

PREPARATION OF FURANS FROM ALKYNOLS UTILIZING PALLADIUM CATALYZED
INTRAMOLECULAR ADDITION OF ALCOHOL TO ACETYLENE AS A KEY REACTION

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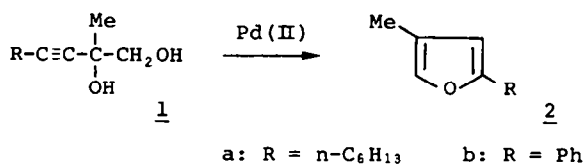
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Abstract - A procedure is described for the synthesis of furans from 3-alkyn-1,2-diols or 2-methoxy-3-alkyn-1-ols by palladium catalyzed intramolecular addition of alcoholic moiety to acetylene linkage followed by elimination of water or methanol. The intermediary 3-furylpalladiums can be trapped with allyl halides affording 3-allylfurans in good yields.

Since Heilbron and coworkers reported the preparation of 2,5-dimethylfuran from 3-hexen-5-yn-1-ol by the catalytic action of mercuric sulphate, substituted furans have been synthesized from acetylenic intermediates.¹⁻¹⁴ Recently 3-furylmercurials were prepared from 3-alkyn-1,2-diols by the action of stoichiometric mercuric chloride.^{15,16} In addition to the formation of 2,3,5-trisubstituted furans by protonolysis with diluted hydrochloric acid, 3-furylmercurials afforded methyl 3-furoates by palladium promoted carbonylation in methanol.¹⁵⁻¹⁷ Though these reports show that 3-alkyn-1,2-diols are useful precursors not only for 2,3,5-trisubstituted furans but for 3-furoic acid derivatives, both mercuric and palladium salts are needed stoichiometrically. Palladium catalysts have been found to be effective for the intramolecular addition of an amine, an alcohol, or a carboxylic acid to an acetylene.¹⁸⁻²⁰ These observations prompted us to reinvestigate the synthesis of furans from acetylenic alcohols under the catalytic action of palladium salts or complexes. This paper describes in detail a facile preparation of substituted furans from 3-alkyn-1,2-diols or 2-methoxy-3-alkyn-1-ols by the catalytic action of palladium(II) species. Trap of the intermediary 3-furylpalladiums with allyl halides producing 3-allylfurans and recycling Pd(II) species is also disclosed.

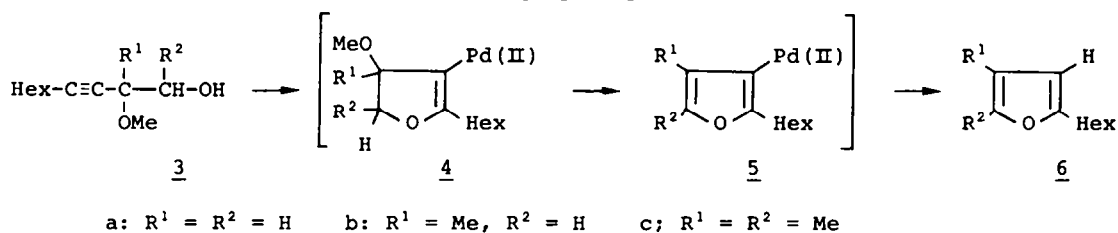
Preparation of furans from 2-methyl-3-alkyn-1,2-diols was examined first. Suitable combinations of catalysts and solvents were surveyed for the preparation of 2-hexyl-4-methylfuran (**2a**) from 2-methyl-3-decyn-1,2-diol (**1a**). Treatment of **1a** with 0.01 equiv. of PdCl₂(PhCN)₂ in THF gave furan **2a** in excellent yield. On the other hand, starting material **1a** was recovered after treatment with Pd(Ph₃P)₄. Results are summarized in Table 1. Utilizing PdCl₂(MeCN)₂ in place of PdCl₂(PhCN)₂ gave furan **2a** in good yield. Analogous treatment of 2-methyl-4-phenyl-3-butyne-1,2-diol (**1b**) with PdCl₂(PhCN)₂ gave 4-methyl-2-phenylfuran (**2b**)²¹ in 82% yield.



Scheme 1

Table 1. 2-Hexyl-4-methylfuran (2a) from 2-Methyl-3-decyn-1,2-diol (1a)

Entry	Catalyst (equiv.)	Solvent	Reaction		Yield (%)
			Temp (°C)	Time (h)	
1	PdCl ₂ (PhCN) ₂ (0.01)	THF	r.t.	10	85 ^a
2	PdCl ₂ (0.05)	MeCN	r.t.	6	60
3	Pd(OAc) ₂ (0.05) + Ph ₃ P (0.2)	THF	r.t.	10	20
4	Pd(Ph ₃ P) ₄ (0.01)	MeCN	r.t.	10	0

^a More than 98% yield before chromatographic purification.

Scheme 2

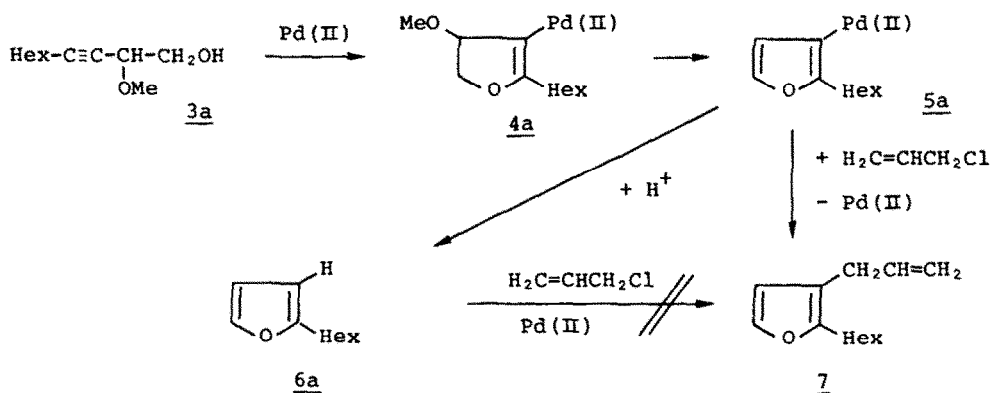
Table 2. Preparation of Furan 6 from 2-Methoxy-3-alkyn-1-ol 3

Entry	Starting material	Solvent	Additive	Reaction		Product ^a	Yield (%)
				Temp.	Time (h)		
1	<u>3a</u>	THF	dil. HCl	r.t.	24	<u>6a</u>	90
2	<u>3a</u>	THF	H ₂ O	r.t.	10		0
3	<u>3a</u>	THF	—	r.t.	10		0
4	<u>3b</u>	THF	H ₂ O	r.t.	5	<u>6b</u>	80
5	<u>3b</u>	THF	—	r.t.	10		0
6	<u>3b</u>	MeCN	—	reflux	10	<u>6b</u>	27
7	<u>3c</u>	THF	H ₂ O	r.t.	5	<u>6c</u>	94
8	<u>3c</u>	MeCN	H ₂ O	r.t.	5	<u>6c</u>	94
9	<u>3c</u>	MeCN	—	reflux	10	<u>6c</u>	24

^a Compound 6a was reported in ref. 22.

As can be seen from Scheme 1, hydroxyl group on C-2 could be substituted with other leaving group. Methoxyl was chosen in place of hydroxyl by the consideration of easy access to the starting material (vide infra). In contrast to the cases of diol 1, formation of furans from 2-methoxy-3-alkyn-1-ols (3) was slow without any additive to the reaction mixture. Addition of a trace amount of water or dil. HCl improved the yields of furan 6. Results are summarized in Table 2.

The above described preparation of furans from acetylenic precursors could be explained as shown in Scheme 2. When organopalladiums 4 or 5 could be trapped with allyl chloride,^{23,24} allyl group would be introduced regioselectively. Oxidation state of palladium catalyst seems unchanged during carbodemetalation. By this assumption cyclization and carbodemetalation of a mixture of alkynols and allyl halides are expected by the action of catalytic amount of palladium(II). A solution of 2-methoxy-3-decyn-1-ol (3a) in 60 equiv. of allyl chloride was treated with 0.05 equiv. of $\text{PdCl}_2(\text{MeCN})_2$ for 40 min at room temperature affording a mixture of 3-allyl-2-hexylfuran (7, 50% yield) and 2-hexylfuran (6a, 47%). When 2-hexylfuran (6a) was treated with allyl chloride in the presence of $\text{PdCl}_2(\text{MeCN})_2$, the starting material 6a was recovered and allylated product 7 could not be detected. The formation of allylfuran 7 could be explained by the coupling of the intermediary organopalladium species 5a with allyl chloride (Scheme 3). Yields of the products are improved by the addition of oxiranes to the reaction mixture as proton scavenger. Combination of the starting materials, allyl halides, oxiranes and product distribution are summarized in Table 3. Thus tetrasubstituted furans are prepared in good yields by the above method.

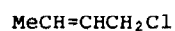
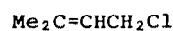
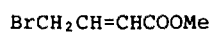
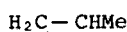
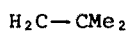
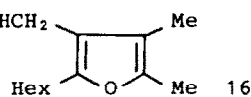
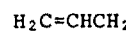
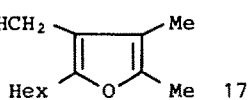
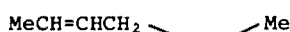
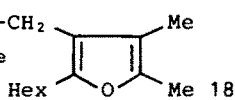
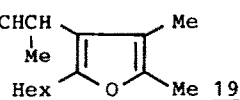


Scheme 3

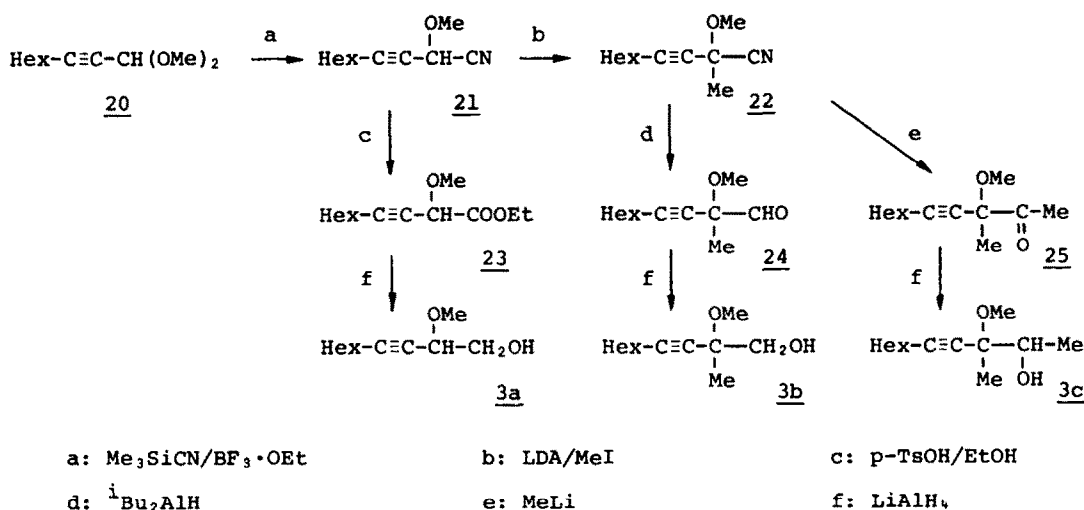
Allyl halides react selectively on γ carbon and crotyl and 1-methyl-2-propyl groups are introduced by the coupling with 3-chloro-1-butene and 1-chloro-2-butene, respectively. These regioselective coupling of allyl halides could be explained by the addition of organopalladium to double bond followed by the elimination of palladium chloride.

Table 3. 3-Allylfurans from 2-Methoxy-3-alkyn-1-ols and Allyl Halides^{a,b}

Entry	Methoxy-alkynol	Halide (equiv.)	Oxirane (equiv.)	Product (% yield)
				Allylated Protonated
1	<u>3a</u>	<u>8</u> (60)	—	<u>7</u> (50) <u>6a</u> (47)
2	<u>3a</u>	<u>8</u> (10)	<u>14</u> (50)	<u>7</u> (68) <u>6a</u> (15)
3	<u>3a</u>	<u>8</u> (10)	<u>15</u> (50)	<u>7</u> (85) <u>6a</u> (5)
4	<u>3a</u>	<u>8</u> (2)	<u>15</u> (5)	<u>7</u> (55) <u>6a</u> (1)
5	<u>3a</u>	<u>8</u> (2)	<u>15</u> (5)	<u>7</u> (63) <u>6a</u> (27) ^c
6	<u>3c</u>	<u>8</u> (60)	—	<u>16</u> (39) <u>6c</u> (4)
7	<u>3c</u>	<u>8</u> (10)	<u>14</u> (50)	<u>16</u> (64) <u>6c</u> (8)
8	<u>3c</u>	<u>8</u> (10)	<u>15</u> (50)	<u>16</u> (79) <u>6c</u> (6)
9	<u>3c</u>	<u>9</u> (10)	<u>15</u> (50)	<u>17</u> (70) ^d <u>6c</u> (5)
10	<u>3c</u>	<u>10</u> (10)	<u>15</u> (50)	<u>18</u> (58) <u>6c</u> (10)
11	<u>3c</u>	<u>11</u> (10)	<u>15</u> (50)	<u>19</u> (48) <u>6c</u> (12)
12	<u>3c</u>	<u>12</u> (10)	<u>15</u> (50)	<u>6c</u> ^e
13	<u>3c</u>	<u>13</u> (10)	<u>15</u> (50)	<u>6c</u> ^e

^a Utilizing 0.05 equiv. of PdCl₂(MeCN)₂ as catalyst.^b Compounds 8 - 19 shown in the Table 3:8910111213141516171819^c THF (2 ml) was used as solvent.^d Ratio of E/Z = 3/1.^e Trace amount of 6c was obtained and starting material was recovered.

Preparation of 2-methoxy-3-decyn-1-ols, starting materials of furan synthesis, is described lastly. Treatment of acetylenic acetal 20 with cyanotrimethylsilane gave methoxynitrile 21 which was utilized as the intermediate for the preparation of alkynols.²⁵ 2-Methoxy-3-decyn-1-ol (3a) was prepared from 21 by transformation of cyano into carbethoxy which was reduced to hydroxymethyl group. Methyl group was introduced by treatment with LDA and iodomethane affording 22 from 21. 2-Methyl-2-methoxy-3-decyn-1-ol (3b) was prepared from 22 by transformation of cyano into hydroxymethyl. 3-Methoxy-3-methyl-4-undecyn-2-ol (3c) was derived from 22 via ketone which was obtained by the action of methyl lithium (Scheme 4).



Scheme 4

In conclusion various types of substituted furans can be prepared from easily accessible acetylenic intermediates in good to excellent yields by utilizing palladium catalyzed intramolecular addition of alcohol to acetylene as a key reaction. Trap of the intermediary 3-furylpalladiums with allyl halides provides a novel strategy of introducing allyl group on C-3 position. This procedure opens a convenient method of constructing tetrasubstituted furans.

EXPERIMENTAL

IR spectra were obtained on a JASCO IR-810 spectrometer. $^1\text{H-NMR}$ (200 MHz) were measured on a Varian XL-200. Mass spectra were obtained on a Hitachi M-80 spectrometer. Elemental analyses were performed at Elemental Analyses Center of Kyoto University.

Preparation of acetylenic diol 1: To a THF solution of 1-lithio-1-alkyn prepared from 1-alkyne (50 mmol) and butyllithium (55 mmol in 35 mL of hexane) in 80 mL of THF was added 1-tetrahydropyranyloxy-2-propanone (9.48 g, 60 mmol) at 0°C and the whole was stirred for 2 h. Hydrolytic work up of the reaction mixture gave coupling product which was treated with catalytic amount of p-toluenesulfonic acid in methanol. The reaction mixture was diluted with ether and ethereal solution was washed with brine, dried (Na_2SO_4) and concentrated.

Preparation of furan 2 from 2-methyl-3-alkyn-1,2-diol 1: To a solution of $\text{PdCl}_2(\text{PhCN})_2$ (7 mg, 0.02 mmol) in 5 mL of THF was added acetylenic diol 1 (2 mmol in 1 mL of THF) at room temperature and the resulting mixture was stirred for 10 h. The reaction mixture was worked up with brine and the whole was extracted with ether. Ethereal solution was dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography (silica gel).

Preparation of furan 6 from 2-methoxy-3-alkyn-1-ol 3: To a solution of $\text{PdCl}_2(\text{PhCN})_2$ (26 mg, 0.1 mmol) in 5 mL of THF or acetonitrile were successively added 0.5 mL of additive and alkynol 3 (2 mmol) and the resulting mixture was stirred at room temperature for 5-24 h. The reaction mixture was worked up with sat. NH_4Cl and extracted with ether. Column chromatography (silica gel) of the concentrate gave the product 6.

2-Hexyl-4-methylfuran (2a): $^1\text{H-NMR}$ (CDCl_3), δ 0.88 (3H, t, $J = 6.5$ Hz), 1.27-1.56 (8H, m), 1.98 (3H, d, $J = 1.1$ Hz), 2.55 (2H, t, $J = 7.8$ Hz), 5.84 (1H, s), 7.06 (1H, t, $J = 1.1$ Hz). IR (neat), 1620, 1555, 1120 cm^{-1} . Anal. Calcd for $\text{C}_{11}\text{H}_{18}\text{O}$: C, 79.74; H, 10.92. Found: C, 79.46; H, 10.92.

5-Hexyl-2,3-dimethylfuran (6c): $^1\text{H-NMR}$ (CDCl_3), δ 0.88 (3H, t, $J = 6.6$ Hz), 1.26-1.55 (8H, m), 1.89 (3H, s), 2.16 (3H, s), 2.51 (2H, t, $J = 7.6$ Hz), 5.74 (1H, s). IR (neat), 1650, 1580, 1230 cm^{-1} . Anal. Calcd for $\text{C}_{12}\text{H}_{20}\text{O}$: C, 79.94; H, 11.18. Found: C, 80.11; H, 11.38.

Preparation of 3-allylfuran (7, 16, 17, 18, and 19) from 2-methoxy-3-alkyn-1-ol (3a and 3c): To a solution of methoxyalkynol 3 in allyl halide (10 equiv.) and oxirane (14 or 15, 50 equiv.), $\text{PdCl}_2(\text{MeCN})_2$ (0.05 equiv.) was added and the reaction mixture was stirred at room temperature for 3 h. Product was obtained by column chromatography (silica gel) of the concentrated reaction mixture.

3-Allyl-2-hexylfuran (7): $^1\text{H-NMR}$ (CDCl_3), δ 0.92 (3H, t, $J = 6.0$ Hz), 1.20–1.60 (8H, m), 2.57 (2H, t, $J = 7.4$ Hz), 3.10 (2H, dt, $J = 6.2, 1.6$ Hz), 5.02 (1H, ddt, $J = 3.6, 16.9, 1.6$ Hz), 5.90 (1H, ddt, $J = 9.8, 16.9, 6.2$ Hz), 6.18 (1H, d, $J = 2.4$ Hz), 7.27 (1H, d, $J = 2.4$ Hz). IR (neat), 1640, 1512, 1466, 1436 cm^{-1} . Anal. (accurate mass) Calcd for $\text{C}_{13}\text{H}_{20}\text{O}$: 192.1513. Found: 192.1540.

3-Allyl-2-hexyl-4,5-dimethylfuran (16): $^1\text{H-NMR}$ (CDCl_3), δ 0.88 (3H, t, $J = 6.0$ Hz), 1.20–1.65 (8H, m), 1.75 (3H, s), 2.16 (3H, s), 2.46 (2H, t, $J = 6.3$ Hz), 3.04 (2H, dt, $J = 6.2, 1.6$ Hz), 4.95 (1H, m), 5.02 (1H, m), 5.85 (1H, m). IR (neat) 1637, 1597, 1457, 1437, 1385, 1260 cm^{-1} . Anal. (accurate mass) Calcd for $\text{C}_{15}\text{H}_{24}\text{O}$: 220.1826. Found: 220.1837.

3-(2-(E)-Butenyl)-2-hexyl-4,5-dimethylfuran (17a): $^1\text{H-NMR}$ (CDCl_3), δ 0.88 (3H, t, $J = 6.0$ Hz), 1.15–1.60 (8H, m), 1.64 (3H, dq, $J = 3.0, 1.5$ Hz), 1.82 (3H, s), 2.15 (3H, s), 2.47 (2H, t, $J = 6.5$ Hz), 2.90–2.98 (2H, m), 5.39–5.47 (2H, m). IR (neat) 1599, 1452, 1438 cm^{-1} . Anal. (accurate mass) Calcd for $\text{C}_{16}\text{H}_{26}\text{O}$: 234.1982. Found: 234.2004.

3-(2-(Z)-Butenyl)-2-hexyl-4,5-dimethylfuran (17b): $^1\text{H-NMR}$ (CDCl_3), δ 0.88 (3H, t, $J = 6.0$ Hz), 1.15–1.65 (8H, m), 1.73 (3H, d, $J = 4.4$ Hz), 1.84 (3H, s), 2.15 (3H, s), 2.52 (2H, t, $J = 6.4$ Hz), 3.04 (2H, d, $J = 6.0$ Hz), 5.30–5.62 (2H, m). IR (neat), 1600, 1445, 1260 cm^{-1} . Anal. (accurate mass) Calcd for $\text{C}_{16}\text{H}_{26}\text{O}$: 234.1982. Found: 234.1766.

3-(2-Methyl-2-propenyl)-2-hexyl-4,5-dimethylfuran (18): $^1\text{H-NMR}$ (CDCl_3), δ 0.88 (3H, t, $J = 6.0$ Hz), 1.15–1.65 (8H, m), 1.70 (3H, s), 1.79 (3H, s), 2.15 (3H, s), 2.47 (2H, t, $J = 6.2$ Hz), 2.95 (2H, s), 4.67 (2H, dq, $J = 18.0, 1.2$ Hz). IR (neat), 1649, 1597, 1448, 1375 cm^{-1} . Anal. (accurate mass) Calcd for $\text{C}_{16}\text{H}_{26}\text{O}$: 234.1982. Found: 234.2007.

3-(1-Methyl-2-propenyl)-2-hexyl-4,5-dimethylfuran (19): $^1\text{H-NMR}$ (CDCl_3), δ 0.88 (3H, t, $J = 6.0$ Hz), 1.15–1.66 (8H, m), 1.30 (3H, s), 1.86 (3H, s), 2.15 (3H, s), 2.52 (2H, t, $J = 6.0$ Hz), 3.25–3.45 (1H, m), 4.96–5.10 (2H, m), 5.93–6.13 (1H, m). IR (neat), 1639, 1591, 1454, 1377 cm^{-1} . Anal. (accurate mass) Calcd for $\text{C}_{16}\text{H}_{26}\text{O}$: 234.1982. Found: 234.2004.

2-Methoxy-2-methyl-3-decynenitrile (22): To a solution of 3.75 g (20.9 mmol) of 2-methoxy-3-decynenitrile (21) and 1.5 mL (24.1 mmol) of iodomethane in 100 mL of THF, was added dropwise a THF-hexane solution of LDA (22.5 mmol in 30 mL solution) at -78°C . After stirring at -78°C for 1.5 h, the reaction mixture was worked up with sat. NH_4Cl affording alkylated product 22 in quantitative yield. $^1\text{H-NMR}$ (CDCl_3), δ 0.90 (3H, t, $J = 6.6$ Hz), 1.15–1.65 (8H, m), 1.82 (3H, s), 2.24 (2H, t, $J = 7.0$ Hz), 3.53 (3H, s). IR (neat), 2280, 2250, 1240, 1200, 1100, 1060 cm^{-1} . Anal. Calcd for $\text{C}_{12}\text{H}_{19}\text{NO}$: C, 74.57; H, 9.91. Found: C, 74.79; H, 10.17.

2-Methoxy-3-decyn-1-ol (3a): Nitrile 21 (1.02 g, 5.67 mmol) was heated at reflux in 15 mL of ethanol containing catalytic amount of p-toluenesulphonic acid for 3 weeks affording ester 23, which was reduced with LiAlH_4 giving 0.97 g (5.3 mmol, 92% yield) of the alcohol 3a. $^1\text{H-NMR}$ (CDCl_3), δ 0.89 (3H, t, $J = 6.5$ Hz), 1.23–1.56 (8H, m), 1.79 (1H, br.s), 2.23 (2H, dt, $J = 1.9, 7.0$ Hz), 3.45 (3H, s), 3.68 (2H, d, $J = 5.6$ Hz), 4.05 (1H, tt, $J = 1.9, 5.6$ Hz). IR (neat), 3600–3050, 1340, 1240, 1105, 1065 cm^{-1} . Anal. (accurate mass) Calcd for $\text{C}_{11}\text{H}_{20}\text{O}_2$: 184.1462. Found: 184.0933.

2-Methoxy-2-methyl-3-decyn-1-ol (3b): To an ice-cooled solution of 594 mg (3.68 mmol) of 22 was added a hexane solution of DIBAL (9.13 mmol in 5.7 mL solution) and the whole was stirred at room temperature for 3 h. Excess DIBAL was decomposed with methanol and the mixture was treated with sat. Na_2SO_4 . Filtrate of the reaction mixture was mixed with 20 mL of 1N-HCl under vigorous stirring. The reaction mixture was extracted with ether and ethereal solution was dried (Na_2SO_4) and concentrated affording 551 mg of crude aldehyde 24, which was reduced with LiAlH_4 giving 492 mg (2.51 mmol, 81% yield) of alcohol 3b. $^1\text{H-NMR}$ (CDCl_3), δ 0.87 (3H, t, $J = 6.9$ Hz), 1.20–1.55 (8H, m), 1.40 (3H, s), 2.22 (2H, t, $J = 7.0$ Hz), 3.36 (3H, s), 3.47 (1H, d, $J = 11.0$ Hz), 3.64 (1H, d, $J = 11.0$ Hz). IR (neat), 3650–3100, 2250, 1380, 1230, 1200, 1170, 1105, 1075 cm^{-1} . Anal. (accurate mass) Calcd for $\text{C}_{12}\text{H}_{22}\text{O}_2$: 198.1618. Found: 198.1550.

3-Methoxy-3-methyl-4-undecyn-2-ol (3c): To a THF solution of 776 mg (4.02 mmol) of 22 in 5 mL of THF was added an ether solution of methylolithium (4.8 mmol in 9.6 mL of solution) at 0°C and the mixture was stirred at 0°C for 30 min. To the reaction mixture was added 10 mL of 10% H_2SO_4 and the whole was stirred vigorously for 10 min. Workup of the reaction mixture with sat. NH_4Cl gave 8.29 mg of ketone 25, which was reduced with LiAlH_4 affording 792 mg (3.74 mmol, 92% yield). This is a mixture of two diastereomers in a ratio of 6:4 of the alcohol 3c. $^1\text{H-NMR}$

(CDCl₃), δ 0.89 (3H, t, J = 6.8 Hz), 1.23 (1.8H, d, J = 6.3 Hz), 1.24 (1.2H, d, J = 6.3 Hz), 1.32 (3H, s), 1.18-1.59 (8H, m), 2.23 (1.2H, t, J = 5.7 Hz), 2.25 (0.8H, t, J = 5.8 Hz), 2.36 (0.6H, br-d, J = 3.2 Hz), 2.56 (0.4H, br-d, J = 3.2 Hz), 3.38 (1.8H, s), 3.39 (1.2H, s), 3.66 (0.4H, dq, J = 3.2, 6.3 Hz), 3.85 (0.6H, dq, J = 3.2, 6.3 Hz). IR (neat), 3600-3200, 2250, 1370, 1280, 1200, 1175, 1165, 1110, 1090, 910 cm⁻¹. Anal. (accurate mass) Calcd for C₁₃H₂₄O₂: 212.1649. Found: 212.1635.

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