

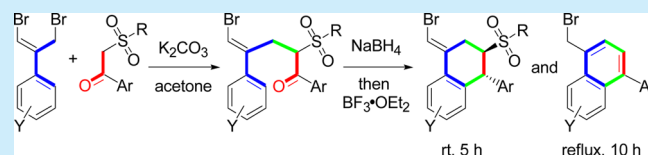
Synthesis of 1-Aryltetralins and 1-Arylnaphthalenes via (4 + 2) Annulation of β -Ketosulfones with Styryl Bromides

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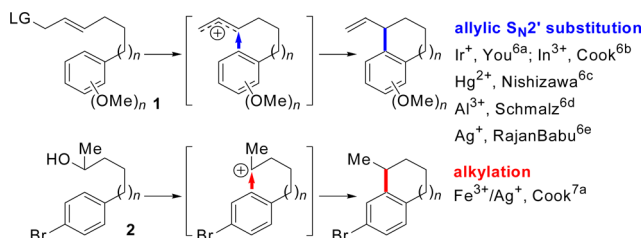
S Supporting Information

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.



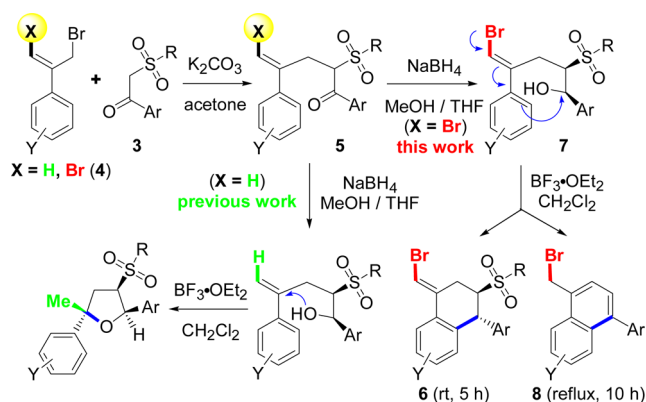
Tetralin is a highly privileged core structure found in many valuable biologically active molecules, pharmaceuticals, and natural products.^{1–4} With regard to the construction of this key building block, the 90-year-old Darzens tetralin synthesis (H_2SO_4 -mediated intramolecular ring-closure of a 1-phenyl-4-pentene)⁵ still stands out as one of the most direct and convenient methods. It uses arenes with an allylic chain⁶ **1** or an unactivated alcohol⁷ **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* $\rightarrow$ *endo* $\rightarrow$ *exo* direction), leading to the formation of a six-membered ring under metal-free catalyzed conditions. This stereospecific annulation reaction provides facile access to

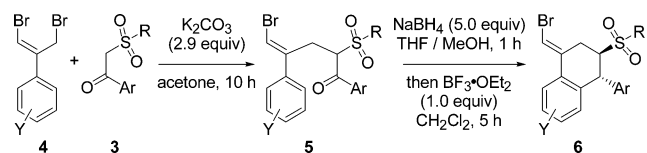
Scheme 2. Synthetic Route of 2,5-Diaryltetrahydrofurans, 1-Aryltetralins, and 1-Arylnaphthalenes



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}

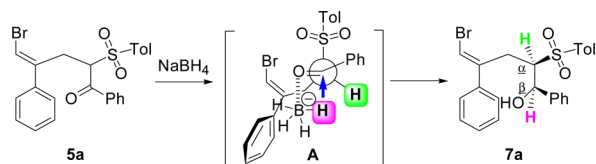
entry	3, Ar = , R =; 4, Y-Ar =	yield ^c (%)	
		5	6
1	3a, Ph, Me; 4a, Ph	5a, 80	6a, 82
2	3b, Ph, Ph; 4a, Ph	5b, 84	6b, 80
3	3c, Ph, Tol; 4a, Ph	5c, 89	6c, 76
4	3d, 4-FC ₆ H ₄ ; Tol; 4a, Ph	5d, 83	6d, 78
5	3e, 4-MeOC ₆ H ₄ ; Tol; 4a, Ph	5e, 86	6e, 74
6	3f, 4-MeC ₆ H ₄ ; Tol; 4a, Ph	5f, 83	6f, 70
7	3g, 4-CF ₃ C ₆ H ₄ ; Tol; 4a, Ph	5g, 80	6g, — ^d
8	3h, 4-NO ₂ C ₆ H ₄ ; Tol; 4a, Ph	5h, 80	6h, — ^d
9	3i, 3-NO ₂ C ₆ H ₄ ; Tol; 4a, Ph	5i, 82	6i, — ^d
10	3j, 4-PhC ₆ H ₄ ; Tol; 4a, Ph	5j, 84	6j, 74
11	3k, 2-naphthalene, Tol; 4a, Ph	5k, 88	6k, 70
12	3l, Ph, 4-FC ₆ H ₄ ; 4a, Ph	5l, 82	6l, 78
13	3m, Ph, 4-MeOC ₆ H ₄ ; 4a, Ph	5m, 80	6m, 81
14	3n, Ph, 3-MeC ₆ H ₄ ; 4a, Ph	5n, 84	6n, 82
15	3o, Ph, 4-EtC ₆ H ₄ ; 4a, Ph	5o, 82	6o, 78
16	3p, Ph, 4- <i>n</i> BuC ₆ H ₄ ; 4a, Ph	5p, 83	6p, 74
17	3b, Ph, Ph; 4b, 4-FC ₆ H ₄	5q, 84	6q, 80
18	3c, Ph, Tol; 4b, 4-FC ₆ H ₄	5r, 83	6r, 80
19	3b, Ph, Ph; 4c, 4-PhC ₆ H ₄	5s, 84	6s, 78
20	3c, Ph, Tol; 4c, 4-PhC ₆ H 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 ₂ C ₆ H ₃ ; Tol; 4a, Ph	5w, 78	6w, 81
24	3r, 2-thiophene, Tol; 4a, Ph	5x, 80	6x, 82

^aAlkylation reaction: **3** (1.0 mmol), K₂CO₃ (2.9 mmol), **4** (1.05 mmol), acetone (15 mL), reflux, 10 h. ^bAnnulation reaction: **5** (0.2 mmol), NaBH₄ (1.0 mmol), THF (5 mL)/MeOH (5 mL), 0 °C, 1 h, then BF₃·OEt₂ (0.2 mmol), CH₂Cl₂ (5 mL), 25 °C, 5 h. ^cIsolated yields. ^dNo 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

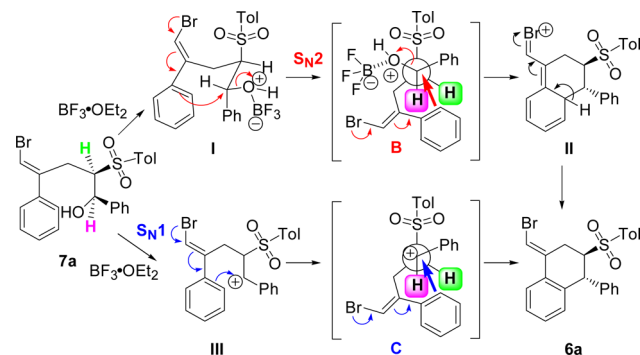
In accord with our previous experience, a single skeleton derived from the sulfonyl 4-alkenol was obtained. On the basis of the Felkin–Anh model **A**,¹⁵ the stereochemical centers of two protons (green H–C_α and pink H–C_β in **7a**) are well established. As shown in Scheme 3, a plausible mechanism

Scheme 3. Proposed Reduction Mechanism



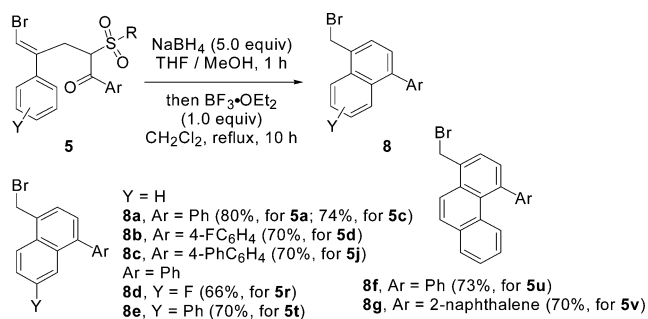
suggests that NaBH₄-mediated carbonyl reduction of **5a** is affected by the steric hindrance of the sulfonyl substituent such that the hydride can only attack the less hindered carbonyl face, yielding a single stereoisomer.^{12b} Subsequently, complexation of the resulting hydroxyl group by BF₃·OEt₂ 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.

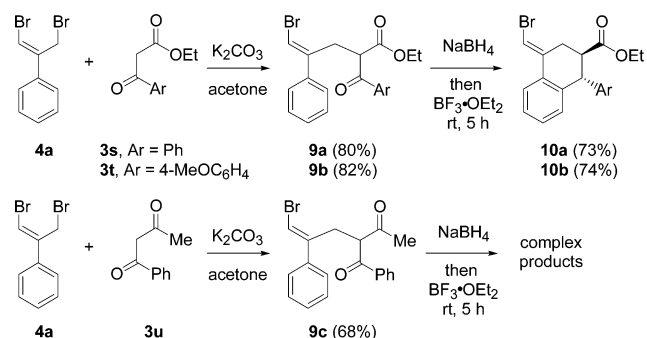
The BF₃·OEt₂-mediated intramolecular annulation of **5** was investigated at elevated temperatures and longer reaction times, as shown in Scheme 5. Specifically, changing the reaction

Scheme 5. Synthesis of **8**

conditions from 25 °C and 5 h to CH₂Cl₂ at reflux for 10 h generated substituted 1-bromomethyl naphthalenes **8a–e** (66–80%) and 1-bromomethyl phenanthrenes **8f,g** (73% and 70%) via BF₃·OEt₂-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

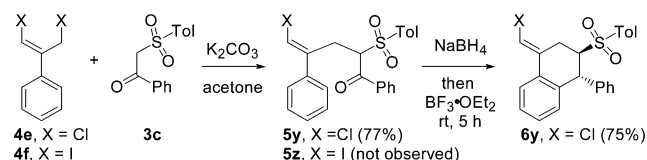
By changing the α-substituent from a sulfonyl group to an ethyl ester group (**3s**, Ar = Ph; **3t**, Ar = 4-MeOC₆H₄), **9a** and **9b** were isolated in 80% and 82% yields, respectively, as shown in Scheme 6. Using the above synthetic protocol, **10a,b** were generated in 73% and 74% yields. Comparing 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.

Scheme 6. Reaction of 4a with 3s–u



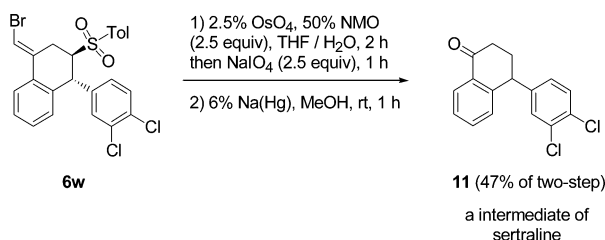
In further work, the bromo group was replaced with a chloro or iodo group, affording facile access to 1-aryltetralins. As shown in Scheme 7, α -styrylation of 3c with chlorostyryl chloride 4e

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 °C²⁰ 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 $\text{BF}_3\cdot\text{OEt}_2$ -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

S 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.

■ ACKNOWLEDGMENTS

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