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Synthesis of 1-Aryltetralins and 1-Arylnaphthalenes via (4 + 2) Annulation of β-Ketosulfones with Styryl Bromides

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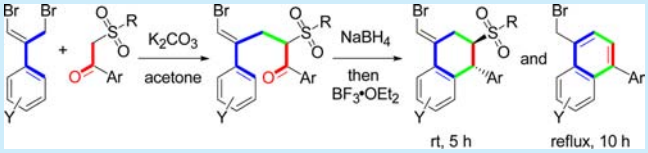
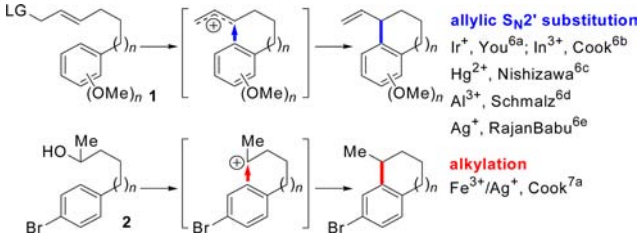
Supporting Information

ABSTRACT: A novel route has been developed for the synthesis of various substituted 1-aryltetralins **6** and 1-arylnaphthalenes **8** via (1) K_2CO_3-mediated α-styrylation of β-ketosulfones **3** with bromostyryl bromides **4** and (2) stereocontrolled $NaBH_4$-promoted reduction of the resulting γ-alkenones **5**, followed by $BF_3{\cdot}OEt_2$-catalyzed intramolecular annulation of the corresponding γ-alkenols **7** under rt/5 h and reflux/10 h conditions, respectively. The key structures of **6** and **8** were confirmed by X-ray crystallographic analysis. A plausible mechanism has been proposed.



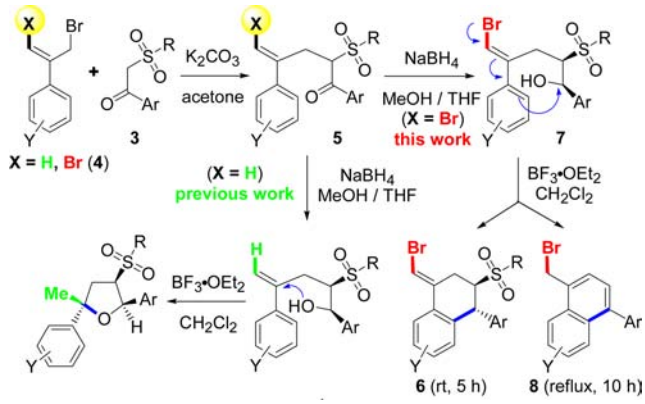
Tetralin is a highly privileged core structure found in many valuable biologically active molecules, pharmaceuticals, and natural products.[1−4] With regard to the construction of this key building block, the 90-year-old Darzens tetralin synthesis (H_2SO_4-mediated intramolecular ring-closure of a 1-phenyl-4-pentene)[5] still stands out as one of the most direct and convenient methods. It uses arenes with an allylic chain[6] **1** or an unactivated alcohol[7] **2** as the starting materials; it is especially effective when combined with the recent remarkable advances in metal-catalyzed annulation. Various catalysts have been employed to accelerate the formation of a carbon−carbon bond via a carbocation-like intermediate (Scheme 1).[8]

Scheme 1. Intramolecular Friedel−Crafts Substitution Reaction



As part of our recent studies of $BF_3{\cdot}OEt_2$-promoted tandem cyclization reactions,[9] we devised an efficient stereocontrolled synthesis of sulfonyl 2,5-diaryltetrahydrofurans in good yield via the Lewis acid promoted intramolecular hydroalkoxylation of sulfonyl γ-alkenols (X = H),[10] as shown in Scheme 2. In this paper, we report the $BF_3{\cdot}OEt_2$-mediated intramolecular Friedel−Crafts electrophilic substitution of sulfonyl γ-alkenols (X = Br) by tethering a β-bromovinyl group to the aryl rings. In contrast to a hydrogen atom, the weakly deactivating bromo group initiates the stereospecific progression of the delocalized electron pair (in an *exo* → *endo* → *exo* direction), leading to the formation of a six-membered ring under metal-free catalyzed conditions. This stereospecific annulation reaction provides facile access to

Scheme 2. Synthetic Route of 2,5-Diaryltetrahydrofurans, 1-Aryltetralins, and 1-Arylnaphthalenes



functionalized 1-aryltetralins. However, to the best of our knowledge, the bromo group-initiated intramolecular Friedel−Crafts alkylation procedure for the preparation of 1-aryltetralins has not been reported,[11] nor has the intramolecular annulation of bromostyryl β-ketosulfones been explored. Herein, we report our studies[12,13] on these two processes for aryltetralin synthesis.

K_2CO_3-mediated α-styrylation of β-ketosulfones **3a−r** with bromostyryl bromides **4a−d** (prepared by *p*-TsOH-catalyzed double bromination of styrene with 2.5 equiv of NBS in CH_2Cl_2 at reflux) in acetone at reflux provided **5a−x** in a yield range of 78−89%, as shown in Table 1. The yields of **5a−x** did not change much as a function of the structure of **3a−r** under these reaction conditions. The bromo group promoted intramolecular ring closure of **5** was examined via a two-step route: stereoselective $NaBH_4$-mediated reduction followed by $BF_3{\cdot}OEt_2$-promoted Friedel−Crafts annulation of the resulting sulfonyl 4-alkenols. For different R substituents on **5**, a diversity of electron-

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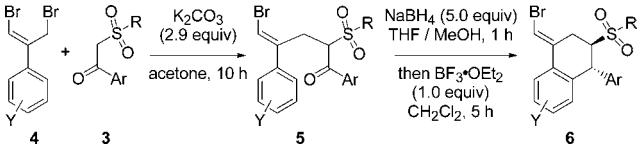
Table 1. Synthesis of 5 and 6[a,b]



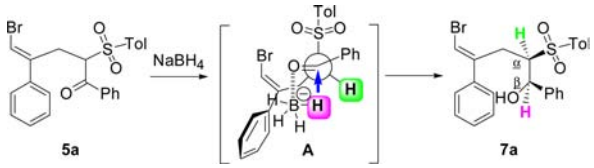
entry	3, Ar = , R =; 4, Y-Ar =	yield[c] (%) 5	6
1	**3a**, Ph, Me; **4a**, Ph	**5a**, 80	**6a**, 82
2	**3b**, Ph, Ph; **4a**, Ph	**5b**, 84	**6b**, 80
3	**3c**, Ph, Tol; **4a**, Ph	**5c**, 89	**6c**, 76
4	**3d**, 4-FC_6H_4, Tol; **4a**, Ph	**5d**, 83	**6d**, 78
5	**3e**, 4-$MeOC_6H_4$, Tol; **4a**, Ph	**5e**, 86	**6e**, 74
6	**3f**, 4-MeC_6H_4, Tol; **4a**, Ph	**5f**, 83	**6f**, 70
7	**3g**, 4-$CF_3C_6H_4$, Tol; **4a**, Ph	**5g**, 80	**6g**, –[d]
8	**3h**, 4-$NO_2C_6H_4$, Tol; **4a**, Ph	**5h**, 80	**6h**, –[d]
9	**3i**, 3-$NO_2C_6H_4$, Tol; **4a**, Ph	**5i**, 82	**6i**, –[d]
10	**3j**, 4-PhC_6H_4, Tol; **4a**, Ph	**5j**, 84	**6j**, 74
11	**3k**, 2-naphthalene, Tol; **4a**, Ph	**5k**, 88	**6k**, 70
12	**3l**, Ph, 4-FC_6H_4; **4a**, Ph	**5l**, 82	**6l**, 78
13	**3m**, Ph, 4-$MeOC_6H_4$; **4a**, Ph	**5m**, 80	**6m**, 81
14	**3n**, Ph, 3-MeC_6H_4; **4a**, Ph	**5n**, 84	**6n**, 82
15	**3o**, Ph, 4-EtC_6H_4; **4a**, Ph	**5o**, 82	**6o**, 78
16	**3p**, Ph, 4-*n*-BuC_6H_4; **4a**, Ph	**5p**, 83	**6p**, 74
17	**3b**, Ph, Ph; **4b**, 4-FC_6H_4	**5q**, 84	**6q**, 80
18	**3c**, Ph, Tol; **4b**, 4-FC_6H_4	**5r**, 83	**6r**, 80
19	**3b**, Ph, Ph; **4c**, 4-PhC_6H_4	**5s**, 84	**6s**, 78
20	**3c**, Ph, Tol; **4c**, 4-PhC_6H_4	**5t**, 85	**6t**, 76
21	**3c**, Ph, Tol; **4d**, 2-naphthalene	**5u**, 82	**6u**, 80
22	**3k**, 2-naphthalene, Tol; **4d**, 2-naphthalene	**5v**, 79	**6v**, 80
23	**3q**, 3,4-$Cl_2C_6H_3$, Tol; **4a**, Ph	**5w**, 78	**6w**, 81
24	**3r**, 2-thiophene, Tol; **4a**, Ph	**5x**, 80	**6x**, 82

[a]Alkylation reaction: **3** (1.0 mmol), K_2CO_3 (2.9 mmol), **4** (1.05 mmol), acetone (15 mL), reflux, 10 h. [b]Annulation reaction: **5** (0.2 mmol), $NaBH_4$ (1.0 mmol), THF (5 mL)/MeOH (5 mL), 0 °C, 1 h, then $BF_3{\cdot}OEt_2$ (0.2 mmol), CH_2Cl_2 (5 mL), 25 °C, 5 h. [c]Isolated yields. [d]No reaction; **7g** (89%), **7h** (92%), and **7i** (90%) were recovered.

withdrawing and electron-donating groups was well-tolerated. However, when the Ar group was strongly electron-withdrawing, none of the desired products **6g–i** were formed, and only **7g–i** were observed. Thus, in the presence of 3- or 4-nitrophenyl and 4-(trifluoromethyl)phenyl groups, no intramolecular annulation was observed. Moreover, for entries 7–9, different substituents on **5** did not affect the annulation, and the isolated yield of **6** was maintained. The structures of **5e**, **6c**, **6f**, **6l**, and **6u** were all determined by single-crystal X-ray crystallography.[14]

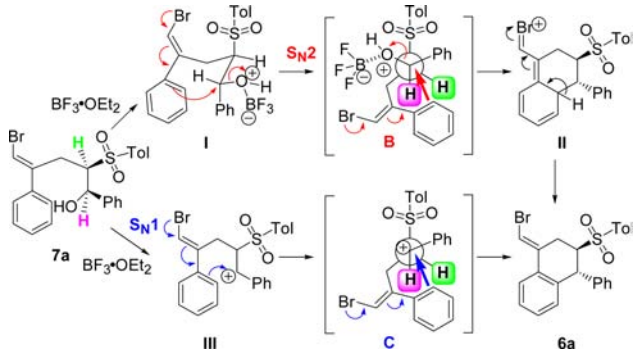
In accord with our previous experience, a single skeleton derived from the sulfonyl 4-alkenol was obtained. On the basis of the Felkin–Anh model **A**,[15] the stereochemical centers of two protons (green H–C_α and pink H–C_β in **7a**) are well established. As shown in Scheme 3, a plausible mechanism

Scheme 3. Proposed Reduction Mechanism



suggests that $NaBH_4$-mediated carbonyl reduction of **5a** is affected by the steric hindrance of the sulfonyl substituent such that the hydride can only attack the less hindered carbonyl face, yielding a single stereoisomer.[12b] Subsequently, complexation of the resulting hydroxyl group by $BF_3{\cdot}OEt_2$ gives **I** (Scheme 4).

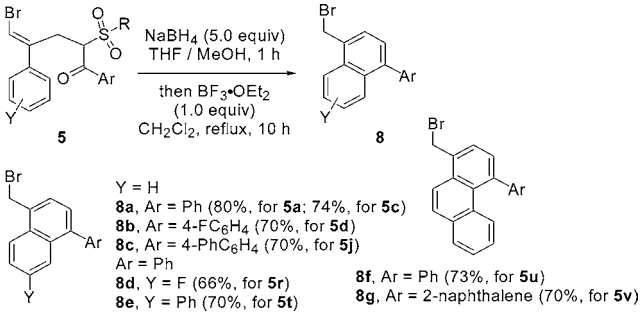
Scheme 4. Proposed Annulation Mechanism



Following model **B**, a stereocontrolled bromo group-initiated intramolecular S_N2 annulation leads to **II** via a six-membered chairlike conformation having two adjacent equatorial protons. An S_N1 process can also be envisioned with **III** (model **C**), deprotonation of **II** affording **6a** via a sequential aromatization process.

The $BF_3{\cdot}OEt_2$-mediated intramolecular annulation of **5** was investigated at elevated temperatures and longer reaction times, as shown in Scheme 5. Specifically, changing the reaction

Scheme 5. Synthesis of 8



conditions from 25 °C and 5 h to CH_2Cl_2 at reflux for 10 h generated substituted 1-bromomethyl naphthalenes **8a–e** (66–80%) and 1-bromomethyl phenanthrenes **8f,g** (73% and 70%) via $BF_3{\cdot}OEt_2$-promoted cascade desulfonative aromatization of **5a,c,d,j,r,t–v**. 1-Arylnaphthalene has been shown to exhibit a wide range of potential biological activities.[16] The present protocol provides a novel synthetic route for the preparation of substituted naphthalenes.[17] The structure of **8d** was determined by single-crystal X-ray crystallography.[14]

By changing the α-substituent from a sulfonyl group to an ethyl ester group (**3s**, Ar = Ph; **3t**, Ar = 4-$MeOC_6H_4$), **9a** and **9b** were isolated in 80% and 82% yields, respectively, as shown in Scheme 6. Using the above synthetic protocol, **10a,b** were generated in 73% and 74% yields. Comparing the 1,3-dicarbonyl synthons, both β-ketosulfones and β-ketoesters provided similar yields of 1-aryltetralins. However, **9c** containing a β-diketone group (prepared by alkylation of **3u** with **4a**) failed to afford the tetralin skeleton under the standard reaction conditions.

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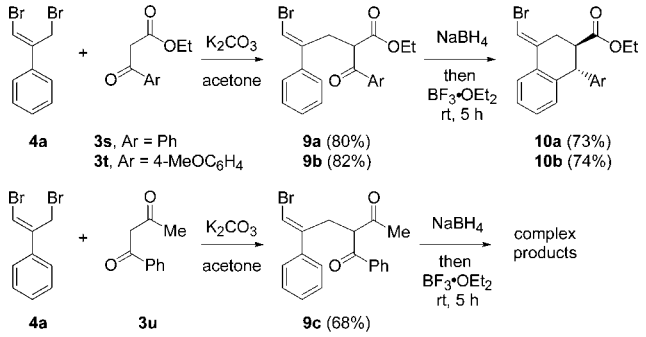
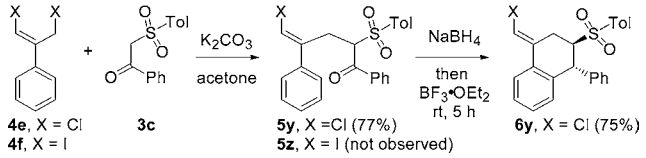
Scheme 6. Reaction of 4a with 3s−u



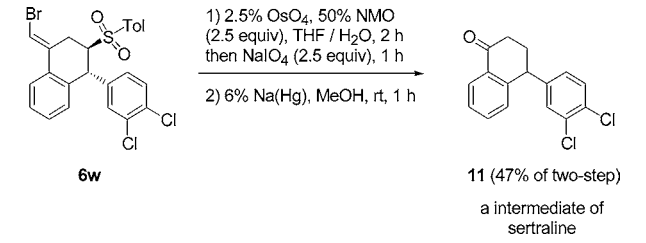
In further work, the bromo group was replaced with a chloro or iodo group, affording facile access to 1-aryltetralins. As shown in Scheme 7, α-styrylation of **3c** with chlorostyryl chloride **4e**

Scheme 7. Reaction of 3c with 4e,f



afforded **5y** in a 77% yield. The formation of **6y** (75%) was achieved via intramolecular annulation of **5y** using the above protocol. The structure of **5y** was determined by single-crystal X-ray crystallography.[14] However, when **3c** was allowed to react with **4f**, a complex mixture was formed and no **5z** was observed. To examine the applicability of this synthetic route, a formal synthesis of sertraline (Zoloft)[18] was developed (Scheme 8).

Scheme 8. Formal Synthesis of Sertraline



Sertraline is a potent selective serotonin reuptake inhibitor (SSRI), which has become a popular synthetic target. For the formal synthesis of sertraline, **6w** was chosen as the starting material. Oxidative cleavage of **6w**, which contains a bromovinyl group, provided the 1-tetralone skeleton via a one-pot reaction in the presence of OsO_4/NMO/$NaIO_4$.[19] Without further purification, removal of the β-sulfonyl group using freshly prepared 6% Na(Hg) (prepared from sodium and mercury in toluene at reflux) in MeOH at 25 °C[20] afforded the known intermediate **11**.[18a] The overall yield of the two-step process was 47%.

In summary, we have developed a novel synthesis of various substituted 1-aryltetralins **6** and 1-arylnaphthalenes **8** via (1) K_2CO_3-mediated α-styrylation of substituted β-ketosulfones **3** with bromostyryl bromides **4** and (2) stereocontrolled $NaBH_4$-promoted reduction of the resulting γ-alkenones **5**, followed by a BF_3·OEt_2-catalyzed intramolecular annulation of the corresponding γ-alkenols. A plausible mechanism has been proposed for these cyclization reactions. The structures of the key products **6** and **8** have been confirmed by X-ray crystallography. Further investigations regarding the asymmetric synthesis of chiral **6** will be studied via the desymmetrical annulation of **5**.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b00603.

X-ray analysis data of **5e** (CIF)
X-ray analysis data of **5y** (CIF)
X-ray analysis data of **6c** (CIF)
X-ray analysis data of **6f** (CIF)
X-ray analysis data of **6l** (CIF)
X-ray analysis data of **6u** (CIF)
X-ray analysis data of **8d** (CIF)
Detailed experimental procedures and spectroscopic data for all new compounds (PDF)

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Notes

The authors declare no competing financial interest.

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