Supporting Information

Remarkable Coordination Behavior of Alkyl Isocyanides Toward Unsaturated Vicinal Frustrated P/B Lewis Pairs

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§ Computational chemistry.
$ Solid state NMR.
‡ X-ray crystal structure analyses.

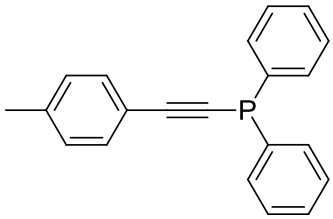
Materials and Methods. All syntheses involving air- and moisture sensitive compounds were carried out using standard Schlenk-type glassware (or in a glove box) under an atmosphere of argon. Solvents were dried with the procedure according to Grubbs (A. B. Pangborn, M. A. Giardello, R. H. Grubbs, R. K. Rosen, F. J. Timmers, *Organometallics* **1996**, *15*, 1518-1520) or were distilled from appropriate drying agents and stored under an argon atmosphere. NMR spectra were recorded on a *Bruker* AV 300 (^{1}H: 300 MHz, ^{13}C: 76 MHz, ^{31}P: 122 MHz, ^{11}B: 96 MHz, ^{19}F: 282 MHz), a *Bruker* AV 400 (^{1}H: 400 MHz, ^{13}C: 101 MHz, ^{31}P: 162 MHz), a *Agilent* DD2- 500 MHz (^{1}H: 500 MHz, ^{13}C: 126 MHz, ^{19}F: 470 MHz, ^{11}B:160 MHz, ^{31}P: 202 MHz) and on a *Agilent* DD2- 600 MHz (^{1}H: 600 MHz, ^{13}C: 151 MHz, ^{19}F: 564 MHz, ^{11}B:192 MHz, ^{31}P: 243 MHz). ^{1}H NMR and ^{13}C NMR: chemical shifts δ are given relative to TMS and referenced to the solvent signal. ^{19}F NMR: chemical shifts δ are given relative to $CFCl_3$ (external reference), ^{11}B NMR: chemical shifts δ are given relative to $BF_3{\cdot}Et_2O$ (external reference), ^{31}P NMR: chemical shifts δ are given relative to H_3PO_4 (85% in D_2O) (external reference). NMR assignments were supported by additional 2D NMR experiments.

Elemental analyses were performed on a *Elementar Vario El III*. IR spectra were recorded on a *Varian* 3100 FT-IR (Excalibur Series). Melting points and decomposition points were obtained with a DSC 2010 (*TA Instruments*). HRMS was recorded on GTC Waters Micromass (Manchester, UK). X-Ray crystal structure analyses: 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). Thermals ellipsoids are shown with 50% probability, *R*-values are given for observed reflections, and wR^2 values are given for all reflections. *Exceptions and special features*: For the compound **4a** an unidentified disordered solvent molecule was found in the asymmetrical unit and could not be satisfactorily refined. The program SQUEEZE (A. L. Spek *J. Appl. Cryst*., **2003**, *36*, 7-13) was therefore used to remove mathematically the effect of the solvent. The n-Butyl group at N4 atom and the two dichloromethane molecule in compound **7a** were found disordered over two positions. Several restraints (EADP, SADI and ISOR) were used in order to improve refinement stability. The two n-Butyl groups at N4A and N4B in compound **7b** were found disordered over two positions. Several restraints (SIMU, SADI and SAME) were used in order to improve refinement stability. Refinement of these atoms was done with ISOR restrain.

Materials: Compound **1** [(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.] was prepared according to modified procedures reported in the literature. $B(C_6F_5)_3$ (**2a**) [(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.], $H_3CB(C_6F_5)_2$ (**2b**) [(a) R. E. v. H. Spence, W. E. Piers, Y. Sun, M. Parvez, L. R. MacGillivray, M. J. Zaworotko, *Organometallics* **1998**, *17*, 2459–2469; (b) T. E. Cole, R. Quintanilla, B. M. Smith, D. Hurst, *Tetrahedron Lett.* **1992**, *33*, 2761-2764.] (caution: the intermediate involved is explosive), **3a** and **3b** [(a) O. Ekkert, G. Kehr, R. Fröhlich, G. Erker, *J. Am. Chem. Soc.*, **2011**, *133*, 4610-4616; (b) O. Ekkert, G. Kehr, R. Fröhlich, G. Erker, *Chem. Commun.* **2011**, *47*, 10482–10484.] were prepared according to procedures reported in the literature.

Caution: many isocyanides are toxic and must be handled with due care.

Synthesis of 1.



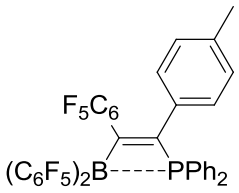
p-Tolylacetylene (1.0 ml, 7.89 mmol) was dissolved in diethyl ether (15 ml). Then *n*-butyllithium solution (1.6 M in hexane, 5.0 ml, 7.89 mmol) was added at 0°C. The solution was stirred for 2h at room temperature. Subsequently the reaction mixture was again cooled to 0°C and chlorodiphenylphosphane (1.41 ml, 7.89 mmol) was added. The reaction mixture was warmed to room temperature and stirred for 3h. The solvent was removed under vacuum and the residue was extracted with pentane (30 ml) and product **1** (1.32 g, 4.38 mmol, 56%) could be isolated as a light brown solid.

^{1}H NMR (400 MHz, 295 K, C_6D_6): δ = 7.78 (m, 4H, *o*−Ph), 7.32 (m, 2H, *o*−Tol), 7.07 (m, 4H, *m*−Ph), 7.03 (m, 2H, *p*−Ph), 6.73 (m, 2H, *m*−Tol), 1.92 (s, 3H, CH_3^{Tol}).

^{13}C{^{1}H} NMR (101 MHz, 298 K, C_6D_6)**:** δ = 139.1 (*p*−Tol), 137.2 (d, $^{1}J_{PC}$ = 7.3 Hz, *i*−Ph), 133.1 (d, $^{2}J_{PC}$ = 21.2 Hz, *o*−Ph), 132.1 (d, $^{4}J_{PC}$ = 1.5 Hz, *o*−Tol), 129.3 (*m*−Tol), 129.1 (*p*−Ph), 128.9 (d, $^{3}J_{PC}$ = 7.2 Hz, *m*−Ph), 120.3 (d, $^{3}J_{PC}$ = 1.2 Hz, *i*−Tol), 108.7 (d, $^{2}J_{PC}$ = 4.2 Hz, ^{Tol}C≡), 86.0 (d, $^{1}J_{PC}$ = 7.3 Hz, ≡C^{P}), 21.3 (CH_3^{Tol}).

^{31}P{^{1}H} NMR (121 MHz, 298 K, C_6D_6)**:** δ = −32.8 ($v_{1/2}$ ~ 2 Hz).

Synthesis of 3a.



Diphenyl(*p*-tolylethinyl)phosphane (**1**) (0.300 g, 0.999 mmol) and $B(C_6F_5)_3$ (**2a**) (0.511 g, 0.999 mmol) were dissolved in toluene (15 ml) and stirred for five hours at 70 ºC. Subsequently evaporation of the solvent *in vacuo* the crude product was washed with pentane (3 × 15 ml). After drying under vacuum gave compound **3a** (0.575 g, 0.708 mmol, 71%) as a light yellow solid. Crystals suitable for X-ray crystal structure analysis were grown by slow diffusion of pentane into a dichloromethane solution of **3a** at −36 ºC. **Anal. Calc.** for $C_{39}H_{17}BF_{15}P$: C, 57.66, H, 2.11. Found: C, 57.68, H, 2.12. **IR** (KBr): $\tilde{\nu}$ / cm^{-1} = 3059 (m), 2924 (m), 2869 (w), 1649 (s), 1518 (s), 1456 (s), 1283 (m), 1112 (s), 1057 (m), 967 (s). **M.p.** (DSC): 251 °C, **decomp.** (DSC): 274 °C.

^{1}H NMR (500 MHz, 299 K, CD_2Cl_2)**:** δ = 7.56 (m, 2H, *p*−Ph), 7.40 (m, 4H, *m*−Ph), 7.37 (m, 4H, *o*−Ph), 7.07 (m, 2H, *m*−Tol), 6.99 (m, 2H, *o*−Tol), 2.33 (s, 3H, CH_3^{Tol}).

^{13}C{^{1}H} NMR (126 MHz, 299 K, CD_2Cl_2)**:** δ = 159.4 (br, ^{B}C=), 148.4 (dm, $^{1}J_{FC}$ ~ 240 Hz, C_6F_5), 143.9 (dm, $^{1}J_{FC}$ ~ 245 Hz, C_6F_5), 143.6 (d, $^{1}J_{PC}$ = 52.7 Hz, =C^{P}), 140.9 (dm, $^{1}J_{FC}$ ~ 250 Hz, C_6F_5), 140.3 (d, $^{5}J_{PC}$ = 0.9 Hz, *p*−Tol), 140.2 (dm, $^{1}J_{FC}$ ~ 250 Hz, C_6F_5), 138.1

(dm, $^{1}J_{FC}$ ~ 250 Hz, C_6F_5), 137.3 (dm, $^{1}J_{FC}$ ~ 250 Hz, C_6F_5), 132.8 (d, $^{4}J_{PC}$ = 3.0 Hz, *p*–Ph), 132.4 (d, $^{2}J_{PC}$ = 9.3 Hz, *o*–Ph), 132.0 (d, $^{2}J_{PC}$ = 2.4 Hz, *i*–Tol), 130.2 (*m*–Tol), 129.6 (d, $^{3}J_{PC}$ = 10.8 Hz, *m*–Ph), 127.2 (d, $^{3}J_{PC}$ = 4.7 Hz, *o*–Tol), 124.8 (d, $^{1}J_{PC}$ = 43.7 Hz, *i*–Ph), 115.7 (br, *i*–C_6F_5), 21.5 (CH_3^{Tol}).

^{19}F NMR (470 MHz, 299 K, CD_2Cl_2)**:** δ = −130.4 (m, 4F, *o*–BC_6F_5), −138.4 (m, 2F, *o*–C_6F_5), −156.2 (t, $^{3}J_{FF}$ = 20.9 Hz, 1F, *p*–C_6F_5), −157.7 (tm, $^{3}J_{FF}$ = 20.4 Hz, 2F, *p*–BC_6F_5), −163.2 (m, 2F, *m*–C_6F_5), −164.9 (m, 4F, *m*–BC_6F_5) [$\Delta\delta^{B}$(m, p) = 7.2].

^{11}B{^{1}H} NMR (160 MHz, 299 K, CD_2Cl_2)**:** δ = −6.6 ($\nu_{1/2}$ ~ 250 Hz).

^{31}P{^{1}H} NMR (202 MHz, 299 K, CD_2Cl_2)**:** δ = 13.1 ($\nu_{1/2}$ ~ 90 Hz).

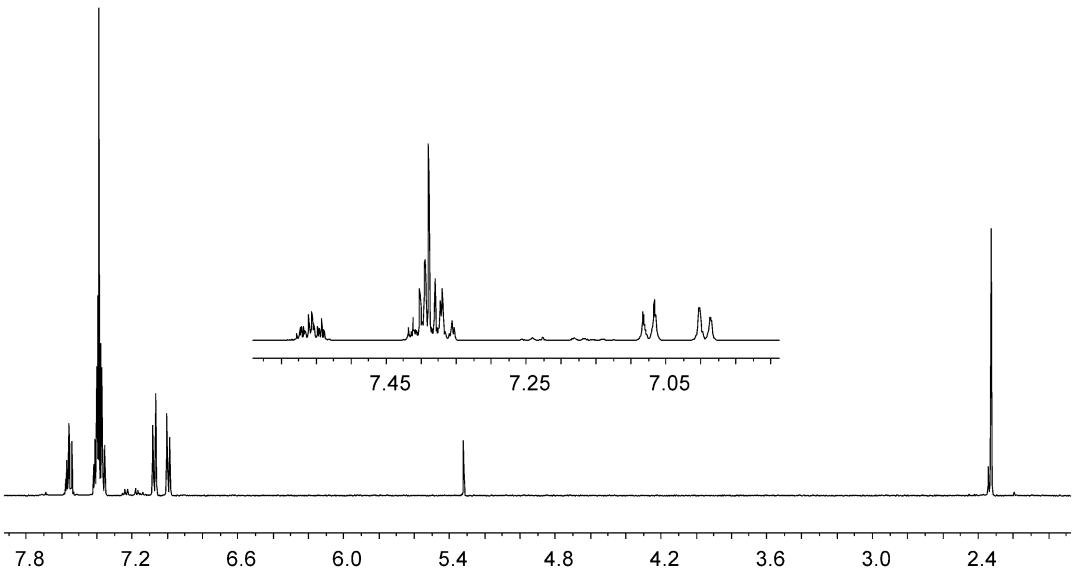
TOCSY (500 MHz / 500 MHz, 299 K, CD_2Cl_2)**:** $\delta^{1}H_{irr.}$ / $\delta^{1}H_{res.}$ = 7.56 / 7.40, 7.37 (*p*–Ph / *m*–, *o*–Ph), 7.07 / 6.99, 2.33 (*m*–Tol / *o*–Tol, CH_3^{Tol}).

NOE (500 MHz / 500 MHz, 299 K, CD_2Cl_2)**:** $\delta^{1}H_{irr.}$ / $\delta^{1}H_{res.}$ = 7.56 / 7.40, 7.37 (*p*–Ph / *m*–, *o*–Ph), 7.40, 7.37 / 7.56 (*m*–, *o*–Ph / *p*–Ph), 7.07 / 6.99, 2.33 (*m*–Tol / *o*–Tol, CH_3^{Tol}), 6.99 / 7.07 (*o*–Tol / *m*–Tol), 2.33 / 7.07 (CH_3^{Tol} / *m*–Tol).

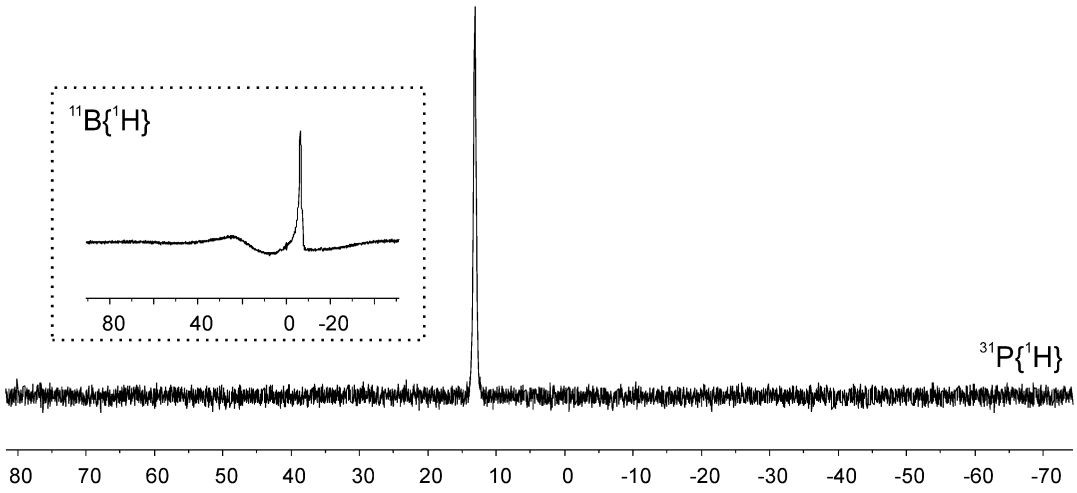
^{1}H,^{1}H COSY (500 MHz / 500 MHz, 299 K, CD_2Cl_2)**:** $\delta^{1}H$ / $\delta^{1}H$ = 7.56 / 7.40, 7.37 (*p*–Ph / *m*–, *o*–Ph), 7.07 / 6.99, 2.33 (*m*–Tol / *o*–Tol, CH_3^{Tol}).

^{1}H,^{13}C GHSQC (500 MHz / 126 MHz, 299 K, CD_2Cl_2)**:** $\delta^{1}H$ / $\delta^{13}C$ = 7.56 / 132.8 (*p*–Ph), 7.40 / 129.6 (*m*–Ph), 7.37 / 132.4 (*o*–Ph), 7.07 / 130.2 (*m*–Tol), 6.99 / 127.2 (*o*–Tol), 2.33 / 21.5 (CH_3^{Tol}).

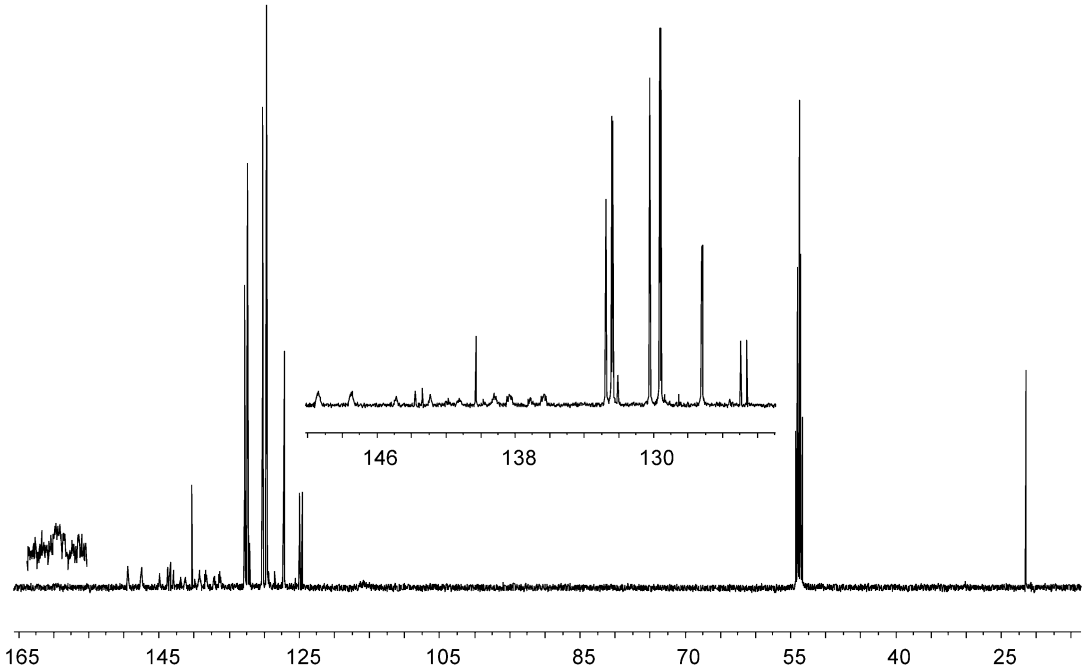
^{1}H,^{13}C GHMBC (500 MHz / 126 MHz, 299 K, CD_2Cl_2)**:** $\delta^{1}H$ / $\delta^{13}C$ = 7.56 / 132.4, 129.6 (*p*–Ph / *o*–, *m*–Ph), 7.40 / 132.8, 124.8 (*m*–Ph / *p*–, *i*–Ph), 7.07 / 132.0, 21.5 (*m*–Tol / *i*–Tol, CH_3^{Tol}), 6.99 / 143.6, 140.3 (*o*–Tol / =C^{P}, *p*–Tol), 2.33 / 140.3, 130.2 (CH_3^{Tol} / *p*–, *m*–Tol).



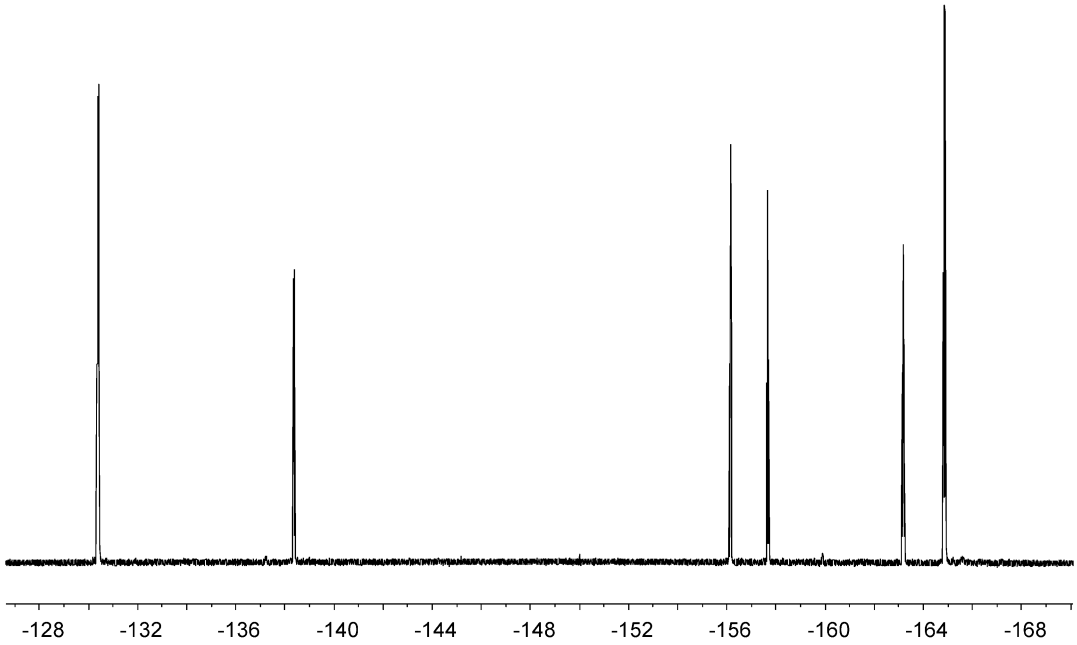
^{1}H NMR (500 MHz, 299 K, CD_2Cl_2) of **3a**.



$^{31}P\{^1H\}$ NMR (202 MHz, 299 K, CD_2Cl_2) and $^{11}B\{^1H\}$ NMR (160 MHz) of **3a**.

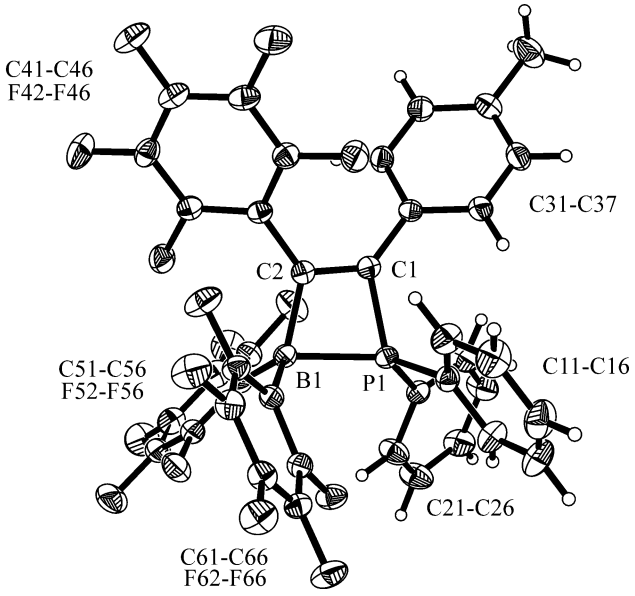


$^{13}C\{^1H\}$ NMR (126 MHz, 299 K, CD_2Cl_2) of **3a**.

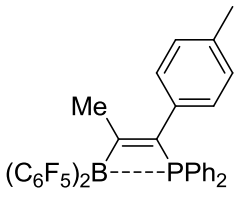


^{19}F NMR (470 MHz, 299 K, CD_2Cl_2) of **3a**.

X-Ray crystal structure analysis of **3a.** formula $C_{39}H_{17}BF_{15}P$, M = 812.31, colourless crystal, 0.33 x 0.33 x 0.27 mm, a = 9.9184(2), b = 20.5507(4), c = 17.2472(4) Å, β = 101.677(2)°, V = 3442.74(13) Å^3, ρ_{calc} = 1.567 gcm^{-3}, μ = 0.192 mm^{-1}, empirical absorption correction (0.939 ≤ T ≤ 0.950), Z = 4, monoclinic, space group $P2_1/n$ (No. 14), λ = 0.71073 Å, T = 223(2) K, ω and φ scans, 32085 reflections collected ($\pm h$, $\pm k$, $\pm l$), [(sinθ)/λ] = 0.66 Å^{-1}, 6016 independent (R_{int} = 0.049) and 5230 observed reflections [I>2σ(I)], 506 refined parameters, R = 0.042, wR^2 = 0.106, max. (min.) residual electron density 0.25 (-0.25) e.Å^{-3}, hydrogen atoms calculated and refined as riding atoms.



Synthesis of 3b.



Diphenyl(*p*-tolylethinyl)phosphane (**1**) (0.212 g, 0.704 mmol) and methyl(pentafluorophenyl)borane (**2b**) (0.254 g, 0.704 mmol) were stirred for 10 h at 100 ºC. Subsequently the solvent was removed and the residue was washed twice with pentane (15 ml) and all volatiles were removed *in vacuo* to yield **3b** (0.122 g, 0.184 mmol, 26%) as a light orange solid. Crystals suitable for X-ray crystal structure analysis were grown by slow evaporation of a dichloromethane solution of **3b** at –36 °C. **Anal. Calc.** for $C_{34}H_{20}BF_{10}P$: C, 61.85, H, 3.05. Found: C, 61.71, H, 3.04. **IR** (KBr): $\tilde{\nu}$ / cm^{-1} = 3060 (w), 2958 (m), 2923 (m), 285 (w), 1643 (s), 1515 (s), 1463 (s), 1281 (m), 1101 (s), 968 (m). **M.p.** (DSC): 159 °C. **Decomp.** (DSC): 287 °C.

^{1}H NMR (600 MHz, 299 K, CD_2Cl_2)**:** δ = 7.50 (m, 2H, *p*-Ph), 7.36 (m, 5H, *m*-Ph), 7.34 (m, 3H, *o*-Ph), 7.18 (m, 2H, *o*-Tol), 7.14 (m, 2H, *m*-Tol), 2.40 (m, 3H, CH_3), 2.33 (s, 3H, CH_3^{Tol}).

^{13}C{^{1}H} NMR (151 MHz, 299 K, CD_2Cl_2)**:** δ = 182.6 (br, ^{B}C=), 148.2 (dm, $^{1}J_{FC}$ ~ 240 Hz, C_6F_5), 140.0 (dm, $^{1}J_{FC}$ ~ 250 Hz, C_6F_5), 138.4 (d, $^{5}J_{PC}$ = 1.1 Hz, *p*-Tol), 137.3 (dm, $^{1}J_{FC}$ ~ 250 Hz, C_6F_5), 132.5 (d, $^{1}J_{PC}$ = 57.1 Hz, =C^{P}), 132.4 (d, $^{2}J_{PC}$ = 9.3 Hz, *o*-Ph), 132.1 (d, $^{2}J_{PC}$ = 4.4 Hz, *i*-Tol), 132.0 (d, $^{4}J_{PC}$ = 3.0 Hz, *p*-Ph), 129.8 (*m*-Tol), 129.3 (d^{3}, J_{PC} = 10.5 Hz, *m*-Ph), 128.2 (d, $^{3}J_{PC}$ = 5.7 Hz, *o*-Tol), 126.4 (d, $^{1}J_{PC}$ = 40.1 Hz, *i*-Ph), 116.6 (br, *i*-BC_6F_5), 21.4 (CH_3^{Tol}), 20.7 (dm, $^{3}J_{PC}$ = 44.1 Hz, CH_3).

^{19}F NMR (564 MHz, 299 K, CD_2Cl_2)**:** δ = -130.4 (m, 2F, *o*-BC_6F_5), -158.7 (tm, $^{3}J_{FF}$ = 20.1 Hz, 1F, *p*-BC_6F_5), -165.1 (m, 2F, *m*-BC_6F_5) [$\Delta\delta^{B}$(m, p) = 6.4].

^{11}B{^{1}H} NMR (192 MHz, 299 K, CD_2Cl_2)**:** δ = -7.6 ($\nu_{1/2}$ ~ 200 Hz).

^{31}P{^{1}H} NMR (243 MHz, 299 K, CD_2Cl_2)**:** δ = 12.3 ($\nu_{1/2}$ ~ 100 Hz).

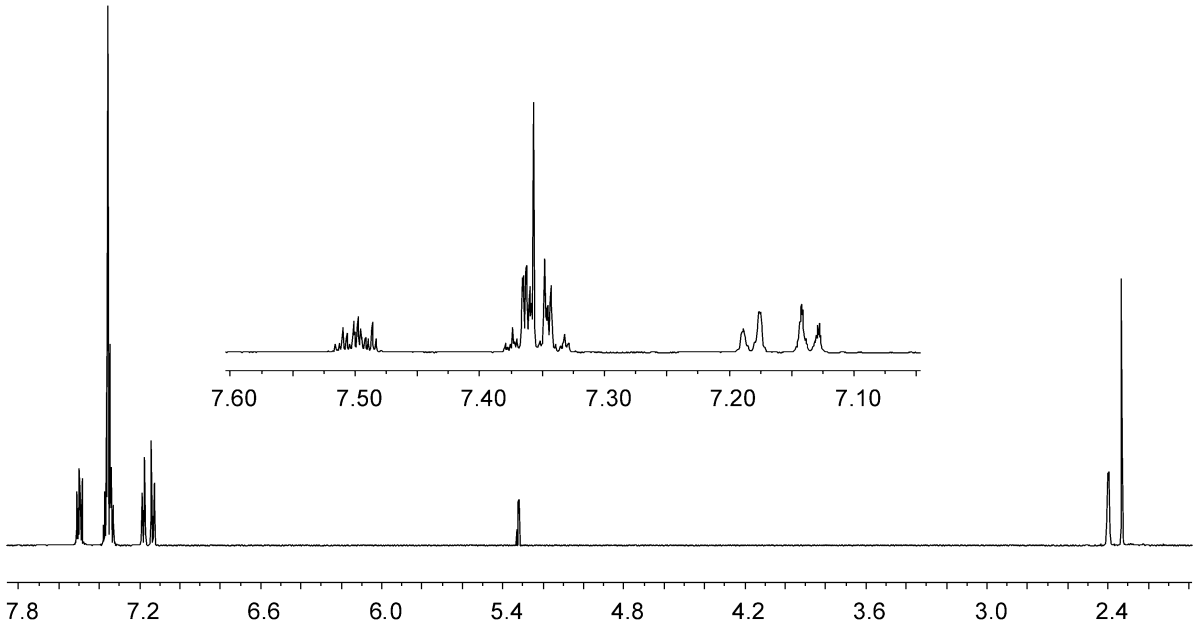
TOCSY (600 MHz, 299 K, CD_2Cl_2): $\delta^{1}H_{irr.}$ / $\delta^{1}H_{res.}$ = 7.50 / 7.36, 7.34 (*p*-Ph / *m*-, *o*-Ph), 7.18 / 7.14, 2.33 (*o*-Tol / *m*-Tol, CH_3^{Tol}).

NOE (600 MHz, 299 K, CD_2Cl_2)**:** $\delta^{1}H_{irr.}$ / $\delta^{1}H_{res.}$ = 7.50 / 7.36 (*p*-Ph / *m*-Ph), 7.36 / 7.50, 7.34 (m-Ph / *p*-, *o*-Ph), 7.18 / 7.14 (*o*-Tol / *m*-Tol), 7.14 / 7.18, 2.33 (*m*-Tol / *o*-Tol, CH_3^{Tol}), 2.40 / 7.34, 7.19 (CH_3 / *o*-Ph, *o*-Tol), 2.33 / 7.14 (CH_3^{Tol} / *m*-Tol).

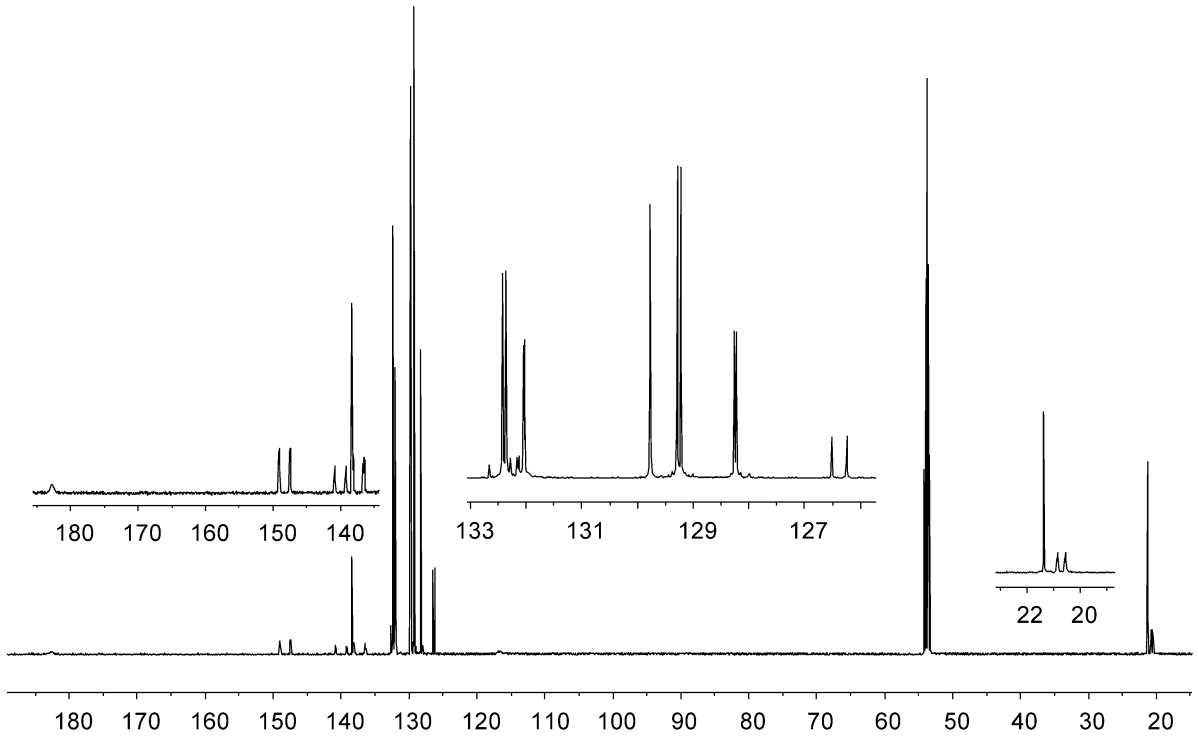
^{1}H,^{1}H COSY (600 MHz / 600 MHz, 299 K, CD_2Cl_2): $\delta^{1}H$ / $\delta^{1}H$ = 7.50 / 7.36, 7.34 (*p*-Ph / *m*-, *o*-Ph), 7.18 / 7.14 (*o*-Tol / *m*-Tol), 7.14 / 7.18, 2.33 (*m*-Tol / *o*-Tol, CH_3^{Tol}), 2.33 / 7.14 (CH_3^{Tol} / *m*-Tol).

^{1}H,^{13}C GHSQC (600 MHz / 151 MHz, 299 K, CD_2Cl_2)**:** $\delta^{1}H$ / $\delta^{13}C$ = 7.50 / 132.0 (*p*-Ph), 7.36 / 129.3 (*m*-Ph), 7.34 / 132.4 (*o*-Ph), 7.18 / 128.2 (*o*-Tol), 7.14 / 129.8 (*m*-Tol), 2.40 / 20.7 (CH_3), 2.33 / 21.4 (CH_3^{Tol}).

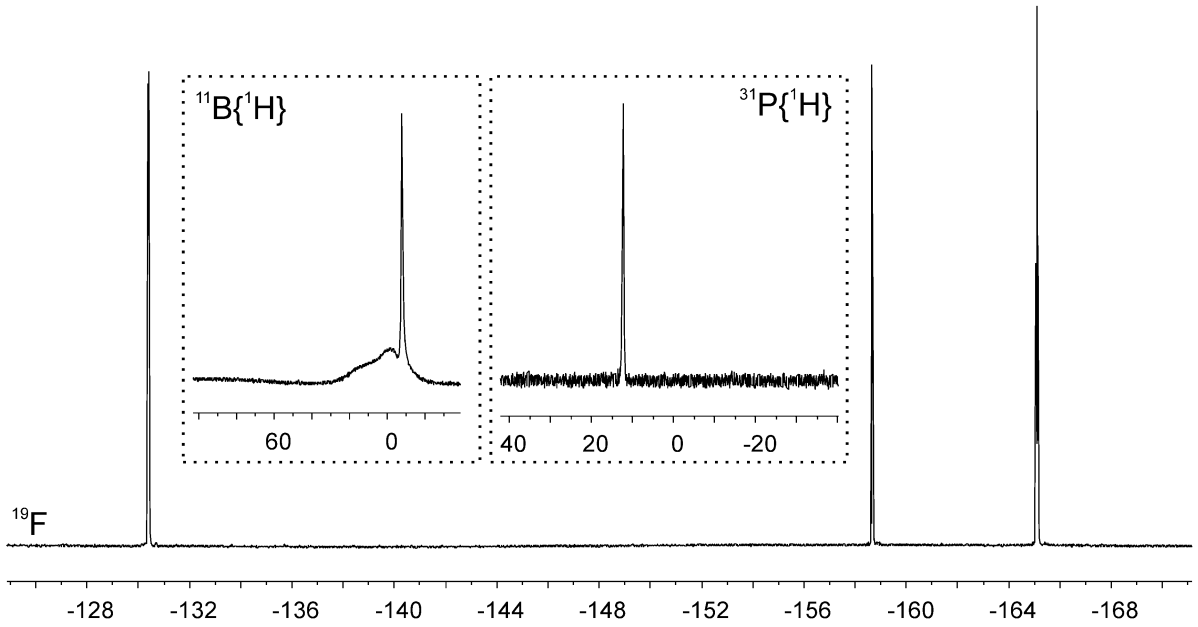
^{1}H,^{13}C GHMBC (600 MHz / 151 MHz, 299 K, CD_2Cl_2): δ^{1}H / δ^{13}C = 7.50 / 132.4 (*p*-Ph / *o*-Ph), 7.36 / 132.0, 126.4 (*m*-Ph / *p*-, *i*-Ph), 7.18 / 138.4 (*o*-Tol / *p*-Tol), 7.14 / 132.1, 21.4 (*m*-Tol / *i*-Tol, CH_3^{Tol}), 2.40 / 182.6, 132.5 (CH_3 / ^{B}C=, =C^{P}), 2.33 / 138.4, 129.8 (CH_3^{Tol} / *i*-, *m*-Tol).



^{1}H NMR (600 MHz, 299 K, CD_2Cl_2) of **3b**.

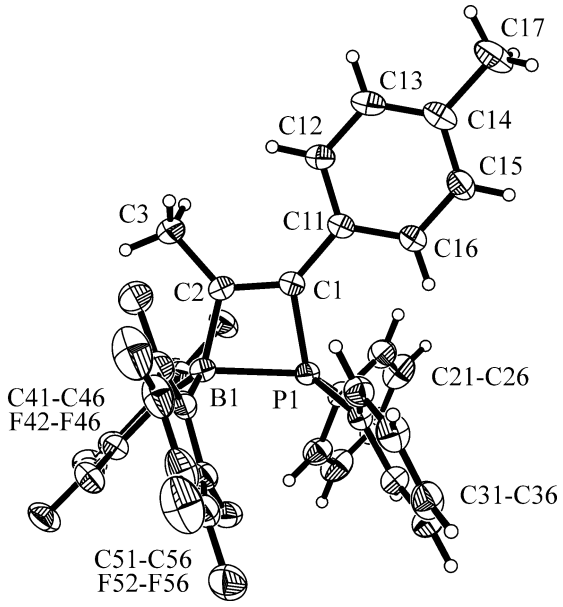


^{13}C{^{1}H} NMR (151 MHz, 299 K, CD_2Cl_2) of **3b**.

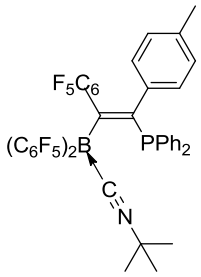


^{19}F NMR (564 MHz, 299 K, CD_2Cl_2), $^{31}P\{^1H\}$ NMR (243 MHz, 299 K, CD_2Cl_2) and $^{11}B\{^1H\}$ NMR (192 MHz, 299 K, CD_2Cl_2) of **3b**.

X-Ray crystal structure analysis of **3b.** formula $C_{34}H_{20}BF_{10}P$, M = 660.28, colourless crystal, 0.15 x 0.04 x 0.02 mm, a = 8.3314(3), b = 23.9142(9), c = 14.7962(9) Å, β = 90.618(4)°, V = 2947.8(2) Å^3, ρ_{calc} = 1.488 gcm^{-3}, μ = 1.624 mm^{-1}, empirical absorption correction (0.792 ≤ T ≤ 0.968), Z = 4, monoclinic, space group $P2_1/c$ (No. 14), λ = 1.54178 Å, T = 223(2) K, ω and φ scans, 27732 reflections collected ($\pm h$, $\pm k$, $\pm l$), [(sinθ)/λ] = 0.60 Å^{-1}, 5156 independent (R_{int} = 0.064) and 3991 observed reflections [I>2σ(I)], 417 refined parameters, R = 0.050, wR^2 = 0.136, max. (min.) residual electron density 0.26 (-0.33) e.Å^{-3}, hydrogen atoms calculated and refined as riding atoms.



Reaction of 3a with *tert*-butyl isocyanide: mixture of 4a and 3a + tBu-NC.



Compound **3a** (0.100 g, 0.123 mmol) was dissolved in dichloromethane (10 ml) and *tert*-butylisocyanide (15.5 mg, 0.185 mmol) was added. After stirring for one hour at room temperature, solvent and all volatiles were removed *in vacuo*. A light yellow solid (**4a**) (65.3 mg, 0.073 mmol, 59%) was isolated. Crystals suitable for X-ray diffraction were grown by slow evaporation of a dichloromethan solution of the light yellow solid (**4a**) at -36 °C. **Anal. Calc.** for $C_{44}H_{26}BF_{15}NP$: C, 59.02; H, 2.93; N, 1.56. Found: C, 59.07; H, 2.85; N, 1.46. **HRMS:** Calc. for $C_{44}H_{26}BF_{15}NPH$: 896.17366. Found: 896.17638. **IR** (KBr): $\tilde{\nu}$ / cm^{-1} = 3057 (br m), 2926 (w), 2359 (w), 2282 (m), 1646 (m), 1518 (s), 1100 (s), 976 (s), 738 (m).

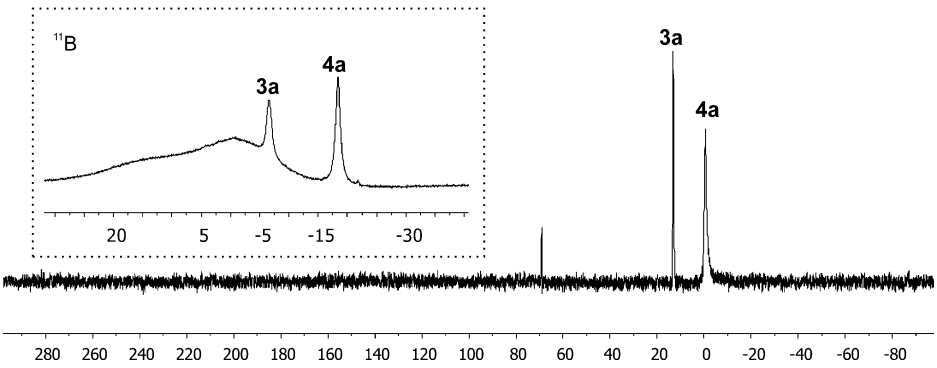
A mixture of **4a** and **3a** + *tert*-butylisocyanide [299K: 63(**4a**):37(**3a**) (^{1}H NMR CH_3^{Tol})] was found after dissolving **4a** in CD_2Cl_2:

^{1}H NMR (600 MHz, 299 K, CD_2Cl_2) **4a**: δ = 7.34 (br m, 2H, *p*-Ph), 7.28 (br m, 4H, *m*-Ph), 7.23 (br m, 4H, *o*-Ph), 6.64 (br d, $^3J_{HH}$ = 7.8 Hz, 2H, *m*-Tol), 6.50 (br d, $^3J_{HH}$ = 7.8 Hz, 2H, *o*-Tol), 2.09 (s, 3H, CH_3^{Tol}), 1.22 (s, 9H, CH_3).

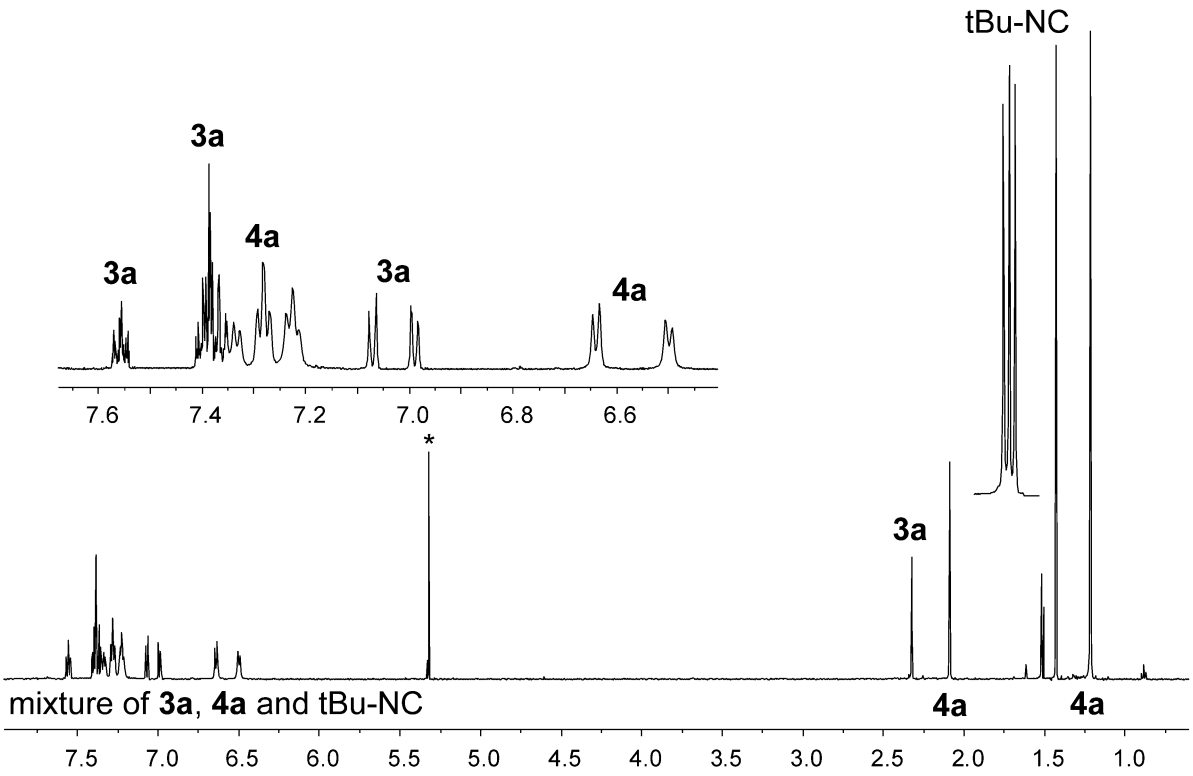
^{19}F NMR (564 MHz, 299 K, CD_2Cl_2) **4a**: δ = -129.9 (br, 4F, *o*-BC_6F_5), -138.1 (br, 2F, *o*-C_6F_5), -157.8 (m, 2F, *p*-BC_6F_5), -158.3 (m, 1F, *p*-C_6F_5), -164.3 (m, 4F, *m*-BC_6F_5), -164.8 (m, 2F, *m*-C_6F_5) [$\Delta\delta^B$(m, p) = 6.5].

^{11}B{^{1}H} NMR (192 MHz, 299 K, CD_2Cl_2): δ = -18.4 ($\nu_{1/2}$ ~ 200 Hz, **4a** [65%]), -6.6 ($\nu_{1/2}$ ~ 250 Hz, **3a** [35%]).

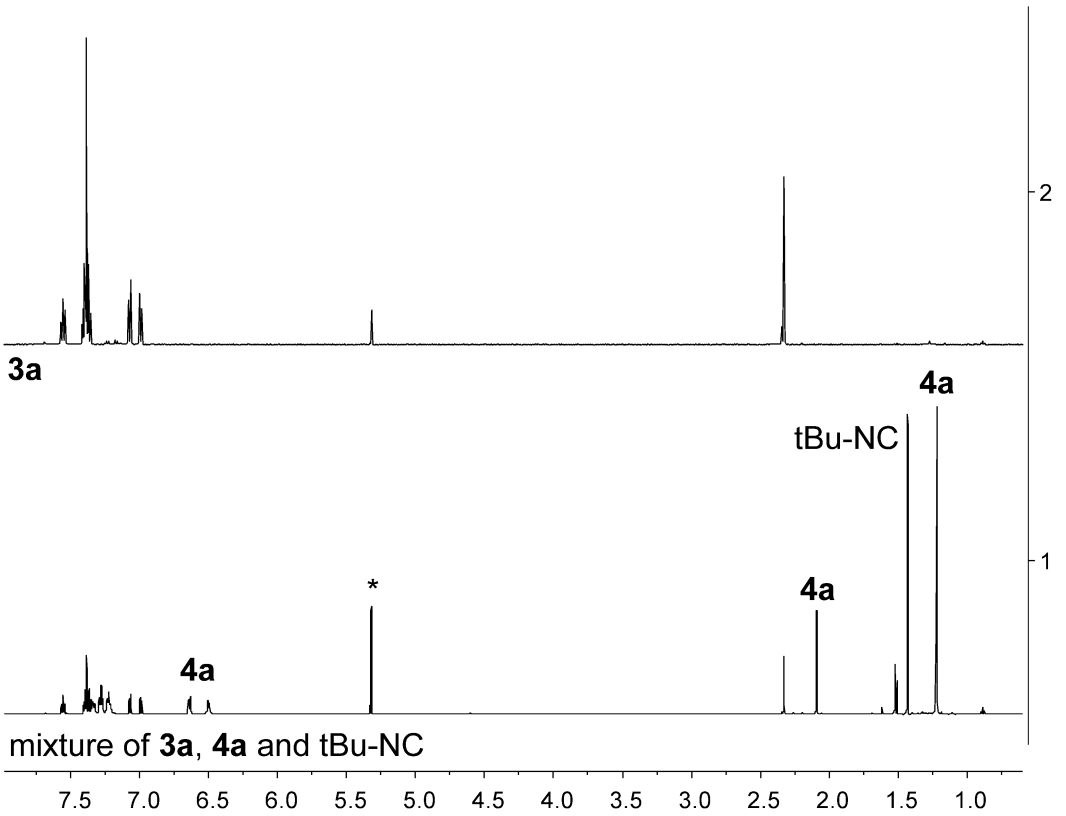
^{31}P{^{1}H} NMR (243 MHz, 299 K, CD_2Cl_2): δ = 13.1 ($\nu_{1/2}$ ~ 100 Hz, **3a** [32%]), -0.5 ($\nu_{1/2}$ ~ 300 Hz, **4a** [68%])



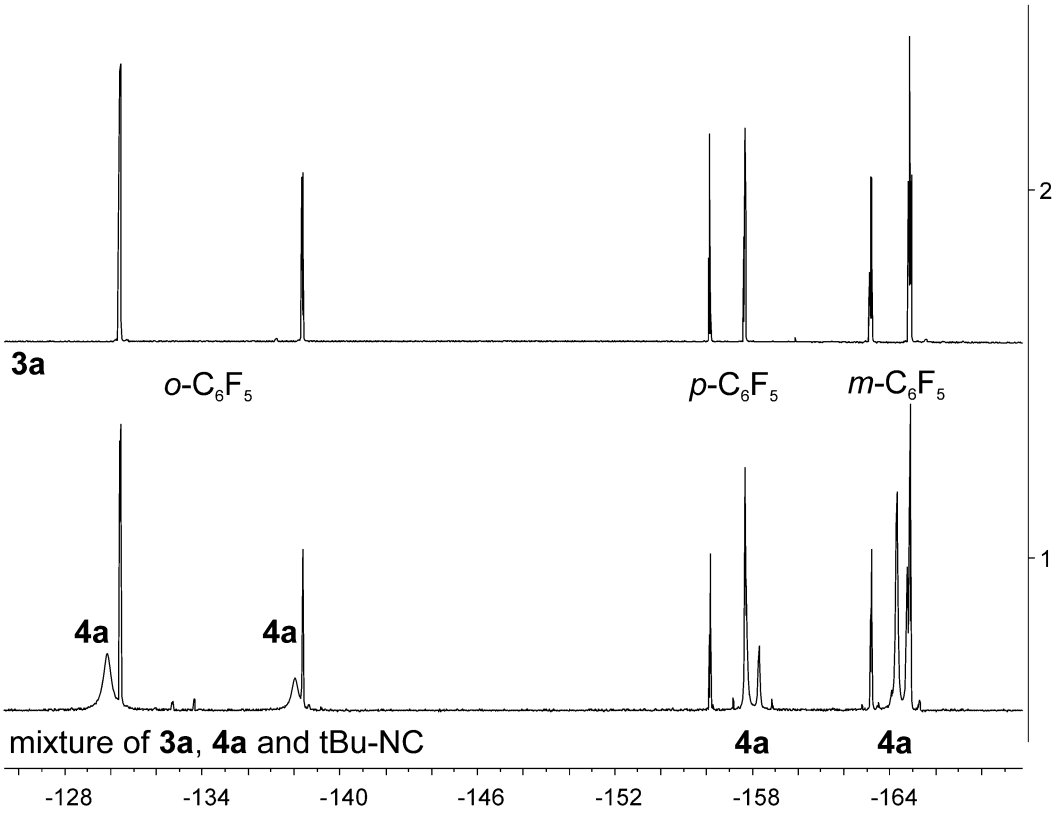
^{31}P{^{1}H} NMR (243 MHz, 299 K, CD_2Cl_2) and ^{11}B NMR (192 MHz) spectrum after dissolving **4a** in CD_2Cl_2.



^{1}H NMR (600 MHz, 299 K, CD_2Cl_2 (*)) spectrum after dissolving **4a** in CD_2Cl_2.

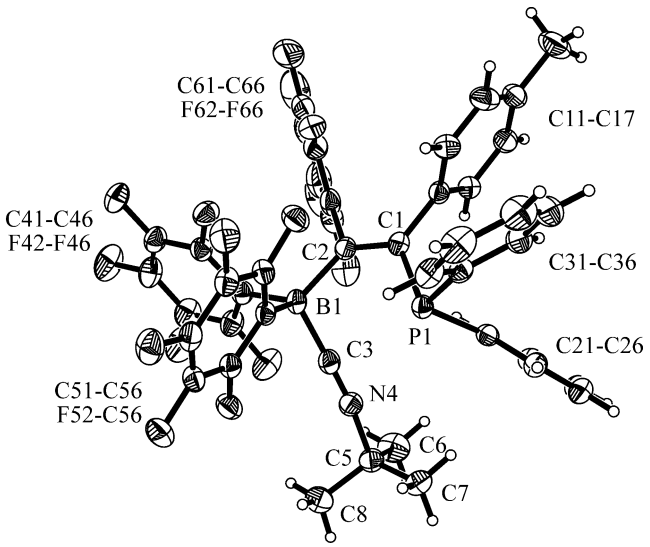


1: ^{1}H NMR (600 MHz, 299 K, CD_2Cl_2 (*)) spectrum after dissolving **4a** in CD_2Cl_2.
2: ^{1}H NMR (500 MHz, 299 K, CD_2Cl_2) spectrum of **3a**.

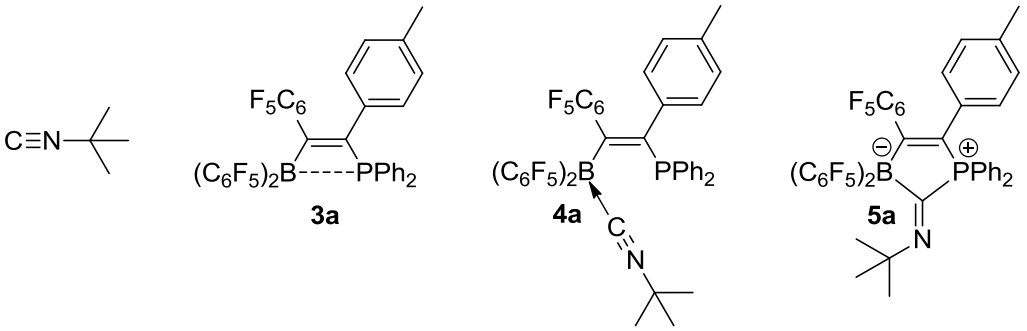


1: ^{19}F NMR (564 MHz, 299 K, CD_2Cl_2) spectrum after dissolving **4a** in CD_2Cl_2.
2: ^{19}F NMR (470 MHz, 299 K, CD_2Cl_2) spectrum of **3a**.

X-Ray crystal structure analysis of **4a.** formula $C_{44}H_{26}BF_{15}NP$, M = 895.44, colourless crystal, 0.30 x 0.17 x 0.07 mm, a = 13.8351(8), b = 11.5678(8), c = 30.5690(30) Å, β = 101.896(3)°, V = 4787.2(6) Å^3, ρ_{calc} = 1.242 gcm^{-3}, μ = 1.298 mm^{-1}, empirical absorption correction (0.696 ≤ T ≤ 0.914), Z = 4, monoclinic, space group $P2_1/c$ (No. 14), λ = 1.54178 Å, T = 223(2) K, ω and φ scans, 33705 reflections collected (±h, ±k, ±l), [(sinθ)/λ] = 0.60 Å^{-1}, 8139 independent (R_{int} = 0.059) and 6278 observed reflections [I>2σ(I)], 564 refined parameters, R = 0.070, wR^2 = 0.207, max. (min.) residual electron density 0.73 (-0.37) e.Å^{-3}, hydrogen atoms calculated and refined as riding atoms.



Dissolving **4a** in CD_2Cl_2 at rt and cooling the obtained mixture of (**3a** + *tert*-butylisocyanide)/**4a** to 213K resulted in a (**3a** + *tert*-butylisocyanide)/**4a**/**5a** mixure [213K: 11(**3a**):72(**4a**):17(**5a**) (**^{1}H NMR** CH_3^{Tol})]:

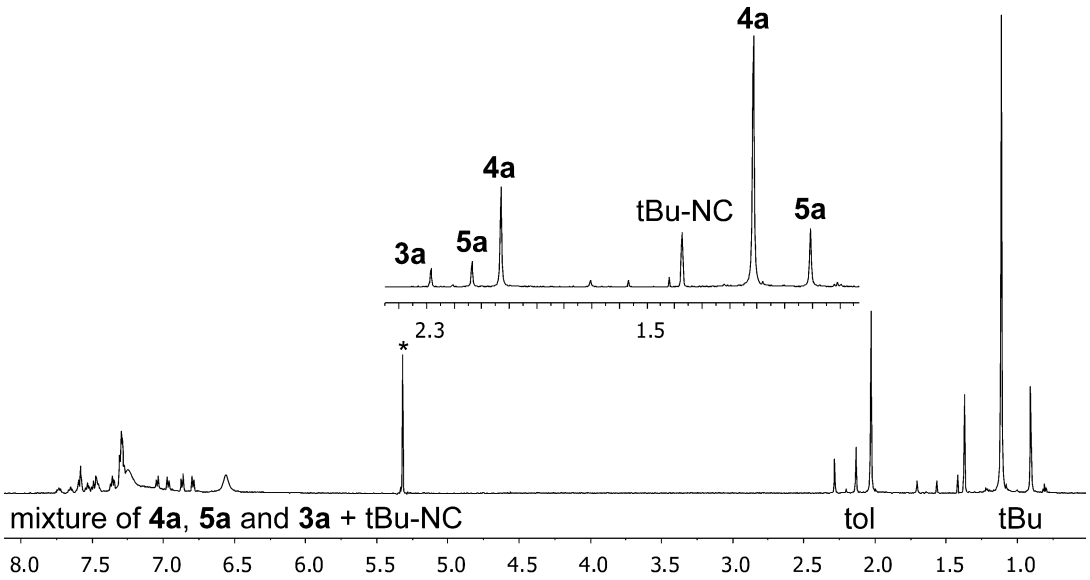


Selected resonances: **^{1}H NMR** (600 MHz, 213 K, CD_2Cl_2) **3a**: δ = 2.29 (s, 3H, CH_3^{Tol}) + *tert*-butylisocyanide: δ = 1.38 (s, 9H, CH_3). **4a**: δ = 2.03 (s, 3H, CH_3^{Tol}), 1.12 (s, 9H, CH_3). **5a:** δ = 2.14 (s, 3H, CH_3^{Tol}), 0.91 (s, 9H, CH_3).

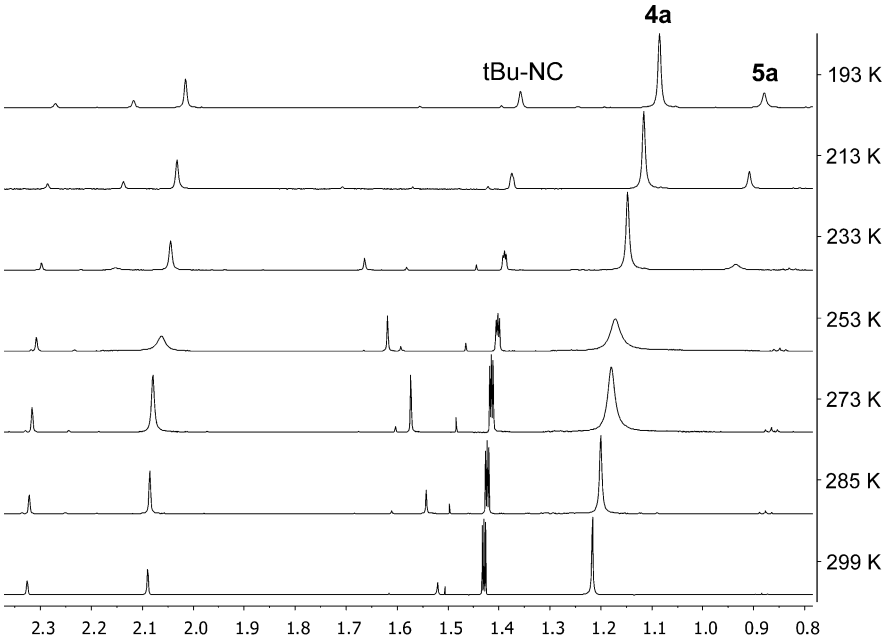
^{13}C{^{1}H} NMR (151 MHz, 213 K, CD_2Cl_2): **3a** + *tert*-butylisocyanide: δ = 21.1 (CH_3^{Tol}), 54.3, 30.2 (tBu). **4a**: δ = 20.6 (CH_3^{Tol}), 60.4, 27.3 (tBu). **5a:** δ = 20.8 (CH_3^{Tol}), 62.2 (d, $^3J_{PC}$ = 37.7 Hz), 28.2 (tBu).

^{11}B{^{1}H} NMR (192 MHz, 213 K, CD_2Cl_2): **3a**: δ = -7.3 (very broad). **5a**: δ = -13.3 (d, J_{PB} ~ 45 Hz, $v_{1/2}$ ~ 20 Hz). **4a**: δ = -19.4 ($v_{1/2}$ ~ 560 Hz).

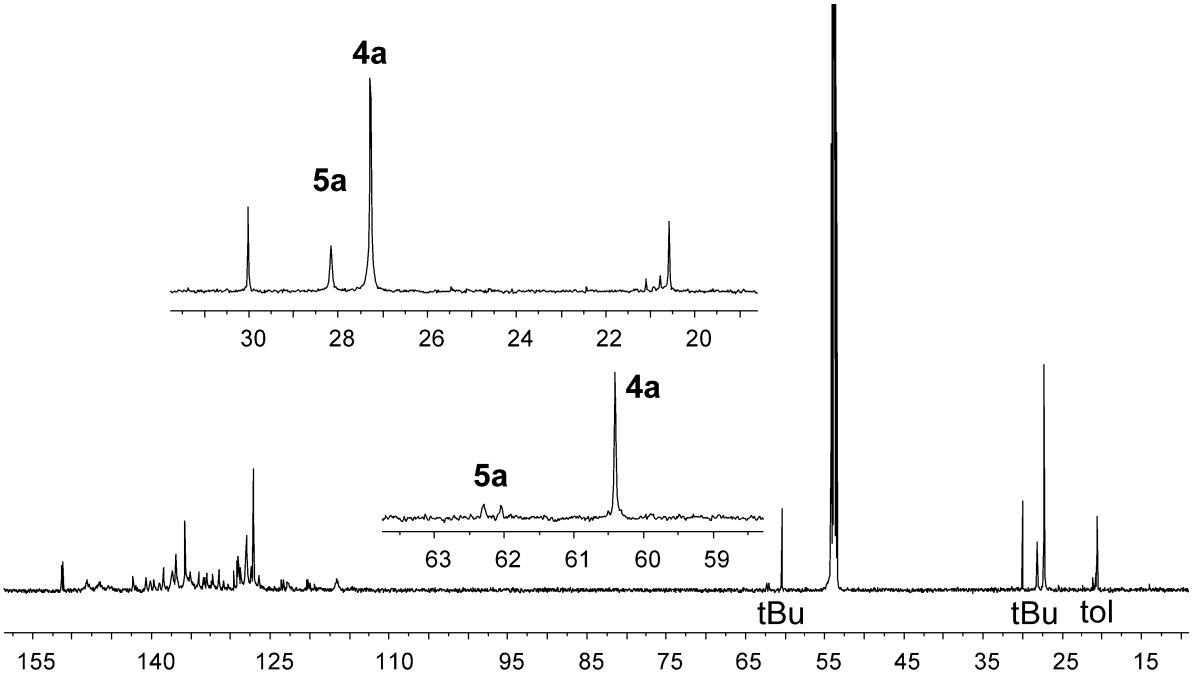
^{31}P{^{1}H} NMR (243 MHz, 213 K, CD_2Cl_2): **3a**: δ = 12.2 ($v_{1/2}$ ~ 50 Hz, [8%]). **5a**: δ = 10.2 (1:1:1:1 q, J_{PB} ~ 45 Hz, $v_{1/2}$ ~ 20 Hz, [17%]). **4a**: δ = -3.1 ($v_{1/2}$ ~ 20 Hz, [74%]).



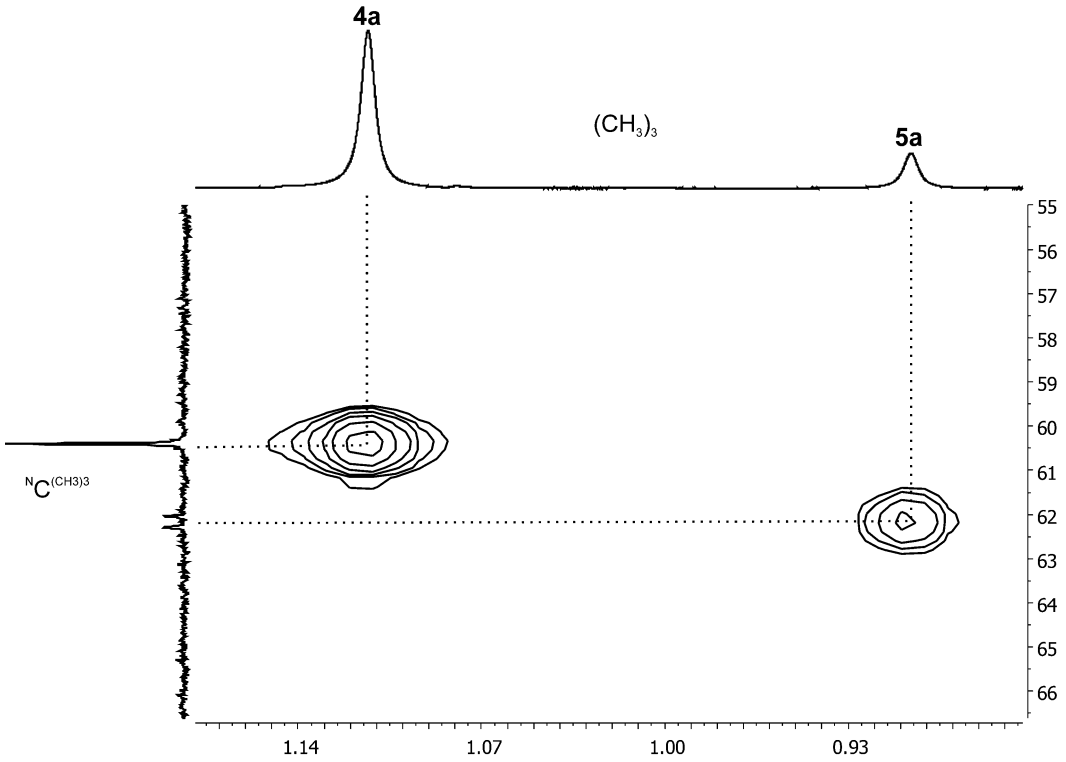
^{1}H NMR (600 MHz, 213 K, CD_2Cl_2 (*)) spectrum after dissolving **4a** in CD_2Cl_2.



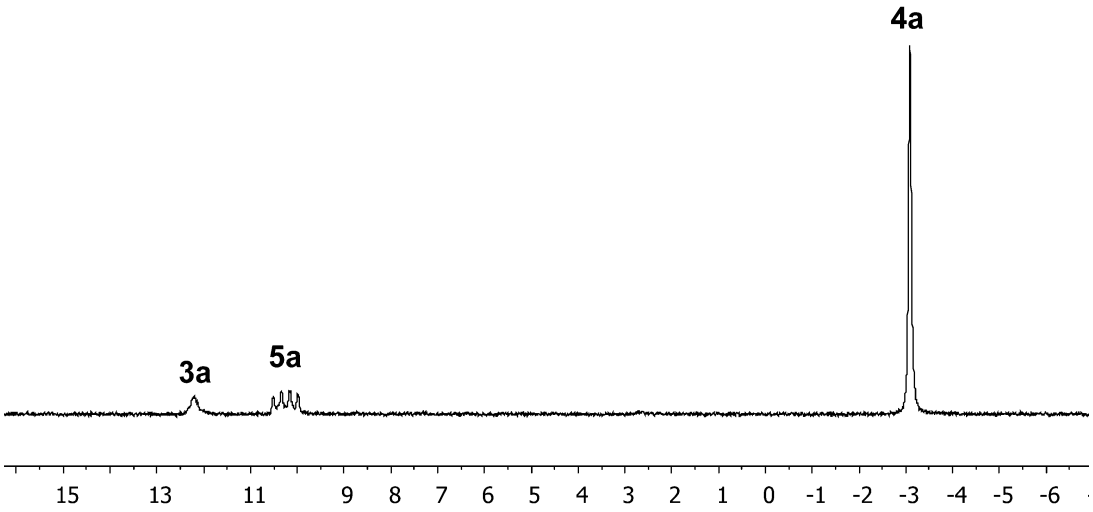
^{1}H NMR (600 MHz, CD_2Cl_2) spectra after dissolving **4a** in CD_2Cl_2.



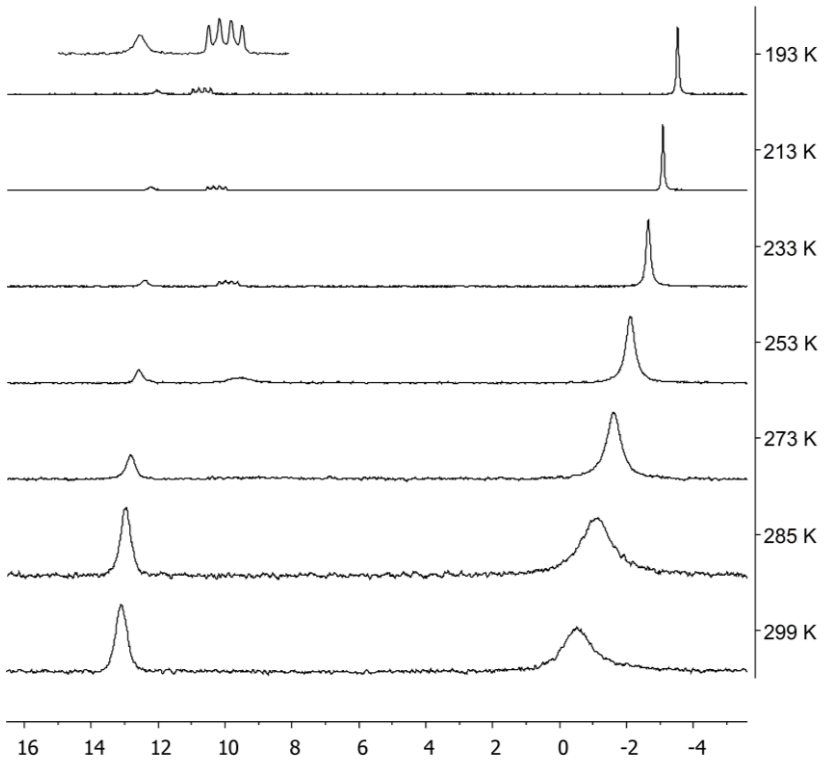
$^{13}C\{^{1}H\}$ NMR (151 MHz, 213 K, CD_2Cl_2) spectrum after dissolving **4a** in CD_2Cl_2.



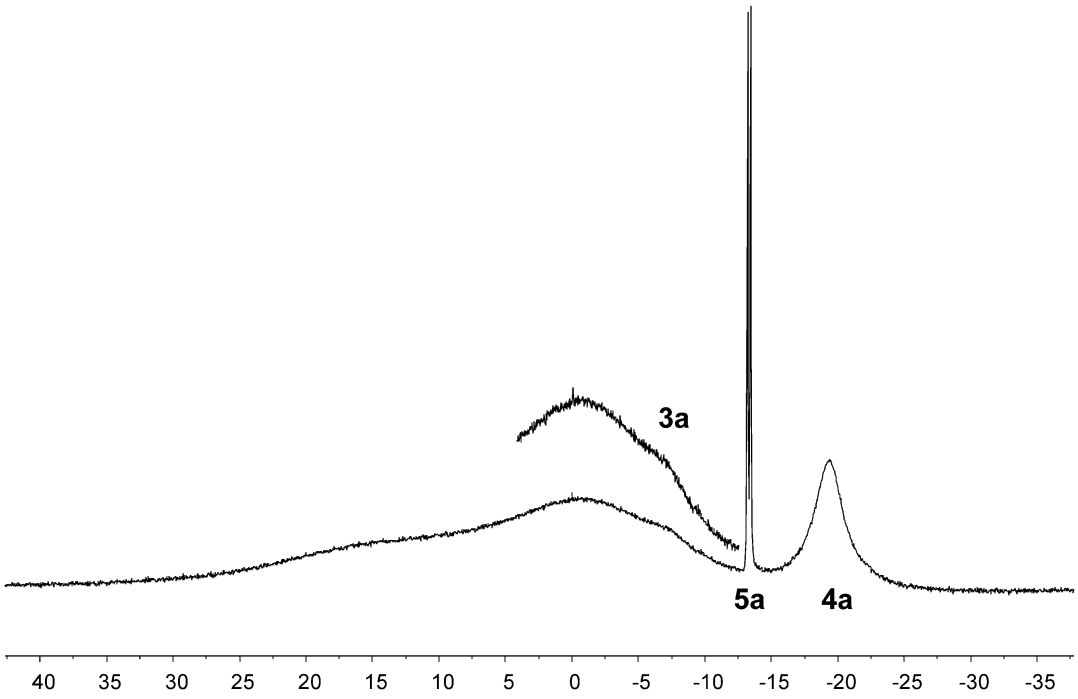
$^{1}H,^{13}C$ GHMBC (600 MHz / 151 MHz, 213 K, CD_2Cl_2) spectrum after dissolving **4a** in CD_2Cl_2.



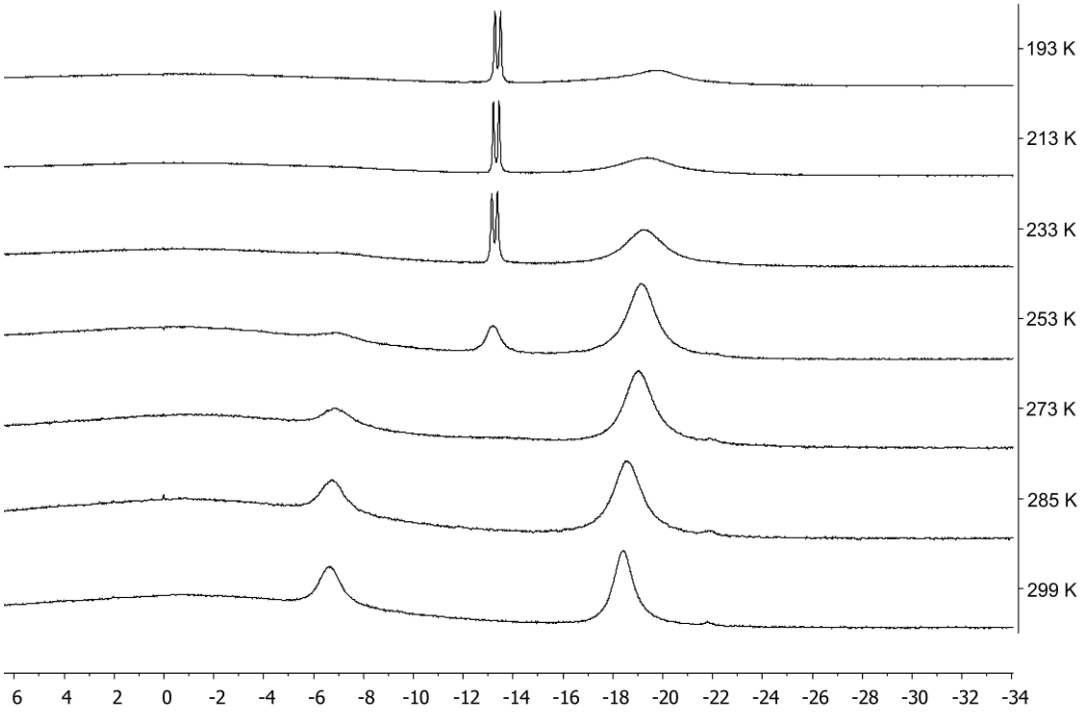
$^{31}P\{^{1}H\}$ NMR (243 MHz, 213 K, CD_2Cl_2) spectrum after dissolving **4a** in CD_2Cl_2.



$^{31}P\{^{1}H\}$ NMR (243 MHz, CD_2Cl_2) spectra after dissolving **4a** in CD_2Cl_2.



$^{11}B\{^{1}H\}$ NMR (192 MHz, 213 K, CD_2Cl_2) spectrum after dissolving **4a** in CD_2Cl_2.



$^{11}B\{^{1}H\}$ NMR (192 MHz, CD_2Cl_2) spectra after dissolving **4a** in CD_2Cl_2.

Reaction of 3b with *tert*-butyl isocyanide: mixture of 5b and 3b + tBu-NC.

Compound **3b** (0.100 g, 0.151 mmol) was dissolved in dichloromethane (10 ml) and *tert*-butylisocyanide (13.5 mg, 0.162 mmol) was added. After stirring for one hour at room temperature, solvent and all volatiles were removed *in vacuo*. A yellow solid (**4b**) (60.5 mg, 0.081 mmol, 54%) was isolated. Crystals suitable for X-ray diffraction were grown by slow evaporation of a dichloromethan solution of **4b** at -36 °C. **Anal. Calc.** for $C_{39}H_{29}BF_{10}NP$: C, 63.01; H, 3.93; N, 1.88. Found: C, 62.25; H, 3.85; N, 1.70. **HRMS:** Calc. for $C_{39}H_{29}BF_{10}NPH$: 744.20457. Found: 744.20499. **IR** (KBr): $\tilde{\nu}$ / cm^{-1} = 3050, 2990, 2871 (br m), 2276 (s), 1890 (w), 1643 (m), 1517 (s), 1374 (m), 1095 (s), 975 (s). **M.p.** (DSC): 167 °C. **Decomp.** (DSC): 171 °C. In the ^{31}P CPMAS solid state NMR experiment only the resonance of adduct **4b** was detected δ = 1.3.

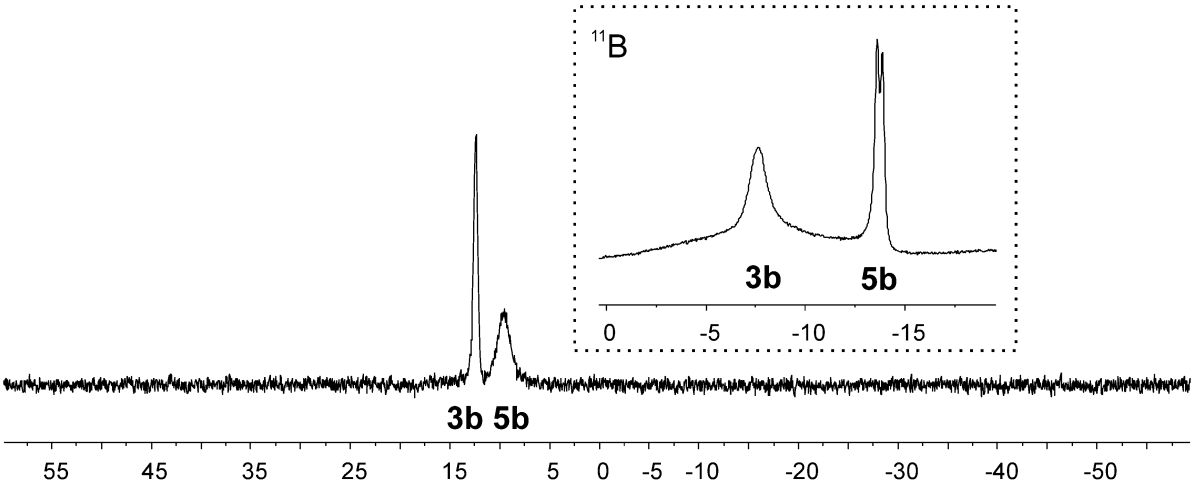
A mixture of **3b** + *tert*-butyl isocyanide and **5b** [299K: 55(**3b**):45(**5b**) (**^{1}H NMR** CH_3^{Tol})] was found after dissolving **4b** in CD_2Cl_2:

^{1}H NMR (500 MHz, 299 K, CD_2Cl_2) **5b**: δ = 7.58 (m, 2H, *p*-Ph), 7.44 (m, 8H, *m*,*o*-Ph), 7.00 (br d, $^{3}J_{HH}$ = 8.0 Hz, 2H, *m*-Tol), 6.67 (br d, $^{3}J_{HH}$ = 8.0 Hz, 2H, *o*-Tol), 2.26 (s, 3H, CH_3^{Tol}), 1.80 (br, 3H, $^{=}CH_3$),1.04 (s, 9H, CH_3). [**3b** + *tert*-butylisocyanide not listed].

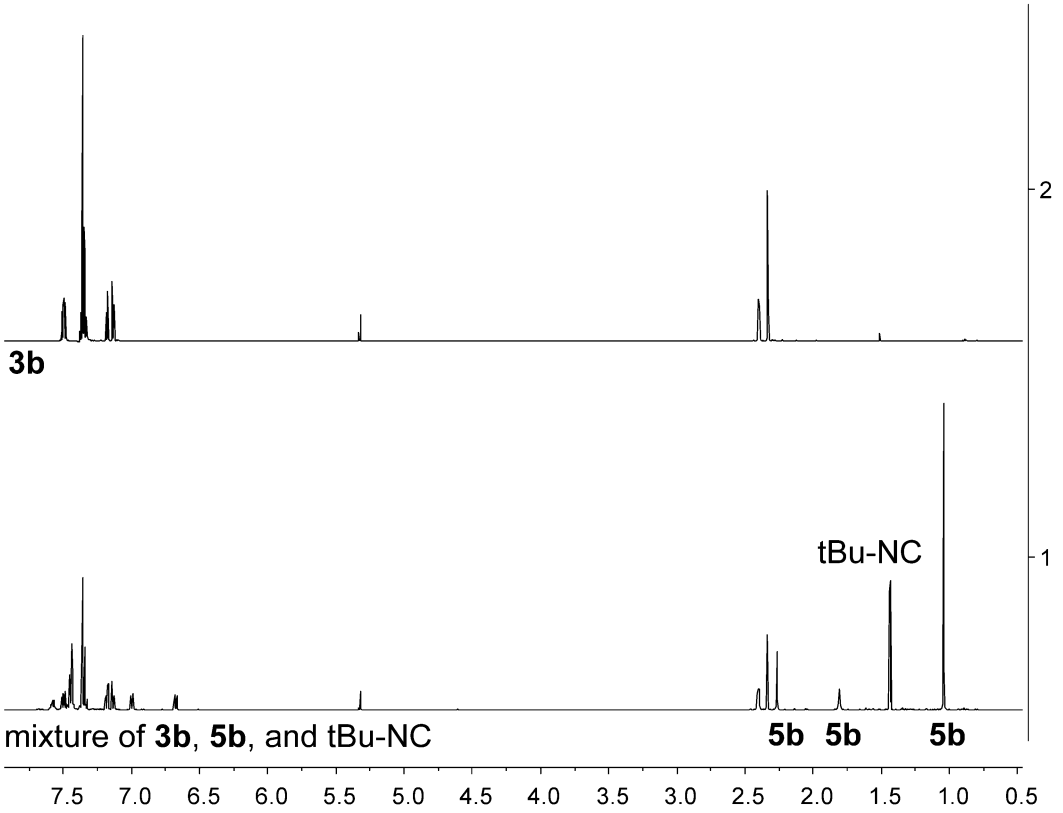
^{19}F NMR (470 MHz, 299 K, CD_2Cl_2) **5b**: δ = -128.4 (br, 2F, *o*-BC_6F_5), -160.4 (t, $^{3}J_{FF}$ = 20.3 Hz, 1F, *p*-BC_6F_5), -165.3 (br m, 2F, *m*-BC_6F_5), [$\Delta\delta^{B}$(m, p) = 4.9], [**3b** not listed].

^{11}B{^{1}H} NMR (160 MHz, 299 K, CD_2Cl_2): δ = -13.7 (d, J_{PB} ~ 41 Hz, $\nu_{1/2}$ ~ 50 Hz, **5b**), -7.6 ($\nu_{1/2}$ ~ 200 Hz, **3b**).

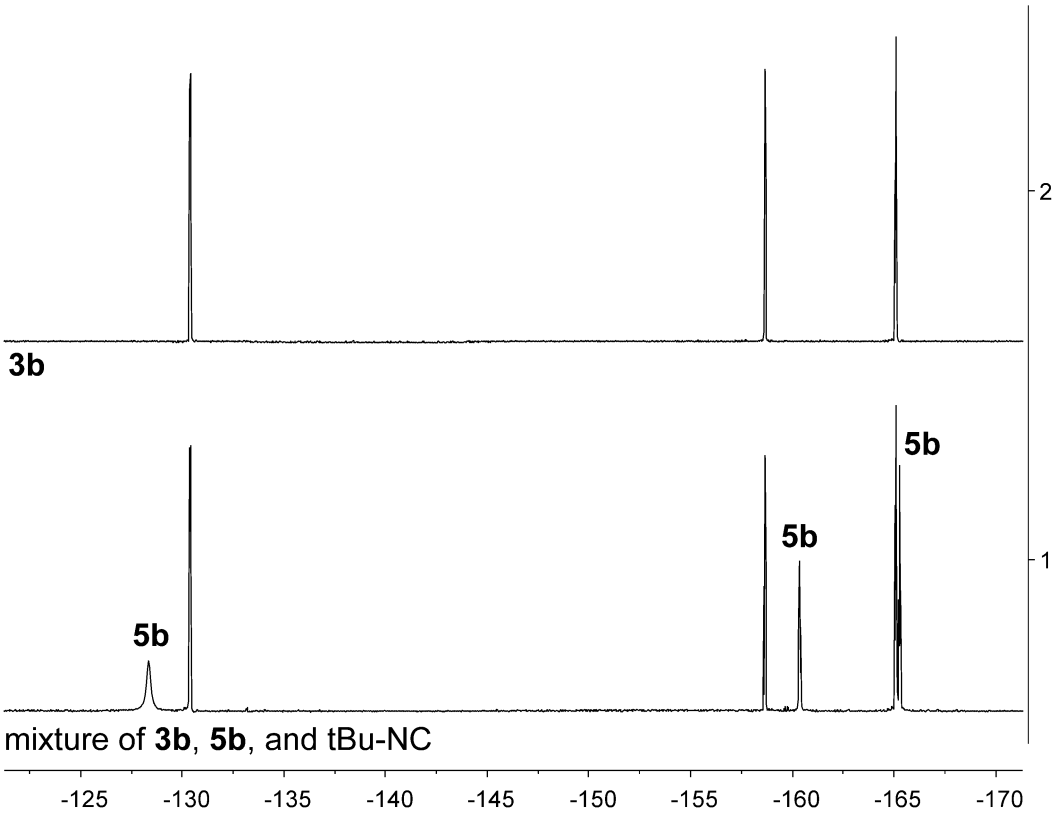
^{31}P{^{1}H} NMR (202 MHz, 299 K, CD_2Cl_2): δ = 12.4 ($\nu_{1/2}$ ~ 120 Hz, **3b** [54%]), 9.6 ($\nu_{1/2}$ ~ 370 Hz, **5b** [46%])



$^{31}P\{^{1}H\}$ NMR (202 MHz, 299 K, CD_2Cl_2) and $^{11}B\{^{1}H\}$ NMR (160 MHz, 299 K, CD_2Cl_2) spectrum after dissolving **4b** in CD_2Cl_2.

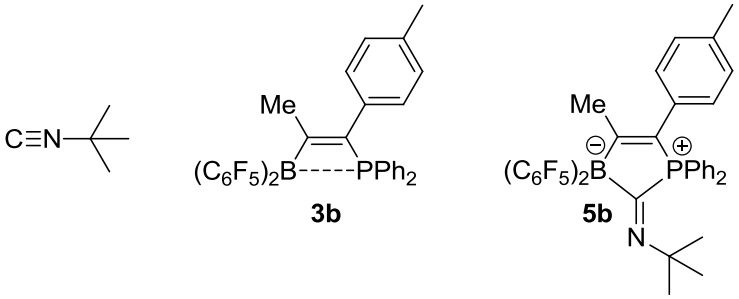


1: ^{1}H NMR (500 MHz, 299 K, CD_2Cl_2 (*)) spectrum after dissolving **4b** in CD_2Cl_2.
2: ^{1}H NMR (600 MHz, 299 K, CD_2Cl_2) spectrum of **3b**.



1: ^{19}F NMR (470 MHz, 299 K, CD_2Cl_2) spectrum after dissolving **4b** in CD_2Cl_2.
2: ^{19}F NMR (564 MHz, 299 K, CD_2Cl_2) spectrum of **3b**.

Dissolving **4b** in CD_2Cl_2 at rt and cooling the obtained mixture of (**3b** + *tert*-butylisocyanide)/**5b** to 193K resulted in a (**3b** + *tert*-butyl isocyanide)/**5b** mixure [193K: 33(**3b**):67(**5b**) (**^{1}H NMR** CH_3^{Tol})]:



^{1}H NMR (500 MHz, 193 K, CD_2Cl_2) **5b**: δ = 7.66 (m, 2H, *p*-Ph), 7.49 (m, 4H, *m*-Ph), 7.44 (m, 4H, *o*-Ph), 6.99 (br d, $^3J_{HH}$ = 8.0 Hz, 2H, *m*-Tol), 6.61 (br d, $^3J_{HH}$ = 8.0 Hz, 2H, *o*-Tol), 2.20 (s, 3H, CH_3^{Tol}), 1.78 (s, 3H, $^{=}CH_3$), 0.88 (s, 9H, CH_3), [**3b** + *tert*-butyl isocyanide not listed].

^{13}C{^{1}H} NMR (126 MHz, 193 K, CD_2Cl_2): δ = 193.4 (br, ^BC=), 191.1 (br, N=C), 137.0 (*p*-Tol), 133.2 (d, J_{PC} = 8.7 Hz, *o*-Ph), 132.7 (*p*-Ph), 131.1 (d, $^2J_{PC}$ = 11.0 Hz, *i*-Tol), 128.9 (*o*-Tol), 128.7 (*m*-Tol), 128.6 (d, $^2J_{PC}$ = 10.1 Hz, *m*-Ph), 123.6 (d, $^1J_{PC}$ = 76.7 Hz, *i*-Ph), 120.7 (d, $^1J_{PC}$ = 85.5 Hz, =C^P), 60.8 (d, $^3J_{PC}$ = 38.3 Hz, $^NC^{(CH3)3}$), 28.0 (CH_3), 20.6 (CH_3^{Tol}), 20.1 (br d, $^3J_{PC}$ = 16.3 Hz, $^{=}CH_3$), [C_6F_5 not listed, **3b** + *tert*-butyl isocyanide not listed].

^{19}F NMR (470 MHz, 193 K, CD_2Cl_2): **5b**: δ = -127.7 (m, 2F, *o*-C_6F_5), -159.7 (t, $^3J_{FF}$ = 21.3 Hz, 1F, *p*-C_6F_5), -164.4 (m, 2F, *m*-C_6F_5), [$\Delta\delta^B$(m, p) = 4.7]. **3b**: δ = -130.7 (m, 2F, *o*-C_6F_5), -157.7 (t, $^3J_{FF}$ = 21.0 Hz, 1F, *p*-C_6F_5), -164.3 (m, 2F, *m*-C_6F_5), [$\Delta\delta^B$(m, p) = 6.6].
(*ole129-3a_110112_193k_19f*)

^{11}B{^{1}H} NMR (160 MHz, 233 K, CD_2Cl_2): **3b**: δ = -8.3 (very broad). **5b**: δ = -13.5 (d, J_{PB} ~ 40 Hz, $v_{1/2}$ ~ 40 Hz).

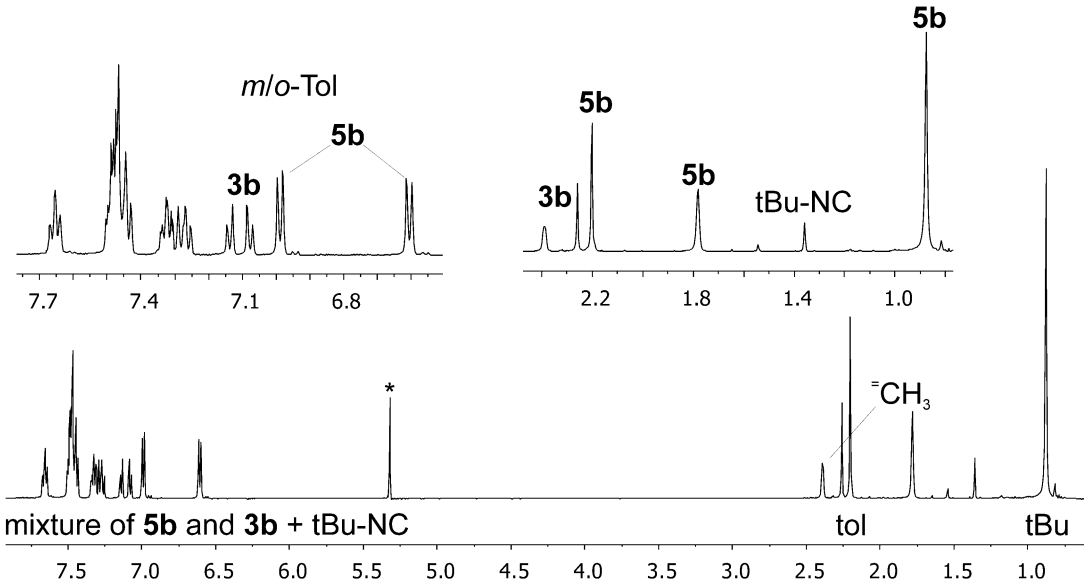
^{31}P{^{1}H} NMR (202 MHz, 193 K, CD_2Cl_2): **5b**: δ = 13.5 (br, [70%]). **3b**: δ = 9.7 ($v_{1/2}$ ~ 50 Hz, [30%]).

ROE (500 MHz, 193 K, CD_2Cl_2): $\delta^1H_{irr.}$ / $\delta^1H_{res.}$ = 7.66 / 7.49 (*p*-Ph / *m*-Ph), 7.49, 7.44 / 6.61 (*m*-, *p*-Ph / *o*-Tol), 6.99 / 6.61, 2.20 (*m*-Tol / *o*-Tol, CH_3^{Tol}), 6.61 / 7.49, 6.99, 1.78 (*o*-Tol / *m*-Ph, *m*-Tol, $^{=}CH_3$), 2.20 / 6.99 (CH_3^{Tol} / *m*-Tol), 1.78 / 6.61 ($^{=}CH_3$ / *o*-Tol), 0.88 / 7.49 (CH_3 / *m*-Ph).

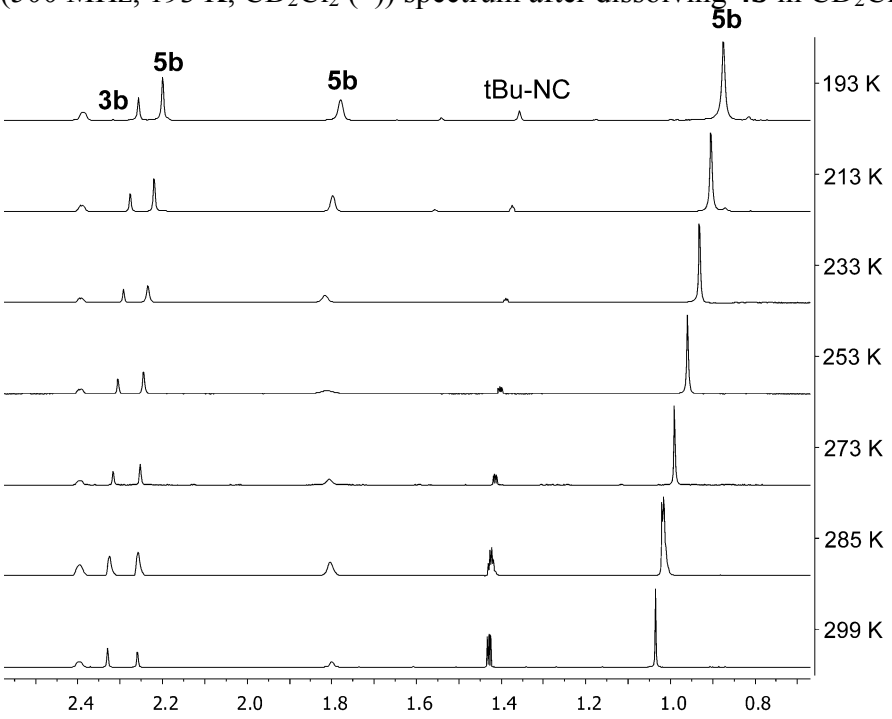
^{1}H,^{1}H GCOSY (500 MHz / 500 MHz, 193 K, CD_2Cl_2): δ^1H / δ^1H = 7.66 / 7.49 (*p*-Ph / *m*-Ph), 7.49 / 7.66, 7.44 (*m*-Ph / *p*-, *o*-Ph), 6.99 / 6.61, 2.20 (*m*-Tol / *o*-Tol, CH_3^{Tol}), 6.61 / 6.99 (*o*-Tol / *m*-Tol), 2.20 / 6.99 (CH_3^{Tol} / *m*-Tol).

^{1}H,^{13}C GHSQC (500 MHz / 126 MHz, 193 K, CD_2Cl_2): δ^1H / δ^{13}C = 7.66 / 132.7 (*p*-Ph), 7.49 / 128.6 (*m*-Ph), 7.44 / 133.2 (*o*-Ph), 6.99 / 128.7 (*m*-Tol), 6.61 / 128.9 (*o*-Tol), 2.20 / 20.6 (CH_3^{Tol}), 1.78 / 20.1 ($^{=}CH_3$), 0.88 / 28.0 (CH_3).

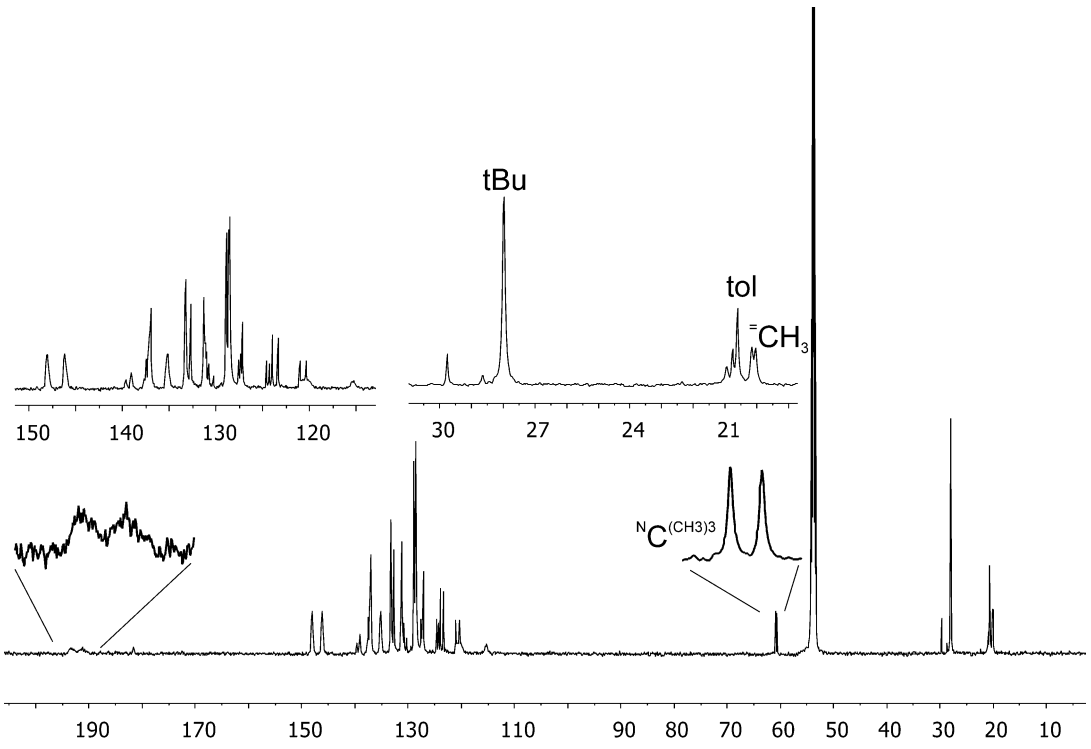
^{1}H,^{13}C GHMBC (500 MHz / 126 MHz, 193 K, CD_2Cl_2): δ^1H / δ^{13}C = 7.66 / 133.2, 128.6 (*p*-Ph / *o*-, *m*-Ph), 7.49 / 132.7, 123.6 (*m*-Ph / *p*-, *i*-Ph), 6.99 / 20.6 (*m*-Tol / CH_3^{Tol}), 6.61 / 137.0, 120.7 (*o*-Tol / *p*-Tol, $=C^P$), 2.20 / 137.0, 128.7 (CH_3^{Tol} / *p*-, *m*-Tol), 1.78 / 193.4, 120.7 ($^{=}CH_3$ / $=C^B$, $=C^P$), 0.88 / 60.8 (CH_3 / $^{N}C^{(CH3)3}$).



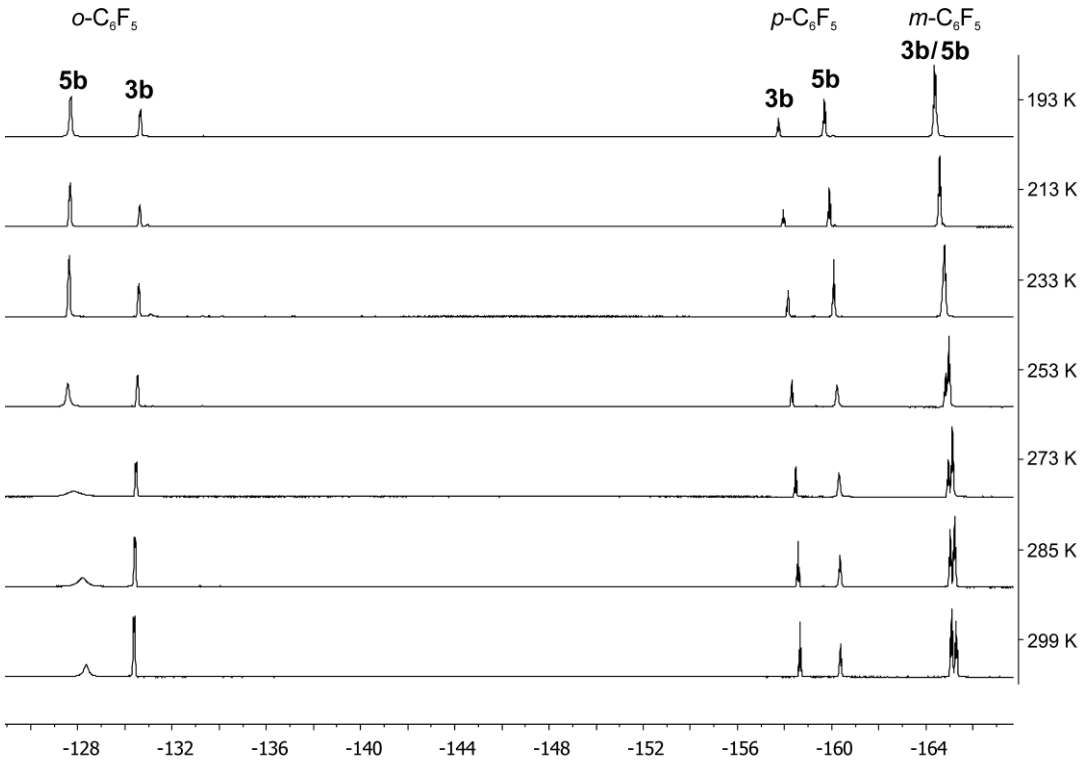
^{1}H NMR (500 MHz, 193 K, CD_2Cl_2 (*)) spectrum after dissolving **4b** in CD_2Cl_2.



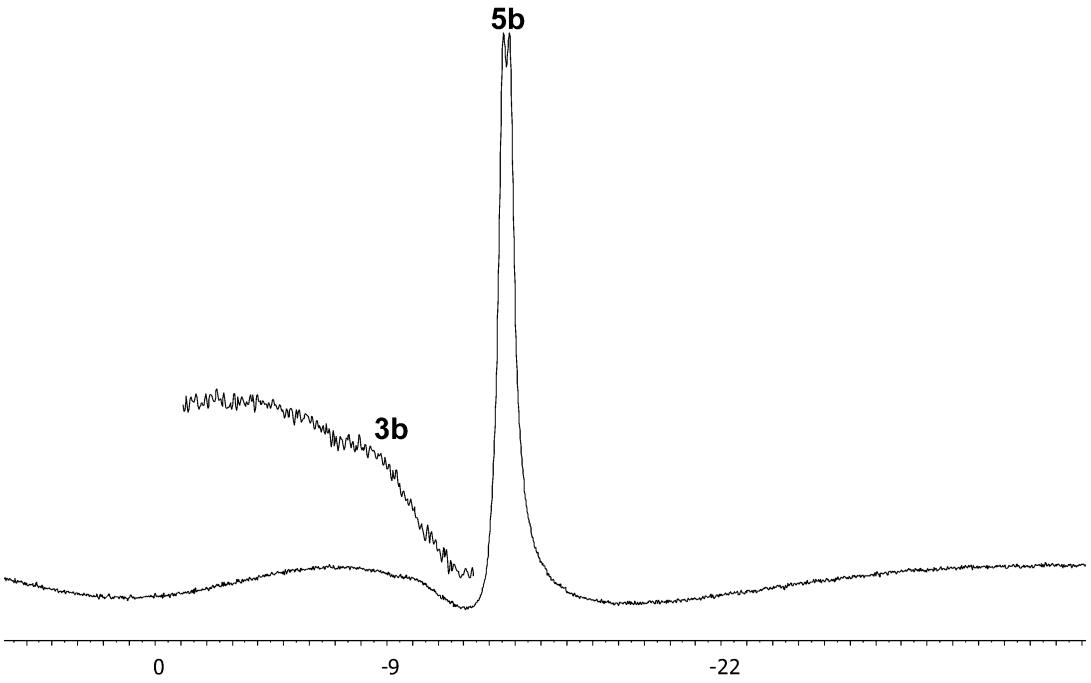
^{1}H NMR (500 MHz, CD_2Cl_2) spectrum after dissolving **4b** in CD_2Cl_2.



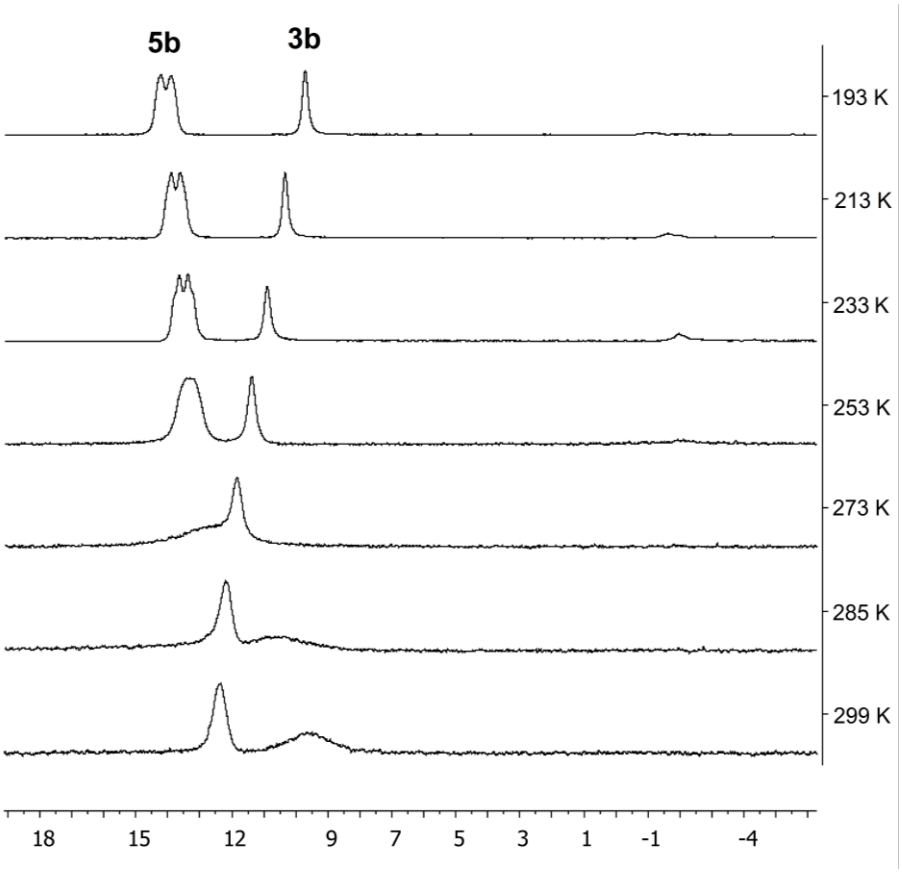
$^{13}C\{^{1}H\}$ NMR (126 MHz, 193 K, CD_2Cl_2) spectrum after dissolving **4b** in CD_2Cl_2.



$^{19}F\{^{1}H\}$ NMR (470 MHz, CD_2Cl_2) spectra after dissolving **4b** in CD_2Cl_2.

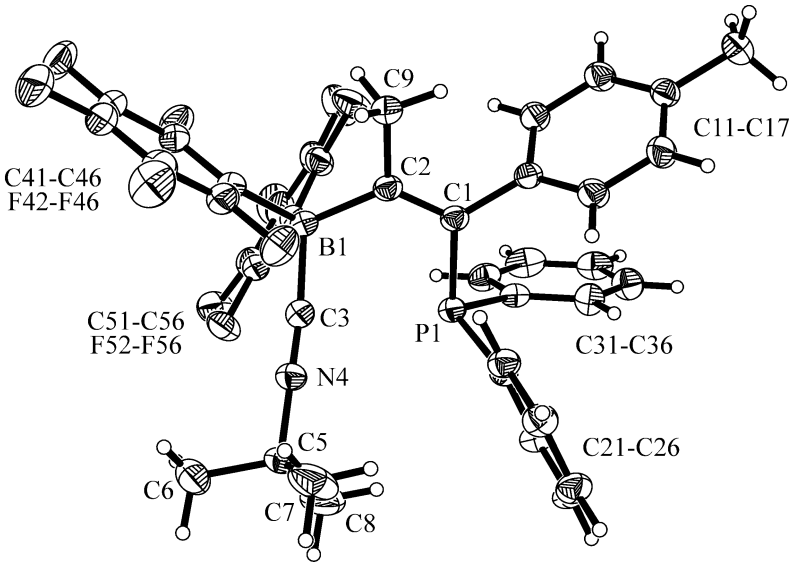


$^{11}B\{^{1}H\}$ NMR (160 MHz, CD_2Cl_2, 193K) spectrum after dissolving **4b** in CD_2Cl_2.

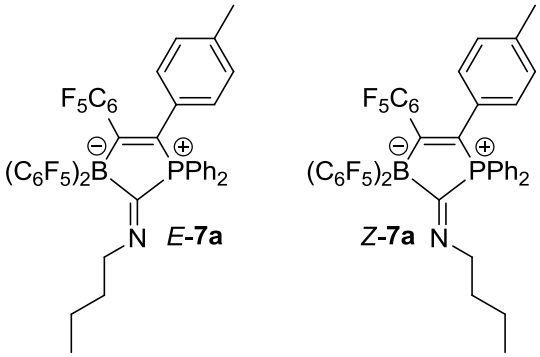


$^{31}P\{^{1}H\}$ NMR (202 MHz, CD_2Cl_2) spectra of **4b**.
[δ $^{31}P\{^{1}H\}$ (193 K): -1.1 (tentatively assigned as **4b**)]

X-Ray crystal structure analysis of **4b.** formula $C_{39}H_{29}BF_{10}NP$, M = 743.41, colourless crystal, 0.27 x 0.17 x 0.05 mm, a = 10.1806(5), b = 10.4896(3), c = 17.7612(8) Å, α = 94.693(3), β = 95.330(5), γ = 112.360(3)°, V = 1732.34(13) Å^3, ρ_{calc} = 1.425 gcm^{-3}, μ = 1.455 mm^{-1}, empirical absorption correction (0.694 ≤ T ≤ 0.930), Z = 2, triclinic, space group $P\bar{1}$ (No. 2), λ = 1.54178 Å, T = 223(2) K, ω and φ scans, 20969 reflections collected (±h, ±k, ±l), [(sinθ)/λ] = 0.60 Å^{-1}, 5917 independent (R_{int} = 0.033) and 5387 observed reflections [I>2σ(I)], 474 refined parameters, R = 0.041, wR^2 = 0.1085, max. (min.) residual electron density 0.27 (-0.24) e.Å^{-3}, hydrogen atoms calculated and refined as riding atoms.



Synthesis of 7a.



Compound **3a** (0.120 g, 0.148 mmol) was dissolved in dichloromethane (10 ml) and *n*-butyl isocyanide (20.4 mg, 0.245 mmol) was added. After stirring for one hour at room temperature, solvent and all volatiles were removed *in vacuo*. Product *E*-/*Z*-**7a** [98:2 (^{19}F NMR in CD_2Cl_2, major isomer tentatively assigned as *E*-**7a**] (93.6 mg, 0.105 mmol, 71%) was isolated as a light yellow solid. Crystals suitable for X-ray diffraction were grown by slow evaporation of a dichloromethane/pentane solution of **7a** at -36 °C. **Anal. Calc.** for $C_{44}H_{26}BF_{15}NP$: C, 59.02;

H, 2.93; N, 1.56. Found: C, 59.48; H, 3.20; N, 1.50. **HRMS:** Calc. for $C_{44}H_{26}BF_{15}NPH$: 896.17411. Found: 896.17435. **IR** (KBr): $\tilde{\nu}$ / cm^{-1} = 3063 (m), 2931, 2861 (br m), 2360 (w), 2273 (w), 1904 (w), 1642 (m), 1599 (m), 1517 (s), 1459 (s), 1279 (m), 1097 (s), 972 (s). **M.p.** (DSC): 153 °C. In the ^{31}P CPMAS solid state NMR experiment the resonance of **7a** was detected at δ = 3.3.

Major isomer:

^{1}H NMR (500 MHz, 299 K, CD_2Cl_2): δ = 7.73 (m, 2H, *p*-Ph), 7.59 (m, 4H, *o*-Ph), 7.56 (m, 4H, *m*-Ph), 6.96 (dm, $^{3}J_{HH}$ = 7.8 Hz, 2H, *m*-Tol), 6.83 (br d, $^{3}J_{HH}$ = 7.8 Hz, 2H, *o*-Tol), 3.52 (m, 2H, $^{N}CH_2$), 2.23 (s, 3H, CH_3^{Tol}), 1.35 (m, 2H, CH_2), 1.19 (m, 2H, CH_2^{CH3}), 0.79 (t, $^{3}J_{HH}$ = 7.3 Hz, CH_3).

^{13}C{^{1}H} NMR (126 MHz, 299 K, CD_2Cl_2): δ = 191.6 (br m, N=C), 174.6 (br s, ^{B}C=), 139.5 (d, $^{5}J_{PC}$ = 1.5 Hz, *p*-Tol), 134.3 (d, $^{4}J_{PC}$ = 3.1 Hz, *p*-Ph), 134.2 (br d, $^{1}J_{PC}$ ~ 85 Hz, =C^{P}), 134.0 (d, $^{2}J_{PC}$ = 9.1 Hz, *o*-Ph), 131.9 (dm, $^{2}J_{PC}$ ~ 12 Hz, *i*-Tol), 129.9 (d, $^{4}J_{PC}$ = 0.7 Hz, *m*-Tol), 129.8 (d, $^{3}J_{PC}$ = 12.1 Hz, *m*-Ph), 128.5 (d, $^{3}J_{PC}$ = 3.5 Hz, *o*-Tol), 121.7 (d, $^{1}J_{PC}$ = 76.1 Hz, *i*-Ph), 61.1 (dm, $^{3}J_{PC}$ ~ 40 Hz, $^{N}CH_2$), 32.5 (d, $^{4}J_{PC}$ = 2.2 Hz, CH_2), 21.3 (CH_3^{Tol}), 21.0 (CH_2^{CH3}), 14.0 (CH_3), [C_6F_5 not listed].

^{19}F NMR (470 MHz, 299 K, CD_2Cl_2): δ = -129.5 (m, 4F, *o*-BC_6F_5), -140.5 (m, 2F, *o*-C_6F_5), -156.9 (t, $^{3}J_{FF}$ = 20.9 Hz, 1F, *p*-C_6F_5), -159.7 (t, $^{3}J_{FF}$ = 20.4 Hz, 2F, *p*-BC_6F_5), -163.8 (m, 2F, *m*-C_6F_5), -165.4 (m, 4F, *m*-BC_6F_5) [$\Delta\delta^{B}$(m, p) = 5.7].

^{11}B{^{1}H} NMR (160 MHz, 299 K, CD_2Cl_2): δ = -13.6 (d, J_{PB} = 41.5 Hz, 95%, *E*-**7a**^{t}), -10.3 (d, J_{PB} = 46.4 Hz, 5%, *Z*-**7a**^{t}) [^{t} tentatively assigned].

^{31}P{^{1}H} NMR (202 MHz, 299 K, CD_2Cl_2): δ = 8.2 (1:1:1:1 q, J_{PB} = 41.5 Hz, *E*-**7a**^{t}), -0.9 (1:1:1:1:1 q, J_{PB} = 46.4 Hz, *Z*-**7a**^{t}) [^{t} tentatively assigned].

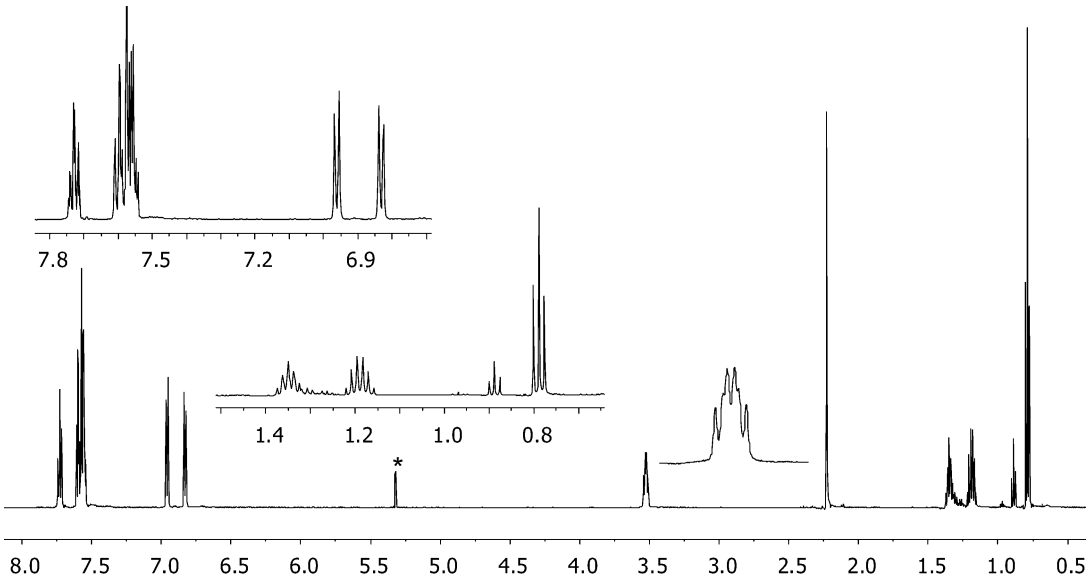
TOCSY (500 MHz, 299 K, CD_2Cl_2): $\delta^{1}H_{irr.}$ / $\delta^{1}H_{res.}$ = 7.73 / 7.59, 7.56 (*p*-Ph / *o*-, *m*-Ph), 6.96 / 6.83, 2.23 (*m*-Tol / *o*-Tol, CH_3^{Tol}), 3.52 / 1.35, 1.19, 0.79 ($^{N}CH_2$ / CH_2, CH_2^{CH3}, CH_3).

NOE (500 MHz, 299 K, CD_2Cl_2): $\delta^{1}H_{irr.}$ / $\delta^{1}H_{res.}$ = 7.73 / 7.56 (*p*-Ph / *m*-Ph), 7.59, 7.56 / 7.73, 6.83 (*o*-, *m*-Ph / *p*-Ph, *o*-Tol), 6.96 / 6.83, 2.23 (*m*-Tol / *o*-Tol, CH_3^{Tol}), 6.83 / 7.56, 6.96 (*o*-Tol / *m*-Ph, *m*-Tol), 3.52 / 7.56, 1.35, 1.19, 0.79 ($^{N}CH_2$ / *m*-Ph, CH_2, CH_2^{CH3}, CH_3), 2.23 / 6.96 (CH_3^{Tol} / *m*-Tol), 1.35 / 7.56, 3.52, 0.79 (CH_2 / *m*-Ph, $^{N}CH_2$, CH_3), 1.19 / 3.52, 0.79 (CH_2^{CH3} / $^{N}CH_2$, CH_3), 0.79 / 3.52, 1.35, 1.19 (CH_3 / $^{N}CH_2$, CH_2, CH_2^{CH3}).

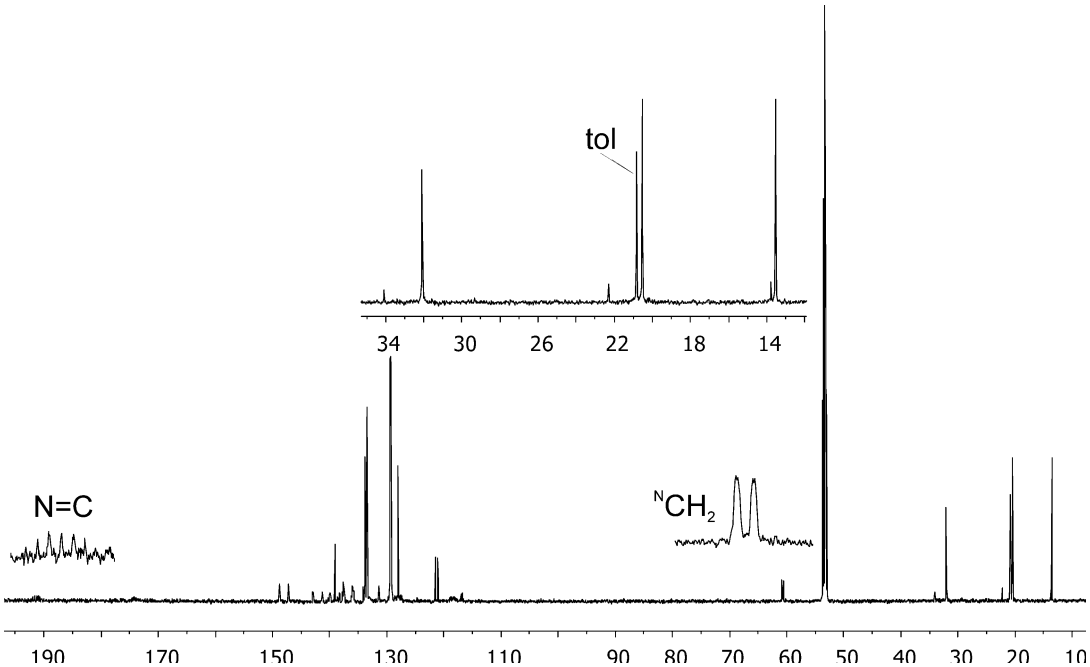
^{1}H,^{1}H GCOSY (500 MHz / 500 MHz, 299 K, CD_2Cl_2): $\delta^{1}H$ / $\delta^{1}H$ = 7.73 / 7.56 (*p*-Ph / *m*-Ph), 7.56 / 7.73, 7.59 (*m*-Ph / *p*-, *o*-Ph), 6.96 / 6.83, 2.23 (*m*-Tol / *o*-Tol, CH_3^{Tol}), 6.83 / 6.96 (*o*-Tol / *m*-Tol), 3.52 / 1.35 ($^{N}CH_2$ / CH_2), 2.23 / 6.96, 6.83 (CH_3^{Tol} / *m*-, *o*-Tol), 1.35 / 3.52, 1.19 (CH_2 / $^{N}CH_2$, CH_2^{CH3}), 1.19 / 1.35, 0.79 (CH_2^{CH3} / CH_2, CH_3), 0.79 / 1.19 (CH_3 / CH_2^{CH3}).

^{1}H,^{13}C GHSQC (500 MHz / 126 MHz, 299 K, CD_2Cl_2): δ^1H / δ^{13}C = 7.73 / 134.3 (*p*-Ph), 7.59 / 134.0 (*o*-Ph), 7.56 / 129.8 (*m*-Ph), 6.96 / 129.9 (*m*-Tol), 6.83 / 128.5 (*o*-Tol), 3.52 / 61.1 ($^{N}CH_2$), 2.23 / 21.3 (CH_3^{Tol}), 1.35 / 32.5 (CH_2), 1.19 / 21.0 (CH_2^{CH3}), 0.79 / 14.0 (CH_3).

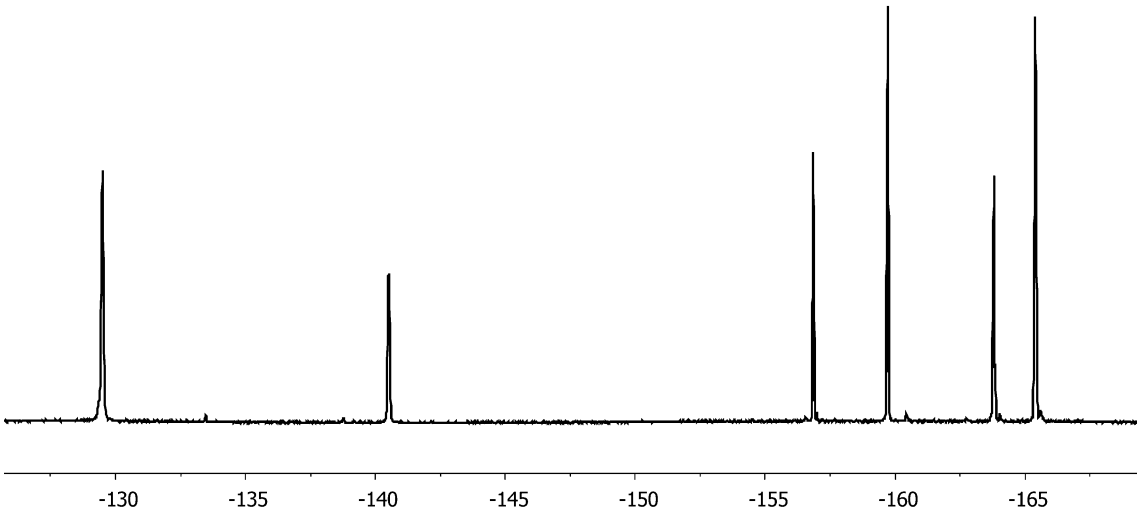
^{1}H,^{13}C GHMBC (500 MHz / 126 MHz, 299 K, CD_2Cl_2): δ^1H / δ^{13}C = 7.73 / 134.0, 129.8 (*p*-Ph / *o*-, *m*-Ph), 7.59 / 134.2 (*o*-Ph / *p*-Ph), 7.56 / 121.7 (*m*-Ph / *i*-Ph), 6.96 / 131.9, 21.3 (*m*-Tol / *i*-Tol, CH_3^{Tol}), 6.83 / 139.5, 134.2 (*o*-Tol / *p*-Tol, $=C^P$), 3.52 / 191.6, 32.5, 21.0 ($^{N}CH_2$ / ^{N}C, CH_2, CH_2^{CH3}), 2.23 / 139.5, 129.9 (CH_3^{Tol} / *p*-, *m*-Tol), 1.35 / 61.1, 14.0 (CH_2 / $^{N}CH_2$, CH_3), 1.19 / 61.1, 14.0 (CH_2^{CH3} / $^{N}CH_2$, CH_3), 0.79 / 32.5, 21.0 (CH_3 / CH_2, CH_2^{CH3}).



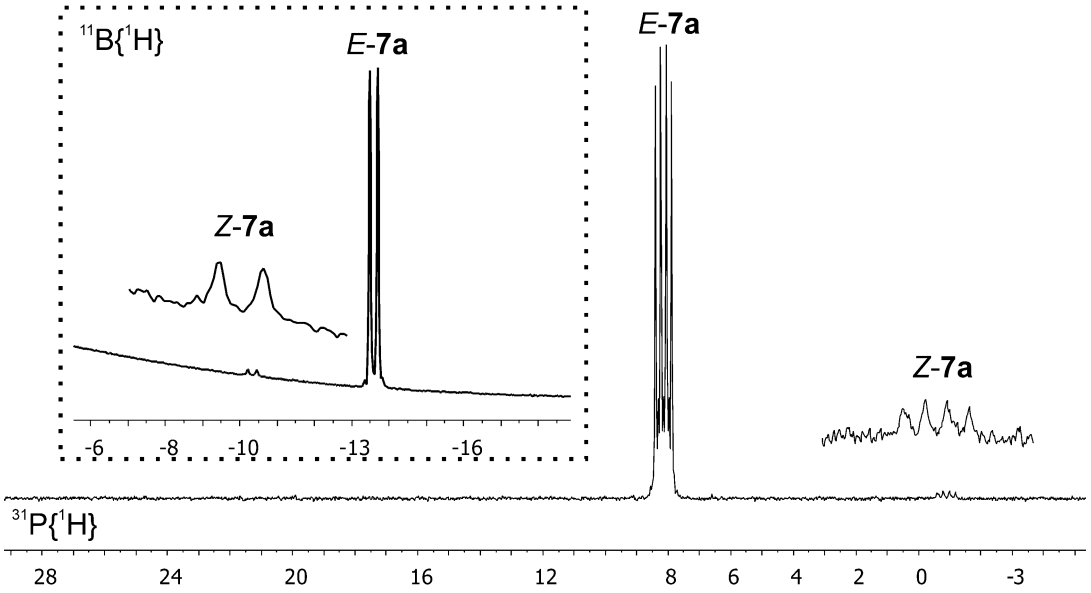
^{1}H NMR (600 MHz, 299 K, CD_2Cl_2 (*)) of **7a**.



^{13}C{^{1}H} NMR (151 MHz, 299 K, CD_2Cl_2) of **7a**.

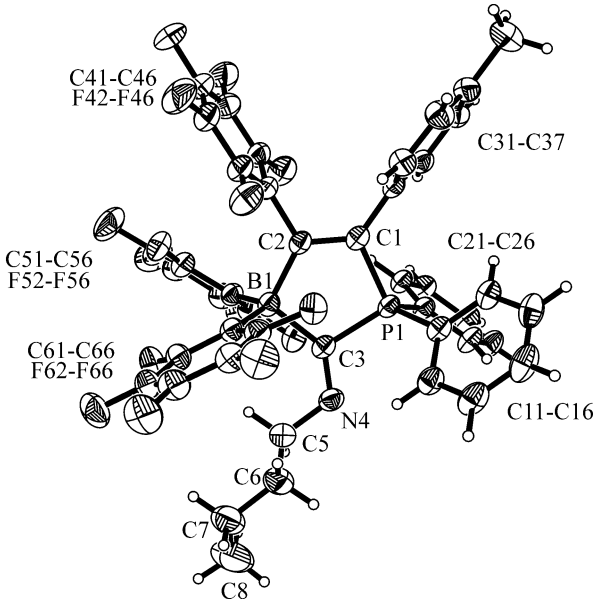


^{19}F NMR (564 MHz, 299 K, CD_2Cl_2) of **7a**.



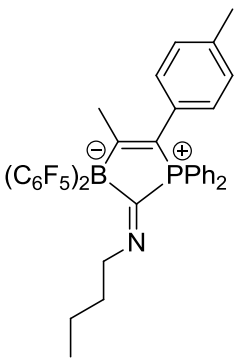
$^{31}P\{^1H\}$ NMR (243 MHz, 299 K, CD_2Cl_2) and $^{11}B\{^1H\}$ NMR (192 MHz) of **7a**.

X-Ray crystal structure analysis of **7a.** formula $C_{44}H_{26}BF_{15}NP$ * 2 CH_2Cl_2, M = 1065.29, colourless crystal, 0.30 x 0.19 x 0.06 mm, a = 17.6472(7), b = 14.6343(4), c = 18.4267(7) Å, β = 103.288(2)°, V = 4631.4(3) Å^3, ρ_{calc} = 1.528 gcm^{-3}, μ = 3.513 mm^{-1}, empirical absorption correction ($0.418 \leq T \leq 0.816$), Z = 4, monoclinic, space group $P2_1/n$ (No. 14), λ = 1.54178 Å, T = 223(2) K, ω and φ scans, 33466 reflections collected ($\pm h$, $\pm k$, $\pm l$), [(sinθ)/λ] = 0.60 Å^{-1}, 7877 independent (R_{int} = 0.056) and 5891 observed reflections [$I>2\sigma(I)$], 646 refined parameters, R = 0.089, wR^2 = 0.272, max. (min.) residual electron density 0.60 (-0.81) e.Å^{-3}, hydrogen atoms calculated and refined as riding atoms.



Synthesis of 7b.

Compound **3b** (45.7 mg, 0.069 mmol) was dissolved in dichloromethane (10 ml) and *n*-butyl isocyanide (15.2 mg, 0.183 mmol) was added. After stirring for one hour at room temperature, solvent and all volatiles were removed *in vacuo*. A product mixture of *E*-/*Z*-**7b** [7:1 (^{19}F NMR in CD_2Cl_2, major isomer tentatively assigned as *E*-**7b**] (26.7 mg, 0.036 mmol, 52%) was isolated as a light brown solid. Crystals suitable for X-ray diffraction were grown by slow evaporation of dichloromethane/pentane solution of *E*-/*Z*-**7b** at -36 °C. **HRMS:** Calc. for $C_{39}H_{29}BF_{10}NPH$: 744.20505. Found: 744.20303. **IR** (KBr): $\tilde{\nu}$ / cm^{-1} = 2931 (br m), 2360 (w), 2147 (w), 1641 (m), 1602 (m), 1515 (m), 1460 (s), 1094 (s), 966 (s). **M.p.** (DSC): 134 °C.



E-**7b**^{t}**: ^{1}H NMR** (500 MHz, 299 K, CD_2Cl_2): δ = 7.64 (m, 2H, *p*-Ph), 7.51 (m, 4H, *o*-Ph), 7.48 (m, 4H, *m*-Ph), 7.09 (m, 2H, *m*-Tol), 6.87 (m, 2H, *o*-Tol), 3.51 (m, 2H, $^{N}CH_2$), 2.31 (s, 3H, CH_3^{Tol}), 1.92 (s, 3H, $^{=}CH_3$), 1.42 (m, 2H, CH_2), 1.21 (m, 2H, CH_2^{CH3}), 0.79 (t, $^{3}J_{HH}$ = 7.4 Hz, 3H, CH_3).

^{13}C{^{1}H} NMR (126 MHz, 299 K, CD_2Cl_2): δ = 193.0 (br m, N=C, ^{B}C=), 138.1 (d, $^{5}J_{PC}$ = 1.6 Hz, *p*-Tol), 133.9 (d, $^{2}J_{PC}$ = 9.1 Hz, *o*-Ph), 133.6 (d, $^{4}J_{PC}$ = 3.2 Hz, *p*-Ph), 132.3 (d, $^{2}J_{PC}$ = 12.5 Hz, *i*-Tol), 129.9 (d, $^{3}J_{PC}$ = 3.6 Hz, *o*-Tol), 129.7 (d, $^{4}J_{PC}$ = 0.7 Hz, *m*-Tol), 129.3 (d, $^{3}J_{PC}$ = 11.8 Hz, *m*-Ph), 123.8 (d, $^{1}J_{PC}$ = 75.3 Hz, *i*-Ph), 122.7 (d, $^{1}J_{PC}$ = 90.8 Hz, =C^{P}), 59.9 (d, $^{3}J_{PC}$ = 38.3 Hz, $^{N}CH_2$), 32.8 (d, $^{4}J_{PC}$ = 2.2 Hz, CH_2), 21.3 (CH_3^{Tol}), 21.0 (CH_2^{CH3}), 20.5 (br d, $^{3}J_{PC}$ = 15.6 Hz, $^{=}CH_3$), 14.0 (CH_3), [C_6F_5 not listed].

^{19}F NMR (470 MHz, 299 K, CD_2Cl_2): δ = -129.8 (m, 2F, *o*-C_6F_5), -160.8 (t, $^3J_{FF}$ = 20.5 Hz, 1F, *p*-C_6F_5), -165.4 (m, 2F, *m*-C_6F_5) [$\Delta\delta^B$(m, p) = 4.6].

^{11}B{^{1}H} NMR (160 MHz, 299 K, CD_2Cl_2): δ = -13.9 (d, J_{PB} ~ 45 Hz).

^{31}P{^{1}H} NMR (202 MHz, 299 K, CD_2Cl_2): δ = 11.7 (1:1:1:1 q, J_{PB} ~ 45 Hz).

TOCSY (500 MHz, 299 K, CD_2Cl_2): $\delta^1H_{irr.}$ / $\delta^1H_{res.}$ = 7.64 / 7.51, 7.48 (*p*-Ph / *o*-, *m*-Ph), 7.09 / 6.87, 2.31 (*m*-Tol / *o*-Tol, CH_3^{Tol}), 3.51 / 1.42, 1.21, 0.79 ($^NCH_2$ / CH_2, CH_2^{CH3}, CH_3).

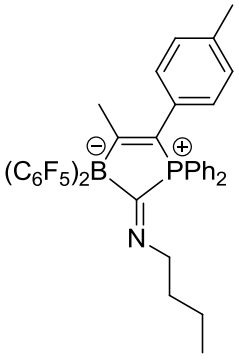
NOE (500 MHz, 299 K, CD_2Cl_2): $\delta^1H_{irr.}$ / $\delta^1H_{res.}$ = 7.64 / 7.48 (*p*-Ph / *m*-Ph), 7.51, 7.48 / 7.64, 6.87 (*o*-, *m*-Ph / *p*-Ph, *o*-Tol), 7.09 / 6.87, 2.31 (*m*-Tol / *o*-Tol, CH_3^{Tol}), 6.87 / 7.51, 7.48, 7.09, 1.92 (*o*-Tol / *o*-, *m*-Ph, *m*-Tol, $^=CH_3$), 3.51 / 7.51, 7.48, 1.42, 1.21, 0.79 ($^NCH_2$ / *o*-, *m*-Ph, CH_2, CH_2^{CH3}, CH_3), 2.31 / 7.09 (CH_3^{Tol} / *m*-Tol), 1.91 / 6.87 ($^=CH_3$ / *o*-Tol), 1.42 / 3.51, 0.79 (CH_2 / $^NCH_2$, CH_3), 1.21 / 3.51 (CH_2^{CH3} / $^NCH_2$).

^{1}H,^{1}H GCOSY (500 MHz / 500 MHz, 299 K, CD_2Cl_2): δ^1H / δ^1H = 7.64 / 7.48 (*p*-Ph / *m*-Ph), 7.48 / 7.64, 7.51 (*m*-Ph / *p*-, *o*-Ph), 7.09 / 6.87, 2.31 (*m*-Tol / *o*-Tol, CH_3^{Tol}), 3.51 / 1.42, 1.21 ($^NCH_2$ / CH_2, CH_2^{CH3}), 1.21 / 3.51, 1.42, 0.79 (CH_2^{CH3} / $^NCH_2$, CH_2, CH_3), 0.79 / 1.21 (CH_3 / CH_2^{CH3}).

^{1}H,^{13}C GHSQC (500 MHz / 126 MHz, 299 K, CD_2Cl_2): δ^1H / $\delta^{13}C$ = 7.64 / 133.6 (*p*-Ph), 7.51 / 133.9 (*o*-Ph), 7.48 / 129.3 (*m*-Ph), 7.09 / 129.7 (*m*-Tol), 6.87 / 129.9 (*o*-Tol), 3.51 / 59.9 ($^NCH_2$), 2.31 / 21.3 (CH_3^{Tol}), 1.92 / 20.5 ($^=CH_3$), 1.42 / 32.8 (CH_2), 1.21 / 21.0 (CH_2^{CH3}), 0.79 / 14.0 (CH_3).

^{1}H,^{13}C GHMBC (500 MHz / 126 MHz, 299 K, CD_2Cl_2): δ^1H / $\delta^{13}C$ = 7.64 / 133.9, 129.3 (*p*-Ph / *o*-, *m*-Ph), 7.51 / 133.6 (*o*-Ph / *p*-Ph), 7.48 / 123.8 (*m*-Ph / *i*-Ph), 7.09 / 132.3, 21.3 (*m*-Tol / *i*-Tol, CH_3^{Tol}), 6.87 / 138.1, 122.7 (*o*-Tol / *p*-Tol, $=C^P$), 3.51 / 193.0, 32.8, 21.0 ($^NCH_2$ / ^NC, CH_2, CH_2^{CH3}), 2.31 / 138.1, 129.7 (CH_3^{Tol} / *p*-, *m*-Tol), 1.92 / 193.0, 122.7 ($^=CH_3$ / $^BC=$, $=C^P$), 1.42 / 59.9, 21.0, 14.0 (CH_2 / $^NCH_2$, CH_2^{CH3}, CH_3), 1.21 / 59.9, 32.8, 14.0 (CH_2^{CH3} / $^NCH_2$, CH_2, CH_3), 0.79 / 32.8, 21.0 (CH_3 / CH_2, CH_2^{CH3}).

^{1}H,^{19}F HOESY (600 MHz / 564 MHz, 299 K, CD_2Cl_2): δ^1H / $\delta^{19}F$ = 7.51, 3.51, 1.92 / -129.8 (*o*-Ph, $^NCH_2$, $^=CH_3$ / *o*-C_6F_5).



Selected resonances: *E*-**7b: ^{1}H NMR** (500 MHz, 299 K, CD_2Cl_2): δ = 7.03 (br d, $^3J_{HH}$ = 7.9 Hz, 2H, *m*-Tol), 6.73 (br d, $^3J_{HH}$ = 7.9 Hz, 2H, *o*-Tol), 3.52 (m, 2H, $^NCH_2$), 2.28 (s, 3H, CH_3^{Tol}), 1.86 (s, 3H, $^=CH_3$), 1.23 (m, 2H, CH_2), 0.90 (m, 2H, CH_2^{CH3}), 0.61 (t, $^3J_{HH}$ = 7.4 Hz, 3H, CH_3).

Selected resonances: ^{13}C{^{1}H} NMR (126 MHz, 299 K, CD_2Cl_2): δ =

191.1 (br m, ^{B}C=), 177.7 (N=C), 138.1 (*p*-Tol), 131.5 (d, $^{2}J_{PC}$ ~ 13 Hz, *i*-Tol), 130.2 (d, $^{3}J_{PC}$ = 3.4 Hz, *o*-Tol), 129.5 (*m*-Tol), 122.9 (d, $^{1}J_{PC}$ = 64.0 Hz, =C^{P}), 66.6 (d, $^{3}J_{PC}$ = 39.4 Hz, $^{N}CH_2$), 32.2 (d, $^{4}J_{PC}$ = 1.5 Hz, CH_2), 20.8 (d, $^{3}J_{PC}$ = 9.1 Hz, $^{=}CH_3$), 20.5 ($CH_2{}^{CH3}$), 13.8 (CH_3).

$^{19}F\{^{1}H\}$ NMR (470 MHz, 299 K, CD_2Cl_2): δ = -130.3 (m, 2F, *o*-C_6F_5), -161.3 (t, $^{3}J_{FF}$ = 20.2 Hz, 1F, *p*-C_6F_5), -165.6 (m, 2F, *m*-C_6F_5) [$\Delta\delta^{B}$(m, p) = 4.3].

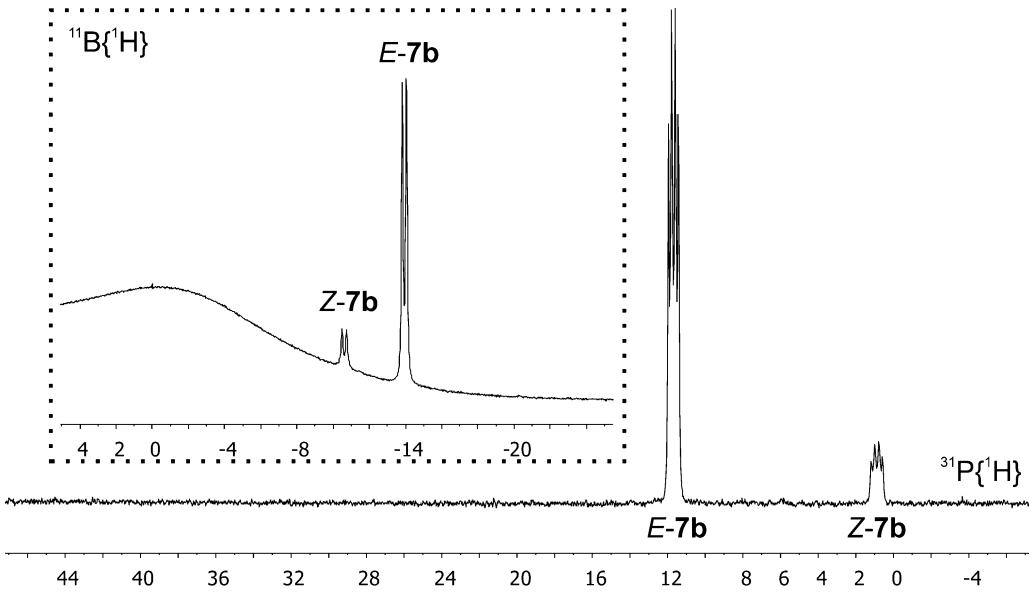
$^{11}B\{^{1}H\}$ NMR (160 MHz, 299 K, CD_2Cl_2): δ = -10.6 (d, J_{PB} ~ 52 Hz).

$^{31}P\{^{1}H\}$ NMR (202 MHz, 299 K, CD_2Cl_2): δ = 0.9 (br 1:1:1:1 q, J_{PB} ~ 52 Hz).

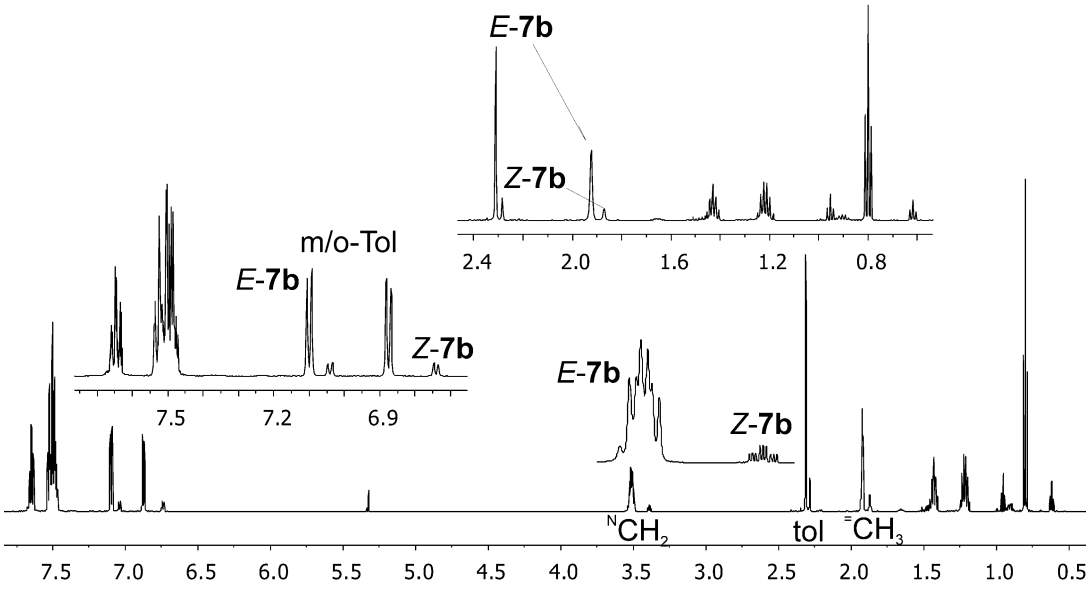
$^{1}H,^{1}H$ GCOSY (500 MHz / 500 MHz, 299 K, CD_2Cl_2): $\delta^{1}H$ / $\delta^{1}H$ = 7.03 / 6.73, 2.28 (*m*-Tol / *o*-Tol, $CH_3{}^{Tol}$), 6.73 / 7.03 (*o*-Tol / *m*-Tol), 1.23 / 0.90 (CH_2 / $CH_2{}^{CH3}$), 0.90 / 1.23, 0.61 ($CH_2{}^{CH3}$ / CH_2, CH_3).

$^{1}H,^{13}C$ GHSQC (500 MHz / 126 MHz, 299 K, CD_2Cl_2): $\delta^{1}H$ / $\delta^{13}C$ = 7.03 / 129.5 (*m*-Tol), 6.73 / 130.2 (*o*-Tol), 3.52 / 66.6 ($^{N}CH_2$), 2.28 / 21.2 ($CH_3{}^{Tol}$), 1.86 / 20.8 ($^{=}CH_3$), 1.23 / 32.2 (CH_2), 0.90 / 20.5 ($CH_2{}^{CH3}$), 0.61 / 13.8 (CH_3).

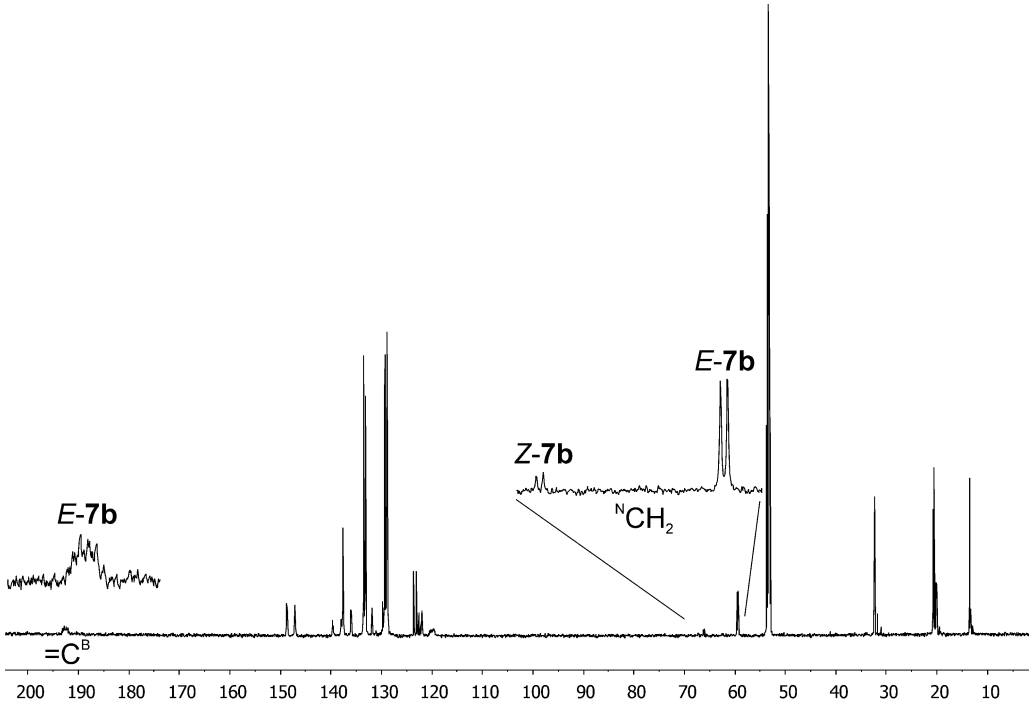
$^{1}H,^{13}C$ GHMBC (500 MHz / 126 MHz, 299 K, CD_2Cl_2): $\delta^{1}H$ / $\delta^{13}C$ = 7.03 / 131.5, 21.2 (*m*-Tol / *i*-Tol, $CH_3{}^{Tol}$), 6.73 / 138.1, 129.5 (*o*-Tol / *p*-, *m*-Tol), 3.52 / 177.7, 32.2, 20.5 ($^{N}CH_2$ / N=C, CH_2, $CH_2{}^{CH3}$), 2.28 / 138.1, 129.5 ($CH_3{}^{Tol}$ / *p*-, *m*-Tol), 1.86 / 191.1, 122.9 ($^{=}CH_3$ / ^{B}C=, =C^{P}), 1.23 / 66.6 (CH_2 / $^{N}CH_2$), 0.90 / 32.2 ($CH_2{}^{CH3}$ / CH_2), 0.61 / 32.2, 20.5 (CH_3 / CH_2, $CH_2{}^{CH3}$).



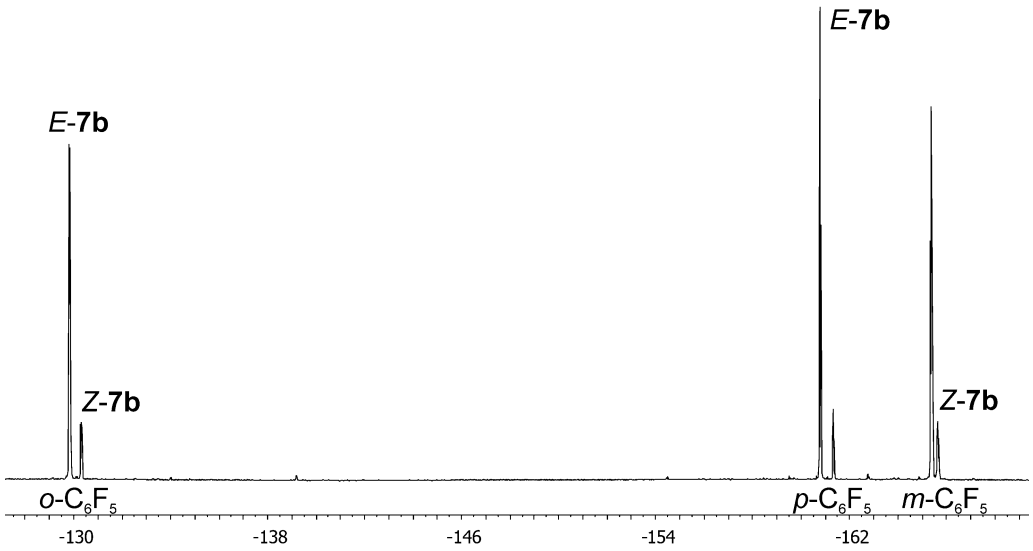
$^{31}P\{^{1}H\}$ NMR (243 MHz, 299 K, CD_2Cl_2) and $^{11}B\{^{1}H\}$ NMR (192 MHz) of *E*-/*Z*-**7b**.



^{1}H NMR (600 MHz, 299 K, CD_2Cl_2) of *E*-/*Z*-**7b**.



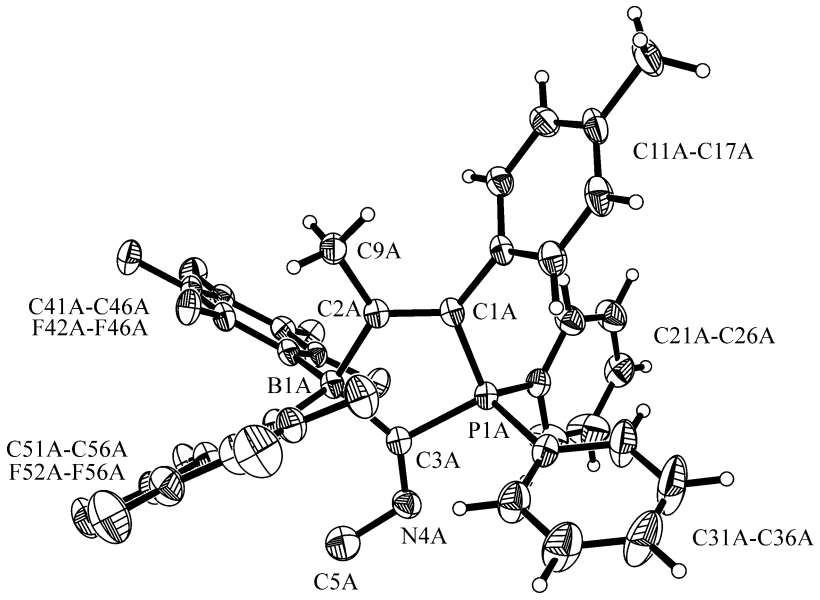
^{13}C{^{1}H} NMR (151 MHz, 299 K, CD_2Cl_2) of *E*-/*Z*-**7b**.



^{19}F NMR (564 MHz, 299 K, CD_2Cl_2) of *E*-/*Z*-**7b**.

X-Ray crystal structure analysis of **7b.** formula $C_{39}H_{29}BF_{10}NP$ * 1/2 CH_2Cl_2, M = 785.88, colourless crystal, 0.32 x 0.27 x 0.18 mm, a = 39.1199(4), b = 16.3061(2), c = 23.4811(4) Å, β = 97.421(1)°, V = 14853.0(3) Å^3, ρ_{calc} = 1.406 gcm^{-3}, μ = 2.034 mm^{-1}, empirical absorption correction (0.562 ≤ T ≤ 0.710), Z = 16, monoclinic, space group $C2/c$ (No. 15), λ = 1.54178 Å, T = 223(2) K, ω and φ scans, 68796 reflections collected ($\pm h$, $\pm k$, $\pm l$), [(sinθ)/λ] = 0.60 Å^{-1}, 12969 independent (R_{int} = 0.042) and 11402 observed reflections [I>2σ(I)], 954 refined parameters, R = 0.061, wR^2 = 0.174, max. (min.) residual electron density 0.68 (-0.59) e.Å^{-3}, hydrogen atoms calculated and refined as riding atoms.

Comment: The two n-butyl groups at N4A and N4B of compound **7b** starting at C5A/B were found disordered over two positions.



Supplemental Materials Section- Solid-State NMR

DFT calculations of NMR parameters

All calculations were carried out using the program packages TURBOMOLE (version 6.3)[1-2] and GAUSSIAN (version GAUSSIAN09)[3]. The geometry optimizations of the proton positions have been performed on a DFT meta-GGA (TPSS[4]) level of theory (starting with the crystal structure) applying the D3(BJ)[5] dispersion correction and Ahlrich's def2-TZVP basis set[6]. The geometry optimization was performed within the TURBOMOLE program suite. In all TURBOMOLE SCF calculations an energy convergence criterion of 10^{-7} E_h and in all geometry optimizations an energy convergence criterion of $5 \cdot 10^{-7}$ E_h was chosen. The integration grid was set to m4[7] and the RI approximation[8-9] was used.

For the calculations of nuclear electric quadrupole coupling the positions of the heavy atoms were taken from the crystal structure, whereas the positions of the hydrogen atoms were optimized (see above). The calculations of the electric field gradients were performed on a GGA DFT level (functional B97-D[10]) using the program package GAUSSIAN09. The def2-TZVP basis set obtained from the EMSL data base[11-12] was modified for the EFG calculations in such a way that tighter basis functions on the boron atom (extracted from the cc-pCVTZ[13-14] basis set, for details see ref. [15]) were included for having a more accurate description of the region near the boron nucleus. The GAUSSIAN output files were analysed by using the program EFGShield, version 2.4[16], for determination of C_Q and η_Q values.

The magnetic shielding calculations were performed within the GIAO (gauge independent atomic orbitals) framework[17-18]. Magnetic shieldings were calculated on the B3-LYP[19-20] level of theory with the def2-TZVP basis set using the TURBOMOLE program package. Chemical shifts are referenced to $BF_3 \cdot Et_2O$ by using B_2H_6 ($\delta(B_2H_6)$= 16.6 ppm vs. $BF_3 \cdot Et_2O$)[21-23] as an external standard ($\sigma^{B3\text{-}LYP}(B_2H_6)$= 84.23 ppm) in case of boron and to phosphoric acid ($\sigma^{B3\text{-}LYP}$= 274.31 ppm) in case of phosphorus. ^{31}P CSA parameters were determined on a B3-LYP/TZVP level of theory using the program package GAUSSIAN. $^{31}P^{\cdots 11}B$ spin-spin coupling constants were also calculated with the GAUSSIAN program on a B3-LYP/TZVP level of theory by using the structures mentioned above.

The ^{13}C shielding of TMS is determined by adding the calculated shielding of methane (B3-LYP/def2-TZVP) to the experimental chemical shift of methane in the gas phase (δ= -11.0 ppm) that is referenced to TMS under consideration of susceptibility corrections (σ_{TMS}=184.1 ppm)[24] (the calculated magnetic shielding of TMS on above discussed level of theory is determined to 179.4 ppm).

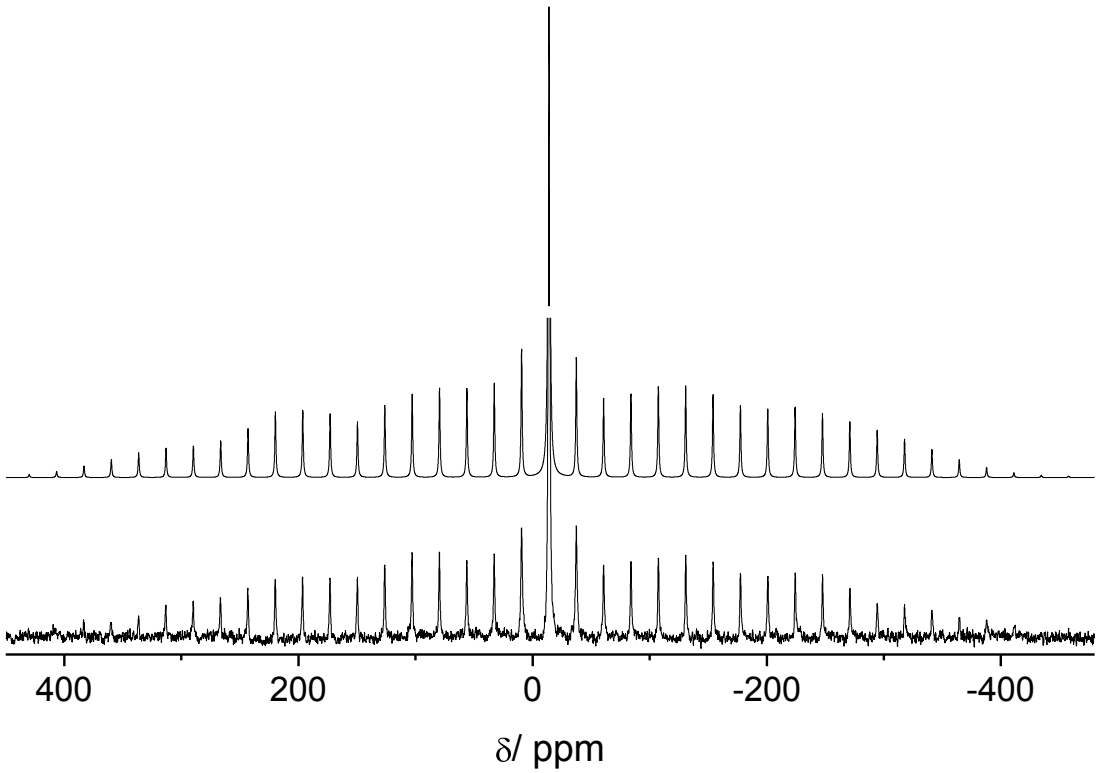


Figure S1: $^{11}B\{^{1}H\}$ SATRAS spectrum of **7a** acquired at 9.4 T with a spinning frequency of 3.0 kHz (a) and simulated spectrum (b) based on the following simulation parameters: C_Q = 0.1 MHz, η_Q= 0.49 and δ^{CS}_{iso}= -14.0 ppm.

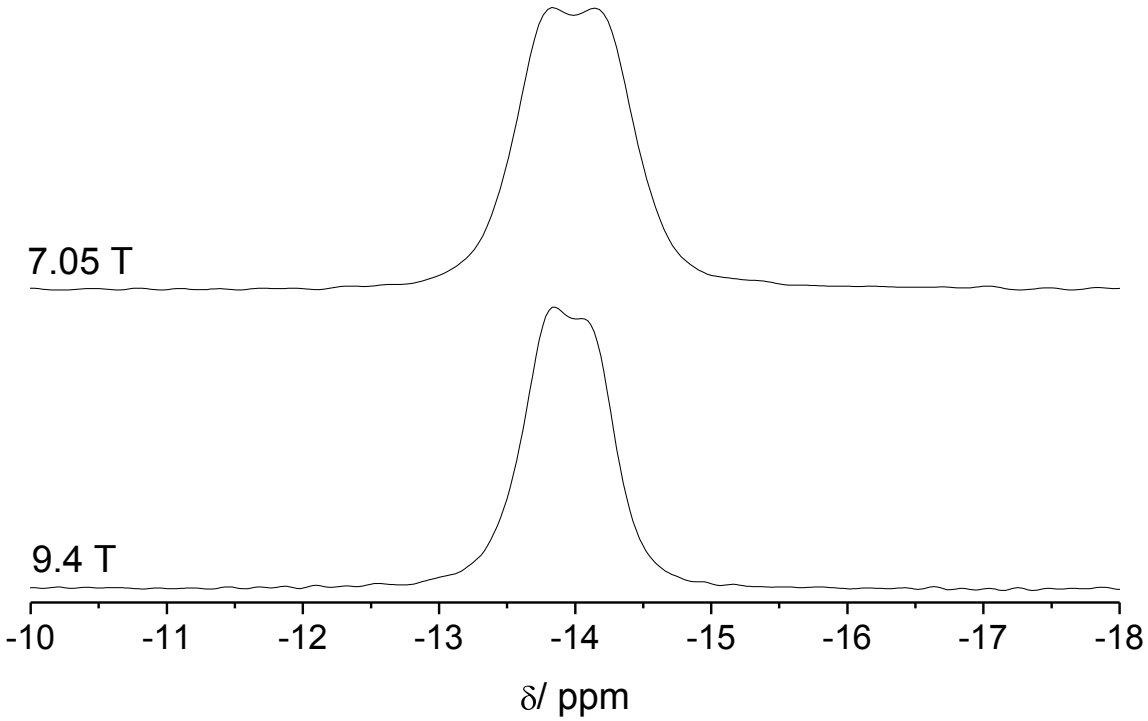


Figure S2: $^{11}B\{^{1}H\}$ MAS NMR spectra of **7a** acquired at 9.4 and 7.05 T with a spinning frequency of 12.0 kHz.

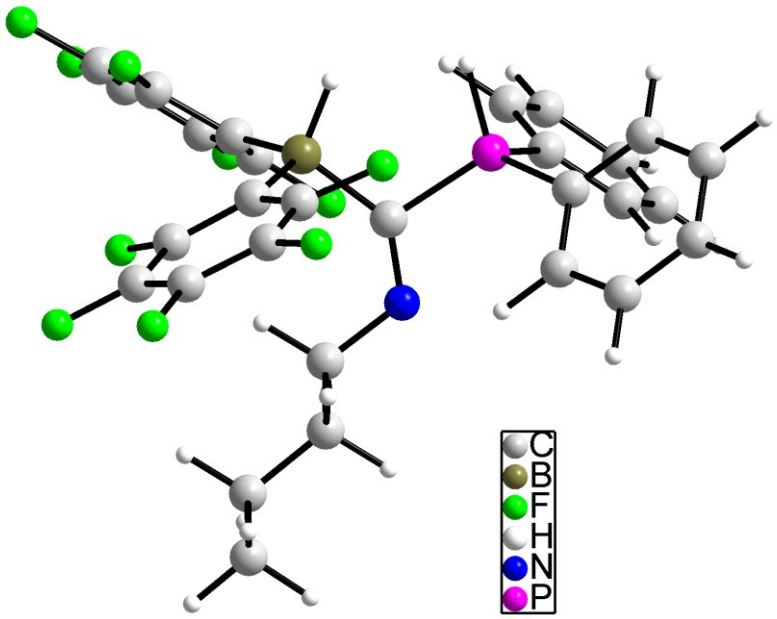


Figure S3: Model of **7a** in which only a $^{2}J(^{11}B^{\cdots 31}P)$ coupling pathway between the phosphorus and boron center via the isonitrile carbon is present. The $^{2}J(^{11}B^{\cdots 31}P)$ coupling constant is calculated to 47.4 Hz (B3-LYP/TZVP) in perfect agreement with the experimental value of 44 ± 5 Hz also assigned to a $^{2}J(^{11}B^{\cdots 31}P)$ coupling pathway.

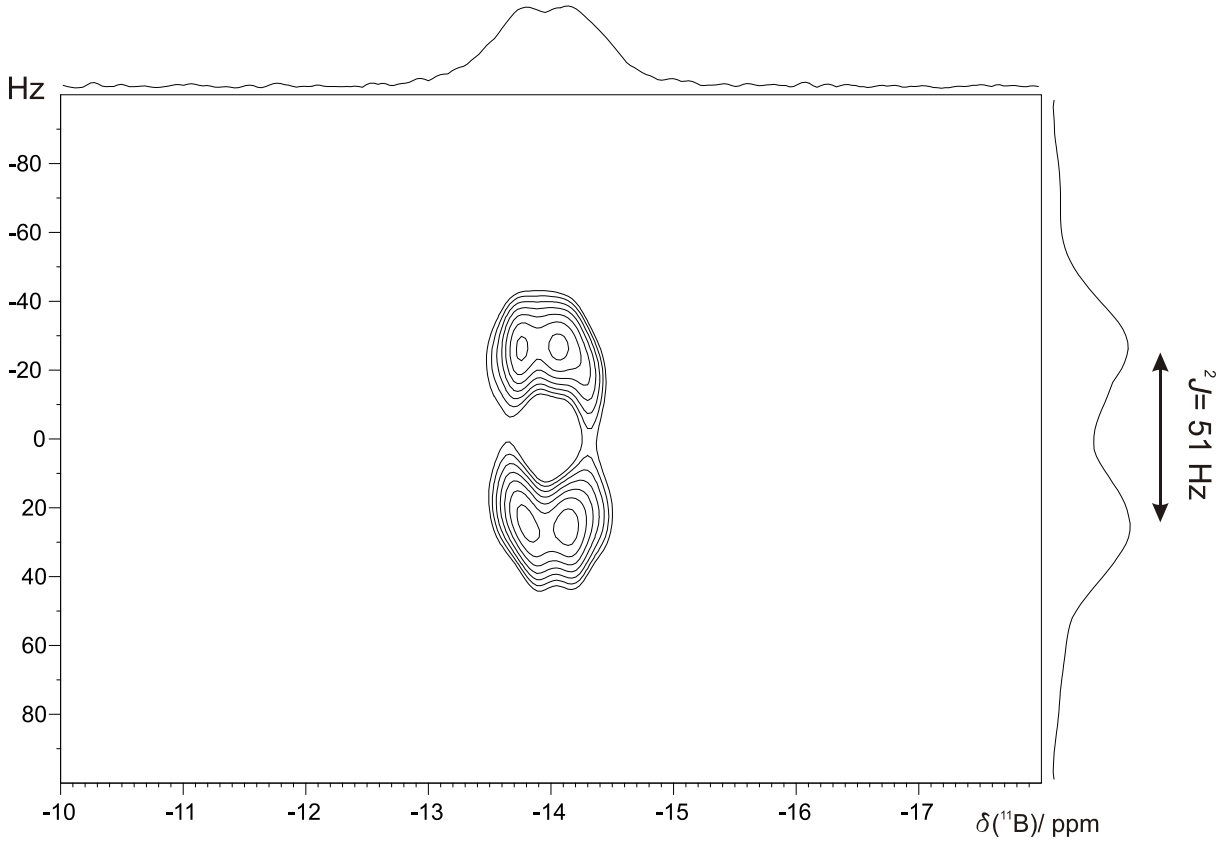


Figure S4: $^{11}B\{^{31}P\}$ heteronculear J-Resolved spectrum of **7a** measured at 7.05 T with a rotation frequency of 10.0 kHz.

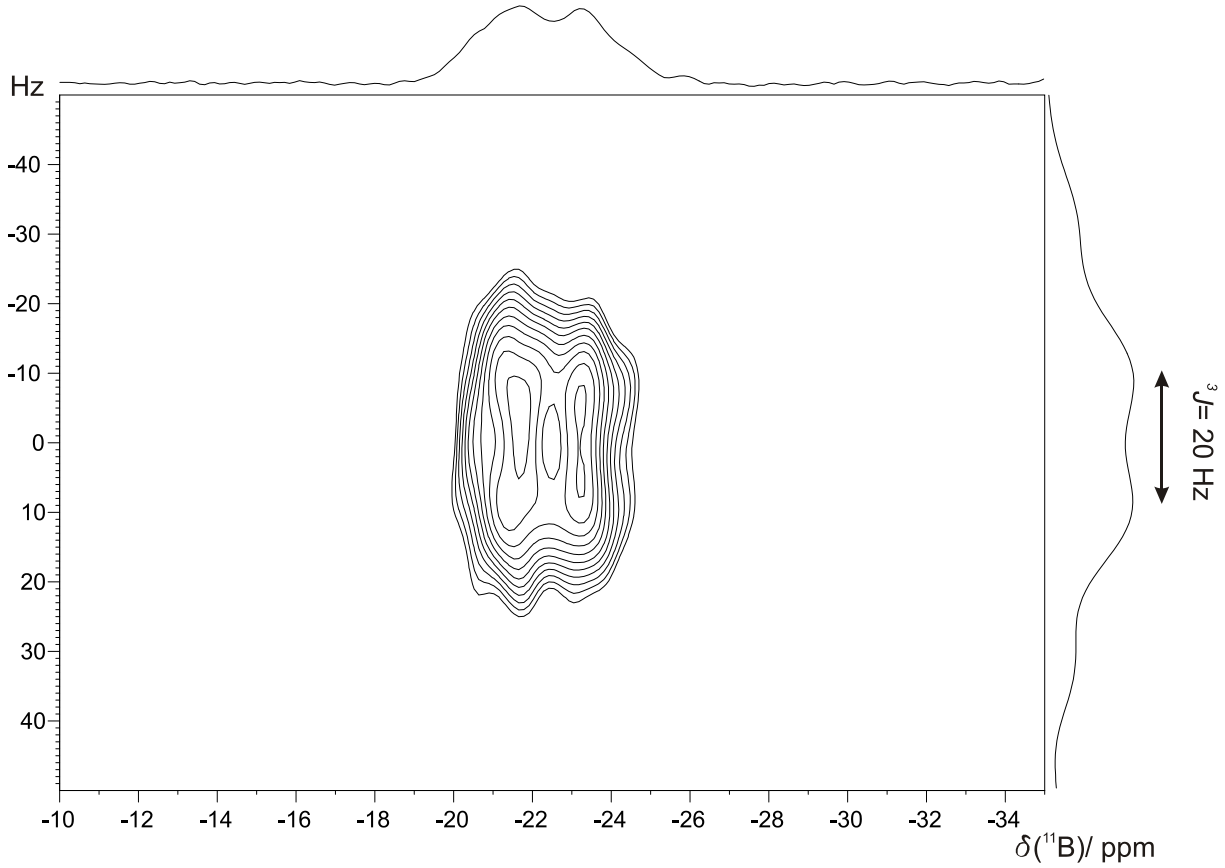


Figure S5: $^{11}B\{^{31}P\}$ heteronculear J-Resolved spectrum of **4a** measured at 7.05 T with a rotation frequency of 10.0 kHz.

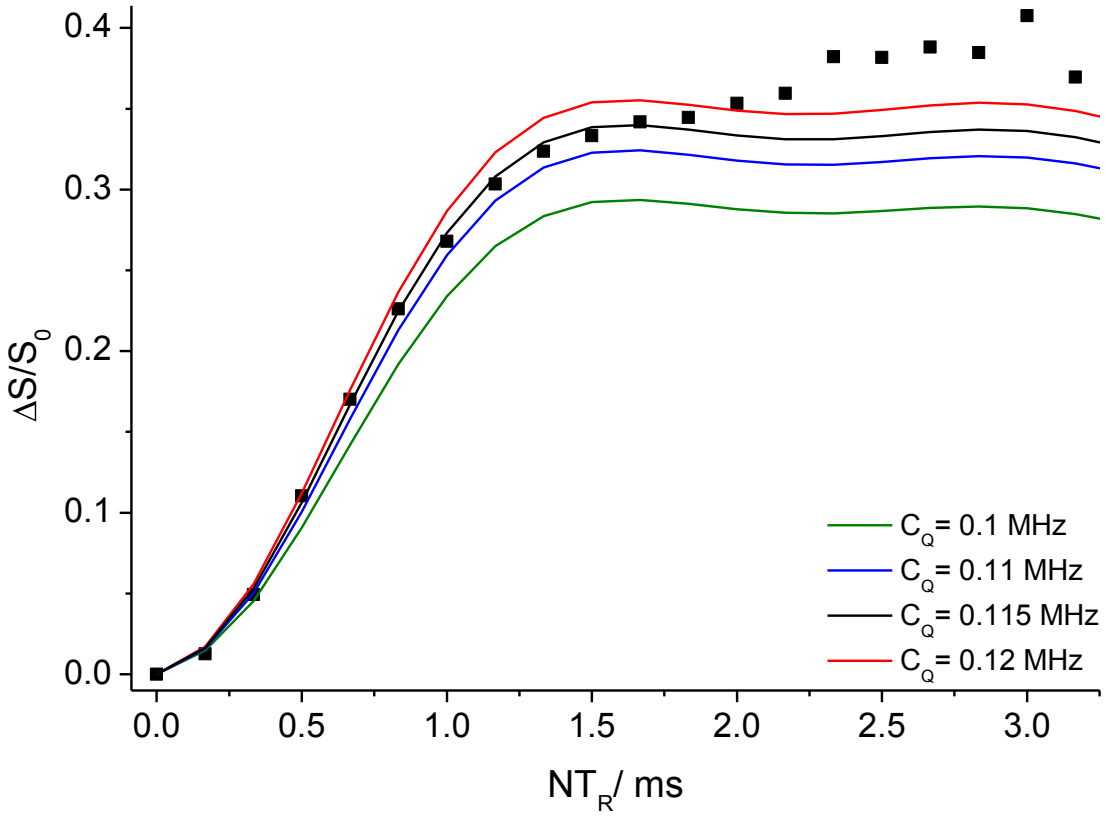


Figure S6: $^{1}H \rightarrow {}^{31}P\{^{11}B\}$ CP-REAPDOR curves for **7a** acquired at 7.05 T with a rotation frequency of 12.0 kHz. Additionally, SIMPSON simulations based on the crystallographic bororn-phosphorus distance and a ΔJ value of 20 Hz are shown, but the quadrupolar coupling constant was varied in those simulations. The best agreement is found for a C_Q value of 0.115 MHz indicating the extremely high sensitivity of those REAPDOR curves on the ^{11}B quadrupolar coupling parameters.

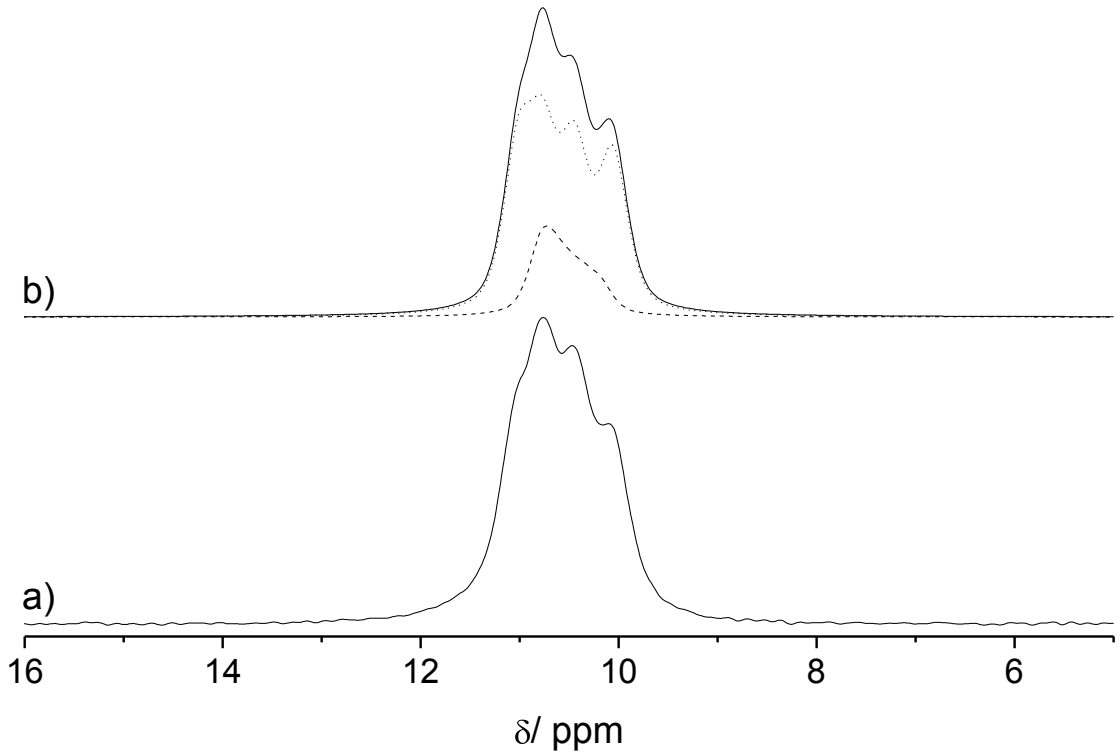


Figure S7: $^{31}P\{^{1}H\}$ CPMAS spectrum of **3a** acquired at 9.4 T with a rotation frequency of 10.0 kHz (a) and corresponding line shape simulation (b) based on the spin-spin coupling mechanisms between phosphorus and the two boron isotopes (dashed line: ^{10}B, dotted line: ^{11}B). The simulation is based on the following parameters: δ_{iso}=10.6 ppm, $J(^{31}P^{\cdots 11}B)$= 52 Hz, $J(^{31}P^{\cdots 10}B)$= 17.2 Hz, $d(^{31}P^{\cdots 11}B)$= -5.5 Hz, $d(^{31}P^{\cdots 10}B)$= -11.5 Hz. The ratios of the peak areas are scaled according to the natural abundances of the two boron isotopes, *d* represents the residual dipolar coupling.

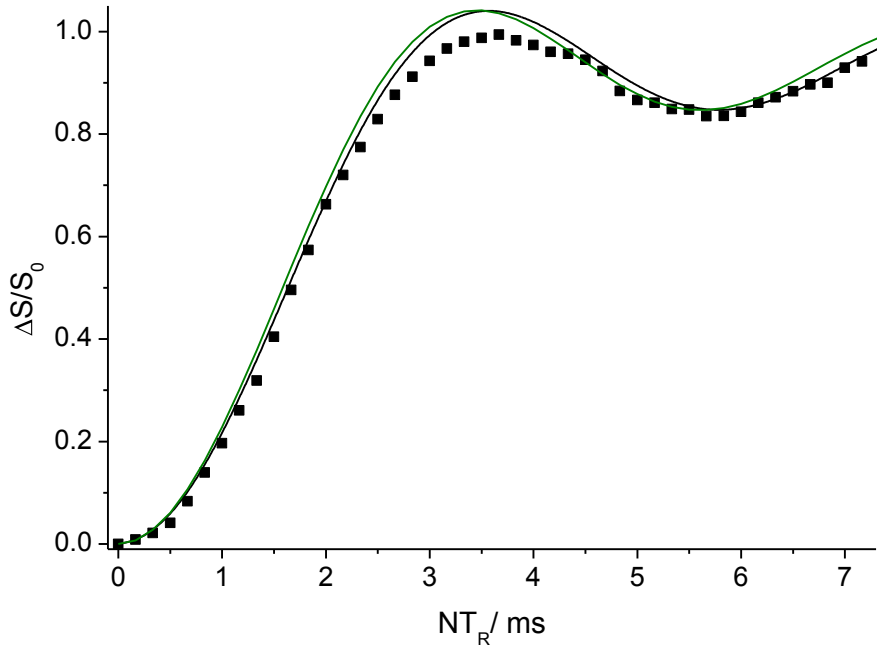


Figure S8a: $^{11}B\{^{31}P\}$ REDOR curve of **4b** and corresponding SIMPSON simulations based on the crystallographic distance, experimental CSA parameters and ΔJ=0 Hz (green curve) and ΔJ=15 Hz (black curve).

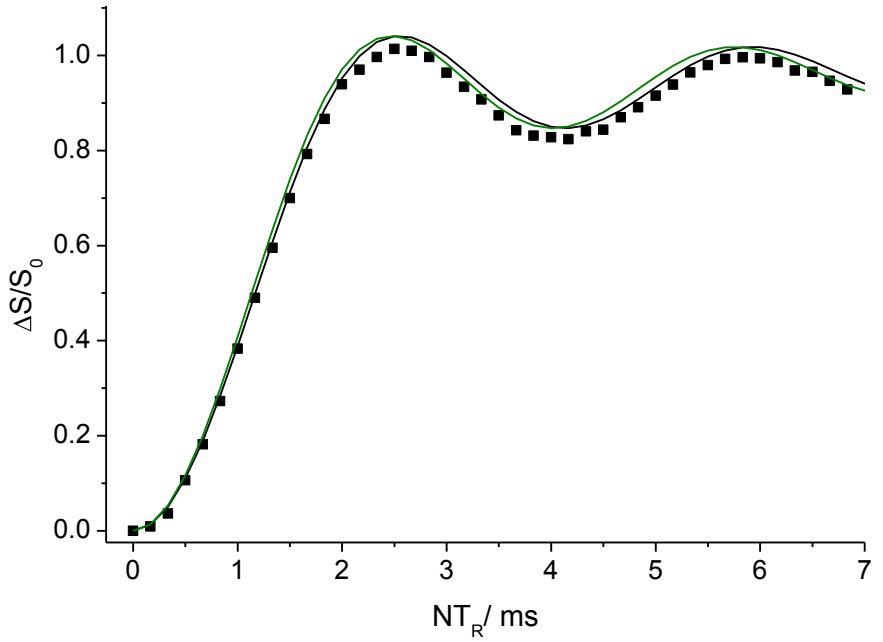


Figure S8b: $^{11}B\{^{31}P\}$ REDOR curve of **7a** and corresponding SIMPSON simulations based on the crystallographic distance, experimental CSA parameters and ΔJ=0 Hz (green curve) and ΔJ=20 Hz (black curve).

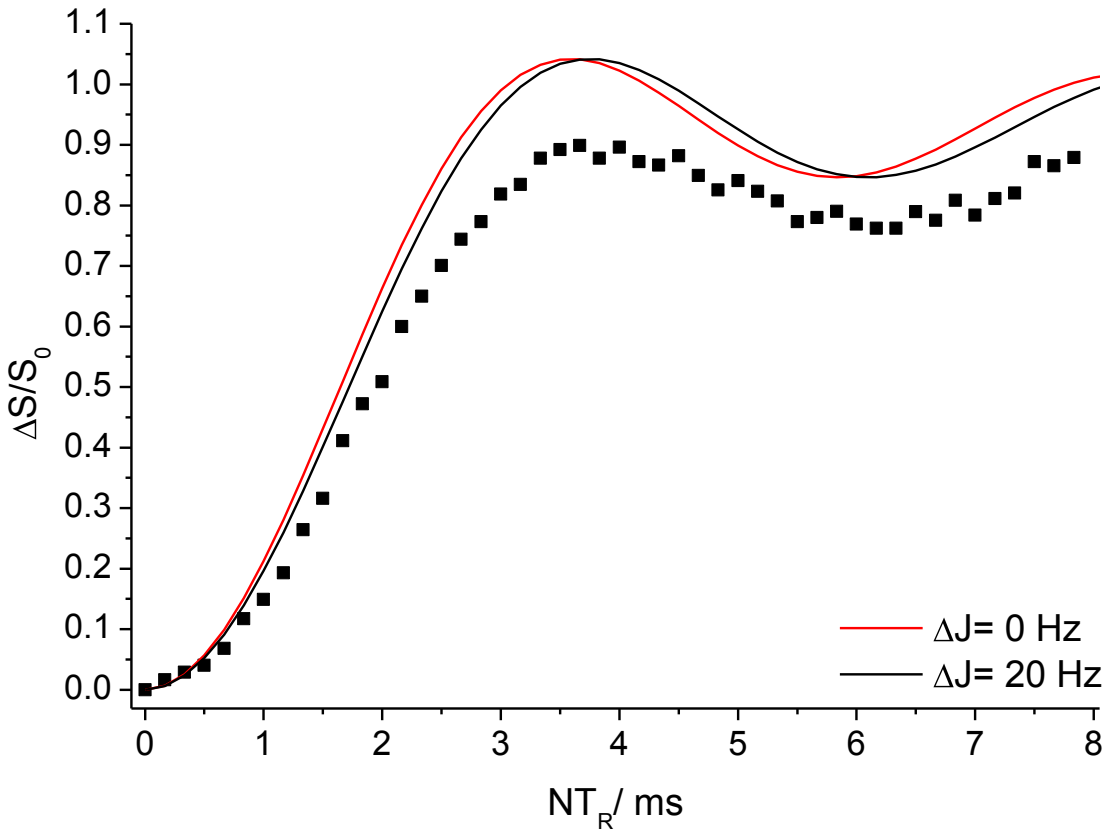


Figure S8c: $^{11}B\{^{31}P\}$ REDOR curve of **4a** and corresponding SIMPSON simulations based on the crystallographic distance, experimental CSA parameters and ΔJ=0 Hz (red curve) and ΔJ=20 Hz (black line).

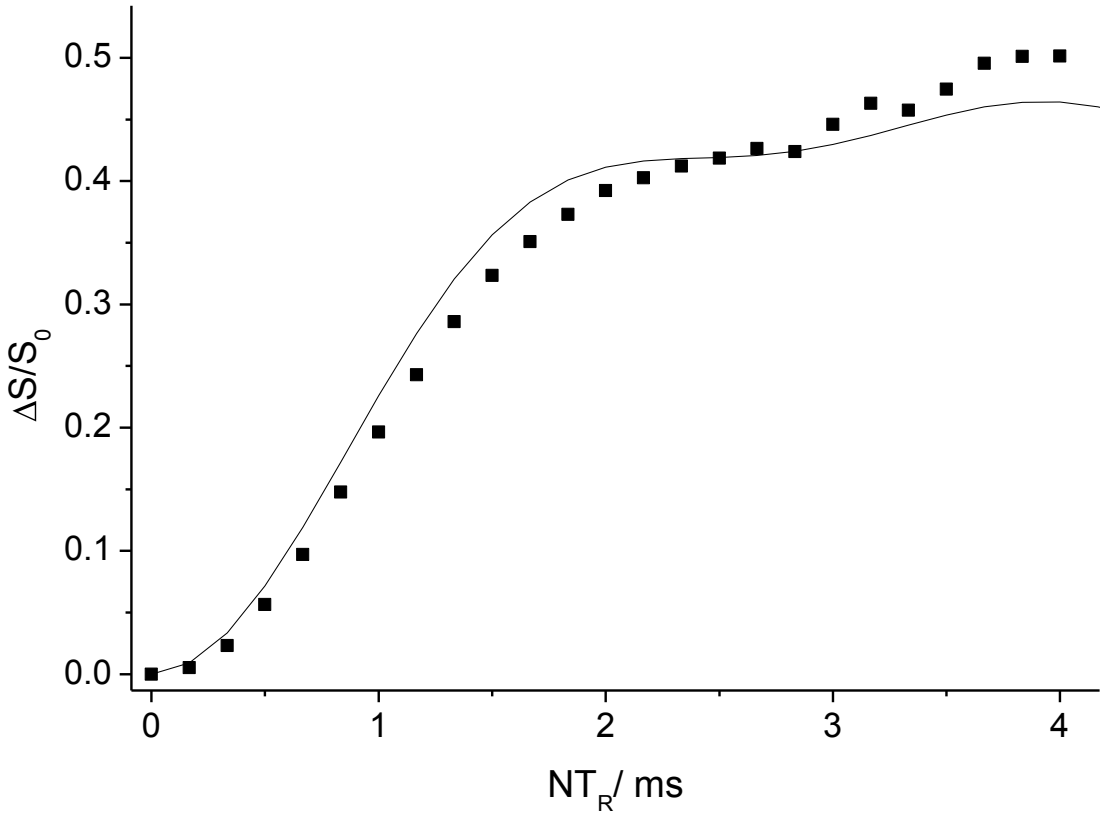


Figure S9: $^{1}H \rightarrow ^{31}P\{^{11}B\}$ CP-REAPDOR experiment of **4a** acquired at 7.05 T with a sample rotation frequency of 12.0 kHz. The solid curve shows the corresponding SIMPSON simulation based on the cyrstallographically determined B···P distance, experimental ^{31}P CSA and ^{11}B quadrupolar coupling parameters.

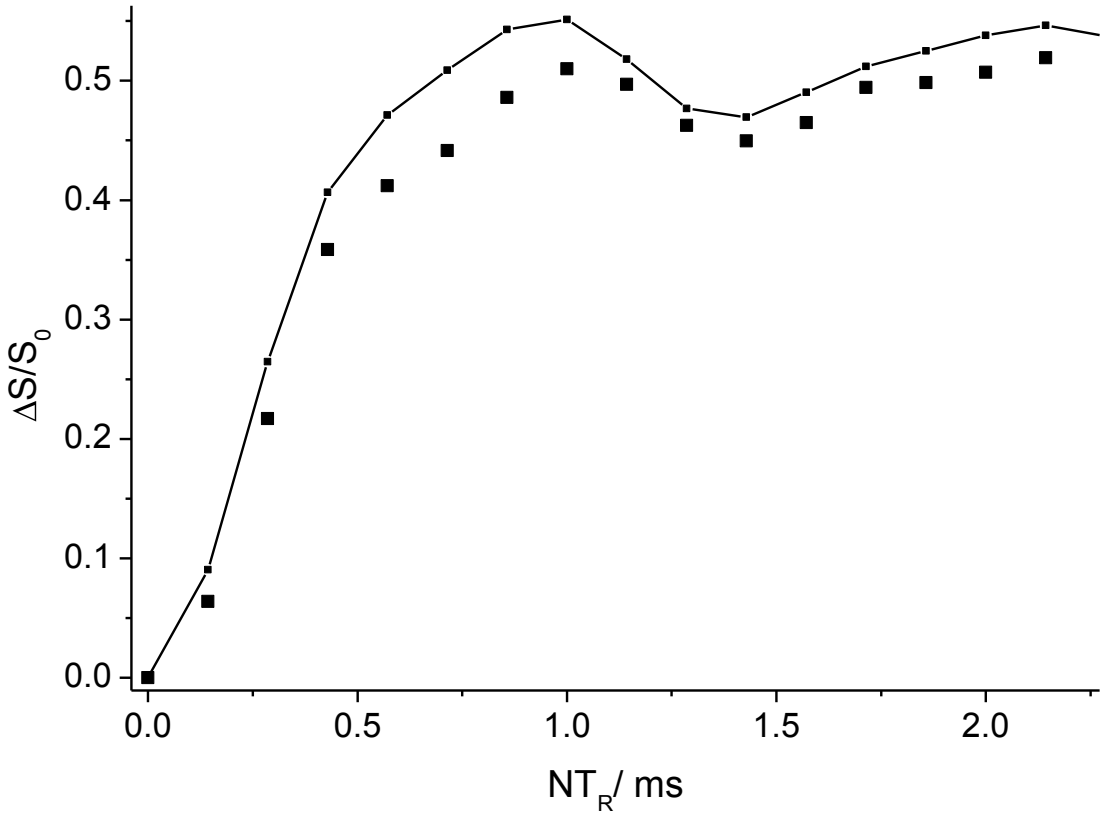


Figure S10: $^{1}H \rightarrow {}^{31}P\{^{11}B\}$ CP-REAPDOR experiment of **3a** acquired at 7.05 T with a sample rotation frequency of 14.0 kHz. The solid curve shows the corresponding SIMPSON simulation based on the crystallographically determined B···P distance, experimental ^{31}P CSA and ^{11}B quadrupolar coupling parameters.

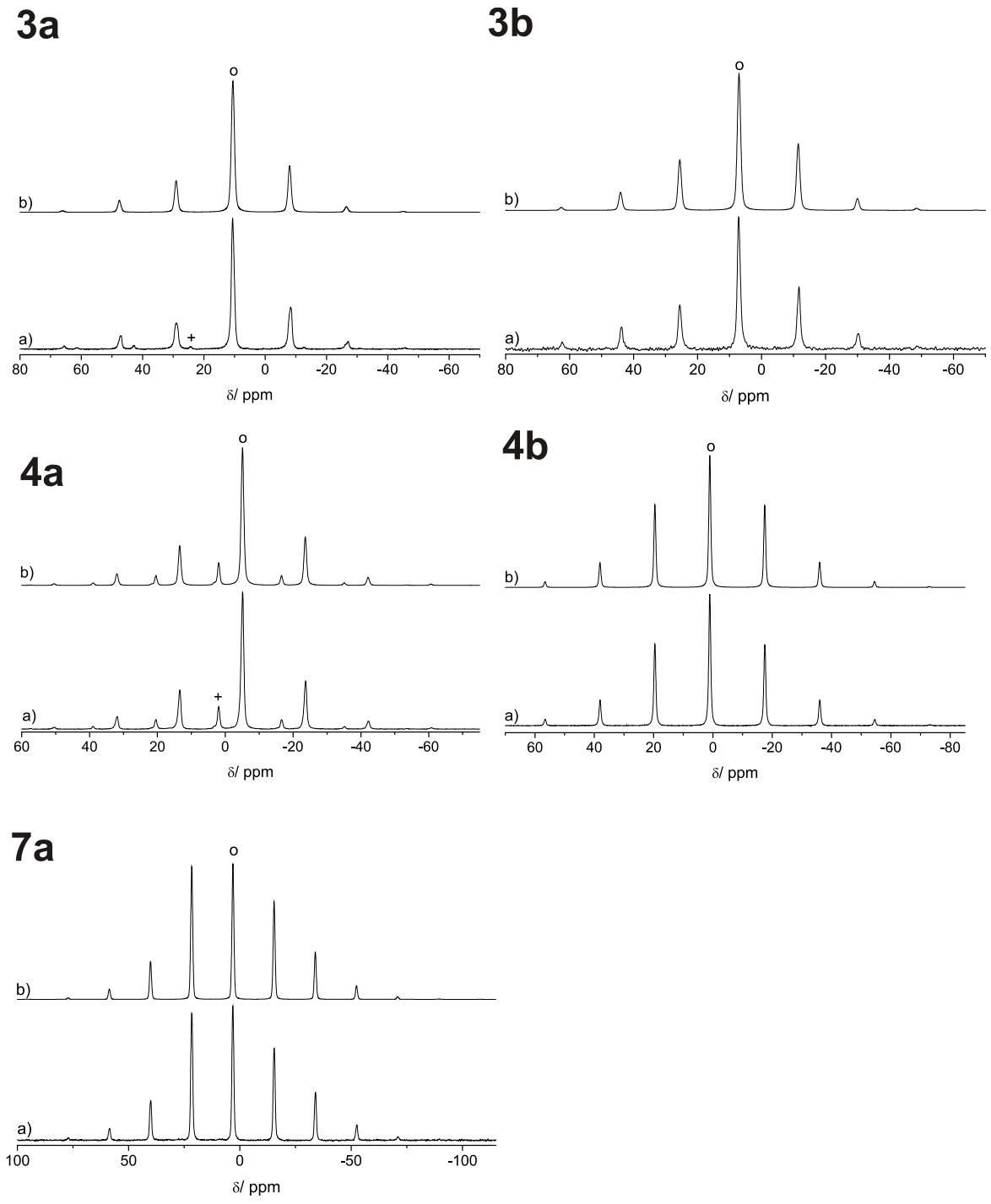


Figure S11: $^{31}P\{^{1}H\}$ slow-spinning CPMAS experiments acquired at 9.4 T with a spinning frequency of 3.0 kHz. The extracted CSA parameters are given in Table 2. o labels the MAS center bands and + impurities.

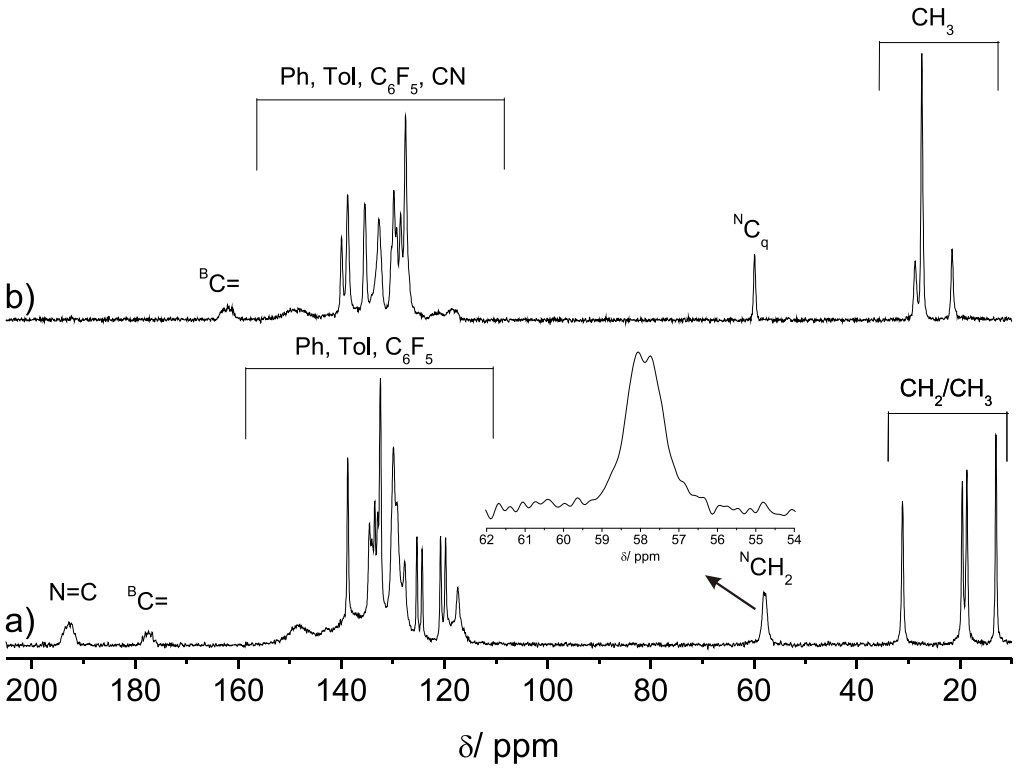


Figure S12a: $^{13}C\{^{1}H\}$ CPMAS NMR spectra of **7a** (a) and **4b** (b) acquired at 7.05 T with a spinning frequency of 10.0 kHz. The inset in (a) shows the doublet due to a $^{3}J(^{13}C\cdots^{31}P)$ spin-spin coupling for the C5 resonance of **7a**. Peak assignments are given in the Figure.

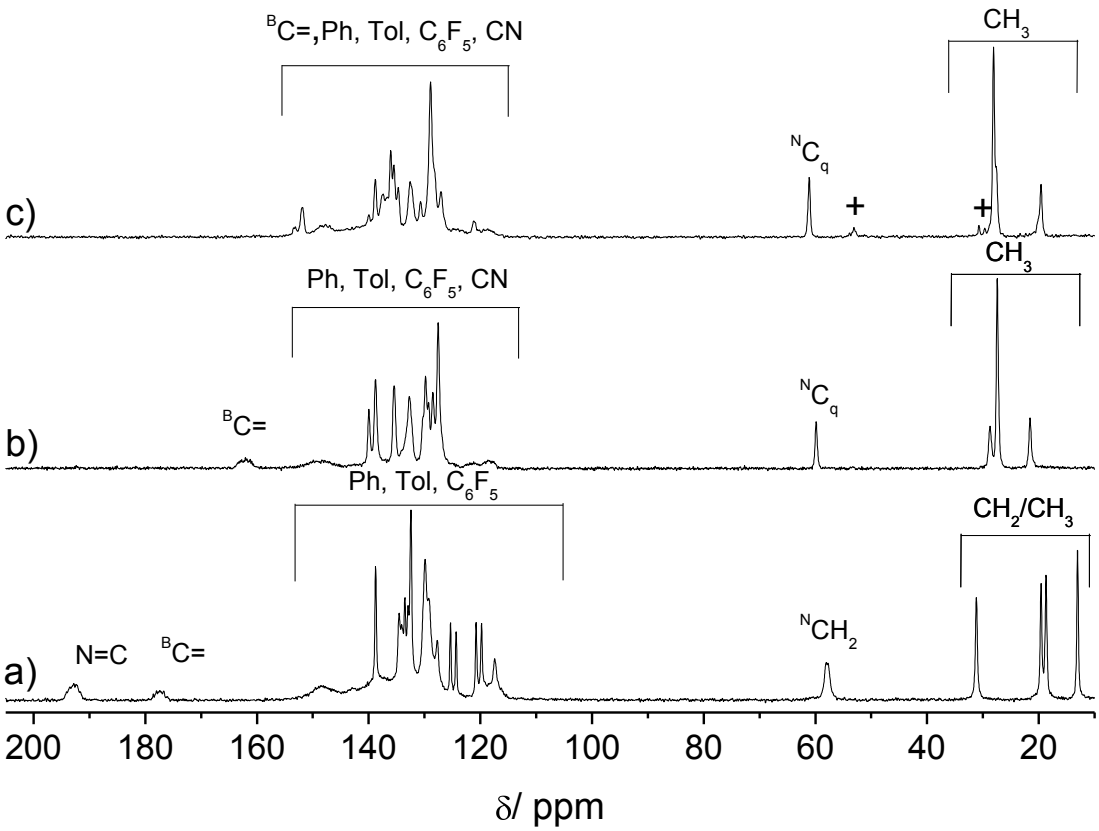


Figure S12b: $^{13}C\{^{1}H\}$ CPMAS NMR spectra of **7a** (a), **4b** (b), and **4a** (c) acquired at 7.05 T with a spinning frequency of 10.0 kHz. Peak assignments are given in the Figure.

	4b	**7a**
δ_{iso}(C3)	132.5	208.1
δ_{iso}(C5)	59.8	62.3
δ_{iso}(C2)	174.1	190.1
δ_{iso}(C1)	144.3	137.5
$\delta_{iso}(C^{i\text{-}Ph})$	140.3/146.2	123.0/127.3
$\delta_{iso}(C^{i\text{-}C6F5})$	121.6/124.2	121.9/ 122.4/ 121.4
$\delta_{iso}(C^{p\text{-}Tol})$	136.7	140.4
$\delta_{iso}(C^{i\text{-}Tol})$	143.0	136.3
$\delta_{iso}(C_6F_5)$	152.5/ 155.1/ 142.6/ 140.2/ 143.9/ 152.3/ 155.5/ 139.8/ 141.7/ 143.2	153.9/ 138.4/ 144.0/ 141.1/ 153.4/ 151.4/ 141.1/ 143.3/ 140.8/ 153.7/ 146.1/ 142.1/ 143.2/ 141.3/ 147.9

Table S13: DFT-calculated ^{13}C shifts (B3-LYP/def2-TZVP, referenced to TMS) for selected ^{13}C nuclei of **4b** and **7a.**

References

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Computational details

All calculations have been performed with the TURBOMOLE 6.3 suite of programs.[1] The structures have been optimized with the *meta*-GGA functional TPSS[2] applying the new D3-dispersion correction with Becke-Johnson damping (denoted as (BJ)).[3,4] Subsequent single point calculations have been carried out with the more accurate double hybrid density functional B2PLYP-D3(BJ) level.[5,6] For both calculations the large Gaussian-AO basis set def2-TZVP[7] and the RI approximation[8,9] have been used. The final level of theory can therefore be abbreviated as B2PLYP-D3(BJ)/def2-TZVP//TPSS-D3(BJ)/def2-TZVP and has an estimated accuracy of about 1-2 kcal/mol.
The thermodynamic corrections are based on harmonic vibrational frequencies calculated at the PM6-D3H level[10,11] as provided in the MOPAC2009 suite of programms.[12] Low-lying frequencies (effectively those below 100 cm^{-1}) are treated in a quasi-free-rotor approximation in order to avoid errors in the entropy calculation. These (free) enthalpy values are denoted $\Delta H(G)$, ΔE marks electronic energies (i.e., not including ZPVE).
For a more detailed description of solvent effects and the accurate treatment of thermodynamic corrections in solvent - here (free) enthalpies of solvation - the COSMO-RS program[13,14,15,16,17] in the parametrization for dichloromethane (CH_2Cl_2) has been used. Both, gas phase and solvational thermal corrections, have been computed once for a temperature of 298.15K (tables 3 and 4) and once for a temperature of 213.15 K (tables 5 and 6).
All values are given in kcal/mol.

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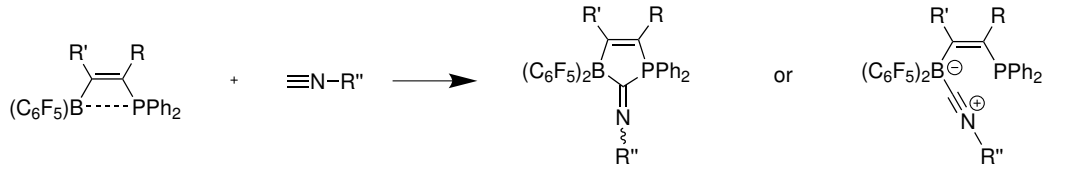

3a: R =ptol, R'=C_6F_5
3b: R =ptol, R'=Me

6a: R"= tBu
6b: R"= nBu

5a: R=ptol, R'=C_6F_5, R"=tBu
5b: R=ptol, R'=Me, R"=tBu
7a: R=ptol, R'=C_6F_5, R"=nBu
7b: R=ptol, R'=Me, R"=nBu

4a: R=ptol, R'=C_6F_5, R"=tBu
4b: R=ptol, R'=Me, R"=tBu
8a: R=ptol, R'=C_6F_5, R"=nBu
8b: R=ptol, R'=Me, R"=nBu

Tab.1: Electronic reaction energies and free reaction enthalpies at two different temperatures in the solvent dichloromethane.

			ΔE_{elec}	$\Delta G^{298.15}_{solution}$	$\Delta G^{213.15}_{solution}$
3a + **6**a	→	**4**a	-20.31	-3.90	-5.85
3a + **6**a	→	*E*-5a	-20.39	-1.75	-4.10
3a + **6**a	→	*Z*-5a	-14.06	3.90	1.44
3a + **6**b	→	**8**a	-19.81	-3.58	-5.56
3a + **6**b	→	*E*-7a	-26.96	-11.26	-13.34
3a + **6**b	→	*Z*-7a	-24.55	-8.15	-10.72
3b + **6**a	→	**4**b	-17.86	1.05	-1.36
3b + **6**a	→	*E*-5b	-18.47	-0.02	-2.47
3b + **6**a	→	*Z*-5b	-17.70	0.85	-1.67
3b + **6**b	→	**8**b	-17.45	0.54	-1.74
3b + **6**b	→	*E*-7b	-27.71	-9.08	-11.71
3b + **6**b	→	*Z*-7b	-26.73	-10.37	-12.84

Tab.2: Electronic reaction energies for the reactions with isonitrile.

			TPSS-D3(BJ)	B2PLYP-D3(BJ)
3a + **6**a	→	**4**a	-19.11	-20.31
3a + **6**a	→	*E*-5a	-19.76	-20.39
3a + **6**a	→	*Z*-5a	-15.82	-14.06
3a + **6**b	→	**8**a	-18.89	-19.81
3a + **6**b	→	*E*-7a	-26.49	-26.96
3a + **6**b	→	*Z*-7a	-25.76	-24.55
3b + **6**a	→	**4**b	-18.34	-17.86
3b + **6**a	→	*E*-5b	-18.88	-18.47
3b + **6**a	→	*Z*-5b	-19.66	-17.70
3b + **6**b	→	**8**b	-18.28	-17.45
3b + **6**b	→	*E*-7b	-28.00	-27.71
3b + **6**b	→	*Z*-7b	-27.82	-26.73

Tab.3: Thermodynamic corrections and reaction (free) enthalpies for the reactions with isonitrile in the gas phase at 298.15K.

			corrections		final B2PLYP-D3(BJ)	
			ΔH	ΔG	ΔH_{gas}	ΔG_{gas}
3a + **6**a	→	**4**a	1.59	15.78	-18.72	-4.53
3a + **6**a	→	*E*-5a	2.25	18.66	-18.14	-1.73
3a + **6**a	→	*Z*-5a	1.75	18.28	-12.31	4.22
3a + **6**b	→	**8**a	1.52	15.96	-18.29	-3.85
3a + **6**b	→	*E*-7a	2.14	16.78	-24.82	-10.18
3a + **6**b	→	*Z*-7a	1.81	18.23	-22.74	-6.32
3b + **6**a	→	**4**b	1.94	18.30	-15.92	0.44
3b + **6**a	→	*E*-5b	2.35	18.79	-16.12	0.32
3b + **6**a	→	*Z*-5b	1.93	18.67	-15.77	0.97
3b + **6**b	→	**8**b	2.05	17.88	-15.40	0.43
3b + **6**b	→	*E*-7b	2.45	19.40	-25.26	-8.31
3b + **6**b	→	*Z*-7b	2.03	18.26	-24.70	-8.47

Tab.4: Solvent corrections and reaction (free) enthalpies for the reactions with isonitrile in the solvent CH_2Cl_2 at 298.15K.

			corrections		final B2PLYP-D3(BJ)	
			ΔH_{solv}	ΔG_{solv}	$\Delta H_{solution}$	$\Delta G_{solution}$
3a + **6**a	→	**4**a	8.34	0.63	-10.38	-3.90
3a + **6**a	→	*E*-5a	7.95	-0.02	-10.19	-1.75
3a + **6**a	→	*Z*-5a	7.44	-0.32	-4.87	3.90
3a + **6**b	→	**8**a	7.98	0.27	-10.31	-3.58
3a + **6**b	→	*E*-7a	6.32	-1.03	-18.50	-11.26
3a + **6**b	→	*Z*-7a	5.43	-1.83	-17.31	-8.15
3b + **6**a	→	**4**b	8.30	0.61	-7.62	1.05
3b + **6**a	→	*E*-5b	7.27	-0.34	-8.85	-0.02
3b + **6**a	→	*Z*-5b	7.56	-0.12	-8.21	0.85
3b + **6**b	→	**8**b	7.72	0.11	-7.68	0.54
3b + **6**b	→	*E*-7b	6.55	-0.77	-18.71	-9.08
3b + **6**b	→	*Z*-7b	5.48	-1.90	-19.22	-10.37

Tab.5: Thermodynamic corrections and reaction (free) enthalpies for the reactions with isonitrile in the gas phase at 213.15K.

			corrections		final B2PLYP-D3(BJ)	
			ΔH	ΔG	ΔH_{gas}	ΔG_{gas}
3a + **6**a	→	**4**a	1.37	11.47	-18.94	-8.84
3a + **6**a	→	*E*-5a	2.23	13.85	-18.16	-6.54
3a + **6**a	→	*Z*-5a	1.70	13.44	-12.36	-0.62
3a + **6**b	→	**8**a	1.30	11.62	-18.51	-8.19
3a + **6**b	→	*E*-7a	2.10	12.42	-24.86	-14.54
3a + **6**b	→	*Z*-7a	1.78	13.46	-22.77	-11.09
3b + **6**a	→	**4**b	1.90	13.54	-15.96	-4.32
3b + **6**a	→	*E*-5b	2.36	14.01	-16.11	-4.46
3b + **6**a	→	*Z*-5b	1.90	13.80	-15.80	-3.90
3b + **6**b	→	**8**b	2.03	13.27	-15.42	-4.18
3b + **6**b	→	*E*-7b	2.47	14.54	-25.24	-13.17
3b + **6**b	→	*Z*-7b	2.01	13.55	-24.72	-13.18

Tab.6: Solvent corrections and reaction (free) enthalpies for the reactions with isonitrile in the solvent CH_2Cl_2 at 213.15K.

			corrections		final B2PLYP-D3(BJ)	
			ΔH_{solv}	ΔG_{solv}	$\Delta H_{solution}$	$\Delta G_{solution}$
3a + **6**a	→	**4**a	9.33	2.99	-9.61	-5.85
3a + **6**a	→	*E*-5a	9.08	2.44	-9.08	-4.10
3a + **6**a	→	*Z*-5a	8.46	2.06	-3.90	1.44
3a + **6**b	→	**8**a	8.96	2.63	-9.55	-5.56
3a + **6**b	→	*E*-7a	7.19	1.20	-17.67	-13.34
3a + **6**b	→	*Z*-7a	6.22	0.37	-16.55	-10.72
3b + **6**a	→	**4**b	9.31	2.96	-6.65	-1.36
3b + **6**a	→	*E*-5b	8.29	1.99	-7.82	-2.47
3b + **6**a	→	*Z*-5b	8.56	2.23	-7.24	-1.67
3b + **6**b	→	**8**b	8.70	2.44	-6.72	-1.74
3b + **6**b	→	*E*-7b	7.41	1.46	-17.83	-11.71
3b + **6**b	→	*Z*-7b	6.29	0.34	-18.43	-12.84

TPSS-D3/def2-TZVP-optimized coordinates of all structures (in bohr):

3a

-0.60165574826045 -2.90808374615756 1.15876098989443 p
4.58322564891832 3.40201297202432 2.63820936507410 f
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1.29479041564867 0.72337419033500 -0.66115156073753 c
2.32505567089077 -1.31149855427183 0.55800206844840 c
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5.48543230812693 -3.35410535744235 3.38393627932374 c
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-1.76996483590559 0.16825326117849 -0.84855161167574 b

3b
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