

Electronic Supplementary Information

**Convenient access to the tricyanoborate dianion $\text{B}(\text{CN})_3^{2-}$ and
selected reactions as a boron-centred nucleophile**

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Experimental Section

Reactions involving air-sensitive compounds were performed either in 100 or 250 mL round bottom flasks or in 20 or 60 mL glass tubes equipped with valves with PTFE stems (Young, London) under argon using standard Schlenk line techniques. ^1H , ^{11}B , ^{19}F and ^{13}C NMR spectra were recorded at 25 °C either in $(\text{CD}_3)_2\text{CO}$ or D_2O on a Bruker Avance 500 spectrometer, a Bruker Avance III 400 spectrometer, a Bruker Avance III HD 300 or on a Bruker DRX 200 spectrometer. The NMR signals were referenced against TMS (^1H and ^{13}C), $\text{BF}_3 \cdot \text{OEt}_2$ in CDCl_3 with $\Xi(^{11}\text{B}) = 32.083974$ MHz and CFCl_3 with $\Xi(^{19}\text{F}) = 94.094011$ MHz as external standards.¹ ^1H and ^{13}C chemical shifts were calibrated against the residual solvent signal and the solvent signal, respectively ($\delta(^1\text{H})$: $(\text{CD}_2\text{H})(\text{CD}_3)\text{CO}$ 2.05 ppm, HDO 4.79 ppm; $\delta(^{13}\text{C})$: $(\text{CD}_3)_2\text{CO}$ 206.26 and 29.84 ppm).² Solid-state ^{11}B MAS rotor-synchronized Hahn-echo and ^{13}C MAS as well as ^{19}F MAS single-pulse NMR spectra were recorded at 22 °C with a Bruker DSX-400 NMR spectrometer with bottom layer rotors of ZrO_2 (diameter 4 mm) containing approximately 30–50 mg of sample (^{13}C , 100.15 MHz; ^{11}B , 128.38 MHz; ^{19}F , 376.47 MHz). The measurements were conducted with a spinning rate of 15 kHz. All chemical shifts were calibrated by setting the ^{13}C low-field signal of adamantane to $\delta = 38.48$ ppm according to the IUPAC recommendations¹ with $\Xi(^{13}\text{C}) = 25.145020$ MHz, $\Xi(^{11}\text{B}) = 32.083974$ MHz and $\Xi(^{19}\text{F}) = 94.094011$ MHz. After correction of the baseline of the ^{11}B MAS NMR spectrum of $\text{K}_2\text{1} \cdot \text{KF}$ the spectrum was fitted with the solids line shape analysis module as implemented in the Bruker TopSpinTM 3.2 NMR software package. IR spectra were measured in the attenuated total reflection (ATR) mode in the region of $4000\text{--}400$ cm^{-1} with an apodized resolution of 1 cm^{-1} with a Bruker Alpha spectrometer equipped with a Bruker diamond single reflection ATR system. Raman spectra were measured using the 1064 nm excitation line of a Nd/YAG laser on crystalline samples contained in melting point capillaries in the region of $3500\text{--}80$ cm^{-1} at room temperature on a Bruker IFS-120 spectrometer with an apodized resolution of 1 cm^{-1} . MALDI mass spectra were acquired on an Autoflex II LRF (Bruker Daltonics). Elemental analysis (C, H, N) were performed either with a Euro EA3000 instrument (HEKA-Tech, Germany) or with a vario MICRO cube (Elementar Analysensysteme, Germany).

Chemicals

All standard chemicals were obtained from commercial sources and used without further purification. Solvents were dried according to standard protocols³ and stored in flasks equipped with valves with PTFE stems (Young, London) under an argon atmosphere. $\text{K}[\text{BF}(\text{CN})_3]$ was synthesized according to known procedures starting from $\text{Na}[\text{BF}_4]$ and $(\text{CH}_3)_3\text{SiCN}$.⁴

Quantum Chemical Calculations

Density functional calculations (DFT)⁵ using the hybrid functional PBE0⁶ and def2-TZVPP basis sets⁷ were performed with the Turbomole V6.5 program package.⁸

Crystal Structure Determination of K3·0.5(CH₃)₂CO, K4·0.5THF, K5 and K6

Colourless crystals of K[BH(CN)₃]·0.5(CH₃)₂CO (K3·0.5(CH₃)₂CO), K[EtB(CN)₃]·0.5THF (K4·0.5THF), K[CH₂=CHCH₂B(CN)₃] (K5) and K[C₆F₅B(CN)₃] (K6) were obtained from solutions in acetone (K3·0.5(CH₃)₂CO and K5) and THF (K4·0.5THF and K6) by slow evaporation of the solvent, respectively. Crystals of K3·0.5(CH₃)₂CO, K4·0.5THF and K5 were investigated with a Bruker X8-Apex II diffractometer, a second crystal of K5 was studied with an Gemini E Ultra Xcalibur diffractometer equipped with an EOS detector and a crystal of K6 was studied with a Stoe IPDS I diffractometer using Mo-K α radiation ($\lambda = 0.71073 \text{ \AA}$) in all cases. All structures were solved either by direct (SHELXS)⁹ or by intrinsic phasing methods (SHELXT).^{9a, 10} Refinements are based on full-matrix least-squares calculations on F^2 .^{9a, 11} All non-hydrogen atoms were refined anisotropically. The positions of all hydrogen atoms were located from electron density difference maps. In the final steps of the refinement idealized bond lengths and angles were introduced. Calculations were performed either with the ShelXle graphical interface¹² or with the WinGX program package.¹³ Molecular structure diagrams were drawn with the program Diamond 3.2i.¹⁴ Experimental details, crystal data, and the CCDC numbers are collected in Table S1. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Table S1. Selected Crystal Data and Details of the Refinement of the Crystal Structures of K[BH(CN)₃]·0.5(CH₃)₂CO (K3·0.5(CH₃)₂CO), K[EtB(CN)₃]·0.5THF (K4·0.5THF), K[CH₂=CHCH₂B(CN)₃] (K5) and K[C₆F₅B(CN)₃] (K6).

	K3·0.5(CH ₃) ₂ CO	K4·0.5THF	K5 ^a	K5 ^b	K6
Formula	C ₉ H ₈ B ₂ K ₂ N ₆ O	C ₂₈ H ₃₆ B ₄ K ₄ N ₁₂ O ₂	C ₆ H ₅ BKN ₃	C ₆ H ₅ BKN ₃	C ₉ BF ₅ KN ₃
M_w	316.03	771.32	169.04	169.04	295.03
T (K)	100	100	150	150	123
Crystal system	monoclinic	monoclinic	triclinic	triclinic	monoclinic
Space group	$P2_1/n$	$P2_1/c$	$P-1$	$P-1$	$P2_1/c$
a (Å)	9.8586(5)	7.5693(15)	9.5267(4)	9.5044(12)	11.894(2)
b (Å)	13.1307(6)	25.062(5)	9.9704(5)	9.9760(13)	12.782(3)
c (Å)	12.1811(6)	22.116(4)	10.1727(5)	10.1345(13)	7.5458(15)
α			81.041(4)	81.033(3)	
β	93.991(2)	94.30(3)	68.165(4)	68.217(3)	103.98(3)
γ			83.636(4)	83.514(3)	
volume (Å ³)	1573.02(13)	4183.6(15)	884.51(7)	879.8(2)	1113.2(4)
Z	4	4	4	4	4
$d(\text{calcd})$ (Mg m ⁻³)	1.334	1.226	1.269	1.276	1.760
μ (mm ⁻¹)	0.603	0.466	0.537	0.540	0.530
$F(000)$	640	1600	344	344	576
No. of collected reflections	21007	49858	10670	16314	9150
No. of unique reflections	3268	8232	5745	3455	1955
$R(\text{int})$	0.018	0.190	0.035	0.021	0.040
No. of parameters / restraints	250 / 32	455 / 12	233 / 20	199 / 0	172 / 0
$R1$ ($I > 2\sigma(I)$)	0.020	0.063	0.032	0.027	0.033
$wR2$ (all)	0.055	0.159	0.079	0.071	0.083
GOF on F^2	1.050	0.906	1.029	1.054	1.017
Largest diff. peak / hole / e Å ⁻³	0.240 / -0.171	1.259 / -0.516	0.299 / -0.326	0.685 / -0.272	0.334 / -0.507
CCDC no.	1039994	1039991	1031561	1039993	1039992

^a Gemini E Ultra Xcalibur diffractometer equipped with an EOS detector. ^b Bruker X8-Apex II diffractometer.

Syntheses

K₂B(CN)₃·KF (K₂1·KF) Method A from K in NH₃: Potassium (2.90 g, 74.2 mmol) was dissolved in liquid ammonia (25 mL) at $-78\text{ }^{\circ}\text{C}$. K[BF(CN)₃] (K2, 5.00 g, 34.0 mmol) was added carefully in several portions over a period of 1–2 hours. Solid yellow K₂B(CN)₃ formed instantaneously and the reaction mixture turned green. The reaction mixture was stirred for one additional hour at $-78\text{ }^{\circ}\text{C}$. The mixture was allowed to warm to room temperature within 5 hours, which resulted in the evaporation of the ammonia. The residue was dried in a vacuum, suspended in THF (20 mL) and filtered under argon. The solid K₂B(CN)₃·KF (K₂1·KF) was washed with THF ($3 \times 10\text{ mL}$) and dried in a vacuum and the yellow powder was stored in a glove box. Yield: 6.85 g (30.4 mmol, 89%).

Elemental analysis: calculated C₃BFK₃N₃: C: 16.00%, N: 18.66%. Found: C: 15.74%, N: 18.43%.

¹¹B MAS NMR (128.38 MHz): $\delta_{\text{iso}} = -43.4\text{ ppm}$ ($C_{\text{quad}} = 1.01\text{ MHz}$, $\eta_{\text{quad}} = 0.19$).

¹³C MAS NMR (100.15 MHz): $\delta_{\text{iso}} = 169.4\text{ ppm}$.

¹⁹F MAS NMR (376.47 MHz): $\delta_{\text{iso}} = -133.7\text{ ppm}$.

IR (ATR): $\tilde{\nu} = 2037\text{ (m, sh, br)}$, $2020\text{ (s, sh, br, } \nu_{\text{asym}}(\text{CN}))$, $2003\text{ cm}^{-1}\text{ (vs, br, } \nu_{\text{asym}}(\text{CN}))$.

Raman: $\tilde{\nu} = 2098\text{ (s, } \nu_{\text{sym}}(\text{CN}))$, 2040 (m, sh) , 2032 (m, sh) , $2022\text{ (s, } \nu_{\text{asym}}(\text{CN}))$, $1997\text{ cm}^{-1}\text{ (s, } \nu_{\text{asym}}(\text{CN}))$.

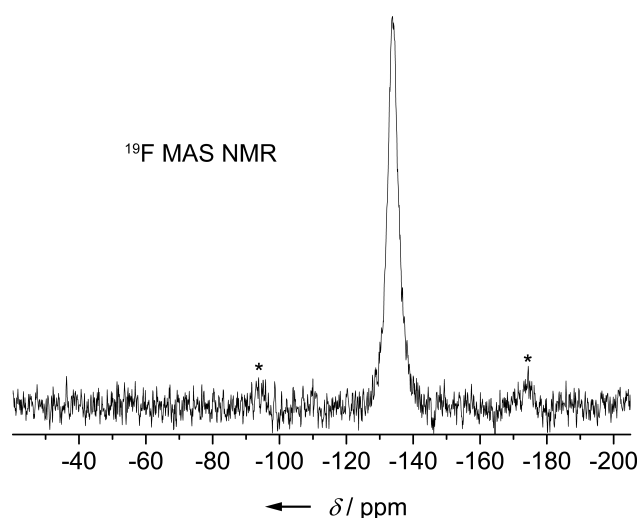


Figure S1. ¹⁹F MAS NMR spectrum of K₂B(CN)₃·KF (K₂1·KF) (asterisks denote spinning side bands). $\delta_{\text{iso}}(^{19}\text{F})$ of -133.7 ppm is similar to the value reported for neat KF of -136 ppm .¹⁵

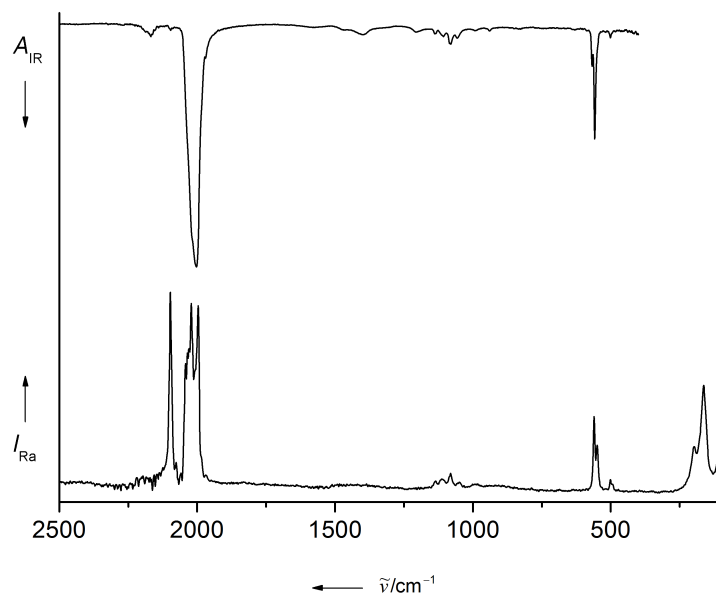


Figure S2. IR and Raman spectrum of $\text{K}_2\text{B}(\text{CN})_3 \cdot \text{KF}$ ($\text{K}_2\mathbf{1} \cdot \text{KF}$).

$\text{K}_2\text{B}(\text{CN})_3 \cdot \text{KF}$ ($\text{K}_2\mathbf{1} \cdot \text{KF}$) Method B from K/naphthalene in THF: $\text{K}[\text{BF}(\text{CN})_3]$ (K_2) (2.00 g, 13.6 mmol) was dissolved in THF (20 mL) and excess potassium (2.00 g, 51.2 mmol) was added in small pieces. Naphthalene (150 mg, 1.17 mmol) was dissolved in THF (5 mL) and added to the reaction mixture in one portion. The suspension was stirred for 2 hours at room temperature. Unreacted potassium was separated from the green suspension and washed with THF (5 mL). The combined suspensions were filtered off under argon and the remainder was washed with THF (2×5 mL). The dark green solid was dried in a vacuum and stored in a glove box. Yield: 3.23 g. The crude product contained a small amount of THF and potassium naphthalide as assessed by $^1\text{H}\{^{11}\text{B}\}$ NMR spectra recorded on solution in $(\text{CD}_3)_2\text{CO}$ after treatment of a small sample of the product with H_2O . However, the purity of the material was sufficient for subsequent reactions with electrophiles similar to the $\text{K}_2\text{B}(\text{CN})_3 \cdot \text{KF}$ ($\text{K}_2\mathbf{1} \cdot \text{KF}$) obtained via method A.

$\text{K}[\text{BH}(\text{CN})_3]$ (K_3) method A: A three necked round-bottom flask equipped with a mechanical stirrer and a connection tube to an argon line was charged with potassium (23.7 g, 607 mmol) and liquid ammonia (250 mL) at -78 °C. K_2 (41.0 g, 279 mmol) was carefully added in several portions during 1–2 hours. In the course of the addition yellow $\text{K}_2\mathbf{1} \cdot \text{KF}$ formed and the colour of the reaction mixture turned green. The mixture was stirred for an additional hour at -78 °C. The mixture was warmed to room temperature within 5 hours and the ammonia was allowed to evaporate. The residue was dried in a vacuum and subsequently washed with THF (3×100 mL) in an argon atmosphere. The solid remainder was suspended in THF (500 mL) and a mixture of water (11.0 g, 610 mmol) in THF (50 mL) was slowly added resulting in the decolourization of the reaction mixture. The mixture was dried with K_2CO_3 and the solid material was filtered off. The volume of the solution was reduced to 50

mL and colourless **K3** precipitated upon addition of CH_2Cl_2 . Yield: 31.8 g (247 mmol, 89%, purity >99.5% according to ^{11}B NMR spectroscopy). [Evaporation of the solvent of the filtrate obtained during isolation of $\text{K}[\text{BH}(\text{CN})_3]$ (**K3**) yielded 2.40 g of a mixture of $\text{K}[\text{BH}(\text{CN})_3]$ (**K3**) (70%) and $\text{K}[\text{BH}_2(\text{CN})_2]$ (30%).]

Elemental analysis: calculated C_3HBKN_3 : C: 27.93%, H: 0.97%, N: 32.54%. Found: C: 27.94%, H: 0.78%, N: 32.58%.

^{11}B NMR (160.46 MHz, $(\text{CD}_3)_2\text{CO}$): $\delta = -40.0$ ppm (d, $^1J(^{11}\text{B}, ^1\text{H}) = 97.0$ Hz, BH, 1B).

^1H NMR (500.13 MHz, $(\text{CD}_3)_2\text{CO}$): $\delta = 1.77$ ppm (q, $^1J(^{11}\text{B}, ^1\text{H}) = 97.0$ Hz, BH, 1H).

$^{13}\text{C}\{^1\text{H}\}$ NMR (125.76 MHz, $(\text{CD}_3)_2\text{CO}$): $\delta = 128.0$ ppm (q, $^1J(^{13}\text{C}, ^{11}\text{B}) = 65.8$ Hz, BCN, 3C).

Raman: $\tilde{\nu} = 2461$ (m, $\nu(\text{BH})$), 2221 cm^{-1} (s, $\nu(\text{CN})$).

$\text{K}[\text{BH}(\text{CN})_3]$ (K3**) method B:** $\text{K}[\text{BF}(\text{CN})_3]$ (**K2**) (25.0 g, 170 mmol) was placed in a three necked round-bottom flask equipped with a mechanical stirrer and it was dissolved in THF (500 mL). Potassium (25.0 g, 63.9 mmol) was added in small pieces and subsequently a solution of naphthalene (2.00 g, 15.6 mmol) in THF (10 mL) was added in a single portion. The suspension was stirred for 6 hours at room temperature. Excess potassium was separated from the green suspension and washed with THF (2×50 mL). A mixture of water (6.0 g, 333 mmol) and THF (15 mL) was slowly added until the suspension became colourless. The suspension was filtered off, washed with THF (2×20 mL) and dried over K_2CO_3 . The volume of the combined organic layers was reduced to 50 mL and colourless $\text{K}[\text{BH}(\text{CN})_3]$ (**K3**) precipitated upon addition of CH_2Cl_2 . Yield: 15.0 g (116 mmol, 68%, purity >99.5% according to ^{11}B NMR spectroscopy).

The analytical data are consistent to those given for the previous example.

$\text{K}[\text{BH}(\text{CN})_3]$ (K3**) method C:** $\text{Na}[\text{BF}(\text{CN})_3]$ (**Na2**) (3.75 g, 28.6 mmol) was dissolved in liquid ammonia (40 mL) at -78 °C. Sodium (1.32 g, 57.4 mmol) was carefully added to the reaction mixture. Subsequently, the mixture was allowed to warm to room temperature, which resulted in the evaporation of the ammonia. The residue was dissolved in a mixture of H_2O (50 mL) and THF (200 mL) at 0 °C and K_2CO_3 (ca. 5 g) was added until two layers had formed. The aqueous layer was separated and saturated with K_2CO_3 (ca. 50 g). The viscous solution was extracted with THF (3×50 mL). The combined organic layers were dried with K_2CO_3 and concentrated to a volume of 10 mL. Colourless $\text{K}[\text{BH}(\text{CN})_3]$ (**K3**) precipitated upon addition of CH_2Cl_2 . Yield: 2.38 g (18.5 mmol, 65%, purity >99.5% according to ^{11}B NMR spectroscopy).

The analytical data are consistent to those given for the previous example.

Comparative study on the reduction of **K2 with Li, Na and K:** $\text{K}[\text{BF}(\text{CN})_3]$ (**K2**) (150 mg, 1.02 mmol) was dissolved in THF (7 mL) and an excess of the respective alkali metal, Li (200 mg, 28.8 mmol), Na (400 mg, 17.4 mmol) and K (550 mg, 14.1 mmol) was added in small pieces

(approximately 10). Naphthalene (30.0 mg, 0.234 mmol) dissolved in THF (2 mL) was added to each reaction mixture in a single portion and the suspensions were stirred at room temperature. The progress of the reactions was monitored via ^{11}B NMR spectroscopy on small amounts of the suspension dissolved in H_2O . The reduction was complete after 2 days (Li), 15 hours (Na) and 0.5 hours (K), respectively.

$\text{K}[\text{C}_6\text{F}_5\text{B}(\text{CN})_3]$ (K6): $\text{K}[\text{BF}(\text{CN})_3]$ (K2) (500 mg, 3.40 mmol) and naphthalene (30.0 mg, 0.234 mmol) were dissolved in THF (7 mL). Small pieces of potassium (500 mg, 12.8 mmol) were added and the reaction mixture was stirred overnight at ambient temperature. Excess potassium was removed and hexafluorobenzene (1.5 mL, 13.0 mmol) was added at $0\text{ }^\circ\text{C}$ with a syringe. The mixture was decanted and the solid residue was washed with THF (2×10 mL). The combined THF layers were concentrated to a volume of 10 mL and pure off-white $\text{K}[\text{C}_6\text{F}_5\text{B}(\text{CN})_3]$ (K6) precipitated upon addition of CH_2Cl_2 . Yield: 670 mg (2.27 mmol, 67%, purity >98% according to ^{11}B NMR spectroscopy).

Elemental analysis: calculated $\text{C}_9\text{BF}_5\text{KN}_3$: C: 36.64%, N: 14.24%. Found: C: 35.19%, N: 13.57%.

^{11}B NMR (96.31 MHz, $(\text{CD}_3)_2\text{CO}$): $\delta = -33.9$ ppm (tt, $^3J(^{19}\text{F}, ^{11}\text{B}) = 7.6$ Hz, $^4J(^{19}\text{F}, ^{11}\text{B}) \approx 2.6$ Hz, 1B).

$^{13}\text{C}\{^{11}\text{B}\}$ NMR (75.49 MHz, $(\text{CD}_3)_2\text{CO}$): $\delta = 148.4$ (dddd, $^1J(^{19}\text{F}, ^{13}\text{C}) = 243.9$ Hz, $^nJ(^{19}\text{F}, ^{13}\text{C}) = 13.6$ Hz, 9.9 Hz, 4.1 Hz, 4.1 Hz, CF, 2C), 141.1 (dtt, $^1J(^{19}\text{F}, ^{13}\text{C}) = 250.1$ Hz, $^nJ(^{19}\text{F}, ^{13}\text{C}) = 20.4$ Hz, 5.7 Hz, C_pF , 1C), 137.9 (dm, $^1J(^{19}\text{F}, ^{13}\text{C}) = 248.4$ Hz, CF, 2C), 126.7 (s, CN, 3C), 111.6 ppm (tdt, $^nJ(^{19}\text{F}, ^{13}\text{C}) = 36.3$ Hz, 3.2 Hz, 3.2 Hz, C_iB).

$^{19}\text{F}\{^{11}\text{B}\}$ NMR (470.59 MHz, $(\text{CD}_3)_2\text{CO}$): $\delta = -132.1$ (m, 2F), -158.5 (tt, $^3J(^{19}\text{F}, ^{19}\text{F}) = 20$ Hz, $^4J(^{19}\text{F}, ^{19}\text{F}) \approx 2$ Hz, 1F), -165.4 (m, 2F).

$\text{K}[\text{C}_2\text{H}_5\text{B}(\text{CN})_3]$ (K4): The synthesis of $\text{K}[\text{C}_2\text{H}_5\text{B}(\text{CN})_3]$ (K4) was performed similar to the preparation of $\text{K}[\text{C}_6\text{F}_5\text{B}(\text{CN})_3]$ (K6) using ethyl iodide instead of hexafluorobenzene. Yield: 170 mg (1.72 mmol, 51%).

Elemental analysis: calculated $\text{C}_5\text{H}_5\text{BKN}_3$: C: 38.25%, H: 3.21%, N: 26.76%. Found: C: 38.30%, H: 3.29%, N: 26.37%.

^{11}B NMR (160.46 MHz, $(\text{CD}_3)_2\text{CO}$): $\delta = -30.0$ ppm (tq, $^2J(^{11}\text{B}, ^1\text{H}) \approx ^3J(^{11}\text{B}, ^1\text{H}) \approx 5$ Hz, 1B).

^1H NMR (500.13 MHz, $(\text{CD}_3)_2\text{CO}$): $\delta = 0.91$ (tq, br, $^3J(^1\text{H}, ^1\text{H}) \approx 7.6$ Hz, $^3J(^{11}\text{B}, ^1\text{H}) \approx 5$ Hz, CH_3 , 3H), 0.44 ppm (qq, br, $^3J(^1\text{H}, ^1\text{H}) \approx 7.6$ Hz, $^2J(^{11}\text{B}, ^1\text{H}) \approx 5$ Hz, CH_2 , 2H).

$^1\text{H}\{^{11}\text{B}\}$ NMR (500.13 MHz, $(\text{CD}_3)_2\text{CO}$): $\delta = 0.91$ (t, $^3J(^1\text{H}, ^1\text{H}) = 7.6$ Hz, CH_3 , 3H), 0.44 ppm (q, $^3J(^1\text{H}, ^1\text{H}) = 7.6$ Hz, CH_2 , 2H).

$^{13}\text{C}\{^1\text{H}\}$ NMR (125.77 MHz, $(\text{CD}_3)_2\text{CO}$): $\delta = 130.6$ (q, br, $^1J(^{13}\text{C}, ^{11}\text{B}) \approx 64$ Hz, BCN, 3C), 13.2 (q, br, $^1J(^{13}\text{C}, ^{11}\text{B}) \approx 45$ Hz, BCH_2 , 1C), 11.5 ppm (s, CH_3 , 1C).

$^{13}\text{C}\{^{11}\text{B}\}$ NMR (75.48 Hz, $(\text{CD}_3)_2\text{CO}$): $\delta = 130.6$ (s, BCN, 3C), 13.2 (s, BCH_2 , 1C), 11.5 ppm (s, CH_3 , 1C).

IR(ATR): $\tilde{\nu} = 2215\text{ cm}^{-1}$ (s, $\nu(\text{CN})$).

K[CH₂=CHCH₂B(CN)₃] (K5): The synthesis of K[CH₂=CHCH₂B(CN)₃] (K5) was performed as described for the preparation of K[C₆F₅B(CN)₃] (K6) using either CH₂=CHCH₂Cl or CH₂=CHCH₂Br instead of hexafluorobenzene. Yield of the synthesis starting from CH₂=CHCH₂Cl: 170 mg (1.01 mmol, 45%, purity >85% according to ¹¹B NMR spectroscopy). Yield of the synthesis starting from CH₂=CHCH₂Br: 115 mg (0.68 mmol, 37%, purity >85% according to ¹¹B NMR spectroscopy). The side product was found to be K3. A few crystals of pure K5 were obtained by recrystallization from acetone and characterized by single-crystal X-ray diffraction.

¹¹B NMR (160.46 MHz, (CD₃)₂CO): δ = -30.8 ppm (t, br, ²J(¹¹B, ¹H) ≈ 6 Hz, 1B).

¹H NMR (500.13 MHz, (CD₃)₂CO): δ = 5.84 (ddt, ³J(¹H, ¹H) = 17.0 Hz, 10.1 Hz, 7.7 Hz, =CH-, 1H), 4.85 (ddt, ³J(¹H, ¹H) = 17.0 Hz, ²J(¹H, ¹H) = 2.6 Hz, ⁴J(¹H, ¹H) = 1.4 Hz, =CH₂, 1H), 4.75 (ddt, ³J(¹H, ¹H) = 10.1 Hz, ²J(¹H, ¹H) = 2.6 Hz, ⁴J(¹H, ¹H) = 1.2 Hz, =CH₂, 1H), 1.33 ppm (m, BCH₂, 2H).

¹H{¹¹B} NMR (500.13 MHz, (CD₃)₂CO): δ = 5.84 (ddt, ³J(¹H, ¹H) = 17.0 Hz, 10.1 Hz, 7.7 Hz, =CH-, 1H), 4.85 (ddt, ³J(¹H, ¹H) = 17.0 Hz, ²J(¹H, ¹H) = 2.6 Hz, ⁴J(¹H, ¹H) = 1.5 Hz, =CH₂, 1H), 4.75 (ddt, ³J(¹H, ¹H) = 10.1 Hz, ²J(¹H, ¹H) = 2.6 Hz, ⁴J(¹H, ¹H) = 1.1 Hz, =CH₂, 1H), 1.33 ppm (ddd, ³J(¹H, ¹H) = 7.7 Hz, ⁴J(¹H, ¹H) = 1.5 Hz, ⁴J(¹H, ¹H) = 1.1 Hz, BCH₂, 2H).

¹³C{¹H} NMR (125.77 MHz, (CD₃)₂CO): δ = 139.8 (s, br, =CH-, 1C), 129.9 (q, ¹J(¹³C, ¹¹B) ≈ 66 Hz, BCN, 3C), 112.8 (s, =CH₂, 1C), 27.5 ppm (q, ¹J(¹³C, ¹¹B) ≈ 43 Hz, BCH₂, 1C).

IR(ATR): $\tilde{\nu}$ = 2219 cm⁻¹ (s, ν(CN)).

References

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