above. Electroreduction of **18b** gave **37** and **38** in 70% and 23% yields, respectively. From **18c** were obtained **19** and **39** in 45% and 47% yields, respectively.

37: IR (neat) 3060, 3020, 2930, 2860, 2850, 1452, 1435, 1372, 1350, 1200, 1120, 1075, 1058, 1030, 993, 970, 902, 865, 810, 733 cm^{-1}; NMR (CCl_4) δ 1.17 (s, 3 H), 1.60 (m, 6 H), 2.76 (dd, 4 H), 3.04–4.03 (m, 4 H), 4.49 (m, 1 H), 6.96 (s, 4 H); mass spectrum, *m/e* 246 (M^+), 145 ($M^+ - C_5H_9O_2$), 144 ($M^+ - C_5H_{10}O_2$), 85 ($C_5H_9O^+$).

38: IR (neat) 3350 (br), 3065, 3020, 2950, 2920, 2870, 1460, 1433, 1378, 1300, 1227, 1035, 978, 795, 735 cm^{-1}; NMR (CCl_4) δ 1.10 (s, 3 H), 2.15 (s, 1 H), 2.67 (dd, 4 H), 3.55 (s, 2 H), 6.93 (s, 4 H); mass spectrum, *m/e* 162 (M^+), 145 (M^+ – OH), 130 ($M^+ - CH_3OH$).

39: IR (neat) 3060, 3020, 2922, 2855, 1475, 1458, 1430, 1323, 1300, 1282, 1245, 1218, 1123, 1082, 1022, 973, 950, 828, 785, 740, 717 cm^{-1}; NMR (CCl_4) δ 3.19 (s, 4 H), 4.49 (s, 4 H), 7.03 (s, 4 H); mass spectrum, *m/e* 160 (M^+), 130 ($M^+ - CH_2O$).

Registry No. 4, 105-53-3; **5**, 1472-85-1; **6**, 64923-67-7; **7**, 1472-09-9; **8**, 13151-27-4; **9**, 607-81-8; **10**, 597-55-7; **11**, 82044-43-7; **12**, 65933-61-1; **13**, 82044-44-8; **14**, 82044-45-9; **15**, 82056-40-4; **16**, 66014-45-7; **17**, 82044-46-0; **18a**, 82044-47-1; **18b**, 82044-48-2; **18c**, 82044-49-3; **19**, 7198-71-2; **20**, 22274-75-5; **21**, 82044-50-6; **22**, 82044-51-7; **23**, 23904-39-4; **24**, 82044-52-8; **25**, 82044-53-9; **26**, 141-97-9; **27**, 5397-53-5; **28**, 82044-54-0; *trans*-**29**, 82056-41-5; *cis*-**29**, 82044-55-1; **30**, 611-10-9; **31**, 36370-13-5; **32**, 82044-56-2; **33**, 82044-57-3; **34**, 4017-60-1; **35**, 82044-58-4; *trans*-**36**, 1731-44-8; *cis*-**36**, 1731-42-6; **37**, 82044-59-5; **38**, 73739-59-0; **39**, 82044-60-8; $PhCH_2Cl$, 100-44-7; *n*-$C_{12}H_{25}Br$, 143-15-7; *n*-$C_6H_{13}Br$, 111-25-1; 1-bromooctane, 111-83-1; allyl bromide, 106-95-6; 1,2-dibromomethylbenzene, 91-13-4; 2-cyclohexenone, 930-68-7; 2-butenal, 4170-30-3.

Allylic vs. Vinylic Deprotonation Reactions of Cyclic Vinyl Ethers. 7-Lithio-2,3,4,5-tetrahydrooxepin: Synthesis and Carbon-13 Nuclear Magnetic Resonance Spectrum

Fred T. Oakes, Fu-An Yang, and John F. Sebastian*

Department of Chemistry, Miami University, Oxford, Ohio 45056

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2,3,4,5-Tetrahydrooxepin treated with either *n*-butyllithium or *tert*-butyllithium affords 7-lithio-2,3,4,5-tetrahydrooxepin. The question of allylic vs. vinylic deprotonation of cyclic vinyl ethers was examined by varying ring size and degree of unsaturation. Carbon-13 spectral data for the anions obtained from 2,5-dihydrofuran, 2,3-dihydrooxepin, and 2,3,4,5-tetrahydrooxepin are reported. In contrast to 2,3,4,5-tetrahydrooxepin, 2,3-dihydrooxepin undergoes allylic deprotonation, possibly as a result of a slightly larger C—C═C bond angle in the dihydrooxepin as compared to that of the tetrahydrooxepin.

Introduction

Recently we reported that vinyl anions obtained from the deprotonation of the cyclic vinyl ethers 2,3-dihydrofuran (**1**) and 2,3-dihydro-4*H*-pyran (**2**) can be readily observed by carbon-13 NMR; formation of the α-lithiated species results in a large downfield shift of the resonance of the vinylic carbon bonded to oxygen.[1] Allylic deprotonation by alkyllithium reagents of these two cyclic ethers has not been observed. The magnitude of the internal angle of the possible vinyl or allyl anions is one of several factors expected to affect the site of deprotonation.[2] For example, a $\overline{C{=}C{-}O}$ angle of about 113° is favored for vinyl anions,[3] whereas a $\overline{C{-}C{-}C}$ angle of approximately 132° is preferred for allyl anions.[4] An important difference between the allyl and vinyl anions, and another factor affecting the site of deprotonation, is the presence of the oxygen atom which is expected to inductively stabilize the vinyl anion and conjugatively destabilize the allyl anion. Allylic deprotonation should be favored by an opening up of the C—C═C angle in vinyl ethers, while vinyl deprotonation is favored by a contraction of the C═C—O angle; calculations suggest that such a contraction occurs upon formation of the vinyl anion.[3] Restricting appreciable contraction, and thus discouraging vinyl deprotonation, might be accomplished by incorporating the C═C—O moiety within a suitable sized ring and by extending the conjugation (compare 2,3,4,5-tetrahydrooxepin (**3**) and 2,3-dihydrooxepin (**4**)). The expected effect of ring size on the internal angle may be approximated by considering the C═C—C angle in relevant cycloalkenes:[5,6] cyclopentene (111.8°), cyclohexene (123.5°), cycloheptene (125.5°), cyclooctene (121.0°), and 1,3-cycloheptadiene (129.1°). These data suggest that in the corresponding cyclic vinyl ether series 2,3,4,5-tetrahydrooxepin (**3**) and 2,3-dihydrooxepin (**4**) are the best candidates for allylic deprotonation provided that the bond angle considerations are not dominated by the effects of the oxygen atom mentioned above.

In the present paper we report on an NMR study of the deprotonation reactions of 2,3,4,5-tetrahydrooxepin (**3**), 2,3-dihydrooxepin (**4**), and 2,5-dehydrofuran (**5**). Deprotonation reactions of **3** have not been previously published to the best of our knowledge. The deprotonation reactions of 2,3-dihydrooxepin and 2,5-dihydrofuran have been noted previously[7,8] although the carbon-13 spectra have not been reported for their respective anions.

Results and Discussion

The carbon-13 chemical shifts for various ethers of interest are shown in Table I along with ${}^1J_{^{13}C-^1H}$ values and the percent *s* character of the carbon orbitals of the C–H bonds. The effect of the oxygen atom is to increase the

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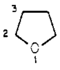
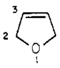
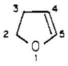
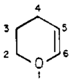
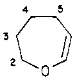
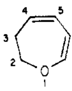
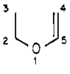
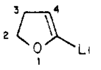
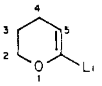
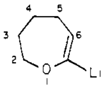
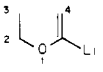
Table I. Carbon-13 Chemical Shifts (δ),[a] $^1J_{^{13}C-^1H}$, and Percent *s* Character of Selected Ethers[b]

		δ $^1J_{^{13}C-^1H}$, Hz % s[c]					
		2	3	4	5	6	7
	(12)	66.9 148.9 29.8	26.0 133.0 26.6				
	(5)	74.5 147.6 29.6	125.9 172.3 34.5				
	(1)	68.6 148.2 29.7	28.4 133.1 26.6	98.4 174.6 34.9	145.6 189.2 37.8		
	(2)	64.9 147.1 29.4	19.3 130.0 26.0	22.6 131.2 26.2	99.2 161.7 32.4	144.0 184.9 37.0	
	(3)	71.7 142.4 28.5	27.0[d] 125[e] 25[e]	26.4[d] 125[e] 25[e]	31.3 126.1 25.2	107.7 154.6 30.9	147.8 183.1 36.6
	(4)	69.4 146.5 29.3	34.1 126.1 25.2	124.2 154.6 30.9	129.4 159.4 31.9	101.7 151.2 30.3	148.4 185.8 37.2
	(6)	62.4 142.8 28.6	13.6 125.7 25.2	84.6 160.8, 156.4 32.2, 31.3	151.4 180.1 36.0		

[a] In parts per million relative to external Me_4Si. [b] Some of the data for 2,3-dihydrofuran, 2,3-dihydro-4*H*-pyran, and ethoxyethene were taken from ref 1. [c] Reference 9 (calculated from 0.2 × $^1J_{^{13}C-^1H}$). [d] These values may be interchanged. [e] Estimated.

Table II. Carbon-13 Chemical Shifts (δ),[a] $^1J_{^{13}C-^1H}$, and Percent *s* Character of Lithiated Vinyl Ethers[b]

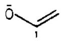
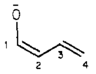
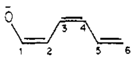
	δ $^1J_{^{13}C-^1H}$, Hz % s[c]					
	2	3	4	5	6	7
	66.04	30.70	109.6 165.5 33.1	202.3		
	62.80 141.7 28.3	20.99 127.5 25.5	24.71 128.8 25.8	111.1 154.8 31.0	201.2	
	68.04 136.3 27.3	28.12[d]	26.98[d]	32.49 124.8 25.0	121.0 150.0 30.0	210.2
	56.83 141.1 28.2	14.45 125[e] 25[e]	93.46 156.0, 154.6 31.2, 30.9	212.1		

[a] In parts per million relative to external Me_4Si. [b] Some of the data for α-lithiated 2,3-dihydrofuran, 2,3-dihydro-4*H*-pyran, and ethoxyethene were taken from ref 1. [c] Reference 9 (calculated from 0.2 × $^1J_{^{13}C-^1H}$). [d] These values may be interchanged. [e] Estimated.

percent *s* character by about 3 (compare percent *s* values for positions 2 and 3 of the ethers in Table I). For example, position 3 of tetrahydrofuran (THF, **12**) has a percent *s* value of 26.6 as compared to 29.8 at position 2. Incorporation of a double bond increases the percent *s* character by about 8. The effect of the oxygen atom and the double bond results in the carbon orbital of the α-vinyl CH bond of the vinyl ethers having a percent *s* character of 36.0–37.8. The range of 1.8 is very likely a result of internal bond angle differences with additional vinyl conjugation a contributing factor to the value obtained for the dihydrooxepin. Ring size does not seem to appreciably affect the *s* character of the C–H carbon orbital at position 2 (range = 1.3% *s*). In contrast, the percent *s* value for the vinyl CH β to oxygen decreases markedly as ring size increases (Table I).

Compounds **1** and **2** and ethoxyethene (**6**) deprotonate at the α-vinyl carbon to yield the corresponding acyl anion equivalents.[1] We have found in the present study that 2,3,4,5-tetrahydrooxepin (**3**) also deprotonates at the α-vinyl position upon treatment with *tert*-butyllithium in pentane/THF at −78 °C. This anion can also be prepared with *n*-butyllithium; reaction of the lithiated species with acetone gives the expected α-substituted alcohol (see Experimental Section). The carbon-13 chemical shifts, $^1J_{^{13}C-^1H}$ values and percent *s* character for various vinyl lithiated

Table III. Carbon-13 Chemical Shifts (δ)[a] and CNDO/2 π-Electron Densities

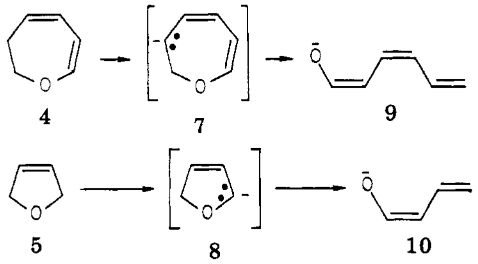
		δ π-electron densities					
		1	2	3	4	5	6
	(11)	158.0 0.801	81.1 1.51				
	(10)	153.4 0.786	103.3 1.40	131.7 0.846	103.3 1.38		
	(9)	156.4 0.782	98.2 1.35	133.7 0.826	117.4 1.33	126.6 0.872	110.1 1.31

[a] In parts per million relative to external Me_4Si. For lithium salts. Assignments based on shift comparisons, off-resonance spectra, and correlations of shifts and π-electron densities.

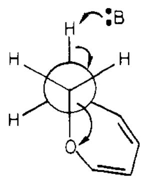
ethers are shown in Table II.

The increased C=C—O bond angle of **3** over that in **1** and **2** is apparently insufficient to result in allylic deprotonation. It should be recognized that while the conjugative destabilization resulting from allylic deprotonation (a π-electron system isoelectronic with the butadiene dianion) is a factor favoring vinyl deprotonation, such destabilization does not prevent allylic deprotonation at the oxygen-activated position 6 in 3,6-dihydro-2*H*-pyran.[10a,b]

2,3-Dihydrooxepin (**4**) and 2,5-dihydrofuran (**5**) undergo allylic deprotonation to presumably form the anions **7** and **8** which rapidly ring open to afford **9** and **10**, respectively.



The lithium salt of dienylate **10** has the *Z,E* configuration,[8] while that of the potassium salt of trienylate **9** is *Z,E,Z,E*.[7] We have found that the lithium trienylate, of undetermined configuration, can be prepared at room temperature by the reaction of **4** with 6-lithio-2,3-dihydro-4*H*-pyran. The carbon-13 chemical shifts and CNDO/2 π-electron densities on the carbon atoms for **9**, **10**, and the enolate (**11**) of acetaldehyde are given in Table III. Ring opening from **4** and **5** or from **7** and **8** can, in principle, proceed by several possible pathways, including the following: (a) ring opening of **7** and **8** might occur via conrotatory and disrotatory processes, respectively, (b) molecular models suggest that **4** may be capable of undergoing an E2 elimination (I) to give **9**. It is known that oxepane can suffer



I

β cleavage upon reaction with *n*-butyllithium to give the lithium alkoxide of 5-hexen-1-ol.[8] The models also suggest that **5** cannot readily assume a favorable anti orientation for E2 elimination. Tetrahydrofuran (**12**), upon treatment with alkyllithium reagents, appears to form an α anion which subsequently ring opens by a $[_\pi 4_s + _\pi 2_s]$ cycloreversion to form the lithium enolate (**11**) of acetaldehyde and ethylene.[8] In contrast to oxepane, β cleavage of THF has not been observed except in the gas phase.[11]

Irrespective of the mechanism of ring opening, allylic deprotonation of **4** and **5** leads to the observed products. It is not surprising that **5** should undergo allylic deprotonation as that position is activated inductively by the electronegative oxygen atom. Since position 3 in compound **4** does not enjoy the same proximity to an oxygen atom, there must be some other factor(s) involved. One possibility is that the α-vinyl anion of **4** is indeed formed but rapidly isomerizes to **7** which irreversibly ring opens. On the other hand, the C—C=C bond angle in **4** is expected to be about 130° (based on comparison with 1,3-cycloheptadiene), some 4° or 5° greater than that of **3** (based on comparison with cycloheptene). Thus, allylic deprotonation of **4** should be favored, relative to **3**, due to the greater bond angle and because the ring system of **4** is relatively rigid and more nearly planar than **3** and therefore less susceptible to bond angle contraction.

Experimental Section

General Comments. All glassware, syringes, syringe stopcocks, and needles were dried in a 110 °C oven for several days before use. All organometallic reactions were carried out in an argon atmosphere. THF was distilled from sodium benzophenone ketyl immediately prior to use. For preparation of carbon-13 NMR samples *n*-butyllithium in pure *n*-hexane from Lithcoa was utilized. Otherwise, *n*-butyllithium in hexanes from Aldrich was used.

Infrared spectra were recorded on a Perkin-Elmer 237B grated infrared spectrophotometer. Mass spectra were recorded on a Hitachi RMU-6 spectrometer.

NMR Measurements. Broad-band (5 W) proton-decoupled carbon-13 spectra were recorded at 22.625 MHz on a Bruker WH-90DS spectrometer equipped with a Nicolet 1180 16 K computer/Diablo disk drive and a Bruker B-ST 100/700 C variable-temperature unit. Carbon-13 spectra were obtained by securely positioning an 8-mm NMR tube containing the sample within a 10-mm NMR rube containing $CDCl_3$ (for lock) and Me_4Si. Chemical shifts (δ) were measured relative to external Me_4Si. A pulse width of 3 μs (27° pulse angle) was used to collect 100–6900 scans in the quadrature detection mode using a sweep width of 5555 Hz (1.36 Hz/pt) or 6024 Hz (1.47 Hz/pt). A line broadening of 1.00 Hz was employed in all cases. Completely coupled carbon-13 spectra were recorded using 1600–61 290 scans. Off-resonance spectra were measured in the CW mode with a decoupler power of 0.5 W.

Proton NMR spectra were obtained on either a Varian EM-360A or a Jeol C-60H 60-MHz spectrometer with shifts (δ) determined relative to internal Me_4Si.

Molecular Orbital Calculations. π-electron densities and total electron densities were calculated by the CNDO/2 method.

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Standard bond lengths and bond angles were employed since only trends in charge densities were of interest.[12]

Preparation of Lithiated Compounds. A. 1-Lithio-(*Z*,-*E*)-1-oxapentadiene (10). To a sample of 2,5-dihydrofuran (**5**; Pfaltz and Bauer; 1.0 g, 0.0143 mol), in an 8-mm NMR tube, was added 4.0 mL (0.0064 mol) of 1.6 M *n*-butyllithium in *n*-hexane at room temperature. The mixture was then shaken carefully for several minutes. The carbon-13 NMR spectrum showed nearly 40% conversion to **10**. See Table III for ^{13}C chemical shifts. When 2,5-dihydrofuran in THF was allowed to react with *tert*-butyllithium (1 equiv) at −78 °C, only the O-silylated dienylate was observed by ^{13}C NMR: 138.8 (d), 129.8 (d), 111.6 (d, t), −1.8 (q).

B. Lithium Enolate of Acetaldehyde (11). To a sample of tetrahydrofuran (**12**) in an 8-mm NMR tube was added 0.5 equiv of 2.4 M *n*-butyllithium in *n*-hexane. The mixture was allowed to react at room temperature for 20 h. After that time most of the *n*-butyllithium had reacted and approximately 50% yield of **11** was observed by ^{13}C NMR. A large peak at 122.1 ppm was also noted and corresponded to ethylene formed in the reaction.

C. 6-Lithio-2,3-dihydro-4*H*-pyran. To a stirred mixture of 2.15 g (0.026 mol) 2,3-dihydro-4*H*-pyran (**2**; Aldrich) and 1.0 g (0.0086 mol) of *N,N,N′,N′*-tetramethylethylenediamine (TMEDA) at ambient temperature in a small reaction vessel was added 22 mL (0.035 mol) of 1.6 M *n*-butyllithium in *n*-hexane. The metalated dihydropyran precipitated after several hours of slight heating to give an off-white solid. The mixture was centrifuged and the hexane layer removed via syringe. The solid was washed with *n*-hexane (5 × 4 mL), giving a white solid which was then dissolved in dry THF to yield a nearly saturated solution. An aliquot was transferred to an 8-mm NMR tube capped with a rubber septum. The ^{13}C NMR spectrum showed 6-lithio-2,3-dihydro-4*H*-pyran free of unreacted starting material.

D. 1-Lithio-1-oxaheptatriene (9). To the 8-mm NMR tube with the 6-lithio-2,3-dihydro-4*H*-pyran described above was added 0.8 g of 2,3-dihydrooxepin (0.008 mol) at room temperature, which resulted in a red-brown solution. Carbon-13 spectra showed essentially complete conversion to **9** after 3 h as well as formation of 2,3-dihydro-4*H*-pyran. At no time was there any evidence for **7** when the progress of the reaction was followed by carbon-13 NMR.

Preparation of 2,3-Dihydrooxepin (4).[13] *n*-Butyllithium (125 mL, 0.20 mol) 1.6 M in hexanes was added dropwise over a period of 4 h to a cold (−10 to −20 °C) mixture of dry 2,3-dihydropyran (25.2 g, 0.30 mol) and dry methylene chloride (25.5 g, 0.30 mol). The mixture was allowed to warm to room temperature overnight. Water (30 mL) was added, the mixture filtered, and then the organic layer separated. The aqueous layer was extracted 3 times with 10-mL portions of hexane. The organic layers were combinded and dried ($MgSO_4$), and then the solvent was distilled away. Freshly distilled quinoline (14.0 g) was added to the residue and the product was distilled as it formed at ca. 50 torr with heating. Overall yield was 12%. **4**: IR (hexane) 2930, 2900, 1595, 1295, 1118, 975, 918, 875, 768, 710 cm^{-1}; ^1H NMR (δ) 6.25 (d, 1.0 H), 5.60 (d of d, 2.0 H), 4.75 (t, 1.2 H), 4.00 (t, 2.1 H), 2.42 (q, 2.1 H); ^{13}C NMR ($CDCl_3$), see Results and Discussion.

Preparation of 2,3,4,5-Tetrahydrooxepin (3).[14] To a melted sample of 1,6-hexanediol (28.0 g, 0.237 mol) were added a mixture of 3.5 g of copper chromite ($2CuO{\cdot}Cr_2O_3$) and 0.5 g of tungstosilicic acid in a 250-mL three-neck flask equipped with a thermometer and a 24-in. Vigreux column wrapped with glass wool. The reaction mixture was heated to 230 °C, without stirring, where evolution of gas began. The temperature was carefully maintained between 238 and 248 °C over a period of 45 min during which distillation of water and 2,3,4,5-tetrahydrooxepin occurred. The fraction boiling at 70–85 °C was collected into a receiver containing 2 g of NaOH pellets (to prevent hydrolysis) until the temperature of the mixture dropped to 180–190 °C and a yellow oil began to distill. After washing twice with 10 mL of H_2O, the crude material (9.0 g, 38% conversion; no detectable side products were observed in the distillate by ^{13}C NMR) was dried with Na_2SO_4, decanted, and dried over CaH_2 and distilled (113–116 °C), giving 7.4 g (32% yield based on diol) of pure 2,3,4,5-tetrahydrooxepin. See Table I for ^{13}C NMR chemical shifts and ${}^1J_{^{13}C-^1H}$ values.

7-Lithio-2,3,4,5-Tetrahydrooxepin. The addition of *tert*-butyllithium (1.7 M; 3.0 mL, 5.1 mmol) to THF (1.0 g) at −78 °C in an 8-mm NMR tube resulted in a yellow precipitate. To this was added 2,3,4,5-tetrahydrooxepin (**3**; 0.4 g, 4.0 mmol) and the resulting mixture was shaken and allowed to warm until homogeneous. The solution was recooled and allowed to warm slowly to ambient. After 2 h the ^{13}C NMR spectrum showed 90% conversion to 7-lithio-2,3,4,5-tetrahydrooxepin; see Table II for ^{13}C NMR chemical shifts and ${}^1J_{^{13}C-^1H}$ values.

7-(2-Hydroxy-2-propyl)-2,3,4,5-tetrahydrooxepin. A mixture of 2,3,4,5-tetrahydrooxepin (1.25 g, 12.8 mmol), 1.25 g of benzene, and 8 mL of THF was cooled to 0 °C; then *n*-butyllithium (1.6 M; 8.0 mL, 12.8 mmol) was added with stirring. After 4 h the ^{13}C NMR spectrum showed 70% conversion to 7-lithio-2,3,4,5-tetrahydrooxepin. The mixture was allowed to stand 3 days and then excess acetone was added followed by H_2O. The layers were separated, and the organic phase was washed, dried (Na_2CO_3), and then evaporated, giving 0.75 g of crude 7-(2-hydroxy-2-propyl)-2,3,4,5-tetrahydrooxepin, 37.6% yield. A sample of 0.65 g of the alcohol was distilled 69–79 °C (0.5 torr) and used for combustion analysis and ^{13}C and ^1H NMR and mass spectra. The clear yellow oil (300 mg) obtained gave the following results: ^{13}C NMR ($CDCl_3$, internal Me_4Si) δ 164.3 (s), 104.1 (d), 73.1 (t), 72.1 (s), 32.1 (t), 27.9 (q), 25.8 (t), 25.6 (t); ^1H NMR ($CDCl_3$) (δ 5.15 (t), 3.90 (t), 2.75 (s), 2.15 (m), 1.75 (m), 1.30 (s); mass spectrum, *m/e* 156 (M^+), 141 ($M^+ - 15$), 138 ($M^+ - 18$), 127 ($M^+ - 29$), 124 ($M^+ - 32$), 123 ($M^+ - 33$), 121 ($M^+ - 35$), 109 ($M^+ - 47$), 99 ($M^+ - 57$), 97 ($M^+ - 59$), 95 ($M^+ - 61$), 82 ($M^+ - 74$), 68 ($M^+ - 88$), 59 ($M^+ - 97$), 55 ($M^+ - 101$).

Anal. Calcd for $C_9H_{16}O$: C, 69.19; H, 10.32. Found: C, 69.06; H, 10.36.

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Registry No. 1, 1191-99-7; **2**, 110-87-2; **3**, 1121-01-3; **4**, 3422-09-1; **5**, 1708-29-8; **6**, 109-92-2; **9**, 82056-37-9; **10**, 82056-38-0; **11**, 2180-63-4; **12**, 109-99-9; 4-lithio-2,3-dihydrofuran, 75213-94-4; 6-lithio-3,4-dihydro-2*H*-pyran, 72081-15-3; 7-lithio-2,3,4,5-tetrahydrooxepin, 82056-39-1; (1-ethoxyethenyl)lithium, 40207-59-8; 7-(2-hydroxy-2-propyl)-2,3,4,5-tetrahydrooxepin, 82064-44-6.

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