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Observations Regarding the Ag(I)-Catalyzed Conversion of Allenones to Furans

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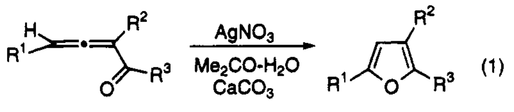
Several years ago we described a new route to furans through Ag(I)-catalyzed isomerization of allenones and allenic aldehydes (eq 1).[1]

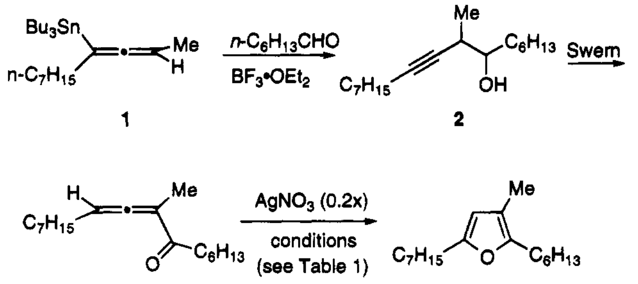


I II

We recently had reason to question the role of water and $CaCO_3$ in this conversion. We were also curious as to possible reaction pathways. We therefore decided to carry out additional studies to clarify these points and possibly develop a more convenient and efficient procedure.

As a starting point we prepared the allenone **3** from allenylstannane **1**.[2] Allenone **3** was selected because it was expected to be relatively nonvolatile and because materials for its preparation are readily available. Addition of stannane **1** to heptanal in the presence of $BF_3{\cdot}OEt_2$ led to the homopropargylic alcohol **2**.[2] Swern oxidation of alcohol **2**[3] followed by brief exposure to additional Et_3N afforded the prototype allenone **3**.

Treatment of **3** with 0.2 equiv of $AgNO_3$ in 3:2 acetone–water in the presence of 0.8 equiv of $CaCO_3$, as previously described,[1] led to the furan **4** in 73% yield after 72 h at rt (Table 1, entry 1). In accord with our suspicions, the reaction proceeded even more efficiently in the absence of $CaCO_3$ (entry 2). Furthermore, a significant rate acceleration was observed with decreasing water content (compare entries 2–4). Furan formation could also be effected in THF as the solvent (entry 5).

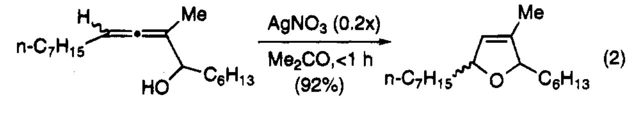


1 2 3 4

Our previously reported conversion of allenylcarbinols to 2,5-dihydrofurans[4] was also more efficient in the absence of $CaCO_3$ and H_2O. The cyclization of alcohol **5**, the reduction product of allenone **3**, to dihydrofuran **6** is illustrative (eq 2). This reaction proceeded in only 73% yield after 31 h of stirring at rt under our reported conditions.[4]

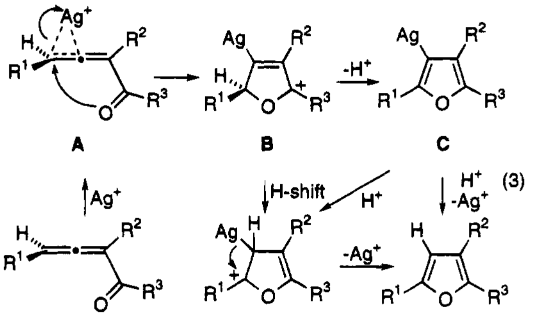
Table 1. Optimization of Allenone–Furan Conversion

entry	condns	t, h	yield, %
1	$Me_2CO–H_2O$ (60:40)/$CaCO_3$	72.0	73
2	$Me_2CO–H_2O$ (75:25)	36.0	84
3	$Me_2CO–H_2O$ (90:10)	4.5	84
4	Me_2CO	<1.0	90
5	THF	3.0	84



5 6

We next addressed the question of reaction pathway. Several possibilities for allenone isomerization are depicted in eq 3. Undoubtedly the process is initiated by coordination of Ag(I) with the allenyl π system as in **A**. Attack by the carbonyl oxygen would lead to the oxo cation **B**. Ensuing proton loss from cation **B** would result in the Ag(I)–furan intermediate **C**. This could undergo direct S_E protonolysis with loss of Ag(I) to afford the furan product **II**. Alternatively, protonolysis of **C** could generate the oxo cation **D** which would undergo E1-type elimination of Ag(I) to give furan **II**. A third possibility entails the formation of oxocation **D** from **B** through 1,2-hydride migration.



A B C I D II

Pathways **B** → **C** → **D** or **II** and **B** → **D** → **II** can be distinguished by a deuterium labeling experiment. An appropriate allenone substrate **13** for this experiment was prepared from enal **7**. Accordingly, Corey–Fuchs–Wittig homologation and subsequent dehydrobromination of the adduct **8** and then debromination of the alkyne **9** followed by D_2O quench of the intermediate lithioalkyne afforded the deuterioalkyne **10** in 91% overall yield.[5] Epoxidation and then S_N' displacement with Bu_3ZnLi led to the deuterated allenylcarbinol **12**, a 72:28 mixture of diastereomers, in high yield.[6,7] Interestingly, when this experiment was carried out on the protonated alkyne (**10**, X = H) the allenol product **12** contained 20–25% of the protonated analogue. In contrast, Bu_3ZnLi additions to propargylic mesylates are reported to incorporate over 90% deuterium upon quenching with D_2O.[6] Evidently

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[1] Marshall, J. A.; Wang, X-j. *J. Org. Chem.* **1991**, *56*, 960.

[2] Marshall, J. A.; Wang, X-j. *J. Org. Chem.* **1990**, *55*, 6246. Marshall, J. A.; Wang, X-j. *J. Org. Chem.* **1992**, *57*, 1242.

[3] Mancuso, A. J.; Huang, S.-L.; Swern, D. *J. Org. Chem.* **1978**, *43*, 2480.

[4] Marshall, J. A.; Wang, X-j. *J. Org. Chem.* **1991**, *56*, 4913.

[5] Corey, E. J.; Fuchs, P. L. *Tetrahedron Lett.* **1972**, 3769. Nicolaou, K. C.; Prasad, C. V. C.; Somers, P. K.; Hwang, C.-K. *J. Am. Chem. Soc.* **1989**, *111*, 5330.

[6] This reaction has previously been employed with propargylic chlorides and mesylates: Katsuhira, T.; Harada, T.; Maejima, K.; Osada, A.; Oku, A. *J. Org. Chem.* **1993**, *58*, 6166.

[7] For previously reported additions of cuprates to alkynyloxiranes, see: Marshall, J. A.; Pinney, K. G. *J. Org. Chem.* **1993**, *58*, 7180.

0022-3263/94/1959-7169$04.50/0

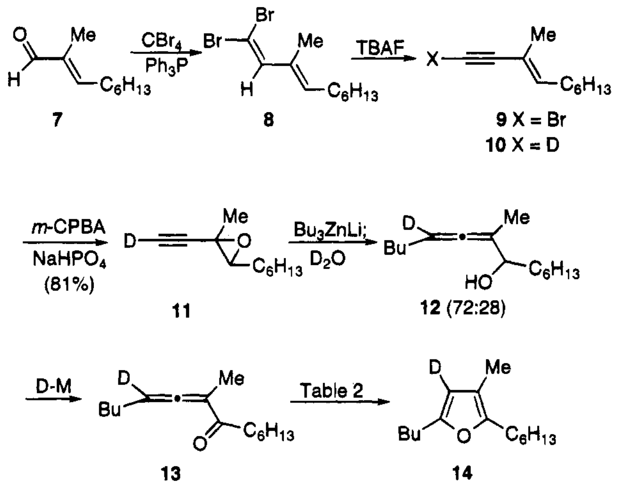
Table 2. Deuterium Incorporation in Allenone–Furan Conversion[a]

entry	D:H in **13**	solvent	yield, %	D:H in **14**
1	91:9	Me_2CO	92	50:50
2	91:9	Me_2CO-H_2O[b]	91	22:78
3	91:9	Me_2CO-D_2O[b]	88	95:5
4	0:100	$(CD_3)_2CO$	91	5:95
5	0:100	Me_2CO-D_2O[a]	92	72:28

[a] See Experimental Section for representative procedure. [b] 95:5 mixture.

the present addition proceeds to some extent by direct S_N2' displacement or sequential addition–elimination.

Dess–Martin oxidation[8] of alcohol **12** afforded the deuterated allenone **13**. Upon treatment with $AgNO_3$ in anhydrous acetone, allenone **13** was converted to furan **14** in 92% yield (Table 2, entry 1). Nearly half of the deuterium was lost in this conversion. Addition of 5% H_2O to the reaction caused even more extensive deuterium loss (entry 2). Conversely, when 5% D_2O was added, the furan product **14** showed a slightly enhanced deuterium content relative to the starting allenone (entry 3). Control experiments indicated that water is the most likely proton (deuteron) source in these conversions (entries 4 and 5). Furthermore, furan **14** and allenone **13** (recovered) showed negligible incorporation or loss of deuterium upon treatment with $AgNO_3$ in acetone–H_2O or D_2O under the reaction conditions. In all cases, deuterium incorporation was established by ^1H NMR analysis and verified by MS data.



The foregoing experiments show that the hydride-shift pathway **B** → **D** → **II** is of minor importance in the furan-forming reaction. Presumably trace contamination of the acetone solvent by H_2O accounts for the significant loss of deuterium shown in entry 1 of Table 2. Acetone itself could also afford some of the protonated product, as indicated by entry 4.

In addition to shedding light on the pathway of the allenone–furan conversion, the foregoing experiments demonstrate a simple route to specifically labeled 3-deuteriofurans. Such derivatives would not be readily accessible from the furans themselves. On the basis of our previous findings, the approach should be applicable to various mono- and disubstituted furans as well as the case at hand.[1]

Experimental Section[9]

8-Methyl-8,9-heptadecadien-7-one (3). To a solution of 98 μL (1.13 mmol) of oxalyl chloride in 2.0 mL of CH_2Cl_2 under nitrogen at −78 °C was added 160 μL (2.25 mmol) of DMSO dropwise. After 15 min, 100 mg (0.375 mmol) of homopropargylic alcohol **2**[2] in 1.0 mL of CH_2Cl_2 was added dropwise, and then after 15 min 1.23 mL (8.85 mmol) of Et_3N was added; the resulting white mixture was stirred at rt. After 16 h, infrared analysis of an aliquot showed complete isomerization to allenone **3**. Ether and water were added, and the aqueous layer was separated and extracted three times with ether. The combined organic solutions were extracted three times with 10% aqueous HCl, washed with saturated $NaHCO_3$ and brine, dried over $MgSO_4$, and concentrated. Flash chromatography on silica gel (hexanes:ether, 30:1) provided allenone **3** (85 mg, 86%): IR (film) ν 1947, 1678 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.46 (tq, J = 6.9, 2.7 Hz, 1H), 2.58 (dd, J = 7.9, 7.2 Hz, 2H), 2.12 (dt, 7.8, 6.9 Hz, 2H), 1.74 (d, J = 2.7 Hz, 3H), 1.56–1.50 (m, 2H), 1.48–1.41 (m, 2H), 1.34–1.20 (m, 14H), 0.86 (t, J = 6.8 Hz, 3H), 0.85 (t, J = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 211.8, 202.2, 104.0, 94.2, 38.8, 31.8–28.2 (seven lines), 25.2, 22.6, 22.5, 14.0–13.5 (three lines). Anal. Calcd for $C_{18}H_{32}O$: C, 81.75; H, 12.20. Found: C, 81.60; H, 12.19.

5-Heptyl-2-hexyl-3-methylfuran (4). To 100 mg (0.378 mmol) of allenone **6** in 3.0 mL of acetone under nitrogen was added 14.1 mg (83.2 μmol) of $AgNO_3$. After refluxing under nitrogen for 30 min, the yellow mixture was cooled to rt and a spatula tip of Na_2CO_3 was added, followed by water and ether. The aqueous layer was separated and extracted three times with ether. The combined organic solutions were washed with brine, dried over $MgSO_4$, and concentrated. Flash chromatography on silica gel (hexanes) afforded furan **4** (92 mg, 92%) as a clear oil: IR (film) ν 1574, 1463, 1378 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.71 (s, 1H), 2.49 (m, 4H), 1.88 (s, 3H), 1.59–1.50 (m, 4H), 1.29–1.26 (m, 14H), 0.88–0.85 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.5, 149.4, 113.8, 107.6, 31.8, 31.7, 29.2, 29.1, 28.9–28.1 (four lines), 25.9, 22.6 (two lines), 14.1, 9.9. Anal. Calcd for $C_{18}H_{32}O$: C, 81.75; H, 12.20. Found: C, 81.62; H, 12.08.

8-Methyl-8,9-heptadecadien-7-ol (5). To 773 mg (2.92 mmol) of allenone **3** in 10.0 mL of THF under nitrogen at −78 °C was added 1.0 M DIBALH in hexanes (3.50 mL, 3.50 mmol) dropwise. After 20 min, the solution was quenched with saturated Rochelle's salt and warmed to rt. The mixture was diluted with water, and the aqueous layer was separated and extracted three times with ether. The combined organic solutions were washed with brine, dried over $MgSO_4$, and concentrated. Flash chromatography on silica gel (hexanes:ether, 6:1) provided allenol **5** (628 mg, 81%) as a 38:62 mixture of diastereomers (by GC analysis): IR (film) ν 3340, 1964 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 5.16 (m, 1H), 3.97 (m, 1H), 1.95 (m, 2H), 1.66–1.64 (m, 3H), 1.66–1.25 (m, 21H); ^{13}C NMR (75 MHz, $CDCl_3$) δ major diastereomer 200.0, 102.6, 93.0, 73.0, 35.1, 31.9, 29.3–29.1 (five lines), 25.5, 22.6 (two lines), 14.1; minor diastereomer, partial 199.5, 103.1, 93.8, 72.5, 35.3, 25.3. Anal. Calcd for $C_{18}H_{34}O$: C, 81.13; H, 12.86. Found: C, 80.88; H, 12.79.

***cis*- and *trans*-5-Heptyl-2-hexyl-3-methyl-2,5-dihydrofurans (6).** To 48.3 mg (0.181 mmol) of allenol **5** in 1.4 mL of acetone was added 6.8 mg (40 μmol) of $AgNO_3$, and the resulting mixture was stirred in the dark under nitrogen at rt for 3 h. The mixture was filtered through a pad of Celite with ether. The filtrate was concentrated and chromatographed on silica gel (hexanes:ether, 40:1–30:1) to afford dihydrofuran **6** (more polar, 16.2 mg, 34%; less polar, 28.0 mg, 58%).

More polar: IR (film) ν 1668, 1091 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.36 (d, J = 1.6 Hz, 1H), 4.64 (brs, 1H), 4.54 (brs, 1H), 1.65 (d, J = 1.1 Hz, 3H), 1.60–1.27 (m, 22H), 0.86 (t, J = 6.7 Hz, 3H), 0.86 (t, J = 6.7 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.9, 124.8, 87.5, 85.3, 37.6, 35.2, 32.2 (two lines), 30.1, 29.9, 29.6, 26.0, 25.4, 23.0 (two lines), 14.5, 12.9. Anal. Calcd for $C_{18}H_{34}O$: C, 81.13; H, 12.86. Found: C, 80.98; H, 12.78.

Less polar: IR (film) ν 1669, 1089 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.37 (d, J = 1.5 Hz, 1H), 4.70 (m, 1H), 4.61 (m, 1H), 1.65 (d, 0.9 Hz, 3H), 1.61–1.26 (m, 22H), 0.85 (t, J = 6.6 Hz, 3H), 0.85 (t, J = 6.6 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.6, 124.7, 87.5, 85.5, 37.0, 34.5, 32.3, 32.2, 30.2, 30.0 (two lines),

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(8) Dess, D. B.; Martin, J. C. *J. Org. Chem.* **1983**, *48*, 4156.

(9) For typical experimental protocols, see ref 4.

25.6, 24.9, 23.0, 14.4, 12.9. Anal. Calcd for $C_{18}H_{34}O$: C, 81.13; H, 12.86. Found: C, 80.92; H, 12.76.

(*E*)-1,1-Dibromo-3-methyl-1,3-decadiene (8). To 24.0 g (91.5 mmol) of Ph_3P in 100 mL of CH_2Cl_2 at rt under nitrogen was added 15.1 g (45.7 mmol) of CBr_4 in one portion causing an exothermic reaction and a red coloration. After being cooled to rt, the solution was cooled to 0 °C and 3.53 g (22.9 mmol) of aldehyde **7** in 40 mL of CH_2Cl_2 was added over a period of 4 min. After 25 min, 15 mL (106 mmol) of Et_3N was added, followed by partial concentration of the mixture under reduced pressure by means of a rotary evaporator. The resulting thick brown slurry was filtered through deactivated silica gel with hexanes. (The silica was deactivated with 50% Et_3N in hexanes.) The filtrate was concentrated to afford a yellow oil containing white Ph_3PO solids. This residue was twice rinsed with hexanes, filtered through scintered glass, and concentrated to provide a yellow oil (7.78 g), which was used without further purification: IR (film) ν 1618, 1456, 897, 839 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 6.92 (s, 1H), 5.61 (tt, J = 6.1, 1.3 Hz, 1H), 2.04 (dt, J = 7.2, 7.1 Hz, 2H), 1.84 (d, J = 0.76 Hz, 3H), 1.36−1.25 (m, 8H), 0.87 (t, J = 6.8 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 141.3, 136.6, 131.8, 85.2, 32.2, 29.4−28.6 (three lines), 23.1, 15.6, 14.5.

(*E*)-1-Bromo-3-methyl-3-decen-1-yne (9). To a solution of 7.10 g (*ca.* 22.9 mmol) of the crude geminal dibromide **8** in 120 mL of THF at rt under nitrogen was added 69 mL (69 mmol) of 1.0 M TBAF in THF, thus darkening the solution. After 45 min, ether and water were added, the aqueous layer was separated and extracted three times with ether, and the combined organic solutions were washed with brine, dried over $MgSO_4$, filtered, and concentrated to provide a brown residue (9.3 g) consisting of an 11:89 mixture (by ^1H NMR analysis) of starting geminal dibromide **8** and bromo enyne **9**. Flash chromatography of the brown residue on silica gel (hexanes:ether, 40:1) afforded bromo enyne **9** (5.78 g), which was used without further purification: IR (film) ν 2187, 1466, 1378, 724 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 5.88 (tq, J = 7.5, 1.5, 1H), 2.04 (dt, J = 7.1, 6.9 Hz, 2H), 1.74 (d, J = 1.4 Hz, 3H), 1.34−1.24 (m, 8H), 0.86 (t, J = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.9, 117.3, 83.1, 45.2, 31.7, 28.9−28.4 (three lines), 22.6, 16.7, 14.1.

(*E*)-1-Deuterio-3-methyl-3-decen-1-yne (10). To 4.93 g (21.5 mmol) of bromo enyne **9** in 90 mL of THF under nitrogen at −78 °C was added 9.5 mL (23.7 mmol) of 2.5 M *n*-BuLi in hexanes. After 50 min, a 25 mL aliquot was transferred to a separate flask for H_2O quench. The remaining solution was quenched with 2.5 mL of D_2O and then warmed to rt. Brine and ether were added, the aqueous layer was separated and extracted three times with ether, and the combined organic solutions were washed with brine, dried over $MgSO_4$, and concentrated. Flash chromatography on silica gel (pentane), followed by concentration of the product-containing fractions under reduced pressure by means of a rotary evaporator at 30 °C for 30 min, afforded the volatile deuterated enyne **10** (2.23 g, 91% adjusted yield) as a colorless mobile liquid (D:H, 97:3 by ^1H NMR): IR (film) ν 2589, 1633 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.93 (tq, J = 7.4, 1.5 Hz, 1H), 2.06 (m, 2H), 1.77 (dt, J = 1.3, 0.7 Hz, 3H), 1.37−1.23 (m, 8H), 0.86 (t, J = 6.9 Hz, 3H); a minor acetylenic proton signal was observed at 2.73 (s); ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.9, 116.6, 86.4 (t, J = 7.5 Hz), 73.0 (t, J = 38.2 Hz), 31.7, 28.9−28.4 (three lines), 22.6, 16.9, 14.0.

(*E*)-1-Deuterio-3,4-epoxy-3-methyl-1-decyne (11). To a well-stirred mixture of 1.63 g (10.8 mmol) of enyne **10** and 4.09 g of Na_2HPO_4 in 100 mL of CH_2Cl_2 under nitrogen at rt was added 4.09 g (11.9 mmol, assuming 50% *m*-CPBA) of 50−60% *m*-CPBA−*m*-chlorobenzoic acid in one portion. After being stirred vigorously for 1 h, the mixture was carefully quenched with saturated $NaHCO_3$. Ether was added, the aqueous layer was separated and extracted three times with ether, and the combined organic solutions were extracted with saturated $NaHCO_3$, washed with brine, dried over $MgSO_4$, and concentrated. Flash chromatography on silica gel (hexanes:ether, 15:1) gave 1.56 g (87%) (D:H, 98:2 by ^1H NMR) of deuterated alkynyloxirane **11**: IR (film) ν 2595, 1978, 1227, 1073 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.02−2.99 (m, 1H, 1.43−1.41 (m, 2H), 1.38 (d, J = 3.2 Hz, 3H), 1.36−1.21 (m, 8H), 0.81−0.78 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ .84.1 (t, 7.4 Hz), 69.5 (t, J = 38.5 Hz), 64.4, 50.2, 31.6, 28.9, 28.0, 26.0, 22.4, 18.1, 13.9.

5-Deuterio-7-methyl-5,6-tetradecadien-8-ol (12). To 3.20 g (23.3 mmol) of $ZnCl_2$ (fused *in vacuo*) in 60 mL of THF under nitrogen at 0 °C was added 28.0 mL (70.0 mmol) of 2.5 M *n*-BuLi in hexanes. After 30 min, the solution was cooled to −78 °C, and 1.56 g (9.33 mmol) of alkynyloxirane **11** in 20 mL of THF was added dropwise. The solution was warmed to 0 °C for 10 min, recooled to −78 °C, and then quenched with 2.2 mL of D_2O. After the mixture was stirred under nitrogen at rt for 24 h, ether and water were added, the mixture was acidified with 10% HCl, and the aqueous layer was separated and extracted three times with ether. The combined organic solutions were washed with brine, dried over $MgSO_4$, and concentrated. Flash chromatography on silica gel (hexanes:ether, 4:1) afforded deuterated allenol **12** (1.68 g, 80%) (D:H, 88:12 by ^1H NMR) as a mixture of diastereomers: IR (film) ν 3333, 1957 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.96−3.92 (m, 1H), 1.94 (t, J = 6.5 Hz, 2H), 1.79 (brs, 1H), 1.63 (s, 3H), 1.56−1.24 (m, 14H), 0.84 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ major diastereomer (**12a**) 199.6, 103.0, 93.3 (t, J = 19.4 Hz), 72.5, 35.3, 31.8, 31.3, 29.2, 28.7, 25.3, 22.6, 22.1, 14.0, 13.8; minor diastereomer (**12b**), partial: 200.0, 102.7, 73.0, 35.0, 25.4.

5-Deuterio-7-methyl-5,6-tetradecadien-8-one (13). To 1.00 g (4.44 mmol) of allenol **12** in 50 mL of CH_2Cl_2 at rt under nitrogen was added 2.80 g (6.70 mmol) of the Dess−Martin periodinane.[3] The mixture was quenched after 1.5 h with saturated $NaHCO_3$, and the aqueous layer was separated and extracted three times with ether. The combined organic solutions were washed with brine, dried over $MgSO_4$, and concentrated. Flash chromatography on silica gel (hexanes:ether, 30:1) gave deuterated allenone **13** (755 mg, 76%) as a yellow oil (D:H, 91:9 by MS): IR (film) ν 1940, 1678 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.58 (dd, J = 7.6, 7.4 Hz, 2H), 2.12 (dd, J = 7.3, 6.8 Hz, 2H), 1.74 (s, 3H), 1.59−1.25 (m, 12H), 0.90 (t, J = 7.1 Hz, 3H), 0.85 (t, J = 6.9 Hz, 3H); a minor allenic proton signal was observed at 5.46 (m); ^{13}C NMR (101 MHz, $CDCl_3$) δ 211.9, 202.4, 104.1, 93.9 (t, J = 24.9 Hz), 38.8, 31.6, 30.0, 27.8, 25.2, 22.5, 22.2, 14.0, 13.7, 13.5; MS *m/z* (rel inten) 223 (M^+, 3), 152 (15), 113 (100), 85 (56).

Representative Procedure for Table 2: 4-Deuterio-5-heptyl-2-hexyl-3-methylfuran (14). To deuterated or non-deuterated allenone **13** (84.4 mg or 84.1 mg, respectively; 0.378 mmol) in the solvent system specified (3.0 mL) was added $AgNO_3$ (14.1 mg, 83.2 μmol). After being refluxed for 30 min, the mixture was cooled to rt and a spatula tip of Na_2CO_3 was added. Water and ether were added, and the aqueous layer was separated and extracted three times with ether. The combined organic solutions were washed with brine, dried over $MgSO_4$, and concentrated. Flash chromatography on silica gel (hexanes) afforded furan **14** in the yields specified: IR (film) ν 1574, 1466, 959, 797 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.73 (s, <1H depending on deuterium content), 2.52 (t, J = 7.6 Hz, 2H), 2.49 (t, J = 7.4 Hz, 2H), 1.89 (s, 3H), 1.61−1.52 (m, 4H), 1.40−1.28 (m, 8H), 0.93−0.86 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.5, 153.4, 149.4, 113.8, 113.7, 107.4 (t, J = 24 Hz), 31.7, 30.3, 28.9, 28.8, 27.7, 25.9, 22.7, 22.3, 14.1, 13.9, 9.9, 9.8; MS *m/z* (rel inten) 223 (M^+, 13), 180 (19), 152 (100).

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Supplementary Material Available: ^1H NMR spectra of **8−14** and ^{13}C NMR spectra of **10** and **11** (11 pages). This material is contained in libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.