32.
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (88% yield) as a white solid. Mp: 65-67 °C. Rf (ethyl acetate: petroleum ether, 1:2): 0.37. [α]D16 = +13.0 (c 1.00, CHCl3). 1H NMR (400 MHz, CDCl3): δ 8.35 (d, J = 8.4 Hz, 1H), 7.84 - 7.78 (m, 2H), 7.46 - 7.35 (m, 5H), 7.28 - 7.23 (m, 1H), 4.44 - 4.40 (m, 1H), 4.25 - 4.09 (m, 2H), 3.96 (s, 1H), 2.13 - 2.08 (m, 1H), 1.92 (d, J = 12 Hz, 1H), 1.66 - 1.61 (m, 2H), 1.49 - 1.42 (t, J = 11.2 Hz, 1H), 1.37 - 1.31 (m, 1H), 1.24 - 1.14 (m, 4H), 1.00 - 0.83 (m, 2H), 0.81 (d, J = 6.8 Hz, 3H), 0.79 (d, J = 6.8 Hz, 3H), 0.51 (d, J = 6.8 Hz, 3H).

13C NMR (100 MHz, CDCl3): δ 161.6, 138.1 (d, JCP = 12.0 Hz), 135.8 (d, JCP = 143.0 Hz), 134.6 (d, JCP = 20.0 Hz), 131.1 (d, JCP = 3.0 Hz), 130.6 (d, JCP = 11.0 Hz), 128.6 (d, JCP = 8.0 Hz), 127.9 (d, JCP = 13.0 Hz), 124.7, 123.8, 122.0, 110.1, 108.0 (d, JCP = 159.0 Hz), 76.3 (d, JCP = 7.0 Hz), 61.5, 49.0 (d, JCP = 5.0 Hz), 43.4, 34.1, 31.8, 31.5, 25.2, 22.7, 22.0, 21.1, 15.3, 13.7. 31P NMR (162 MHz, CDCl3): δ 28.14. HRMS Calcd for C28H37NO4P [M + H]+ 482.2455, found 482.2458.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (65% yield) as a white solid. Mp: 232-234 °C. Rf (ethyl acetate: petroleum ether, 1:2): 0.25. [α]D16 = +2.0 (c 1.00, CHCl3). 1H NMR (400 MHz, CDCl3): δ 10.61 (b, 1H), 8.58 (d, J = 8.0 Hz, 1H), 7.83 - 7.77 (m, 2H), 7.44 - 7.40 (m, 1H), 7.35 - 7.30 (m, 3H), 7.27 - 7.23 (m, 1H), 4.49 - 4.40 (m, 1H), 4.07 - 4.03 (m, 2H), 2.18 - 2.12 (m, 1H), 1.92 (d, J = 10.4 Hz, 1H), 1.63 - 1.61 (m, 2H), 1.51 (t, J = 11.2 Hz, 1H), 1.31 - 1.16 (m, 2H), 1.04 - 0.77 (m, 11H), 0.51 (d, J = 6.8 Hz, 3H). 13C NMR (100 MHz, CDCl3): δ 160.5, 136.7 (d, JCP = 145.0 Hz), 133.6 (d, JCP = 12.0 Hz), 134.6, 131.4, 131.2 (d, JCP = 3.0 Hz), 130.8, 130.7, 130.6, 130.5, 127.9, 127.8, 125.7, 124.6 122.3, 111.8, 109.6 (d, JCP = 156.0 Hz), 76.4 (d, JCP = 7.0 Hz), 61.6, 49.0 (d, JCP = 5.0 Hz), 43.6, 34.1, 31.5, 25.4, 22.7, 22.0, 21.1, 15.3, 13.9. 31P NMR (162 MHz, CDCl3): δ 29.26. HRMS Calcd for C27H35NO4P [M + H]+ 468.2298, found 468.2291.
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (86% yield) as a white solid. Mp: 60-62 °C. Rf (ethyl acetate: petroleum ether, 1:2): 0.22. [α]D16 = +85.0 (c 1.00, CHCl3). 1H NMR (400 MHz, CDCl3): δ 7.89 - 7.78 (m, 3H), 7.49 - 7.44 (m, 1H), 7.48 - 7.37 (m, 3H), 7.34 - 7.26 (m, 1H), 4.34 - 4.25 (m, 1H), 3.73 (s, 3H), 2.80 (s, 3H), 2.06 - 1.98 (m, 2H), 1.63 - 1.60 (m, 2H), 1.42 (t, J = 11.2 Hz, 1H), 1.35 - 1.30 (m, 1H), 1.26 - 1.20 (m, 1H), 0.99 - 0.82 (m, 2H), 0.81 - 0.77 (m, 3H), 0.73 (d, J = 7.2 Hz, 3H), 0.46 (d, J = 6.8 Hz, 3H). 13C NMR (100 MHz, CDCl3): δ 161.8, 138.1 (d, Jc,p = 13.0 Hz), 135.6 (d, Jc,p = 143.0 Hz), 134.0 (d, Jc,p = 21.0 Hz), 132.3, 131.1 (d, Jc,p = 3.0 Hz), 130.8, 130.5, 130.4, 128.7, 128.3, 127.9, 127.8, 124.8, 123.7, 122.1, 110.2, 108.8 (d, Jc,p = 159.0 Hz), 76.2 (d, Jc,p = 7.0 Hz), 68.0, 49.0 (d, Jc,p = 6.0 Hz), 43.3, 34.0, 31.7, 31.4, 25.2, 22.9, 21.9, 21.0, 15.3. 31P NMR (162 MHz, CDCl3): δ 28.01. HRMS Calcd for C27H35NO4P [M + H]+ 468.2298, found 468.2292.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (67% yield) as a white solid. Mp: 227-229 °C. Rf (ethyl acetate: petroleum ether, 1:2): 0.19. [α]D16 = -110.0 (c 1.00, CHCl3). 1H NMR (400 MHz, CDCl3): δ 10.49 (brs, 1H), 8.56 - 8.53 (m, 1H), 7.85 - 7.79 (m, 2H), 7.51 - 7.40 (m, 1H), 7.36 - 7.31 (m, 3H), 7.28 - 7.23 (m, 1H), 4.44 - 4.41 (m, 1H), 3.54 (s, 3H), 2.23 - 2.10 (m, 1H), 1.87 - 1.84 (m, 1H), 1.67 - 1.51 (m, 3H), 1.26 - 1.17 (m, 2H), 1.02 - 0.87 (m, 2H), 0.82 (d, J = 6.8 Hz, 3H), 0.76 (d, J = 6.4 Hz, 3H), 0.57 (d, J = 6.4 Hz, 3H). 13C NMR (100 MHz, CDCl3): δ 160.8, 136.2, 136.0, 134.7, 131.6, 131.4, 130.8, 130.6, 130.4, 130.3, 127.8, 127.7, 125.4, 124.2 122.1, 112.3, 109.1 (d, Jc,p = 156.0 Hz), 76.4 (d, Jc,p = 6.0 Hz), 61.5, 48.9 (d, Jc,p = 5.0 Hz), 43.5, 34.0, 31.4, 25.3, 22.6, 21.9, 21.0, 15.2. 31P NMR (162 MHz, CDCl3): δ 28.54. HRMS Calcd for C25H33NO4P [M + H]+ 454.2142, found 454.2147.
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (85% yield) as a yellow oil. Rf (ethyl acetate: petroleum ether, 1:2): 0.45. [α]D16 = +10.0 (c 1.00, CHCl3).  

**1H NMR (400 MHz, CDCl3):** δ 8.20 (s, 1H), 7.83 - 7.77 (m, 2H), 7.45 - 7.37 (m, 3H), 7.30 - 7.27 (m, 1H), 7.20 (d, J = 8.4 Hz, 1H), 4.48 - 4.39 (m, 1H), 4.22 - 4.03 (m, 2H), 3.93 (s, 3H), 2.48 (s, 3H), 2.17 - 2.10 (m, 1H), 1.95 (d, J = 12.0 Hz, 1H), 1.64 (d, J = 10.8 Hz, 1H), 1.49 - 1.36 (m, 2H), 1.26 - 1.16 (m, 1H), 1.21 - 1.11 (m, 4H), 1.05 - 0.85 (m, 2H), 0.83 (d, J = 7.2 Hz, 3H), 0.81 (d, J = 6.4 Hz, 3H), 0.55 (d, J = 7.2 Hz, 3H), 13C NMR (100 MHz, CDCl3): δ 161.5, 136.6, 136.6 (d, JC-P = 12.0 Hz), 136.0 (d, JC-P = 144.0 Hz), 134.1(d, JC-P = 21.0 Hz), 131.5, 131.0 (d, JC-P = 3.0 Hz), 130.4 (d, JC-P = 11.0 Hz), 128.9 (d, JC-P = 9.0 Hz), 127.9, 127.7, 126.7, 123.1 109.7, 107.4 (d, JC-P = 159.0 Hz), 76.2 (d, JC-P = 6.0 Hz), 61.3, 48.9 (d, JC-P = 6.0 Hz), 43.4, 34.0, 31.8, 31.4, 25.2, 22.6, 22.0, 21.6, 21.0, 15.2, 13.7. 31P NMR

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (80% yield) as a yellow oil. Rf (ethyl acetate: petroleum ether, 1:2): 0.39. [α]D16 = -8.0 (c 1.00, CHCl3). 

**1H NMR (400 MHz, CDCl3):** δ 7.92 - 7.86 (m, 2H), 7.47 - 7.35 (m, 3H), 7.19 - 7.14 (m, 2H), 7.40 - 7.37 (m, 2H), 7.34 (s, 2H), 4.49 - 4.39 (m, 1H), 4.24 - 4.07 (m, 2H), 3.96 (s, 3H), 2.14 - 2.07 (m, 1H), 1.85 (d, J = 12 Hz, 1H), 1.68 - 1.61 (m, 2H), 1.51 - 1.45 (t, J = 11.2 Hz, 1H), 1.37 - 1.32 (m, 1H), 1.21 - 1.11 (m, 4H), 1.01 - 0.84 (m, 2H), 0.80 (d, J = 6.4 Hz, 3H), 0.71 (d, J = 7.2 Hz, 3H), 0.51 (d, J = 6.8 Hz, 3H). 

**13C NMR (100 MHz, CDCl3):** δ 163.9, 140.2 (d, JC-P = 16.0 Hz), 137.8 (d, JC-P = 12.0 Hz), 135.3 (d, JC-P = 138.0 Hz), 132.9, 131.5 (d, JC-P = 11.0 Hz), 131.4 (d, JC-P = 3.0 Hz), 128.1, 128.0, 126.8 (d, JC-P = 11.0 Hz), 123.8, 123.6, 107.5, 103.5 (d, JC-P = 158.0 Hz), 76.1 (d, JC-P = 7.0 Hz), 62.3, 48.8 (d, JC-P = 5.0 Hz), 43.2, 34.2, 31.5, 31.2, 25.0, 22.6, 22.0, 21.7, 21.0, 15.0, 14.0.  

**31P NMR (162 MHz, CDCl3):** δ 27.48. 

**HRMS** Calcd for C29H39NO4P [M + H]+ 496.2611, found 496.2615.
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (84% yield) as a white solid. Mp: 58-60 °C. R_f (ethyl acetate: petroleum ether, 1:2): 0.34. [α]_D^{16} = +24.0 (c 1.00, CHCl₃). ¹H NMR (400 MHz, CDCl₃): δ 7.89 (d, J = 2.4 Hz, 1H), 7.82 - 7.76 (m, 2H), 7.45 - 7.42 (m, 1H), 7.41 - 7.36 (m, 2H), 7.33 - 7.27 (m, 1H), 7.07 - 7.04 (m, 1H), 4.44 - 4.40 (m, 1H), 4.20 - 4.14 (m, 1H), 4.07 - 4.01 (m, 1H), 3.96 (s, 3H), 3.87 (s, 3H), 2.14 - 2.12 (m, 1H), 1.92 (d, J = 12.0 Hz, 1H), 1.64 (d, J = 11.6 Hz, 2H), 1.44 - 1.33 (m, 3H), 1.23 - 1.16 (m, 1H), 1.12 (t, J = 7.2 Hz, 3H), 1.00 - 0.85 (m, 2H), 0.83 - 0.79 (m, 3H), 0.55 (d, J = 6.8 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 161.4, 155.6, 136.0 (d, J_2-13C = 144.0 Hz), 134.0 (d, J_2-13C = 20.0 Hz), 133.5 (d, J_2-13C = 13.0 Hz), 131.3 (d, J_2-13C = 3.0 Hz), 130.4 (d, J_2-13C = 11.0 Hz), 129.4 (d, J_2-13C = 9.0 Hz), 128.0, 127.8, 116.4, 111.0 107.4 (d, J_2-13C = 159.0 Hz), 103.9, 76.1 (d, J_2-13C = 7.0 Hz), 61.3, 55.6, 49.2 (d, J_2-13C = 5.0 Hz), 43.7, 34.1, 32.1, 31.5, 25.3, 22.6, 22.0, 21.1, 15.2, 13.7. ³¹P NMR (162 MHz, CDCl₃): δ 29.11. HRMS Calcd for C₂₉H₃₉NO₄P [M + H]^+ 512.1560, found 512.2555.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (73% yield) as a yellow solid. Mp: 94-96 °C. R_f (ethyl acetate: petroleum ether, 1:2): 0.33. [α]_D^{16} = +26.0 (c 1.00, CHCl₃). ¹H NMR (400 MHz, CDCl₃): δ 8.05 (dd, J = 10.0 Hz, 2.4 Hz, 1H), 7.82 - 7.76 (m, 2H), 7.47 - 7.33 (m, 3H), 7.15 (td, J = 8.8 Hz, 2.4 Hz, 1H), 4.44 - 4.40 (m, 1H), 4.23 - 4.08 (m, 2H), 3.96 (s, 3H), 2.12 - 2.07 (m, 1H), 1.88 - 1.84 (m, 1H), 1.68 - 1.62 (m, 2H), 1.48 - 1.44 (m, 1H), 1.35 - 1.33 (m, 1H), 1.22 - 1.13 (m, 4H), 1.01 - 0.84 (m, 2H), 0.83 - 0.79 (m, 6H), 0.55 (d, J = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 161.2, 159.0 (d, J_2-13C = 236.0 Hz), 135.6 (d, J_2-13C = 20.0 Hz), 135.5 (d,
$J_{C,P} = 143.0$ Hz), 134.8 (d, $J_{C,P} = 11.0$ Hz), 131.3 (d, $J_{C,P} = 3.0$ Hz), 130.6 (d, $J_{C,P} = 11.0$ Hz), 129.1, 128.9 (d, $J_{C,P} = 8.0$ Hz), 128.1, 127.9, 114.4 (d, $J_{C,P} = 27.0$ Hz), 111.1 (d, $J_{C,P} = 10.0$ Hz), 108.2 (d, $J_{C,P} = 164.0$ Hz), 108.5 (d, $J_{C,P} = 26.0$ Hz), 76.5 (d, $J_{C,P} = 7.0$ Hz), 61.7, 49.0 (d, $J_{C,P} = 5.0$ Hz), 43.4, 34.1, 32.2, 31.5, 25.3, 22.7, 22.0, 21.0, 15.3, 13.7. \textsuperscript{31}P NMR (162 MHz, CDCl\textsubscript{3}): $\delta$ 27.98. \textsuperscript{19}F NMR (376 MHz, CDCl\textsubscript{3}): $\delta$ -120.53. HRMS Calcd for C\textsubscript{28}H\textsubscript{36}FNO\textsubscript{4}P [M + H]\textsuperscript{+} 500.2360, found 500.2367.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (78\% yield) as a yellow solid. Mp: 89-91 °C. $R_f$ (ethyl acetate: petroleum ether, 1:2): 0.33. $[\alpha]_D^{16} = +17.0$ (c 1.00, CHCl\textsubscript{3}). \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}): $\delta$ 8.39 (s, 1H), 7.82 - 7.76 (m, 2H), 7.48 - 7.40 (m, 1H), 7.40 - 7.37 (m, 2H), 7.34 (s, 2H), 4.49 - 4.39 (m, 1H), 4.24 - 4.07 (m, 2H), 3.96 (s, 3H), 2.14 - 2.07 (m, 1H), 1.85 (d, $J = 12$ Hz, 1H), 1.68 - 1.61 (m, 2H), 1.51 - 1.45 (t, $J = 11.2$ Hz, 1H), 1.37 - 1.32 (m, 1H), 1.21 - 1.11 (m, 4H), 1.05 - 0.85 (m, 2H), 0.83 (d, $J = 7.2$ Hz, 3H), 0.81 (d, $J = 6.4$ Hz, 3H), 0.55 (d, $J = 7.2$ Hz, 3H). \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}): $\delta$ 161.2, 136.6, 136.5, 135.6 (d, $J_{C,P} = 144.0$ Hz), 135.4 (d, $J_{C,P} = 21.0$ Hz), 134.9, 131.3 (d, $J_{C,P} = 2.0$ Hz), 130.6 (d, $J_{C,P} = 11.0$ Hz), 129.4 (d, $J_{C,P} = 8.0$ Hz), 128.1 (d, $J_{C,P} = 14.0$ Hz), 128.0, 125.4, 123.2 111.3, 108.1 (d, $J_{C,P} = 159.0$ Hz), 76.6 (d, $J_{C,P} = 7.0$ Hz), 61.7, 49.0 (d, $J_{C,P} = 5.0$ Hz), 43.4, 34.1, 33.2, 31.5, 25.4, 22.7, 22.0, 21.1, 15.4, 13.8. \textsuperscript{31}P NMR (162 MHz, CDCl\textsubscript{3}): $\delta$ 27.75. HRMS Calcd for C\textsubscript{28}H\textsubscript{36}ClNO\textsubscript{4}P [M + H]\textsuperscript{+} 516.2065, found 516.2072.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (83\% yield) as a white solid. Mp: 62-64 °C. $R_f$ (ethyl acetate: petroleum ether, 1:2): 0.31. $[\alpha]_D^{16} = +37.0$ (c 1.00, CHCl\textsubscript{3}). \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}): $\delta$ 8.57 (d, $J = 1.6$ Hz, 1H), 7.82 - 7.76 (m, 2H), 7.48 - 7.37 (m, 4H), 7.29 - 7.26 (m, 1H), 4.46 - 4.42 (m, 1H), 4.22 - 4.09 (m,
2H), 3.94 (s, 3H), 2.14 - 2.09 (m, 1H), 1.86 (d, J = 12.0 Hz, 1H), 1.69 - 1.61 (m, 2H),
1.52 - 1.32 (m, 2H), 1.21 - 1.12 (m, 4H), 1.01 - 0.88 (m, 2H), 0.85 (d, J = 7.2 Hz, 3H),
0.81 (d, J = 6.8 Hz, 3H), 0.56 (d, J = 6.8 Hz, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$
161.1, 136.7 (d, $J_{C>P}$ = 12.0 Hz), 155.5 (d, $J_{C>P}$ = 144.0 Hz), 135.2 (d, $J_{C>P}$ = 11.0 Hz),
131.3 (d, $J_{C>P}$ = 2.0 Hz), 130.5 (d, $J_{C>P}$ = 11.0 Hz), 129.9 (d, $J_{C>P}$ = 8.0 Hz), 128.0,
127.9, 127.8, 126.2 115.6, 108.0 (d, $J_{C>P}$ = 158.0 Hz), 76.5 (d, $J_{C>P}$ = 6.0 Hz), 61.7,
48.9 (d, $J_{C>P}$ = 6.0 Hz), 43.4, 34.0, 32.1, 31.5, 25.4, 22.7, 21.9, 21.0, 15.3, 13.7. $^{31}$P
NMR (162 MHz, CDCl$_3$): $\delta$ 27.75. HRMS Calcd for C$_{28}$H$_{36}$BrNO$_4$P [M + H]$^+$
560.1560, found 560.1567.

Prepared according to the general procedure. Flash column chromatography on a
silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (79% yield) as a
yellow solid. Mp: 101-103 °C. $\text{R}_f$ (ethyl acetate: petroleum ether, 1:2): 0.43. $[\alpha]_D^{16}$
+34.0 (c 1.00, CHCl$_3$). $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.73 (s, 1H), 7.83 - 7.77 (m,
2H), 7.60 (d, J = 8.8 Hz, 1H), 7.52 - 7.45 (m, 2H), 7.43 - 7.38 (m, 2H), 4.48 - 4.44 (m,
1H), 4.25 - 4.16 (m, 2H), 4.00 (s, 3H), 2.13-2.08 (m, 1H), 1.80 (d, J = 12.0 Hz, 1H),
1.69 - 1.61 (m, 2H), 1.50 - 1.46 (m, 1H), 1.35 - 1.27 (m, 2H), 1.18 (t, (d, J = 7.2 Hz,
4H), 1.01 - 0.85 (m, 2H), 0.81 (d, J = 7.2 Hz, 3H), 0.80 (d, J = 6.4 Hz, 3H), 0.58 (d, J
= 6.8 Hz, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 161.0, 139.2 (d, $J_{C>P}$ = 11.0 Hz),
136.2 (d, $J_{C>P}$ = 20.0 Hz), 135.2 (d, $J_{C>P}$ = 144.0 Hz), 131.4 (d, $J_{C>P}$ = 3.0 Hz), 130.5 (d,
$J_{C>P}$ = 11.0 Hz), 128.1, 127.9, 127.5 (d, $J_{C>P}$ = 8.0 Hz), 124.8 (d, $J_{C>P}$ = 270.0 Hz),
124.4 (q, $J_{C>P}$ = 32.0 Hz) 121.7 (d, $J_{C>P}$ = 5.0 Hz), 121.3 (d, $J_{C>P}$ = 3.0 Hz), 110.8,
109.6 (d, $J_{C>P}$ = 158.0 Hz), 76.6 (d, $J_{C>P}$ = 7.0 Hz), 61.8, 48.9 (d, $J_{C>P}$ = 5.0 Hz), 43.3,
34.0, 32.2, 31.5, 25.3, 22.6, 21.9, 20.8, 15.3, 13.7. $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$
27.52. $^{19}$F NMR (376 MHz, CDCl$_3$): $\delta$ -60.76. HRMS Calcd for C$_{30}$H$_{36}$F$_3$NO$_4$P [M
+ H]$^+$ 550.2329, found 550.2336.

Prepared according to the general procedure. Flash column chromatography on a
silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (79% yield) as a white
solid. Mp: 119-121 °C. Rf (ethyl acetate: petroleum ether, 1:2): 0.45. [α]D^16 = +22.0 (c 1.00, CHCl₃). ^1H NMR (400 MHz, CDCl₃): δ 8.03 (d, J = 8 Hz, 1H), 7.97 - 7.91 (m, 2H), 7.53 - 7.48 (m, 1H), 7.46 - 7.40 (m, 3H), 7.37 - 7.34 (m, 1H), 7.25 - 7.22 (m, 1H), 4.46 - 4.37 (m, 1H), 3.95 (s, 3H), 2.30 - 2.20 (m, 1H), 1.97 - 1.93 (m, 1H), 1.70 - 1.53 (m, 3H), 1.35 - 1.27 (m, 2H), 1.04 - 0.94 (m, 2H), 0.88 (d, J = 7.2 Hz, 3H), 0.82 (d, J = 5.6 Hz, 3H), 0.64 (d, J = 6.8 Hz, 3H). ^13C NMR (100 MHz, CDCl₃): δ 139.0 (d, J_C-P = 1.0 Hz), 133.2 (d, J_C-P = 142.0 Hz), 132.1, 132.0, 131.4 (d, J_C-P = 10.0 Hz), 128.0 (d, J_C-P = 13.0 Hz), 127.4 (d, J_C-P = 10.0 Hz), 126.3, 122.9 (d, J_C-P = 13.0 Hz), 115.5 (d, J_C-P = 154.0 Hz), 114.6, 114.5, 112.2, 110.2, 77.7 (d, J_C-P = 7.0 Hz), 48.5 (d, J_C-P = 5.0 Hz), 43.2, 33.9, 31.9, 31.6, 25.5, 22.7, 21.9, 21.0, 15.4. ^31P NMR (162 MHz, CDCl₃): δ 21.75. HRMS Calcd for C_{26}H_{32}N_{2}O_{3}P [M + H]^+ 435.2196, found 435.2190.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (52% yield) as a yellow oil. Rf (ethyl acetate: petroleum ether, 1:2): 0.28. [α]D^16 = +13.0 (c 1.00, CHCl₃). ^1H NMR (400 MHz, CDCl₃): δ 11.55 (br, 1H), 11.36 (br, 1H), 7.81 - 7.75 (m, 2H), 7.62 - 7.59 (m, 1H), 7.50 - 7.37 (m, 4H), 7.29 - 7.24 (m, 1H), 7.05 (t, J = 7.6 Hz, 1H), 4.29 - 4.24 (m, 1H), 3.15 (d, J = 4.0Hz, 3H), 2.28 (s, 1H), 1.90 - 1.86 (m, 1H), 1.65 - 1.59 (m, 3H), 1.50 - 1.40 (m, 2H), 0.91 (d, J = 5.6 Hz, 3H), 0.68 (d, J = 6.8 Hz, 3H), 0.24 (d, J = 6.8 Hz, 3H). ^13C NMR (100 MHz, CDCl₃): δ 161.2, 137.6 (d, J_C-P = 21.0 Hz), 135.1(d, J_C-P = 10.0 Hz), 133.9 (d, J_C-P = 148.0 Hz), 131.9 (d, J_C-P = 3.0 Hz), 130.6, 130.5, 129.4, 129.2, 128.5, 128.4, 124.4, 122.1, 121.6, 112.5, 101.6 (d, J_C-P = 153.0 Hz), 78.3 (d, J_C-P = 8.0 Hz), 48.7 (d, J_C-P = 6.0 Hz), 43.8, 33.9, 31.6, 29.7, 26.8, 25.3, 22.6, 22.0, 20.9, 14.9. ^31P NMR (162 MHz, CDCl₃): δ 31.16. HRMS Calcd for C_{27}H_{36}N_{2}O_{3}P [M + H]^+ 453.2302, found 453.2297.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (73% yield) as a
yellow oil. $R_f$ (ethyl acetate: petroleum ether, 1:1): 0.25. $\{\alpha\}_D^{16} = -61.0$ (c 1.00, CHCl$_3$). $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 14.32 (br, 1H), 11.23 (br, 1H), 7.80 (dd, $J = 13.6$ Hz, 7.2 Hz, 2H), 7.62 (d, $J = 8.4$ Hz, 1H), 7.52 - 7.47 (m, 1H), 7.44 - 7.37 (m, 3H), 7.30 - 7.25 (m, 1H), 7.05 (t, $J = 7.6$ Hz, 1H), 4.30 - 4.21 (m, 1H), 3.98 (s, 3H), 2.35 - 2.31 (m, 1H), 1.88 - 1.80 (m, 1H), 1.65 - 1.59 (m, 2H), 1.50 - 1.41 (m, 3H), 0.92 - 0.86 (m, 5H), 0.68 (d, $J = 6.8$ Hz, 3H), 0.19 (d, $J = 6.8$ Hz, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 158.3, 135.5 (d, $J_{C-P} = 12.0$ Hz), 135.1, 134.9, 133.2 (d, $J_{C-P} = 150.0$ Hz), 132.2 (d, $J_{C-P} = 2.0$ Hz), 130.7 (d, $J_{C-P} = 11.0$ Hz), 129.0 (d, $J_{C-P} = 10.0$ Hz), 128.5 (d, $J_{C-P} = 13.0$ Hz), 124.8, 121.9 (d, $J_{C-P} = 10.0$ Hz), 112.6, 101.7 (d, $J_{C-P} = 151.0$ Hz), 78.7 (d, $J_{C-P} = 6.0$ Hz), 64.1 (d, $J_{C-P} = 5.0$ Hz), 48.7 (d, $J_{C-P} = 7.0$ Hz), 43.8, 33.9, 31.7, 25.2, 22.6, 20.9, 14.8. $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 30.67. HRMS Calcd for C$_{27}$H$_{38}$N$_2$O$_3$P $[M + H]^+$ 453.2302, found 453.2297.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (82% yield) as a yellow solid. Mp: 173-175 oC. $R_f$ (ethyl acetate: petroleum ether, 1:2): 0.59. $[\alpha]_D^{16} = -58.0$ (c 1.00, CHCl$_3$). 1H NMR (400 MHz, CDCl$_3$): $\delta$ 10.86 (s, 1H), 7.68 (d, $J = 8.4$ Hz, 1H), 7.88 - 7.82 (m, 2H), 7.49 - 7.38 (m, 5H), 7.24 - 7.20 (m, 1H), 4.47 - 4.37 (m, 1H), 4.14 (s, 3H), 2.10 - 1.97 (m, 2H), 1.64 (d, $J = 10.8$ Hz, 2H), 1.53 - 1.47 (m, 1H), 1.36 - 1.25 (m, 2H), 1.02 - 0.92 (m, 2H), 0.90 (d, $J = 6.4$ Hz, 3H), 0.85 (d, $J = 6.0$ Hz, 3H), 0.76 (d, $J = 7.2$ Hz, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 186.6(d, $J_{C-P} = 18.0$ Hz), 139.4 (d, $J_{C-P} = 12.0$ Hz), 138.2 (d, $J_{C-P} = 22.0$ Hz), 134.9 (d, $J_{C-P} = 142.0$ Hz), 131.9, 130.7 (d, $J_{C-P} = 11.0$ Hz), 128.4 (d, $J_{C-P} = 13.0$ Hz), 127.6 (d, $J_{C-P} = 9.0$ Hz), 126.9, 123.6, 122.4, 115.6 (d, $J_{C-P} = 153.0$ Hz), 110.6, 77.5, 49.0 (d, $J_{C-P} = 5.0$ Hz), 43.6, 34.0, 32.5, 31.6, 25.5, 22.7, 22.0, 21.0, 15.2. $^{31}$P NMR (162 MHz, CDCl$_3$): $\delta$ 25.95. HRMS Calcd for C$_{26}$H$_{33}$NO$_3$P $[M + H]^+$ 438.2193, found 438.2199.
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (82% yield) as a yellow oil. Rf (ethyl acetate: petroleum ether, 1:0.0) 0.55. [α]D16 = +20.0 (c 1.00, CHCl3). 1H NMR (400 MHz, CDCl3): δ 7.89 - 7.83 (m, 2H), 7.80 (d, J = 8 Hz, 1H), 7.49 - 7.44 (m, 1H), 7.43 - 7.29 (m, 4H), 7.20 - 7.15 (m, 1H), 4.34 - 4.24 (m, 1H), 3.74 (s, 3H), 2.80 (s, 3H), 2.05 - 1.97 (m, 2H), 1.61 (d, J = 10 Hz, 2H), 1.45 - 1.39 (m, 1H), 1.35 - 1.20 (m, 2H), 0.99 - 0.82 (m, 2H), 0.80 (d, J = 2.8 Hz, 3H), 0.74 (d, J = 7.2 Hz, 3H), 0.45 (d, J = 6.8 Hz, 3H). 13C NMR (100 MHz, CDCl3): δ 198.4, 144.5 (d, JC-P = 22.0 Hz), 137.5 (d, JC-P = 11.0 Hz), 134.6 (d, JC-P = 141.0 Hz), 131.6 (d, JC-P = 3.0 Hz), 131.0 (d, JC-P = 11.0 Hz), 128.2 (d, JC-P = 13.0 Hz), 127.5 (d, JC-P = 10.0 Hz), 124.1, 122.4, 121.9, 110.2, 104.3 (d, JC-P = 159.0 Hz), 77.0, 49.0 (d, JC-P = 6.0 Hz), 43.4, 34.0, 33.4, 31.5, 31.3, 25.5, 22.7, 22.0, 21.0, 15.1. 31P NMR (162 MHz, CDCl3): δ 26.08. HRMS Calcd for C27H35NO3P [M + H]+ 452.2349, found 452.2354.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (68% yield) as a yellow oil. Rf (ethyl acetate: petroleum ether, 1:1): 0.27. [α]D16 = -42.0 (c 1.00, CHCl3). 1H NMR (400 MHz, CDCl3): δ 14.24 (br, 1H), 11.15(br, 1H), 7.81 - 7.74 (m, 2H), 7.56 - 7.51 (m, 2H), 7.48 - 7.41 (m, 3H), 7.25 - 7.24 (m, 1H), 4.31 - 4.22 (m, 1H), 3.97 (s, 3H), 2.30 - 2.26 (m, 1H), 1.92 - 1.83 (m, 1H), 1.66 - 1.62 (m, 2H), 1.54 - 1.51 (m, 1H), 1.49 - 1.37 (m, 2H), 0.98-0.85 (m, 5H), 0.75 (d, J = 6.8 Hz, 3H), 0.28 (d, J = 6.8 Hz, 3H). 13C NMR (100 MHz, CDCl3): δ 157.9, 136.0 (d, JC-P = 20.0 Hz), 133.8 (d, JC-P = 12.0 Hz), 132.9 (d, JC-P = 150.0 Hz), 132.5 (d, JC-P = 3.0 Hz), 130.6 (d, JC-P = 12.0 Hz), 129.9 (d, JC-P = 11.0 Hz), 128.7 (d, JC-P = 13.0 Hz), 127.8, 125.5, 121.3, 113.8, 102.0 (d, JC-P = 152.0 Hz), 79.2 (d, JC-P = 7.0 Hz), 64.2, 48.7 (d, JC-P = 7.0 Hz), 43.7, 33.9, 31.7, 25.5, 22.7, 22.0, 20.9, 14.9. 31P NMR (162 MHz, CDCl3): δ 29.95. HRMS Calcd for C27H35ClN2O4P [M + H]+ 503.1861, found 503.1855.
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (76% yield) as a yellow solid. Mp: 177-179 °C. R$_f$ (ethyl acetate: petroleum ether, 1:2): 0.51. [$\alpha$]$_D^{16}$ = +17.0 (c 1.00, CHCl$_3$). $^1$H NMR (400 MHz, CDCl$_3$): δ 10.78 (s, 1H), 8.03 (d, J = 1.2 Hz, 1H), 7.88 - 7.80 (m, 2H), 7.52 - 7.47 (m, 1H), 7.45 - 7.34 (m, 4H), 4.50 - 4.41 (m, 1H), 4.11 (s, 3H), 2.10 - 2.00 (m, 2H), 1.70 - 1.65 (m, 2H), 1.57 - 1.50 (m, 1H), 1.42 - 1.23 (m, 2H), 1.05 - 0.87 (m, 2H), 0.86 - 0.80 (m, 6H), 0.85 (d, J = 6.0 Hz, 3H), 0.76 (d, J = 7.2 Hz, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$): δ 186.2, 138.7 (d, J$_{C-P}$ = 21.0 Hz), 137.8 (d, J$_{C-P}$ = 12.0 Hz), 134.6 (d, J$_{C-P}$ = 142.0 Hz), 132.1 (d, J$_{C-P}$ = 2.0 Hz), 130.7 (d, J$_{C-P}$ = 11.0 Hz), 128.7, 128.5, 128.4, 127.5, 122.8, 115.2 (d, J$_{C-P}$ = 153.0 Hz), 111.9, 78.0 (d, J$_{C-P}$ = 7.0 Hz), 49.0 (d, J$_{C-P}$ = 6.0 Hz), 43.6, 34.0, 32.8, 31.6, 25.7, 22.8, 21.9, 21.0, 15.3. $^{31}$P NMR (162 MHz, CDCl$_3$): δ 25.44. HRMS Calcd for C$_{26}$H$_{32}$ClNO$_3$P [M + H]$^+$ 472.1803, found 472.1796.

Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (56% yield) as a yellow oil. R$_f$ (ethyl acetate: petroleum ether, 1:2): 0.20. [$\alpha$]$_D^{16}$ = +25.0 (c 1.00, CHCl$_3$). $^1$H NMR (400 MHz, CDCl$_3$): δ 9.78, (s, 1H), 8.58 (d, J = 1.6 Hz, 1H), 7.72 (d, J = 13.6 Hz, 1H), 7.63 (d, J = 13.6 Hz, 1H), 7.45 - 7.34 (m, 4H), 5.91 (d, J = 16.8 Hz, 1H), 4.52 - 4.42 (m, 1H), 4.21 - 4.11 (m, 2H), 2.37 (s, 3H), 2.37 (s, 3H), 2.14 - 2.07 (m, 1H), 1.85 (d, J = 11.6 Hz, 1H), 1.70 - 1.52 (m, 3H), 1.41 - 1.17 (m, 2H), 1.14 (t, J = 11.6 Hz, 3H), 1.05 - 0.81 (m, 8H), 0.53 (d, J = 6.8 Hz, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$): δ 159.9, 149.7, 138.8 (d, J$_{C-P}$ = 14.0 Hz), 135.9, 134.5, 134.2 (d, J$_{C-P}$ = 11.0 Hz), 133.3, 133.1 (d, J$_{C-P}$ = 10.0 Hz), 132.9, 130.7 (d, J$_{C-P}$ = 7.0 Hz), 129.7, 128.4, 127.5 (d, J$_{C-P}$ = 11.0 Hz), 126.4, 123.5, 117.8, 113.3, 97.3, 77.6 (d, J$_{C-P}$ = 6.0 Hz), 61.9, 49.1 (d, J$_{C-P}$ = 7.0 Hz), 43.7, 33.9, 31.9, 31.5, 25.6, 22.7, 21.9, 21.2, 21.1, 15.1, 13.8. $^{31}$P NMR (162 MHz, CDCl$_3$): δ 27.02. HRMS Calcd for C$_{22}$H$_{35}$NO$_3$P [M + H]$^+$ 567.2174, found 567.2179.
5. Charts of products.
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