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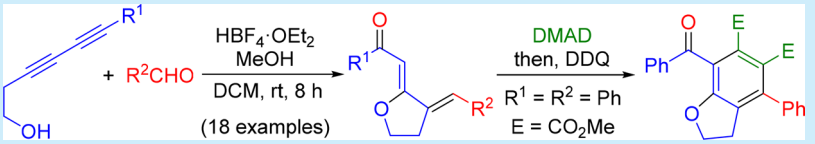
Domino Synthesis of 2,3-Dialkylidenetetrahydrofurans via Tandem Prins Cyclization−Skeletal Reorganization

Mizuki Kato and Akio Saito*

Division of Applied Chemistry, Institute of Engineering, Tokyo University of Agriculture and Technology, 2-24-16 Naka-cho, Koganei, Tokyo 184-8588, Japan

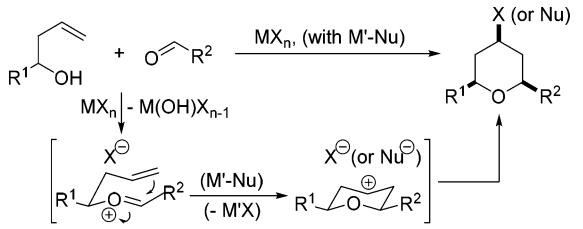
Supporting Information



ABSTRACT: A domino synthesis of 2,3-dialkylidenetetrahydrofurans based on Prins-type cyclization of 3,5-diynols and aldehydes is described. In the present reaction, skeletal reorganization of the Prins-cyclized intermediates proceeds via a ring-opening reaction followed by intramolecular (hemi)acetalization of the resulting 4-en-1-yn-3-ones. Furthermore, a representative product undergoes a Diels−Alder reaction with dimethyl acetylenedicarboxylate (DMAD) to afford a highly substituted 2,3-dihydrobenzofuran.

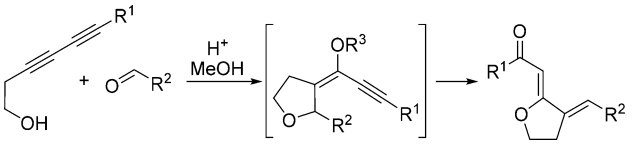
The cyclization of an oxocarbenium intermediate generated from a homoallylic alcohol and an aldehyde in the presence of an acid (MX_n) is known as the Prins cyclization (Scheme 1), which is widely utilized in natural product synthesis as a powerful method for constructing oxygen-containing heterocycles.[1] The cyclization generally proceeds via a six-membered chairlike transition state to produce tetrahydropyrans with high stereoselectivities.[2] Because the acid can act not only as a promoter but also as a source of the nucleophilic anion depending on the reaction conditions, various functional groups such as halides,[3] oxygen-centered,[4] sulfur-centered,[5] nitrogen-centered,[6] and carbon-centered nucleophiles[7] can be introduced into position 4 of the products by modulating the nature of the acid and the addition of a nucleophile. Furthermore, the Prins cyclization has been extended to its variants of other unsaturated alcohols with functionalities such as silylated alkenes,[1e,8] alkynes,[9] allenes,[10] and conjugated dienes,[11] although these have been less studied. Also, tetrahydrofurans can be obtained by the varying substitution styles of the unsaturated alcohols.[2a,3b,8a,9a−c,10e,11b] However, to the best of our knowledge, Prins-type cyclization of conjugated diynyl alcohols and its application to domino reactions[1d,12] have not been reported.

Scheme 1. Prins Cyclization



As a part of our continuing studies on efficient syntheses of cyclic compounds, we have developed domino reactions involving the acid-catalyzed metathesis between alkynes and carbonyls or imines.[13] In the course of investigating the metathesis of conjugated diynes and aldehydes, it was found that 2,3-dialkylidenetetrahydrofurans were formed in the reaction between 3,5-diynols and aldehydes (Scheme 2).

Scheme 2. This Work



Although a flash vacuum pyrolysis of 3-acyloxymethyl-2-methylfuran,[14a] an olefination of 2-alkylidenebutyrolactone,[14b] and a cyclocondensation of 2-cinnamoyl ketene dithioacetals with oxalyl chloride[14c] have been known as preparations of 2,3-dialkylidenetetrahydrofurans that have a further functionalizable diene moiety and are expected to be converted into various biologically interesting polycyclic furans,[15] these methods have limitations in reactants.[14] Herein, we report the domino synthesis of 2,3-dialkylidenetetrahydrofurans via skeletal reorganization of the Prins-cyclized intermediates derived from 3,5-diynols and aldehydes.

In the initial study, acids and additives were evaluated for the domino reaction of 3,5-diynol **1a** and benzaldehyde (**2a**, 2 equiv) in dichloromethane (DCM, Table 1). The use of trimethylsilyl trifluoromethanesulfonate (TMSOTf), HOTf, and $HBF_4·OEt_2$ as the acids (1 equiv) afforded the desired

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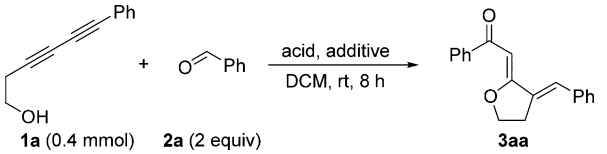
Table 1. Evaluation of Acids and Additives



1a (0.4 mmol) **2a** (2 equiv) **3aa**

entry	acid (equiv)	additive	3aa[a] (%)	1a[a] (%)
1	TMSOTf (1)		17	6
2	HOTf (1)		21	6
3	$HBF_4{\cdot}OEt_2$ (1)		25	27
4	$HBF_4{\cdot}OEt_2$ (1)	MeOH (1)	37	46
5	$HBF_4{\cdot}OEt_2$ (1)	MeOH (2)	21	79
6	$HBF_4{\cdot}OEt_2$ (1)	MeOH (5)	–	100
7	$HBF_4{\cdot}OEt_2$ (3)	MeOH (3)	80[b]	–
8	$HBF_4{\cdot}OEt_2$ (3)	^{i}PrOH (3)	57[b]	–
9	$HBF_4{\cdot}OEt_2$ (3)	^{t}BuOH (3)	trace	trace
10	$HBF_4{\cdot}OEt_2$ (3)	H_2O (3)	25	3

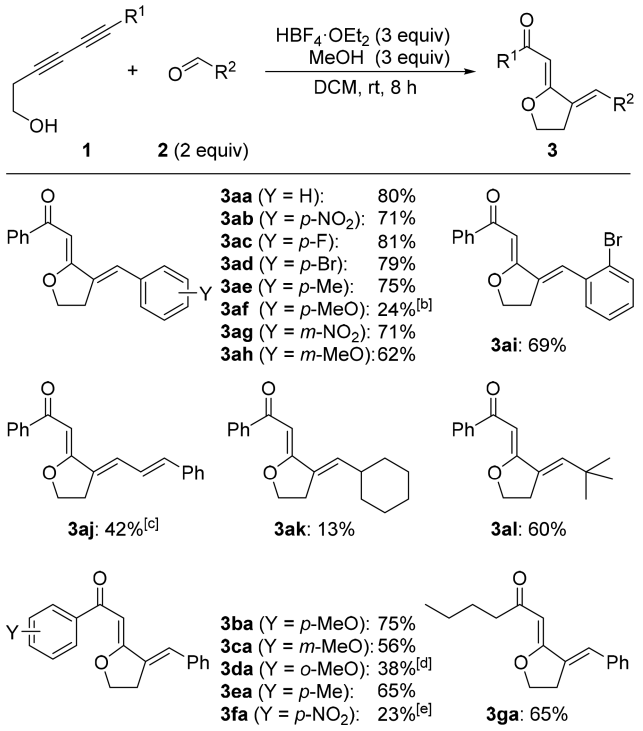
[a]Determined by ^{1}H NMR. [b]Isolated yield.

furans **3aa** in 17−25% yields at room temperature (entries 1−3, see also the Supporting Information for details of other acids). Furthermore, the addition of methanol (1 equiv) with $HBF_4{\cdot}OEt_2$ (1 equiv) showed a better result, in which the yield of **3aa** was improved up to 37% (entry 4). Unfortunately, the reaction with an increased amount of methanol (2 or 5 equiv) was sluggish (entry 5 or 6). In contrast, increasing the amounts of both methanol and HBF_4 by 1 equiv led to good conversions to **3aa**, and finally, the use of 3 equiv each afforded **3aa** in high yield (80%, entry 7). Furthermore, the present reaction could be successfully scaled up (**1a**, 5 mmol; **3aa**, 81%; see entry 25 in Table S-1). Notably, the addition of other alcohols or H_2O instead of methanol led to reduced yields of **3aa** (entries 8−10).

With the optimal conditions in hand, we next investigated the scope of 3,5-diynols **1** and aldehydes **2** in the domino synthesis of tetrahydrofurans **3** (Scheme 3). Similar to benzaldehyde (**2a**), the *para*-substituted **2b−e** and the *meta*-substituted **2g,h** reacted smoothly with 3,5-diynol **1a** in the presence of HBF_4 and methanol to give the corresponding furans **3ab−ae**, **3ag**, and **3ah** in 62−81% yields. With *p*-anisaldehyde (**2f**) as the substrate, lower conversion to **3af** (24% yield, 17% recovery of **1a**) was observed, probably because of its diminished electrophilicity as a result of the *p*-methoxy substituent. On the other hand, sterically hindered *o*-bromobenzaldehyde (**2i**) and pivalaldehyde (**2l**) afforded the desired **3ai** and **3al** in good yields (69% and 60%), respectively, although lower yields of **3aj** (42%) or **3ak** (13%) were attained from the reactions with cinnamaldehyde (**2j**) or cyclohexanecarbaldehyde (**2k**). It should be mentioned that 4-en-1-yn-3-ones **4aj** and **4ak** were formed in relatively good yields (56 and 61%) in the reaction of **1a** with **2j** and **2k**, respectively, at a lower temperature (−40 °C) in the absence of methanol (Scheme 4).

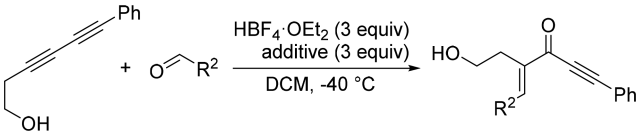
To our delight, the present method could be applied to the domino reactions of other 3,5-diynols **1b−g** and aldehyde **2a** (Scheme 3). In particular, *p*- and *m*-anisyl derivatives **3ba** and **3ca**, *p*-tolyl derivative **3ea**, and butyl derivative **3ga** were obtained in moderate to good yields (56−75%) at room temperature. On the other hand, the formation of *p*-nitrophenyl derivative **3fa** proceeded slowly (23% yield) because the nitro group reduced the electron density of the

Scheme 3. Scope of the Domino Synthesis of 3[a]



[a]Yields of isolated products are given. [b]Recovery of **1a**: 17%. [c]Reaction time: 18 h. [d]**4da** was obtained as a byproduct in 25% yield. [e]Reaction time, 24 h; recovery of **1f**, 62%.

Scheme 4. Formation of 4-En-2-ynones 4



diyne, and consequently, 62% of the substrate **1f** was recovered. In the case of *o*-anisyl derivative **3da** (38% yield), 4-en-1-yn-3-one **4da** was detected as a byproduct (25% yield). Considering the reaction of **1a** with **2j** or **2k** at −40 °C (Scheme 4), it is evident from these results that the 4-en-1-yn-3-one **4** was an intermediate in the domino synthesis of 2,3-dialkylidenetetrahydrofurans **3**.

To gain insight into the involvement of **4** as an intermediate, the formation of **4aa** from 3,5-diynol **1a** with aldehyde **2a** and the formation of **3aa** from **4aa** were examined as control experiments (Scheme 5, see also the Supporting Information for other control experiment). As expected, in the presence of $HBF_4{\cdot}OEt_2$, the reaction of **1a** with aldehyde **2a** at −40 °C afforded the corresponding 4-en-1-yn-3-one **4aa** in 72% yield. Although the addition of methanol and isopropanol led to the slow formation of **4aa** (Scheme 5a), likely because of the reduced acidity of the promoter by these alcohols and the competition between **1a** and these alcohols for addition to **2a**, the 4-en-1-yn-3-ones **4aa** were obtained in 70% and 65% yields, respectively (Scheme 5a). Furthermore, treatment of the isolated **4aa** with $HBF_4{\cdot}OEt_2$ in the presence of methanol at room temperature gave the desired furans **3aa** in 75% yield.

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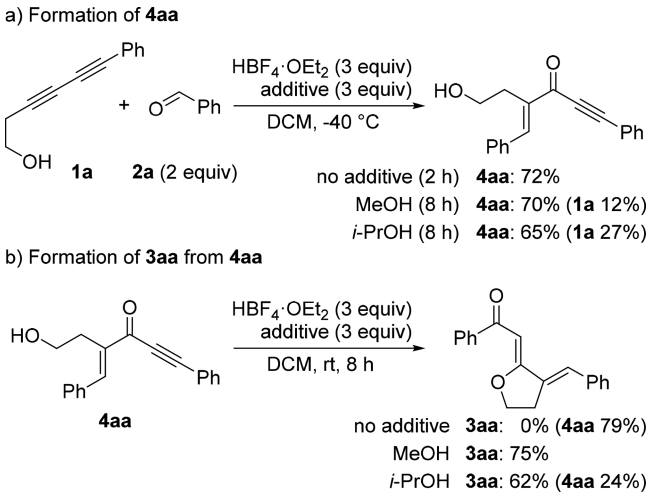
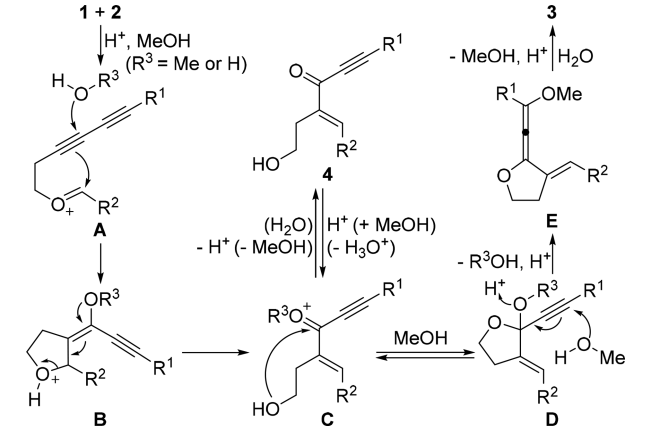
Scheme 5. Control Experiments



These results support the involvement of **4** as the intermediate. The use of isopropanol instead of methanol hindered the conversion of **4aa** to **3aa**, which was not produced at all in the absence of alcohols (Scheme 5b). Therefore, it was concluded that alcohol additives are essential for the cyclization of **4aa** to **3aa**.

On the basis of these results and previous reports of the Prins cyclization,[1,4a] a proposed mechanism for the domino synthesis of 2,3-dialkylidenetetrahydrofurans **3** is shown in Scheme 6. In this mechanism, 5-*endo* cyclization of the

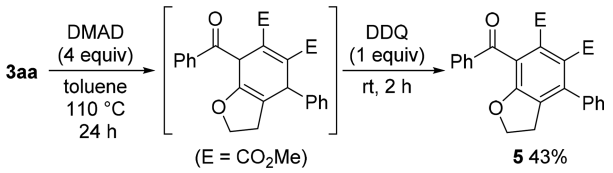
Scheme 6. Proposed Mechanism



oxocarbenium intermediates **A** derived from 3,5-diynols **1** and aldehydes **2** followed by addition of methanol (R^3 = Me) or the generated H_2O (R^3 = H) to carbon of inner alkyne give the Prins-cyclized intermediates **B**. The intermediates **B** are then converted into oxocarbenium intermediates **C** via the ring-opening of furan rings by acid. Subsequently, intramolecular (hemi)acetalization of intermediates **C** in the 5-*exo* mode leads to ring-closing intermediates **D**, which then undergo acid-mediated propargylic substitution reaction with methanol to produce allenyl ethers **E**.[16] Finally, the intermediates **E** are hydrolyzed to give furans **3**. Because the intramolecular acetalization and/or the propargylic substitution reaction did not proceed at −40 °C, intermediates **C** and/or **D** were hydrolyzed to yield 4-en-1-yn-3-ones **4**. Accordingly, methanol would play the role of nucleophiles in both Prins cyclization[4a] and propargylic substitution reactions.[16] Therefore, less sterically hindered methanol showed results superior to isopropanol in both conversions, namely, of **4aa** from **1a** with **2a** and of **3aa** from **4aa** (Scheme 4).

As an application of the present domino reaction, synthesis of 2,3-dihydrobenzofurans via the Diels−Alder reaction was also investigated (Scheme 7). The Diels−Alder reaction of

Scheme 7. Synthesis of 5 via Diels−Alder Reaction



diene **3aa** with dimethyl acetylenedicarboxylate (DMAD, 4 equiv) proceeded at 110 °C to give 4,5,6,7-tetrasubstituted 2,3-dihydrobenzofuran **5** in 43% yield after oxidative treatment using 2,3-dichloro-5,6-dicyano-*p*-benzoquinone (DDQ). Because the synthetic methods of 2,3-dihydrobenzofurans have been primarily focused on the construction of tetrahydrofuran rings,[17] this finding provides a novel procedure for the construction of aromatic rings.[18]

In conclusion, we developed a synthetic method for 2,3-dialkylidenetetrahydrofurans from 3,5-diynols and aldehydes under mild conditions. On the basis of control experiments, it was proposed that the domino synthesis of these heterocycles likely proceeded via Prins cyclization of 3,5-diynols and aldehydes followed by a skeletal reorganization that involved ring-opening of the Prins-cyclized intermediates and intramolecular (hemi)acetalization of the 4-en-1-yn-3-one intermediates. Furthermore, as one of the few construction methods of aromatic rings in the synthesis of 2,3-dihydrobenzofurans, the Diels−Alder reaction of the present product was demonstrated. Studies on the formation of other regioisomers in the Prins-type cyclization of 3,5-diynols are underway.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.8b02114.

Experimental procedures and compound characterization data (PDF)

■ AUTHOR INFORMATION

Corresponding Author

*E-mail: akio-sai@cc.tuat.ac.jp.

ORCID

Akio Saito: 0000-0002-8291-2059

Notes

The authors declare no competing financial interest.

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