Supporting Information

Catalyst- and solvent-free bisphosphinylation of isothiocyanates: A practical method for the synthesis of bisphosphinoylaminomethanes

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Molecular structure and crystallographic data of 3o

Table S1. Crystal data and structure refinement for 3o

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General methods

Unless noted, all commercial reagents and solvents were used without further purification. Melting points were recorded on a RY-1 microscopic melting apparatus and uncorrected. $^1$H NMR spectra were recorded on 500 MHz and $^{13}$C NMR spectra were recorded on 125 MHz by using a Bruker Avance 500 spectrometer. Chemical shifts were reported in parts per million (δ) relative to tetramethylsilane (TMS). Mass spectras were obtained on an Ultima Global spectrometer with an ESI source. The X-ray single-crystal diffraction was performed on Saturn 724+ instrument. Silica gel (200–300 mesh) for column chromatography and silica GF254 for TLC were produced by Qingdao Marine Chemical Company (China). The analysis of elemental sulfur was performed on a JSM-6700F scanning electron microscope (SEM) equipped with a X-MaxN-80 energy dispersive X-ray spectrometer (EDS).

Preparation of starting materials

Aryl isothiocyanates$^1$ and phosphine oxides$^2$ were prepared according to the literatures.

General procedure for the synthesis of bisphosphinoylaminomethanes 3

To a 15 mL sealed tube was charged with a mixture of isothiocyanate 1 (0.4 mmol) and phosphine oxide 2 (0.8 mmol). The reaction mixture was stirred at 110 °C for 6 h. After completion, the mixture was cooled to room temperature, added with EtOAc (1.0 mL), and stirred for 15 min. The crude solid was then filtered and washed with EtOAc. After dried in vacuum, the product 3 was obtained as a solid.

(Phenylaminomethylene)bis(diphenylphosphine oxide) (3a)$^3$

![Chemical Structure](image)

Following the general procedure, 3a was isolated as a white solid from phenyl isothiocyanate 1a (54 mg, 0.4 mmol) and diphenylphosphine oxide 2a (162 mg, 0.8 mmol). Mp 230–232 °C; R$_f$ = 0.25 (DCM/MeOH = 30:1 v/v); 142 mg, 70% yield.

$^1$H NMR (CDCl$_3$, 500 MHz): δ 7.66–7.94 (m, 8H), 7.23–7.46 (m, 12H), 6.83–6.91 (m, 2H), 6.51–6.61 (m, 1H), 6.21–6.33 (m, 2H), 4.97–5.31 (m, 1H), 4.64 (s, 1H).

$^{13}$C NMR (125 MHz, CDCl$_3$): δ 146.0 (s, 1C), 131.9 (s, 4C), 131.8 (t, J = 4.3 Hz, 4C), 131.6 (t, J = 4.7 Hz, 4C), 131.1 (dd, J = 101.5, 46.8 Hz, 4C), 128.8 (s, 2C), 128.3 (d, J = 4.9 Hz, 4C), 128.2 (d, J = 5.0 Hz, 4C), 119.1 (s, 1C), 114.0 (s, 2C), 56.9 (t, J = 64.9 Hz, 1C). HRMS (ESI-TOF, [M + H]$^+$): calcd for C$_{31}$H$_{28}$NO$_2$P$_2$, 508.1589, found 508.1589.
Methyl 4-((bis(diphenylphosphoryl)methyl)amino)benzoate (3b)

Following the general procedure, 3b was isolated as a white solid from methyl 4-isothiocyanatobenzoate 1b (77 mg, 0.4 mmol) and diphenylphosphine oxide 2a (162 mg, 0.8 mmol). Mp 270–272 °C; R_f = 0.20 (DCM/MeOH = 20:1 v/v); 197 mg, 87% yield.

^1H NMR (CDCl_3, 500 MHz): δ 7.73–7.87 (m, 8H), 7.46–7.66 (m, 2H), 7.17–7.45 (m, 12H), 6.26–6.36 (m, 2H), 5.61 (s, 1H), 5.23–5.35 (m, 1H), 3.79 (s, 3H).

^13C NMR (125 MHz, CDCl_3): δ 166.9 (s, 1C), 149.7 (s, 1C), 132.2 (s, 4C), 131.7 (s, 4C), 131.4 (s, 4C), 130.9 (s, 2C), 130.5 (dd, J = 103.7, 86.3 Hz, 4C), 128.3 (s, 8C), 119.9 (s, 1C), 112.5 (s, 2C), 55.7 (t, J = 63.8 Hz, 1C), 51.6 (s, 1C). HRMS (ESI-TOF, [M + Na]^+): calcd for C_{33}H_{29}NO_4NaP_2, 588.1470, found 588.1474.

(4-Acetylphenylaminomethylene)bis(diphenylphosphine oxide) (3c)

Following the general procedure, washed with petroleum ether/EtOAc (3: 1), 3c was isolated as a white solid from 1-(4-isothiocyanatophenyl)ethan-1-one 1c (71 mg, 0.4 mmol) and diphenylphosphine oxide 2a (162 mg, 0.8 mmol). Mp 214–216 °C; R_f = 0.25 (DCM/MeOH = 20:1 v/v); 182 mg, 83% yield.

^1H NMR (CDCl_3, 500 MHz): δ 7.62–8.01 (m, 8H), 7.49–7.62 (m, 2H), 7.19–7.48 (m, 12H), 6.32−6.37 (m, 2H), 5.41 (s, 1H), 5.21−5.30 (m, 1H), 2.41 (s, 3H).

^13C NMR (125 MHz, CDCl_3): δ 196.3 (s, 1C), 149.9 (s, 1C), 132.2 (s, 4C), 131.7 (s, 4C), 131.4 (s, 4C), 130.4 (s, 1C), 130.1 (s, 2C), 129.6 (s, 1C), 128.4 (s, 8C), 128.1 (s, 1C), 112.5 (s, 2C), 55.7 (t, J = 64.3 Hz, 1C), 26.0 (s, 1C). HRMS (ESI-TOF, [M + H]^+): calcd for C_{33}H_{30}NO_3P_2, 550.1695, found 550.1696.

(4-Cyanophenylaminomethylene)bis(diphenylphosphine oxide) (3d)

Following the general procedure, 3d was isolated as a white solid from 4-isothiocyanatobenzonitrile 1d (64 mg, 0.4 mmol) and diphenylphosphine oxide 2a (162 mg, 0.8 mmol). Mp 264–266 °C; R_f = 0.30 (DCM/MeOH = 10:1 v/v); 162 mg, 76% yield.

^1H NMR (CDCl_3, 500 MHz): δ 7.70−7.84 (m, 8H), 7.22−7.47 (m, 12H), 6.25−6.40 (m, 2H), 5.23−5.65 (m, 1H), 5.11−5.22 (m, 1H). ^13C NMR (125 MHz, CDCl_3): δ
149.3 (s, 1C), 133.1 (s, 2C), 132.2 (s, 4C), 131.7 (s, 4C), 131.3 (s, 4C), 129.9 (dd, $J = 106.1, 15.3$ Hz, 4C), 128.4 (s, 8C), 119.6 (s, 1C), 113.3 (s, 2C), 100.8 (s, 1C), 56.0 (t, $J = 63.3$ Hz, 1C).

HRMS (ESI-TOF, [M + Na]$^+$): calcd for C$_{32}$H$_{26}$N$_2$O$_2$NaP$_2$, 555.1367, found 555.1371.

(4-Trifluoromethylphenylaminomethylene)bis(diphenylphosphine oxide) (3e)

Following the general procedure, 3e was isolated as a white solid from 1-isothiocyanato-4-(trifluoromethyl)benzene 1e (92 mg, 0.4 mmol) and diphenylphosphine oxide 2a (162 mg, 0.8 mmol). Mp 270−272 °C; $R_f = 0.15$ (DCM/MeOH = 30:1 v/v); 189 mg, 82% yield.

$^1$H NMR (CDCl$_3$, 500 MHz): $\delta$ 7.60−8.00 (m, 8H), 7.27−7.46 (m, 12H), 7.07−7.14 (m, 2H), 6.24−6.46 (m, 2H), 5.10−5.24 (m, 1H), 5.03 (s, 1H).

$^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ 148.6 (s, 1C), 132.1 (s, 4C), 131.7 (s, 4C), 131.4 (s, 6C), 130.5 (s, 1C), 129.8 (s, 1C), 128.3 (s, 8C), 126.1 (s, 2C), 124.4 (d, $J_{FC} = 270.2$ Hz, 1C), 120.3 (d, $J_{FC} = 35.6$ Hz, 1C), 113.0 (s, 2C), 56.3 (t, $J_{PC} = 63.6$ Hz, 1C).

HRMS (ESI-TOF, [M + H]$^+$): calcd for C$_{32}$H$_{27}$F$_3$NO$_2$P$_2$, 576.1463, found 576.1464.

(4-Nitrophenylaminomethylene)bis(diphenylphosphine oxide) (3f)

Following the general procedure, 3f was isolated as a yellow solid from 1-isothiocyanato-4-nitrobenzene 1f (72 mg, 0.4 mmol) and diphenylphosphine oxide 2a (162 mg, 0.8 mmol). Mp 263−265 °C; $R_f = 0.30$ (DCM/MeOH = 15:1 v/v); 172 mg, 78% yield.

$^1$H NMR (CDCl$_3$, 500 MHz): $\delta$ 7.56−8.01 (m, 10H), 7.17−7.52 (m, 12H), 6.28−6.42 (m, 2H), 5.69 (s, 1H), 5.16−5.28 (m, 1H).

$^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ 151.4 (s, 1C), 139.4 (s, 1C), 132.4 (s, 4C), 131.8 (s, 4C), 131.4 (s, 4C), 129.9 (dd, $J = 102.7, 26.9$ Hz, 4C), 128.5 (s, 8C), 125.6 (s, 2C), 112.3 (s, 2C), 56.2 (t, $J = 62.8$ Hz, 1C).

HRMS (ESI-TOF, [M + Na]$^+$): calcd for C$_{31}$H$_{26}$N$_2$O$_4$NaP$_2$, 575.1265, found 575.1268.

(4-Fluorophenylaminomethylene)bis(diphenylphosphine oxide) (3g)

Following the general procedure, 3g was isolated as a white solid from 1-fluoro-4-isothiocyanatobenzene 1g (61 mg, 0.4 mmol) and diphenylphosphine oxide 2a (162 mg, 0.8 mmol). Mp 220−222 °C; $R_f = 0.20$ (DCM/MeOH = 30:1 v/v); 158 mg, 75% yield.
**1H NMR (CDCl₃, 500 MHz):** δ 7.60–8.00 (m, 8H), 7.26–7.56 (m, 12H), 6.42–6.75 (m, 2H), 6.21 (s, 2H), 4.93–5.08 (m, 1H), 4.56 (s, 1H). **13C NMR (125 MHz, CDCl₃):** δ 156.5 (d, J_{FC} = 237.4 Hz, 1C), 142.7 (s, 1C), 132.0 (s, 4C), 131.9 (s, 4C), 131.5 (s, 4C), 131.0 (dd, J_{PC} = 103.1, 64.4 Hz, 4C), 128.3 (s, 10C), 115.4 (d, J_{PC} = 20.9 Hz, 2C), 58.4 (t, J = 64.3 Hz, 1C). HRMS (ESI-TOF, [M + H]+): calcd for C₃₁H₂₇FNO₂P₂, 526.1496, found 526.1496.

**1H NMR (CDCl₃, 500 MHz):** δ 7.75–7.87 (m, 8H), 7.27–7.48 (m, 12H), 6.69–6.98 (m, 2H), 6.18–6.28 (m, 2H), 5.02–5.13 (m, 1H), 4.80 (s, 1H). **13C NMR (125 MHz, CDCl₃):** δ 144.8 (s, 1C), 132.0 (s, 4C), 131.8 (s, 4C), 131.4 (s, 4C), 130.9 (dd, J = 101.7, 76.8 Hz, 4C), 128.6 (s, 2C), 128.3 (s, 8C), 123.6 (s, 1C), 115.0 (s, 2C), 57.4 (t, J = 64.3 Hz, 1C). HRMS (ESI-TOF, [M + H]+): calcd for C₃₁H₂₇ClNO₂P₂, 542.1200, found 542.1202.

**1H NMR (CDCl₃, 500 MHz):** δ 7.67–7.94 (m, 8H), 7.25–7.50 (m, 12H), 6.82–7.09 (m, 2H), 6.15–6.21 (m, 2H), 5.02–5.12 (m, 1H), 4.80 (s, 1H). **13C NMR (125 MHz, CDCl₃):** δ 145.2 (s, 1C), 132.0 (s, 4C), 131.8 (s, 4C), 131.5 (s, 4C), 130.7 (dd, J = 102.7, 78.8 Hz, 4C), 128.3 (s, 10C), 115.6 (s, 2C), 110.9 (s, 1C), 57.1 (t, J = 64.8 Hz, 1C). HRMS (ESI-TOF, [M + H]+): calcd for C₃₁H₂₇BrNO₂P₂, 586.0695, found 586.0695.
methylbenzene 1j (60 mg, 0.4 mmol) and diphenylphosphine oxide 2a (162 mg, 0.8 mmol). Mp 225–227 °C; Rf = 0.30 (DCM/MeOH = 30:1 v/v); 115 mg, 55% yield.

**1H NMR (CDCl$_3$, 500 MHz):** δ 7.65–7.95 (m, 8H), 7.18–7.48 (m, 12H), 6.61–6.73 (m, 2H), 6.14–6.23 (m, 2H), 5.05–5.15 (m, 1H), 4.53 (s, 1H), 2.10 (s, 3H).

**13C NMR (125 MHz, CDCl$_3$):** δ 143.8 (s, 1C), 131.8 (s, 8C), 131.6 (s, 6C), 130.9 (s, 1C), 130.6 (s, 1C), 129.3 (s, 3C), 128.2 (s, 8C), 114.2 (s, 2C), 57.5 (t, J = 65.0 Hz, 1C), 20.3 (s, 1C).

**HRMS (ESI-TOF, [M + H]$^+$):** calcd for C$_{32}$H$_{30}$NO$_2$P$_2$, 522.1746, found 522.1749.

(4-Methoxyphenylaminomethylene)bis(diphenylphosphine oxide) (3k)

Following the general procedure, the reaction temperature was 90 °C, 3k was isolated as a light yellow solid from 1-isothiocyanato-4-methoxybenzene 1k (66 mg, 0.4 mmol) and diphenylphosphine oxide 2a (162 mg, 0.8 mmol). Mp 210–212 °C; Rf = 0.15 (DCM/MeOH = 15:1 v/v); 122 mg, 57% yield.

**1H NMR (CDCl$_3$, 500 MHz):** δ 7.75–7.88 (m, 8H), 7.27–7.50 (m, 12H), 6.43–6.48 (m, 2H), 6.19–6.24 (m, 2H), 4.95–5.07 (m, 1H), 4.38–4.45 (m, 1H), 3.64 (s, 3H).

**13C NMR (125 MHz, CDCl$_3$):** δ 153.0 (s, 1C), 140.3 (s, 1C), 131.9 (s, 8C), 131.5 (s, 4C), 130.5 (dd, J = 107.6, 65.8 Hz, 4C), 128.2 (s, 8C), 115.7 (s, 2C), 114.3 (s, 2C), 58.4 (t, J = 51.4 Hz, 1C), 55.6 (s, 1C).

**HRMS (ESI-TOF, [M + Na]$^+$):** calcd for C$_{32}$H$_{29}$NO$_3$NaP$_2$, 560.1520, found 560.1521.

(2-Chlorophenylaminomethylene)bis(diphenylphosphine oxide) (3l)

Following the general procedure, 3l was isolated as a white solid from 1-chloro-2-isothiocyanatobenzene 1l (68 mg, 0.4 mmol) and diphenylphosphine oxide 2a (162 mg, 0.8 mmol). Mp 280–282 °C; Rf = 0.30 (DCM/MeOH = 30:1 v/v); 143 mg, 66% yield.

**1H NMR (CDCl$_3$, 500 MHz):** δ 7.77–7.89 (m, 8H), 7.21–7.48 (m, 12H), 6.86–7.14 (m, 1H), 6.78–6.84 (m, 1H), 6.44–6.50 (m, 1H), 6.33–6.36 (m, 1H), 5.17–5.23 (m, 2H).

**13C NMR (125 MHz, CDCl$_3$):** δ 141.6 (s, 1C), 132.1 (s, 4C), 131.7 (s, 8C), 130.8 (dd, J = 102.7, 23.9 Hz, 4C), 129.1 (s, 1C), 128.4 (t, J = 5.3 Hz, 4C), 128.3 (t, J = 5.3 Hz, 4C), 127.3 (s, 1C), 120.2 (s, 1C), 118.8 (s, 1C), 111.9 (s, 1C), 56.5 (t, J = 64.3 Hz, 1C).

**HRMS (ESI-TOF, [M + H]$^+$):** calcd for C$_{31}$H$_{27}$ClNO$_2$P$_2$, 542.1200, found 542.1201.
Following the general procedure, 3m was isolated as a white solid from 1-chloro-3-isothiocyanatobenzene 1m (68 mg, 0.4 mmol) and diphenylphosphine oxide 2a (162 mg, 0.8 mmol). Mp 268–270 °C; Rf = 0.30 (DCM/MeOH = 30:1 v/v); 154 mg, 71% yield.

\[ ^1H \text{ NMR (CDCl}_3, \text{ 500 MHz)}: \delta 8.18 (s, 2H), 7.84 (s, 11H), 7.34–7.43 (m, 6H), 6.61–6.86 (m, 2H), 6.42–6.49 (m, 1H), 5.99–5.29 (m, 1H), 5.05–3.24 (m, 1H). \]

\[ ^{13}C \text{ NMR (CDCl}_3): \delta 147.2 (s, 1C), 134.5 (s, 1C), 132.0 (s, 4C), 131.8 (s, 4C), 131.5 (s, 4C), 130.7 (dd, J = 103.7, 70.8 Hz, 4C), 129.7 (s, 1C), 128.3 (s, 8C), 118.8 (s, 1C), 113.8 (s, 1C), 112.1 (s, 1C), 56.8 (t, J = 64.3 Hz, 1C). \]

HRMS (ESI-TOF, [M + Na]^+): calcd for C\textsubscript{31}H\textsubscript{26}NO\textsubscript{2}NaP\textsubscript{2}Cl, 564.1025, found 564.1031.

(2-Bisphenylaminomethylene)bis(diphenylphosphine oxide) (3n)

Following the general procedure, 3n was isolated as a yellow solid from 2-isothiocyanato-1,1'-biphenyl 1n (84 mg, 0.4 mmol) and diphenylphosphine oxide 2a (162 mg, 0.8 mmol). Mp 61–63 °C; Rf = 0.25 (DCM/MeOH = 30:1 v/v); 112 mg, 48% yield.

\[ ^1H \text{ NMR (CDCl}_3, \text{ 500 MHz)}: \delta 7.78–7.85 (m, 4H), 7.57–7.64 (m, 4H), 7.43–7.48 (m, 2H), 7.28–7.37 (m, 9H), 7.16–7.22 (m, 4H), 6.95–6.99 (m, 1H), 6.83–6.90 (m, 3H), 6.59–6.67 (m, 1H), 6.50–6.56 (m, 1H), 5.28–5.39 (m, 1H), 4.59–4.67 (m, 1H). \]

\[ ^{13}C \text{ NMR (CDCl}_3): \delta 141.9 (s, 1C), 138.0 (s, 1C), 131.9 (s, 4C), 131.7 (s, 6C), 131.5 (s, 4C), 130.9 (dd, J = 104.1, 26.2 Hz, 4C), 129.9 (s, 1C), 129.0 (s, 2C), 128.8 (s, 2C), 128.4 (s, 4C), 128.1 (s, 4C), 127.4 (s, 1C), 118.1 (s, 1C), 110.4 (s, 1C), 56.2 (t, J = 64.3 Hz, 1C). \]

HRMS (ESI-TOF, [M + H]^+): calcd for C\textsubscript{37}H\textsubscript{32}NO\textsubscript{2}P\textsubscript{2}, 584.1903, found 584.1902.

(2-Phenylethynylphenylaminomethylene)bis(diphenylphosphine oxide) (3o)

Following the general procedure, 3o was isolated as a white solid from 1-isothiocyanato-2-
(phenylethynyl)benzene 1o (94 mg, 0.4 mmol) and diphenylphosphine oxide 2a (162 mg, 0.8 mmol). Mp 214–216 °C; Rf = 0.20 (DCM/MeOH = 30:1 v/v); 136 mg, 56% yield.

\(^1H\) NMR (CDCl\(_3\), 500 MHz): \(\delta\) 7.79–7.96 (m, 8H), 7.24–7.49 (m, 13H), 7.14–7.21 (m, 4H), 7.08–7.12 (m, 1H), 6.85–6.93 (m, 1H), 6.47–6.55 (m, 1H), 6.30–6.37 (m, 1H), 5.51–5.62 (m, 1H).

\(^13C\) NMR (125 MHz, CDCl\(_3\)): \(\delta\) 146.3 (s, 1C), 132.0 (s, 2C), 131.9 (s, 4C), 131.6 (s, 4C), 130.8 (dd, \(J = 104.1, 42.7\) Hz, 4C), 129.2 (s, 2C), 128.4 (s, 8C), 128.1 (s, 4C), 122.8 (s, 1C), 117.9 (s, 2C), 110.2 (s, 2C), 109.2 (s, 2C), 96.4 (s, 1C), 84.9 (s, 1C), 56.1 (t, \(J = 65.3\) Hz, 1C).

HRMS (ESI-TOF, [M + H]\(^+\)): calcd for C\(_{39}\)H\(_{32}\)NO\(_2\)P\(_2\), 608.1903, found 608.1905.

(2,4-Difluorophenylaminomethylene)bis(diphenylphosphine oxide) (3p)

Following the general procedure, 3p was isolated as a white solid from 2,4-difluoro-1-isothiocyanatobenzene 1p (68 mg, 0.4 mmol) and diphenylphosphine oxide 2a (162 mg, 0.8 mmol). Mp 258–260 °C; Rf = 0.30 (DCM/MeOH = 30:1 v/v); 161 mg, 74% yield.

\(^1H\) NMR (CDCl\(_3\), 500 MHz): \(\delta\) 7.61–8.00 (m, 8H), 6.99–7.57 (m, 12H), 6.20–6.58 (m, 3H), 4.99–5.08 (m, 1H), 4.63–4.71 (m, 1H).

\(^13C\) NMR (125 MHz, CDCl\(_3\)): \(\delta\) 155.1 (d, \(J_{FC} = 230.2\) Hz, 1C), 151.0 (d, \(J_{FC} = 243.3\) Hz, 1C), 132.0 (s, 4C), 131.6 (s, 4C), 131.5 (s, 4C), 130.7 (dd, \(J_{PC} = 103.3, 42.2\) Hz, 4C), 128.3 (s, 8C), 113.9 (s, 2C), 110.4 (d, \(J_{FC} = 21.7\) Hz, 1C), 103.4 (t, \(J_{FC} = 24.9\) Hz, 1C), 57.6 (t, \(J_{PC} = 64.8\) Hz, 1C).

HRMS (ESI-TOF, [M + H]\(^+\)): calcd for C\(_{31}\)H\(_{26}\)F\(_2\)NO\(_2\)P\(_2\), 544.1403, found 544.1403.

((benzo[d][1,3]dioxol-5-ylamino)methylene)bis(diphenylphosphine oxide) (3q)

Following the general procedure, 3q was isolated as a white solid from 5-isothiocyanatobenzo[d][1,3]dioxole 1q (72 mg, 0.4 mmol) and diphenylphosphine oxide 2a (162 mg, 0.8 mmol). Mp 237–239 °C; Rf = 0.15 (DCM/MeOH = 30:1 v/v); 143 mg, 65% yield.

\(^1H\) NMR (CDCl\(_3\), 500 MHz): \(\delta\) 7.89–8.11 (m, 1H), 7.75–7.88 (m, 7H), 7.25–7.47 (m, 12H), 6.25–6.40 (m, 1H), 5.86–5.93 (m, 1H), 5.77–5.81 (m, 1H), 5.76 (s, 2H), 5.03–5.16 (m, 1H), 4.55 (s, 1H).

\(^13C\) NMR (125 MHz, CDCl\(_3\)): \(\delta\) 147.8 (s, 1C), 141.8 (s, 1C), 140.6 (s, 1C), 131.9 (s, 6C), 131.5 (s, 4C), 131.0 (s, 1C), 130.9 (s, 1C), 130.8 (dd, \(J = 105.7, 53.9\) Hz, 4C), 128.2 (s, 8C), 108.0 (s, 1C), 106.6 (s, 1C), 100.6 (s, 1C), 97.5 (s, 1C), 58.5 (t, \(J = 64.8\) Hz, 1C).

HRMS (ESI-TOF, [M + H]\(^+\)): calcd for C\(_{32}\)H\(_{28}\)NO\(_4\)P\(_2\), 552.1488, found 552.1488.
((Pyridin-2-ylamino)methylene)bis(diphenylphosphine oxide) (3r)

Following the general procedure, the reaction temperature was 90 °C and the reaction time was 1 h, 3r was isolated as a white solid from 2-isothiocyanatopyridine 1r (54 mg, 0.4 mmol) and diphenylphosphine oxide 2a (162 mg, 0.8 mmol). Mp 240–242 °C; R_f = 0.15 (DCM/MeOH = 30:1 v/v); 116 mg, 57% yield.

^1H NMR (CDCl₃, 500 MHz): δ 7.91–8.03 (m, 1H), 7.75–7.84 (m, 9H), 7.29–7.41 (m, 4H), 7.19–7.26 (m, 7H), 6.93–7.15 (m, 1H), 6.70–6.78 (m, 1H), 6.38–6.48 (m, 1H), 5.88–6.18 (m, 1H), 5.27–5.70 (m, 1H). ^13C NMR (125 MHz, CDCl₃): δ 155.3 (s, 1C), 146.7 (s, 2C), 136.8 (s, 2C), 131.6 (s, 10C), 131.0 (s, 1C), 130.8 (s, 1C), 128.1 (s, 2C), 127.9 (s, 2C), 113.9 (s, 4C), 109.7 (s, 4C), 50.0 (t, J = 69.3 Hz, 1C). HRMS (ESI-TOF, [M + Na]^+): calcd for C_{30}H_{26}N_{2}O_{2}NaP_{2}, 531.1367, found 531.1370.

((Phenylamino)methylene)bis(bis(4-fluorophenyl)phosphine oxide) (3s)

Following the general procedure, 3s was isolated as a white solid from phenyl isothiocyanate 1a (54 mg, 0.4 mmol) and bis(4-fluorophenyl)phosphine oxide 2b (190 mg, 0.8 mmol). Mp 183–185 °C; R_f = 0.30 (DCM/MeOH = 30:1 v/v); 162 mg, 70% yield.

^1H NMR (CDCl₃, 500 MHz): δ 7.74–7.90 (m, 8H), 6.88–7.15 (m, 10H), 6.62–6.69 (m, 1H), 6.27–6.30 (m, 2H), 4.99–5.08 (m, 1H), 4.53–4.59 (m, 1H). ^13C NMR (125 MHz, CDCl₃): δ 165.2 (dd, J_{PC} = 255.3, 10.0 Hz, 4C), 145.6 (s, 1C), 134.3 (s, 4C), 134.0 (s, 4C), 129.0 (s, 2C), 126.5 (dd, J_{PC} = 105.7, 35.9 Hz, 4C), 119.7 (s, 1C), 115.9 (s, 4C), 115.7 (s, 4C), 114.1 (s, 2C), 57.4 (t, J_{PC} = 66.3 Hz, 1C). HRMS (ESI-TOF, [M + H]^+): calcd for C_{31}H_{24}F_{4}NO_{2}P_{2}, 580.1213, found 580.1212.

((Phenylamino)methylene)bis(di-p-tolylphosphine oxide) (3t)

Following the general procedure, the reaction temperature was 90 °C and the reaction time was 2 h, 3t was isolated as a white solid from phenyl isothiocyanate 1a (54 mg, 0.4 mmol) and di-p-tolylphosphine oxide 2c (184 mg, 0.8 mmol). Mp 250–252 °C; R_f = 0.30 (DCM/MeOH = 30:1
v/v); 140 mg, 62% yield.

$^1$H NMR (CDCl$_3$, 500 MHz): $\delta$ 7.61–7.77 (m, 8H), 7.02–7.19 (m, 8H), 6.85–6.92 (m, 2H), 6.55–6.60 (m, 1H), 6.26–6.32 (m, 2H), 5.01–5.12 (m, 1H), 4.48–4.64 (m, 1H), 2.32 (d, $J = 27.5$ Hz, 12H).

$^{13}$C NMR (CDCl$_3$): $\delta$ 146.3 (s, 1C), 142.1 (s, 4C), 131.8 (s, 4C), 131.6 (s, 4C), 128.8 (s, 8C), 128.6 (s, 2C), 127.9 (s, 1C), 127.6 (s, 1C), 118.6 (s, 1C), 114.1 (s, 4C), 57.3 (t, $J = 69.0$ Hz, 1C), 21.4 (s, 4C).

HRMS (ESI-TOF, [M + Na]$^+$): calcd for C$_{35}$H$_{35}$NO$_2$NaP$_2$, 586.2041, found 586.2039.

$^1$(Di-$o$-tolylphosphoryl)(phenylamino)methyl)(-tolyl)(-tolyl)phosphine oxide (3u)

Following the general procedure, the reaction time was 0.5 h, 3u was prepared as a light yellow solid from phenyl isothiocyanate 1a (54 mg, 0.4 mmol) and di-$o$-tolylphosphine oxide 2d (184 mg, 0.8 mmol), the mixture was diluted and washed with petroleum ether/EtOAc (4: 1). The crude solid was purified by thin layer chromatograph on silica gel with DCM/MeOH = 30: 1 as eluent.

Mp 248–250 °C; $R_f$ = 0.30 (DCM/MeOH = 30:1 v/v); 79 mg, 35% yield.

$^1$H NMR (CDCl$_3$, 500 MHz): $\delta$ 7.75–7.89 (m, 4H), 7.00–7.34 (m, 12H), 6.74–6.96 (m, 2H), 6.40–6.70 (m, 1H), 6.13–6.29 (m, 2H), 5.46–5.58 (m, 1H), 5.06–5.15 (m, 1H), 4.60–4.65 (m, 1H), 2.21 (d, $J = 32.2$ Hz, 24H).

$^{13}$C NMR (CDCl$_3$): $\delta$ 144.9 (s, 1C), 143.1 (s, 2C), 142.4 (s, 2C), 133.1 (s, 2C), 132.2 (s, 2C), 131.8 (s, 4C), 131.7 (s, 4C), 130.4 (dd, $J = 98.7$, 70.8 Hz, 4C), 128.7 (s, 2C), 125.1 (s, 4C), 118.4 (s, 1C), 113.1 (s, 2C), 54.4 (t, $J = 65.0$ Hz, 1C), 21.8 (s, 2C), 21.4 (s, 2C).

HRMS (ESI-TOF, [M + Na]$^+$): calcd for C$_{35}$H$_{35}$NO$_2$NaP$_2$, 586.2041, found 586.2048.

$^1$N$^1$(phenylamino)methylene)bis(bis(3,5-dimethylphenyl)phosphine oxide) (3v)

Following the general procedure, the reaction time was 0.5 h, 3v was isolated as a white solid from phenyl isothiocyanate 1a (54 mg, 0.4 mmol) and bis(3,5-dimethylphenyl)phosphine oxide 2e (206 mg, 0.8 mmol). Mp 238–240 °C; $R_f$ = 0.25 (DCM/MeOH = 30:1 v/v); 161 mg, 65% yield.

$^1$H NMR (CDCl$_3$, 500 MHz): $\delta$ 7.28–7.44 (m, 8H), 6.80–7.10 (m, 6H), 6.48–6.68 (m, 1H), 6.35–6.43 (m, 2H), 5.06–5.15 (m, 1H), 4.60–4.65 (m, 1H), 5.06–5.15 (m, 1H), 4.60–4.65 (m, 1H), 2.21 (d, $J = 32.2$ Hz, 24H).

$^{13}$C NMR (CDCl$_3$): $\delta$ 146.4 (s, 1C), 137.7 (d, $J = 7.0$ Hz, 4C), 133.4 (s, 8C), 130.9 (dd, $J = 102.8$, 76.9 Hz, 4C), 129.2 (s, 4C), 129.0 (s, 4C), 128.5 (s, 2C), 128.5 (s, 2C), 118.6 (s, 1C), 114.0 (s, 2C), 57.2 (t, $J = 64.3$ Hz, 1C), 21.1 (s, 8C).

HRMS (ESI-TOF, [M + Na]$^+$): calcd for C$_{39}$H$_{43}$NO$_2$NaP$_2$, 642.2667, found
Procedure for the synthesis of thioamide 4

To a 15 mL sealed tube was charged with a mixture of phenyl isothiocyanate 1a (54 mg, 0.4 mmol) and diphenylphosphine oxide 2a (162 mg, 0.8 mmol). The reaction mixture was stirred at 60 °C for 0.5 h. After completion, the mixture was cooled to room temperature, added with CH$_3$CN (1.0 mL), and stirred for 10 min. The crude solid was then filtered and washed with CH$_3$CN. After dried in vacuum, the thioamide 4 was obtained as a yellow solid (117 mg, 87%).

1-(diphenylphosphoryl)-N-phenylmethanethioamide (4)$^4$
Yellow solid; mp 160−162 °C (lit.$^4$ mp 161−162 °C); R$_f$ = 0.25 (PE/EA = 4:1 v/v); 117 mg, 87% yield.

$^1$H NMR (CDCl$_3$, 500 MHz): $\delta$ 11.06−11.38 (m, 1H), 8.06−8.15 (m, 2H), 7.95−8.05 (m, 4H), 7.55−7.65 (m, 2H), 7.46−7.53 (m, 4H), 7.38−7.45 (m, 2H), 7.27−7.34 (m, 1H).

Procedure for the synthesis of bisphosphinoylaminomethanes 5

To a 15 mL sealed tube was charged with a mixture of phenyl isothiocyanate 1a (54 mg, 0.4 mmol) and diphenylphosphine oxide 2a (81 mg, 0.4 mmol). The reaction mixture was stirred at 60 °C for 1.0 h. Then, the P-reagent (0.4 mmol) was added to the reaction mixture and stirred at 90 °C or 110 °C. After completion, the mixture was cooled to room temperature, diluted with petroleum ether/EtOAc (4: 1). After filtration, the crude solid was then purified by thin layer chromatograph on silica gel with DCM/MeOH = 30:1 as eluent, the product 5 was afforded as a white solid.

((Di-p-tolylphosphoryl)(phenylamino)methyl)diphenylphosphine oxide (5a)

Following the general procedure, di-p-tolylphosphine oxide (92 mg, 0.4 mmol) was used in the second step, the reaction temperature was 90 °C and reaction time was 2 h. Mp 237−239 °C; R$_f$ = 0.25 (DCM/MeOH = 30:1 v/v); 118 mg, 55% yield.

$^1$H NMR (CDCl$_3$, 500 MHz): $\delta$ 7.62−7.89 (m, 8H), 7.27−7.47 (m, 6H), 7.04−7.18 (m, 4H), 6.82−6.92 (m, 2H), 6.54−6.62 (m, 1H), 6.24−6.36 (m, 2H), 5.01−5.24 (m, 1H), 4.51−4.73 (m, 1H), 2.32 (d, $J$ = 25.6 Hz, 6H). $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ 146.1 (s, 1C), 142.3 (s, 1C), 142.2 (s,
Following the general procedure, bis(3,5-dimethylphenyl)phosphine oxide (103 mg, 0.4 mmol) was used in the second step, the reaction temperature was 110 °C and the reaction time was 0.5 h. Mp 232–234 °C; R_f = 0.25 (DCM/MeOH = 30:1 v/v); 160 mg, 71% yield.

^1^H NMR (500 MHz, CDCl_3): δ 7.90–8.00 (m, 2H), 7.78–7.85 (m, 2H), 7.26–7.41 (m, 10H), 7.00–7.06 (m, 1H), 6.89–6.99 (m, 3H), 6.55–6.64 (m, 1H), 6.33–6.40 (m, 2H), 5.09–5.18 (m, 1H), 4.58–4.64 (m, 1H), 2.21 (d, J = 33.0 Hz, 12H).

^13^C NMR (125 MHz, CDCl_3): δ 146.0 (s, 1C), 137.7 (s, 2C), 137.6 (s, 2C), 133.8 (s, 2C), 132.7 (s, 2C), 132.0 (s, 2C), 131.7 (s, 2C), 130.7 (dd, J = 97.7, 40.9 Hz, 2C), 129.2 (dd, J = 124.7, 19.0 Hz, 2C), 129.5 (s, 2C), 129.0 (s, 2C), 128.5 (s, 4C), 128.0 (s, 2C), 118.5 (s, 1C), 114.2 (s, 2C), 56.7 (t, J = 64.3 Hz, 1C), 21.2 (s, 2C), 21.1 (s, 2C).

HRMS (ESI-TOF, [M + Na]^+): calcd for C_{33}H_{31}NO_2NaP_2, 558.1728, found 558.1730.

Dimethyl ((diphenylphosphoryl)(phenylamino)methyl)phosphonate (5c)

Following the general procedure, dimethyl phosphonate 2f (44 mg, 0.4 mmol) was used in the second step, the reaction temperature was 110 °C and reaction time was 6.5 h. Mp 198–200 °C; R_f = 0.20 (DCM/MeOH = 30:1 v/v); 75 mg, 45% yield.

^1^H NMR (CDCl_3, 500 MHz): δ 7.73–8.05 (m, 4H), 7.36–7.59 (m, 6H), 7.07–7.17 (m, 2H), 6.70–6.77 (m, 1H), 6.56–6.60 (m, 2H), 4.72–4.81 (m, 1H), 4.30–4.35 (m, 1H), 3.53–3.71 (m, 6H).

^13^C NMR (125 MHz, CDCl_3): δ 145.7 (s, 1C), 132.3 (s, 1C), 132.2 (s, 1C), 131.6 (s, 2C), 131.5 (s, 2C), 130.6 (dd, J = 99.7, 29.9 Hz, 2C), 129.2 (s, 2C), 128.5 (s, 2C), 128.4 (s, 2C), 119.1 (s, 1C), 113.7 (s, 2C), 53.5 (t, J = 33.4 Hz, 1C), 52.5 (s, 1C), 52.0 (s, 1C). HRMS (ESI-TOF, [M + Na]^+): calcd for C_{21}H_{23}NO_2NaP_2, 438.1000, found 438.1003.

Ethyl((diphenylphosphoryl)(phenylamino)methyl)(phenyl)phosphinate (5d)

Ethyl((diphenylphosphoryl)(phenylamino)methyl)(phenyl)phosphinate (5d)
Following the general procedure, ethyl phenylphosphinate 2g (68 mg, 0.4 mmol) was used in the second step, the reaction temperature was 90 °C and reaction time was 0.5 h. Mp 206–208 °C; R_f = 0.35 (DCM/MeOH = 30:1 v/v); 99 mg, 52% yield.

^1^H NMR (500 MHz, CDCl₃): δ 7.67–8.00 (m, 6H), 7.29–7.58 (m, 9H), 6.83–7.06 (m, 2H), 6.22–6.70 (m, 3H), 4.76–4.93 (m, 1H), 4.23–4.48 (m, 1H), 3.77–4.01 (m, 2H), 2.04–2.19 (m, 3H).

^1^C NMR (125 MHz, CDCl₃): δ 146.0 (s, 1C), 132.8 (d, J = 9.7 Hz, 1C), 132.5 (s, 2C), 131.9 (s, 1C), 131.8 (s, 1C), 131.6 (d, J = 8.6 Hz, 1C), 131.4 (d, J = 8.9 Hz, 1C), 131.2 (d, J = 8.7 Hz, 1C), 130.0 (dd, J = 131.6, 54.9 Hz, 2C), 128.9 (s, 1C), 128.3 (s, 9C), 118.8 (d, J = 10.0 Hz, 1C), 113.8 (d, J = 14.0 Hz, 1C), 61.8 (s, 1C), 55.3 (t, J = 54.4 Hz, 1C), 29.7 (s, 1C). HRMS (ESI-TOF, [M + Na]^+): calcd for C_{27}H_{27}NO_3NaP_2, 498.1364, found 498.1370.

References

Profile of the reaction of 1b with 2a

![Diagram of the reaction of 1b with 2a]

**Figure S2.** Profile of the reaction of 1b with 2a under solvent-free conditions.
Hammett correlation study

A mixture of the equimolar amount of substituted thioamide 4 (0.1 mmol) and diphenylphosphine oxide (0.1 mmol) was stirred at 110 °C for 20 min. The reaction mixture was cooled to room temperature. The resulting mixture was analyzed by 1H NMR for determination of yield using 1, 3, 5-trimethoxybenzene (16.8 mg, 0.1 mmol) as the internal standard. The values were listed below:

![Graph showing Hammett correlation study.](image)

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**Figure S3.** Hammett correlation study.

Testing the generation of H₂S with lead acetate strip

To prove H₂S generation, lead acetate test strip was pasted on the Teflon cover of the sealed tube before reaction. Subsequently, the reaction mixture of isothiocyanate and diphenylphosphine oxide was heated to 110 °C. With the reaction proceeding, the lead acetate strip gradually turned to black. After 3 h, the whole paper became totally dark black which confirmed the elimination of
H₂S.

**EDS spectrum for the detection of elemental sulfur**

*Procedure for the Separation of Elemental Sulfur.* A 5 mmol (based on 1a) scale reaction was conducted under standard conditions. After filtration, the filtrate was purified by silica gel column chromatography, the resultant mixture was dissolved in methanol, then the solid were precipitated (52 mg) and used for EDS analysis.

![Figure S5. The EDS spectrum and data analysis.](image)

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Time                 14.28
INSTRUM              spect
PROBHD               5 mm PABBO BB-
PULPRES
TD                   2g
SOLVENT             CDC13
NS                   8
DS                   2
SWH                  10000.000 Hz
FIDRES              0.610352 Hz
AQ                   0.8193000 sec
RG                   181
DW                   50.000 usec
DE                   8.00 usec
TE                   673.2 K
D1                   2.00000000 sec
TD0                  1

---------- CHANNEL f1 ----------
NUC1                 1H
P1                   13.00 usec
PL1                  2.00 dB
SFO1                 500.0335000 MHz
SI                   16384
SF                   500.0300052 MHz
WDW                  EM
SSB                  0
LB                   0.30 Hz
GB                   0
PC                   1.00
NAME  SUN-P-ME
EXPN0    1
PROCNO    1
Date_    20170419
Time   17.39
INSTRUM  spect
PROBHD  5 mm PABBO BB-
PULPROG  zg
TD     16384
SOLVENT  CDC13
NS      8
DS      2
SWH     10000.000  Hz
FIDRES  0.610352   Hz
AQ      0.8193000  sec
RG      256
DW      50.000  usec
DE      8.000   usec
TE      673.2  K
D1     2.00000000  sec
TDO     1

---------- CHANNEL f1 ----------
NUC1     1H
P1      13.00  usec
PL1     2.00  dB
SFO1    500.035000  MHz
SI     16384
SF     500.0300013  MHz
WDW    EM
SSB     0
LB      0.30  Hz
GB      0
PC      1.00