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A highly diastereoselective approach to tetrahydrofurans via [3+2] cycloadditions of silylmethyl-substituted cyclopropanes with aldehydes and ketones

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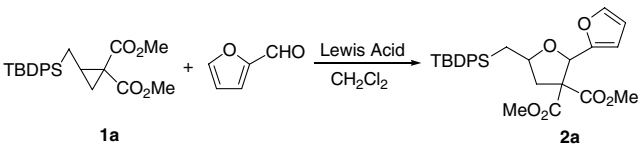
Abstract—An efficient and highly diastereoselective synthesis of highly substituted tetrahydrofurans from the reaction of a vicinal *t*-butyldiphenylsilylmethyl-substituted cyclopropyl diester with aldehydes and ketones has been developed. The 2,5-*cis*-disubstitution predominates over the 2,5-*trans*-disubstitution by as much as 12:1. The reaction with cyclic ketones generates spiro-fused tetrahydrofurans in good yields.

Many natural products and therapeutic agents possessing antitumor, anthelmintic, antimalarial, antimicrobial, and antiprotozoal activities contain the tetrahydrofuran subunit.[1] This subunit is also present in polyether antibiotics,[2] lignans,[3] and *C*-glycosidases.[4] The development of easy and efficient methods to construct this skeleton has been a matter of much research.[5] The high degree of reactivity of a donor–acceptor-substituted cyclopropane makes it a versatile building block.[6,7] Herein, we report on the generation of 1,3-dipoles from the cyclopropane derivatives **1a**–**c**, wherein the negative charge is stabilized by the malonyl diester function and the positive charge is stabilized by silicon through a β-effect, and their reactions with aldehydes and ketones to generate highly substituted tetrahydrofurans. Although these studies were in progress, Pohlhaus and Johnson reported the preparation of 2,5-disubstituted tetrahydrofurans from donor–acceptor-substituted cyclopropanes and aldehydes;[8] however, this chemistry was restricted to the stabilization of the cation by an aryl substituent. As the carbon–silicon bond is preserved in our product, further manipulation into other functional groups, including a hydroxyl group, would be possible.[9] The oxidative cleavage of carbon–silicon bonds under basic conditions to generate a hydroxyl group has also been reported from our laboratory.[10]

We have reported previously on the $TiCl_4$-promoted efficient and regioselective ring opening of vicinal silylmethyl-substituted cyclopropylmalonates and other similar substrates to furnish dihydrofurans through intramolecular ring closure.[11] We envisioned the interception of an in situ generated 1,3-dipole with a carbonyl species to generate highly substituted tetrahydrofurans in a single step.

When **1a** was reacted with furfural in the presence of $TiCl_4$ at −30 °C, only a little of the desired tetrahydrofuran product was formed. The search for a suitable Lewis acid to accomplish the task (Scheme 1) was, therefore, undertaken. $Cu(OTf)_2$ and $Yb(OTf)_3$ were completely ineffective. $Sn(OTf)_2$, $BF_3{\cdot}OEt_2$ and Et_2AlCl were also unsatisfactory. The reactions with the first two Lewis acids were very slow; 40–45% of the cyclopropane substrate was recovered from a 3 h reaction in each instance. The reaction with Et_2AlCl was complete in 3 h,



1a 2a

Scheme 1. [3+2] Cycloaddition of **1a** with furfural.

Keywords: 1,3-Dipole; Cycloaddition; 2,3,5- and 2,3,4,5-Substituted tetrahydrofurans.
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Table 1. Screening of Lewis acids for the reaction of **1a** with furfural

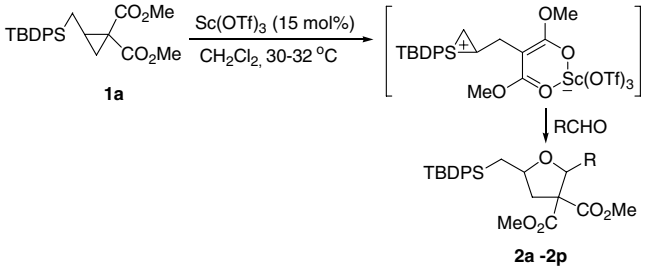
Entry	Lewis acid	equiv	dr (% yield)
1	$Sn(OTf)_2$[a,b]	0.15	6.3:1 (34)
2	$BF_3{\cdot}OEt_2$[b,c,d]	1.2	2.7:1 (23)
3	Et_2AlCl[a]	1.2	2.4:1 (38)
4	$SnCl$[c]	0.05	6.3:1 (59)
5	$Sc(OTf)_3$[a]	0.15	3.5:1 (100)

[a] Reaction was carried out at rt for 3 h.
[b] The yields are based on the reacted starting cyclopropane derivative.
[c] The reaction was carried out at 0 °C–rt for 4 h.
[d] 3-Carbomethoxy-5-*t*-butyldiphenylsilylmethyl tetrahydrofuran-2-one was obtained in a 30% isolated yield.

however, it generated a rearranged dihydrofuran derivative and the desired product in 38% and 30% yields, respectively. Although $SnCl_4$ gave the desired product with reasonably high diastereoselectivity, moderate yields of the desired product and the formation of 3-carbomethoxy-5-*t*-butyldiphenylsilylmethyltetrahydrofuran-2-one precluded its further use. $Sc(OTf)_3$ was the best Lewis acid and the desired product was isolated in near quantitative yield as a 3.5:1 diastereomeric mixture from a 3 h reaction at room temperature. The results are compiled in Table 1.

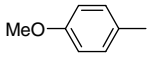
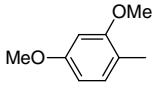
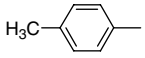
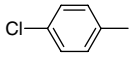
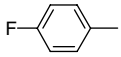
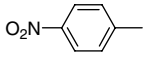
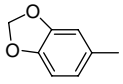
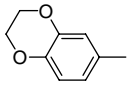
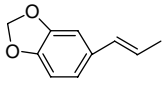
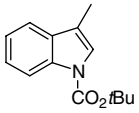
We next investigated the substrate-dependence of the above one-pot tetrahydrofuran synthesis. The cyclopropane derivative **1a** reacted smoothly with 3 equiv of several aromatic and heteroaromatic aldehydes to generate the desired products with high cis-selectivity (Scheme 2, Table 2). The diastereoselectivity was determined from NOE measurements. Aromatic aldehydes with electron-donating and electron-withdrawing substituents were reacted: electron-donating substituent facilitated the reaction (entries c–f), while electron-withdrawing substituent retarded it (entries g–k).

The reaction proceeded smoothly with piperonal without affecting the acid-sensitive acetal linkage (entry l). Vinylogous aromatic aldehydes also reacted well to furnish the expected products in good yields (entries n–o). Although pyridine-2-carboxaldehyde and *N*-methylindole-3-carboxaldehyde (not shown) did not react, *N*-Boc-indole-3-carboxaldehyde reacted well and the product was obtained in a good yield (entry p). The failures with pyridine-2-carboxaldehyde and *N*-methylindole-3-carboxaldehyde are likely to be associated with the possible coordination of $Sc(OTf)_3$ with the ring-nitrogen in the former and the carbonyl oxygen in the latter in preference to the carboxylates of the cyclopropane substrate.



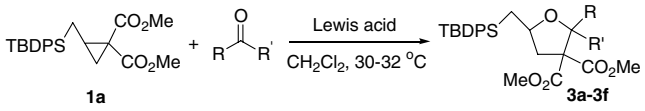
Scheme 2. [3+2] Cycloaddition of **1a** with aldehydes.

Table 2. [3+2] Cycloaddition of **1a** with aldehydes

Entry	R	Time (h)	Yield[a] (%)	dr (*cis*:*trans*)[b]
a		3	100	3.5:1
b		3	85	9:1
c		6	85	5.4:1
d		6	98	5.2:1
e		6	90	6.7:1
f		6	85	3.8:1
g		6	75	12.5:1
h		6	70	3.8:1
i		6	60	9:1
j		20	30[c]	10:1
k		20	40	12:1
l		6	97	5:1
m		6	98	4.5:1
n		3	80	5:1
o		3	80	2.6:1
p		6	75	4.3:1

[a] Isolated yields. The starting cyclopropyl substrate was recovered to the extent of 22%, 28%, and 47% from the reactions in entries h, i, and k, respectively.
[b] The diastereomeric ratios were estimated from the ^1H integrals of the isomeric mixtures.
[c] Unlike the reaction at entry k, all the cyclopropane reactant was consumed, however, a substantial amount of **4a** was formed (see Scheme 7).

Saturated aliphatic aldehydes such as *n*-butanal and α, β-unsaturated aliphatic aldehydes such as citral did not react under the $Sc(OTf)_3$ conditions. The reaction of *n*-butanal using $SnCl_4$ was complicated at both room temperature and in the temperature range −78 °C to 0 °C.



Scheme 3. [3+2] Cycloaddition of **1a** with ketones.

Table 3. [3+2] Cycloaddition of **1a** with ketones

Entry	Acid	Ketone	Time (h)	Yield (%)	dr[d]
a	$Sc(OTf)_3$[a]		3	84	
b	$Sc(OTf)_3$[a]		3	82	
c	$Sc(OTf)_3$[a]		3	75	1:1
d	$SnCl_4$[b]		6	70	
e	$SnCl_4$[b]		6	75	3.7:1
f	$SnCl_4$[c]		4	78	3.5:1

[a] 15 mol % of $Sc(OTf)_3$ was used.
[b] 5 mol % of $SnCl_4$ was used.
[c] The reaction was conducted at −78 °C to 0 °C.
[d] The diastereomeric ratios were estimated from the ^1H integrals of the isomeric mixtures.

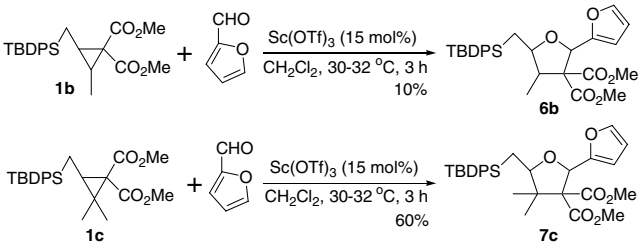
Next, we constructed spiro skeletons in good yields by reacting **1a** with cyclic ketones (Scheme 3, Table 3). 2-Cyclohexenone reacted to generate a 1:1 diastereomeric mixture of the products. (*R*)-(−)-Carvone (not shown) was inert as a substrate; the methyl substituent at the α-position possibly caused enough steric crowding to suppress the reaction completely. The reaction with 3-methyl-2-cyclohexenone was very slow in comparison to the reaction with 2-cyclohexenone; only one-quarter of the cyclopropane had reacted after 20 h at room temperature to generate a 1:1 diastereomeric mixture.

Acyclic ketones did not react under the $Sc(OTf)_3$-promoted condition. However, a catalytic amount of $SnCl_4$ proved efficient and **1a** reacted with the selected acyclic ketones to generate tetrahydrofurans in good yields; small amounts of 3-carbomethoxy-5-*t*-butyldiphenylsilylmethyltetrahydrofuran -2-one, **4a**, (Scheme 7) were also formed. The reaction with 2-butanone furnished a 3.7:1 diastereomeric mixture.

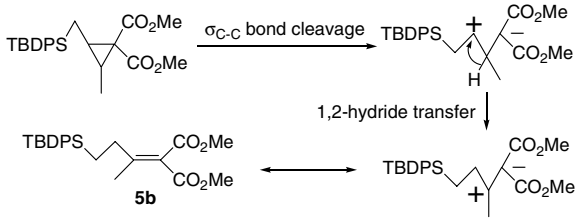
Alkyl aryl ketones such as methyl phenyl ketone also reacted well under the $SnCl_4$ conditions. The reaction, however, was sensitive to temperature and proceeded cleanly at −78 °C to 0 °C over 4 h. $Sc(OTf)_3$ was completely ineffective even at room temperature. Sugita et al. had earlier employed the above $SnCl_4$ conditions for the cycloaddition of alkyl aryl ketones with donor–acceptor substituted cyclopropanes.[12]

To assess the further scope of the present protocol, in particular, its tolerance to substituents in the cyclopropane reactant, we examined the reactions of **1b** and **1c** with furfural to generate all-substituted tetrahydrofurans (Scheme 4). The reaction of **1b** with furfural, however, generated methyl 5-*t*-butyldiphenylsilyl-2-carbomethoxy-3-methyl-2-pentenoate, **5b**, as the main product in a 85% yield; the desired cycloaddition product **6b** was formed but only in a 10% yield as a mixture of diastereomers. 1,2-Hydride transfer from the methyl-bearing carbon to the silylmethyl-substituted carbon in the 1,3-dipole, as shown in Scheme 5, may explain the formation of the major product.

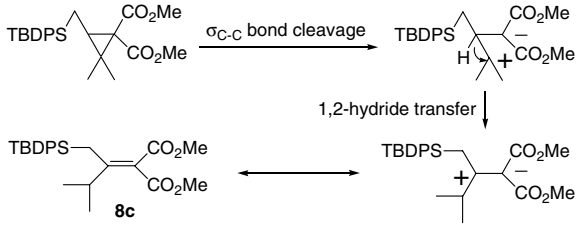
In order to prevent the hydride shift we studied substrate **1c**, wherein the central carbon is substituted by two methyl groups. The reaction with furfural furnished the cycloaddition product **7c** in a moderate yield (60%) with 14:1 diastereoselectivity and methyl 3-*t*-butyldiphenylsilyl methyl-2-carbomethoxy-4-methyl-2-pentenoate, **8c**, in a 28% yield. The reaction of **1c** with cyclic ketones such as cyclopentanone was unsuccessful; the only product formed was the above pentenoate **8c** in a quantitative yield. Pentenoate **8c** may tentatively be considered to arise from cleavage of the alternate cyclopropane bond followed by 1,2-hydride transfer from the silylmethyl-bearing carbon to the carbocation as shown in Scheme 6.



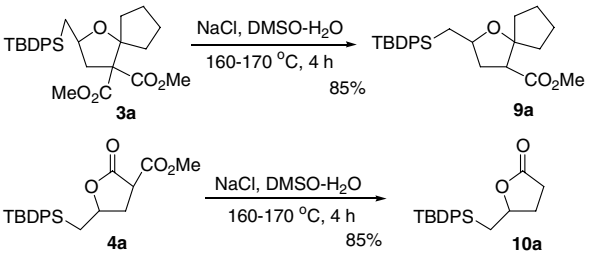
Scheme 4. Construction of 2,3,4,5-tetrasubstituted tetrahydrofurans.



Scheme 5. Tentative pathway for the generation of **5b**.



Scheme 6. Tentative pathway for the generation of **8c**.



Scheme 7. Decarboxylation of **3a** and **4a**.

The major utility of the present cycloaddition protocol is in the formation of tetrahydrofuran derivatives bearing substituents that are amenable to further rapid chemical transformations, including decarboxylation of one ester group. On heating with NaCl in wet DMSO, **3a** and **4a** underwent a smooth reaction to generate **9a** (1:1 diastereomeric mixture) and **10a**, respectively, in excellent yields (Scheme 7).[13] The alcohol that can be generated from the oxidative cleavage of the carbon–silicon bond in **10a** is an important intermediate for further elaboration into useful products.[14]

In summary, 2-*t*-butyldiphenylsilylmethyl-substituted cyclopropyl diesters reacted efficiently with both aldehydes and ketones in the presence of a catalytic amount of either $Sc(OTf)_3$ or $SnCl_4$ to generate highly substituted tetrahydofuran products in good to excellent yields. The present methodology allows the introduction of two different groups at the 2- and 5-positions of the tetrahydrofuran ring which enhances its utility for further synthetic applications.[15–17]

Acknowledgments

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15. General procedure for the $Sc(OTf)_3$-catalyzed reactions of **1a/1b/1c** with aldehydes and ketones: $Sc(OTf)_3$ (0.0185 g, 15 mol %) was added to a stirred solution of **1a/1b/1c** (0.25 mmol) and an aldehyde or ketone (0.75 mmol) in anhydrous CH_2Cl_2 (2 mL). The reaction was stirred at room temperature (30–32 °C) until the disappearance of **1a/1b/1c** was confirmed by TLC. The reaction mixture was concentrated and the residue was chromatographed over silica gel (EtOAc/hexanes).
16. General procedure for the $SnCl_4$-assisted reaction of silylmethyl-substituted cyclopropyl diester **1** with ketones: A freshly prepared solution of $SnCl_4$ in anhydrous CH_2Cl_2 (0.1 mL, 5 mol %) was added to a stirred solution of **1** (0.25 mmol) and a ketone (0.75 mmol) in anhydrous CH_2Cl_2 (2 mL). The reaction was stirred at room temperature (30–32 °C) until the disappearance of **1** by TLC. The reaction mixture was concentrated and the residue was chromatographed over silica gel (EtOAc/hexanes).
17. General procedure for dealkyldecarboxylation. NaCl (0.017 g, 0.28 mmol) and H_2O (10 μL) were added to a solution of the substrate (0.141 mmol) in DMSO (1 mL). After refluxing for 4 h (oil bath temperature 160–170 °C), the mixture was cooled to room temperature and taken up in EtOAc (10 mL). This was washed with water (2 × 5 mL) and brine (1 × 5 mL). The solvent was removed and the residue was purified by silica gel chromatography (EtOAc/hexanes) to furnish the pure product.
Spectral data of *cis*-**2a**. ^1H NMR (400 MHz, $CDCl_3$): δ 7.66–7.59 (4H, m), 7.42–7.26 (7H, m), 6.29–6.28 (2H, m),

5.46 (1H, s), 3.93–3.86 (1H, m), 3.59 (3H, s), 3.37 (3H, s), 2.36–2.31 (1H, dd, $J = 13.2$, 10.5 Hz), 2.02–1.98 (1H, dd, $J = 14.4$, 3.9 Hz), 1.72–1.67 (1H, dd, $J = 13.2$, 5.1 Hz), 1.66–1.60 (1H, dd, $J = 14.4$, 10.0 Hz), 1.04 (9H, s). ^{13}C NMR (100 MHz, $CDCl_3$, major isomer): δ 170.4, 168.6, 151.1, 142.6, 136.1, 134.1, 133.7, 129.3, 129.2, 127.6, 110.2, 108.7, 77.1, 65.0, 52.8, 52.7, 41.0, 29.7, 27.6, 18.1, 17.0. IR (thin film): 1737, 1431, 1266, 1150, 1105, 1054, 1016, 737, 700 cm^{-1}. Anal. Calcd for $C_{29}H_{34}O_6Si$: C, 68.75; H, 6.76%. Found: C, 68.60; H, 6.80%.

Characteristic partial ^{1}H data of *trans*-**2a**: δ 6.25–6.22 (1H, m), 6.14 (1H, d, $J = 3.4$ Hz), 5.72 (1H, s), 4.66–4.62 (1H, m), 3.71 (3H, s), 3.36 (3H, s), 2.55–2.50 (1H, dd, $J = 13.4$, 6.8 Hz), 1.86–1.81 (1H, dd, $J = 14.4$, 4.4 Hz), 1.47–1.39 (2H, m).

Spectral data of **3d**. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67–7.63 (4H, m), 7.40–7.33 (6H, m), 4.24–4.17 (1H, m), 3.71 (3H, s), 3.61 (3H, s), 2.03–1.98 (1H, dd, $J = 13.9$, 7.8 Hz), 1.91–1.84 (2H, m), 1.66–1.60 (1H, dd, $J = 14.4$, 10.4 Hz), 1.31 (3H, s), 1.16 (3H, s), 1.04 (9H, s). ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.7, 169.9, 136.1, 134.6, 134.0, 129.1, 127.5, 82.8, 73.5, 66.9, 52.3, 52.2, 39.9, 27.7, 25.5, 24.2, 18.8, 18.0. IR (thin film): 1738, 1461, 1432, 1372, 1265, 1146, 1102, 1060, 1013, 926, 736, 702 cm^{-1}. Anal. Calcd for $C_{27}H_{36}O_5Si$: C, 69.20; H, 7.74%. Found: C, 69.00; H, 7.70%.

Spectral data of *trans*-**7c**. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82–7.80 (2H, m), 7.70–7.67 (2H, m), 7.40–7.32 (6H, m), 7.26–7.25 (1H, m), 6.23–6.22 (1H, dd, $J = 3.2$, 1.7 Hz), 6.15–6.14 (1H, d, $J = 3.2$ Hz), 5.73 (1H, s), 3.83 (3H, s), 3.55–3.51 (1H, m), 3.29 (3H, s), 1.83–1.78 (1H, dd, $J = 15.1$, 3.4 Hz), 1.01 (9H, s), 0.97–0.93 (1H, dd, $J = 15.1$, 10.7 Hz), 0.89 (3H, s), 0.81 (3H, s). ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.5, 168.9, 152.1, 142.4, 142.3, 136.6, 136.3, 136.2, 134.9, 133.5, 129.2, 127.4, 110.2, 108.4, 83.5, 76.2, 70.3, 52.5, 49.3, 29.7, 28.4, 27.7, 22.1, 18.6. IR (thin film): 1733, 1431, 1258, 1175, 1101, 1020, 929, 815, 738, 656 cm^{-1}. Anal. Calcd for $C_{31}H_{38}O_6Si$: C, 69.63; H, 7.16%. Found: C, 69.50; H, 7.20%.

Characteristic partial ^{1}H data of *cis*-**7c**. δ 5.27 (1H, s), 4.34–4.28 (1H, m), 3.69 (3H, s), 3.39 (3H, s), 1.61 (3H, s), 1.37 (3H, s).

Spectral data of **10a**. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64–7.59 (4H, m), 7.44–7.33 (6H, m), 4.57–4.54 (1H, m), 2.35–2.23 (2H, m), 2.04–1.99 (1H, dd, $J = 14.4$, 3.9 Hz), 1.67–1.60 (1H, m), 1.53–1.41 (2H, m), 1.03 (9H, s). ^{13}C NMR (100 MHz, $CDCl_3$): δ 177.0, 135.9, 133.8, 132.8, 129.61, 129.56, 129.5, 127.92, 127.86, 127.8, 127.7, 80.2, 30.5, 29.7, 27.6, 18.3. IR (thin film): 1774, 1464, 1424, 1431, 1271, 1168, 1107, 1003, 971, 909, 859, 816, 735, 703 cm^{-1}. Anal. Calcd for $C_{21}H_{26}O_2Si$: C, 74.51; H, 7.74%. Found: C, 74.40; H, 7.65%.