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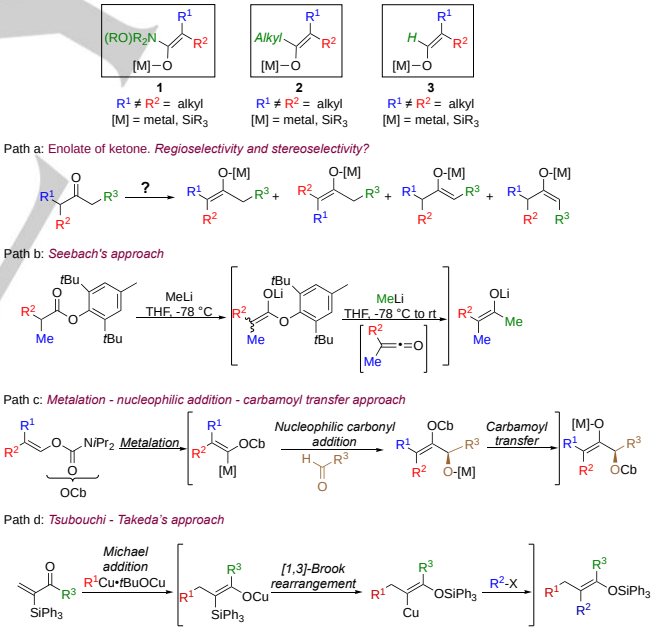
Regio- and Stereoselective Synthesis of Fully Substituted Silyl Enol Ethers of Ketones and Aldehydes in Acyclic Systems

Peter-Yong Wang,[a][b] Guillaume Duret,[a][b] and Ilan Marek[a]*

Dedicated to Professor Yitzhak Apeloig on the occasion of his 75th birthday

Abstract: *The regio- and stereoselective preparation of fully substituted and stereodefined silyl enol ethers of ketone and aldehyde are reported through an allyl-Brook rearrangement. This fast and efficient strategy proceeds from a mixture of E and Z-isomers of easily accessible starting materials.*

Enolate derivatives are important key intermediates in organic synthesis, widely used in the stereoselective formation of carbon-carbon bonds.[1] Most of the conventional methods for their preparation rely on the selective formation of kinetic enolates by deprotonation of α-substituted carbonyl compounds.[2] However, the generation of fully-substituted enolates **1-3**, or enolate equivalent, in acyclic systems is a more complex task and has been the focus of few independent studies.[3] Any solution allowing for the preparation of geometrically defined β,β-disubstituted enolates **1-3** would, by addition of the appropriate electrophile, not only empower the aldol reaction but also provide an efficient and straightforward access to the synthesis of a large family of products possessing quaternary carbon stereocenters.[4] From the reductive ring-opening of diastereoisomerically pure bicyclic lactams[5] to the stereospecific enolization of acyclic α-alkylbutyramides,[6] or conjugated addition[7] through the double stereodifferentiation deprotonation of enantiomerically enriched α-branched esters,[8] the formation of stereodefined fully substituted enolates of amides and esters **1** have been achieved with a great efficiency. In this context, we have reported the carbometalation reaction of ynamides[9] followed by a stereo-retentive oxidation reaction[10] providing the expected fully-substituted enolate of amides **1** as a single isomer where the stereochemistry of the enolate was predetermined during the regio- and stereoselective carbometalation step.[11] Although few additional studies were reported,[12] one could safely consider that the stereoselective formation of acyclic β,β-disubstituted enolates of amides and esters (**1**) doesn't represent anymore a major synthetic problem. On the other hand, and despite all these efforts, the stereoselective formation of fully-substituted enolates of *ketones* **2** is still challenging as one needs to control the regio- and stereoselectivity of the enolization process (Scheme 1, path a). Attractive solutions to this problem were reported through either the *in-situ* generation and trapping of ketene intermediates (Scheme 1, path b),[13] via a combined metalation – addition of a carbonyl and carbamoyl transfer (Scheme 1, path c),[14] or through a Michael-addition followed by a sp^2-type Brook rearrangement (Scheme 1, path d).[15] Although these methods provided the desired stereodefined enolates of ketones, they intrinsically present some limitations. For instance, the stereochemistry of the enolate resulting from the reaction of methyllithium to ketene depends on the steric bulk between the two substituents on the ketene (R^2 *versus* Me). The transformation proceeding through the carbamoyl transfer is restricted to the formation of β'-alkoxy-substituted enolate and finally, the Michael addition followed by the Brook rearrangement proceeds only for reactive electrophiles (R^2-X). Even more difficult is the stereodefined preparation of acyclic fully-substituted enolate of *aldehydes* (**3**, Scheme 1) which is still in its complete infancy.[16] As we have been involved in the last few years in the design of new strategies allowing the preparation of stereodefined β,β-disubstituted enolates as a new source of quaternary carbon stereocenters, we report herein a unified approach for the regio-and stereoselective preparation of fully substituted silyl enol ether of ketones and aldehydes.



Scheme 1. Various strategies for the synthesis of fully substituted enolates

Based on the pioneering work of Kuwajima,[17] we hypothesized that stereodefined fully substituted silyl enol ether of ketone **2** should be obtained through the addition of a catalytic amount of base to α-hydroxy alkenyl silane **4**. The resulting alcoholate **5** should promote an intramolecular allylmetal-Brook rearrangement[18] to lead to the chelated silyl ether **6**. The starting material **4** would then protonate **6** to give the expected stereodefined silyl enol ether **2** and regenerate **5** to further

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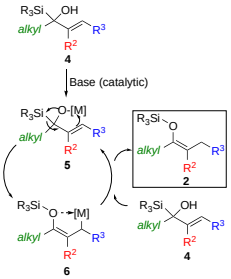
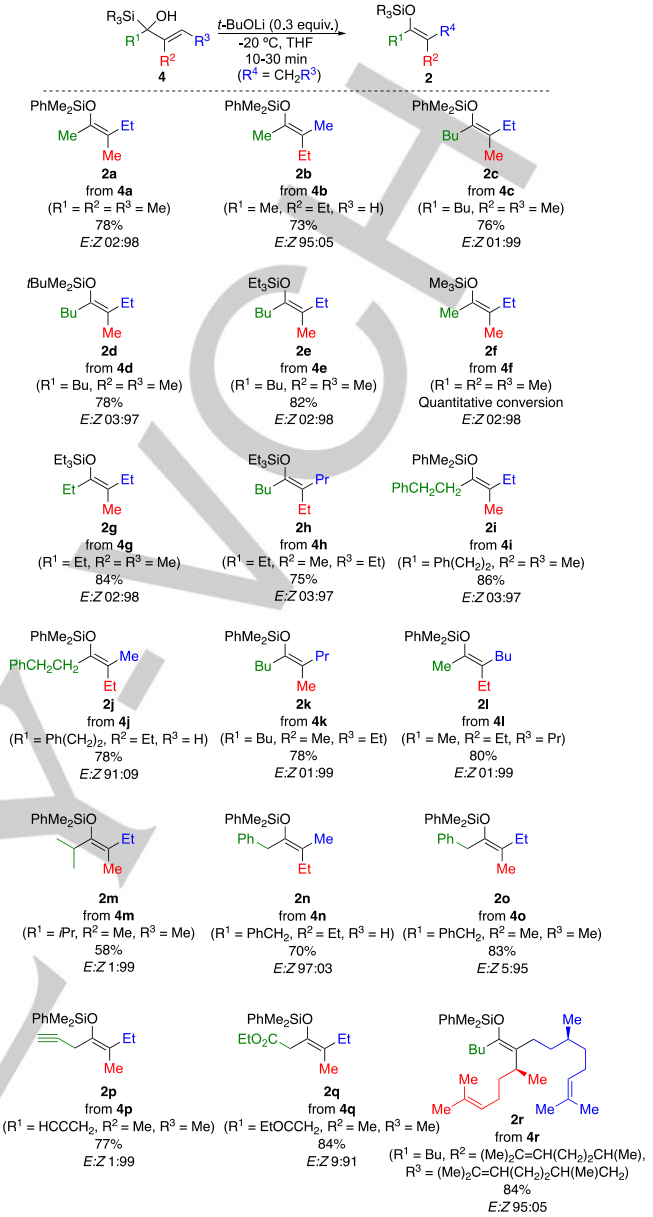
continue the catalytic cycle (Scheme 2). The final stereochemistry of **2** depending on the existence of the intramolecular chelation in **6**. Conceptually very simple, this flexible and convenient approach should provide an answer to the formation of polysubstituted silyl enol ether of ketones **2**.[19] It should be noted that the preparation of all starting α-hydroxy alkenyl silanes **4** is easy and straightforward (see Supporting Information).[20]



Scheme 2. Proposed catalytic cycle for the preparation of stereodefined fully substituted silyl enol ether of ketones

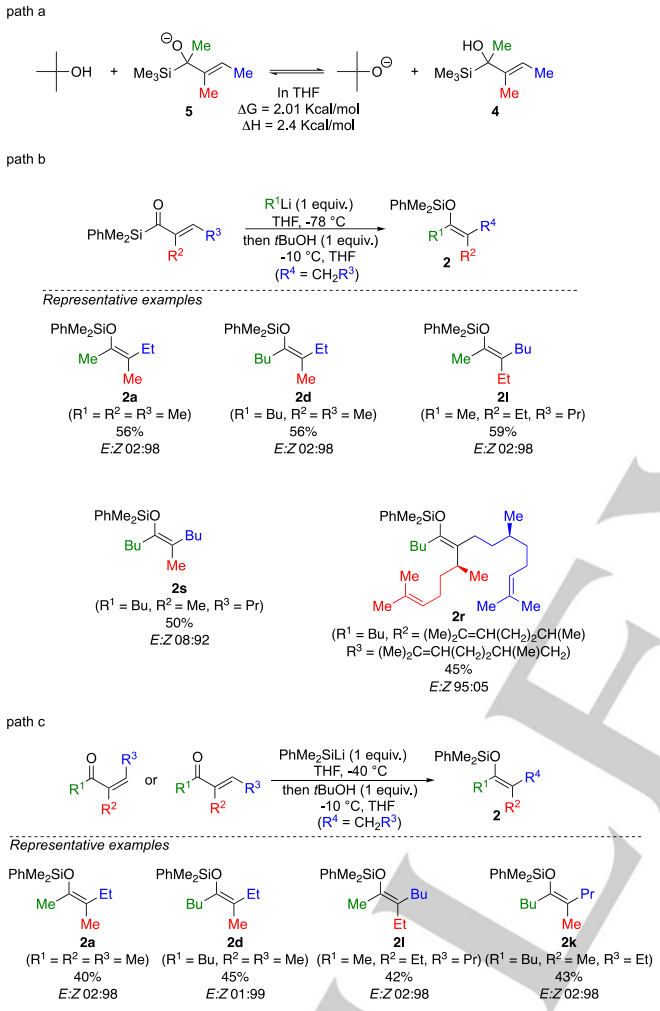
However, and to our surprise, the experimental conditions originally reported for the Brook rearrangement[17] didn't provide the expected products in decent yields, particularly that our ultimate goal was to have a one-pot procedure allowing access to all possible stereoisomers, and we had to reoptimize the conditions for this transformation on our model substrate **4a** ($R^1 = R^2 = R^3$ = Me, see Supporting Information for all details and optimization). Following this optimization, we found that when α-hydroxy alkenyl silane **4a** was treated with 0.3 equivalent of *t*-BuOLi in THF at -20 °C, the silyl enol ether **2a** was obtained in 74% yield in 10 to 30 min with an excellent 02:98 *E/Z* ratio (**2a**, Scheme 3). At the outset, a scale-up experiment established our confidence in the robustness of the current method as **2a** could be prepared on a 2g scale. The opposite stereoisomer **2b** could equally be prepared with similar selectivity by changing the nature of the two substituents R^2 and R^3 (R^1 = Me, R^2 = Et, R^3 = H). The nature of the silyl group doesn't influence the reaction as similar yields and *E:Z* ratios were found for dimethylphenylsilyl tert-butyldimethylsilyl, triethylsilyl, or trimethylsilyl groups respectively (**2c**-**2f**, Scheme 3). It should be noted that in the latter case (**2f**), the silyl enol ether decomposes upon purification by column chromatography. Either the *E* or the *Z*-isomers of structurally similar silyl enol ethers with different R^1 alkyl groups (**2i** and **2j**, **2n** and **2o**) could be easily prepared underlining the power of the proposed strategy. All *E:Z* ratios were determined by GC analysis and the stereochemistry of the two isomers **2i** and **2j** was unambiguously determined by NOESY experiments (see Supplementary Information).



Scheme 3. Scope for the formation of stereodefined fully substituted ketone silyl enol ether **2**

Interestingly, the alkyl group R^1 could be secondary (**2m**, Scheme 3), showing that this proposed approach allows the formation of a stereo- and regioselective enolate derivative as single isomer that would be inaccessible through classical deprotonation of β,β'-branched disubstituted ketone. Even more appealing is the formation of stereodefined fully substituted silyl enol ethers in the presence of acidic protons (**2n**-**2q**, Scheme 3,). In the latter case (**2q**), a lower catalytic amount of base needs to be employed (10 mol%) for a longer period of time (7 h) to reach good yields. As illustrative example, stereodefined fully substituted silyl enol ether **2r** possessing an allylic stereocenter was prepared without racemization. Finally, we were delighted that the desired silyl enol ethers could be directly prepared in a

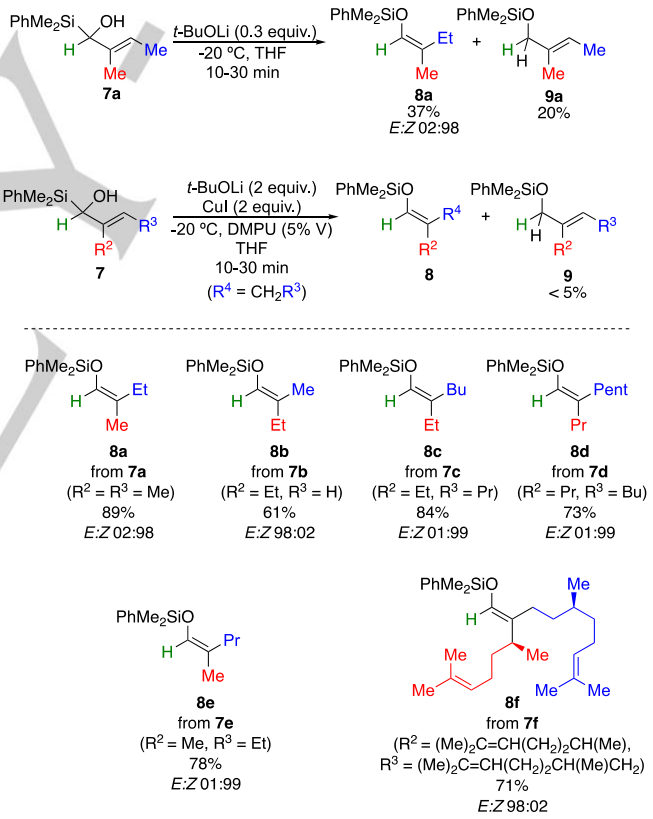
one-pot procedure from either simple acylsilanes or α,β-unsaturated ketones (Scheme 4). Based on calculated isodesmic reaction (Scheme 4, path a), **5** is slightly more basic that *t*BuOH and therefore should be able to undergo the Li-allyl Brook rearrangement before being *in-situ* hydrolyzed by *t*BuOH into the desired silyl enol ether **2**. Using this simple one-pot procedure several fully substituted silyl enol ethers **2** were obtained with very high *E*:*Z* isomeric ratios by simply adding an alkyllithium to the acylsilane followed by addition of *t*BuOH (Scheme 4, path b).



Scheme 4. One-pot synthesis of silyl enol ether **2a** from acylsilanes or α,β-unsaturated ketones.

Alternatively, fully substituted silyl enol ethers **2** could be similarly *in-situ* prepared with high isomeric ratios from α,β-unsaturated ketones by successive addition of $PhMe_2SiLi$[21] derivative and *t*BuOH (Scheme 4, path c). Although yields are lower due to the addition of $PhMe_2SiLi$ to enones, this transformation underline that the stereochemistry of the initial enone has no effect on the stereochemistry of the final silyl enol ether **2** (Scheme 2) and simple enones can be used as starting materials.

Having successfully demonstrated that we could prepare stereodefined polysubstituted silyl enol ethers of ketones in acyclic systems by a Li allyl-Brook rearrangement, we wondered if the same strategy could lead to the preparation of their aldehyde analogs. While being very interesting building blocks for subsequent transformations,[22] the lack of stability as well as the difficulties to control the stereoselectivity make them challenging to prepare.[16] Using our optimized conditions on **7a**, the expected stereodefined fully substituted silyl enol ether of aldehyde **8a** was obtained in only 37% yield, still with an outstanding 02:98 *E*:*Z* ratio, along the formation of **9a** in almost 20% and unidentified side products (Scheme 5). After extensive optimization of the experimental conditions (See supporting information for all details), we found that the best condition for the reaction to proceed was to treat **7a** with 2 equiv. of *t*BuOCu with DMPU (5% in volume) in THF at -20 °C. In this condition, **8a** was obtained in 89% as almost a single isomer with less than 5% of **9a**.

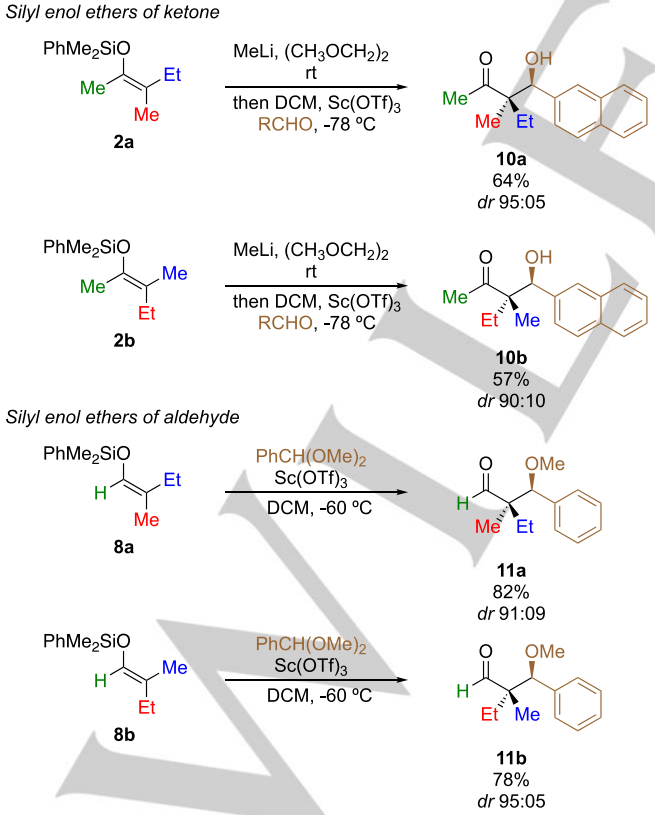


Scheme 5. Scope for the formation of stereodefined fully substituted silyl enol ether of aldehyde **8**

We applied this new optimized condition to several α-hydroxy alkenyl silanes **7a-f**, and we were delighted to obtain the corresponding silyl enol ethers of aldehydes **8a-f** in good yields and excellent *E*/*Z* selectivity (Scheme 5). Here again, both stereoisomers could be independently prepared by simply changing the nature of the R^1 and R^2 groups (see formation of

8a and **8b**, Scheme 5). We could perform the reaction on both isomers independently as well as on a 1:1 mixture of the starting materials (**4** and **7**). In all cases, the same products (**2** and **8** respectively) were obtained with similar stereochemistries and yields. This stereoconvergent reaction could easily be rationalized by an equilibration of the configurationally labile carbanion **6** (Scheme 2) into a chelated species, controlling the final stereochemistry of the Brook-products.

To illustrate the potential of these diversely polysubstituted silyl enol ethers in stereoselective synthesis, an aldol reaction has been performed on the two independent isomers **2a** and **2b**. When methyllithium in dimethoxyethane was added to **2a** at room temperature, the lithium enolate was formed without losing the initial stereochemistry.[23] Then, the subsequent addition of scandium triflate in DCM and naphthaldehyde at low temperature afforded the aldol **10a** in 70% yield with an excellent 95:05 diastereoselectivity (Scheme 6).[24] Alternatively, when the same transformation was performed on **2b**, the opposite diastereoisomer **10b** was formed with similar selectivities. In the case of silyl enol ether of aldehydes **8a** and **8b**, the direct Mukaiyama aldol transformation has been tested with benzaldehyde dimethyl acetal and provide **11a** in 82% yield with a diastereoisomeric ratio of 91:09 and **11b** in similar selectivity from **8a** and **8b** respectively.[25] These representative examples illustrate the power of the preparation of stereodefined silyl enol ether of ketones and aldehydes through the formation of quaternary carbon stereocenters in acyclic systems via the classical aldol-type chemistry.



Scheme 6. Representative examples of stereodefined silyl enol ethers of ketones and aldehydes in stereoselective synthesis

In conclusion, we have described a fast and efficient reaction sequence allowing the preparation of a wide range of fully substituted stereodefined silyl enol ethers of ketones and aldehydes with excellent *E:Z* ratios from easily accessible starting materials through a postulated allylmetal Brook rearrangement. Efforts to exploit this new approach in asymmetric catalysis for the creation of quaternary carbon stereocenters will be reported in due course.

Acknowledgements

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Keywords: hydroxy silane • silyl enol ether • Brook rearrangement • stereochemistry • migration

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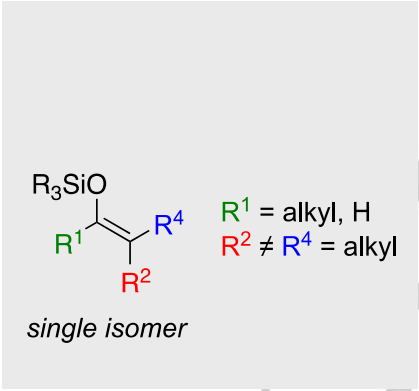

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