Supporting Information

Experimental Section

All experiments were carried out under a dry Argon atmosphere using standard Schlenk techniques or in a glovebox. Solvents (including deuterated solvents used for NMR) were dried and distilled prior to use. $^1$H, $^{13}$C, $^{11}$B, $^{19}$F, $^{31}$P NMR spectra were recorded on a Bruker AV 300 MHz, Varian 500 MHz INOVA or a Varian 600 MHz UNITY plus NMR spectrometer at ambient temperature unless otherwise stated. Chemical shifts are given in ppm relative to solvents ($^1$H and $^{13}$C) or an external standard [$\delta$(BF$_3$·OEt$_2$) = 0 for $^{11}$B NMR, $\delta$(CFCl$_3$) = 0 for $^{19}$F NMR and $\delta$(85% H$_3$PO$_4$) = 0 for $^{31}$P NMR). Coupling constants are in Hz. Elemental analysis data was recorded on a Elementar Vario El III. HRMS was recorded on GTC Waters Micromass (Manchester, UK). IR spectra were recorded on a Varian 3100 FT-IR (Excalibur Series). Melting points were obtained with a DSC 2010 (TA Instruments). X-ray structure analysis: Data sets were collected with a Nonius KappaCCD diffractometer. Programs used: data collection COLLECT (Nonius B.V., 1998), data reduction Denzo-SMN (Z. Otwinowski, W. Minor, Methods in Enzymology, 1997, 276, 307-326), absorption correction Denzo (Z. Otwinowski, D. Borek, W. Majewski & W. Minor, Acta Cryst. 2003, A59, 228-234), structure solution SHELXS-97 (G.M. Sheldrick, Acta Cryst. 1990, A46, 467-473), structure refinement SHELXL-97 (G.M. Sheldrick, Acta Cryst. 2008, A64, 112-122), graphics XP (BrukerAXS, 2000). Graphics show the thermal ellipsoids with 50 % probability, $R$ values are given for the observed reflections, $wR^2$ values for all reflections. CCDC reference numbers 826907 - 826909.

B(C$_6$F$_5$)$_3$: was prepared according to procedures reported in the literature (caution: the intermediate involved is explosive) [(a) A. G. Massey, A. J. Park, J. Organomet. Chem. 1964, 2, 245-250. (b) C. Wang, G. Erker, G. Kehr, K. Wedeking and R. Fröhlich, Organometallics, 2005, 24, 4760-4773]. Compound 3c was prepared according to modified procedures reported in the literature [(a) A. D. Miller, S. A. Johnson, K. A. Tupper, J. L. McBee, T. D. Tilley, Organometallics, 2009, 28, 1252–1262; (b) A. Samb, B. Demerseman, P. H. Dixneuf, C. Mealli, Organometallics, 1988, 7, 26–33].
Synthesis of 3c. p-Tolylacetylene (0.49 ml, 3.84 mmol) was dissolved in diethylether (15 ml). Then n-butyllithium solution (1.6 M in hexane, 2.4 ml, 3.84 mmol) was added at -78°C. The solution was stirred for 2h at room temperature. Subsequently the reaction mixture was again cooled to -78°C and a solution of chlorodimesitylphosphane (1.17 g, 3.84 mmol) in diethylether (15 ml) was added. The reaction mixture was warmed to room temperature and stirred for 3h. The solvent was removed in vacuum and the residue was extracted with pentane (30 ml) and the product (0.584 g, 1.52 mmol, 40%) could be isolated as a white-yellow solid. Crystals suitable for X-ray crystal structure analysis were grown by slow diffusion of pentane into a solution of 3c in dichloromethane at –36 °C. 

Anal. Calc. for C_{27}H_{29}P: C, 84.34; H, 7.60. Found: C, 84.05; H, 7.43.

IR (KBr): $\tilde{\nu}$/ cm$^{-1}$ = 2917 (br m), 2148 (w), 1913 (w), 1601 (m), 1463 (s), 1226 (m), 1103 (m), 849 (s), 618 (s), 534 (s).

M.p. (DSC): 124°C.

$^1$H NMR (500 MHz, 298 K, C$_6$D$_6$): $\delta$ = 7.23 (d, $^3$J$_{HH}$ = 8.1 Hz, 2H, o-Tol), 6.71 (d, $^3$J$_{HH}$ = 8.1 Hz, 2H, m-Tol), 6.69 (d, $^4$J$_{PH}$ = 3.3 Hz, 4H, m-Mes), 2.58 (s, 12H, o-CH$_3$Mes), 2.05 (s, 6H, p-CH$_3$Mes), 1.91 (s, 3H, Me).

$^{13}$C{$^1$H} NMR (126 MHz, 298 K, C$_6$D$_6$): $\delta$ = 142.3 (d, $^2$J$_{PC}$ = 15.7 Hz, o-Mes), 138.4 (p-Tol), 138.3 (p-Mes), 131.4 (d, $^4$J$_{PC}$ = 2.1 Hz, o-Tol), 130.6 (d, $^1$J$_{PC}$ = 12.7 Hz, i-Mes), 130.4 (d, $^3$J$_{PC}$ = 3.6 Hz, m-Mes), 129.3 (m-Tol), 121.3 (d, $^3$J$_{PC}$ = 1.5 Hz, i-Tol), 107.6 (d, $^2$J$_{PC}$ = 8.4 Hz, TolC≡), 87.8 (d, $^1$J$_{PC}$ = 6.2 Hz, ≡C≡), 23.3 (d, $^3$J$_{PC}$ = 13.3 Hz, o-CH$_3$Mes), 21.2 (Me), 20.9 (p-CH$_3$Mes).

$^{31}$P{$^1$H} NMR (202 MHz, 298 K, C$_6$D$_6$): $\delta$ = -55.7 ($v_{1/2}$ ~ 1 Hz).

$^1$H,$^1$H GCOSY (500 MHz / 500 MHz, 298 K, C$_6$D$_6$): $\delta^1$H / $\delta^1$H = 7.23 / 6.71 (o-Tol / m-Tol), 6.42 / 2.58, 2.05 (m-Mes / o-CH$_3$Mes, p-CH$_3$Mes).

$^1$H,$^{13}$C GHSQC (500 MHz / 126 MHz, 298 K, C$_6$D$_6$): $\delta^1$H / $\delta^{13}$C = 7.23 / 131.4 (o- Tol, m-Tol), 6.71 / 129.3 (m- Tol), 6.69 / 130.4 (m-Mes), 2.58 / 23.3 (o-CH$_3$Mes), 2.05 / 20.9 (p-CH$_3$Mes), 1.91 / 21.2 (Me).

$^1$H,$^{13}$C GHMBC (500 MHz / 126 MHz, 298 K, C$_6$D$_6$): $\delta^1$H / $\delta^{13}$C = 7.23 / 138.4, 107.6 (o-Tol / p-Tol, TolC≡), 6.71 / 121.3, 21.2 (m-Tol / i-, p-Tol), 6.69 / 130.6, 23.3, 20.9 (m-Mes / i-Mes, o-, p-CH$_3$Mes), 2.58 / 142.3, 130.4 (o-CH$_3$Mes / i- , m -Mes), 2.05 / 138.3, 130.4, 121.4 (p-CH$_3$Mes / p-, m-Mes), 1.91 / 138.4, 129.3, 121.3 (Me / p-Tol, m-Tol, i-Tol).
X-Ray crystal structure analysis of 3c. Crystal data for C_{27}H_{29}P, \(M = 384.47\), triclinic, \(P\overline{1}\) (No. 2), \(a = 10.1751(5)\) Å, \(b = 10.6783(4)\) Å, \(c = 11.6493(7)\) Å, \(\alpha = 96.462(2)\), \(\beta = 107.570(4)\), \(\gamma = 109.165(4)\)°, \(V = 1107.87(10)\) Å^3, \(D_c = 1.153\) g cm\(^{-3}\), \(\mu = 1.142\) mm\(^{-1}\), \(F(000) = 412\), \(Z = 2\), \(\lambda = 1.54178\) Å, \(T = 223(2)\) K, 15949 reflections collected (\(\pm h, \pm k, \pm l\), \([\sin\theta/\lambda] = 0.60\) Å\(^{-1}\), 3784 independent (\(R_{int} = 0.048\)), and 3388 observed reflections \([I \geq 2\sigma(I)]\), 260 refined parameters, \(R = 0.044\), \(wR^2 = 0.123\), \(GoF = 1.039\).
**Synthesis of 5.** B(C₆F₅)₃ (0.400 g, 0.780 mmol) and 3c (0.300 g, 0.780 mmol) were suspended in pentane (15 ml) and stirred for 30 minutes at room temperature. The suspension was filtered and the residue washed twice with pentane (20 ml). Drying under vacuum gave the product 5 (0.534 g, 0.596 mmol, 76%) as a white solid. Crystals suitable for X-ray crystal structure analysis were grown by slow diffusion of pentane into a solution of 5 in dichloromethane at –36 °C. **Anal. Calc.** for C₄₅H₂₉BF₁₅P: C, 60.29; H, 3.26. Found: C, 60.37; H, 3.51. **IR (KBr):** ν̃/cm⁻¹ = 2926 (br m), 2360 (w), 1643 (m), 1515 (s), 1464 (s), 1092 (s), 980 (s), 823 (m), 648 (m), 564 (m). **Decomp. (DSC):** 189 °C.

**¹H NMR** (500 MHz, 298 K, C₆D₆): δ = 6.96 (d, 3J_HH = 8.1 Hz, 2H, o-Tol), 6.80 (d, 3J_HH = 8.1 Hz, 2H, m-Tol), 6.42 (d, 4J_PC = 5.2 Hz, 4H, m-Mes), 2.08 (s, 12H, o-CH₃Mes), 1.92 (s, 3H, Me), 1.87 (s, 6H, p-CH₃Mes).

**¹³C{¹H} NMR** (126 MHz, 298 K, C₆D₆): δ = 151.6 (br, B=C=), 148.7 (dm, 1J_FC ~ 240 Hz, o-C₆F₅), 144.0 (d, 4J_PC = 2.9 Hz, p-Mes), 142.9 (d, 2J_PC = 11.7 Hz, o-Mes), 141.2 (p-Tol), 139.7 (dm, 1J_FC ~ 250 Hz, p-C₆F₅), 137.4 (dm, 1J_FC ~ 245 Hz, m-C₆F₅), 136.7 (TolC=), 130.6 (d, 3J_PC = 13.4 Hz, m-Mes), 129.6 (m-Tol), 128.4 (d, 3J_PC = 5.3 Hz, o-Tol), 125.4 (d, 2J_PC = 3.7 Hz, i-Tol), 121.5 (br, i-C₆F₅), 121.4 (d, 1J_PC = 88.0 Hz, i-Mes), 22.8 (d, 3J_PC = 7.2 Hz, o-CH₃Mes), 21.1 (Me), 20.8 (p-CH₃Mes).

**¹⁹F NMR** (470 MHz, 298 K, C₆D₆): δ = -130.3 (m, 6F, o-BC₆F₅), -159.5 (t, 3J_FF = 20.7 Hz, 3F, p-BC₆F₅), -164.5 (m, 6F, m-BC₆F₅) [Δδ¹⁹F_m,p = 5.0].

**¹¹B{¹H} NMR** (160 MHz, 298 K, C₆D₆): δ = -16.5 (ν/2 ~ 50 Hz).

**³¹P{¹H} NMR** (121 MHz, 300 K, C₆D₆): δ = -137.8 (ν/2 ~ 10 Hz).
**TOCSY** (500 MHz, 298 K, C₆D₆): $\delta^{1}H_{\text{irr.}} / \delta^{1}H_{\text{res.}} = 6.96 / 6.80$ ($o$-Tol / $m$-Tol), 6.80 / 6.96, 1.92 ($m$-Tol / $o$-Tol, Me), 6.42 / 2.08, 1.87 ($m$-Mes / $o$-, $p$-CH₃Mes).

**NOE** (500 MHz, 298 K, C₆D₆): $\delta^{1}H_{\text{irr.}} / \delta^{1}H_{\text{res.}} = 6.96 / 6.80$, 2.08 ($o$-Tol / $m$-Tol, $o$-CH₃Mes), 6.80 / 6.96, 1.92 ($m$-Tol / $o$-Tol, Me), 6.42 / 2.08, 1.87 ($m$-Mes / $o$-, $p$-CH₃Mes).

**¹H,¹H GCOSY** (500 MHz / 500 MHz, 298 K, C₆D₆): $\delta^{1}H / \delta^{1}H = 6.96 / 6.80$ ($o$-Tol / $m$-Tol), 6.80 / 6.96, 1.92 ($m$-Tol / $o$-Tol, Me), 6.42 / 2.08, 1.87 ($m$-Mes / $o$-, $p$-CH₃Mes).

**¹H,¹³C GHSQC** (500 MHz / 126 MHz, 298 K, C₆D₆): $\delta^{1}H / \delta^{13}C = 6.96 / 128.4$ ($o$-Tol), 6.80 / 129.6 ($m$-Tol), 6.42 / 130.6 ($m$-Mes), 2.08 / 22.8 ($o$-CH₃Mes), 1.92 / 21.1 (Me), 1.87 / 20.8 ($p$-CH₃Mes).

**¹H,¹³C GHMBC** (500 MHz / 126 MHz, 298 K, C₆D₆): $\delta^{1}H / \delta^{13}C = 6.96 / 141.2, 136.7, 129.6$ ($o$-Tol / $p$-Tol, TolC=, $m$-Tol), 6.80 / 128.4, 125.4, 21.1 ($m$-Tol / $o$-Tol, i-Tol, Me), 6.42 / 142.9, 121.4, 22.8, 20.8 ($m$-Mes / $o$-, i-Mes, $o$-, $p$-CH₃Mes), 2.08 / 142.9, 130.6, 121.4 ($o$-CH₃Mes / $o$-, $m$-, i-Mes), 1.92 / 141.2, 129.6, 125.4 (Me / $p$-Tol, $m$-Tol, i-Tol), 1.87 / 144.0, 130.6, 121.4 ($p$-CH₃Mes / $p$-, $m$-, i-Mes).

**¹H NMR** (500 MHz, 298 K, C₆D₆) of **5**.
$^{13}$C\{\textsuperscript{1}H\} NMR (126 MHz, 298 K, C$_6$D$_6$) of 5.

$^{19}$F NMR (470 MHz, 298 K, C$_6$D$_6$) of 5.

$^{31}$P\{\textsuperscript{1}H\} NMR (121 MHz, 300 K, C$_6$D$_6$) and $^{11}$B\{\textsuperscript{1}H\} NMR (160 MHz) of 5.
X-Ray crystal structure analysis of 5. Crystal data for C₄₅H₂₉BF₁₅P * ½ CH₂Cl₂ * ½ C₅H₁₂,
M = 975.00, orthorhombic, Pca₂₁ (No. 29), a = 21.7497(6), b = 22.5390(4), c = 17.9477(6) Å,
V = 8798.3(4) Å³, D_c = 1.472 g cm⁻³, μ = 1.998 mm⁻¹, F(000) = 3968, Z = 8, λ = 1.54178 Å,
T = 223(2) K, 47800 reflections collected (±h, ±k, ±l), [(sinθ)/λ] = 0.60 Å⁻¹, 14469 independent (R_int = 0.074), and 12099 observed reflections [I ≥ 2σ(I)], 1206 refined parameters, R = 0.059, wR² = 0.157, GoF = 1.033, Flack parameter 0.50(2).
Synthesis of 4c. B(C₆F₅)₃ (0.332 g, 0.650 mmol) and 3c (0.250 g, 0.650 mmol) were dissolved in toluene (20 ml) and stirred for 6h at 105 °C. Subsequently the solvent was removed and the residue was washed twice with pentane (15 ml) and all volatiles were removed in vacuo to yield 4c (0.379 g, 0.423 mmol, 65%) as a white solid [admixed with 15% of A: see O. Ekkert, R. Fröhlich, G. Kehr, G. Erker, J. Am. Chem. Soc. 2011, 133, 4610-4616]. Crystals suitable for X-ray crystal structure analysis were grown by slow diffusion of pentane into a solution of 4c in dichloromethane at −36 °C. HRMS: Calc. for C₄₅H₂₉BF₁₅PAg: 1005.09161. Found: 1005.09180. IR (KBr): ν̃/cm⁻¹ = 2924 (br m), 2624 (w), 2398 (w), 2175 (w), 1643 (s), 1520 (br s), 1277 (s), 1095 (s), 982 (s), 694 (m), 506 (m). Decomp. (DSC): 227 °C. 

$^1$H NMR (500 MHz, 298 K, CD₂Cl₂): δ = 6.97 (m, 2H, m-Tol), 6.86 (m, 2H, o-Tol), 6.82 (d, $^4$Jₚₜ = 3.6 Hz, 4H, m-Mes), 2.27 (s, 9H, p-CH₃Mes / Me), 2.14 (s, 12H, o-CH₃Mes).

$^{13}$C{¹H} NMR (126 MHz, 298 K, CD₂Cl₂): δ = 146.9 (d, $^1$Jₚₚ = 48.0 Hz, =Cₚ), 143.7 (d, $^2$Jₚₚ = 9.1 Hz, o-Mes), 142.3 (d, $^4$Jₚₚ = 2.7 Hz, p-Mes), 139.3 (p-Tol), 134.9 (i-Tol), 131.0 (d, $^3$Jₚₚ = 9.3 Hz, m-Mes), 129.6 (m-Tol), 124.0 (d, $^3$Jₚₚ = 4.0 Hz, o-Mes), 123.2 (d, $^1$Jₚₚ = 36.0 Hz, i-Mes), 24.0 (d, $^3$Jₚₚ = 5.6 Hz, o-CH₃Mes), 21.4 (Me), 20.9 (p-CH₃Mes), n.o. ($^5$B=C=), [C₆F₅ not listed].

$^{19}$F{¹H} NMR (470 MHz, 298 K, CD₂Cl₂): δ = -127.3 (br., 4F, o), -158.0 (t, $^3$Jₚₚ = 20.5 Hz, 2F, p), -165.2 (m, 4F, m) (B(C₆F₅)₂) [Δδ$^{19}$Fₚₚ = 7.2], -136.7 (m, 2F, o), -156.3 (t, $^3$Jₚₚ = 21.0 Hz, 1F, p), -163.7 (m, 2F, m) (C₆F₅), [Δδ$^{19}$Fₚₚ = 7.4]; {A [key resonance]: -188.2 (br, B-F)}.

$^{11}$B{¹H} NMR (160 MHz, 298 K, CD₂Cl₂): δ = -0.1 (v₁/₂ ~ 350 Hz).

$^{31}$P{¹H} NMR (202 MHz, 298 K, CD₂Cl₂): δ = 14.6 (v₁/₂ ~ 50 Hz);

{A: δ = 27.3 (v₁/₂ ~ 60 Hz)}. 

TOCSY (500 MHz, 298 K, CD₂Cl₂): δ$^1$H$^{\text{irr.}}$/δ$^1$H$^{\text{res.}}$ = 6.97 / 6.86 (m-Tol / o-Tol), 6.86 / 6.97, 2.27 (o-Tol / m-Tol, Me), 6.82 / 2.27, 2.14 (m-Mes / p-, o-CH₃Mes).

NOE (500 MHz, 298 K, CD₂Cl₂): δ$^1$H$^{\text{irr.}}$/δ$^1$H$^{\text{res.}}$ = 6.97 / 6.86, 2.27 (m-Tol / o-Tol, Me), 2.27, 2.14 / 6.82 (p-CH₃Mes, o-CH₃Mes / m-Mes).

$^1$H,$^1$H GCOSY (500 MHz / 500 MHz, 298 K, CD₂Cl₂): δ$^1$H / δ$^1$H = 6.97 / 6.86 (m-Tol / o-Tol), 6.82 / 2.27, 2.14 (m-Mes / p-, o-CH₃Mes).
$^{1}H,^{13}C$ GHSQC (500 MHz / 126 MHz, 298 K, CD$_2$Cl$_2$): \( \delta^{1}H / \delta^{13}C = 6.97 / 129.6 \) (m-Tol), 6.86 / 127.3 (o-Tol), 6.82 / 131.0 (m-Mes), 2.27 / 21.4 (Me), 2.27 / 20.9 (p-CH$_3$Mes), 2.14 / 24.0 (o-CH$_3$Mes).

$^{1}H,^{13}C$ GHMBC (500 MHz / 126 MHz, 298 K, CD$_2$Cl$_2$): \( \delta^{1}H / \delta^{13}C = 6.97 / 134.9, 21.4 \) (m-Tol / i-Tol, Me), 6.86 / 146.9, 139.3 (o-Tol / =C, i-Tol), 6.82 / 143.7, 123.2, 24.0, 20.9 (m-Mes / o-, i-Mes, o-, Me), 2.27 / 142.3, 139.3, 131.0, 129.6 (Me, p-CH$_3$Mes / p-Mes, p-Tol, m-Mes, m-Tol), 2.14 / 143.7, 131.0, 123.2 (o-CH$_3$Mes / o-, m-, i-Mes).

$^1$H NMR (500 MHz, 298 K, CD$_2$Cl$_2$) of 4c.

$^{13}$C\textsubscript{$^1$H} NMR (126 MHz, 298 K, CD$_2$Cl$_2$) of 4c.
\[^1\text{H}^\text{19F}\] NMR (470 MHz, 298 K, CD\textsubscript{2}Cl\textsubscript{2}) of \textit{4c}.

\[^1\text{H}^\text{11B}\] NMR (160 MHz, 298 K, CD\textsubscript{2}Cl\textsubscript{2}) and \[^1\text{H}^\text{31P}\] NMR (202 MHz) of \textit{4c}.

**X-Ray crystal structure analysis of 4.** Crystal data for C\textsubscript{45}H\textsubscript{29}BF\textsubscript{15}P, \(M = 896.46\), triclinic, \(P\bar{T}\) (No. 2), \(a = 12.4342(5)\), \(b = 13.4995\) (6), \(c = 14.2303(6)\) Å, \(\alpha = 82.805(2)\), \(\beta = 66.678(2)\), \(\gamma = 64.037(2)\)°, \(V = 1968.75(14)\) Å\(^3\), \(D_c = 1.512\) g cm\(^{-3}\), \(\mu = 1.570\) mm\(^{-1}\), \(F(000) = 908\), \(Z = 2\), \(\lambda = 1.54178\) Å, \(T = 223(2)\) K, 28030 reflections collected (±\(h\), ±\(k\), ±\(l\)), [(sin\(\theta\)/\(\lambda\)] = 0.60 Å\(^{-1}\), 6739 independent (\(R_{int} = 0.038\)), and 6261 observed reflections [\(I \geq 2\sigma(I)\)], 566 refined parameters, \(R = 0.037\), w\(R^2 = 0.109\), GoF = 1.034.
NMR-scale reactions:

a) Heating of 5 (31.0 mg, 0.035 mmol) in d₈-toluene (1 ml) for 6h at 105 °C yielded a mixture of 4c and A in a 1:0.1 ratio (monitored by ³¹P NMR).

NMR spectra before heating

![NMR spectra](image)

¹H NMR (300 MHz, 298 K, D₈-toluene) and ¹¹B{¹H} NMR (96 MHz): before heating of 5

![NMR spectra](image)

¹⁹F NMR (282 MHz, 298 K, D₈-toluene): before heating of 5

![NMR spectra](image)

³¹P NMR (122 MHz, 298 K, D₈-toluene): before heating of 5
NMR spectra after heating at 105°C for 6h

\[ ^1H \text{ NMR} (300 \text{ MHz}, 298 \text{ K}, \text{D}_8\text{-toluene}) \text{ and } ^{11}B\{^1H\} \text{ NMR} (96 \text{ MHz}): \]

after heating of 5 for 6h

\[ ^19F \text{ NMR} (282 \text{ MHz}, 298 \text{ K}, \text{D}_8\text{-toluene}): \text{after heating of 5 for 6h} \]

\[ ^{31}P\{^1H\} \text{ NMR} (122 \text{ MHz}, 298 \text{ K}, \text{D}_8\text{-toluene}): \text{after heating of 5 for 6h} \]
b) Heating of 5 (43.0 mg, 0.048 mmol) and 3a (17.9 mg, 0.062 mmol) in d₈-toluene (1 ml) for 48h at 70 °C resulted in a reaction mixture of 4c, 4a, 3c and 3a in a ca. 5.8 : 1 : 1 : 3.4 ratio [traces of 5 and A] (monitored by ³¹P NMR).

*NMR spectra before heating*

- **¹H NMR (300 MHz, 298 K, D₈-toluene) and ¹¹B NMR (96 MHz):** before heating the reaction mixture of 5 and 3a

- **¹⁹F NMR (282 MHz, 298 K, D₈-toluene):** before heating the reaction mixture of 5 and 3a

- **³¹P NMR (122 MHz, 298 K, D₈-toluene):** before heating the reaction mixture of 5 and 3a
NMR spectra after heating at 70°C for 48h

$^1$H NMR (300 MHz, 298 K, D$_8$-toluene): heating the reaction mixture of 5 and 3a (70°C, 48h)

$^{19}$F NMR (282 MHz, 298 K, D$_8$-toluene): heating the reaction mixture of 5 and 3a (70°C, 48h)

$^{11}$B NMR (96 MHz, 298 K, D$_8$-toluene): heating the reaction mixture of 5 and 3a (70°C, 48h)

$^{31}$P NMR (122 MHz, 298 K, D$_8$-toluene): heating the reaction mixture of 5 and 3a (70°C, 48h)