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# Highly Regio- and Stereoselective Cyclic Iodoetherification of 4,5-Alkadienols. An Efficient Preparation of 2-(1′(*Z*)-Iodoalkenyl)tetrahydrofurans

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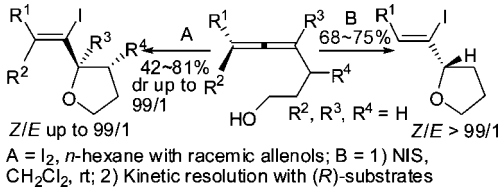


In this paper, an efficient way to synthesize 2-(1′(*Z*)-iodoalkenyl)tetrahydrofurans from 4,5-alkadienols and $I_2$ was developed. The reaction of the 4,5-allenols with a substituent in the 3-position afforded the *trans*-2,3-disubstituted tetrahydrofurans with very high diastereoselectivity. However, when the axially optically active 4,5-allenol was treated with $I_2$ in *n*-hexane, the efficiency for chirality transfer was low. This problem was circumvented by conducting the reaction in $CH_2Cl_2$ at room temperature and applying *N*-iodosuccinimide as the electrophilic reagent; however, the *Z*/*E* ratio for the products is much lower. Highly optically active *Z*-products may be prepared via the kinetic resolution via a Sonogashira coupling reaction with propargyl alcohol.

Although allenes were considered to be highly unstable for a long period of time, during the last 10 years, much attention has been paid to this area demonstrating the potential of allenes in organic synthesis.[1] Recently, we have reported electrophilic cyclization of functionalized allenes with high regioselectivity.[2] On the other hand, tetrahydrofuran is a very important unit in many potentially useful natural products.[3,4] Recently, many groups have focused on the synthesis of substituted tetrahydrofuran derivatives.[5] Bäckvall et al. reported the $Pd(OAc)_2$-catalyzed bromoetherification of 4,5-allenols in which the *Z*/*E* selectivity ranged from 84/16 to 94/6.[6] Herein, we wish to report an efficient cyclic iodoetherification of 4,5-allenols with $I_2$ or *N*-iodosuccinimide (NIS), which is cheap/readily available and forms the tetrahydrofuran derivatives including the optically active ones with very high regio- and stereoselectivity.

The starting 4,5-allenols were prepared according to the published procedure from propargylic alcohols **1**.[6] Claisen rearrangement and the subsequent reduction would form 3,4-allenols **2a**−**n**, which were tosylated and subsequently treated with NaCN to afford 4,5-allenylic nitriles. Hydrolysis of the nitriles with NaOH and reduction of the acids with $LiAlH_4$ would finally afford 4,5-allenols **5a**−**n** (Tables S1−4 in the Supporting Information). Such methods were also used for the preparation of optically active 4,5-allenols (*R*)-**5b** and (*R*)-**5c** using readily available propargylic alcohols (*R*)-**1b** and (*R*)-**1c** as the starting material (Scheme 1).

**Electrophilic Cyclization.** The initial electrophilic cyclization experiment was conducted by stirring a mixture of 4,5-decadienol **5a** and $I_2$ in $CH_2Cl_2$ at room temperature. Fortunately, we observed the formation of tetrahydrofuran product **6a** in 81% yield with a *Z*/*E* ratio of 96/4. The *Z*-configuration of the carbon−carbon double bond in **6a** was determined by its $^1H-^1H$ NOESY analysis (Figure 1 and Supporting Information). Seven-membered ring 2-butyl-3-iodo-2,5,6,7-tetrahydrooxepin **7a** was not formed (entry 1, Table 1). Encouraged by these results, we screened a series of solvents including $CH_3CN$, THF, and DMF; however, they

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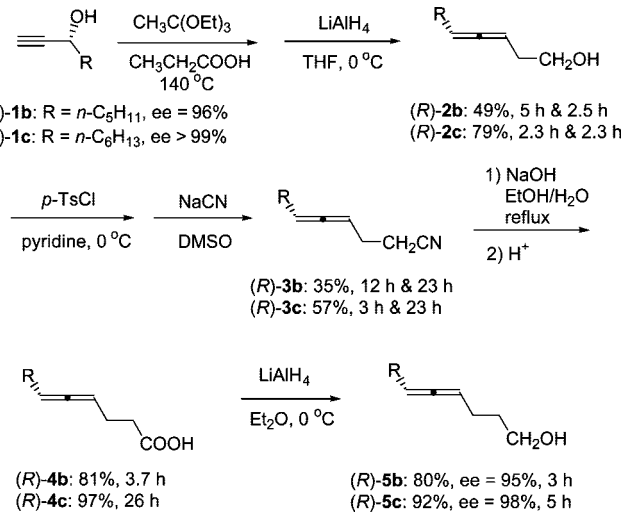
**SCHEME 1**




all showed poor stereoselectivity for the formation of the carbon−carbon double bond (entries 2−4, Table 1). Further study revealed that the reaction in *n*-hexane could afford the product **6a** in a higher stereoselectivity, although the yield was lower (entry 6, Table 1). With 1 equiv of $I_2$, the yield is 88% with the same level of stereoselectivity (entry 7, Table 1). However, the reaction at −20 °C afforded **6a** in relatively lower yield and stereoselectivity (entry 8, Table 1).

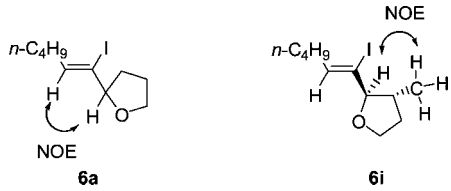
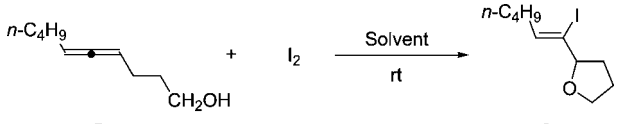



**FIGURE 1.** $^1H-^1H$ NOESY analysis of **6a** and **6i**.

The scope of the cyclic iodoetherification of 4,5-allenols was explored under the optimized conditions (entry 7, Table 1). Some typical results are summarized in Table 2. It is noteworthy that the reaction afforded 2-(1′(*Z*)-iodoalkenyl)tetrahydrofuran derivatives in good yield and fairly high *Z*-stereoselectivity. $R^1$ may be alkyl group and $R^2$ as well as $R^3$ can be H or alkyl group. In addition, if a substituent $R^3$ was introduced at the 3-position, only one diastereoisomer was formed highly diastereoselectively (entries 6−9, Table 2) and the relative configurations of these products **6f**−**i** were confirmed to be *trans* according to $^1H-^1H$ NOESY analysis of **6i** (Figure 1 and Supporting Information).

**TABLE 1. Cyclic Iodo-etherification of 5a under Different Conditions**

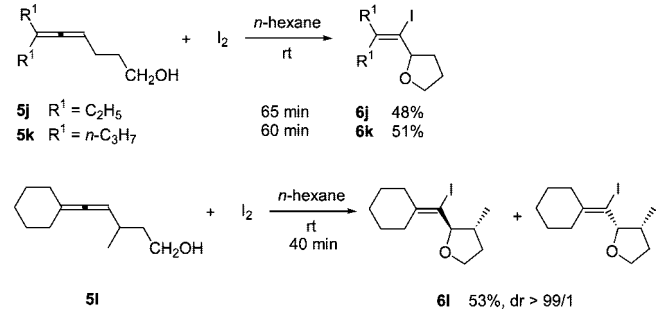



| entry | $I_2$ (equiv) | solvent | time (h) | yield of **6a**[a] (%) | *Z*/*E*[a] |
|---|---|---|---|---|---|
| 1 | 1.2 | $CH_2Cl_2$ | 1.5 | 81 | 96/4 |
| 2 | 1.2 | $CH_3CN$ | 1.4 | 89 | 91/9 |
| 3 | 1.2 | THF | 1.8 | 85 | 89/11 |
| 4 | 1.2 | DMF | 16 | 82 | 89/11 |
| 5 | 1.2 | *c*-hexane | 1.2 | 76 | 97/3 |
| 6 | 1.2 | *n*-hexane | 1.9 | 58 | 99/1 |
| 7 | 1.0 | *n*-hexane | 1.5 | 88 | 98/2 |
| 8[b] | 1.0 | *n*-hexane | 2 | 75 | 95/5 |

[a] Determined by $^1H$ NMR analysis (400 MHz). [b] This experiment was carried out at −20 °C.

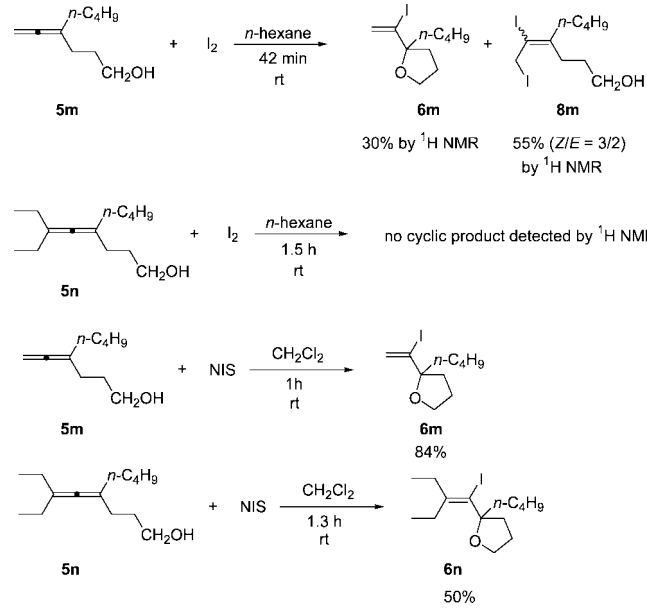
The reactions of 6,6-disubstituted-4,5-allenols **5j**,**k** were also conducted under the optimized conditions to afford the electrophilic cyclization products **6j**,**k**. The reaction of **5l** with a methyl substituted at the 3-position also afforded the *trans*-product **6l** with a dr of >99/1 (Scheme 2).

**SCHEME 2**



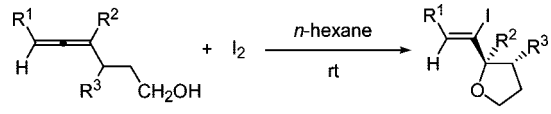

However, 4-monosubstituted 4,5-allenol **5m** showed poor selectivity toward $I_2$ under the optimized conditions affording a mixture of the expected tetrahydrofuran derivative **6m** and a *E*/*Z* mixture of 5,6-diiodo-4-(*n*-butyl)-4-hexenol **8m**. In addition, the reaction of 4,6,6-trisubstituted allenol **5n** under the standard reaction failed to afford cyclization product. Luckily, by introducing NIS the iodoetherification reaction in $CH_2Cl_2$ afforded the expected cyclization products **6m** and **6n** easily at room temperature in 84% and 50% yields, respectively (Scheme 3).

**SCHEME 3**




In addition, it is surprising for us to observe that when optically active 4,5-allenol (*R*)-**5b** was used, its reaction with $I_2$ in *n*-hexane afforded the related products (*S*)-**6b** in 80% yield with only 20% ee. Further screening led to the observations that the reaction of (*R*)-**5b** and (*R*)-**5c** with NIS in $CH_2Cl_2$ afforded (*S*)-*Z*-**6b**, (*R*)-*E*-**6b** and (*S*)-*Z*-**6c**, (*R*)-*E*-**6c** in a *Z*/*E* ratio of 90/10 without obvious loss of the chirality. Further kinetic

**TABLE 2. Iodocyclization Reaction of Differently Substituted 4,5-Allenols with $I_2$**

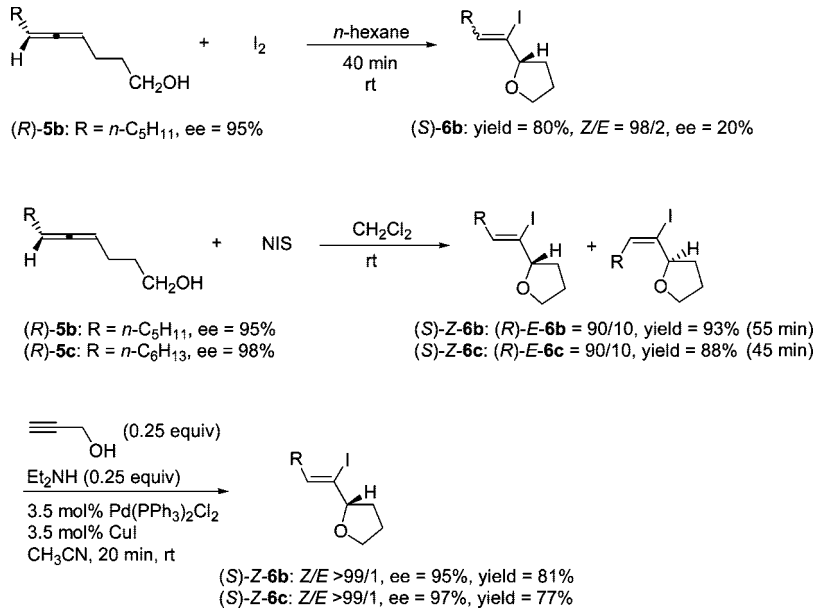



**5** **6**

| entry | $R^1$ | $R^2$ | $R^3$ | | yield of **6** %[a] | time (min) | *Z*/*E*[b] | dr (*trans*/*cis*)[b] |
|---|---|---|---|---|---|---|---|---|
| 1 | *n*-$C_4H_9$ | H | H | (**5a**) | 81 (**6a**) | 45 | 97/3 | |
| 2 | *n*-$C_5H_{11}$ | H | H | (**5b**) | 79 (**6b**) | 50 | 97/3 | |
| 3 | *n*-$C_6H_{13}$ | H | H | (**5c**) | 76 (**6c**) | 60 | 97/3 | |
| 4 | $CH_3$ | $C_2H_5$ | H | (**5d**) | 42 (**6d**) | 40 | >99/1 | |
| 5 | *n*-$C_3H_7$ | *n*-$C_4H_9$ | H | (**5e**) | 78 (**6e**) | 60 | >99/1 | |
| 6 | $CH_3$ | H | *n*-$C_3H_7$ | (**5f**) | 73 (**6f**) | 30 | 97/3 | >98/2 |
| 7 | $CH_3$ | H | $C_2H_5$ | (**5g**) | 59 (**6g**) | 40 | 97/3 | >97/3 |
| 8 | *n*-$C_6H_{13}$ | H | *n*-$C_3H_7$ | (**5h**) | 81 (**6h**) | 25 | 98/2 | >98/2 |
| 9 | *n*-$C_4H_9$ | H | $CH_3$ | (**5i**) | 81 (**6i**) | 33 | 96/4 | >96/4 |

[a] Isolated yield after flash chromatography on silica gel. [b] Determined by $^1H$ NMR analysis and $^1H-^1H$ NOESY analysis.

**SCHEME 4**

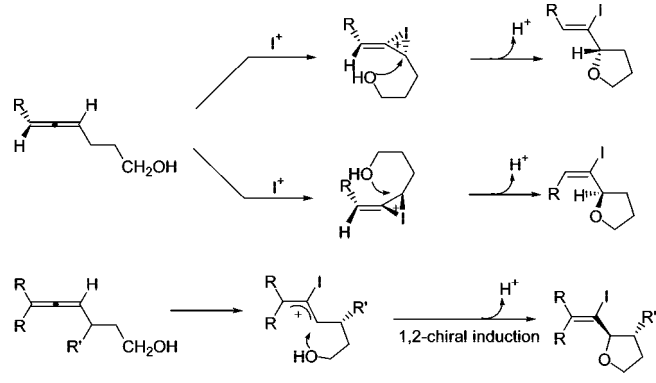



resolution via Sonogashira coupling[7] afforded (*S*)-*Z*-**6b** and (*S*)-*Z*-**6c** with >99/1 *Z*/*E* ratio and the same enantiopurity of the starting 4,5-allenols (Scheme 4).

The stereoselectivity may be explained via the protocol shown in Scheme 5: the three-membered iodonium intermediate formed was intramolecularly attacked by the oxygen atom to form the five-membered ring. The steric interaction between the R group and the hydroxy group determines the *Z*-selectivity.[2b] Deprotonation would lead to the formation of the substituted tetrahydrofuran products. With NIS as the electrophile, the steric repulsion between the R group and NIS becomes another major issue, which reduced the *Z*/*E* selectivity. However, the diastereoselectivity observed in the formation of **6f**−**i**,**l** must be formed via a 2-iodo-$\pi$-allylic cationic intermediate (Scheme 5).

In conclusion, we have developed a convenient method for the efficient and stereoselective synthesis of 2-(1′(*Z*)-iodoalkenyl)tetrahydrofurans by the cyclic iodoetherification reaction of 4,5-allenols with high regio- and high stereoselectivity. For the substrates with a substituent at the 3-position, the reaction with $I_2$ in *n*-hexane afforded the *trans*-2,3-disubstituted tetrahydrofurans highly diastereoselectively. For the transfer of axial chirality, the reaction should be carried out with NIS in $CH_2Cl_2$. Further studies in this area are being conducted in our laboratory.

**SCHEME 5**




(7) For kinetic resolution using Sonogashira coupling, see: Ma, S.; Ma, Z. *Synlett* **2006**, *6*, 1263. For recent reviews of Sonogashira reaction, see: (a) Chinchilla, R.; Nájera, C. *Chem. Rev.* **2007**, *107*, 874. (b) Doucet, H.; Hierso, J.-C. *Angew. Chem., Int. Ed.* **2007**, *46*, 834.

## Experimental Section

**Preparation of 4,5-Decadienol (5a). Typical Procedure.** The reaction of $LiAlH_4$ (0.84 g, 22.1 mmol) and **4a** (2.45 g, 14.6 mmol) in anhydrous ether (37 mL) was monitored by TLC. After complete consumption of the starting material, the mixture was quenched with water, extracted with ether for three times, washed with brine, and dried over anhydrous $Na_2SO_4$. Filtration, evaporation, and flash column chromatographic separation on silica gel (petroleum ether/ethyl acetate = 5:1) afforded **5a** (1.41 g, 63%) as a liquid:[8] $^1$H NMR (400 MHz, $CDCl_3$) $\delta$ 5.12−5.05 (m, 2H), 3.68 (t, $J$ = 6.4 Hz, 2H), 2.10−2.02 (m, 2H), 2.01−1.93 (m, 2H), 1.72−1.60 (m, 3H), 1.42−1.28 (m, 4H), 0.89 (t, $J$ = 7.0 Hz, 3H).

**Preparation of 2-(1′-Iodo-1′(*Z*)-hexenyl)tetrahydrofuran (6a). Typical Procedure.** To a solution of **5a** (61.9 mg, 0.40 mmol) in 1 mL of *n*-hexane were added sequentially iodine (103.1 mg, 0.41 mmol) and 1 mL of *n*-hexane at room temperature. After complete conversion of the starting material as monitored by TLC, the reaction mixture was quenched with 10 mL of $H_2O$ and saturated aqueous $Na_2S_2O_3$ to remove the excess $I_2$. The mixture was extracted with 15 mL of ether, washed with 10 mL of water and 10 mL of brine, and dried over anhydrous $Na_2SO_4$. After filtration and evaporation, the *Z*/*E* ratio of **6a** was determined by $^1$H NMR analysis using 1,3,5-trimethylbenzene as the internal standard (18.5 $\mu$L, 0.13 mmol). Chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 40/1) of the crude product afforded **6a** (91.2 mg, 81%, *Z*/*E* = 97/3) as a liquid: $^1$H NMR (400 MHz, $CDCl_3$) $\delta$ [6.30 (t, $J$ = 7.6 Hz, *E*-isomer, 0.03H); 5.89 (t, $J$ = 6.8 Hz, *Z*-isomer, 1H)], 4.17 (t, $J$ = 6.2 Hz, 1H), 4.03−3.96 (m, 1H), 3.88−3.81 (m, 1H), 2.20−2.10 (m, 2H), 2.09−1.80 (m, 4H), 1.43−1.25 (m, 4H), 0.89 (t, $J$ = 7.0 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 135.5, 112.4, 84.7, 69.2, 35.2, 32.8, 30.3, 25.7, 22.2, 13.9; IR (neat) $\nu$ ($cm^{-1}$): 2956, 2926, 2870, 1642, 1457, 1349, 1266, 1234, 1180, 1061; MS (70 eV, EI) *m*/*z* 280 ($M^+$, 8.36), 153 ($M^+$ − I, 68.17), 97 (100); HRMS calcd for $C_{10}H_{18}OI$ ($M^+$ + H) 281.0397, found 281.0391.

**Preparation of Optically Active 2-(1′-Iodo-1′-heptenyl)tetrahydrofuran ((*S*)-(*Z*)-6b, (*R*)-(*E*)-6b). Typical Procedure.** To a solution of (*R*)-(−)-**5b** (33.4 mg, 0.20 mmol, 95%ee) in 1 mL of $CH_2Cl_2$ were added sequentially NIS powder (54.4 mg, 0.24 mmol) and an additional 1 mL of $CH_2Cl_2$. The resulting mixture was stirred at room temperature. Upon completion of the reaction as monitored by TLC, the reaction mixture was quenched with 10 mL of $H_2O$ and washed with saturated aqueous $Na_2S_2O_3$ to remove the excess NIS. The mixture was extracted with 25 mL of ether, washed with 10 mL of water and 10 mL of brine, and dried over anhydrous $Na_2SO_4$. Filtration, evaporation, and flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 40/1) afforded (*S*)-*Z*-**6b** and (*R*)-*E*-**6b** (54.3 mg, 93%, *Z*/*E* = 90/10) as a liquid: $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ [6.31, (t, $J$ = 7.8 Hz, *E*-isomer, 0.1H), 5.90 (t, $J$ = 6.8 Hz, *Z*-isomer, 0.9H)], 4.18 (t, $J$ = 6.5 Hz, 1H), 4.08−3.95 (m, 1H), 3.92−3.80 (m, 1H), 2.21−2.10 (m, 2H), 2.10−1.80 (m, 4H), 1.46−1.22 (m, 6H), 0.88 (t, $J$ = 6.8 Hz, 3H).

**Kinetic Resolution of *Z*,*E*-Isomers of Optically Active (*S*)-(*Z*)-6b, (*R*)-(*E*)-6b. Typical Procedure.** A mixture of $Pd(PPh_3)_2Cl_2$ (4.9 mg, 0.007 mmol), CuI (1.6 mg, 0.008 mmol), $Et_2NH$ (4.4 mg, 0.06 mmol), prop-2-yn-1-ol (3.2 mg, 0.057 mmol), and a mixture of (*S*)-(Z)-**6b**, (*R*)-(*E*)-**6b** (54.3 mg, 0.18 mmol, *Z*/*E* = 90/10) in 2 mL of $CH_3CN$ was stirred at room temperature (20 °C) for 20 min under nitrogen. Filtration, washed by $Et_2O$, evaporation, and flash column chromatography on silica gel (petroleum ether/ethyl acetate = 40:1) afforded (*S*)-*Z*-**6b** (44.1 mg, 81% recovered, *Z*/*E* > 99/1, 95%ee). HPLC conditions: Chiralpak AD-H, rate: 0.5 mL/min, $\lambda$ = 254 nm, *n*-hexane/*i*-PrOH = 99/1: $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 5.90 (t, $J$ = 6.6 Hz, 1H), 4.18 (t, $J$ = 6.3 Hz, 1H), 4.05−3.95 (m, 1H), 3.92−3.81 (m, 1H), 2.21−2.10 (m, 2H), 2.10−1.80 (m, 4H), 1.46−1.22 (m, 6H), 0.88 (t, $J$ = 6.2 Hz, 3H); $[\alpha]^{20}_D$ = +14.0 ($c$ = 0.89, $CHCl_3$).

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**Supporting Information Available:** Experimental procedures, analytical data, and copies of $^1$H and $^{13}$C NMR spectra of 3,4-allenols, 4,5-allenylic nitriles, 4,5-allenoic acids, 4,5-allenols and all 2-(1′(*Z*)-iodoalkenyl)tetrahydrofurans. This material is available free of charge via the Internet at http://pubs.acs.org.

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