Synthesis of Functionalized Borate Building Blocks for the Anionic Derivatization of Neutral Compounds

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Abstract: Mono- and bifunctional fluorinated borate building blocks were prepared in three to five steps with good to excellent overall yields. Compounds with both nucleophilic and electrophilic functionalities are presented, which can be used for the anionic derivatization of neutral molecules.

Key words: boron, fluorine, organometallic reagents, anions

Fluorinated tetraarylborates constitute an easily accessible class of weakly coordinating anions (WCA), which have found widespread use in the synthesis of highly efficient single-site olefin polymerization catalysts. In addition, they were successfully applied in catalytic asymmetric transformations such as the iridium-catalyzed hydrogenation of unfunctionalized alkenes and Diels–Alder reactions. In most cases, the well-established representatives tetrakis(pentafluorophenyl)borate or tetraakis[3,5-bis(trifluoromethyl)phenyl]borate were employed. Furthermore, related anions with four identical aryl moieties at the boron center have recently been developed. However, there are only a few examples of borates having the composition \([\text{B}(\text{Ar})_3(\text{R})^-]\) with different boron substituents, and only simple alkyl derivatives \([R = \text{Me}, \text{Et}, \text{i-Pr}, \text{n-Bu}, \text{CH}_2\text{C}(\text{CH}_3)_3]\) and the phenyl analogue \((R = \text{Ph})\) have been described in detail.

In principle, the synthesis of \([\text{B}(\text{Ar})_3(\text{R})^-]\) species can be accomplished via two synthetic pathways (Scheme 1), differing in the order of steps through which the two substituents at the boron center are introduced.

![Scheme 1](image)

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If the borane \(\text{B}(\text{Ar})_3\) is easily accessible, the desired borate can be obtained according to pathway A, in one step, by addition of the appropriate metal organyl compound. Alternatively, a neutral \((\text{RBX}_2)\) or anionic intermediate \((\text{RBX}_3^-)\) can be generated first and subsequently transformed into the borate by reaction with the corresponding aryl reagent according to pathway B.

In the course of our studies concerning the effect of the counter-ion in iridium-catalyzed asymmetric hydrogenation reactions, functionalized borate building blocks were required to prepare anionic derivatives of neutral ligands. In this report, we describe the synthesis of a series of functionalized borates bearing nucleophilic or electrophilic functional groups, which can be used for the anionic functionalization of neutral compounds.

Figure 1 Functionalized borates with two different linkers

Initial attempts to prepare compounds of type 1 with a benzyl linker derived from the commercially available tris(pentafluorophenyl)borane (Figure 1) via pathway A, proved unsuccessful. The intermediate borates suffered from partial decomposition, presumably by proton-induced deborylation of the benzyl moiety, even under only slightly acidic conditions, for example during silica gel chromatography. Therefore, we turned our attention to the corresponding derivatives 2 with perfluorinated linkers. We assumed that the reduced π-basicity of these compounds would slow down the acid-driven cleavage of the carbon–boron bond and thus enable chromatographic purification.

Starting from the known benzyl alcohol 3, prepared in two steps from pentafluorobenzaldehyde (see experimental section), the silyl ethers 4 and 5 were synthesized (Scheme 2). While the tert-butyltrimethylsilyl ether 4 was obtained in virtually quantitative yield, protection with tert-butyl(pentafluorophenyl)silyl chloride at room temperature furnished the desired aryl bromide 5 in 72% yield after 26 hours along with 27% of re-isolated starting material 3.

Both silyl ethers 4 and 5 were subsequently metalated at low temperature and the resulting aryllithium compounds
were quenched with tris(pentafluoro-phenyl)borane. The isolated borates 6 and 7 were contaminated by varying amounts of lithium [tetrakis(pentafluoro-phenyl)borate], which was easily removed after the next step. This side-product probably resulted from aryl exchange between the product and the borane reagent. The amount of the impurity was dependent on the protecting group and decreased with increasing reaction temperature. However, due to the explosive nature of similar compounds, the transformations were not performed at temperatures above –50 °C. Under optimized conditions, using tert-butyl(dimethyl)silyl ether 4 and slowly adding the electrophile at –50 °C, the ratio of product 6 to lithium [tetrakis(pentafluoro-phenyl)borate] was found to be 13:1, according to 19F NMR spectroscopy.

The crude silyl ethers 6 and 7 were deprotected and the resulting tertbutylammonium borate 8 was purified by chromatography on silica gel without any problems. As expected, fluorine substitution of the benzyl linker reduced the acid sensitivity and prevented decomposition during chromatography as observed with compounds 1. Using the tert-butyl(dimethyl)silyl protecting group, the hydroxyl-functionalized borate 8 was readily synthesized in 85% yield over three steps, starting from aryl bromide 3.

Benzyl bromide 9 was prepared in high yield by treating alcohol 8 with phosphorus tribromide and tetrabutylammonium bromide (TBAB); the reaction without addition of TBAB was less efficient.

Interestingly, borates 6–9 showed more than the expected five signals in their 19F NMR spectra. This can be ascribed to hindered rotation of the aryl substituents around the boron–carbon bonds with concomitant collapse of the dynamic C3-symmetry. A different route was chosen for the preparation of the corresponding tris[3,5-bis(trifluoromethyl)phenyl]borates 14 and 16. So far, no practical synthesis of tris[3,5-bis(trifluoromethyl)phenyl]borane has been described in the literature. For this reason, we decided to develop a general reaction sequence according to pathway B in Scheme 1. As an intermediate of type RBrX, the air- and moisture-stable aryltrifluoroborate 10 was chosen (Scheme 3).

Initial attempts to synthesize the desired aryltrifluoroborate 10 from aryl bromide 4, yielded mainly the corresponding diaryldifluoroborate 11. Similarly, desired bisarylations of trialkyl borates have been described in the literature for a few cases. However, after careful optimization of the reaction conditions, we were able to obtain both compounds 10 and 11 selectively (Scheme 3).

The aryltrifluoroborate 10, prepared according to Scheme 3, contained about 10% of 11 as a side-product. However, the corresponding mixed tetraarylboration 12 and 13 (Scheme 4) could be easily separated by column chromatography after the next step. Interestingly, the tert-butyl(dimethyl)silyl ether proved to be stable under the fluorination conditions, but only if all the tetrahydrofuran present in the reaction mixture was removed before addition of potassium hydrogen difluoride.

Addition of the appropriate arylmagnesium reagent, prepared by halogen-magnesium exchange, to the respective fluoroborates 10 and 11 yielded the mixed tetraarylboration 12 and 13 (Scheme 4). These were subsequently transformed into the bromides 16 and 17 via the corresponding benzylic alcohols 14 and 15, using the procedures described in Scheme 2. In this way, the hydroxyl- and bromo-functionalized tetraarylboration 14–17 were obtained in 81–71% yield over three steps.

In summary, a series of mono- and bifunctional fluorinated borate building blocks have been prepared, containing nucleophilic hydroxyl or electrophilic benzylic bromide functionalities. The compounds are readily accessible in three to five steps with 85–38% overall yield, starting from the literature-known alcohol 3. The inert nature and lipophilicity, which ensures high solubility in apolar media, are attractive features of the borate unit. Reagents of

![Scheme 2 Synthesis of building blocks derived from tris(pentafluoro-phenyl)borane](image)
this type can be used for the anionic derivatization of any neutral compound possessing at least one nucleophilic or electrophilic functional group. Thus they may find use in ESI-MS studies or mechanistic investigations of anion effects. Application of these borate reagents in the synthesis of anionic ligands for metal-catalyzed asymmetric transformations will be reported in due course.

All reactions were performed in flame-dried glassware under argon using Schlenk techniques. Solvents, Et$_3$N, LiBr and TBAB were dried employing standard procedures.$^{20}$ All other commercial reagents were used without further purification. Chromatography was performed on Merck silica gel 60 (Darmstadt, 40–63 nm). For TLC analyses, pre-coated Macherey–Nagel Polygram SIL G/UV$_{254}$ plates were used and the compounds were visualized with the help of UV light. All NMR experiments were performed on Bruker Avance 400 and 500 MHz spectrometers. $^1$H and $^{13}$C NMR spectra are referenced relative to TMS using the residual solvent peaks and the solvent signals, respectively, as internal standards.$^{21}$ The $^{19}$F and $^{11}$B spectra were calibrated using CFCl$_3$ and BF$_3$·OEt$_2$ as external standards. Mass spectra were measured on VG70-250, Finnigan MAT 95Q (EI) or Finnigan MAT LCQ apparatus (ESI). Elemental
analyses were performed by the Micro Analysis Laboratory of the University of Basel. IR spectra were measured on a Perkin-Elmer 1600 FTIR spectrometer. Melting points were determined on a Büchi 535 apparatus and are uncorrected. Benzylalcohol 3 was prepared following literature procedures. The abbreviation ArF refers to any fluorinated aryl moiety, whilst the abbreviations mc and dmc refer to centered multiplet and doublet of centered multiplet, respectively.

4-Bromo-2,3,5,6-tetrafluorobenzaldehyde

A solution of pentafluorobenzaldehyde (11.2 mL, 17.7 g, 90.3 mmol) and dried LiBr (8.90 g, 102 mmol) in anhyd NMP (50 mL) was stirred at 160 °C for 3 h. After the brown mixture had been cooled to r.t., it was filtered through Celite. The filtrate was poured into H2O (200 mL) and the resulting brownish solid was collected by filtration, washed with H2O (3 × 20 mL) and dried over P2O5 in a desiccator. The filtrate was evaporated under reduced pressure. The remaining sticky solid was washed with H2O (3 × 10 mL) and dried as described above. The combined crude products were washed with Et2O (60 mL) and dried. Concentration of the washing solutions to about half volume and isolation of the precipitate formed yielded a second crop of product. Aldehyde 18 was isolated as a pale-yellow solid in total yield of 13.7 g (59%).

IR (KBr): 2915, 1702, 1636, 1476, 1406, 1370, 1270, 1060, 916, 818, 703 cm–1.

4-Bromo-2,3,5,6-tetrafluorobenzyloxy)-tert-butylidiphenyldisilane (5)

To a solution of benzylalcohol 3 (5.18 g, 20.0 mmol) and anhyd Et3N (4.18 mL, 30.0 mmol) in anhyd DMF (40 mL), TBDBPSiCl (6.14 mL, 24.0 mmol) was slowly added at 0 °C. After the mixture had been stirred for 26 h at r.t., it was diluted with Et2O (150 mL) and poured into aqueous HCl (0.4 M, 80 mL). The phases were separated and the aqueous phase was extracted with Et2O (3 × 70 mL). The combined extracts were washed with sat. NaHCO3 and evaporated under reduced pressure. Purification of the remaining yellow oil by column chromatography (silica gel, 4 × 24 cm, hexanes–EtOAc, 15:1) yielded silyl ether 4 as a colorless, viscous oil, which solidified at –22 °C to a waxy solid (2.57 g, 98%).

IR (NaCl): 2955, 2935, 2891, 2860, 1637, 1487, 1261, 1100, 1051, 917, 837, 780, 717 cm–1.

1H NMR (400 MHz, CDCl3): δ = 0.24 (hexanes–EtOAc, 3:1).

IR (KBr): 3347, 2980, 2954, 2894, 1637, 1476, 1406, 1370, 1270, 1060, 916, 818, 703 cm–1.

1H NMR (100 MHz, CDCl3): δ = 0.24 (hexanes–EtOAc, 3:1).

IR (KBr): 2915, 1702, 1636, 1476, 1406, 1370, 1270, 1060, 916, 818, 703 cm–1.

1H NMR (100 MHz, CDCl3): δ = 0.24 (hexanes–EtOAc, 3:1).
Lithium [4-(tert-Butyldimethoxyloxymethyl)-2,3,5,6-tetrafluorophenyl]tris(pentafluorophenyl)borate (6)

To a solution of aryl bromide (4.01 g, 2.71 mmol) in anhyd Et2O (15 mL), n-BuLi in hexanes (1.6 M, 1.69 mL, 2.71 mmol) was added dropwise at –78 °C over a period of 12 min. After the mixture had been stirred for 60 min at –78 °C, the temperature was raised to 0 °C and then for 14 h (overnight) at r.t. After the solution had been stirred for 2 h at 0 °C, the mixture was stirred for a further 3.5 h at r.t., 2,4,6-triisopropylbenzenesulfonic acid (3.25 mmol) was added. The mixture was stirred at 0 °C for 3 h then for 16 h (overnight) at r.t. A colorless, foamy solid was obtained, which consisted of borate 7 and Li[B(C6F5)4] in a ratio of 9:1 according to 19F NMR spectroscopy.

1H NMR (400 MHz, acetone-d6): δ = 1.00 (s, 9 H, CH3), 4.79 (s, 2 H, CH2), 7.37–7.49 (m, 6 H, Ph-m-H and Ph-p-H), 7.70 (d, J = 7.3 Hz, 4 H, Ph-o-H).

11B [1H] NMR (375 MHz, acetone-d6): δ = –167.7 (m, 6 F, C6F5–m–F), –164.0 (t, J = 20 Hz, 2 F, C6F5–p–F), –163.9 (t, J = 18 Hz, 1 F, C6F5–p–F), –149.6 (dd, J = 23, 13 Hz, 2 F, Ar-m–F), –133.3 (m, 2 F, Ar2–o–F), –132.3 (m, 2 F, Ar2–o–F), –132.0 (m, 2 F, Ar2–o–F), –131.7 (m, 2 F, Ar2–o–F).

13C {1H} NMR (125 MHz, acetone-d6): δ = –150.0 (dd, J = 22, 14 Hz, 2 F, Ar–o–F), –133.2 (m, 2 F, Ar2–o–F), –132.3 (m, 2 F, Ar2–o–F), –131.9 (m, 2 F, Ar2–o–F), –131.7 (m, 2 F, Ar2–o–F).

Tetraethylammonium (4-Bromomethyl-2,3,5,6-tetrafluorophenyl)tris(pentafluorophenyl)borate (8)

To a solution of borate 8 (4.67 g, 5.00 mmol) in anhyd CH2Cl2 (35 mL), PBr3 (305 mL, 3.25 mmol) was added dropwise at 0 °C. After the solution had been stirred for 2 h at 0 °C, dried TBAB (2.42 g, 7.50 mmol) was added. The mixture was stirred for a further 3.5 h at 0 °C and then for 14 h (overnight) at r.t. After the resulting colorless solution had been diluted with Et2O (300 mL), it was successively washed with H2O (50 mL), half-sat. NaHCO3 (50 mL) and brine (50 mL). The combined aqueous phases were re-extracted with Et2O (2 × 80 mL) and the combined extracts were dried over MgSO4, filtered and evaporated under reduced pressure. Purification of the crude product by column chromatography (silica gel, 4 × 11 cm, CH2Cl2) yielded benzylic bromide 9 as a colorless, foamy solid (4.85 g, 97%).

Mp 54–55 °C; Rf: 0.15 (CH2Cl2), tailing.

IR (KBr): 2971, 2881, 1645, 1515, 1483, 1380, 1268, 1219, 1159, 1090, 979, 833, 766, 694, 665, 613, 549 cm–1.

1H NMR (400 MHz, acetone-d6): δ = 0.98 (t, J = 7.4 Hz, 12 H, CH3), 1.43 (sext, J = 7.4 Hz, 8 H, CH2CH2CH2CH3), 1.84 (m, 8 H, CH2CH2CH2CH3), 3.46 (m, 8 H, CH2CH2CH2CH3), 4.29 (t, J = 6.1 Hz, 1 H, OH), 4.64 (d, J = 6.1, 1.4 Hz, 2 H, CH2OH).

19F {1H} NMR (375 MHz, acetone-d6): δ = –168.0 to –167.6 (m, 6 F, C6F5–m–F), –161.5 (t, J = 20 Hz, 2 F, C6F5–p–F), –163.9 (t, J = 19 Hz, 1 F, C6F5–p–F), –150.0 (dd, J = 22, 14 Hz, 2 F, Ar–m–F), –133.5 (m, 2 F, Ar–o–F), –132.3 (m, 2 F, Ar2–o–F), –131.9 (m, 2 F, Ar2–o–F), –131.7 (m, 2 F, Ar2–o–F).

Anal. Calcd for C52H56BF29O: C, 52.75; H, 4.21; N, 1.50. Found: C, 52.86; H, 4.24; N, 1.52.

Potassium [{4-[(tert-Butyldimethylsilanylmethyl)-2,3,5,6-tetrafluorophenyl]trifluoroborate} (10)
To a solution of aryl bromide (4.14 g, 3.82 mmol) in anhyd Et2O (9 mL), i-PrMgCl in THF (2 M, 1.91 mL, 3.82 mmol) was added dropwise within 9 min at r.t. The mixture was stirred for 2.5 h and the resulting colorless suspension was diluted with more Et2O (10 mL) and cooled to –78 °C. Bis(Oi-Pr)2 (1.76 mL, 7.64 mmol) was quickly added and the mixture was stirred for 22 h (overnight) at r.t. To the viscous, colorless suspension, MeOH (100 mL) was added in order to destroy any residual aryllithium reagent. Then all volatiles were removed under reduced pressure. The remaining solid was suspended in Et2O (30 mL) in a polypropylene vessel and KH2F (2.54 g, 32.5 mmol) in H2O (15 mL) was added dropwise at r.t. The mixture was stirred for 90 min at this temperature, B(Oi-Pr)3 (1.12 g, 14.3 mmol) in H2O (7 mL) was slowly added. The mixture had been stirred for 90 min at this temperature, B(Oi-Pr)3 (649 mg, 1.62 mmol) in anhyd Et2O (28 mL) and the resulting solution was stirred for 135 h at r.t. The mixture was poured into a solution of Na2CO3 (4.40 g) in H2O (55 mL) and the two-phase system was vigorously stirred for 30 min at r.t. The phases were separated and the aqueous phase was extracted with CH2Cl2 (3 × 50 mL). The combined organic extracts were dried over Na2SO4, filtered, evaporated under reduced pressure and the remaining oil was re-dissolved in CH2Cl2 (30 mL). TBAB (627 mg, 1.94 mmol) in CH2Cl2 (10 mL) was added and the suspension was stirred for 30 min at r.t., filtered and concentrated under reduced pressure. Purification of the resulting oil by column chromatography (silica gel, 4 × 11 cm, 240 mL Et2O then CH2Cl2) yielded borate 12 as a colorless solid (1.73 g, 90%).

Tetrabutylammonium Bis[4-(tert-butyl(dimethyl)silyl)anisoyl-methyl]-2,3,5,6-tetrafluoroquinoline]bis[3,5-bis(trifluoro-methyl)phenyl]borate (13)

In analogy to the synthesis of 12, bromo-3,5-bis(trifluoromethyl)benzene (792 µL, 4.59 mmol) was reacted with i-PrMgCl in THF (20 M, 2.04 mL, 4.08 mmol) and diaryl difluoroborate 11 (689 mg, 1.02 mmol) in anhyd THF (4 mL) and anhyd Et2O (12 mL) for 135 h at rt. After aqueous workup as described above, the magnesium borate was treated with TBAB (395 mg, 1.22 mmol) in CH2Cl2 (25 mL) for 30 min at rt. Purification of the yellow crude product by column chromatography (silica gel, 3 × 12 cm, 140 mL Et2O then CH2Cl2) furnished borate 13 as a colorless, sticky foam (1.16 g, 90%).

\[ R_f \leq 0.74 \text{ (CH}_2\text{Cl}_2, \text{tailing)}. \]

IR (NaCl): 2962, 2885, 1612, 1443, 1359, 1277, 1128, 1038, 937, 886, 841, 778, 735, 679 cm⁻¹.

1H NMR (500 MHz, CDCl3): \( \delta = 0.07 \text{ [s, 12 H, Si(CH}_3\text{)l], 0.88 [s, 18 H, C(CH}_3\text{)l], 0.88 [t, J = 7.3 H z, 12 H, CH}_2\text{CH}_3\text{], 1.24 [sext, J = 7.4 H z, 8 H, CH}_2\text{CH}_2\text{CH}_3\text{], 1.47 [m, 8 H, CH}_2\text{CH}_2\text{CH}_2\text{CH}_3\text{], 2.93 [m, 8 H, CH}_2\text{CH}_2\text{CH}_2\text{CH}_3\text{], 4.70 [s, 4 H, CH}_2\text{O], 7.47 [s, 2 H, ArF-}^2\text{H}, 7.88 [s, 4 H, ArF-}^2\text{-O-H}. \]

13C{1H} NMR (125 MHz, CDCl3): \( \delta = 23.7 \text{ (CH}_3\text{)}, 18.6 [C(CH}_3\text{)l], 19.6 (CH}_2\text{CH}_3\text{), 23.7 (CH}_2\text{CH}_2\text{CH}_3\text{), 26.0 [C(CH}_3\text{)l], 53.7 (CH}_2\text{CH}_2\text{CH}_3\text{), 113.9 [t, J = 18 Hz, Ar-p-C], 117.3 [sept, J = 4 Hz, ArF-}^2\text{p-C}, 124.8 [q, J = 273 Hz, CF}_3\text{], 128.6 [q, J = 31 Hz, ArF-}^2\text{m-C}, 133.3 (ArF-}^2\text{o-C}, 144.7 [dd, J = 245, 17 Hz, Ar-m-C], 147.7 [dt, J = 237, 12 Hz, Ar-}^2\text{o-C}, 159.3 (br, ArF-}^2\text{i-C). Despite prolonged data acquisition time the signal for Ar-p-C was not detected. \]

19F{1H} NMR (375 MHz, CDCl3): \( \delta = -148.3 \text{ (dd, J = 24, 13 Hz, 4 F, Ar-}^2\text{m-F), -130.0 [dd, J = 24, 13 Hz, 4 F, Ar-o-F], -62.3 [s, 12 F, CF}_3\text{]. \]

MS (ESI): \( m/z (%) = 1023 (100) \text{ [M – NBu}_4\text{]+}. \]

Tetrabutylammonium (2,3,5,6-Tetrafluoro-4-hydroxy-methylphenyl)tris[3,5-bis(trifluoromethyl)phenyl]borate (14)

In analogy to the synthesis of 8, silyl ether 12 (1.56 g, 1.31 mmol) was treated with TBAF·3H 2O (380 mg, 2.63 mmol) in anhyd THF (15 mL) for 14 h (overnight) at rt. Purification of the yellowish crude product by column chromatography (silica gel, 3 × 8 cm, 200 mL Et2O then CH2Cl2) yielded benzylic alcohol 14 as a colorless, sticky foam, which gradually solidified to a vitreous solid (1.26 g, 90%).

Mp 120–121 °C; \( R_f = 0.24 \text{ (CH}_2\text{Cl}_2\text{).} \]

IR (KBr): 3627, 3368, 2971, 2883, 1612, 1447, 1361, 1279, 1128, 934, 886, 839, 722, 679, 628 cm⁻¹.

1H NMR (500 MHz, CDCl3): \( \delta = 0.08 [t, J = 7.3 H z, 12 H, CH}_2\text{Cl}_2\text{], 1.25 [sext, J = 7.3 H z, 8 H, CH}_2\text{CH}_2\text{CH}_3\text{], 1.48 [m, 8 H, CH}_2\text{CH}_2\text{CH}_2\text{CH}_3\text{], 1.92 [br s, 2 H, OH], 2.95 [m, 8 H, CH}_2\text{CH}_2\text{CH}_2\text{CH}_3\text{], 3.48 [s, 4 H, CH}_2\text{O}], 7.48 [s, 2 H, ArF-}^2\text{p-H}, 7.91 [s, 4 H, ArF-}^2\text{o-H}. \]

11B{1H} NMR (160 MHz, CDCl3): \( \delta = 11.8 (100) \text{ [M – NBu}_4\text{]+}. \]


MS (ESI): \( m/z (%) = 795 (100) \text{ [M – NBu}_4\text{]+}. \]

Tetrabutylammonium (4-Bromomethyl-2,3,5,6-tetrafluoroquinolino)tris[3,5-bis(trifluoromethyl)phenyl]borate (16)

In analogy to the synthesis of 9, borate 14 (6.13 g, 5.72 mmol) was treated with Tetrabutylammonium (2,3,5,6-Tetrafluoro-4-hydroxymethyl)tris[3,5-bis(trifluoromethyl)phenyl]borate (16)
Tetrabutylammonium Bis(4-bromomethyl-2,3,5,6-tetrafluorophenyl)bis[3,5-bis(trifluoromethyl)phenyl]borate (17)

In analogy to the synthesis of 9, diol 15 (2.21 g, 2.13 mmol) was treated with PBr3 (260 μL, 2.77 mmol) and subsequently TBAB (1.71 g, 5.33 mmol) in anhyd CH2Cl2 (25 mL) at 0 °C for 3.5 h in total and then for 15 h (overnight) at r.t. Purification of the yellowish crude product by column chromatography (silica gel, 4 × 10 cm, CH2Cl2) furnished dibromide 17 as a colorless, foamy solid (2.29 g, 92%).

Mp 57–62 °C; Rf ≤ 0.72 (CH2Cl2, tailing).

IR (KBr): 2971, 2881, 1645, 1612, 1576, 1448, 1360, 1277, 1127, 971, 888, 840, 725, 680, 610, 544 cm⁻¹.

1H NMR (400 MHz, CDCl3): δ = –146.7 (dd, J = 22, 11 Hz, 4 F, Ar-β-F), –129.3 (dd, J = 24, 13 Hz, 4 F, Ar-α-F), –62.4 (s, 12 F, CF3).

1B(1H) NMR (160 MHz, CDCl3): δ = –10.5.

MS (ESI): m/z (%) = 921 (100) [M – NBu4]+.

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