Synthesis of Highly Functionalizable Symmetrically and Unsymmetrically Substituted Triarylboran es from Bench-Stable Boron Precursors

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Abstract: A novel and convenient methodology for the one-pot synthesis of sterically congested triarylboran es by using bench-stable aryltrifluoroborates as the boron source is reported. This procedure gives systematic access to symmetrically and unsymmetrically substituted triarylboran es of the types $\text{BAr}_3\text{Ar}'$ and $\text{BArAr}'\text{Ar}''$, respectively. Three unsymmetrically substituted triarylboran es as well as their iridium-catalyzed C–H borylation products are reported. These borylated triarylboran es contain one to three positions that can subsequently be orthogonally functionalized in follow-up reactions, such as Suzuki-Miyaura cross-couplings or Sonogashira couplings.

Introduction

Triarylboran es or compounds containing this structural motif can potentially find applications in various fields as functional materials.[1–9] The interesting properties of these functional materials originate from the vacant $p_z$ orbital of the boron center that can efficiently conjugate with adjacent $z$-systems. The electronic and optical, physical, biological, and chemical properties of such systems can be tuned by variation of the substituents on the three aryl rings attached to the boron atom.[4–9] In general, there are three types of triarylboran es (Figure 1). The first type bears three identical substituents on the boron center ($\text{BAr}_3$) resulting in a high symmetry ($D_3$) and, due to its simplicity, this type of triarylborate was the first to be reported in 1885.[10] A reliable methodology for the synthesis of these compounds was reported by Krause and coworkers in 1922 wherein aryl Grignard reagents were reacted with gaseous boron trifluoride to form the corresponding triarylborate.[11] By changing the boron source to the more convenient bench-stable potassium aryltrifluoroborates as the boron source.[12–14] This strategy became widely applicable. Less symmetrical triarylboran es of the type $\text{BAr}_3\text{Ar}'$ ($C_1$) have been systematically accessible since 1972.[15] In this procedure, sterically demanding aryl met alates, for example, Grignard reagents, are reacted with $\text{BX}_3$ species to form the corresponding $\text{XBAr}_3$ derivative. Due to the steric congestion, the reaction stops after the attachment of two aryl moieties to the boron center. Then, a second, more reactive aryl met alate, for example, an organolithium reagent, is used to attach the third aryl ring to the boron center. This is still the most common way to synthesize $C_3$ symmetrically substituted triarylboran es.[16] The third class of triarylborate bears three different aryl substituents ($\text{BArAr}'\text{Ar}''$) and will, in the following, be referred to as unsymmetrically substituted triarylboran es. Only a few examples of unsymmetrically substituted $\text{BArAr}'\text{Ar}''$ triarylboran es have been reported in the literature, and no general synthetic methodology is known. We have recently published a detailed review on the historical development of synthetic strategies for the preparation of triarylboran es.[16] The unsymmetrically substituted triarylboran es reported to date have been synthesized via extensive, multistep procedures,[17,18] from symmetrically substituted precursors,[19,20] or with sterically less demanding substituents attached to the boron center,[22–25] mainly starting from highly reactive, and therefore not bench-stable boron halides. In contrast, the work presented herein provides a systematic, one-pot approach to $\text{BArAr}'\text{Ar}''$ triarylboran es starting from bench-stable potassium aryltrifluoroborates as the boron source.

The optional functional groups to incorporate, and the possibilities to do follow-up reactions on triarylboran es, have increased considerably over the past years. The appl icability of triarylboran es in many fields is limited by their instability towards hydrolysis. One of the easiest and most common ways to stabilize triarylboran es kinetically is to introduce ortho-methyl groups around the boron center. It was demonstrated that compounds bearing six ortho-methyl groups around the boron are stable in pure water for several days.[26,27] Due to the kinetic stabilization of the boron center, the $\text{BX}_3$ ($\text{X} = 2,6-\text{MeC}_6\text{H}_3$) moiety exhibits a high tolerance to different reaction conditions.
conditions employed in functionalization reactions. An overview of the numerous reported reaction conditions and selected functional groups attached to the BXyl$_3$ moiety is given in Figure 2.

The most notable effect on the properties of triarylboranes is obtained by functionalization of the para-position on the 2,6-dimethylphenyl substituents. This can be achieved, for example, by iridium-catalyzed C–H borylation which exhibits very high, sterically driven regioselectivity.$^{[76,77]}$ The resulting boronate

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**Figure 1.** General types of triarylboranes$^{[16]}$ and a schematic summary of this work.

**Figure 2.** Overview of reported follow-up reactions, to which the BXyl$_3$ moiety (BAR$_3$ or BAR$_2$Ar') is tolerant, and selected functional groups that have been attached. Examples of each path are given in the following references: a, $^{[26–30]}$ b, $^{[27,29–33]}$ c, $^{[34,35]}$ d, $^{[36–51]}$ e, $^{[52–57]}$ f, $^{[42,58–62]}$ and h, $^{[63–75]}$.
ester moieties can be employed in various functional group transformations as well as Suzuki-Miyaura cross-coupling reactions (Figure 2b). In this way, triarylboranes can also be attached to chromophores or stationary phases. Even deprotonation or lithium-halide exchange reactions can be employed to functionalize triarylboranes due to their high tolerance to functional groups. A maximum of two functional groups is available, such as CO$_2$H or COH, can be introduced. In general, introduction of electron donating or accepting groups has a strong effect on the optoelectronic properties of triarylboranes. By carefully choosing the specific substituent, the properties can be fine-tuned to meet different requirements. For example, water solubility of triarylboranes can be facilitated by the introduction of cationic ammonium or phosphonium substituents (Figure 2e and f). Consequently, even though a more reactive corresponding aryl lithium reagent, BAr$_3$H, was isolated in a slightly higher yield (23%) after stirring for 3 days at room temperature. It is well documented that by reaction of trimethylsilyl chloride (TMSCl) and aryltrifluoroborates, our first approach was to exchange the Grignard reagent for the more reactive corresponding aryl lithium reagent, BAr$_3$H. This compound is frequently used in our work as a starting material for the synthesis of water-soluble chromophores for biological applications. Based on these results, we present a synthetic route to symmetrically and unsymmetrically substituted, sterically congested and, therefore, air-stable triarylboranes starting from bench-stable boron precursors, namely potassium arylltrifluoroborates.

Results and Discussion

Synthesis of symmetrically substituted triarylboranes

To optimize the formation of para-substituted BXyl$_3$ from potassium arylltrifluoroborates, our first approach was to examine the synthesis of the compound BAr$_3$BF$_3$. Using a Grignard reagent in THF as the metalate, BAr$_3$BF$_3$ was isolated in 19% yield after stirring for 2 days at 90°C in a sealed vessel. The reaction time can be significantly reduced to 1 h when using microwave irradiation at 200°C. Exchanging the Grignard reagent for the more reactive corresponding aryl lithium reagent, BAr$_3$BF$_3$, was isolated in a slightly higher yield (23%) after stirring for 3 days at room temperature. It is well documented that by reaction of trimethylsilyl chloride (TMSCl) and arylltrifluoroborates, the reactivity of the latter is increased due to partial formation of difluoroborates. Therefore, it was possible to increase the isolated yield of BAr$_3$BF$_3$ to 43% when activating ArBF$_3$K with TMSCl in diethyl ether prior to reaction with the aryl lithium reagent. This yield was further increased to 51% by changing the solvent for the activation to THF, as the solubility of 4-bromo-2,6-dimethylphenyl lithium is low in diethyl ether.

To demonstrate further the utility of our reaction, the reaction conditions were applied in the synthesis of BAr$_3$BF$_3$. This compound is frequently used in our work as a starting material for the synthesis of water-soluble chromophores for biological applications. BAr$_3$BF$_3$ was previously only accessible in a yield of 33% over two steps,

![Scheme 1. Synthesis of the C$_2$ symmetric triarylboranes BAr$_3$Ar$^+$ and BAr$_3$Ar$^{\text{Me}}$.](Image)

| Table 1. Summary of conditions applied for the synthesis of the symmetrically substituted triarylboranes BAr$_3$Ar$^+$ and BAr$_3$Ar$^{\text{Me}}$. |
|-------------------|----------------|----------------|
| BAr$_3$Ar$^+$     | M               | R              | Solvent | T [°C] | t [h]  | Yield [%] |
| 1 Ar$_3$BF$_3$K   | MgBr            | Br             | THF     | 90     | 2      | 19 |
| 2 Ar$_3$BF$_3$K   | MgBr            | Br             | THF     | 200$^{[b]}$ | 1      | 19 |
| 3 Ar$_3$BF$_3$K   | Li              | Br             | THF     | RT     | 3      | 23 |
| 4$^{[a]}$ Ar$_3$BF$_3$K | Li | Br | ET$_2$O | RT | 2      | 43 |
| 5$^{[a]}$ Ar$_3$BF$_3$K | Li | Br | ET$_2$O | RT | 3      | 51 |
| 6 Ar$_3$Ar$^{\text{Me}}$ | Li | NE$_3$ | THF | RT | 18 h–2 d | 63 |

[a] Activation of Ar$_3$BF$_3$K performed in ET$_2$O; [b] Activation of Ar$_3$BF$_3$K performed in THF; [c] Isolated yield; [d] Microwave irradiation in a sealed tube.
as we reported in 2016.[27,28] Using our new approach, 4-(N,N-dimethylamino)-2,6-dimethylphenyl lithium was reacted with ArBF₃K according to Scheme 1, to give BAr⁷Ar'Ar''Br in a good yield of up to 63% in one step. This reaction shows that the activation of the potassium aryltrifluoroborates is not required for highly reactive lithiated species, such as 4-(N,N-dimethylamino)-2,6-dimethylphenyl lithium.

From these reactions, it can be concluded that sterically very congested triarylboranes can be synthesized from ArBF₃K and two equivalents of aryl Grignard reagent at elevated temperatures. In contrast, at room temperature, the reaction of ArBF₃K with two equivalents of an aryl Grignard reagent stops after the attachment of one additional aryl ring to the boron center, for reaction times of up to five days. This leads to a diarylfluoroborane (BFArAr'') with four ortho-methyl groups. To synthesize symmetrically substituted triarylboranes (BAr₁⁺Ar'⁺), from aryltrifluoroborates at room temperature without a loss in yield, more reactive lithiated arenes must be used.

**Synthesis of unsymmetrically substituted triarylboranes**

As aryl Grignard and aryl lithium reagents differ significantly in their reactivities towards potassium aryltrifluoroborates, this approach was used for the synthesis of unsymmetrically substituted triarylboranes according to Scheme 2 and Table 2.

To this end, ArBF₃K was reacted with one equivalent of (4-bromo-2,6-dimethylphenyl)magnesium bromide at room temperature, to form the corresponding fluoroborane (BFArAr''), as indicated by in situ ³²F and ¹¹B NMR spectroscopy. Then, the more reactive 2,4,6-trimethylphenyl lithium (MesLi) was added and the unsymmetrically substituted triarylbforen BAr⁷Ar'Ar'' was isolated in 18% yield. The formation of BFArAr' is rather fast, and the same yields were obtained for shorter reaction times (Table 2 entry 1 vs. entry 2). Thus, the synthesis of BAr⁷Ar'Ar'' is possible in a one pot process within 24 h. To optimize the conditions further, the influence of the para substituents on the potassium aryltrifluoroborate was examined. The synthesis of BAr⁷Ar'Ar'' was carried out starting from the respective aryltrifluoroborate ArBF₃K, potassium mesityltrifluoroborate ArBF₃K and potassium 4-bromo-(2,6-dimethylphenyl)trifluoroborate ArBF₃K. Our reaction procedure was performed starting from each potassium aryltrifluoroborate according to Scheme 3 and Table 2, entries 2–4.

For each reaction, the desired product BAr⁷Ar'Ar'' was isolated. Using ArBF₃K as the starting material, we obtained a yield of 18% over two steps (see above). Starting from ArBF₃K, the yield doubled to 35% over two steps. With ArBF₃K as the starting material, the yield over two steps decreased to 3%. These drastic differences in yield were surprising, as the electronic differences of the potassium aryltrifluoroborates were considered minor in comparison to the equal steric demand at the boron centers of all aryltrifluoroboranes. The electronic differences for substituents at the para position can be assessed by the Hammett values and are α₂ = −0.17 for CH₃, 0 for H and 0.23 for bromide. The trend found in the isolated yields of BAr⁷Ar'Ar'' are in good accordance with the trend given by the para Hammett values of the respective substituents in the potassium aryltrifluoroborates ArBF₃K, ArBF₃K and ArBF₃K. It is well documented that electron donating groups (EDGs) enhance solvolysis of aryltrifluoroborates, whereas electron withdrawing groups (EWGs) hinder solvolysis.[86,87] In analogy to their benzo trifluoride analogues,[88,89] this is explained by the proactivity of aryltrifluoroborate to lose a fluorine atom in the

![Scheme 2](image)

**Scheme 2.** Summary of synthesis of the unsymmetrically substituted triarylboranes BAr⁷Ar'Ar'', BAr⁷Ar'Ar''Br, and BAr⁷Ar'Ar''Me.

| Table 2. Summary of conditions applied for the synthesis of the unsymmetrically substituted triarylboranes BAr⁷Ar'Ar'', BAr⁷Ar'Ar''Br, and BAr⁷Ar'Ar''Me. |
|---|---|---|---|---|---|---|---|
| **BAr⁷Ar'Ar''** | **R** | **R'** | **R''** | **T** | **t A** | **t B** | **Yield [%]** |
| 1 | BAr⁷Ar'Ar'' | H | Br | Me | RT | 18 h | 3 d | 18 |
| 2 | BAr⁷Ar'Ar'' | H | Br | Me | RT | 30 min | 18 h | 18 |
| 3 | BAr⁷Ar'Ar'' | Me | Br | H | RT | 30 min | 18 h | 35 |
| 4 | BAr⁷Ar'Ar'' | Br | H | Me | RT | 30 min | 18 h | 3 |
| 5 | BAr⁷Ar'Ar'' | H | Br | SiMe₂ | RT | 30 min | 18 h | 16 |
| 6 | BAr⁷Ar'Ar'' | H | Me | NMe₂ | RT | 18 h | 3 d | trace |
| 7 | BAr⁷Ar'Ar'' | H | Me | NMe₂ | 70 °C | 18 h | 2 d | 5 |
| 8 | BAr⁷Ar'Ar'' | H | NMe₂ | Me | RT | 18 h | 3 d | 10 |
| 9 | BAr⁷Ar'Ar'' | H | NMe₂ | Me | RT | 18 h | 18 h–3 d | 51 |

[a] Intermediate diaryl fluoroborane was isolated. [b] Isolated yield.

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first step of the solvolysis. The first step of the solvolysis is described as an equilibrium between the aryldifluoroborate and the short-lived aryldifluoroborane. This intermediate is stabilized by electron donating groups, whereas electron withdrawing groups stabilize the negative charge at the boron in the case of aryldifluoroborates. Even though the mechanism of our triarylborane synthesis is unknown, both a dissociative as well as an associative mechanism would energetically benefit from electron donating groups at the para position of the aryldifluoroborate salts, as B–F bond cleavage seems to be the critical factor. Thus, while our method provides considerable flexibility, to enhance the yield, electron-rich potassium aryldifluoroborates should be chosen as the boron source, if possible.

The unsymmetrically substituted triarylborane \( \text{BAr}_3^\text{H} \) allows further orthogonal functionalization at two positions, namely the para bromide and the para proton (Figure 2). The mesitylene unit does not possess a position that can be conveniently functionalized. To obtain a triarylborane that can be orthogonally functionalized at all three aromatic rings, the para methyl group of mesitylene was exchanged for a trimethylsilyl group. This moiety permits many different functionalization reactions\(^{20-24} \) and should be robust enough to tolerate the reaction conditions required for the triarylborane formation. We chose \( \text{Ar}^\text{B} \text{F}_3 \text{K} \) as the starting material and synthesized compound \( \text{BAr}_3^\text{H} \text{Ar}^\text{B} \text{F}_3 \text{K} \), in a yield of 16% over two steps in a one pot synthesis (Scheme 2, Table 2 entry 5).

To test whether our methodology can be applied for the synthesis of a compound containing an electron donating group, triarylborane \( \text{BAr}_3^\text{H} \text{Ar}^\text{B} \text{F}_3 \text{K} \) was chosen as a model system in analogy to \( \text{BAr}_3^\text{H} \text{Ar}^\text{B} \text{F}_3 \text{K} \). Therefore, in the first step, \( \text{Ar}^\text{B} \text{F}_3 \text{K} \) was reacted with a slight excess of mesitylmagnesium bromide. The conversion of the 4-aryl Grignard reagent will not form triarylboranes (see above). Therefore, the reaction temperature in the second step was increased to 70°C to give \( \text{BAr}_3^\text{H} \text{Ar}^\text{B} \text{F}_3 \text{K} \) in a 5% isolated yield (Scheme 2, Table 2 entry 7). This was further improved as the choice of Grignard and aryl lithium reagent was changed. When the 4-N,N-dimethylamino-2,6-dimethylphenyl motif was introduced as the Grignard reagent and the mesityl moiety was added as the lithium reagent, the reaction sequence gave the triarylborane \( \text{BAr}_3^\text{H} \text{Ar}^\text{B} \text{F}_3 \text{K} \) in 10% isolated yield in a one pot approach at room temperature (Scheme 2, Table 2 entry 8). This demonstrates that the choice of aryl Grignard and aryl lithium reagents is crucial to improve the isolated yields.

Given the fact that using more electron-rich potassium aryldifluoroborates as starting materials results in higher yields (see above), the synthesis of \( \text{BAr}_3^\text{H} \text{Ar}^\text{B} \text{F}_3 \text{K} \) from potassium (N,N-dimethylamino-2,6-dimethylphenyl)trifluoroborate \( \text{Ar}^\text{B} \text{F}_3 \text{K} \) should give higher yields. However, it was not possible to synthesize \( \text{Ar}^\text{B} \text{F}_3 \text{K} \) via our methodology as proteodeborylation during fluorination of the intermediate \( \text{Ar}^\text{B} \text{F}_3 \text{K} \) occurs. This indicates that not all potassium aryldifluoroborates are readily available.

However, to increase the isolated yield, the isolation of the proposed intermediate fluoroborate \( \text{BFAr}_3^\text{H} \text{Ar}^\text{B} \text{F}_3 \text{K} \) from a reaction mixture of \( \text{Ar}^\text{B} \text{F}_3 \text{K} \) and (4-(N,N-dimethylamino)-2,6-dimethylphenyl)magnesium bromide was attempted. It was possible to isolate \( \text{BFAr}_3^\text{H} \text{Ar}^\text{B} \text{F}_3 \text{K} \) in 89% yield, still containing residual Grignard reagent. At room temperature, the remaining Grignard reagent will not form triarylboranes (see above). Therefore, without further purification, the fluoroborate \( \text{BFAr}_3^\text{H} \text{Ar}^\text{B} \text{F}_3 \text{K} \) was added to a solution of mesityl lithium. After stirring at room temperature for 3 days, \( \text{BAr}_3^\text{H} \text{Ar}^\text{B} \text{F}_3 \text{K} \) was isolated in up to 51% yield. Thus, the isolated yield can be increased from 10%
in the one pot approach to 51% by one additional step, in which purely inorganic metal salts are separated from BFArNMe. Further experiments showed that the reaction time of the second step can be decreased from 3 days to 18 h without any loss in yield.

Interestingly, activation of ArBF3K prior to reaction with the Grignard reagent did not improve the yield of BFArNMe. This might be attributed to the increased reactivity of (4-(N,N-dimethylamino)-2,6-dimethylphenyl)magnesium bromide compared to Grignard reagents without strong electron donating groups, as similar behavior was observed for the synthesis of BArHArNMe. In turn, increasing the reactivity of ArBF3K by activation with trimethylsilyl chloride does not affect the isolated yield of BArHArNMe.

However, it is apparent that the yield depends not only on the choice of potassium aryltrifluoroborate, but also on the choice of Grignard and aryl lithium reagent (Table 2). In addition, all triarylboranes synthesized bear at least one position that can be functionalized after the formation of the triarylborane, making these compounds versatile building blocks for the synthesis of larger, boron-containing compounds.

Selected examples of post-functionalization

Iridium-catalyzed C–H borylation using [Ir(COD)OMe]2 as the precatalyst, 4,4′-di-tert-butylpyridine as the ligand and Bpin as the boron source exhibits very high sterically driven selectivity and has been applied in our group for the synthesis of many different functional materials, including triarylboranes. To demonstrate the utility of our novel compounds, we applied iridium-catalyzed C–H borylation to BArHArBrArNMe, BArHArBrArMe, BArHArBrBr and BArHArBrSiMe3. This borylation takes place selectively at the para C–H bond of the 2,6-dimethylphenyl ring, thus yielding triarylboranes BArHArBrArNMe, BArHArBrArMe, BArHArBrBr and BArHArBrSiMe3. The products shown in Scheme 4a were synthesized in up to 89% yield. These compounds represent a small library of A, AB, ABB, and ABC type, orthogonally functionalizable triarylboranes, wherein each letter indicates a different, orthogonally functionalizable group, in this case Bpin, bromine or trimethyl silyl.

As shown in Figure 2, borylated triarylboranes have already been employed successfully in Suzuki-Miyaura cross-coupling reactions with different aryl halides. However, the bromide, which is present in three of our four borylated triarylboranes, might lead to unwanted side reactions. To see if this can be circumvented by choice of an appropriate, more reactive aryl halide, we employed BArHArBrArNMe in a Suzuki-Miyaura cross-coupling of BArHArBrArNMe with iodo methylbenzoate.
coupling reaction with methyl iodobenzoate (Scheme 4b). The coupling product $\text{BAr}_3\text{Ar}_3\text{Ar}^\text{Me}$ was isolated in a good yield of 79% and the presence of the bromide in $\text{BAr}_3\text{Ar}_3\text{Ar}^\text{Me}$ was confirmed.

As mentioned above, the trimethylsilyl group permits many functionalization reactions. However, this functional group has not been used for functionalizations when attached to a $\text{Bx}_3\text{y}$ moiety. To demonstrate the applicability of this functional group in our system, we converted the trimethylsilyl group of triarylborane can be improved with one additional step in preparation of the Arts for generous financial support, the Deutsche Forschungsgemeinschaft (DFG; GRK 2112) and the Julius-Maximilians-Universität Würzburg. Open access funding enabled by Projekt DEAL.

**Conflict of Interest**

The authors declare no conflict of interest.

**Keywords:** boranes – borylation – functionalization – synthetic methods

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