extracts were washed with saturated sodium bicarbonate and water, dried over $MgSO_4$, and concentrated in vacuo. The residue was purified by flash chromatography, eluting with ethyl acetate–hexane (1:2) to afford 1.78 g of the hydrated aldehyde which was distilled in a Kugelrohr apparatus [bp 90–95 °C (0.05 torr)] to yield 1.46 g (70%) of **7**: IR (film) (hydrate) 3400, 2950, 1740, 1440, 1220 cm^{-1}; ^1H NMR ($CDCl_3$, 60 MHz) δ 1.8–2.5 (6 H, m), 5.2 (1 H, m), 5.6 (2 H, m), 9.4 (1 H, s); mass spectrum, *m/z* (relative intensity) 154 (1), 125 (1), 81 (100), 80 (88), 79 (36), 77 (11).

Synthesis of Nitrones 1Z and 1E. A mixture of 2.79 g (0.02 mol) of *N*-benzylhydroxylamine and 0.200 g of calcium chloride in 75 mL of ether was cooled to 0 °C.[3] To this mixture was added 3.50 g (0.02 mol) of freshly distilled glyoxylate **7** in 30 mL of ether. After being stirred at 0 °C for 2 h, the mixture was filtered through Na_2SO_4 and concentrated in vacuo to provide a clear oil. This oil was dissolved in 100 mL of benzene and water was azeotroped off for 4 h. Removal of the solvent in vacuo yielded 5.65 g (96%) of a white waxy solid which was a 2:1 mixture of *E* and *Z* nitrone isomers, respectively: IR (film) 2950, 1710, 1550, 1200, 1170 cm^{-1}; ^1H NMR ($CDCl_3$, 360 MHz) δ 1.7 (1 H, m), 1.8 (1 H, m), 2.1 (3 H, m), 2.4 (1 H, m), 4.9 (2 H, s, minor isomer), 5.1 (1 H, m), 5.6 (1 H, m), 5.65 (1 H, m), 5.7 (2 H, s, major isomer), 7.09 (1 H, s, minor isomer), 7.20 (1 H, s, major isomer), 7.35 (2 H, m), 7.43 (2 H, m), 7.53 (1 H, m); mass spectrum, *m/z* (relative intensity) 259 (1), 242 (2), 215 (1), 180 (3), 162 (10), 91 (100), 80 (95); exact mass calcd for $C_{15}H_{17}NO_3$ 259.1208, found 259.1205.

(±)-(3α,3aβ,5α,7aβ)-Hexahydro-1-(phenylmethyl)-3,5-ethano-7*H*-pyrano[3,4-*c*]isoxazol-7-one (2). A magnetically stirred solution of 0.960 g (3.7 mmol) of the nitrone mixture **1** in 400 mL of dry chlorobenzene was heated at reflux for 16 h. The solvent was removed in vacuo and the residue was purified by flash chromatography on silica gel, eluting with ethyl acetate–hexane (1:1), to yield 0.115 g of starting nitrone mixture **1** along with 0.534 g (56% based on recovered nitrone) of the adduct **2**: IR (film) 2950, 1735, 1495, 1390, 1230, 1075 cm^{-1}; ^1H NMR ($CDCl_3$, 360 MHz) δ 1.6–2.1 (6 H, m), 3.11 (1 H, m), 3.89 (1 H, d, J = 8.2 Hz), 4.05 (1 H, d, J = 13.7 Hz), 4.18 (1 H, d, J = 13.7 Hz), 4.60 (1 H, m), 4.81 (1 H, m), 7.3–7.5 (5 H, m); ^{13}C NMR ($CDCl_3$) δ 20.8, 23.6, 25.5, 36.4, 60.1, 66.2, 73.6, 74.6, 127.4, 128.3, 128.8, 136.5, 168.7; mass spectrum, *m/z* (relative intensity) 259 (5), 215 (7), 160 (30), 91 (100); exact mass calcd for $C_{15}H_{17}NO_3$ 259.1208, found 259.1221.

Cleavage of Isoxazolidine 2. A solution of 0.190 g (0.7 mmol) of isoxazolidine **2** in 35 mL of aqueous THF (THF:H_2O/10:1) was cooled to 0 °C. Small strips of aluminum foil (0.380 g) were sequentially exposed for 15–30 s to 1 M NaOH, distilled water, 0.5% aqueous mercuric chloride, distilled water, and THF and then added to the solution of **2**. After being stirred at 0 °C for 14 h, the mixture was filtered through Celite and the solvent was removed in vacuo. The residue was dissolved in ethyl acetate and washed with 20 mL of water. The organic extract was dried over Na_2SO_4 and evaporated in vacuo. Purification of the residue by column chromatography on 30 g of silica gel, eluting with ethyl acetate, afforded 0.150 g (78%) of γ-lactone **8** as a white crystalline product. A sample which was crystallized from benzene–hexane had mp 83 °C: IR (film) 3300, 2950, 1775, 1650, 1450, 1190, 960 cm^{-1}; ^1H NMR ($CDCl_3$, 200 MHz) δ 1.6–2.3 (7 H, m), 3.59 (1 H, d, J = 5.8 Hz), 3.80 (1 H, m), 3.90 (1 H, d, J = 13.1 Hz), 3.99 (1 H, d, J = 13.1 Hz), 4.65 (1 H, m), 7.30–7.37 (5 H, m); mass spectrum, *m/z* (relative intensity) 261 (1), 217 (3), 170 (4), 160 (2), 146 (15), 106 (59), 91 (100); exact mass calcd for $C_{15}H_{19}NO_3$ 261.1365, found 261.1380.

Hydrogen bromide gas was bubbled through a solution of 0.035 g (0.13 mmol) of the amino alcohol **8** in 8 mL of ether for 2 min. The mixture was stirred at room temperature for an additional 5 min during which time a white precipitate formed. Removal of the solvent in vacuo afforded 0.046 g (100%) of the white crystalline hydrobromide which was recrystallized from acetone–hexane: mp 142–143 °C; IR (KBr) 3400, 3150, 2950, 1780, 1570, 1440, 1180, 1000 cm^{-1}; ^1H NMR ($(CD_3)_2SO$, 200 MHz) δ 1.5–1.8 (6 H, m), 2.8 (1 H, m), 3.3 (3 H, bs), 3.8 (1 H, m), 4.4 (3 H, m), 4.8 (1 H, m), 7.4–77.5 (5 H, m); mass spectrum, *m/z* (relative intensity) 261 (1), 217 (4), 170 (5), 146 (21), 106 (88), 91 (100), 82 (12).

X-ray Crystal Structure Determination of the HBr Salt of 8. Unit cell dimensions determined from 25 reflections at moderate 2θ angles indicated a monoclinic cell of dimensions: a = 9.298 (2) Å; b = 15.613 (3) Å; c = 11.095 (5) Å; B = 96.83 (4)°, and v = 1599 (1) Å^3. The observed volume was consistent with that expected for Z = 4, using a calculated density of 1.496 g cm^{-3}. Observed systematic absence of $h\phi l$ for $l = 2n + 1$ and $\phi k\phi$ for $k = 2n + 1$ gave the space group $P2_1/c$. Applications of the zero-moment tests of Howells, Phillips, and Rogers indicated a centric cell.[9]

Data was collected using molybdenum K_α radiation [λ 0.71073 Å]. A total of 2236 reflections were collected to a 2θ of 44.32°; scan width: (0.60 + 0.347 tan θ); scan range: (1.0–5.0° min^{-1}); of these 1764 were unique nonzero reflections. Three standard reflections collected every hour were used to rescale the data to account for changes in orientation, temperature, etc., during data collection (drift correction 0.956–1.048). φ scans indicated absorption was not severe (N = 27.42 cm^{-1}) and no absorption corrections were applied. A total of 1396 observed reflections with intensities $I \gtrsim 2\sigma$ were used for refinement of 256 variables. These data were corrected for Lorentz and polarization factors and used in the refinement of the structure.

Inclusion of the hydrogens at fixed isotropic factors (B = 5.0 Å^2) and refinement of positional parameters along the full matrix anisotropic refinement of the non-hydrogen atoms using unit weights converged to R_1 = 0.037 and R_2 = 0.040 with an esd = 1.744 with a maximum shift of δ 0.17. A final difference electron density map showed no residual electron density greater than 0.15 e Å^{-3}. Final positional and thermal parameters are available as supplementary material.

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Registry No. **1E**, 102285-01-8; **1Z**, 102284-98-0; **2**, 102284-99-1; **5**, 102285-02-9; **6**, 102285-03-0; **7**, 102285-04-1; **8**, 102285-05-2; **8**·HBr, 102285-00-7; 3-cyclohexen-1-ol, 822-66-2; bromoacetyl bromide, 598-21-0; 3-cyclohexenyl iodoacetate, 102306-08-1; *N*-benzylhydroxylamine, 622-30-0.

Supplementary Material Available: Tables of X-ray crystallographic data on the HBr salt of **8** (5 pages). Ordering information is given on any current masthead page.

(9) Howells, E. R.; Phillips, D. C.; Rogers, D. *Acta Crystallogr.* **1950**, *3*, 210.

A Facile Intramolecular Michael Addition Reaction[1]

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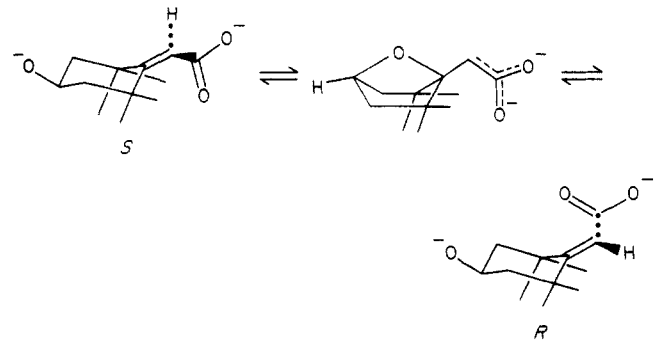
The *p*-methoxybenzyl and 3,4-dimethoxybenzyl groups are useful protecting groups for alcohols because they can be removed simply, under neutral conditions at room temperature, by oxidation with a mild oxidizing reagent, 2,3-dichloro-5,6-dicyanobenzoquinone (DDQ).[2] It was the method of choice for the protection of the 4-hydroxyl group in (4-hydroxy-2,2,6,6-tetramethylcyclohexylidene)acetic acid ((±)-**1**). The treatment of (±)-**1** with 2.2 equiv each of sodium hydride and 3,4-dimethoxybenzyl chloride at ambient temperature overnight resulted in the formation of 3,4-dimethoxybenzyl [4-((3,4-dimethoxylbenzyl)oxy)-

(1) The support of this work by a grant from the National Science Foundation is gratefully acknowledged.

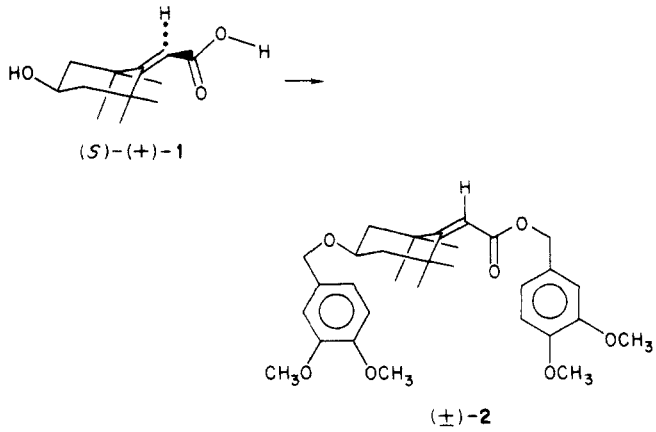
(2) Oikawa, Y.; Yoshioka, T.; Yonemitsu, O. *Tetrahedron Lett.* **1982**, 885.

0022-3263/86/1951-2605$01.50/0

Scheme I

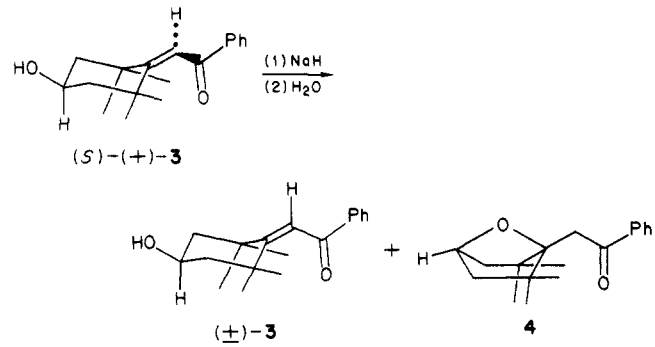


2,2,6,6-tetramethylcyclohexylidene]acetate ((±)-2). When this same reaction was carried out with the chiral acid (*S*)-(+)-1 the product of the reaction was not chiral 2 but instead the product was racemic (±)-2.



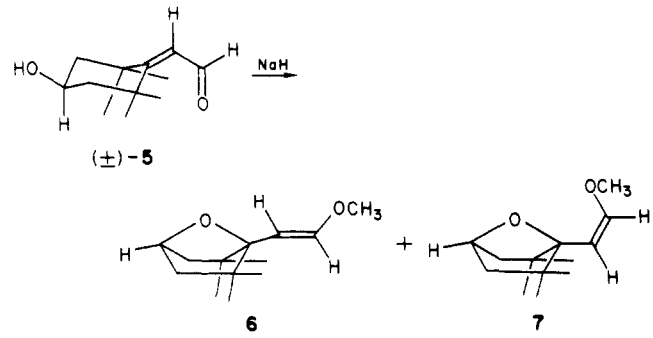
It was suspected that the racemization occurred by an intramolecular Michael addition of the 4-alkoxide group to the α,β-unsaturated carboxylate leading to an achiral intermediate which was in equilibrium with the *R* and *S* configurations of the alkoxide (Scheme I). In support of this hypothesis was the observation that treatment of the methyl ester of (*S*)-(+)-1, at ambient temperature overnight, with an equivalent of sodium hydride resulted in the isolation of the racemic ester. Although not isolated there was some NMR evidence that indicated the presence of the 7-oxabicyclo[2.2.1]heptane structure. The bridgehead proton appeared as a triplet at 4.3 ppm and the four methyl singlets in 1 became two singlets (see Experimental Section).

A clearer picture emerged from the reaction of (4-hydroxy-2,2,6,6-tetramethylcyclohexylidene)acetophenone ((*S*)-(+)-3) with sodium hydride. Here a 60% yield of



1-phenacyl-2,2,6,6-tetramethyl-7-oxabicyclo[2.2.1]heptane (4) was isolated and characterized (see Experimental Section). Of equal significance was the isolation (25% yield) of the starting ketone which was found to be racemic.

Treatment of (4-hydroxy-2,2,6,6-tetramethylcyclohexylidene)acetaldehyde ((±)-5) with sodium hydride gave a complex mixture of products probably due to, inter alia, aldol condensations. When the reaction was carried out in the presence of methyl iodide, two compounds were isolated which have been assigned structure 6 (24% yield) and 7 (12% yield). Neither 6 nor 7 have absorption bands



in the infrared for O–H or C=O stretching. Compound 6 has intense absorption band at 1660 and 1680 cm^{-1} which is characteristic of a *trans*-vinyl ether whereas 7 has only a moderate absorption at 1678 cm^{-1} which is typical for a *cis*-vinyl ether.[3] ^1H NMR is also consistent with the assigned structures. The C-4 proton in (±)-5 is a multiplet at 4.12 ppm whereas in compounds 6 and 7, as expected, the bridgehead proton at C-4 appears as a triplet at about 4.35 ppm. Moreover, whereas one observes four methyl absorptions in (±)-5, in 6 and 7 only two methyl absorptions are found which integrate for 12 protons. Finally the vinyl protons in 6 appear as two doublets at 4.70 and 6.47 ppm with J = 12.69 Hz, indicative of a trans double bond, and in 7 the vinyl protons are found at 4.20 and 5.94 ppm with J = 7.57 Hz, which is consistent with a cis double bond.[4] This reinforces the IR observations for these compounds. ^{13}C NMR spectra are consistent with the two bridgehead carbons being attached to an oxygen (see Experimental Section).

This is a good example of how the use of a chiral molecule has uncovered what would ordinarily have been a hidden reaction. Scheme I is a reasonable mechanism for the observed racemization of (*S*)-(+)-1 and its methyl ester.

Experimental Section

Melting points were determined with a Mel-Temp apparatus. Infrared (IR) spectra were measured with Perkin-Elmer Model 257 grating spectrophotometer using the polystyrene 1601-cm^{-1} band for calibration. Nuclear magnetic resonance (NMR) spectra were recorded on a Brucker 200- or 270-MHz spectrometer. The solvent used was $CDCl_3$ unless noted otherwise, with Me_4Si as internal standard. The chemical shifts are given in δ (ppm) downfield from Me_4Si and the coupling constants are in Hz. The microanalyses were performed by Beller Laboratories, Gottingen, Germany.

Ultraviolet (UV) spectra were recorded with a cary 219 spectrophotometer. The cell path length used was 1 cm. For column chromatography silica gel 70–230 mesh (Merck) was used unless noted otherwise. Radial chromatography thin-layer separations were performed with Merck silica gel 60 PF_{254} using Harrison Research Chromatotran Model 7924T.

3,4-Dimethoxybenzyl [4-((3,4-Dimethoxybenzyl)oxy)-2,2,6,6-tetramethylcyclohexylidene]acetate (2). A solution of (*S*)-(+)-(4-hydroxy-2,2,6,6-tetramethylcyclohexylidene)acetic acid ($[\alpha]^{31}_D$ +30.80 ± 0.20°, 37.52% ee) (5 g) and 3,4-dimeth-

(3) Silverstein, R. M.; Bassler, G. C.; Morrill, T. C. *Spectrometric Identification of Organic Compounds*; Wiley: New York, 1981; p 116.
(4) Jackman, L. M.; Sternhell, S. *Applications of Nuclear Magnetic Resonance Spectroscopy in Organic Chemistry*; Pergamon: 1969; p 302.

oxybenzyl chloride (8.80 g) in 25 mL of DMF was added for 30 min to a cooled (0 °C) suspension of NaH (2.36 g of 60% dispersion) in 75 mL of DMF and stirred at 25 °C. After 1 h 20% excess of 3,4-dimethoxybenzyl chloride (0.88 g) was added and left overnight. The reaction mixture was poured over ice and after usual workup the crude product was purified by passing through silica gel column with hexane–ether. Fractions corresponding to the pure product was collected to give racemic ester **2** as a colorless liquid (11.50 g, 95%): IR (film) 2900 (m), 1720, 1612, 1596, 1520, 1470, 1425, 1400–1040, 970–785 cm^{-1}; ^1H NMR (CCl_4, 60 MHz) 1.20 (s, 3 H), 1.25 (s, 3 H), 1.30 (s, 6 H), 1.35–2.00 (m, 4 H), 3.70 (m, 1 H), 3.75 (s, 12 H), 4.39 (s, 2 H), 5.07 (s, 2 H), 5.78 (s, 1 H), 6.60–6.80 (m, 6 H) ppm.

Reaction of Methyl (*S*)-(+)-(4-Hydroxy-2,2,6,6-tetramethylcyclohexylidene)-acetate with NaH in THF. A mixture of 226 mg of methyl (*S*)-(+)-(4-hydroxy-2,2,6,6-tetramethylcyclohexylidene)acetate ($[\alpha]^{25}_D$ +31.31 ± 0.20°, 37.52% ee) and 100 mg of NaH in 5 mL of THF was stirred overnight at 25 °C under N_2 atmosphere. The reaction mixture was diluted with wet ether (50 mL) and filtered through 5 g of silica gel to give mixture of several compounds. The crude mixture on radial chromatography separation using hexane–ether solvent mixtures gave two main fractions. The ^1H NMR spectrum of the less polar fraction (70 mg) showed signal corresponding to bridged bicyclic compounds (^1H NMR 1.00–1.50 (several methyl s), 1.50–2.00 (m), 2.80 (s), 2.84 (s), 3.68 (s), 3.71 (s), 3.72 (s), 4.30 (t), 5.20 (br), 5.81 (s), 5.85 (s) ppm). No further attempts was made to separate these compounds. The polar fraction (120 mg) was found to be racemic starting material methyl ester.

NaH Treatment of (*S*)-(+)-(4-Hydroxy-2,2,6,6-tetramethylcyclohexylidene)acetophenone (3) in THF. A mixture of 272 mg of (*S*)-(+)-(4-hydroxy-2,2,6,6-tetramethylcyclohexylidene)acetophenone (**3**) ($[\alpha]^{31}_D$ +65.63 ± 0.30°, 37.52% ee) and excess NaH (150 mg) in 5 mL of THF was stirred at 25 °C for 7 days. Water was added, and the mixture of products was extracted with ether (3 × 50 mL). The combined ether solution was washed with water, dried (Na_2SO_4), and evaporated. The crude mixture on separation by radial chromatography gave two fractions.

The less polar fraction I, 0.165 g (60%), solidified on standing to give 1-phenacyl-2,2,6,6-tetramethyl-7-oxabicyclo[2.2.1]heptane (**4**): mp 70–71 °C; IR (CCl_4) 3080 (w), 3060 (w), 3000 (w), 2980, 2950, 2880, 1950 (w), 1900 (w), 1810 (w), 1700, 1600, 1580 (w), 1480, 1460, 1430, 1400, 1390, 1380, 1370, 1115, 1230, 1200, 1130, 1055, 1005–900 cm^{-1}; ^1H NMR 1.03 (s, 6 H), 1.52 (s, 6 H), 1.40–1.60 (m, 2 H), 1.70–1.90 (dd, J = 6 Hz, J = 16 Hz, 2 H), 3.60 (s, 2 H), 4.23 (t, J = 6.10 Hz, 1 H), 7.40–7.60 (m, 3 H), 7.99 (m of d, J = 9 Hz, 2 H) ppm; ^{13}C NMR 26.43 (2 CH_3), 32.46 (2 CH_3), 38.96 (CH_2), 44.95 (2 C), 50.43 (2 CH_2), 74.95 (CH), 92.64 (C), 128.00 (2 CH), 128.59 (2 CH), 132.88 (CH), 138 (C), 197.39 (C=O) ppm; UV (c 9.19 × 10^{-5}, 9.19 × 10^{-3}, CH_3CN) λ 317 nm (ε 35), 287 (630), 278 (960), 270 (914), 241 (13 000).

Anal. Calcd for $C_{18}H_{24}O_2$: C, 79.41; H, 8.82. Found: C, 79.34; H, 8.84.

Fraction II, 70 mg (25%), was identified as (±)-(4-hydroxy-2,2,6,6-tetramethylcyclohexylidene)acetophenone (**3**).

Reaction of (±)-(4-Hydroxy-2,2,6,6-tetramethylcyclohexylidene)acetaldehyde (5) with NaH in the Presence of CH_3I. A mixture of (4-hydroxy-2,2,6,6-tetramethylcyclohexylidene)acetaldehyde (**5**) (392 mg), NaH (200 mg), and CH_3I (1 mL) in 10 mL of THF was stirred at 25 °C for 12 h. The reaction mixture was diluted with water and extracted with ether (three times). The ether layer washed with water, dried (Na_2SO_4), and concentrated. The ^1H NMR spectrum of the crude mixture showed the presence of two main components in the ratio of 65:35. Careful radial chromatography separation of the mixture using hexane–ether (10:1) gave two pure fractions.

The less polar fraction yielded 1-(2′-methoxy-*trans*-ethylene)-2,2,6,6-tetramethyl-7-oxabicyclo[2.2.1]heptane (**6**) (101 mg, 24%) as a solid: mp 68–70 °C; IR ($CHCl_3$) 2900 (m), 1695 (w), 1680, 1660, 1470, 1455, 1395, 1388, 1374, 1340, 1315, 1290, 1267, 1170, 1120, 1040–920, 872, 842 cm^{-1}; ^1H NMR 0.90 (s, 6 H), 1.23 (s, 6 H), 2.08 (d, J = 10.5 Hz, 2 H), 2.47 (dd, J = 6.10 Hz, J = 10.49 Hz, 2 H), 3.56 (s, 3 H), 4.35 (t, J = 5.86 Hz, 1 H), 4.70 (d, J = 12.69 Hz, 1 H), 6.47 (d, J = 12.69 Hz, 1 H) ppm; ^{13}C NMR 25.63 (2 CH_3), 33.27 (2 CH_3), 44.30 (2 C), 48.48 (2 CH_2), 56.15 (OCH_3), 75.05 (OCH), 97.53 (=CH), 113.70 (OC), and 147.86 (=$CHOCH_3$) ppm.

Anal. Calcd for $C_{13}H_{22}O_2$: C, 74.28; H, 10.47. Found: C, 74.30; H, 10.55.

More polar fraction gave 1-(2′-methoxy-*cis*-ethylene)-2,2,6,6-tetramethyl-7-oxabicyclo[2.2.1] heptane (**7**) (51 mg, 12%) as a colorless liquid: IR ($CHCl_3$) 2950 (m), 1678, 1470, 1405, 1385, 1370, 1285, 1155-1130, 1095, 1050–950, 870 cm^{-1}; ^1H NMR 1.01 (s, 6 H), 1.26 (s, 6 H), 1.33 (d, J = 10.74 Hz, 2 H), 1.75 (dd, J = 5.86 Hz, J = 10.50 Hz, 2 H), 3.58 (s, 3 H), 4.20 (d, J = 7.57 Hz, 1 H), 4.39 (t, J = 5.86 Hz, 1 H), 5.94 (d, J = 7.57 Hz, 1 H) ppm; ^{13}C NMR 25.52 (2 CH_3), 32.42 (2 CH_3), 45.24 (2 C), 48.08 (2 CH_2), 59.84 (OCH_3), 75.09 (OCH), 101.74 (=CH), 122.18 (OC), 146.07 (=C-$HOCH_3$) ppm.

Anal. Calcd for $C_{13}H_{22}O_2$: C, 74.28; H, 10.47. Found: C, 74.20; H, 10.61.

The Stereoselective Synthesis of Ethyl 2(*E*),4(*Z*)-Decadienoate

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Ethyl (2*E*,4*Z*)-decadienoate (**6**) is widely known as the chemical found in Bartlett pears (*Pyrus communis*) causing the characteristic taste.[1] Previous syntheses of **6** suffered from low yields[2] or low stereoselectivities.[3,4] Equally important is that the earlier work depended upon starting materials that were either expensive or not commercially available. We report a synthesis for ester **6** which alleviates the above shortcomings and provides a process versatile enough to accommodate the synthesis of other structurally similar compounds of interest to us.

The process (Scheme I) began with commercially available 1-heptyne (**1**), which was converted to the acetal 1,1-diethoxy-2-octyne (**2**). Acetal **2** was reduced stereoselectively to 1,1-diethoxy-2(*Z*)-octene (**3**), which was deprotected under mild conditions to 2(*Z*)-octenal (**4**). Reacting **4** with the carbanion generated from **5** gave the desired ester **6**.

Preparation of Alkynal Acetals. Howk and Sauer[5] described the preparation of 1,1-diethoxy-3-phenyl-2-propyne (**7**) in good yields (72–78%) starting with phenylacetylene and triethyl orthoformate, using zinc iodide as a catalyst. However, when the authors extended the reaction to the lower boiling 1-hexyne under "autogeneous" pressure (190 °C for 3 h), acetal **11** was obtained in only a 32% yield.

We were able to improve the yield of **11** to 42.8% by azeotropically removing ethanol with 1-hexyne to drive the reaction toward completion. This required greater than stoichiometric amounts of alkyne. Similarly, 1-heptyne and 1-octyne gave the corresponding acetals **2** and **10** in 70.2% and 74.3% yields, respectively. The higher yields

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(3) Ohloff, G.; Pawlak, M. *Helv. Chem. Acta* **1973**, *56*, 1176.
(4) Sensory evaluations of ethyl 2(*E*),4(*E*)-decadienoate demonstrated the negative influence it has on flavors containing greater than 25% of the 2*E*,4*E* isomer.
(5) Howk, B. W.; Sauer, J. C. *Organic Syntheses*; Wiley: New York, 1963; Collect. Vol. 4, p 801.

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