Supporting Information

* N-Triflylphosphorimidoyl trichloride: A Versatile Reagent for the Synthesis of Strong Chiral Brønsted acids

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1. General information

All reactions were carried out under argon atmosphere in flame dried glassware with magnetic stirring. Unless otherwise stated, all reagents were purchased from commercial suppliers and used without further purification. All solvents employed in the reactions were distilled from appropriate drying agents prior to use. Organic solutions were concentrated under reduced pressure on a Büchi rotary evaporator. Reactions were monitored by thin layer chromatography using 0.2 mm Macherey-Nagel silica gel precoated plates (POLYGRAM SIL G/UV254). Visualization was accomplished by irradiation with UV light at 254 nm or staining with PMA or anisaldehyde solution. Flash column chromatography was performed using Merck silica gel 60 (particle size 0.040–0.063 mm). Chemical yields refer to pure isolated substances. All NMR spectra were recorded at 25 °C on a Bruker Avance III 500 MHz, a Bruker Avance III 400 MHz and a Bruker Avance III HD 300 MHz spectrometers using CD$_2$Cl$_2$ and acetone-d$_6$ as solvent. The ppm of solvent residues was assigned as internal reference in $^1$H NMR spectra (e.g. CD$_2$Cl$_2$ = 5.32 ppm, acetone-d$_6$ = 2.05 ppm). $^{13}$C, $^{19}$F, and $^{31}$P NMR spectra were referenced according to δ-values (IUPAC recommendations 2008) relative to the internal references set in $^1$H NMR spectra. The following abbreviations were used to designate chemical shift multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, hept = heptet, m = multiplet, b = broad. MS (EI): Mass spectra were recorded on a Finnigan MAT 8200 at 70 eV in the EI mode. High resolution mass spectra were determined on a Bruker APEX III FT-MS (7 T magnets).
2. General procedure for the synthesis of \( N \)-triflylphosphoramides

In a flame dried vial under Ar, the corresponding \((S)\)- or \((R)\)-BINOL (1.0 equiv) was dissolved in anhydrous \( \text{CH}_2\text{Cl}_2 \) (0.20 M). \( \text{TfNPCl}_3 \) (1.1 equiv) and DIPEA (5.0 equiv) were added and the mixture was stirred for 10 min at ambient temperature. After the full consumption of the starting material was observed (TLC), \( \text{H}_2\text{O} \) (20 \( \mu \)L) was added. With an additional 10 min stirring, the reaction mixture was dried over \( \text{Na}_2\text{SO}_4 \), filtered, concentrated, and purified by column chromatography on silica gel to afford the desired product as a salt. Acidification in \( \text{CH}_2\text{Cl}_2 \) with \( \text{HCl} \) (3.0 M) followed by drying under reduced pressure afforded the desired product as a free acid.

\((S)-[3,3'\text{-Diphenyl-1,1'\text{-binaphthalen-2,2'\text{-yl}]\text{-N-triflyl phosphoramid}}e \ (6a)}\)

0.05 mmol scale, 31 mg, 98%, brown solid

\(^1\text{H NMR} \ (501 \text{ MHz, CD}_2\text{Cl}_2) \ \delta \ 8.14 \ (s, \ 1\text{H}), \ 8.09 \ (s, \ 1\text{H}), \ 8.05 \ (dd, \ J = 8.4, \ 1.1 \text{ Hz, 1H}), \ 8.00 \ (dd, \ J = 8.2, \ 1.1 \text{ Hz, 1H}), \ 7.72 - 7.67 \ (m, \ 2\text{H}), \ 7.60 (dd, \ J = 8.6, \ 1.1 \text{ Hz, 1H}), \ 7.29 \ (dd, \ J = 8.6, \ 6.7 \text{ Hz, 1H}).

\(^{13}\text{C NMR} \ (126 \text{ MHz, CD}_2\text{Cl}_2) \ \delta \ 143.53, \ 142.73, \ 136.01, \ 135.98, \ 133.53, \ 133.36, \ 133.34, \ 131.96, \ 131.93, \ 131.83, \ 131.80, \ 130.00, \ 129.71, \ 128.54, \ 128.53, \ 128.47, \ 128.11, \ 128.09, \ 127.87, \ 126.95, \ 126.94, \ 126.79, \ 126.69, \ 126.62, \ 126.38, \ 122.21 \ (d, \ J = 2.0 \text{ Hz}), \ 122.19 \ (d, \ J = 3.0 \text{ Hz}), \ 118.71 \ (qd, \ J = 322.1, \ 1.6 \text{ Hz}).

\(^{19}\text{F NMR} \ (471 \text{ MHz, CD}_2\text{Cl}_2) \ \delta \ -77.8.

\(^{31}\text{P NMR} \ (203 \text{ MHz, CD}_2\text{Cl}_2) \ \delta \ -5.8.

\text{HRMS (ESI, C}_{33}\text{H}_{20}\text{F}_{3}\text{NO}_{5}\text{PS, } M-H^+) \ : m/z \text{ calcld. 630.0757, found 630.0759.}

\((R)-[3,3'\text{-Di(4-phenylphenyl-1,1'\text{-binaphthalen-2,2'\text{-yl}]\text{-N-triflyl phosphoramid}}e \ (6b)}\)

0.05 mmol scale, 38 mg, 97%, brown solid

\(^1\text{H NMR} \ (501 \text{ MHz, CD}_2\text{Cl}_2) \ \delta \ 8.22 \ (s, \ 1\text{H}), \ 8.12 \ (s, \ 1\text{H}), \ 8.07 \ (ddd, \ J = 9.8, \ 8.4, \ 1.1 \text{ Hz, 2H}), \ 7.78 \ (d, \ J = 8.4 \text{ Hz, 2H}), \ 7.72 \ (d, \ J = 8.3 \text{ Hz, 2H}), \ 7.69 \ (d, \ J = 8.3 \text{ Hz, 2H}), \ 7.67 - 7.58 \ (m, \ 5\text{H}), \ 7.56 - 7.51 \ (m, \ 3\text{H}), \ 7.44 - 7.37 \ (m, \ 4\text{H}), \ 7.37 - 7.27 \ (m, \ 3\text{H}), \ 7.17 - 7.08 \ (m, \ 3\text{H}).

\(^{13}\text{C NMR} \ (126 \text{ MHz, CD}_2\text{Cl}_2) \ \delta \ 144.1, \ 144.0, \ 143.6, \ 143.5, \ 141.5, \ 141.1, \ 140.9, \ 135.7, \ 135.4, \ 133.8, \ 133.8, \ 133.6, \ 133.6, \ 132.7, \ 132.6, \ 132.5, \ 132.4, \ 132.3, \ 131.1, \ 130.7, \ 129.3, \ 129.2, \ 129.1, \ 128.0, \ 127.8, \ 127.8, \ 127.7, \ 127.6, \ 127.5, \ 127.4, \ 127.4, \ 127.3, \ 127.3, \ 127.1, \ 123.2, \ 123.1, \ 122.8, \ 122.8, \ 120.6, \ 118.1.

\(^{19}\text{F NMR} \ (471 \text{ MHz, CD}_2\text{Cl}_2) \ \delta \ -78.1.

\(^{31}\text{P NMR} \ (203 \text{ MHz, acetone-d}_6) \ \delta \ -5.2.

\text{HRMS (ESI, C}_{45}\text{H}_{28}\text{F}_{3}\text{NO}_{5}\text{PS, } M-H^+) \ : m/z \text{ calcld. 782.1383, found 782.1389.}
(S)-[3,3′-Di(1-naphtyl)-1,1′-binaphthalen-2,2′-yl]-N-triflyl phosphoramidate (6c)

0.05 mmol scale, 33 mg, 90%, brown solid

The spectrum is complicated due to the rotatory isomers.

\(^1^H\) NMR (501 MHz, CD\(_2\)Cl\(_2\)) δ 8.20 – 7.99 (m, 4H), 7.78 (dd, J = 12.6, 8.0 Hz, 1H), 7.72 – 7.27 (m, 16H).

\(^{13}\)C NMR (126 MHz, CD\(_2\)Cl\(_2\)) δ 144.5, 143.6, 134.4, 134.3, 134.1, 133.9, 133.7, 133.6, 133.6, 133.2, 133.2, 133.1, 133.0, 132.8, 132.3, 132.2, 132.1, 132.0, 131.9, 131.8, 131.8, 131.4, 129.1, 128.9, 128.8, 128.7, 128.6, 128.6, 128.5, 128.5, 128.4, 128.4, 128.3, 128.2, 128.0, 127.2, 127.1, 127.1, 127.1, 127.0, 127.0, 126.7, 126.7, 126.6, 126.5, 126.3, 126.2, 126.1, 126.1, 126.0, 125.9, 125.8, 125.7, 125.7, 125.6, 125.6, 125.5, 125.4, 125.3, 125.1, 125.0, 124.9, 124.8, 121.5.

\(^{19}\)F NMR (471 MHz, CD\(_2\)Cl\(_2\)) δ –78.0, –78.2, –78.5.

\(^{31}\)P NMR (203 MHz, CD\(_2\)Cl\(_2\)) δ –4.4, –5.4, –6.2.

HRMS (ESI, C\(_{41}\)H\(_{24}\)F\(_3\)NO\(_5\)PS, M–H\(^+\)): m/z calcd. 730.1070, found 730.1075.

(R)-[3,3′-Di(2-naphtyl)-1,1′-binaphthalen-2,2′-yl]-N-triflyl phosphoramidate (6d)

0.05 mmol scale, 35 mg, 96%, brown solid

\(^1^H\) NMR (501 MHz, CD\(_2\)Cl\(_2\)) δ 8.27 (s, 1H), 8.22 (s, 1H), 8.17 – 8.15 (m, 1H), 8.14 (dd, J = 8.4, 1.1 Hz, 1H), 8.08 (dd, J = 8.0, 1.2 Hz, 1H), 8.05 – 8.03 (m, 1H), 7.93 – 7.86 (m, 2H), 7.85 – 7.76 (m, 4H), 7.70 – 7.62 (m, 2H), 7.60 (d, J = 8.3 Hz, 1H), 7.56 (ddd, J = 8.1, 6.6, 1.3 Hz, 1H), 7.52 (dd, J = 8.5, 1.1 Hz, 1H), 7.49 – 7.42 (m, 3H), 7.39 (dd, J = 8.5, 1.1 Hz, 1H), 7.34 (ddt, J = 8.0, 6.8, 1.3 Hz, 2H), 7.10 (t, J = 7.6 Hz, 1H).

\(^{13}\)C NMR (126 MHz, CD\(_2\)Cl\(_2\)) δ 143.7, 143.6, 143.1, 143.0, 143.0, 133.6, 133.6, 133.5, 133.5, 133.3, 133.0, 132.8, 132.7, 132.1, 132.0, 132.0, 131.9, 129.5, 128.9, 128.6, 128.2, 128.2, 127.9, 127.7, 127.5, 127.5, 127.4, 127.2, 127.0, 126.9, 126.8, 126.7, 126.7, 126.5, 126.2, 126.2, 126.1, 126.0, 122.5, 122.5, 122.3, 122.3, 119.9, 117.3.

\(^{19}\)F NMR (471 MHz, CD\(_2\)Cl\(_2\)) δ –78.5.

\(^{31}\)P NMR (203 MHz, CD\(_2\)Cl\(_2\)) δ –5.3.

HRMS (ESI, C\(_{41}\)H\(_{24}\)F\(_3\)NO\(_5\)PS, M–H\(^+\)): m/z calcd. 730.1070, found 730.1077.
(S)-[3,3'-Di(9-phenanthryl)-1,1'-binaphthalen-2,2'-yl]-N-triflyl phosphoramide (6e)

0.05 mmol scale, 37 mg, 89%, brown solid

The spectrum is complicated due to the rotatory isomers.

^1H NMR (501 MHz, CD2Cl2) δ 8.85 – 8.58 (m, 4H), 8.30 – 8.14 (m, 2H), 8.14 – 8.03 (m, 2H), 8.00 (s, 1H), 7.99 – 7.92 (m, 1H), 7.92 – 7.73 (m, 3H), 7.73 – 7.43 (m, 15H).

^13C NMR (126 MHz, CD2Cl2) δ 144.7, 133.7, 133.3, 133.2, 133.0, 132.9, 132.8, 132.5, 132.5, 132.4, 132.3, 132.3, 132.2, 132.2, 132.1, 131.4, 131.3, 131.3, 131.1, 131.0, 131.0, 130.9, 130.5, 130.4, 130.2, 130.1, 130.1, 130.0, 129.9, 129.9, 129.9, 129.7, 129.4, 129.2, 128.8, 128.6, 128.6, 128.6, 127.3, 127.2, 127.2, 127.1, 127.1, 127.1, 127.0, 126.9, 126.9, 126.8, 126.7, 126.7, 126.6, 126.6, 126.6, 126.4, 126.4, 126.3, 123.1, 122.8, 122.6, 122.6, 122.4, 122.3, 122.3, 122.2, 121.9, 121.6, 121.6.

^19F NMR (471 MHz, CD2Cl2) δ −78.0, −78.3, −78.5.

^31P NMR (203 MHz, CD2Cl2) δ −5.0, −5.4, −5.8.

HRMS (ESI, C49H28F3NO5PS, M−H^+) : m/z calcd. 830.1383, found 830.1391.

(S)-[3,3'-Di[3,5-bis(trifluoromethyl)phenyl]-1,1'-binaphthalen-2,2'-yl]-N-triflyl phosphoramide (6f)

0.05 mmol scale, 44 mg, 97%, white solid

^1H NMR (501 MHz, acetone-d6) δ 8.48 (d, J = 1.0 Hz, 2H), 8.47 – 8.44 (m, 2H), 8.40 – 8.35 (m, 2H), 8.23 (ddt, J = 8.2, 5.0, 0.8 Hz, 2H), 8.07 (ddt, J = 13.5, 1.7, 0.9 Hz, 2H), 7.67 – 7.60 (m, 2H), 7.52 – 7.46 (m, 1H), 7.43 (ddd, J = 7.2, 5.2, 1.2 Hz, 2H), 7.37 (dd, J = 8.6, 1.0 Hz, 1H).

^13C NMR (126 MHz, acetone-d6) δ 143.5, 143.4, 143.4, 143.1, 143.0, 138.4, 138.4, 138.4, 131.6, 131.5, 131.2, 131.2, 130.7, 130.6, 130.3, 130.3, 130.3, 130.3, 130.1, 130.1, 130.0, 129.8, 129.7, 129.5, 129.5, 128.1, 128.1, 126.6, 126.5, 125.6, 125.6, 125.5, 125.4, 123.8, 123.7, 122.1, 122.1, 121.8, 121.8, 121.6, 121.6, 120.5, 120.5, 120.4, 120.4, 120.4, 120.3, 120.3, 120.2, 120.2, 120.2. (The spectrum is complicated due to C-F coupling.)

^19F NMR (471 MHz, acetone-d6) δ −64.1 (12F), −81.56 (3F).

^31P NMR (203 MHz, acetone-d6) δ −1.2.

HRMS (ESI, C37H16F15NO3PS, M−H^+) : m/z calcd. 902.0253, found 902.0261.
(S)-[3,3'-Di(bis(2,4,6-triisopropyl)phenyl)-1,1'-binaphthalen-2,2'-yl]-N-triflyl phosphoramidate (6g)

A reaction time was prolonged to 1 h for each step.

0.05 mmol scale, 36 mg, 82%, brown solid

\(^1\)H NMR (501 MHz, CD\(_2\)Cl\(_2\)) \(\delta\) 8.00 (dd, \(J = 8.4, 2.9\) Hz, 4H), 7.57 (dddd, \(J = 8.1, 6.8, 3.3, 1.2\) Hz, 2H), 7.35 (ddd, \(J = 8.5, 7.0, 1.3\) Hz, 2H), 7.29 (d, \(J = 8.6\) Hz, 1H), 7.25 (d, \(J = 8.5\) Hz, 1H), 7.20 (d, \(J = 1.8\) Hz, 1H), 7.15 (dd, \(J = 9.4, 1.8\) Hz, 2H), 7.08 (d, \(J = 1.7\) Hz, 1H), 3.01 – 2.90 (m, 2H), 2.78 – 2.65 (m, 3H), 2.57 (hept, \(J = 6.8\) Hz, 1H), 1.33 – 1.25 (m, 15H), 1.25 – 1.15 (m, 12H), 1.10 (d, \(J = 6.8\) Hz, 3H), 0.99 (d, \(J = 6.8\) Hz, 3H), 0.95 (d, \(J = 6.8\) Hz, 3H).

\(^{13}\)C NMR (126 MHz, CD\(_2\)Cl\(_2\)) \(\delta\) 149.74, 149.16, 148.01, 147.73, 147.14, 146.53, 145.26 (d, \(J = 11.2\) Hz), 144.41 (d, \(J = 8.8\) Hz), 133.18, 133.08, 132.16, 132.11, 132.08, 131.60, 131.22, 130.28, 130.25, 130.19, 129.94, 128.35, 128.31, 127.14, 127.09, 126.72, 126.66, 126.40, 126.33, 121.68, 121.22, 121.09, 121.03, 120.28, 118.46 (q, \(J = 248.3\) Hz), 34.46, 34.36, 31.40, 31.03, 31.01, 30.51, 26.63, 26.47, 24.99, 24.80, 23.80, 23.71, 23.67, 23.61, 22.84, 22.62, 22.52, 22.30.

\(^{19}\)F NMR (471 MHz, CD\(_2\)Cl\(_2\)) \(\delta\) –77.4.

\(^{31}\)P NMR (203 MHz, CD\(_2\)Cl\(_2\)) \(\delta\) –4.5.

HRMS (ESI, C\(_{51}\)H\(_{56}\)F\(_3\)NO\(_5\)PS, M–H\(^+\)): \(m/z\) calcd. 882.3574, found 882.3580.
3. The synthesis of N-triflylthiophosphoramides and N,N'-bis(triflyl)phosphoramidimidates

In a flame dried vial under Ar, the corresponding (S)-BINOL (1.0 equiv) was dissolved in anhydrous CH₂Cl₂ (0.20 M). TfNPCl₃ (1.1 equiv) and DIPEA (5.0 equiv) were added and the reaction mixture was stirred for 10 min at ambient temperature. After the full consumption of the starting material was observed (TLC), H₂S (0.8 M in THF, 2.0 equiv) or TfNH₂ (2.0 equiv) was added. With an additional 10 min stirring, the reaction mixture was filtered through celite, concentrated, and purified by column chromatography on silica gel to afford the desired product as a salt. Acidification in CH₂Cl₂ with HCl (3.0 M) followed by drying under reduced pressure afforded the desired product as a free acid.

(S)-[3,3'-Di(3,5-bis(trifluoromethyl)phenyl)-1,1'-binaphthalen-2,2'-yl]-N-triflyl thiophosphoramide (7a)

0.05 mmol scale, 44 mg, 96%, white solid

**¹H NMR** (501 MHz, CD₂Cl₂) δ 8.28 – 8.22 (m, 4H), 8.15 (d, J = 1.6 Hz, 2H), 8.14 – 8.10 (m, 2H), 7.99 (dd, J = 8.6, 1.8 Hz, 2H), 7.66 (dq, J = 8.2, 4.1 Hz, 2H), 7.49 – 7.44 (m, 4H).

**¹³C NMR** (126 MHz, CD₂Cl₂) δ 144.27, 144.14, 142.05, 141.98, 138.77, 137.97, 132.54, 132.52, 132.28, 132.21, 132.08, 131.95, 131.83, 131.68, 131.56, 131.43, 131.41, 131.30, 131.03, 130.56, 130.53, 130.48, 130.35, 130.32, 128.94, 128.91, 127.95, 127.33, 127.15, 126.89, 126.63, 126.60, 124.46, 124.44, 123.52, 123.49, 123.07, 122.30, 122.27, 122.03, 122.00, 121.97, 121.84, 121.81, 121.78, 120.14, 119.97, 117.42. (The spectrum is complicated due to C-F coupling.)

**¹⁹F NMR** (471 MHz, CD₂Cl₂) δ –63.3 (6F), –63.3 (6F), –76.7 (3F).

**³¹P NMR** (203 MHz, CD₂Cl₂) δ 54.9.

**HRMS** (ESI, C₃₇H₁₆F₁₅NO₄PS₂, M–H⁺): m/z calcd. 918.0024, found 918.0030.

(S)-[3,3'-Bis(2,4,6-trisopropylphenyl)-1,1'-binaphthalen-2,2'-yl]-N-triflyl thiophosphoramide (7b)

The reaction time was prolonged to 1 h for the first step and 24 h for the reaction with H₂S.

0.05 mmol scale, 34 mg, 75%, brown solid

**¹H NMR** (501 MHz, CD₂Cl₂) δ 8.05 – 7.98 (m, 4H), 7.59 – 7.53 (m, 2H), 7.32 (m, 2H), 7.24 – 7.20 (m, 2H), 7.19 (d, J = 1.8 Hz, 1H), 7.16 (d, J = 1.8 Hz, 1H), 7.15 – 7.11 (m, 1H), 7.06 (d, J = 1.8 Hz, 1H), 3.09 – 2.90 (m, 3H), 2.82 (hept, J = 6.7 Hz, 1H), 2.76 (hept, J = 6.8 Hz, 1H), 2.57 (hept, J = 6.8 Hz, 1H), 1.35 – 1.28 (m, 21H), 1.23 (d, J = 6.8 Hz, 3H), 1.18 (d, J = 6.8 Hz, 3H), 1.10 (d, J = 6.8 Hz, 3H), 0.97 (d, J = 6.8 Hz, 3H), 0.86 (d, J = 6.8 Hz, 3H).

**¹³C NMR** (126 MHz, CD₂Cl₂) δ 149.75, 149.02, 148.51, 147.43, 147.06, 146.23, 146.12, 146.00, 144.55, 144.48, 133.41, 133.00, 132.40, 132.34, 132.14, 132.12, 131.42, 131.20, 130.60, 129.95, 129.92, 129.83, 128.31, 126.97, 126.74, 126.61, 126.32, 126.26, 122.42, 122.39, 121.83, 121.64, 121.62, 121.19, 120.11, 118.65 (d, J = 323.0 Hz), 34.40, 34.30, 31.47, 30.97, 30.88, 30.49, 27.12, 26.73, 25.01, 24.87, 23.90, 23.72, 23.59, 23.55, 23.42, 23.27, 22.72, 21.85.
$^{19}$F NMR (471 MHz, CD$_2$Cl$_2$) δ –75.6.

$^{31}$P NMR (203 MHz, CD$_2$Cl$_2$) δ 52.2.

HRMS (ESI, C$_{51}$H$_{56}$F$_3$NO$_4$PS$_2$, M–H$^+$): m/z calcd. 898.3346, found 898.3354.

(S)-3,3'-Di(3,5-bis(trifluoromethyl)phenyl)-[1,1'-binaphthalene]-2,2'-dinaphthyl-N,N'-bis-((trifluoromethyl)sulfonyl)phosphoramidimidate (8a)

The reaction time was prolonged to 1 h for the first step and 24 h for the reaction with H$_2$S.

0.05 mmol scale, 50 mg, 97%, white solid

$^1$H NMR (501 MHz, CD$_2$Cl$_2$) δ 8.25 (s, 2H), 8.17 (d, $J$ = 1.6 Hz, 4H), 8.14 (dd, $J$ = 8.4, 1.1 Hz, 2H), 8.00 (d, $J$ = 1.8 Hz, 2H), 7.68 (ddd, $J$ = 8.1, 6.4, 1.4 Hz, 2H), 7.52 – 7.41 (m, 4H).

$^{13}$C NMR (126 MHz, CD$_2$Cl$_2$) δ 142.38, 142.29, 137.66, 132.83, 132.25, 132.16, 131.90, 131.64, 131.37, 130.43, 130.41, 130.28, 130.25, 128.97, 128.15, 127.54, 126.90, 126.51, 124.34, 122.36, 122.34, 122.21, 122.18, 122.15, 122.12, 122.09, 120.01, 119.96, 117.47. (The spectrum is complicated due to C-F coupling.)

$^{19}$F NMR (471 MHz, CD$_2$Cl$_2$) δ –63.4 (12F), –76.2 (6F).

$^{31}$P NMR (203 MHz, CD$_2$Cl$_2$) δ –3.7.

HRMS (ESI, C$_{38}$H$_{16}$F$_{18}$N$_2$O$_6$PS$_2$, M–H$^+$): m/z calcd. 1032.9906, found 1032.9913.

(S)-3,3'-Di(2,4,6-triisopropylphenyl)-[1,1'-binaphthalene]-2,2'-dinaphthyl-N,N'-bis((trifluoromethyl)sulfonyl)phosphoramidimidate (8b)

The reaction time was prolonged to 1 h for the first step and 24 h for the reaction with H$_2$S.

0.05 mmol scale, 37 mg, 72%, brown solid

$^1$H NMR (501 MHz, CD$_2$Cl$_2$) δ 7.98 – 7.95 (m, 2H), 7.92 (s, 2H), 7.53 (ddd, $J$ = 8.0, 6.7, 1.0 Hz, 2H), 7.30 (ddd, $J$ = 8.3, 6.8, 1.3 Hz, 2H), 7.18 (d, $J$ = 1.8 Hz, 2H), 7.12 – 7.06 (m, 4H), 2.95 (hept, $J$ = 6.8 Hz, 2H), 2.87 (hept, $J$ = 6.5 Hz, 2H), 2.58 (hept, $J$ = 6.8 Hz, 2H), 1.29 (dd, $J$ = 6.9, 1.2 Hz, 12H), 1.25 (t, $J$ = 6.8 Hz, 12H), 1.11 (d, $J$ = 6.8 Hz, 6H), 0.89 (d, $J$ = 6.8 Hz, 6H).

$^{13}$C NMR (126 MHz, CD$_2$Cl$_2$) δ 149.04, 147.53, 147.42, 145.15, 145.07, 133.20, 132.47, 131.21, 131.16, 131.12, 130.77, 128.20, 126.68, 126.65, 126.10, 121.14, 121.12, 121.10, 120.35, 34.38, 30.90, 30.80, 26.37, 24.72, 23.75, 23.66, 22.94, 21.82, 19.05, 18.96.

$^{19}$F NMR (471 MHz, CD$_2$Cl$_2$) δ –79.4.

$^{31}$P NMR (203 MHz, CD$_2$Cl$_2$) δ –1.7.

HRMS (ESI, C$_{52}$H$_{56}$F$_3$N$_2$O$_6$PS$_2$, M–H$^+$): m/z calcd. 1013.3227, found 1013.3237.