C–H Functionalization

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$(C_6F_5)_3B$ Catalyzed Chemoselective and *ortho*-Selective Substitution of Phenols with α-Aryl α-Diazoesters

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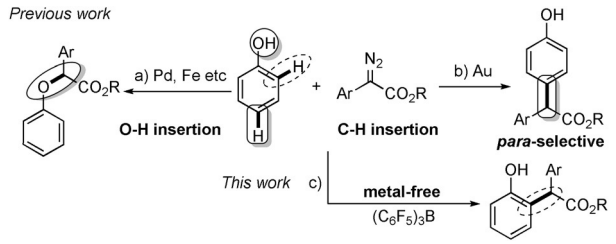
Abstract: *The development of an efficient method for the site-selective substitution of unprotected phenols has long been considered as an attractive but challenging task. Herein, we describe a highly chemo- and ortho-selective substitution reaction of phenols with α-aryl α-diazoacetates with commercially available $(C_6F_5)_3B$ as the catalyst. This reaction proceeds under simple and mild conditions with high efficiency, it features a wide substrate scope and can be easily scaled up.*

Diazo compounds are essential and useful reactive substrates that can undergo a series of transformations, including alkene cyclopropanation, metal carbene migratory insertion, C–H bond functionalization, X–H insertion (X = O, N, Si, etc.), and ylide formation.[1] Among these transformations, transition-metal-catalyzed C(sp^2)–H bond insertions with carbenes represent atom- and step-economic methods for carbon–carbon bond formation.[2] However, direct C–H bond substitution reactions of aromatic compounds with X–H bonds, such as phenols, which are widely found in numerous natural products, bioactive compounds, pharmaceuticals, and polymers and also constitute common versatile building blocks in organic synthesis,[3] are rather challenging as X–H insertion is more favorable in the presence of various metal catalysts, such as those based on Rh, Cu, Ru, Fe, or Pd (Scheme 1).[4] The Fu[5] and Zhou[6] groups have developed elegant metal-catalyzed enantioselective versions for this type of reaction. The development of methods for the site-selective C–H bond substitution of phenols, on the other hand, has long been considered as an attractive but challenging task. Recently, our group[7] and Shi[8] and co-workers independently developed gold-catalyzed highly chemoselective and *para*-selective substitution reactions of phenols by making use of the specific carbophilicity of gold and the strong directing ability of hydroxy groups. In continuation of our interest in C–H bond substitution by carbene transfer,[9] we wished to develop a new catalytic system to realize an intermolecular *ortho*-selective C–H bond substitution of phenols with diazoesters.[10] However, this *ortho*-selective substitution reaction poses more challenges than the *para*-selective one owing to the small differences in the nucleophilicities of the *ortho* and *para* positions of phenols and the greater steric hindrance for the *ortho* position.[11]



Scheme 1. Transformations of phenols with diazoesters.

To overcome the problems described above, we reasoned that a bifunctional hydrogen-bonding catalyst, in which the hydrogen-bond acceptor recognizes the hydrogen-bond donor of the phenol and orients the *ortho*-C–H bond,[12] would facilitate the desired *ortho* substitution of phenols. With this idea in mind, $(C_6F_5)_3B$, a strong Lewis acid that is used for H–H and Si–H bond activation and alkene polymerization and plays a significant role as a component of frustrated Lewis pairs, attracted our attention.[13,14] We hypothesized that a hydrogen bond between a fluorine atom and the hydroxy group could direct the diazo compound to the *ortho* position of phenol, and the boron catalyst could serve as a Lewis acid to activate the diazo compound.[15] Herein, we present the first boron-catalyzed highly chemoselective and *ortho*-selective C–H bond substitution reaction of phenols with α-aryl α-diazoacetates under mild conditions. This method provides reliable and efficient access to diaryl acetates, which are important motifs in biologically active compounds, pharmaceuticals, and natural products (Figure 1).[16]

Our initial experiment was performed with phenol (**1a**) and α-phenyl α-diazoacetate **2a** in the presence of $(C_6F_5)_3B$ (10 mol%) in CH_2Cl_2 at room temperature. As expected, the desired *ortho*-C–H bond substitution product **3aa** was

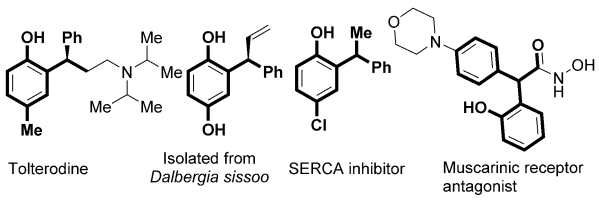


Figure 1. Diaryl acetate subunits in natural products, pharmaceuticals, and bioactive molecules.

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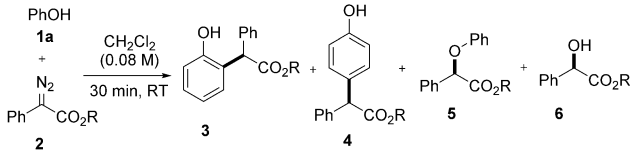
obtained as a single regioisomer in promising yield (57%), along with the O–H insertion product **5aa** (9.5%) and the water insertion product **6aa** (18%; Table 1, entry 1). Then, various boron catalysts were screened but much poorer results were obtained (entries 2–4). These results indicated that the nature of the boron catalyst is crucial for the desired

Table 1: Optimization of the reaction conditions.

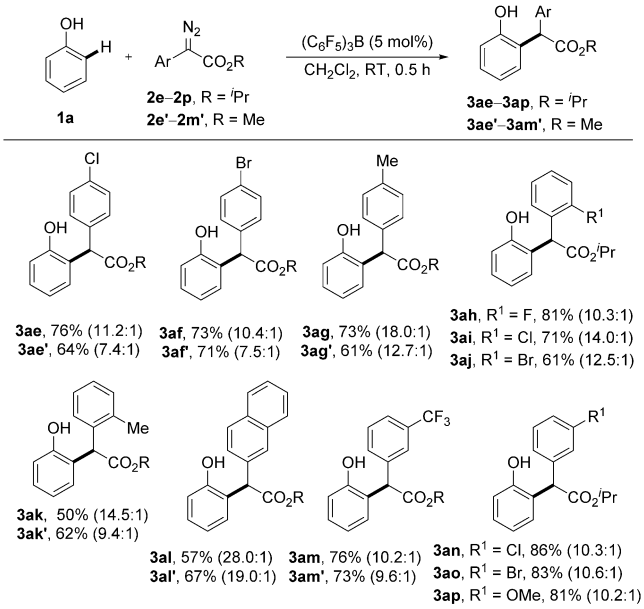


Entry	R (**2**)	Catalyst	Yield[e] [%] **3**/**4**/**5**/**6**
1[a]	Me (**2a**)	$(C_6F_5)_3B$	57/0/9.5/18
2[a,b]	Me (**2a**)	Ar_3B	0/0/5/69
3[a,b]	Me (**2a**)	$(C_6F_5)_2BCl$	0/0/6/92
4[a,b]	Me (**2a**)	$BF_3{\cdot}Et_2O$	17/0/39/8
5[a,c]	Me (**2a**)	$(C_6F_5)_3B$	44/0/8.8/37
6[a,b]	Me (**2a**)	$FeCl_3$	5/15/23/25
7[d]	Me (**2a**)	$(C_6F_5)_3B$	84(75)/0/13/–
8[d]	Et (**2b**)	$(C_6F_5)_3B$	82(74)/0/11/–
9[d]	^{i}Pr (**2c**)	$(C_6F_5)_3B$	92 (90)/0/8.5/–
10[d]	^{t}Bu (**2d**)	$(C_6F_5)_3B$	0/11/0/–

[a] **1a** (0.6 mmol), **2a** (0.4 mmol), catalyst (10 mol%). [b] Run for 12 h. [c] With 4 Å M.S. [d] **1a** (0.4 mmol), **2** (0.6 mmol), catalyst (5 mol%). [e] Determined by NMR spectroscopy using CH_2Br_2 as an internal standard. Yields of isolated products are given in parentheses. Ar = 2,6-$F_2C_6H_3$.

reaction to occur. A solvent screen showed that CH_2Cl_2 is best for this transformation. To suppress the formation of the water-insertion side product **6aa**, 4 Å molecular sieves (M.S.) were added. Unfortunately, this was not beneficial to the reaction (entry 5). We wondered whether certain metals are also suitable catalysts for this unprecedented process. However, no satisfactory results were obtained upon testing a series of metals (see the Supporting Information). The *ortho*-C–H bond substitution product **3aa** was observed only when using $FeCl_3$ as the catalyst, albeit in low yield with low site and chemoselectivity (entry 6). Having identified $(C_6F_5)_3B$ as the best catalyst and CH_2Cl_2 as the best solvent, we turned our attention to other reaction variables. Gratifyingly, the yield was improved to 84% when the amount of diazo compound **2a** was increased, and the catalyst loading could be reduced to 5 mol% (entry 7). Further studies demonstrated that the ester substituent of diazo ester **2** has a significant effect on yield and selectivity. The best result was obtained with an isopropyl group. (entry 9). Astonishingly, only the *para*-C–H substitution product **4ad** (11%) was formed when the more bulky *tert*-butyl diazo ester was used (entry 10).

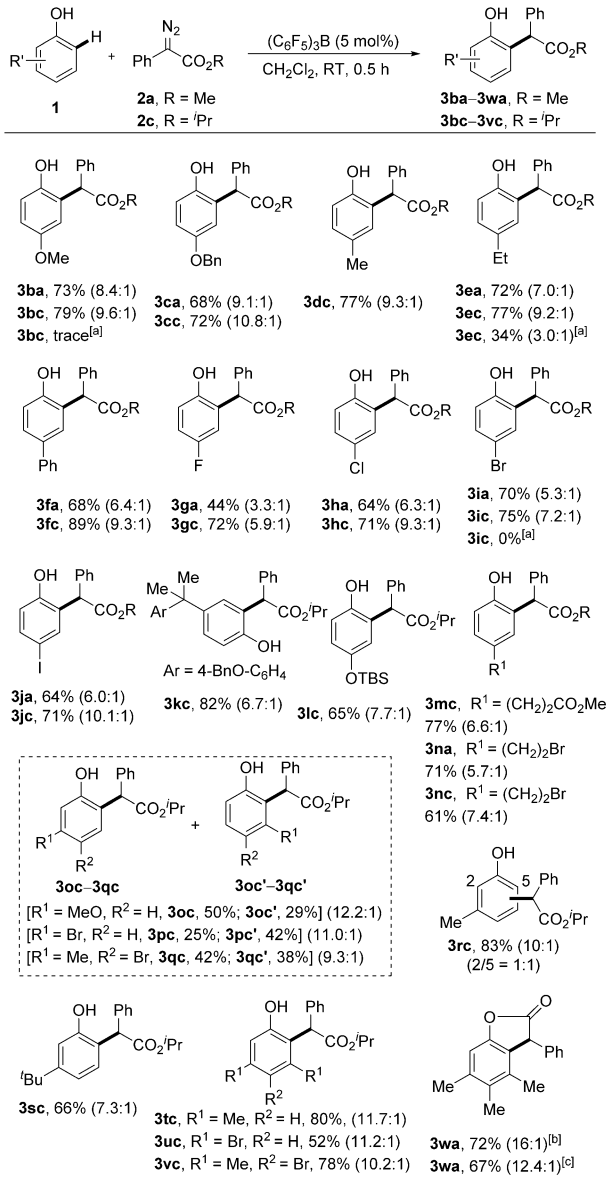
With optimized reaction conditions in hand, we next investigated the scope of this highly chemoselective and *ortho*-selective C–H bond substitution reaction of phenol **1a** with various isopropyl-substituted α-aryl α-diazoacetates **2** (Scheme 2). Our strategy is indeed applicable to a range of



Scheme 2. Variation of the diazo coupling partner. The ratios in parentheses are the ratio of *ortho*-C–H bond substitution to O–H insertion product. Yields of isolated *ortho*-C–H bond substitution products are given.

isopropyl diazo esters with both electron-donating and electron-withdrawing groups on the aryl ring, affording the desired *ortho*-C–H bond substitution products in moderate to good yields (50% to 83%) with high chemoselectivity (>10:1). The amount of diazo compound had to be increased (2.0 equiv) when sterically hindered diazo esters, such as **2k** and **2k′**, were employed. α-Naphth-2-yl α-diazoacetate **2l** also worked well, providing product **3al** in 57% yield with very high chemoselectivity (28:1). Aside from isopropyl α-aryl α-diazoacetates, methyl α-aryl α-diazoacetates **2e′–2m′** could also be used in this transformation but reacted with lower chemoselectivities and gave the C–H substitution products in reduced yields, except for **3ak′** and **3al′**. The structure of **3aj** was confirmed by single-crystal X-ray crystallography.[17] It is noteworthy that all of the reactions are *ortho*-selective, and that the *para*-C–H bond functionalization products were not observed.

The reactions between various substituted phenols and α-phenyl α-diazoacetates were then examined. As depicted in Scheme 3, the reactions proceeded smoothly, affording the desired *ortho*-functionalized products in moderate to good yields and chemoselectivity. The reactions also showed a remarkable substituent effect. For *meta*-substituted phenols, two regioisomers were usually obtained. However, only one isomer (**3sc**)was formed through substitution of the C–H bond *para* to the substituent when a phenol derivative with a bulky *tert*-butyl substituent in the *meta* position was employed. On the other hand, *ortho*-substituted phenols were found to be incompatible with this transformation, furnishing only trace amounts or no product at all. We surmised that this might be due to the hydrogen-bonding interaction being prevented by steric hindrance. Interestingly, lactone **3wa** was obtained by tandem C–H substitution and

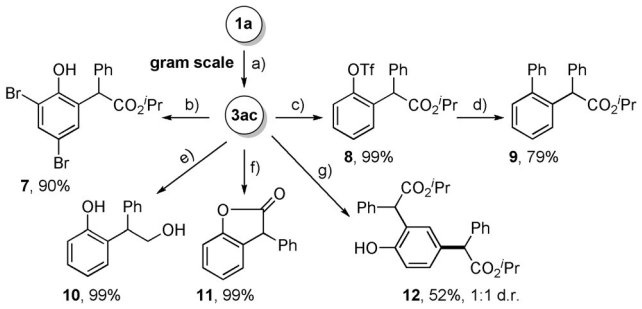


Scheme 3. Variation of the phenol component. The ratios in parentheses are the ratio of the *ortho*-C–H bond substitution to the O–H insertion product. Yields of isolated *ortho*-C–H bond substitution products are given. [a] (2,4-^{t}Bu$_2$C$_6$H$_3$O)$_3$PAuSbF$_6$ (5 mol%), CH_2Cl_2 (0.08 M), RT. [b] **2a** was used. [c] **2c** was used. Bn = benzyl, TBS = *tert*-butyldimethylsilyl.

cyclization when sterically congested 3,4,5-trimethylphenol was used.

In previous work, we had disclosed the gold-catalyzed *ortho*-C–H bond functionalization of *para*-substituted phenols with diazoacetates.[7] We thus compared the gold-catalyzed process with the present catalyst system, and the results revealed that the present $(C_6F_5)_3B$ catalyst system has obvious advantages over the gold method (Scheme 3; **3bc**, **3ec**, **3ic**).

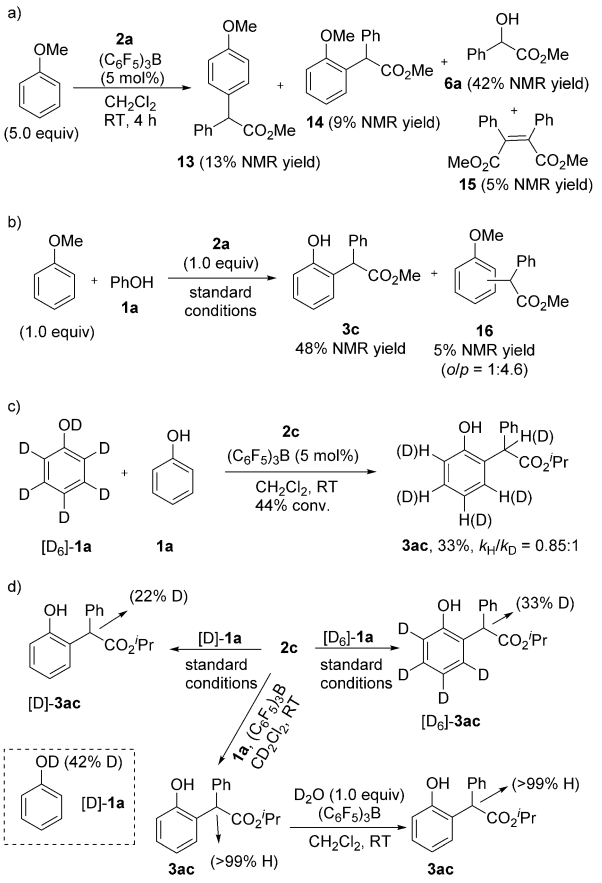
To our delight, the present method can be easily scaled up (Scheme 4). A gram-scale reaction of **1a** with **2c** was performed at lower catalyst loading (1 mol%), and afforded the desired product **3ac** (1.1 g, 77%). Moreover, these *ortho*-



Scheme 4. Gram-scale reaction and synthetic applications. Reaction conditions: a) $(C_6F_5)_3B$ (1 mol%), **2c** (1.5 equiv), CH_2Cl_2 (0.53 M), RT; b) **3ac**, NBS (2.5 equiv), CH_2Cl_2/DMF (5:1), 0 °C; c) **3ac**, pyridine (2.0 equiv), Tf_2O (2.0 equiv), CH_2Cl_2, 0 °C to RT; d) **8**, phenylboronic acid (1.5 equiv), $Pd(PPh_3)_4$ (10 mol%), Cs_2CO_3 (1.5 equiv), THF/H_2O (10:1), 70 °C; e) **3ac**, $LiAlH_4$ (2.0 equiv), THF, 0 °C; f) **3ac**, TFA (20 mol%), toluene, 90 °C; g) **3ac**, (2,4-^{t}Bu$_2$C$_6$H$_3$O)$_3$PAuSbF$_6$ (5 mol%), **2c** (0.67 equiv), CH_2Cl_2, RT. NBS = *N*-bromosuccinimide, Tf = trifluoromethanesulfonyl, TFA = trifluoroacetic acid.

C–H bond substitution products could be used as versatile synthons. For example, bromination of **3ac** afforded **7** in 90% yield. In addition, hydroxy groups are common precursors for coupling reactions. Coupling product **9** was obtained in 79% yield after converting the hydroxy group into triflate **8**. Reduction of **3ac** with $LiAlH_4$ gave alcohol **10** in 99% yield, and TFA-catalyzed lactonization could afford benzofuranone **11** in excellent yield, which is a prominent structural motif in natural products.[18] Finally, further *para*-C–H functionalization could be achieved with a gold catalyst, furnishing **12** in 52% yield.

To gain mechanistic insight, several control experiments were carried out (Scheme 5). First and foremost, we wondered whether the *ortho* selectivity arise from hydrogen-bonding interactions or not. With this mind, NMR titration experiments were carried out (see the Supporting Information for details). ^{19}F NMR analysis showed that the single ^{19}F resonance of $(C_6F_5)_3B$ was shifted downfield when more phenol was mixed with $(C_6F_5)_3B$ while the ^{11}B resonance (−0.68 ppm) of $(C_6F_5)_3B$ did not undergo any changes; this finding is incompatible with the formation of a four-coordinate boron intermediate.[19] These results indicated that the predominant interaction between phenol and $(C_6F_5)_3B$ was due to hydrogen bonding and not to coordination between B and OH. Based on the above results, we hypothesized that the key to *ortho*-C–H substitution is the hydrogen bond between a fluorine atom and the hydroxy group. Subsequently, the reaction between anisole and **2a** was performed, which gave the *para*- and *ortho*-C–H functionalization products in only 13% and 9% yield, respectively (determined by NMR spectroscopy, Scheme 5a). Furthermore, competition experiments between phenol and anisole with **2a** under the standard reaction conditions showed that the anisole has a much lower reactivity than the corresponding phenol. These results further support the hypothesis that the *ortho* selectivity is due to a hydrogen-bonding interaction between the phenol and the reactive intermediate (Scheme 5b). Moreover, the reaction does not exhibit a kinetic isotope effect, revealing



Scheme 5. Preliminary mechanistic studies.

that the C–H cleavage is not involved in the rate-determining step, and the reaction may proceed by electrophilic addition (Scheme 5c). Furthermore, control experiments showed that the hydroxy group acts as a proton source during the reaction as a deuterium was incorporated in the final product when [D]-**1a** was used. The proton at the α-position of the acetate in the product, however, did not undergo proton–deuterium exchange under the reaction conditions, indicating that no enolization occurred (Scheme 5d). Stephan and co-workers have demonstrated that a very interesting C_6F_5 group migration reaction occurs upon mixing $(C_6F_5)_3B$ with α-alkyl diazo esters.[20] To determine whether a similar C_6F_5 group migration reaction takes place in our process, control experiments were carried out, which showed that the water insertion reaction is preferred over C_6F_5 migration with the α-phenyl diazo ester as the water insertion product was obtained in 99% yield (see the Supporting Information). It is noteworthy that the migration product was indeed formed under the same conditions.[20b] To our surprise, when α-methyl diazoester **2q** was subjected to the reaction with phenol, the *ortho* substitution and the OH insertion product were obtained in 8 and 11% yield, respectively (determined by NMR spectroscopy; see the Supporting Information for details). These results are consistent with previous observations that α-aryl diazo esters often react very differently to α-alkyl diazo esters.[1,21]

In conclusion, we have described the first catalytic *ortho*-selective C–H substitution of unprotected phenols with $(C_6F_5)_3B$ as the catalyst. This transformation constitutes a simple, efficient, and reliable approach to a variety of useful diaryl acetates under mild conditions. This reaction is a hydrogen-bond-directed process, which was supported by NMR studies and control experiments. This work represents a rare example of $B(C_6F_5)_3$ catalyzed C–C bond formation[14] and will broaden the application of $(C_6F_5)_3B$ in organic synthesis.

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[17] CCDC 1495563 (**3aj**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre.

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