Electronic Supplementary Material (ESI) for Dalton Transactions.

Formation, Structural Characterization, and Reactions of a Unique Cyclotrimeric Vicinal Lewis Pair Containing $(C_6F_5)_2$P-Lewis Base and (C_6F_5)BH-Lewis Acid Components

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Experimental work carried out by the Eckert group:

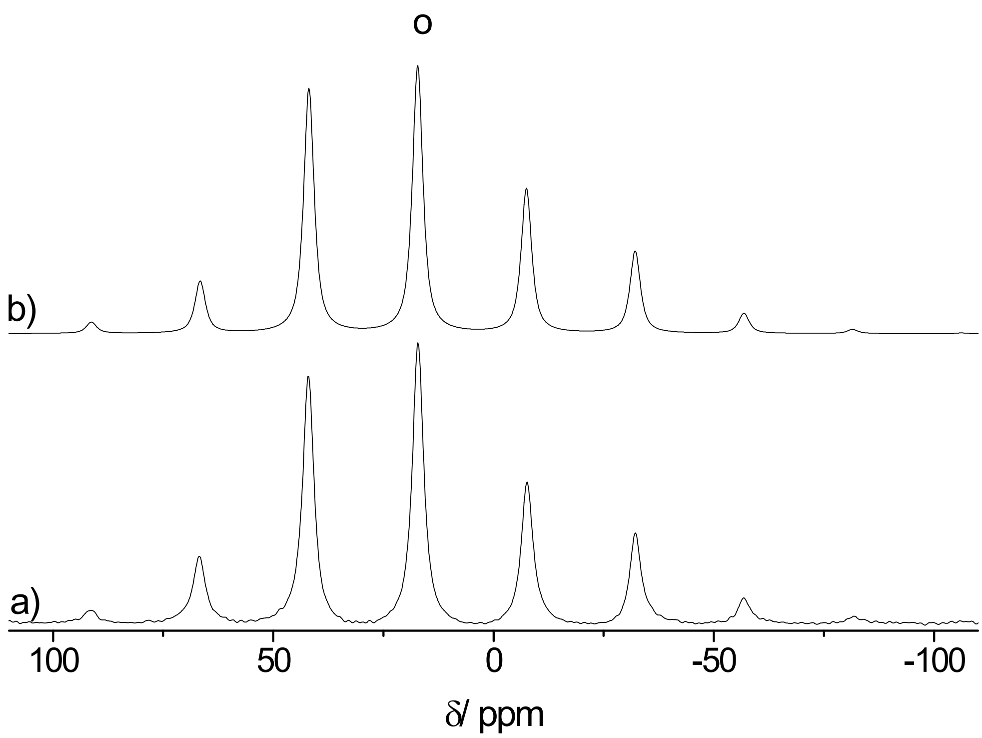


Figure S1: $^{31}P\{^{1}H\}$ CPMAS NMR spectrum of (**5**)$_3$ acquired at 7.05 T with a spinning frequency of 3.0 kHz (a) and corresponding line shape simulation based on the parameters listed in Table 1.

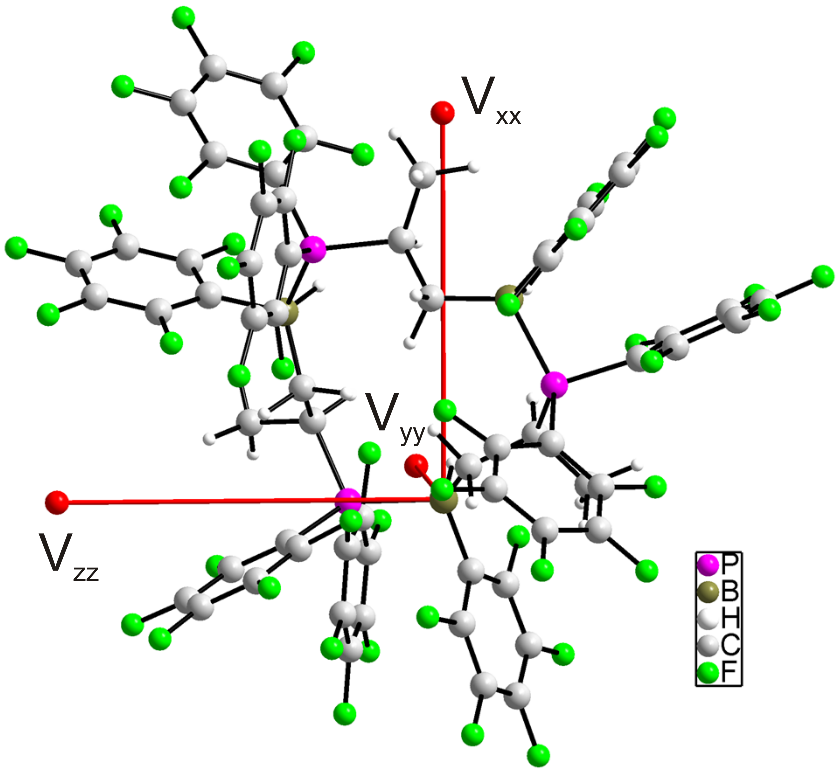


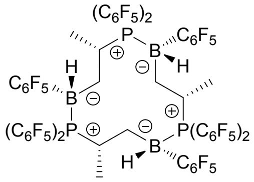
Figure S2: Orientation of the calculated [11]B electric field gradient tensor in the molecular geometry for compound **(5)₃**. The principal component of the tensor (V_{zz}) points nearly parallel to the B-P bond (the angle V_{zz}-B-P is determined to 14.5°).

Experimental work carried out by the Erker group:

General Information

All reactions were carried out under argon atmosphere with Schlenk-type glassware. Solvents were dried and stored in an argon atmospere. Benzaldehyde, pyridine and *tert*-butylacetylene were dried over molecular sieves (4 Å). Bis(pentafluorophenyl)-borane was prepared according to a modified literature procedure ((a) D. J. Parks, R. E. v. H. Spence, W. E. Piers, *Angew. Chem. Int. Ed. Engl.* 1995, **34**, 809-811; (b) W. E. Piers, T. Chivers, *Chem. Soc. Rev.* 1997, **26**, 345-354; (c) D. J. Parks, W. E. Piers, G. P. A. Yap, *Organometallics* 1998, **17**, 5492-5503). $(C_6F_5)_2P\text{-}C(CH_3){=}CH_2$ was synthesized by Dr. Annika Stute (A. Stute, L. Heletta, R. Fröhlich, C. G. Daniliuc, G. Kehr and G. Erker, *Chem. Commun.* 2012, **48**, 11739-11741). The following instruments were used for physical characterization of the compounds. Elemental analyses: Foss-Heraeus CHN-O-Rapid and Vario EL III CHNS instrument. Melting Point: Differential Scanning Calorimeter 2010 from TA-instruments DSC Q-20. IR: Varian 3100 FT-IR. NMR: Varian Inova 500 (^{1}H, 500 MHz; ^{13}C, 126 MHz, ^{31}P, 202 MHz, ^{10}B, 54 MHz, ^{19}F, 470 MHz), Varian UnityPlus 600 (^{1}H, 600 MHz; ^{13}C, 151 MHz, ^{31}P, 242 MHz, ^{10}B, 64 MHz, ^{19}F, 564 MHz). Assignments of the resonances were supported by 2D experiments. **X-Ray diffraction:** 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 Enzymol.* **1997**, *276*, 307-326); absorption correction, Denzo (Z. Otwinowski, D. Borek, W. Majewski, W. Minor, *Acta Crystallogr.* **2003**, *A59*, 228-234); structure solution SHELXS-97 (G. M. Sheldrick, *Acta Crystallogr.* **1990**, *A46*, 467-473); structure refinement SHELXL-97 (G. M. Sheldrick, *Acta Crystallogr.* **2008**, *A64*, 112-122) and graphics, XP (BrukerAXS, 2000). Thermal ellipsoids are shown with 15% probability, *R*-values are given for observed reflections, and wR^2 values are given for all reflections. CCDC deposition numbers are 1001587 to 1001589.

Preparation of Compound (5)$_3$



$(C_6F_5)_2P\text{-}C(CH_3)=CH_2$ (80.0 mg, 0.20 mmol, 1.0 eq) and bis(pentafluorophenyl)borane (68.1 mg, 0.20 mmol, 1.0 eq) were dissolved in toluene (10 mL). After stirring for 15 min 9-BBN (24.0 mg, 0.20 mmol, 1.0 eq) was added. The solution was stirred overnight and the formed colourless precipitate was isolated and dried *in vacuo* (53.2 mg, 45%). Crystals suitable for the X-ray single crystal structure analysis were obtained simultaneously with the isolated colourless precipitate.

IR (KBr): $\tilde{\upsilon}$ = 2951 (m), 2353 (m), 1644 (s), 1521 (s), 1468 (s) 1389 (s) 1301 (s), 1269 (s), 1235 (m), 1188 (m), 1095 (s), 1027 (s), 965 (s), 937 (s), 890 (m), 850 (m), 797 (m), 767 (m), 721 (m), 691 (w), 633 (m), 590 (w), 553 (w), 530 (m), 486 (m), 454 (m), 422 (w).

Melting point: 192 °C.

Elemental analysis: calc. for $C_{63}H_{21}B_3F_{45}P_3$ (1758.12 g/mol): C, 43.04; H, 1.20.

Found: C, 42.99; H, 1.39.

X-ray crystal structure analysis of compound 5: formula $C_{63}H_{21}B_3F_{45}P_3$, M = 1758.14, colourless crystal, 0.40 x 0.15 x 0.10 mm, a = 25.0360(4), b = 25.0360(4), c = 35.7640(10) Å, α = 90, β = 90, γ = 120 °, V = 19413.6(7) Å^3, ρ_{calc} = 1.805 gcm^{-3}, μ = 2.463 mm^{-1}, empirical absorption correction (0.439 ≤ T ≤ 0.790), Z = 12, trigonal, space group R-3c (No. 167), λ = 1.54178 Å, T = 223(2) K, ω and φ scans, 59983 reflections collected (±h, ±k, ±l), [(sinθ)/λ] = 0.60 Å^{-1}, 3823 independent (R_{int} = 0.058) and 3189 observed reflections [I>2σ(I)], 348 refined parameters, R = 0.042, wR^2 = 0.115, max. (min.) residual electron density 0.31 (-0.23) e.Å^{-3}, the hydrogen atom at B1 was refined freely; others were calculated and refined as riding atoms.

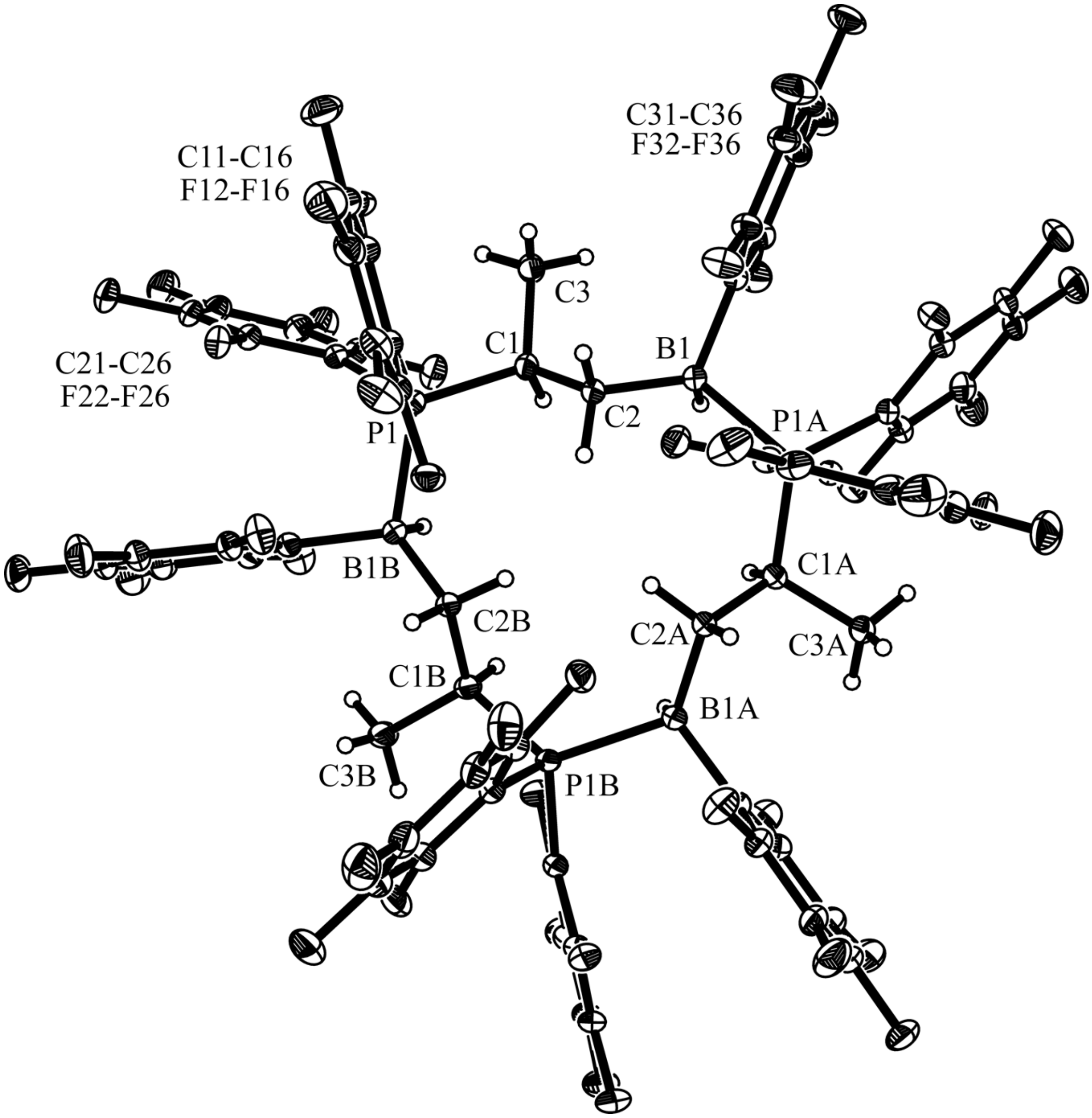
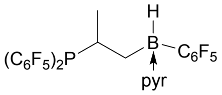


figure 1: X-ray crystal structure of compound **5**.

Preparation of Compound 6



Pyridine (7.0 µL, 0.09 mmol, 3.0 eq) was added to a suspension of compound $(\mathbf{5})_3$ (50.0 mg, 0.03 mmol, 1.0 eq) in dichloromethane (10 mL) and stirred overnight. Then all volatiles were removed *in vacuo* and the remaining solid was dissolved in pentane (ca. 5 mL) and the formed suspension stored at -34 °C. After two days the formed colourless precipitate was isolated and dried *in vacuo* (24.1 mg, 43%). In the CD_2Cl_2 solution of the obtained solid two diastereomers $\mathbf{6_{major}}$ and $\mathbf{6_{minor}}$ were observed (major/minor ~ 2/1). Crystals suitable for the X-ray single crystal structure analysis were obtained by slow crystallisation from a dichloromethane solution of compound **6**.

IR (KBr): υ = 2968 (w), 2890 (w), 2419 (w), 1641 (m), 1517 (s), 1474 (s), 1379 (m), 1284 (m), 1216 (w), 1087 (s), 1023 (m), 976 (s), 895 (w), 871 (w), 831 (w), 762 (m), 731 (m), 691 (s), 637 (w), 511 (m), 421 (m).

Melting point: no minimum observed.

Elemental analysis: calc. for $C_{26}H_{12}BF_{15}NP$ (665.15 g/mol): C, 46.95; H, 1.82; N, 2.11. Found: C, 47.39; H, 1.93; N, 2.19.

Diastereomer $\mathbf{6_{major}}$:

^{1}H NMR (500 MHz, CD_2Cl_2, 299 K): δ = 8.55 (m, 2H, *o*-Py), 8.05 (m, 1H, *p*-Py), 7.60 (m, 2H, *m*-Py), 3.71 (br d, $^{1}J_{BH}$ ~ 90 Hz, 1H, BH), 3.00 (m, 1H, CH), 1.20 (dd, $^{4}J_{PH}$ = 21.2 Hz, $^{3}J_{HH}$ = 6.7 Hz, 3H, CH_3), 1.08, 0.81 (each m, each 1H, CH_2B).

^{13}C{^{1}H} NMR (126 MHz, CD_2Cl_2, 299 K): δ = 147.0 (*o*-Py), 141.4 (*p*-Py), 126.3 (*m*-Py), 25.9 (m, CH), 25.6 (br, CH_2B), 18.5 (d, $^{2}J_{PC}$ = 26.1 Hz, CH_3), [C_6F_5 not listed].

^{31}P NMR (202 MHz, CD_2Cl_2, 299 K): δ = -37.7 ($\nu_{1/2}$ ~ 90 Hz).

^{31}P{^{1}H} NMR (202 MHz, CD_2Cl_2, 299 K): δ = -37.7 (quin, $^{3}J_{PF}$ = 30.6 Hz).

^{10}B NMR (54 MHz, CD_2Cl_2, 299 K): δ = -5.1 ($\nu_{1/2}$ ~ 150 Hz).

^{10}B{^{1}H} NMR (54 MHz, CD_2Cl_2, 299 K): δ = -5.1 ($\nu_{1/2}$ ~ 150 Hz).

^{19}F NMR (564 MHz, CD_2Cl_2, 299 K): δ = -129.4 (m, 2F, *o*-$C_6F_5^P$), -151.5 (tm, $^{3}J_{FF}$ = 20.4 Hz, 1F, *p*-$C_6F_5^P$), -162.1 (m, 2F, *m*-$C_6F_5^P$), [$\Delta\delta^{19}F_{pm}^{P}$ = 10.6]; -130.0 (m, 2F, *o*-$C_6F_5^P$), -152.1 (tm, $^{3}J_{FF}$ = 20.4 Hz, 1F, *p*-$C_6F_5^P$), -161.9 (m, 2F, *m*-$C_6F_5^P$), [$\Delta\delta^{19}F_{pm}^{P}$ = 9.8]; -134.4 (m, 2F, *o*-$C_6F_5^B$), -159.5 (t, $^{3}J_{FF}$ = 19.9 Hz, 1F, *p*-$C_6F_5^B$), -165.0 (m, 2F, *m*-$C_6F_5^B$), [$\Delta\delta^{19}F_{pm}^{B}$ = 5.5].

^{19}F, ^{19}F GCOSY (564 MHz/564 MHz, CD_2Cl_2, 299 K)[selected traces]**:** $\delta^{19}F/\delta^{19}F$ = -161.9/-152.1, -130.0 (*m*-$C_6F_5^P$/*o*-$C_6F_5^P$, *p*-$C_6F_5^P$), -162.1/-129.4, -151.5 (*m*-$C_6F_5^P$/*o*-$C_6F_5^P$, *p*-$C_6F_5^P$), -165.0/-134.4, -159.9 (*m*-$C_6F_5^B$/*o*-$C_6F_5^B$, *p*-$C_6F_5^B$).

^{1}H, ^{1}H GCOSY (500 MHz/500 MHz, CD_2Cl_2, 299 K)[selected trace]: δ^1H/ δ^1H = 7.60/8.55, 8.05 (*m*-Py/ *o*-Py, *p*-Py).

^{1}H{^{1}H} TOCSY (600MHz, CD_2Cl_2, 299 K)[selected experiment]: δ^1H_{irr}/ δ^1H_{res} = 0.81/3.70, 3.01, 1.21, 1.08, 0.81 (CH_2B/BH, CH, CH_3, CH_2B, CH_2B).

^{1}H, ^{13}C GHSQC (500 MHz/126 MHz, CD_2Cl_2, 299 K): δ^1H/ δ^{13}C = 8.55/147.0 (*o*-Py), 8.05/141.4 (*p*-Py), 7.60/126.3 (*m*-Py), 3.00/25.9 (CH), 1.20/18.5 (CH_3), 0.81/25.6 (CH_2B).

Diastereomer 6_{minor}:

^{1}H NMR (500 MHz, CD_2Cl_2, 299 K): δ = 8.55 (m, 2H, *o*-Py), 8.11 (m, 1H, *p*-Py), 7.64 (m, 2H, *m*-Py), 3.67 (br, 1H, BH), 2.93 (m, 1H, CH), 1.16 (dd, $^4J_{PH}$ = 21.2 Hz, $^3J_{HH}$ = 6.6 Hz, 3H, CH_3), 1.06, 1.00 (each m, each 1H, CH_2B).

^{13}C{^{1}H} NMR (126 MHz, CD_2Cl_2, 299 K): δ = 147.1 (*o*-Py), 141.6 (*p*-Py), 126.5 (*m*-Py), 26.8 (br, CH_2B), 25.9 (m, CH), 18.2 (d, $^2J_{PC}$ = 25.3 Hz, CH_3), [C_6F_5 not listed].

^{31}P NMR (202 MHz, CD_2Cl_2, 299 K): δ = -36.9 ($\nu_{1/2}$ ~ 70 Hz).

^{31}P{^{1}H} NMR (202 MHz, CD_2Cl_2, 299 K): δ = -36.9 (quin, $^3J_{PF}$ = 28.4 Hz).

^{10}B NMR (54 MHz, CD_2Cl_2, 299 K): δ = -5.1 ($\nu_{1/2}$ ~ 150 Hz).

^{10}B{^{1}H} NMR (54 MHz, CD_2Cl_2, 299 K): δ = -5.1 ($\nu_{1/2}$ ~ 150 Hz).

^{19}F NMR (564 MHz, CD_2Cl_2, 299 K): δ = -129.7 (m, 2F, *o*-$C_6F_5^P$), -151.6 (tm, $^3J_{FF}$ = 20.6 Hz, 1F, *p*-$C_6F_5^P$), -161.76 (m, 2F, *m*-$C_6F_5^P$), [$\Delta\delta^{19}F_{pm}^P$ = 10.2]; -129.8 (m, 2F, *o*-$C_6F_5^P$), -151.8 (tm, $^3J_{FF}$ = 20.3 Hz, 1F, *p*-$C_6F_5^P$), -161.80 (m, 2F, *m*-$C_6F_5^P$), [$\Delta\delta^{19}F_{pm}^P$ = 10.1]; -133.3 (m, 2F, *o*-$C_6F_5^B$), -159.7 (t, $^3J_{FF}$ = 20.1 Hz, 1F, *p*-$C_6F_5^B$), -165.0 (m, 2F, *m*-$C_6F_5^B$), [$\Delta\delta^{19}F_{pm}^B$ = 5.3].

^{19}F, ^{19}F GCOSY (564 MHz/564 MHz, CD_2Cl_2, 299 K)[selected traces]: δ^{19}F/ δ^{19}F = -161.76/-129.7, -151.6 (*m*-$C_6F_5^P$/*o*-$C_6F_5^P$, *p*-$C_6F_5^P$), -161.80/-129.8, -151.8 (*m*-$C_6F_5^P$/*o*-$C_6F_5^P$, *p*-$C_6F_5^P$), -165.0/-133.3, -159.7 (*m*-$C_6F_5^B$/*o*-$C_6F_5^B$, *p*-$C_6F_5^B$).

^{1}H, ^{1}H GCOSY (500 MHz/500 MHz, CD_2Cl_2, 299 K)[selected trace]**:** δ^1H/ δ^1H = 7.64/8.55, 8.11 (*m*-Py/ *o*-Py, *p*-Py).

^{1}H{^{1}H} TOCSY (600MHz, CD_2Cl_2, 299 K)[selected experiment]: δ^1H_{irr}/ δ^1H_{res} = 1.00/3.67, 2.93, 1.16, 1.06, 1.00 (CH_2B/BH, CH, CH_3, CH_2B, CH_2B).

^{1}H, ^{13}C GHSQC (500 MHz/126 MHz, CD_2Cl_2, 299 K): δ^1H/ δ^{13}C = 8.55/147.1 (*o*-Py), 8.11/141.6 (*p*-Py), 7.64/126.5 (*m*-Py), 2.93/25.9 (CH), 1.16/18.2 (CH_3), 1.00/26.8 (CH_2B).

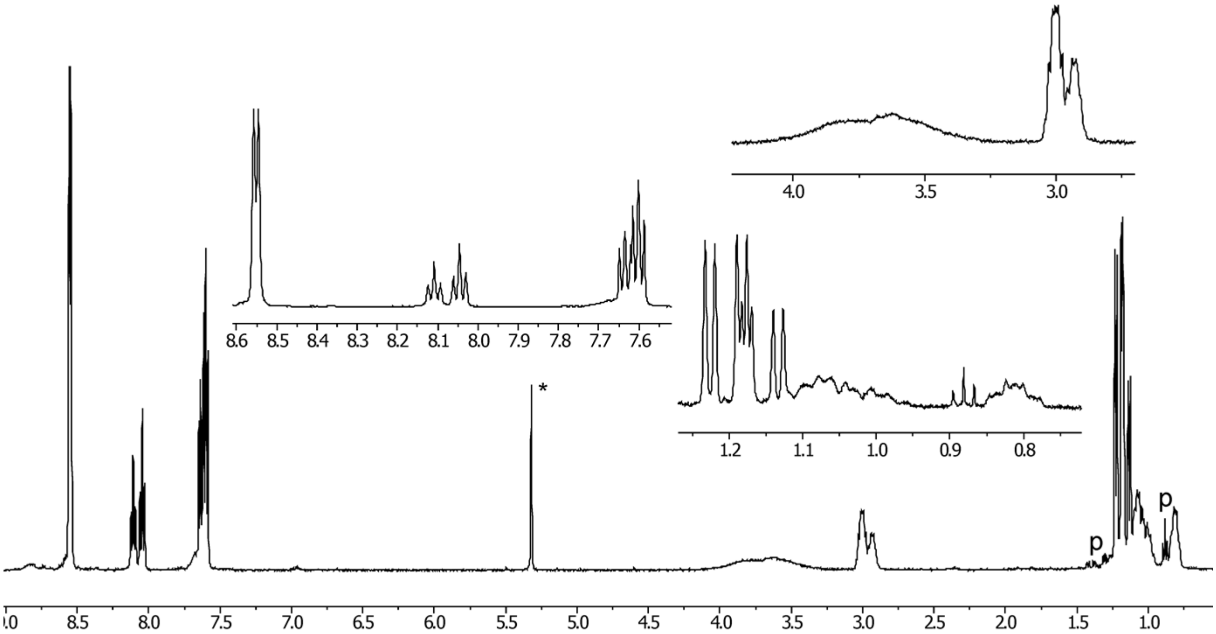


figure 2: ^{1}H NMR (500 MHz, CD_2Cl_2, 299 K, p = pentane).

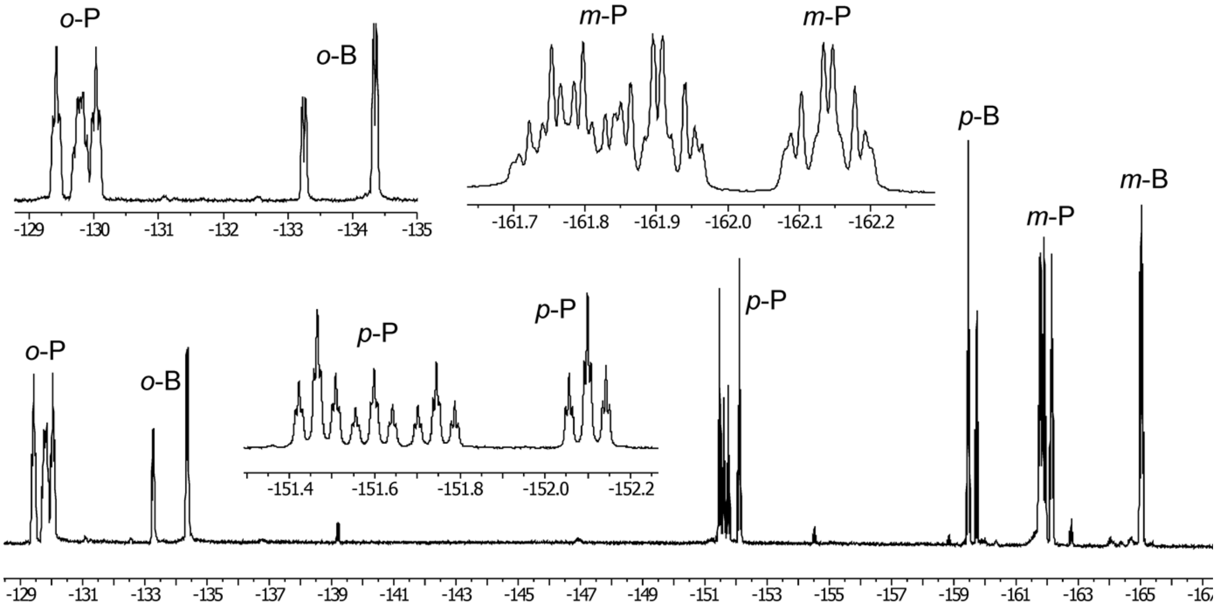


figure 3: ^{19}F NMR (470 MHz, CD_2Cl_2, 299 K).

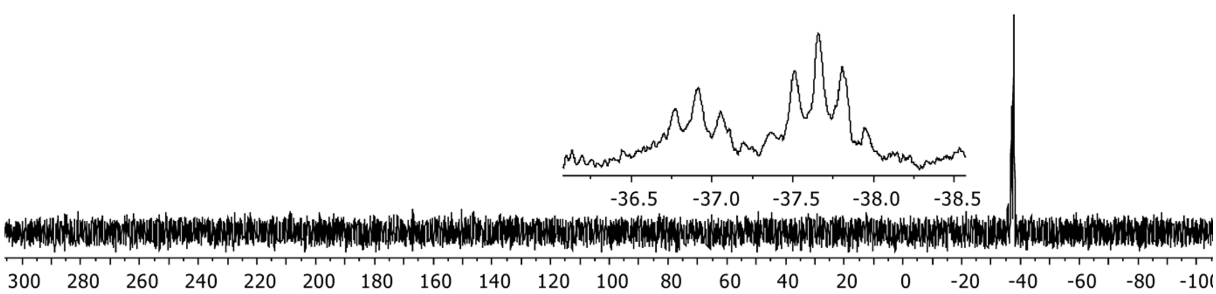


figure 4: $^{31}P\{^{1}H\}$NMR (202 MHz, CD_2Cl_2, 299 K).

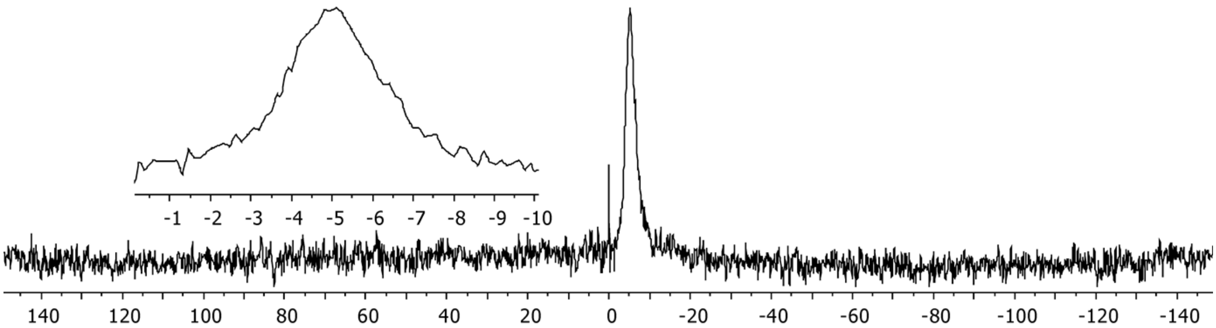


figure 5: $^{10}B\{^{1}H\}$ NMR (54 MHz, CD_2Cl_2, 299 K).

X-ray crystal structure analysis of compound 6: formula $C_{26}H_{12}BF_{15}NP$, M = 665.15, colourless crystal, 0.18 x 0.11 x 0.05 mm, a = 10.1327(3), b = 18.2392(7), c = 14.6954(5) Å, β = 98.180(2)°, V = 2688.3(2) Å^3, ρ_{calc} = 1.643 gcm^{-3}, μ = 2.070 mm^{-1}, empirical absorption correction (0.707 ≤ T ≤ 0.903), Z = 4, monoclinic, space group $P2_1/c$ (No. 14), λ = 1.54178 Å, T = 223(2) K, ω and φ scans, 22071 reflections collected (±h, ±k, ±l), [(sinθ)/λ] = 0.60 Å^{-1}, 4724 independent (R_{int} = 0.049) and 3795 observed reflections [I>2σ(I)], 402 refined parameters, R = 0.043, wR^2 = 0.116, max. (min.) residual electron density 0.18 (-0.18) e.Å^{-3}, the hydrogen atom at B1 was refined freely; others were calculated and refined as riding atoms.

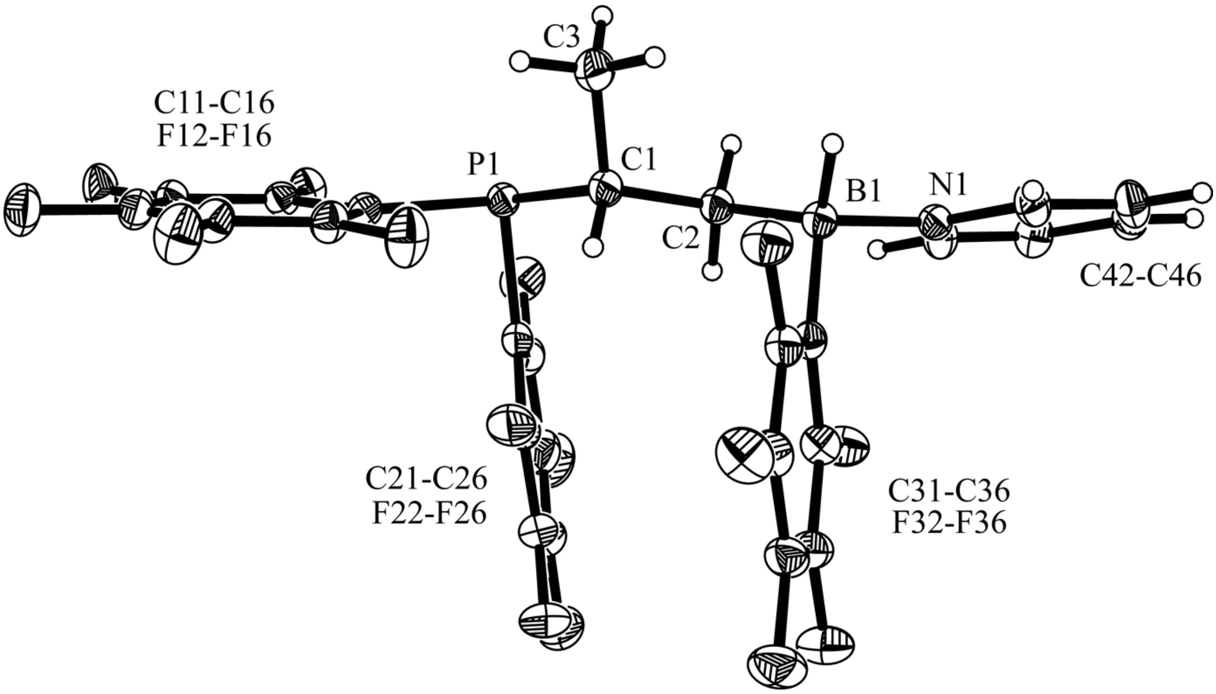
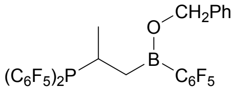


figure 6: X-ray crystal structure of compound **6**.

Preparation of Compound 7



Benzaldehyde (8.6 µL, 0.09 mmol, 3.0 eq) was added to a suspension of compound (**5**)$_3$ (50.0 mg, 0.03 mmol, 1.0 eq) in CD_2Cl_2 (0.8 mL) and reacted overnight. Then all volatiles were removed and the obtained solid was dried *in vacuo* to give compound **7** (47.1 mg, 76%).

IR (ATR): υ = 1642 (w), 1515 (m), 1467 (s), 1414 (w), 1394 (w), 1379 (w), 1341 (w), 1327 (w), 1303 (w), 1231 (w), 1090 (m), 1080 (m), 971 (s), 827 (w), 817 (w), 757 (w), 700 (m).
Melting point: 89 °C.
Elemental analysis: calc. for $C_{28}H_{13}BF_{15}OP$ (692.17 g/mol): C, 48.59; H, 1.89.
Found: C, 48.63; H, 1.82.
^{1}H NMR (500 MHz, CD_2Cl_2, 299 K): δ = 7.38 (m, 2H, *m*-Ph), 7.32 (m, 1H, *p*-Ph), 7.31 (m, 2H, *o*-Ph), 5.12, 5.11 (each d, $^2J_{HH}$ = 12.7 Hz, each 1H, CH_2O), 3.62 (m, 1H, CH), 1.39 (m, 2H, CH_2B), 1.17 (dd, $^4J_{PH}$ = 20.1 Hz, $^3J_{HH}$ = 6.9 Hz, 3H, CH_3).
^{13}C{^{1}H} NMR (126 MHz, CD_2Cl_2, 299 K): δ = 137.9 (*i*-Ph), 129.0 (*m*-Ph), 128.4 (*p*-Ph), 127.1 (*o*-Ph), 71.4 (CH_2O), 27.9 (br, CH_2B), 23.6 (m, CH), 19.6 (d, $^2J_{PC}$ = 24.0 Hz, CH_3), [C_6F_5 not listed].
^{31}P NMR (202 MHz, CD_2Cl_2, 299 K): δ = -35.5 ($\nu_{1/2}$ ~ 100 Hz).
^{31}P{^{1}H} NMR (202 MHz, CD_2Cl_2, 299 K): δ = -35.5 (quin, $^3J_{PF}$ =30.4 Hz).
^{10}B NMR (54 MHz, CD_2Cl_2, 299 K): δ = 48.0 ($\nu_{1/2}$ ~ 450 Hz).
^{10}B{^{1}H} NMR (54 MHz, CD_2Cl_2, 299 K): δ = 48.0 ($\nu_{1/2}$ ~ 450 Hz).
^{19}F NMR (470 MHz, CD_2Cl_2, 299 K): δ = -129.9 (m, 2F, *o*-$C_6F_5^P$), -150.7 (tm, $^3J_{FF}$ = 20.4 Hz, 1F, *p*-$C_6F_5^P$), -161.2 (m, 2F, *m*-$C_6F_5^P$), [$\Delta\delta^{19}F_{pm}^P$ = 10.5]; -129.8 (m, 2F, *o*-$C_6F_5^P$), -150.6 (br t, $^3J_{FF}$ = 19.1 Hz, 1F, *p*-$C_6F_5^P$), -161.2 (m, 2F, *m*-$C_6F_5^P$), [$\Delta\delta^{19}F_{pm}^P$ = 10.6]; -133.3 m, 2F, *o*-$C_6F_5^B$), -152.5 (tm, $^3J_{FF}$ = 19.6 Hz, 1F, *p*-$C_6F_5^B$), -161.5 (m, 2F, *m*-$C_6F_5^B$), [$\Delta\delta^{19}F_{pm}^B$ = 9.0].
^{19}F, ^{19}F GCOSY (470 MHz/470 MHz, CD_2Cl_2, 299 K) [selected traces]: δ^{19}F/ δ^{19}F = -161.2/129.9, -150.7 (*m*-$C_6F_5^P$/*o*-$C_6F_5^P$, *p*-$C_6F_5^P$), -161.2/-129.8, -150.6, (*m*-$C_6F_5^P$/*o*-$C_6F_5^P$, *p*-$C_6F_5^P$), -161.5/-133.3, -152.5 (*m*-$C_6F_5^B$/*o*-$C_6F_5^B$, *p*-$C_6F_5^B$).
^{1}H, ^{1}H GCOSY (500 MHz/500 MHz, CD_2Cl_2, 299 K)[selected traces]: δ^{1}H/ δ^{1}H = 7.38/7.31, 5.12 (*m*-Ph/*o*-Ph, CH_2O), 3.62/1.39, 1.17 (CH/CH_2B, CH_3).
^{1}H, ^{13}C GHSQC (500 MHz/126 MHz, CD_2Cl_2, 299 K): δ^{1}H/ δ^{13}C = 7.38/129.0 (*m*-Ph), 7.32/128.4 (*p*-Ph), 7.31/127.1 (*o*-Ph), 5.12, 5.11/71.4 (CH_2O), 3.62/23.6 (CH), 1.39/27.9 (CH_2B), 1.17/19.6 (CH_3).

^{1}H, ^{13}C GHMBC (500 MHz/126 MHz, CD_2Cl_2, 299 K) δ^1H/ δ^{13}C = 7.38/137.9, 129.0, 127.1 (*m*-Ph/*i*-Ph, *m*-Ph, *o*-Ph), 7.31/128.4, 127.1, 71.4 (*o*-Ph/*p*-Ph, *o*-Ph, CH_2O).

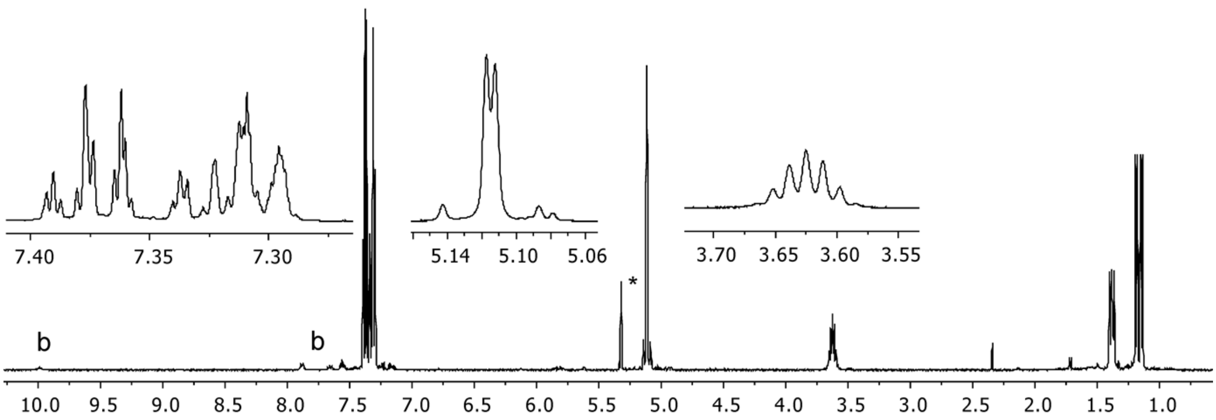


figure 7: ^{1}H NMR (500 MHz, CD_2Cl_2, 299 K), b: benzaldehyde.

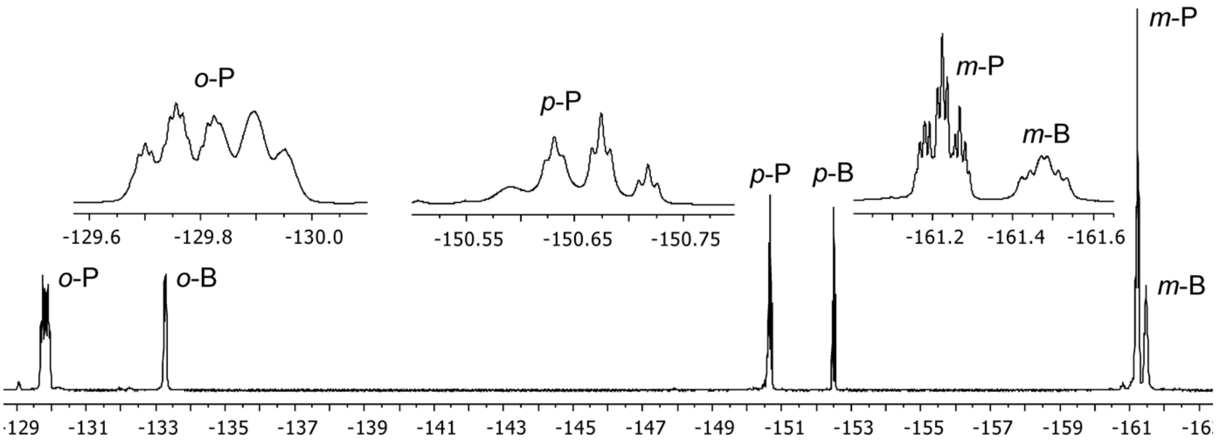


figure 8: ^{19}F NMR (470 MHz, CD_2Cl_2, 299 K).

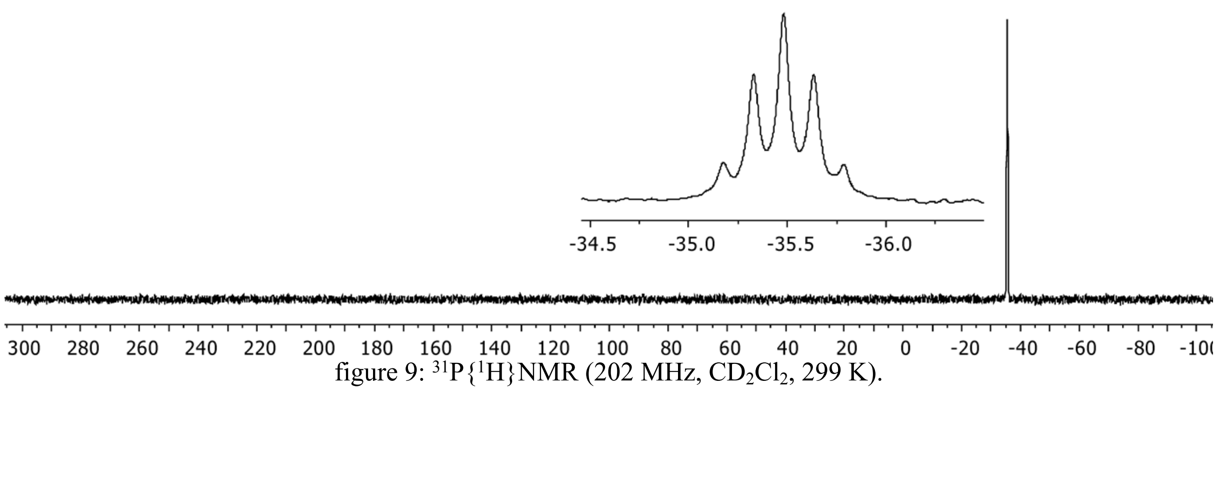


figure 9: $^{31}P\{^1H\}$NMR (202 MHz, CD_2Cl_2, 299 K).

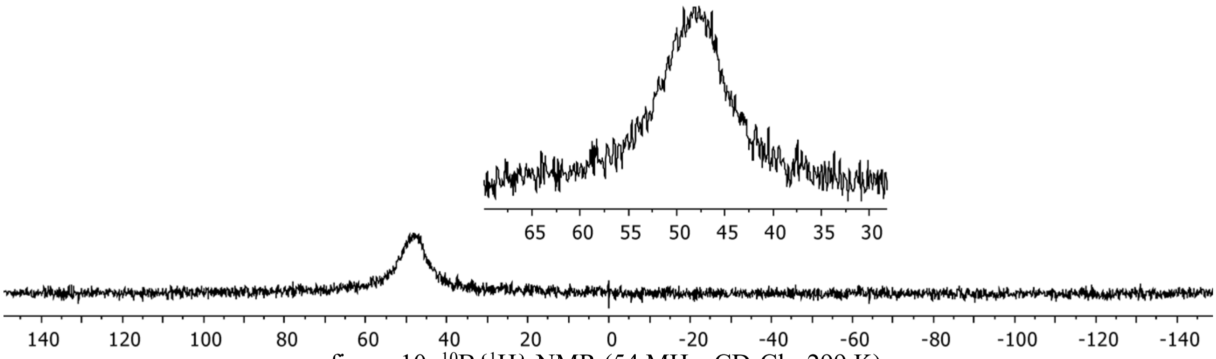
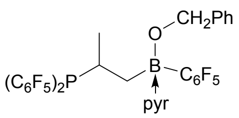


figure 10: $^{10}B\{^{1}H\}$ NMR (54 MHz, CD_2Cl_2, 299 K).

Preparation of compound 8



Benzaldehyde (8.6 µL, 0.09 mmol, 3.0 eq) was added to a suspension of compound (**5**)$_3$ (50.0 mg, 0.03 mmol, 1.0 eq) in dichloromethane (10 mL) and stirred overnight. Then pyridine (7.0 µL, 0.09 mmol, 3.0 eq) was added and the reaction mixture was stirred overnight again. All volatiles were removed *in vacuo* and the remaining residue was dissolved in pentane (ca. 5 mL) and stored at -34 °C. After two days the formed oil was isolated and dried *in vacuo* (55.4 mg, 84%).

IR (KBr): υ = 3442 (w), 3430 (w), 3032 (w), 2960 (w), 2922 (w), 2878 (w), 1641 (m), 1518 (s), 1474 (s), 1380 (m), 1352 (m), 1285 (m), 1211 (m), 1138 (m), 1087 (s), 1025 (m), 976 (s), 889 (w), 833 (m), 731 (m), 695 (m), 635 (w), 595 (w), 546 (w), 511 (m), 420 (m).
Elemental analysis: calc. for $C_3H_{18}BF_{15}NOP$ (771.27 g/mol): C, 51.39; H, 2.35; N, 1.82. Found: C, 51.65; H, 2.23; N, 1.74.
^{1}H NMR (500 MHz, CD_2Cl_2, 299 K): δ = 8.68 (br m, 2H, *o*-Py), 8.05 (br m, 1H, *p*-Py), 7.60 (br m, 2H, *m*-Py), 7.33 (m, 2H, *o*-Ph), 7.30 (m, 2H, *m*-Ph), 7.21 (m, 1H, *p*-Ph), 4.52, 4.29 (each d, $^2J_{HH}$ = 13.0 Hz, each 1H, CH_2O), 2.86 (m, 1H, CH), 1.29, 1.00 (each m, each 1H, CH_2B), 1.10 (dd, $^4J_{PH}$ = 21.7 Hz, $^3J_{HH}$ = 6.2 Hz, 3H, CH_3).
^{13}C{^{1}H} NMR (126 MHz, CD_2Cl_2, 299 K): δ = 145.9 (*o*-Py), 142.0 (*i*-Ph), 140.7 (*p*-Py), 128.4 (*m*-Ph), 127.0 (*p*-Ph), 126.7 (*o*-Ph), 125.7 (*m*-Py), 65.9 (CH_2O), 25.9 (br, CH_2B), 24.5 (m, CH), 18.5 (d, $^2J_{PH}$ = 23.5 Hz, CH_3), [C_6F_5 not listed].
^{31}P NMR (202 MHz, CD_2Cl_2, 299 K): δ = -36.1 ($\nu_{1/2}$ ~ 100 Hz).
^{31}P{^{1}H} NMR (202 MHz, CD_2Cl_2, 299 K): δ = -36.1 (quin, $^3J_{PF}$ =27.5 Hz).
^{10}B NMR (54 MHz, CD_2Cl_2, 299 K): δ = 10.5 ($\nu_{1/2}$ ~ 150 Hz).
^{10}B{^{1}H} NMR (54 MHz, CD_2Cl_2, 299 K): δ = 10.5 ($\nu_{1/2}$ ~ 150 Hz).
^{19}F NMR (470 MHz, CD_2Cl_2, 299 K): δ = -129.2 (m, 2F, *o*-$C_6F_5^P$), -151.0 (tm, $^3J_{FF}$ = 20.5 Hz, 1F, *p*-$C_6F_5^P$), -161.5 (m, 2F, *m*-$C_6F_5^P$), [$\Delta\delta^{19}F_{pm}^P$ = 10.4]; -129.8 (m, 2F, *o*-$C_6F_5^P$), -151.7 (tm, $^3J_{FF}$ = 19.8 Hz, 1F, *p*-$C_6F_5^P$), -161.7 (m, 2F, *m*-$C_6F_5^P$), [$\Delta\delta^{19}F_{pm}^P$ = 10.0]; -133.0 (m, 2F, *o*-$C_6F_5^B$), -157.8 (t, $^3J_{FF}$ = 19.6 Hz, 1F, *p*-$C_6F_5^B$), -164.2 (m, 2F, *m*-$C_6F_5^B$), [$\Delta\delta^{19}F_{pm}^B$ = 6.4].
^{19}F, ^{19}F GCOSY (470 MHz/470 MHz, CD_2Cl_2, 299 K)[selected traces]: δ^{19}F/ δ^{19}F = -161.5/-151.0, 129.2 (*m*-$C_6F_5^P$/*o*-$C_6F_5^P$, *p*-$C_6F_5^P$), -161.7/-151.7, -129.8 (*m*-$C_6F_5^P$/*o*-$C_6F_5^P$, *p*-$C_6F_5^P$), -164.2/ -157.8, -133.0 (*m*-$C_6F_5^B$/*o*-$C_6F_5^B$, *p*-$C_6F_5^B$).
^{1}H, ^{1}H GCOSY (500 MHz/500 MHz, CD_2Cl_2, 299 K)[selected traces]: δ^1H/ δ^1H = 8.68/7.60 (*o*-Py/*m*-Py), 8.05/7.60 (*p*-Py/*m*-Py), 2.86/1.29, 1.10, 1.00 (CH/CH_2B, CH_3, CH_2B).

^{1}H, ^{13}C GHSQC (500 MHz/126 MHz, CD_2Cl_2, 299 K): δ^1H/ δ^{13}C = 8.68/145.9 (*o*-Py), 8.05/140.7 (*p*-Py), 7.60/125.7 (*m*-Py), 7.33/126.7 (*o*-Ph), 7.30/128.4 (*m*-Ph), 7.21/127.0 (*p*-Ph), 4.52, 4.29/65.9 (CH_2O), 2.86/24.5 (CH), 1.10/18.5 (CH_3).

^{1}H, ^{13}C GHMBC (500 MHz/126 MHz, CD_2Cl_2, 299 K)[selected traces]: δ^1H/δ^{13}C = 7.33/127.0, 126.7, 65.9 (*o*-Ph/*p*-Ph, *o*-Ph, CH_2O), 7.30/142.0, 128.4 (*m*-Ph/*i*-Ph, *m*-Ph), 4.52/142.0, 126.7 (CH_2O/*i*-Ph, *o*-Ph).

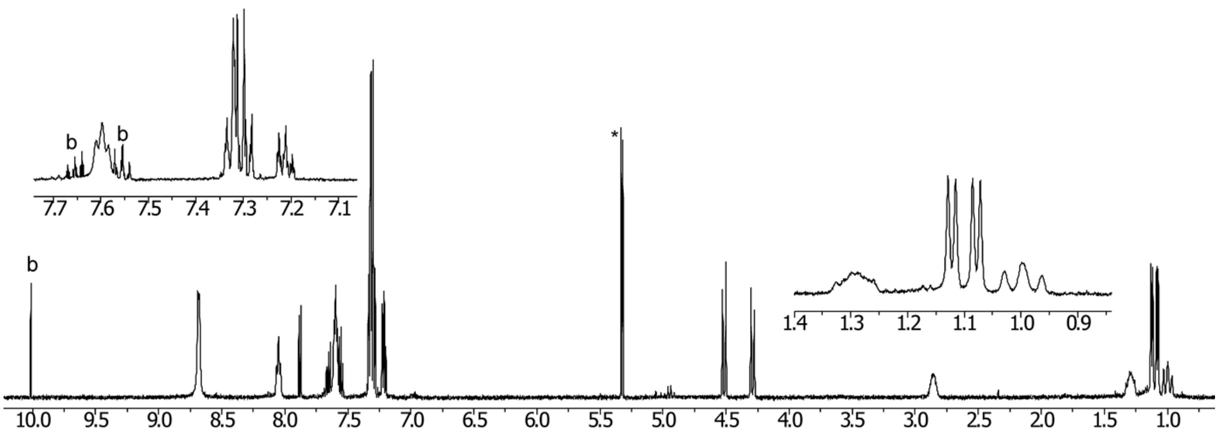


figure 11: ^{1}H NMR (500 MHz, CD_2Cl_2, 299 K, b: benzaldehyde).

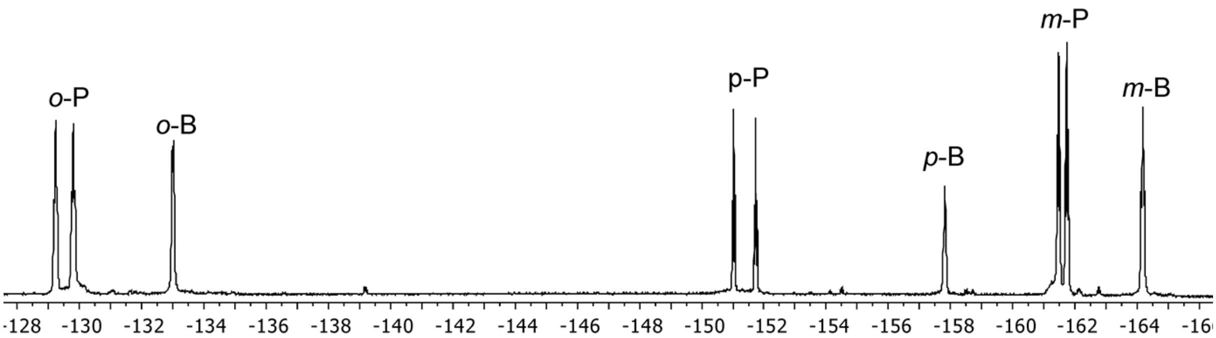


figure 12: ^{19}F NMR (470 MHz, CD_2Cl_2, 299 K).

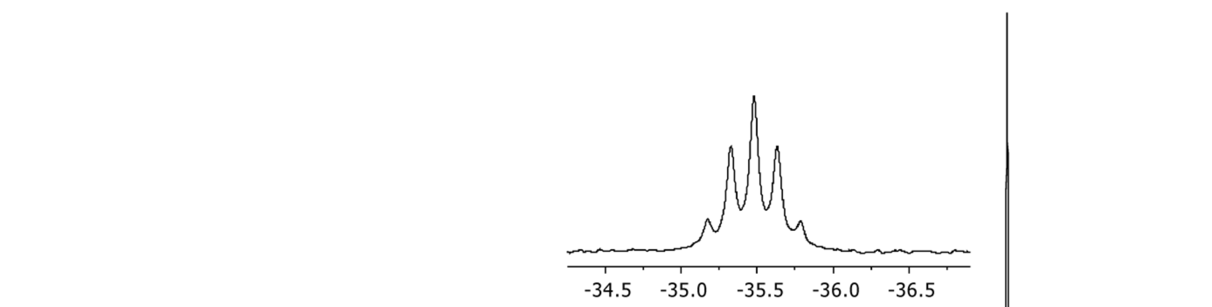


figure 13: ^{31}P{^{1}H}NMR (202 MHz, CD_2Cl_2, 299 K).

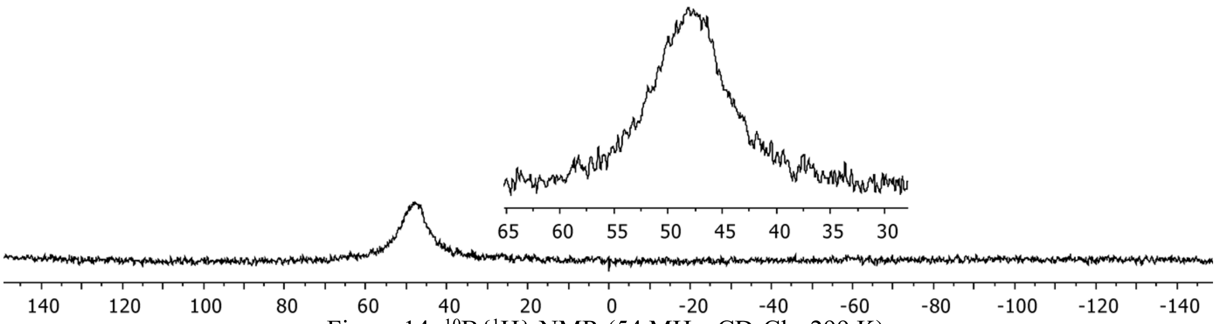
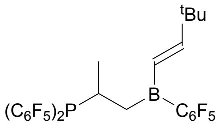


Figure 14: $^{10}B\{^{1}H\}$ NMR (54 MHz, CD_2Cl_2, 299 K).

Compound 9



tert-Butylacetylene (5.3 µL, 0.09 mmol, 3.0 eq.) was added to a suspension of compound $(\mathbf{5})_3$ (25.0 mg, 0.03 mmol, 1.0 eq) in CD_2Cl_2 (0.8 mL) and reacted overnight. All volatiles were removed *in vacuo* to obtain compound **9** as a colourless oil (25.7 mg, 90%).

IR (ATR): υ = 2967 (m), 1646 (m), 1602 (m), 1521 (s), 1474 (s), 1383 (m), 1306 (m), 1264 (w), 1207 (w), 1089 (s), 977 (s), 912 (w), 831 (m), 763 (w), 731 (m), 676 (w), 638 (m), 608 (w), 541 (w), 512 (m), 425 (w).

Elemental analysis: calc. for $C_{27}H_{17}BF_{15}P$ (668.19 g/mol): C, 48.53; H, 2.56.

Found: C, 47.09; H, 2.32.

^{1}H NMR (500 MHz, CD_2Cl_2, 299 K): δ = 6.82 (d, $^3J_{HH}$ = 17.7 Hz, 1H, =CH), 6.45 (d, 17.7 Hz, 1H, BCH), 3.64 (m, 1H, CH), 1.83 (dt, $^2J_{HH}$ = 16.2 Hz, $^3J_{PH}$ = $^3J_{HH}$ = 10.8 Hz, 1H, CH_2B), 1.61 (m, 1H, CH_2B), 1.14 (dd, $^3J_{PH}$ = 20.2 Hz, $^3J_{HH}$ = 6.8 Hz, 3H, CH_3), 1.08 (s, 9H, tBu).

^{13}C{^{1}H} NMR (126 MHz, CD_2Cl_2, 299 K): δ = 176.3 (=CH), 128.2 (br, BCH), 36.3 (tBu), 33.1 (br d, $^2J_{PH}$ = 20.7 Hz, CH_2B), 28.5 (tBu), 25.3 (m, CH), 20.1 (d, $^2J_{PH}$ = 25.6 Hz, CH_3), [C_6F_5 not listed].

^{31}P NMR (202 MHz, CD_2Cl_2, 299 K): δ = -36.4 ($\nu_{1/2}$ ~ 100 Hz).

^{31}P{^{1}H} NMR (202 MHz, CD_2Cl_2, 299 K): δ = -36.4 (quin, $^3J_{PF}$ =31.0 Hz).

^{10}B NMR (54 MHz, CD_2Cl_2, 299 K): δ = 71.5 ($\nu_{1/2}$ ~ 600 Hz).

^{10}B{^{1}H} NMR (54 MHz, CD_2Cl_2, 299 K): δ = 71.5 ($\nu_{1/2}$ ~ 550 Hz).

^{19}F NMR (470 MHz, CD_2Cl_2, 299 K): δ = -129.5 (m, 2F, *o*-$C_6F_5^P$), -150.3 (tm, $^3J_{FF}$ = 20.3 Hz, 1F, *p*-$C_6F_5^P$), -161.1 (m, 2F, *m*-$C_6F_5^P$), [$\Delta\delta^{19}F_{pm}^P$ = 10.8]; -129.9 (m, 2F, *o*-$C_6F_5^P$), -150.8 (tm, $^3J_{FF}$ = 20.3 Hz, 1F, *p*-$C_6F_5^P$), -161.2 (m, 2F, *m*-$C_6F_5^P$), [$\Delta\delta^{19}F_{pm}^P$= 10.5]; -132.5 (m, 2F, *o*-$C_6F_5^B$), -153.6 (t, $^3J_{FF}$ = 20.3 Hz, 1F, *p*-$C_6F_5^B$), -162.5 (m, 2F, *m*-$C_6F_5^B$), [$\Delta\delta^{19}F_{pm}^B$= 8.9].

^{19}F, ^{19}F GCOSY (470 MHz/470 MHz, CD_2Cl_2, 299 K)[selected traces]: δ^{19}F/ δ^{19}F = -161.1/-129.5, -150.3 ((*m*-$C_6F_5^P$/*o*-$C_6F_5^P$, *p*-$C_6F_5^P$), -161.2/-129.9, -150.8 (*m*-$C_6F_5^P$/*o*-$C_6F_5^P$, *p*-$C_6F_5^P$), -162.5/-132.5, 153.6 (*m*-$C_6F_5^B$/*o*-$C_6F_5^B$, *p*-$C_6F_5^B$).

^{1}H, ^{1}H GCOSY (500 MHz/500 MHz, CD_2Cl_2, 299 K)[selected traces]: δ^1H/ δ^1H = 6.82/6.45, 1.83, 1.61 (=CH/BCH, CH_2B, CH_2B), 6.45/1.08 (BCH/tBu), 3.64/1.83, 1.61, 1.14 (CH/CH_2B, CH_2B, CH_3).

^{1}H, ^{13}C GHSQC (500 MHz/126 MHz, CD_2Cl_2, 299 K): δ^1H/ δ^{13}C = 6.82/176.3 (=CH), 6.45/128.2 (BCH), 3.64/25.3 (CH), 1.83, 1.61/33.1 (CH_2B), 1.14/20.1 (CH_3), 1.08/28.5 (tBu).
^{1}H, ^{13}C GHMBC (500 MHz/126 MHz, CD_2Cl_2, 299 K)[selected trace]: δ^1H/ δ^{13}C = 6.82/128.2, 36.3, 28.5 (=CH/tBu, tBu).

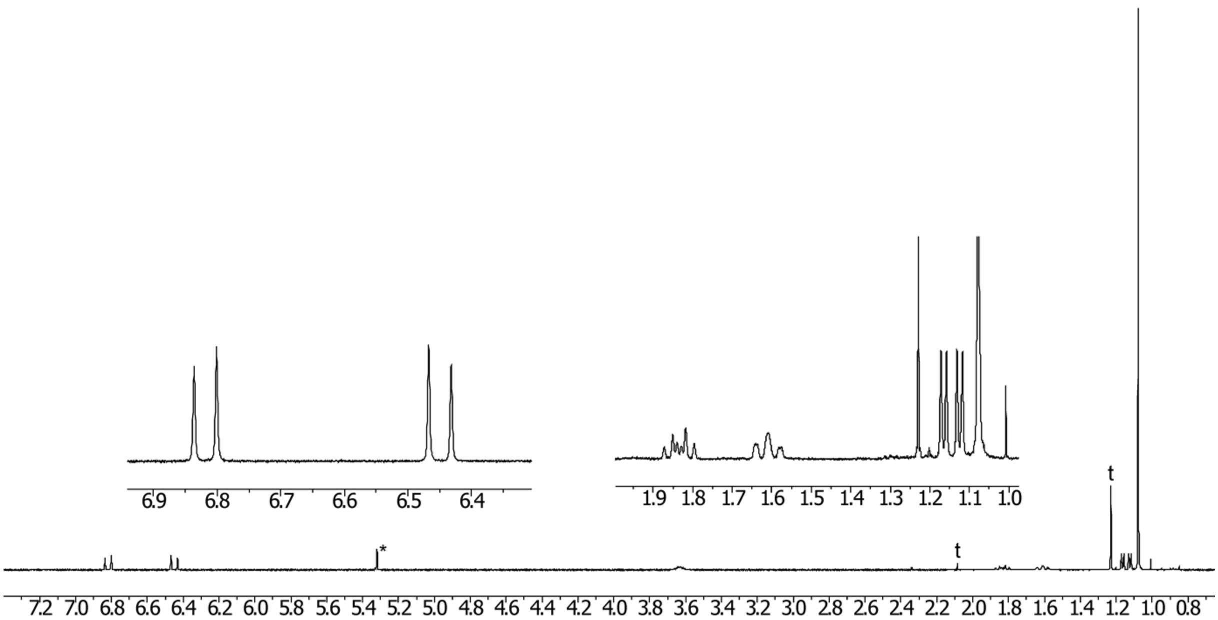


figure 15: ^{1}H NMR (500 MHz, CD_2Cl_2, 299 K), t: *tert*-butylacetylene.

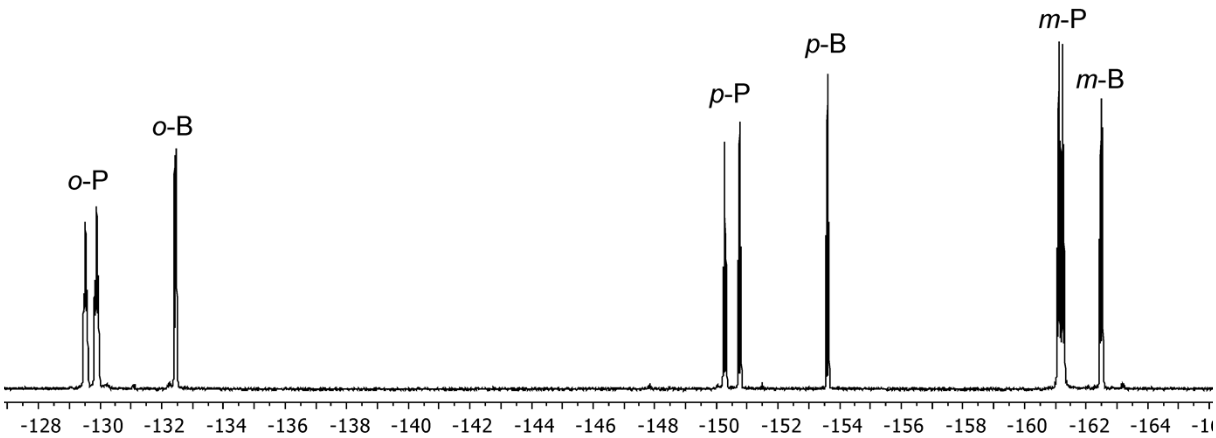


figure 16: ^{19}F NMR (470 MHz, CD_2Cl_2, 299 K).

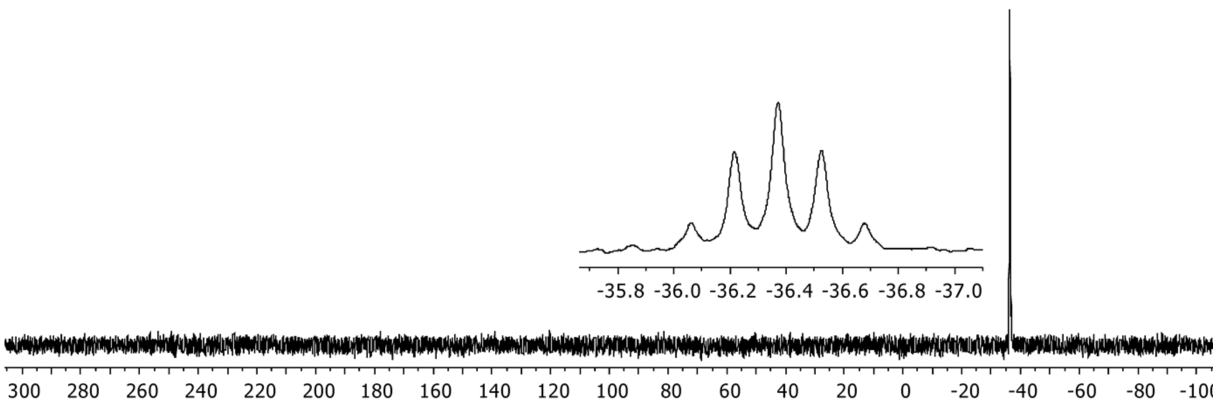


figure 17: ^{31}P{^{1}H}NMR (202 MHz, CD_2Cl_2, 299 K).

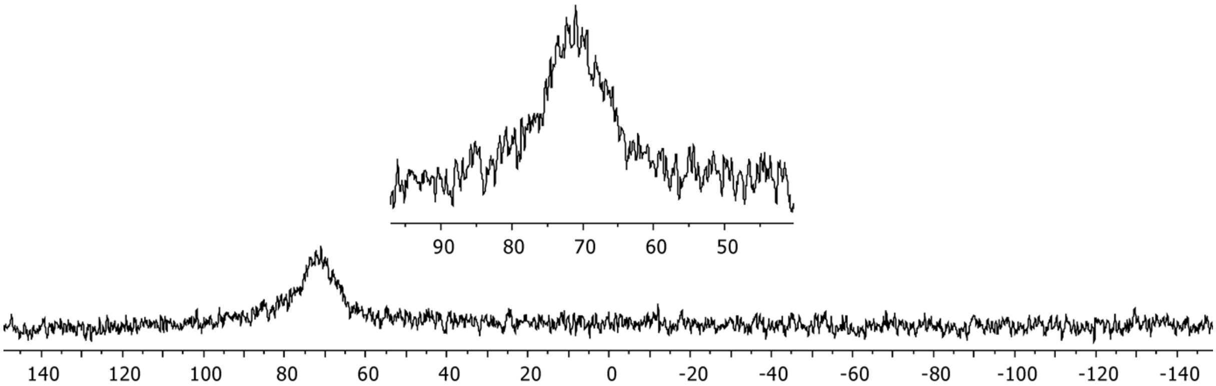
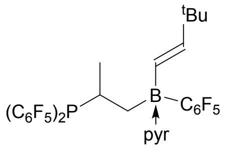


figure 18: $^{10}B\{^{1}H\}$ NMR (54 MHz, CD_2Cl_2, 299 K).

Preparation of Compound 10



tert-Butylacetylene (10.5 µL, 0.09 mmol, 3.0 eq) was added to a suspension of compound (**5**)$_3$ (50.0 mg, 0.03 mmol, 1.0 eq) in dichloromethane (10 mL) and stirred overnight. Then pyridine (7.0 µL, 0.09 mmol, 3.0 eq) was added and the reaction mixture was stirred overnight again. All volatiles were removed *in vacuo* and the remaining residue was dissolved in pentane (ca. 5 mL) and stored at -34 °C. After two days the formed colourless precipitate was isolated and dried *in vacuo* (39.8 mg, 63%). In the CD_2Cl_2 solution of the obtained solid two diastereomers $\mathbf{10_{major}}$ and $\mathbf{10_{minor}}$ were observed (major/minor ~ 5/3). Crystals suitable for the X-ray single crystal structure analysis were obtained by slow crystallisation from a heptane/dichloromethane solution of **10**.

IR (ATR): υ = 2956 (w), 1639 (w), 1514 (s), 1473 (s), 1457 (s), 1378 (m), 1282 (m), 1252 (w), 1216 (w), 1083 (s).

Melting point: 259 °C.

Elemental analysis: calc. for $C_{32}H_{22}BF_{15}NP$ (747.29 g/mol): C, 51.43; H, 2.97; N, 1.87. Found: C, 51.23; H, 2.71; N, 1.81.

Diastereomer $\mathbf{10_{major}}$:

^{1}H NMR (600 MHz, CD_2Cl_2, 299 K): δ = 8.47 (m, 2H, *o*-Py), 8.06 (m, 1H, *p*-Py), 7.60 (m, 2H, *m*-Py), 6.21 (dt, $^3J_{HH}$ = 17.7 Hz, $^4J_{HH}$ = 2.2 Hz, 1H, BCH), 4.84 (d, $^3J_{HH}$ = 17.7 Hz, 1H, =CH), 3.10 (m, 1H, CH), 1.03 (m, each 2H, CH_2B), 1.00 (dd, $^4J_{PH}$ = 21.6 Hz, $^3J_{HH}$ = 6.5 Hz, 3H, CH_3), 0.92 (s, 9H, tBu).

^{13}C{^{1}H} NMR (151 MHz, CD_2Cl_2, 299 K): δ = 146.8 (=CH), 146.2 (*o*-Py), 141.3 (*p*-Py), 133.8 (br, BCH), 126.0 (*m*-Py), 34.1 (tBu), 29.7 (tBu), 28.7 (br, CH_2B), 25.3 (br m, CH), 19.2 (d, $^3J_{PC}$ = 25.9 Hz, CH_3), [C_6F_5 not listed].

^{31}P NMR (243 MHz, CD_2Cl_2, 299 K): δ = -37.7 ($\nu_{1/2}$ ~ 100 Hz).

^{31}P{^{1}H} NMR (243 MHz, CD_2Cl_2, 299 K): δ = -37.7 (quin, $^3J_{PF}$ =29.8 Hz).

^{10}B NMR (64 MHz, CD_2Cl_2, 299 K): δ = 0.4 (br, $\nu_{1/2}$ ~ 200 Hz).

^{10}B{^{1}H} NMR (64 MHz, CD_2Cl_2, 299 K): δ = 0.4 (br, $\nu_{1/2}$ ~ 200 Hz).

^{19}F NMR (564 MHz, CD_2Cl_2, 299 K): δ = -129.2 (m, 2F, *o*-$C_6F_5^P$), -151.2 (tm, $^3J_{FF}$ = 20.3 Hz, 1F, *p*-$C_6F_5^P$), -161.9 (m, 2F, *m*-$C_6F_5^P$), [$\Delta\delta^{19}F_{pm}{}^P$ = 10.7]; -130.0 (m, 2F, *o*-$C_6F_5^P$), -152.1 (tm, $^3J_{FF}$ = 20.3 Hz, 1F, *p*-$C_6F_5^P$), -161.9 (m, 4F, *m*-$C_6F_5^P$), [$\Delta\delta^{19}F_{pm}{}^P$= 9.8]; -132.4 (m, 2F, *o*-$C_6F_5^B$), -159.7 (t, $^3J_{FF}$ = 20.2 Hz, 1F, *p*-$C_6F_5^B$), -164.9 (m, 2F, *m*-$C_6F_5^B$), [$\Delta\delta^{19}F_{pm}{}^B$= 5.2].

^{19}F, ^{19}F GCOSY (564 MHz/564 MHz, CD_2Cl_2, 299 K)[selected traces]: δ^{19}F/ δ^{19}F = -161.9/129.2, -151.2 (*m*-$C_6F_5^P$/*o*-$C_6F_5^P$, *p*-$C_6F_5^P$), -161.9/-130.0, 152.1 (*m*-$C_6F_5^P$/*o*-$C_6F_5^P$, *p*-$C_6F_5^P$), -164.9/-132.4, -159.7 (*m*-$C_6F_5^B$/*o*-$C_6F_5^B$, *p*-$C_6F_5^B$).

^{1}H, ^{1}H GCOSY (600 MHz/600 MHz, CD_2Cl_2, 299 K)[selected traces]: δ^{1}H/ δ^{1}H = 8.47/8.06, 7.60 (*o*-Py/*p*-Py, *m*-Py), 6.21/4.84, 0.92 (BCH/=CH, tBu), 3.10/1.03, 1.00 (CH/CH_2B, CH_3).

^{1}H, ^{13}C GHSQC (600 MHz/151 MHz, CD_2Cl_2, 299 K): δ^{1}H/ δ^{13}C = 8.47/146.2 (*o*-Py), 8.06/141.3 (*p*-Py), 7.60/126.0 (*m*-Py), 6.21/133.8 (BCH), 4.84/146.8 (=CH), 3.10/25.3 (CH), 1.03/28.7 (CH_2B), 1.00/19.2 (CH_3), 0.92/29.7 (tBu).

^{1}H, ^{13}C GHMBC (600 MHz/151 MHz, CD_2Cl_2, 299 K)[selected trace]: δ^{1}H/ δ^{13}C = 6.21/146.8, 34.1 (BCH/=CH, tBu).

Diastereomer 10_{minor}:

^{1}H NMR (600 MHz, CD_2Cl_2, 299 K): δ = 8.47 (m, 2H, *o*-Py), 8.10 (m, 1H, *p*-Py), 7.60 (m, 2H, *m*-Py), 6.12 (d $^3J_{HH}$ = 17.8 Hz, 1H, BCH), 5.05 (d, $^3J_{HH}$ = 17.8 Hz, 1H, =CH), 2.92 (m, 1H, CH), 1.31, 0.91 (each m, each 1H, CH_2B), 1.00 (dd, $^4J_{PH}$ = 21.6 Hz, $^3J_{HH}$ = 6.5 Hz, 3H, CH_3), 0.95 (s, 9H, tBu).

^{13}C{^{1}H} NMR (151 MHz, CD_2Cl_2, 299 K): δ = 147.9 (=CH), 146.2 (*o*-Py), 141.3 (*p*-Py), 132.1 (br, BCH), 126.0 (*m*-Py), 34.2 (tBu), 29.7 (tBu), 28.7 (br, CH_2B), 25.3 (br m, CH), 19.2 (d, $^3J_{PC}$ = 23.1 Hz, CH_3), [C_6F_5 not listed].

^{31}P NMR (243 MHz, CD_2Cl_2, 299 K): δ = -35.7 ($\nu_{1/2}$ ~ 100 Hz).

^{31}P{^{1}H} NMR (243 MHz, CD_2Cl_2, 299 K): δ = -35.7 (quin, $^3J_{PF}$ =28.0 Hz).

^{10}B NMR (64 MHz, CD_2Cl_2, 299 K): δ = 0.4 ($\nu_{1/2}$ ~ 200 Hz).

^{10}B{^{1}H} NMR (64 MHz, CD_2Cl_2, 299 K): δ = 0.4 ($\nu_{1/2}$ ~ 200 Hz).

^{19}F NMR (564 MHz, CD_2Cl_2, 299 K): δ = -129.1 (m, 2F, *o*-$C_6F_5^P$), -151.5 (tm, $^3J_{FF}$ = 20.2 Hz, 1F, *p*-$C_6F_5^P$), -161.4 (m, 2F, *m*-$C_6F_5^P$), [$\Delta\delta^{19}F_{pm}{}^P$ = 9.9]; -129.6 (m, 2F, *o*-$C_6F_5^P$), -151.8 (tm, $^3J_{FF}$ = 21.0 Hz, 1F, *p*-$C_6F_5^P$), -161.8 (m, 2F, *m*-$C_6F_5^P$), [$\Delta\delta^{19}F_{pm}{}^P$= 10.0]; -132.2 (m, 2F, *o*-$C_6F_5^B$), -160.2 (t, $^3J_{FF}$ = 20.2 Hz, 1F, *p*-$C_6F_5^B$), -165.0 (m, 2F, *m*-$C_6F_5^B$), [$\Delta\delta^{19}F_{pm}{}^B$= 4.8].

^{19}F, ^{19}F GCOSY (564 MHz/564 MHz, CD_2Cl_2, 299 K)[selected traces]: δ^{19}F/ δ^{19}F = -161.4/-129.1, -151.5 (*m*-$C_6F_5^P$/*o*-$C_6F_5^P$, *p*-$C_6F_5^P$), -161.8/-129.6, -151.8 (*m*-$C_6F_5^P$/*o*-$C_6F_5^P$, *p*-$C_6F_5^P$), -165.0/-132.2, -160.2 (*m*-$C_6F_5^B$/*o*-$C_6F_5^B$, *p*-$C_6F_5^B$).

^{1}H, ^{1}H GCOSY (600 MHz/600 MHz, CD_2Cl_2, 299 K)[selected traces]: δ^{1}H/ δ^{1}H = 8.47/8.10, 7.60 (*o*-Py/*p*-Py, *m*-Py), 6.12/5.05 (BCH/=CH), 2.92/1.31, 1.00, 0.91 (CH/CH_2B, CH_3, CH_2B).

^{1}H, ^{13}C GHSQC (600 MHz/151 MHz, CD_2Cl_2, 299 K): δ^1H/ δ^{13}C = 8.47/146.2 (*o*-Py), 8.10/141.3 (*p*-Py), 7.60/126.0 (*m*-Py), 5.05/147.9 (=CH), 2.92/25.3 (CH), 1.31, 0.91/28.7 (CH_2B), 1.00/19.2 (CH_3), 0.95/29.7 (tBu).

^{1}H, ^{13}C GHMBC (600 MHz/151 MHz, CD_2Cl_2, 299 K)[selected traces]: δ^1H/ δ^{13}C = 6.12/147.9, 34.2 (BCH/=CH, tBu).

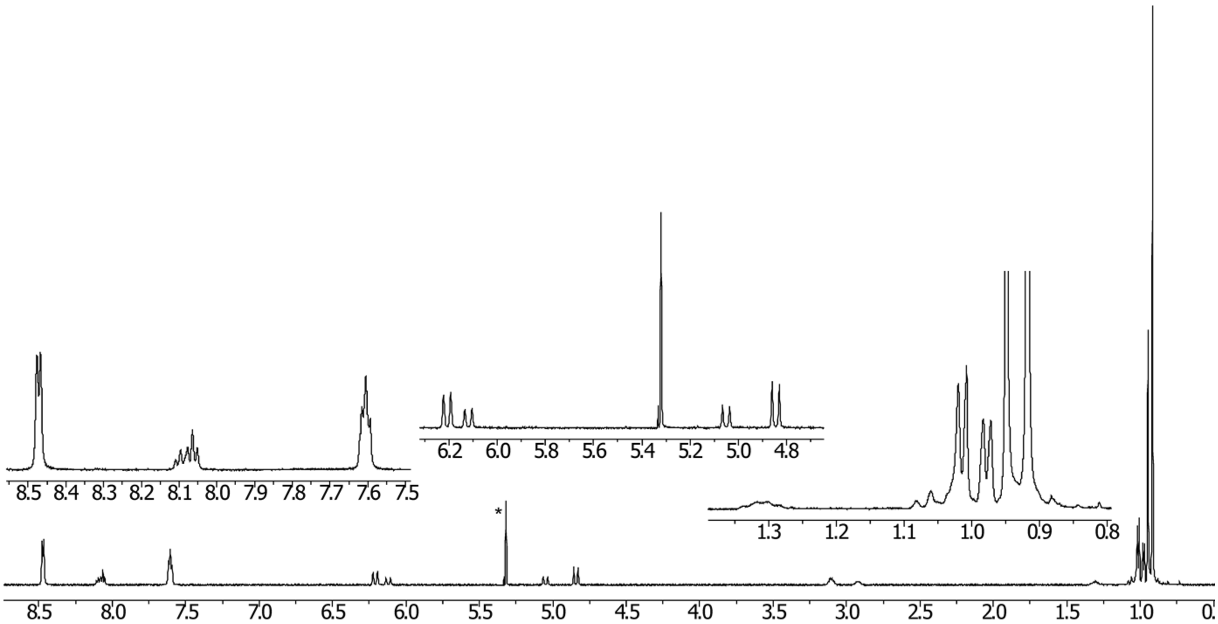


figure 19: ^{1}H NMR (500 MHz, CD_2Cl_2, 299 K).

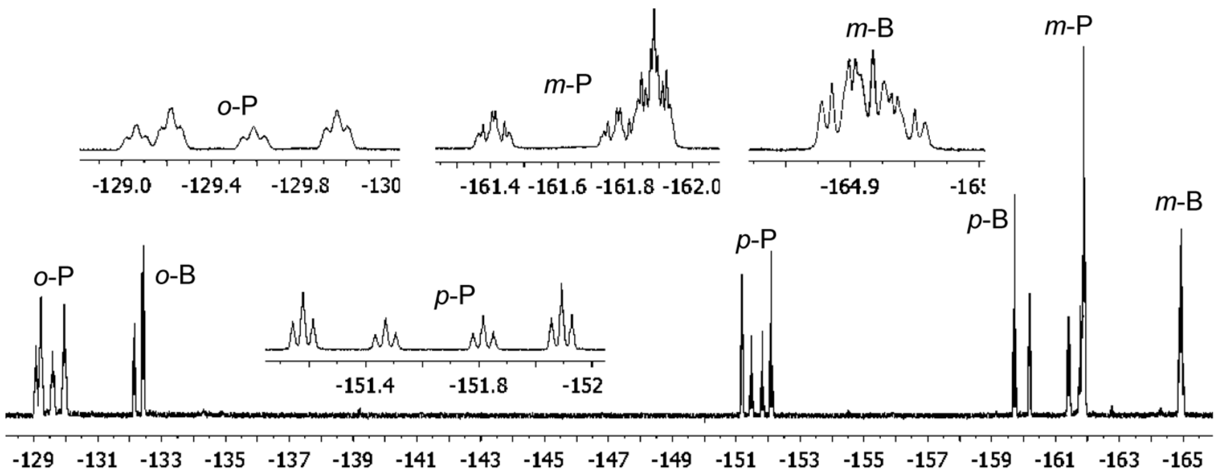


figure 20: ^{19}F NMR (470 MHz, CD_2Cl_2, 299 K).

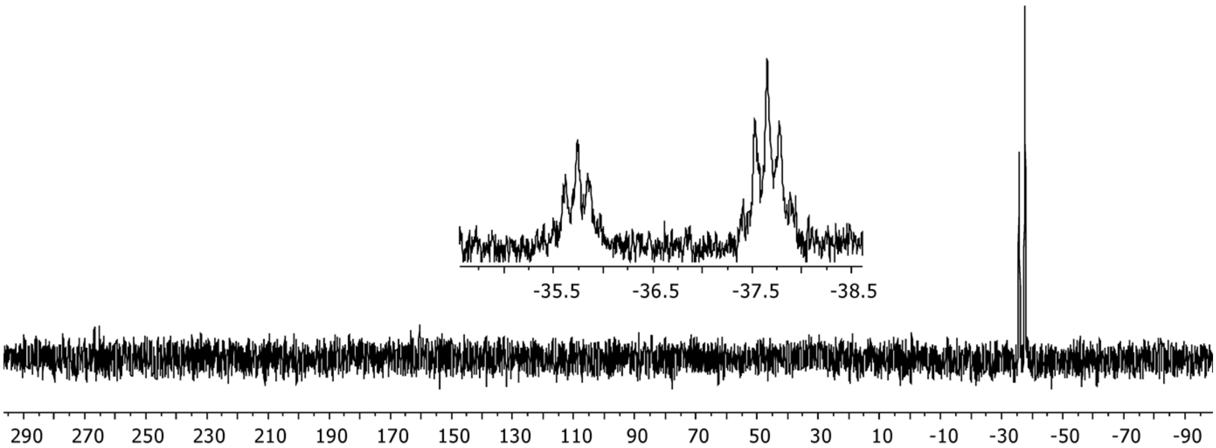


figure 21: $^{31}P\{^{1}H\}$NMR (202 MHz, CD_2Cl_2, 299 K).

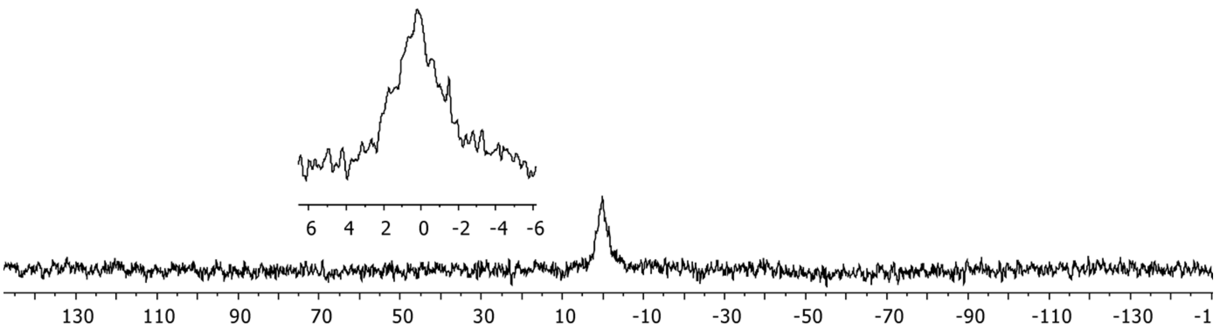


figure 22: $^{10}B\{^{1}H\}$ NMR (54 MHz, CD_2Cl_2, 299 K),

X-ray crystal structure analysis of compound 10: formula $C_{32}H_{22}BF_{15}NP$, M = 747.29, colourless crystal, 0.20 x 0.10 x 0.04 mm, a = 9.5845(2), b = 11.8194(4), c = 15.2277(4) Å, α = 105.663(2), β = 102.191(2), γ = 90.409(1)°, V = 1619.75(8) Å^3, ρ_{calc} = 1.532 gcm^{-3}, μ = 0.196 mm^{-1}, empirical absorption correction (0.961 ≤ T ≤ 0.992), Z = 2, triclinic, space group $P\bar{1}$ (No. 2), λ = 0.71073 Å, T = 223(2) K, ω and φ scans, 13030 reflections collected (±h, ±k, ±l), [(sinθ)/λ] = 0.59 Å^{-1}, 5560 independent (R_{int} = 0.035) and 4162 observed reflections [I>2σ(I)], 455 refined parameters, R = 0.069, wR^2 = 0.141, max. (min.) residual electron density 0.29 (-0.21) e.Å^{-3}, hydrogen atoms calculated and refined as riding atoms.

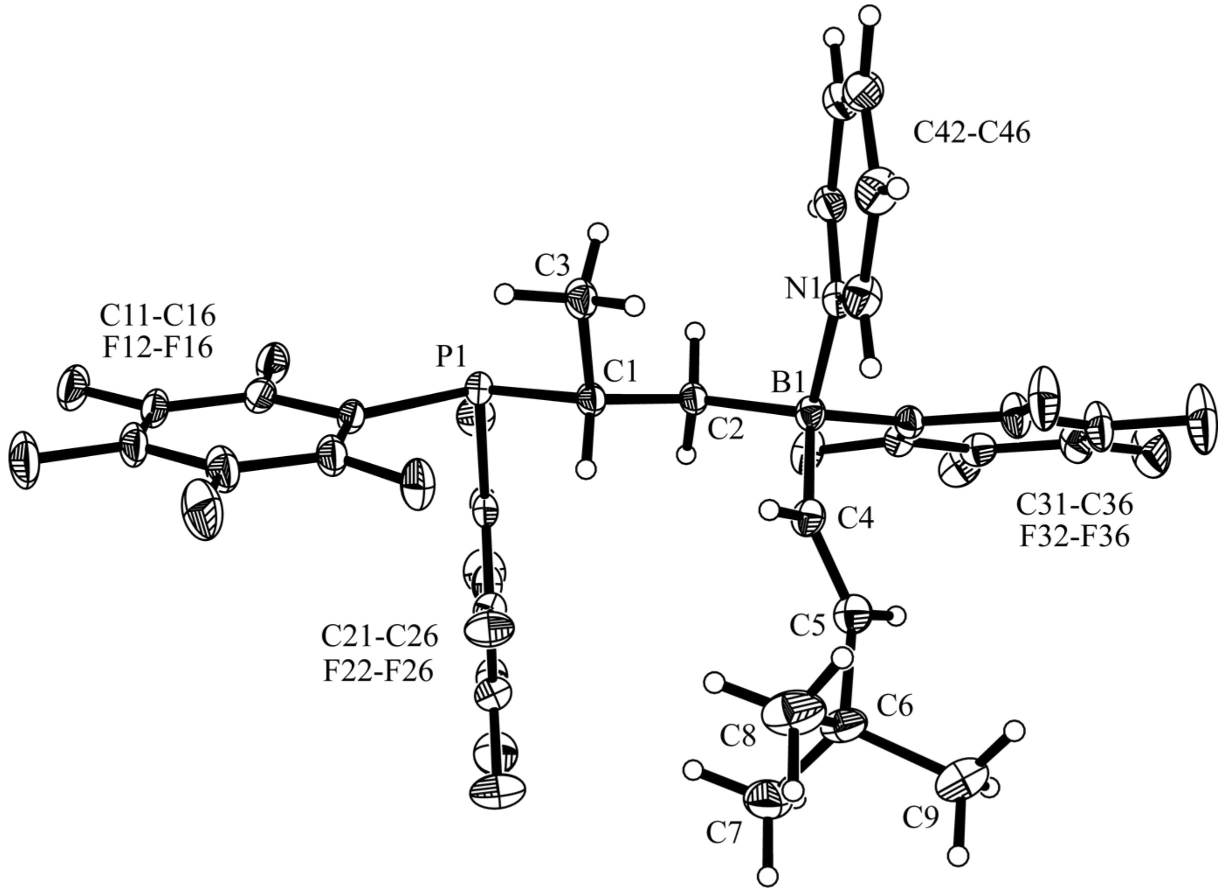


figure 23: X-ray crystal structure of compound **10**.