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Synthesis, Structure and Reactivity of 5-(Aryl)dibenzothiophenium Triflates

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Abstract: A synthetic protocol for the preparation of 5-(aryl)dibenzothiophenium salts starting from inexpensive dibenzothiophene *S*-oxide and simple arenes is reported. The scope of the method regarding the nature of the arene is evaluated, intermediates along the reaction sequence have been trapped, and side reactions limiting the scope identified. In addition, the X-ray structures of a complete set of these salts are reported and their reactivity studied. Specifically, chemoselective Suzuki coupling over iodides is observed when the dibenzothiophenium (DBT) unit is used as linchpin for this transformation.

Introduction

Although the use of sulfonium moieties as linchpins in cross coupling chemistry^[1] and as source of organic radicals has been known for more than two decades,^[2] it has not been until very recently that the actual synthetic potential of these reagents has been broadly recognized. Variants of the Pd-catalyzed Suzuki-Miyaura,^[3] Mizoroki-Heck,^[4] Sonogashira,^[5] Negishi,^[6] Stille,^[7] and related reactions such as Buchwald-Hartwig aminations^[8,9] are now available employing aryl sulfonium salts as electrophilic partners. Moreover, either by direct reaction with nucleophiles or through photoredox catalyzed processes, the sulfonium moiety can be selectively exchanged by alkyl groups, halogens or amines under really soft reaction conditions.^[10,11] Aside from this wide range of applications, additional beneficial attributes are associated with the use of sulfonium salts. Namely, their easy and regioselective preparation from commercially available sulfides^[12] or sulfoxides,^[13] non-volatility, and comparatively higher thermal stability than diazonium salts.

Very recently, our research group reported on the synthesis of related 5-(cyano)- and 5-(alkynyl) dibenzothiophenium salts via triflic acid anhydride activation of the parent dibenzothiophene S-oxide.^[14] Given the synthetic potential of S-(aryl)sulfonium salts and the lack of a detailed study on the reaction sequence leading to their formation from dibenzothiophene S-oxide 1; we decided to carefully study this synthetic route, evaluate its scope regarding the nature of the arene, and further explore the reactivity of the sulfonium salts obtained.^[15]

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Figure 1. Schematic synthesis of 5-(aryl)dibenzothiophenium triflates.

Results and Discussion

Trapping of bistriflate 2 and characterization of its thermally induced decomposition products. We started our investigation by attempting the isolation of compound 2, the species expected from the activation of sulfoxide 1 with Tf₂O. Bistriflates of that general structure are usually proposed as the products of activation of sulfoxides when treated with Tf₂O; however, no structural characterization of salts of such connectivity has been reported to date. Thus, we slowly added Tf₂O to a cooled (-50°C) dichloromethane solution of sulfoxide 1 obtaining an intensive red suspension.^[14] All our attempts to obtain monocrystals of 2 under these conditions failed, while warming the reaction mixture to -20 °C provoked the disappearance of the red color and precipitation of a beige solid. Along this transformation the sample remained EPR silent. Chromatographic analysis of the reaction mixture allowed the isolation of salts 4 and 5 in moderate yields, together with a highly insoluble material (Scheme 1a).

Formation of sulfone 4 and bis(sulfonium) salt 5 speaks for a disproportionation of 2 into the corresponding sulfide and sulfone, followed by reaction of both species with one or two equivalents of remaining 2, respectively (Scheme 1a). The X-ray structure of 4, in which the sulfonium substituent adopts a metadisposition relative to the SO2 group, evidences that this compound was not formed by oxidation of 6 (Scheme 1b). On the other hand, the necessary intermediate for the formation of 5, sulfonium salt 6 was not observed in this experiment, but it can be obtained in nearly quantitative yield by reaction of 2 with one equivalent of dibenzothiophene. Moreover, isolated 6 is able to further react with a second equivalent of 2 to afford 5 (Scheme 1a). Hence, we believe that the disproportionation of 2 is a slow process if compared with the electrophilic aromatic substitution steps leading to 4 and 5. Finally, MS analysis of the remaining insoluble fraction indicated the formation of highly charged oligomeric species containing four and five dibenzothiophene units, which can be explained by further reaction of 5 with additional 2 (See the SI). Attempts to inhibit these side reactions by blocking positions 2- and 8- of the dibenzothiophene core with fluorine substituents did not improve the reaction outcome,

instead sulfonium salt 8 was the only product isolated from the complex mixture (Scheme 1c). Further analysis is still necessary to explain the formation of 8.

a) Isolated species



Scheme 1. Thermally induced decomposition products of 2. Reagents and conditions: (a) Tf₂O (1.1 equiv.), -50°→-20°C, 4h.; 4, 11%; 5, 19%; (b) Tf₂O (1.1 equiv.), -50° and then dibenzothiophene (1.1 equiv.), 6, 95%; (c) Tf₂O (1.1 equiv.), -50° and then 6, 5, 62%. Molecular structures of compounds 4, 6 and 8 in the solid state. Anisotropic displacement shown at 50% probability level. Hydrogen atoms and triflate anions omitted for clarity. [16]

Finally, a plausible reaction pathway for the formation of sulfone derivatives is depicted in Scheme 1d. We hypothesize an equilibrium for the formation of 2 from 1 and Tf₂O. The attack of sulfoxide 1 to the sulfur atom in 2, leads to the formation of an oxygen-bridged disulfonium salt, [17] which presumably rearranges to dibenzothiophene and dibenzothiophene-5,5dioxide by asymmetric cleavage of the bridge.

Being aware of the difficulty that the isolation of 2 entails, we decided to chemically trap it. It was hypothesized that the use of a monodentate neutral ligand able to engage in strong odonation to the S(IV) center of 2 might provide sufficient stabilization to allow the isolation of the corresponding adduct before side reactions take place. Hence, one equivalent of DMAP was added to a freshly prepared suspension of 2 at -50 °C. This causes the slow formation of an orange/yellow solid 9, which could be obtained in analytically pure form after washing with dry dichloromethane. X-ray diffraction analysis of monocrystals obtained by cooling a saturated dichloromethane solution confirmed the formation of the expected Lewis adduct 9 (Scheme 2a).



Scheme 2. Coordination of Lewis bases to 2. Reagents and conditions: (a) DMAP (1. equiv.), -50 °C →rt, DCM, 9, 37%; (b) 1,3-Diisopropyl-4,5-dimethylimidazol-2-ylidene (1.0 equiv.), THF 10, 20%. Molecular structure of compound 9. Anisotropic displacement shown at 50% probability level. Hydrogen atoms, non-coordinating triflate anion and solvent omitted for clarity. Selected bond lengths [Å]: C1-S1, 1.773(2); C4-S1, 1.777(2); S1-N1, 1.734(1); S1...O1, 2.991(1); S1...O4, 3.821(1); S1...O7, 2.769(1); S1...O8, 3.232(1); S2-C20, 1.778(2), S2-C27, 1.781(1); S2-N3, 1.735(1); S2-··O3, 2.986(1); S2-··O6, 2.963(1); S2-··O8, 2.534(1).^[16]

In the solid state, adduct 9 adopts a dimeric structure in which the geometry around each dibenzothiophene S-atom can be defined as distorted octahedral. Two vertexes are occupied by the chelating biphenyl rest from the dibenzothiophene unit while the pyridine ligand is positioned in a third vertex adopting a cis- relation with both arene substituents. The other three vertexes from the octahedron are each occupied by one triflate anion, which additionally act as bridges with a second S center. Five from the six S-O contacts (S1...O1, 2.991(1); S1...O4, 3.821(1); S1...O7, 2.769(1); S2...O3, 2.986(1); S2...O6,

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2.963(1); S2...O8, 2.534(1)) are significantly shorter than the sum of the van der Walls radii of the corresponding elements (3.32 Å) manifesting a significant Lewis acid character at the S-atom. The isolation of **9** unequivocally demonstrates that the primary product of activation of sulfoxide **1** with Tf₂O is a dicationic S(IV) species. Moreover, **9** is still electrophilic and upon treatment with one equivalent of a stronger σ -donor, such as 1,3-diisopropyl-4,5-dimethyl-imidazol-2-ylidene, ligand exchange takes place affording the carbene adduct **10**.^[18]

Synthesis of 5-(aryl)dibezothiophenium triflates: Scope and

limitations. The next step in our investigation was the study of the reaction between **2** and aromatic substrates. Hence, a series of functionalized arenes were carefully added to freshly prepared suspensions of **2** at -50°C, and the mixture allowed to warm up to -20°C during a period of 12 h. Under these conditions, deleterious side reactions were minimized and a remarkable scope of sulfonium salts **3a-r** was obtained. This synthetic route allows access to the desired compounds without the use of I(III)-reagents.^[19]



Scheme 3. Synthesis of 5-(aryl)dibenzothiophenium triflates. If not indicated, only one regioisomer was detected. Reagents and conditions: (a) Arene (1.1 equiv.) $-50^{\circ} \rightarrow -20^{\circ}$ C, 15h. [#]Isolated as a 1:2 mixture of rotamers.

Ideal substrates for this transformation are moderate electron rich arenes and heteroarenes such as anisole derivatives, alkyl- or aryl-substituted benzenes, haloarenes, and thiophenes. In most of the cases the reaction proceeded in good to excellent yields and satisfactory *para-/ortho-* ratios. In addition, it is tolerant to halogens, ethers, esters, and heteroaromatic skeletons (Scheme 3). The reactivity of **2** and **9** is similar in terms of conversion to **3a** when benzene is used as substrate. For this range of aromatic compounds we have no evidence against the formation of sulfonium salts **3** via electrophilic aromatic substitution, even the cyclopropane ring of **3h** survives; however, single electron oxidation of the substrate by **2** followed by immediate recombination of the two radicals cannot be excluded (Scheme 4).^[20]



Scheme 4. Proposed mechanism for the sulfonium salt formation when using substrates of moderate electron richness.

Figure 2 depicts the molecular diagrams of salts **3m** and **3o** in the solid state; see the SI for the crystallographic structures of **3b-I**. It is worth noting that interactions between the S-atom and the triflate counteranion, reminiscent to the ones observed in **9**, are again detected along the complete series; S1...O1 distances (2.990(1)Å for **3m**, and 3.057(2) Å for **3o**, respectively) are shorter than the sum of the van der Waals radii, revealing the still remarkable electrophilic character at that position.



Figure 2. Molecular structures of compounds **3m** (left) and **3o** (right) in the solid state. Anisotropic displacement shown at 50% probability level. Hydrogen atoms omitted for clarity. Selected bond lengths [Å] and angles [deg]: **3m**, C1-S1, 1.776(1); S1...O1, 2.990(1); C1-S1-O1, 80.8(1); **3o**, C1-S1, 1.763 (3); S1...O1, 3.057(2); C1-S1-O1, 77.1(1).^[16]

Electron poor rings such as 1,2-dichlorobenzene are still able to render the corresponding sulfonium salt (3k) by reaction with 2, albeit in moderate to low yields. Careful column chromatography is necessary to separate 3k from side products 4 and 5, which are formed in considerable amounts. Electron

poorer substrates such as 1,4-difluorobenzene or trifluoromethylbenzene are inert to the attack of **2**. In these essays only decomposition products **4** and **5** together with polycationic dibenzothiophene oligomers are observed.

(Hetero)aromatic rings from the other side of the electron richness spectrum such as anilines or indols are problematic as well, but for different reasons. For example, reaction of 2 with Nmethylindole (11) afforded a complex mixture of products from which dibenzothiophene is the main product; sulfonium salts 6 and 3s, and bisindole 12 were isolated as well. Additionally, triand tetraindolic structures were detected by MS analysis. This outcome suggests, as reported by Kita when pyrroles are treated with hypervalent I(III) reagents,^[21] that single electron oxidation of methylindole by 2 takes place yielding an indole radical cation, which oxidatively couples with more indole producing 12 and indole oligomers. Simultaneously, the dibenzothiophene being formed partially reacts with 2 to afford 6. Additional evidence of the feasibility of a radical mechanism when easy to oxidize substrates are employed derives from the reaction of 2 with stoichiometric amounts of TEMPO. This essay affords a clean mixture of 6 (75% isolated yield) and TEMPO oxoamonium triflate 13.



Scheme 5. Reaction of **2** with 1-methylindole. Reagents and conditions: (a) 1-methylindole (1.1 equiv.), CH_2Cl_2 , $-50 \rightarrow -20^\circ C$; b) CH_2Cl_2 , $-50 \rightarrow -20^\circ C$.

modification of Post-synthetic sulfonium salts via nucleophilic aromatic substitution and synthetic applications. Given their electron richness, aniline substrates are not well-tolerated by the protocol just described. However, sulfonium salts containing these substituents might be synthetically useful compounds because halo-anilines are not always easy to obtain. In fact, classical Friedel-Crafts aniline halogenation is a difficult to control reaction, with polyhalogenation taking place readily.^[22] We rationalized at this stage that aniline-substituted sulfonium salts might be obtained in a two-step sequence starting from fluorobenzene derivative 3c. In this salt the fluoride is expected to be activated towards nucleophilic aromatic substitution due to the strong electron withdrawing nature of the para-located sulfonium moiety. Should that substitution be feasible, reaction of **3c** with amines may deliver the desired aniline-derived sulfonium salts. Actually, it was found that a variety of secondary amines were suitable for that reaction leading to a set of sulfonium salts **14a-e** containing structurally differentiated aniline substituents (Scheme 6). Chlorobenzene derivative **3d** is unable to deliver products **14a-e** under identical conditions.



Scheme 6. Synthesis of aniline substituted dibenzothiophenium triflates by nucleophilic aromatic substitution.

Finally, it has been recently reported that thianthrenium cations are able to led site-control over typical linchpins such as C-Br and C-OTf bonds in coupling reactions.^[11b] We rationalized that DBT, being an electron poorer unit than the thianthrenium one, should favor even more the insertion of Pd along the C-S bond. Hence, we decided to challenge the well-established C-I site selectivity in the Suzuki coupling versus the C-DBT one.^[23] Specifically, salt **3I** was chosen as substrate because the iodo and dibenzothiophenium units are in identical stereoelectronic environments but independent rings. One equivalent of 4- (trifluoromethyl)phenyl boronic acid was used for this experiment (Scheme 7).



Scheme 7. Chemoselective C-DBT Suzuki coupling over C-I bonds.

From this assay, carried out at 35°C and employing only 0.5 mol% of Pd(AcO)₂ as catalyst, the product of selective arylation at the C-DBT bond, **15**, was isolated in 90% yield. By MS spectrometry we were able to detect traces of thioether **16**, which derives from initial insertion of Pd into one of the internal C-S bonds of the dibenzothiophene moiety, and traces of the product resulting from coupling at the C-I site. This result demonstrates intrinsic chemoselectivity of C-DBT over C-I positions in the Suzuki reaction. From the mild conditions and simple catalyst employed to promote the coupling, and the exquisite site-selectivity observed, we envision the use of DBT units as a promising linchpin to enable rapid access to densely functionalized polyarenes via coupling reactions.

Conclusions

In summary, we report herein a straightforward method for the synthesis 5-(aryl)dibenzothiophenium triflates by metal free sulfenylation of C-H bonds. Preliminary mechanistic studies based on intermediate trapping and the careful isolation of decomposition products suggests that an electrophilic aromatic substitution is operative for the sulfenylation of electron neutral or moderately rich arenes. The generation of radicals via single electron transfer between 2 and the substrate is more conceivable for electron richer aromatics, leading to complex reaction mixtures. Comparison of the reactivity of the DBT linchpin with that of C-I bonds in a model Suzuki reaction reveals full control of the DBT moiety over the competing C-I coupling site. Hence, we anticipate a widespread range of application for 5-(aryl)dibenzothiophenium triflates in Pd-chemistry; in particular for the execution of sequential biaryl couplings leading to the construction of polyarenes.

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Keywords: sulfonium salts • dibenzothiophenium cations • linchpin • Pd-catalysis • Site-selective Suzuki coupling

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Easy to put on, easier to take off: 5-(aryl)dibenzothiophenium triflates were prepared by highly selective metal free C-H sulfenylation of arenes. Interestingly, these salts undergo site selective Suzuki-Miyaura coupling in the presence of C-I bonds, enabling the iterative synthesis of polyaromatics.

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