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Convergent Synthesis of Silylated Allylic Alcohols by a Stereoselective Domino, Sequential Radical-Coupling Reaction**

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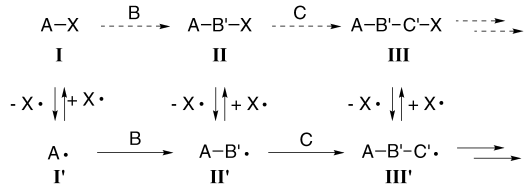
The construction of multiple carbon – carbon bonds by tandem reactions represents an efficient approach to the synthesis of complex molecular structures from simple organic building blocks.[1] Although radical-mediated intramolecular tandem cyclization exemplifies such an approach,[2] extensions to the intermolecular reaction (Scheme 1; **I'** – **III'**) have been severely limited, and require careful choice of



Scheme 1. A – X: Radical precursor; B, C: radical acceptors.

coupling partners.[3] This limitation could be attributed to the difficulty in the selective reaction of the transient radicals, for example, **I'**, **II'**, and **III'**, with certain coupling partners in radical chain reactions. The problem might be solved if we could prepare the radical intermediates **I'**, **II'**, and **III'** as their radical precursors **I**, **II**, and **III**, and subsequently couple them with radical acceptors in an atom- or group-transfer manner (Scheme 1).[4] The reaction would then be an iterative atom- or group-transfer radical reaction.[5] However, there have been no reports of such transformations, with the exception of living radical polymerization, in which the same alkenes react consecutively.[6]

We have reported the group-transfer coupling of trimethylsilyl phenyl telluride (**1**), carbonyl compounds, and isonitriles, which involves the selective carbon – carbon bond formation of the α-siloxy radical **2** with isonitriles.[7] It should be noted that the reaction of silyl tellurides with carbonyl compounds in the absence of isonitrile afforded the α-siloxy telluride **3**. This unique feature of the reaction prompted us to investigate alkynes as a third coupling partner (Scheme 2). Here we report a novel group-transfer coupling of **1**, carbonyl compounds, and alkynes to give the silylated allylic alcohol **5**. As the product also possesses a reactive carbon – tellurium bond, radical-mediated transformation via the vinyl radical **4**

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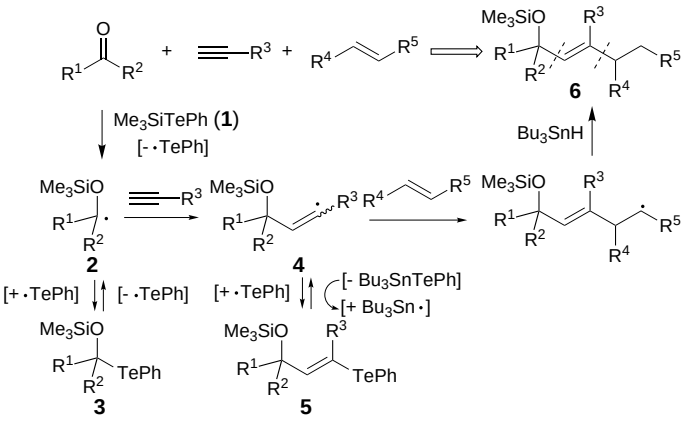


Scheme 2.

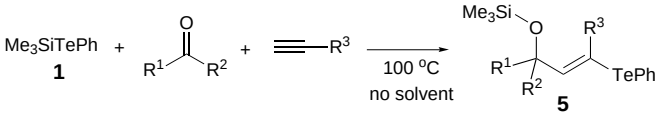
would effect further carbon–carbon bond formation, thus providing a convergent and stereoselective synthetic route to allylic alcohols with tri-substituted carbon–carbon double bonds.[8]

The sequential reaction to form **5** (method A) was initially examined by using **1**, benzophenone, and phenyl acetylene as substrates. Compound **1** and benzophenone were treated at room temperature in MeCN to give **3a** (R^1, R^2 = Ph), which was treated with phenyl acetylene at 100 °C to give **5a** (R^1, R^2, R^3 = Ph) in 85% yield.[9] Alternatively, the reaction was accomplished in one step by heating a mixture of **1** (1.2 equivalents), benzophenone (1.0 equivalents), and phenyl acetylene (1.2 equivalents) without solvent at 100 °C for 12 h (method B) to give **5a** in 93% yield after recrystallization. Owing to its simplicity, we routinely used method B. The reaction does not require the use of a solvent, although various solvents, such as EtCN, $(CH_2Cl)_2$, and supercritical CO_2, could be also used.

The reaction showed high *E* selectivity (96%), which was confirmed by X-ray structural analysis.[10] The X-ray analysis also confirmed the regioselectivity; the addition of the α-siloxy radical **2a**, which is generated from **1** and benzophenone, took place at the less hindered side of the alkyne. The subsequent group-transfer reaction of the resulting vinyl radical **4** gave rise to the formation of **5** (Scheme 2). As the stereochemistry is controlled kinetically (see below), the group-transfer takes place from the less hindered side of **4**, to avoid steric interaction with the bulky siloxyalkyl moiety.

The present reaction is applicable to a variety of carbonyl compounds and alkynes, and its scope and efficiency are summarized in Table 1. Since a variety of R^1, R^2, and R^3 groups can be used, the present reaction would be useful for the diversity-oriented synthesis of allylic alcohol derivatives. Aromatic ketones and aldehydes (entries 1–8 and 10) are especially good substrates. The reaction with electron-rich alkynes was completed within 4–12 h at 100 °C in most cases and gave the desired coupling product in good to excellent yields. Aliphatic ketones and aldehydes were less reactive and required longer reaction times, but the reaction eventually afforded the desired coupling products in good yields (entries 9 and 11). Aryl- and heteroaryl-substituted alkynes and conjugated enynes were excellent acceptors, and the

Table 1. Synthesis of silylated allylic alcohols **5**.[a]



Entry	Ketone (R^1, R^2)	Alkyne (R^3)	Time [h]	Yield [%]	(E:Z)[b]
1	Ph, Ph	Ph	12	93	96:4
2[c]	Ph, Ph	*p*-EtO_2C-C_6H_4	12	78	93:7
3[c]	Ph, Ph	*p*-Br-C_6H_4	12	97	93:7
4[c]	Ph, *p*-MeO-C_6H_4	Ph	12	77 (93)[d]	95:5
5[c]	Ph, *p*-Me_2N-C_6H_4	Ph	12	61	90:10
6[c]	Ph, Ph	3-pyridyl	12	82	80:20
7[c]	Ph, Ph	1-cyclohexenyl	12	64 (87)[d]	92:8
8	Ph, *i*Pr	Ph	36	51	93:7
9	-$(CH_2)_3$-	Ph	40	66	87:13
10	Ph, H	Ph	4	74 (82)[d]	74:26
11	*c*-C_6H_{11}, H	Ph	36	82	67:33
12[c,e]	Ph, Ph	CO_2Et	2	69	85:15

[a] A mixture of **1** (1.3 equiv), carbonyl compound (1.0 equiv) and alkyne (2.0 equiv) was heated at 100 °C in a sealed tube. Full experimental details and characterization data can be found in the Supporting Information. [b] The ratio was determined by ^{1}H NMR of the crude reaction mixture. [c] The reaction was carried out in propionitrile or CD_3CN (about 0.6 M concentration of starting carbonyl compound). [d] Yield based on the converted carbonyl compounds. [e] The reaction was carried out in a stepwise manner.

reaction gave the desired coupling products in good to excellent yields (entries 1–3, 6 and 7; where R^1 = R^2 = Ph). The reaction with electron-deficient alkynes such as acetylene carboxylic esters, however, did not give the desired coupling products because of the preferential reaction of **1** with the alkynes. Method A was effective in such cases, and the coupling of **3a** with ethyl propiolate at 100 °C for 2 h afforded the desired coupling product **5b** (R^1, R^2 = Ph, R^3 = CO_2Et) in good yield (entry 12).

The high *E* stereoselectivity of the olefinic moiety of the products is remarkable, when it is considered that these reactions took place at 100 °C. Because the isolated (*E*)-**5b** and (*Z*)-**5b** did not show any signs of stereochemical isomerization even under the harsher reaction conditions of 100–130 °C for 13 h, the stereochemistry is controlled kinetically, although we have already reported that structurally related vinyl tellurides undergo stereochemical isomerization under photo-irradiation.[8e] The level of selectivity is more than 9:1 in the reaction with most of the ketones used, while in the reaction with aldehydes and sterically less hindered ketones, such as cyclobutanone, the level is slightly lower. The observed decrease in selectivity is consistent with the reduced steric bulk of the siloxyalkyl moiety in **3** in the group-transfer step.

Because the coupling product **5** is a radical precursor of the vinyl radical **4**, we examined the radical generation from **5** and subsequent C–C coupling reactions next. However, while alkyl tellurides are superior precursors for carbon-centered radicals,[11] there are no reports on vinyl-radical generation from vinyl tellurides. We found that the tributyltin radical selectively cleaved the vinylic carbon–tellurium bond in **5** to generate the vinyl radical **4**, which could be used for further carbon–carbon bond formations (Scheme 3).[12] Thus, treatment of (*E*)-**5a** with tributyltin hydride[11a] in the presence of

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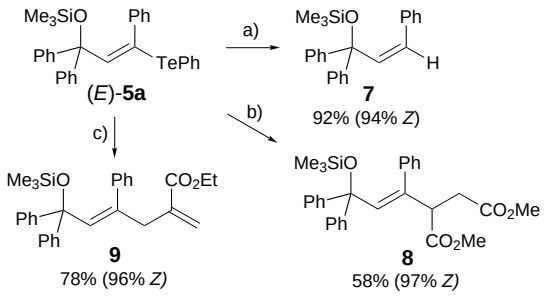

Scheme 3. a) Bu_3SnH (1.2 equiv), azobisisobutyronitrile (AIBN) (0.1 equiv), 80 °C, 1 h (no solvent); b) dimethyl fumarate (5 equiv), Bu_3SnH (2.4 equiv), AIBN (0.1 equiv), benzene, 80 °C, 1 h (syringe pump addition of Bu_3SnH solution over 1 h); c) ethyl 2-tributylstannylmethylacrylate (3 equiv), AIBN (0.1 equiv), 80 °C, 3 h (no solvent).

AIBN gave **7** in 92% yield. Because the stereochemical isomerization of vinyl radicals takes place very rapidly,[13] the observed high *Z* stereoselectivity can be also ascribed to the selective hydrogen atom abstraction from **4**, which is stereoselective because of the bulky siloxyalkyl group. The vinyl radical **4a** also reacted with electron-deficient alkenes.[11] Thus, treatment of (*E*)-**5a** with dimethyl fumarate in the presence of tributyltin hydride afforded the corresponding coupling product **8** (R^3, $R^4 = CO_2Me$; see Scheme 3) in good yield. The coupling of (*E*)-**5a** with ethyl 2-tributylstannylmethylacrylate also resulted in the desired coupling product **9** in good yield. In both cases, the olefinic stereochemistry of **5a** was highly preserved in the products.

In summary, we have developed a highly convergent and stereocontrolled synthesis of silylated allyl alcohols by domino, sequential radical-coupling reactions. Because the coupling reaction can be carried out with a variety of combinations of carbonyl compounds, alkynes, and agents to trap the vinyl radicals, this method provides a novel and powerful strategy for the diversity-oriented synthesis of allylic alcohols. Moreover, since the reaction does not require the use of a solvent, it also has the advantage of being environmentally friendly.[14]

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[9] Addition of 0.1 equivalents of 4-oxo-TEMPO (4-oxo-2,2,6,6-tetramethyl-1-piperidinoxyl; radical chain inhibitor) had a marginal effect on the formation of **5a**, which indicates that any contribution of the chain pathway is negligible.

[10] CCDC 176516 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

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