CHEMISTRY
A EUROPEAN JOURNAL

ChemPubSoc
Europe

DOI: 10.1002/chem.201300205

Direct Syntheses of Spiro- and Fused-Hydrofurans by a Tunable Tandem Semipinacol Rearrangement/Oxa-Michael Addition Protocol

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A significant number of biologically important natural products and potential medicinal molecules contain cycloketone spiro- and fused-dihydrofuran units,[1,2] with examples including sieboldine A[1f] and the trichodermaketones A and C[2d] (Figure 1). Over the past few years, several different methods have been developed for the construction of these units,[3,4] including a variety of asymmetric syntheses.[5,6] Unfortunately, however, these methods generally require the preparation of complex substrates and subsequent multistep derivations of the resulting products. With this in mind, the development of a divergent and concise synthesis for the construction of these structural units is still highly desirable.

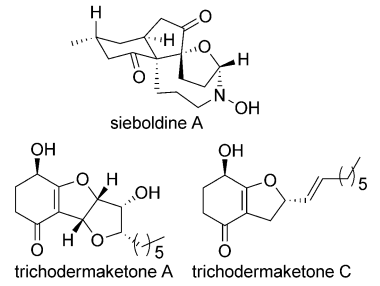
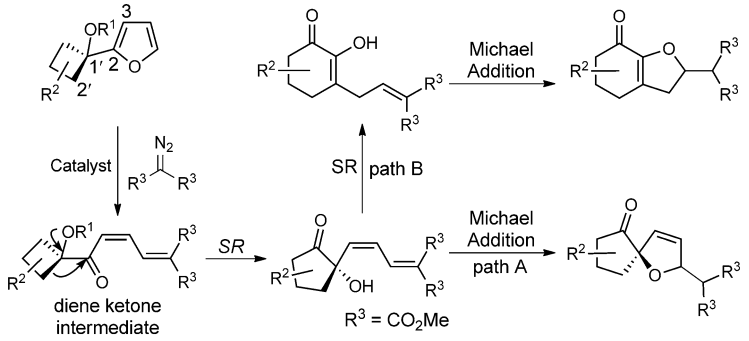


Figure 1. Representative natural products containing the spiro- or fused-hydrofuran units.

The semipinacol rearrangement is a well-known chemical reaction[7] and represents a class of versatile transformations for the construction of complex structural motifs, especially those with crowded quaternary centers.[8] As a result, this reaction has been applied to the efficient syntheses of many natural products and bioactive compounds.[9] Over the past decade, our own group[7c] and several other chemists[7a,b,d] have directed a great deal of effort towards the development of semipinacol rearrangement based synthetic methodologies for the formation of structurally diverse quaternary centers. In connection with our ongoing investigations regarding this rearrangement process and interests in constructing cycloketone spiro- and fused-dihydrofuran units, we hypothesized that these units might be derived from a readily available diene–ketone intermediate[10,11] through a tandem semipinacol rearrangement/oxa-Michael addition protocol under appropriate conditions (Scheme 1). This tandem process would be unprecedented and efficient for the synthesis of the aforementioned structural motifs. Herein, we present our preliminary results for this tandem protocol.



Scheme 1. A tunable tandem reaction towards the syntheses of dihydrofuran derivatives. SR = semipinacol rearrangement.

Our preliminary evaluation of the strategy was carried out by using phenylfuranyl cyclobutanol **1a** (R^1 = trimethylsilyl, TMS) and dimethyl diazomalonate as the model substrates (Table 1). Initially, a variety of different metal catalysts were investigated, including copper and silver salts. Unfortunately, these attempts failed to afford any of the expected intermediate in dichloromethane. Pleasingly, when $[Rh_2(Oct)_4]$ (1 mol%) was used as the catalyst, substrate **1a** reacted with dimethyl diazomalonate to form the intermediate **1e** in 92% yield.[10]

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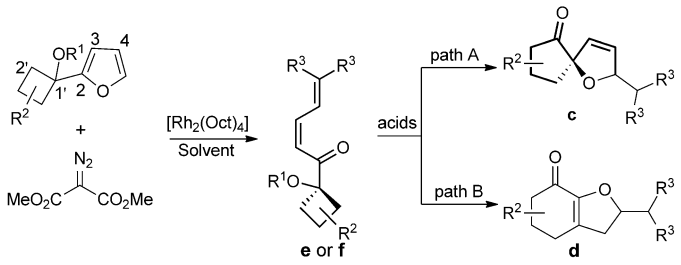
Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/chem.201300205.

Table 1. Optimization of reaction conditions.[a]



Entry	Solvent	Acid	R	t [h][b]	Yield [%] of **1c**/**1c′**[c]	Yield [%] of **1d**/**1d′**[d]
1	DCM	$Et_2O{\cdot}BF_3$ (1.1 equiv)	TMS	4	–	–
2	DCM	$THF{\cdot}BF_3$ (1.1 equiv)	TMS	24	61	–
3	DCM	$THF{\cdot}BF_3$ (3.0 equiv)	TMS	8	78	–
4	DCM	$THF{\cdot}BF_3$ (3.0 equiv)	TBS	8	59	–
5	DCM	$Et_2O{\cdot}BF_3$ (3.0 equiv)	TBS	4	–	71
6	DCM	$2\,AcOH{\cdot}BF_3$ (3.0 equiv)	TBS	4	–	66
7	DCM	TBSOTf (3.0 equiv)	TBS	8	–	53
8	DCM	TFA (3.0 equiv)	TBS	8	–	–
9	1,2-DCE	$Et_2O{\cdot}BF_3$ (3.0 equiv)	TBS	8	–	45
10	CCl_4	$Et_2O{\cdot}BF_3$ (3.0 equiv)	TBS	4	–	60
11	C_6H_5F	$Et_2O{\cdot}BF_3$ (3.0 equiv)	TBS	6	–	18
12	mesitylene	$Et_2O{\cdot}BF_3$ (3.0 equiv)	TBS	6	–	39
13	toluene	$Et_2O{\cdot}BF_3$ (3.0 equiv)	TBS	6	–	55

[a] All reactions were carried out with 0.1 mmol of the substrate in 1.0 mL of solvent under $[Rh_2(Oct)_4]$ catalysis (1.0 mol%). [b] The time required for the transformation of intermediate to products **c** or **d**. [c] Isolated total yield of the spiro products **c**/**c′** through path A. [d] Isolated total yield of the fused products **d**/**d′** through path B. 1,2-DCE = 1,2-dichloroethane; DCM = dichloromethane.

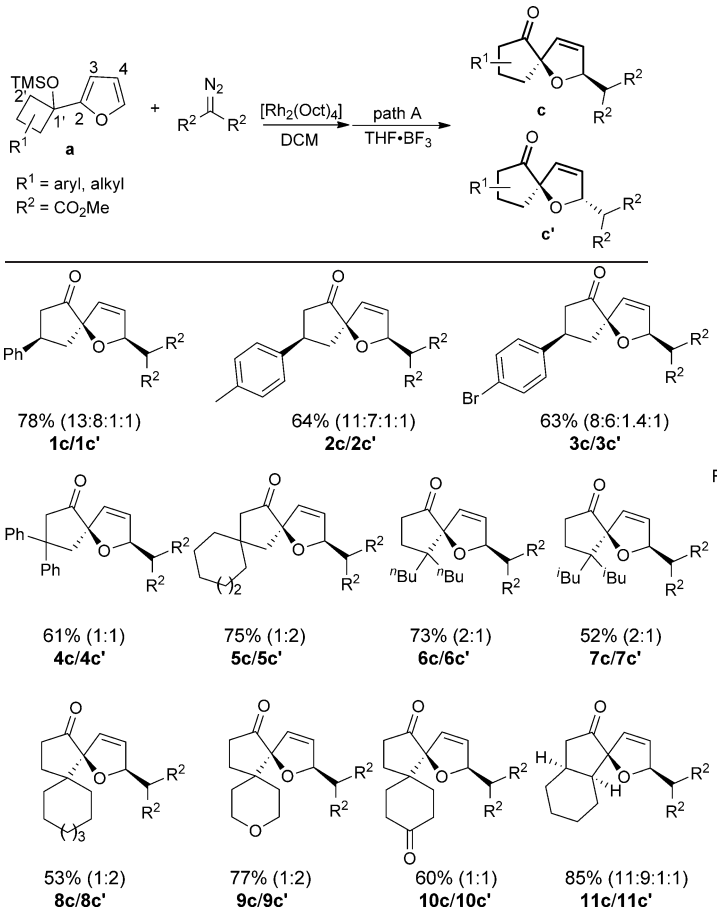
With the intermediate **1e** in hand, we proceeded to optimize the reaction conditions for the conversion of **1e** to either **1c** or **1d** in a one-pot manner. As shown in Table 1, when $Et_2O{\cdot}BF_3$ (1.1 equiv) was used as the Lewis acid to promote the tandem reaction, none of the expected products, **1c** or **1d**, was formed; however, decomposition of the substrate was observed (Table 1, entry 1). Fortunately, when $THF{\cdot}BF_3$ (1.1 equiv) was used as a replacement for $Et_2O{\cdot}BF_3$, the intermediate successfully afforded the spiro-ring products **1c**/**1c′** in a combined yield of 61% following a 24 h reaction time (entry 2). In addition, the reaction time was further reduced to 8 h and the yield was increased to 78% when three equivalents of $THF{\cdot}BF_3$ were used (entry 3).

To investigate the influence of protecting groups on this tandem reaction, substrate **1b** (R^1 = TBS) was subjected to the current reaction conditions (3 equiv of $THF{\cdot}BF_3$ were used). The results revealed that the use of the more stable protecting group *tert*-butyldimetylsilyl (TBS) also provided the spiro-ring products **1c**/**1c′**, albeit with a lower total yield of 59% (Table 1, entry 4). Interestingly, the fused-ring products **1d**/**1d′** (entry 5) were obtained in good yield (71%) from intermediate **1f** by the simple replacement of $THF{\cdot}BF_3$ with $Et_2O{\cdot}BF_3$. A further investigation of the solvents and several other acids demonstrated that substrate **1b** with a TBS protecting group always gave the fused hydrofuran products **1d**/**1d′** in 18–71% yields (entries 5–12). The use of trifluoroacetic acid (TFA; entry 8) provided the only exception, resulting only in the decomposition of the substrate. In conclusion, the chemoselectivity of this one-pot reaction was highly dependent on the acidity of the $THF{\cdot}BF_3$ or $Et_2O{\cdot}BF_3$ additive and the stability of the protecting group.

With the optimized reaction conditions (Table 1, entry 3) in hand, we proceeded to investigate the general scope of the reaction for the conversion of compounds of type **a** to the corresponding spiro-products **c** by means of path A. Subsequently, a series of TMS-protected substrates **2a**–**11a** containing a variety of different substituents on the cyclobutanol moiety were synthesized and subjected to the reaction conditions. As shown in Table 2, the substrates with *cis*-*p*-Me-phenyl, *cis*-*p*-Br-phenyl, diphenyl, and spiro-cycloalkyl at the C3′-position gave the products in good yields of 64 (**2c**/**2c′**), 63 (**3c**/**3c′**), 61 (**4c**/**4c′**), and 75% (**5c**/**5c′**). Furthermore, the reaction exhibited a trend in migratory aptitude similar to that observed in the normal semipinacol rearrangement, in that the tertiary carbon atom migrated preferentially to the secondary carbon atom.[7d,12] Accordingly, substrates bearing different substituents (e.g., di-*n*-butyl, di-*i*-butyl, spiro-cycloalkyl, spiro-pyranyl, and ketospiro-cycloalkyl) at the C2′-position tended to undergo C2′ carbon atom migration reactions to give the corresponding spirofurans **6c**/**6c′**, **7c**/**7c′**, **8c**/**8c′**, **9c**/**9c′**, and **10c**/**10c′** containing two contiguous quaternary centers in reasonable yields. Importantly, the 2′,3′-cyclohexane-fused cyclobutanol substrate **11a** also proved to be effective for generating the corresponding tricyclic products **11c**/**11c′** in the best yield (85%) of all of the products. Furthermore, the products **11c**/**11c′** might serve as a building block in the synthesis of the biologically important sieboldine A (Figure 1). The relative configurations of all of the spiro-furan products **c** and **c′** were deduced based on their ^1H NMR spectra and X-ray diffraction of the products **1c**, **3c**, **4c**, **6c**, **7c**, and **3c′**.[13]

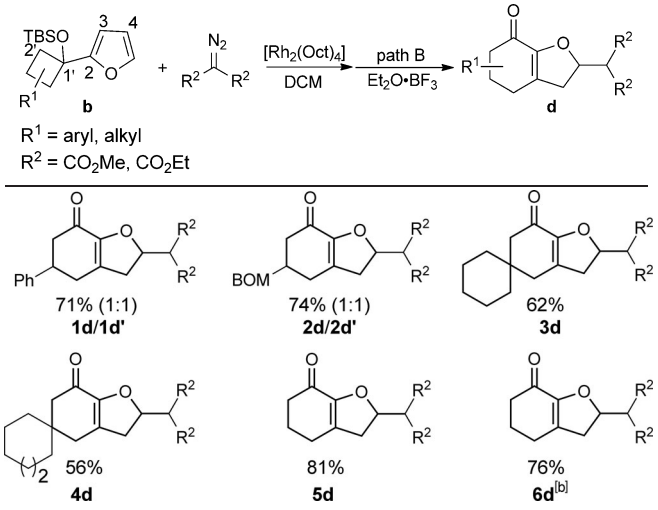
We then applied this one-pot protocol to the syntheses of the fused hydrofurans **d** from various TBS-protected substrates **2b**–**6b** (Table 3). Pleasingly, this series of substrates also produced the desired products in moderate to good yields. When the larger 3′-BOM-substituted substrate **2b** (BOM = benzyloxymethyl) was subjected to the standard conditions, the reaction afforded the desired products **2d**/**2d′** in a combined yield of 74%. In addition, the reaction of substrates **3b** and **4b** containing 3′,3′-spiro-alkyls of different sizes led to the formation of **3d** and **4d** in acceptable yields

Table 2. The spiro-dihydrofuran products **c** and **c′** synthesized through path A.[a]



[a] All reactions were carried out under standard conditions in DCM. Isolated yields ($R^2 = CO_2Me$). The relative configurations of the products **c** were shown. The diastereomeric ratio (d.r.) values were confirmed by ^1H NMR spectroscopy.

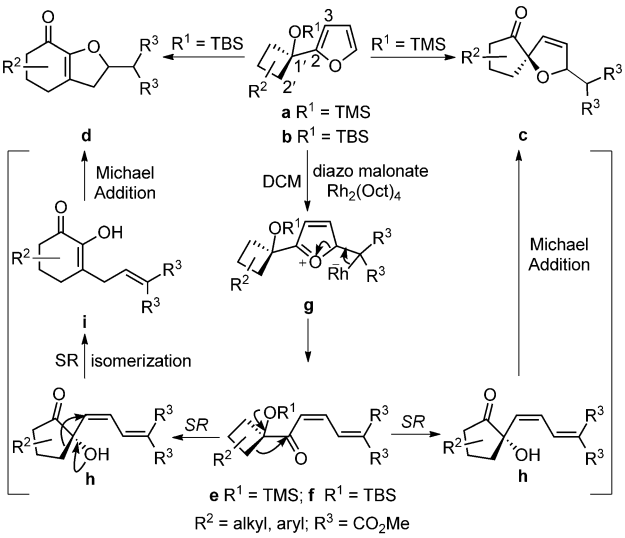
Table 3. The fused-dihydrofuran products **d** and **d′** synthesized through path B.[a]



[a] All reactions were carried out under standard conditions in DCM. Isolated yields (the single products **d** or **d′** could not be isolated by column chromatography). The d.r. values were confirmed by ^1H NMR spectroscopy; $R^2 = CO_2Me$. [b] $R^2 = CO_2Et$.

(62 and 56%, respectively) relative to the yield (53%) of **8c/8c′**. Furthermore, the application of the conditions to substrate **5b**, which did not contain any substituents on the cyclobutanol moiety, gave the desired product **5d** in the best yield of all of the examples (81%). When diethyl diazomalonate and **5b** were subjected to the reaction conditions, the corresponding product **6d** was also produced in a good yield of 76%. Unfortunately, however, the 3′,3′-diphenyl-substituted substrate **6b**[13] did not provide any of the desired product under the same reaction conditions, with the reaction resulting only in decomposition.

On the basis of the experimental results above, a plausible reaction mechanism was proposed (Scheme 2). The



Scheme 2. A plausible mechanism for the tandem reaction.

formation of the spiro and fused products might be attributed to the stronger acidity of $Et_2O•BF_3$[14] relative to that of $THF•BF_3$, with the former capable of inducing the second 1,2-migration of the intermediate **h** and the subsequent oxa-Michael addition of the intermediate, leading in turn to the formation of the fused-dihydrofuran product **d**. In contrast, however, the latter directly underwent an oxa-Michael addition of intermediate diene–ketone **h** and resulted in the spiro-dihydrofuran product **c**.

In summary, we have successfully developed a novel one-pot tandem process for the construction of spiro- or fused-dihydrofuran products from simple furan derivatives under mild reaction conditions. The method represents a useful and highly efficient strategy for the syntheses of differentially substituted spiro and fused compounds. Further studies towards the application of this one-pot tandem reaction are currently underway in our group.

Experimental Section

General procedure: Under an Ar atmosphere and ice bath, $[Rh_2(Oct)_4]$ (1.0 mol%) was added to a solution of substrate **a** or **b** (0.1 mmol mL^{-1})

in DCM; then diazomalonate (1.2 equiv) was added dropwise to the reaction system and stirred vigorously at RT. After the substrate **a** or **b** had been fully transformed to intermediate **e** or **f** as monitored by TLC analysis, $THF{\cdot}BF_3$ or $Et_2O{\cdot}BF_3$ (3.0 equiv) was injected slowly into the reaction solution under an ice bath. The reaction was warmed to RT until full conversion of the intermediate **e** or **f** to product as monitored by TLC analysis. The reaction was quenched by careful addition of saturated $NaHCO_3$ aqueous solution under an ice bath. The resulting solution was extracted with DCM, washed with brine, dried over $MgSO_4$, and chromatographed (ethyl acetate/petroleum ether) to give the product.

Acknowledgements

We greatly acknowledge financial support from the "973" Program (grant no. 2010CB833203), the NSFC (grant nos. 21072085, 20921120404, 21102061, 20972059, 21202073, 21290180, and 21272097), the Key National S&T Program "Major New Drug Development" of Ministry of Health of China (2012ZX09201101-003), and the "111" Program of the MOE, the fundamental research funds for the central universities (lzujbky-2012-60, 86).

Keywords: chemoselectivity · dihydrofurans · Michael addition · semipinacol rearrangements · tandem reactions

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Received: January 19, 2013
Published online: March 13, 2013

