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One-Pot Access to Sulfonylmethyl Arylpyrroles via the Domino Aerobic Wacker-Type Aminocyclization/1,4-Sulfonyl Migration

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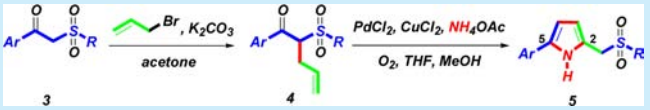
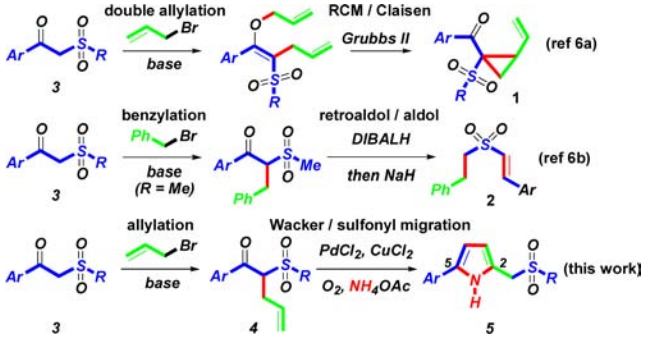
Supporting Information

ABSTRACT: $PdCl_2/CuCl_2/NH_4OAc$-mediated the domino aerobic Wacker-type aminocyclization of α-allyl-β-ketosulfones **4** in cosolvents THF and MeOH afforded 2-(sulfonylmethyl)-arylpyrroles **5** via 1,4-sulfonyl migration with moderate to good yields.



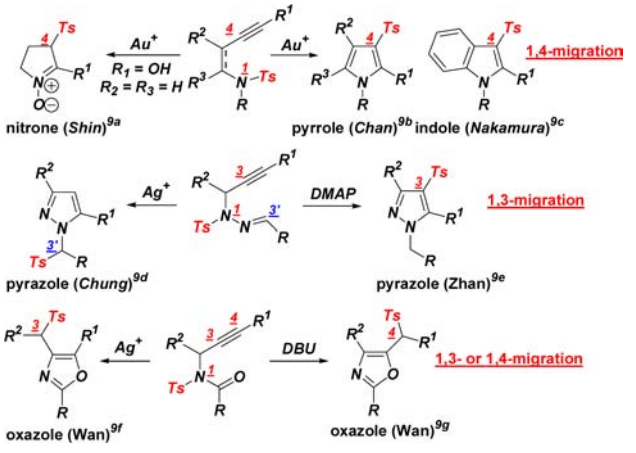
The skeleton of pyrrole is a key structural core in numerous biologically active molecules, pharmaceuticals, synthetic intermediates, and natural products.[1] Routes such as the Hantzch and Paal−-Knorr reactions have been developed to prepare pyrroles, but the drawbacks of these methods (multistep operations, regioselectivity, and harsh conditions) have encouraged researchers to explore more efficient synthetic protocols.[2] In several cases, the synthesis of 2-arylpyrroles is also of interest in different applications.[3,4] In particular, the skeleton of arylpyrrole is active against some cancer cells, such as murine P388 lymphocytic leukemia.[5] However, the development of an efficient protocol for sulfonylmethyl arylpyrroles remains a challenge in the synthetic field. In continuation of our investigation with the application of β-ketosulfones **3** (e.g., vinylcyclopropanes (VCP) **1** and arylethyl (*E*)-styrylsulfones **2**),[6a,b] the novel and facile $PdCl_2/CuCl_2/NH_4OAc$-mediated domino aerobic Wacker oxidative aminocyclization/1,4-sulfonyl migration is employed to construct the framework of (sulfonylmethyl)arylpyrroles **5** (Scheme 1).[6]

Scheme 1. Our Synthetic Applications of β-Ketosulfones 3



β-Ketosulfones (α-sulfonyl ketones) are important intermediates in organic synthesis, which can be easily prepared through substitution of α-haloketones with RSO_2Na, aerobic difunctionalization of sulfides, and direct oxysulfonylation of terminal alkynes or alkenes.[7,8] The sulfonyl group is a flexible substituent, and it can migrate to the appropriate position and facilitate the formation of a new C−S bond. However, wanting to examine recent synthetic examples of sulfonyl migration, we found that there are some reports on synthesizing the frameworks of nitrones,[9a] pyrroles,[9b] indoles,[9c] pyrazoles,[9d,e] oxazoles,[9f,g] and others[9h−k] via transition metal (Au^+, Ag^+) mediated or base (DBU, DMAP) promoted five-membered ring-closure and 1,3- or 1,4-sulfonyl migration (N−S bond cleavage/C−S bond formation) of *N*-sulfonylamino alkynes, as shown in Scheme 2.

Scheme 2. Routes to 1,3- or 1,4-Sulfonyl Migrations



After further comparison of literature reports on the sulfonyl migrations of N-sulfonylamino alkynes, we initiated our studies with the screening of conditions based on our previous experiences. For the aerobic Wacker reaction of skeleton **4** with a terminal olefin motif, substrate **4a** was first examined.[10] A number of review articles have highlighted fascinating developments based on an aerobic Wacker-type oxidative reaction.[11,12] As shown in Table 1, entry 1, we find that this $PdCl_2/CuCl_2$ system could catalyze the transformation from terminal alkene

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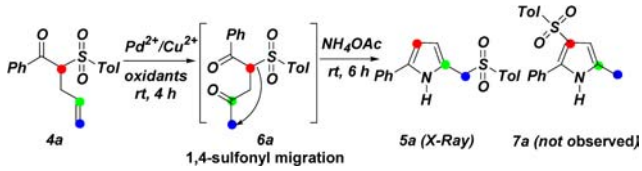
Table 1. Conditions for the Construction of 5a[a]



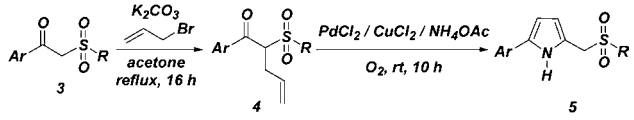
entry	catalyst (mol %), $CuCl_2$ (equiv), oxidants (equiv)	yield[b] (%)
1	$PdCl_2$ (10), $CuCl_2$ (1.5), O_2 (bubbled)	88
2	$PdCl_2$ (5), $CuCl_2$ (1.5), O_2 (bubbled)	80
3	$Pd(OAc)_2$ (10), $CuCl_2$ (1.5), O_2 (bubbled)	74
4	$Pd(MeCN)_2Cl_2$ (5), $CuCl_2$ (1.5), O_2 (bubbled)	68
5	$PdCl_2$ (10), $CuCl_2$ (1.5), *t*-BuO_2H (1.0)	28[c]
6	$PdCl_2$ (10), $CuCl_2$ (1.5), DDQ (1.0)	43[c]
7	$PdCl_2$ (10), $CuCl_2$ (1.5), $PhI(OAc)_2$ (1.0)	35[c]
8	$PdCl_2$ (5), $CuCl_2$ (1.5), air	66 (78)[d]
9	$PdCl_2$ (5), $CuCl_2$ (0), air	10[e]

[a]The reactions were run on a 1.0 mmol scale with **4a** at rt for 10 h. [b]Isolated yields. [c]The unknown products (for entry 5, 40%; entry 6, 24%; entry 7, 38%) were obtained. [d]The reaction was run at reflux and 15 h. [e]68% of **4a** was recovered.

4a to ketone **6a** with molecular oxygen (O_2) as the co-oxidant in the cosolvent of THF and MeOH (v/v = 1/1) at rt for 4 h, and the yield of **6a** provided an excellent 95% yield. When the reaction mixture was further involved with NH_4OAc at rt for 6 h, an 88% yield of pyrrole **5a** was isolated via 1,4-sulfonyl migration. Unexpectedly, we did not obtain the desired pyrrole **7a** regardless of which condition was used. The formation of **5a** was confirmed by X-ray crystallography.[13] After screening different conditions, we found that Pd^{2+}/Cu^{2+} system mediated Wacker-type oxidation of substrate **5a** with three similar catalysts ($PdCl_2$, $Pd(OAc)_2$, $Pd(MeCN)_2Cl_2$) and three different oxidants (*t*-BuO_2H, DDQ, $PhI(OAc)_2$) provided entries 1−4 in 68−88% yields of **5a**. Entries 5−7 provided lower yields of **5a** along with the unknown reaction mixture. By involvement of air, entry 8 showed that **5a** was produced in a 68% yield. By elevating the reaction temperature and time, the yield was increased to 78%. When no $CuCl_2$ was added to the reaction mixture as a co-oxidant, only 10% of **5a** was obtained, and the starting material **4a** was isolated in 68% yield. We envisioned that the optimized domino aerobic Wacker-type aminocyclization oxidation/1,4-sulfonylmigration process for synthesizing pyrrole **5a** should involve a combination of $PdCl_2$/$CuCl_2$/NH_4OAc.

With optimized conditions in hand (Table 1, entry 1), we further explored the substrate scope of the reaction, and the results are shown in Table 2. For the Ar and R groups of skeleton **3** (Ar = Ph, 4-FPh, 4-MeOPh, 4-MePh, 4-CF_3Ph, 4-PhPh, 2-naphthalene; R = 4-MePh, Ph, Me, 4-*i*-Pr, 4-*n*-Bu, 4-MeOPh, 4-FPh), the phenyl ring, with both electron-withdrawing and electron-donating substituents, was well tolerated, providing the desired products **4** and **5** in moderate to good yields. First, by controlling the use of 1.1 equiv of allyl bromide, *α*-allylated **4a**−**p** were obtained in good yields (entries 1−16). The reaction of **4** with the combination of $PdCl_2$/$CuCl_2$/NH_4OAc was further examined. All entries 6 showed that **5a**−**p** were isolated in a range of 70−86% yields when Ar and R were the alkyl or aryl groups. The involvement of different substituents did not affect the procedure and the isolated yield was maintained. No obvious yield changes were exhibited for the generation of **5a**−**p**. The structures of **5b** and **5e** were

Table 2. Synthesis of 5[a,b]

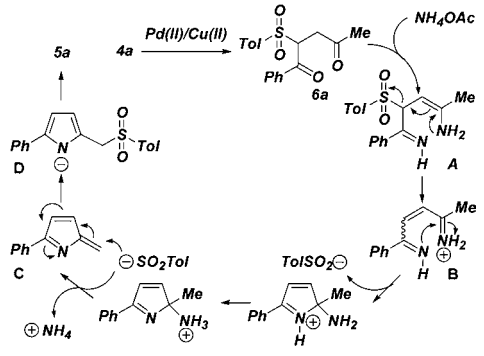


entry	**3**, Ar =, R =	**4**, yield[c] (%)	**5**, yield[c] (%)
1	**3a**, Ph, 4-MePh	**4a**, 86	**5a**, 86
2	**3b**, 4-FPh, 4-MePh	**4b**, 87	**5b**, 80
3	**3c**, 4-MeOPh, 4-MePh	**4c**, 90	**5c**, 82
4	**3d**, 4-MePh, 4-MePh	**4d**, 90	**5d**, 78
5	**3e**, 4-CF_3Ph, 4-MePh	**4e**, 85	**5e**, 70
6	**3f**, 4-PhPh, 4-MePh	**4f**, 86	**5f**, 82
7	**3g**, 2-naphthalene, 4-MePh	**4g**, 87	**5g**, 78
8	**3h**, 4-FPh, Ph	**4h**, 86	**5h**, 74
9	**3i**, 4-MeOPh, Ph	**4i**, 80	**5i**, 74
10	**3j**, 4-PhPh, Ph	**4j**, 86	**5j**, 72
11	**3k**, 4-MePh, Me	**4k**, 83	**5k**, 80
12	**3l**, 4-PhPh, Me	**4l**, 85	**5l**, 82
13	**3m**, Ph, 4-*i*-PrPh	**4m**, 89	**5m**, 73
14	**3n**, Ph, 4-*n*-BuPh	**4n**, 82	**5n**, 80
15	**3o**, Ph, 4-MeOPh	**4o**, 86	**5o**, 82
16	**3p** Ph, 4-FPh	**4p**, 93	**5p**, 76

[a]The *α*-allylation was run on a 1.0 mmol scale with **3**, K_2CO_3 (2.0 mmol), and allyl bromide (1.1 mmol), acetone (15 mL), reflux, 16 h. [b]The pyrrole synthesis was run on a 1.0 mmol scale with **4**, $PdCl_2$ (10 mol %), $CuCl_2$ (1.5 mmol), and NH_4OAc (2.1 mmol), THF (18 mL), MeOH (2 mL), bubbled O_2, rt, 10 h. [c]Isolated yield.

determined by single-crystal X-ray crystallography.[13] On the basis of the experimental results, a possible reaction mechanism is shown in Scheme 3. How is **5a** produced? Initially,

Scheme 3. Possible Mechanism



complexation of $PdCl_2$/$CuCl_2$ with the terminal olefin of substrate **4a** was found to yield **6a**. Intermediate **A** should be afforded via NH_4OAc-mediated intramolecular condensation. After desulfonation of **A** and then isomerization of **B**, removal of NH_3 and $TolSO_2Na$ affords **C**. Subsequently, $TolSO_2Na$ could be added into **C** to generate **5a** via the protonation of **D**.

According to the above conditions, the intermolecular control cross-coupling experiments could be supported to demonstrate the proposed mechanism. First, the reactions of **4b** and **4c** with the combination of $PdCl_2$/$CuCl_2$/NH_4OAc afforded two products, **5b** (73%) and **5c** (78%), with a 1:1 yield ratio, as shown in Scheme 4. In another way, cross-coupling of **4d** and **4k** or $MeSO_2Na$ also provided a nearly 1:1 yield ratio of **5d** (71%) and **5k** (73%) or **5d** (30%) and **5k** (36%) under the same conditions. We believe that the released sulfinic acid ammonium salt ($TolSO_2NH_4$ and $MeSO_2NH_4$) is the key

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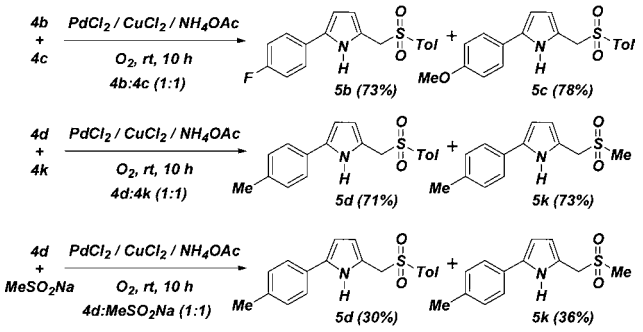
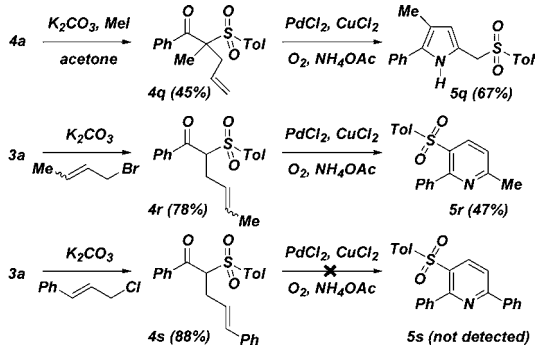
Scheme 4. Control Cross-Coupling Experiments



factor triggering the cross coupling of **C** and two sulfonyl groups. By mixing 1 equiv reactant of **4b**/**4c**, **4d**/**4k**, or **4d**/$MeSO_2Na$, we found that the provided yield distributions of the isolated products were consistent, which should be due to the generation of **C** with a fulvene skeleton in a one-pot process.

To examine the limitation of this route (see Scheme 5), α-methylation of **4a** was investigated. C-Methylation of **4a**

Scheme 5. Synthetic Applications



(K_2CO_3, MeI) provided **4q** in only 45% along with O-methylated enol ether *E*-/*Z*-isomers.[6a] By combination of $PdCl_2/CuCl_2/NH_4OAc$, **5q**, with trisubstituted groups, was isolated in a 67% yield. In particular, after the α-crotylation of **3a** with K_2CO_3 in boiling acetone (78%), one-pot pyrrole synthesis of **4r**, with the combination, provided **5r** with a pyridine structure in a 47% yield along with trace amounts of the pyrrole skeleton. When the substituent was changed from a methyl to phenyl group, α-cinnamylation of **3a** provided **4s** in an 88% yield. However, attempts to examine the one-pot protocol on **5s** failed due to the steric hindrance of the allylic phenyl substituent.

In summary, we have developed a $PdCl_2/CuCl_2/NH_4OAc$-mediated synthesis of (sulfonylmethyl)arylpyrroles **5**. A series of α-allyl-β-arylketosulfones **3** provided good yields. The pyrrole skeleton was developed on the basis of the domino aerobic Wacker-type aminocyclization of **4**. Further investigation regarding synthetic applications of β-arylketosulfones will be conducted and published in due course.

■ ASSOCIATED CONTENT

Supporting Information

Experimental data and scanned photocopies of ^1H and ^{13}C NMR spectral data. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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(13) CCDC 1029291 (**5a**), 1029290 (**5b**), and 1030255 (**5e**) contain the supplementary crystallographic data for this paper. This data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; fax: 44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk).