B. From 1c,9c-Dimethyl-7c-isopropenyl-10H*r*)-decal-3-one (2).[12f]—A solution of 100 mg (0.455 mmol) of ketone **2** in 10 ml of ether was reduced as outlined above to give 100 mg (98%) of alcohols (a 50:50 mixture of epimers by gas chromatographic analysis):[12d] λ_{max}^{film} 3.00 (OH), 3.25 (vinyl H), 6.07, 8.91, 9.57, 9.73, 10.14, and 11.26 μ.

The above mixture of alcohols was converted into the mesylate derivative as outlined above: λ_{max}^{film} 3.24, 6.08, 7.39, 8.50, 10.24, 10.6–11.2 (broad), 11.61, 12.80 and 13.7 μ.

A solution of this material in 0.7 ml of ethanol was reduced as outlined above affording, after chromatography and distillation, 61 mg (66%) of decalin **7**.[2]

3,3-(Propane-1,3-dithio)-10*r*-methyl-(9H*t*)-decal-2-one (9).[12f]—The procedure outlined above for the preparation of thioketal ketone **3** was followed using 265 mg of the hydroxymethylene ketone obtained (95% yield) from decalone **8**[15] by the procedure of Turner.[13] The crude product was filtered through 40 ml of Fisher alumina with 300 ml of benzene. Removal of the solvent afforded crystalline material which was recrystallized from ethanol affording 332 mg (90%) of thioketal ketone **9**. Recrystallization from hexane and sublimation at 80° (0.05 mm) afforded material of mp 133.5–135°; λ_{max}^{KBr} 5.92 (CO), 8.07, 8.46, 8.59, 8.94, 9.13, 9.33, 9.89, 10.88, 11.00, 11.41, 11.56, 12.20, 13.54, and 14.43 μ; $\delta_{TMS}^{CDCl_3}$ 1.07 ppm (C-10 CH_3).

Anal. Calcd for $C_{14}H_{22}OS_2$: C, 62.16; H, 8.20; S, 23.71. Found: C, 62.44; H, 8.26; S, 23.65.

3,3-(Propane-1,3-dithio)-10*r*-methyl(9H*t*)-decal-3*c*-ol (10).[12f]—A solution of 798 mg (2.95 mmol) of thioketal ketone **9** in 30 ml of ether was added dropwise to 114 mg (3.0 mmol) of lithium aluminum hydride in 30 ml of ether. The mixture was stirred for 15 hr, treated with water and base,[12e] stirred for 1 hr, and then dried over anhydrous magnesium sulfate. The salts were removed by filtration and washed well with ether and chloroform. Removal of the solvent afforded 800 mg (100%) of crystalline alcohol **10**. Two recrystallizations from hexane and sublimation at 80° (0.07 mm) afforded 536 mg of material with mp 105.5–107.5°; λ_{max}^{KBr} 2.85 (OH), 9.12, 9.29, 9.74, 9.93, 10.55, 10.91, 11.39, and 13.14 μ; $\delta_{TMS}^{CDCl_3}$ 3.83 (CHOH, X of ABX, $J_{AX} + J_{BX}$ = 15 Hz) and 1.11 ppm (C-10 CH_3).

Anal. Calcd for $C_{14}H_{24}OS_2$: C, 61.70; H, 8.88; S, 23.53. Found: C, 61.97; H, 8.96; S, 23.56.

3,3-Propane-1,3-dithio)-10*r*-methyl-(9H*t*)-decal-2*c*-yl Acetate (11).[12f]—A solution of 247 mg (0.945 mmol) of thioketal alcohol **10** and 200 mg of sodium acetate in 2.5 ml of acetic anhydride was heated at reflux for 4 hr.[12a] The cooled mixture was poured into aqueous sodium bicarbonate and stirred to hydrolyze the acetic anhydride. The aqueous layer was extracted with ether and the combined organic extracts were washed with aqueous sodium bicarbonate and brine and dried over anhydrous magnesium sulfate. Removal of the solvent afforded 300 mg (100%) of crystalline acetate **11**. Two recrystallizations from hexane and sublimation at 90° (0.07 mm) afforded 260 mg of material with mp 137.5–138.5°; λ_{max}^{KBr} 5.75 (CO), 8.09, 9.35, 9.69, 10.94, 11.15, 13.01, and 15.00 μ; $\delta_{TMS}^{CDCl_3}$ 5.16 (CHOAc, X of ABX, $J_{AX} + J_{BX}$ = 15 Hz), 2.14 (CH_3CO), and 1.14 ppm (C-10 CH_3).

Anal. Calcd for $C_{16}H_{26}O_2S_2$: C, 61.10; H, 8.33; S, 20.39. Found: C, 61.12; H, 8.27; S, 20.42.

2-Acetoxy-10*r*-methyl-(9H*t*)-decal-3-one (12).[12f]—Application of the hydrolysis procedure outlined above for thioketal **6** using 188 mg (0.60 mmol) of thioketal **11** afforded 139 mg (100%) of crystalline keto acetate **12** as a mixture of epimers. Two recrystallizations from hexane and sublimation at 80° (0.05 mm) afforded 113 mg (84%) of material with mp 137–148°; λ_{max}^{KBr} 5.71 (CO), 5.82 (CO), 7.97, 9.16, 9.49, 9.60, 9.97, 9.65, 11.13, and 15.15 μ; $\delta_{TMS}^{1:1\ CDCl_3-CCl_4}$ 5.4–5.0 (CHOAc, multiplet), 2.23 and 2.17 (CH_3CO), and 0.80 ppm (C-10 CH_3).

A 30-mg sample of this material was chromatographed on 10 ml of Fisher alumina. Elution with 10% ether in benzene afforded material, mp 145–149°, after recrystallization from hexane and sublimation at 90° (0.05 mm).

Anal. Calcd for $C_{13}H_{20}O_3$: C, 69.61; H, 8.99. Found: C, 69.65; H, 8.91.

10*r*-Methyl-(9H*t*)-decal-2-one (13).[12f]—A solution of 85 mg (0.38 mmol) of keto acetate **12** (a mixture of epimers) in 15 ml of ether was added over 10 min to a solution of 200 mg of calcium in 75 ml of distilled ammonia. The addition funnel was rinsed with 5 ml of ether. The mixture was stirred an additional 5 min and was then quenched with solid ammonium chloride. Brine was added to the residue which remained after evaporation of the ammonia, and the product was isolated with ether.[12d] Removal of the solvent afforded a mixture of alcohol and ketone **13**, λ_{max}^{film} 2.91 and 5.85 μ.

This mixture was dissolved in 5 ml of acetone and cooled to 0°. Jones reagent[16] (~10 drops) was added with rapid stirring until the red color remained. After stirring for 3 min, the mixture was treated with isopropyl alcohol to destroy the excess oxidizing agent and the product was isolated with ether.[12d] Short-path distillation at 70° (0.05 mm) afforded 51 mg (80%) of ketone **13** which was pure by gas chromatographic analysis:[12d] λ_{max}^{film} 5.82 (CO), 8.11, 8.40, 10.09, and 11.13 μ; $\delta_{TMS}^{CCl_4}$ 0.79 ppm (C-9 CH_3). The infrared and nmr spectra and the gas chromatographic retention time, by peak enhancement, were identical with those of an authentic sample of ketone **11**.[17]

Attempted Formation of Mesylate 14.—A solution of 90 mg (0.33 mmol) of thioketal alcohol **10** in 0.2 ml of pyridine was cooled to 0° and treated with 0.03 ml of methanesulfonyl chloride.[12a] The mixture was stirred for 2 hr at room temperature, ice was added, and stirring was continued for an additional 5 min. The mixture was poured into ether, washed with 4% sulfuric acid until acidic and with aqueous sodium bicarbonate until neutral, and dried over anhydrous magnesium sulfate. Removal of the solvent afforded 90 mg of a crystalline product: λ_{max}^{KBr} 6.19, 6.91, 7.03, 7.67, 7.92, 8.11, 10.05, 11.54, 11.44, 11.71, 13.31, and 14.20 μ; $\delta_{TMS}^{CDCl_3}$ 5.80 (C=CH, $W_{1/2}$ = 1.5 Hz), 3.88 (CHS, poorly resolved doublet, J = 4 Hz), and 0.96 ppm (C-10 CH_3).

Registry No.—**2**, 21736-21-0; 2,4-dinitrophenylhydrazone derivative of **2**, 21736-22-1; **3**, 21779-09-9; **4**, 21736-23-2; **5**, 21779-10-2; **6**, 21736-24-3; 2,4-dinitrophenylhydrazone of **6**, 21779-58-8; epimer of **6**, 21740-16-9; **7**, 21740-17-0; **9**, 21740-18-1; **10**, 21740-19-2; **11**, 21740-20-5; **12**, 21740-21-6; **13**, 21740-22-7.

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(15) *Cf.* M. Yanagita and K. Yamakawa, *J. Org. Chem.*, **21**, 500 (1956).

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(17) J. A. Marshall and R. D. Carroll, *J. Org. Chem.* **30**, 2748 (1965).

Enolene Rearrangements. II. Rearrangement of 3-Ethyl-4-pentenophenone to 3-Methyl-4-hexenophenone[1]

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The parent example of the "abnormal Claisen rearrangement" is now known to involve the isomerization of an *o*-(α-ethylallyl)phenol (**1a**, **1b**, or **1c**) to an *o*-(α,γ-dimethylallyl)phenol (**3a**, **3b**, or **3c**) by sigmatropic [1,5] hydrogen shifts, *via* a spirodienone intermediate (**2a**, **2b**, or **2c**), as shown in Scheme I.[2-6] In support of this mechanism, when the lower homologs **1d**

(1) Acknowledgment is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for support of this research.

(2) (a) W. M. Lauer and W. F. Filbert, *J. Amer. Chem. Soc.*, **58**, 1388 (1936); (b) C. D. Hurd and M. A. Pollack, *J. Org. Chem.*, **3**, 550 (1939); (c) E. N. Marvell, D. R. Anderson, and J. Ong, *ibid.*, **27**, 1109 (1962).

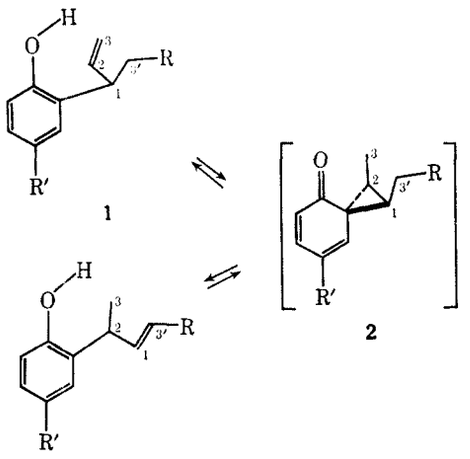
(3) W. M. Lauer and H. E. Ungenade, *J. Amer. Chem. Soc.*, **61**, 3047 (1939).

(4) R. M. Roberts and R. G. Landolt, *J. Org. Chem.*, **31**, 2699 (1966).

(5) A. Habich, R. Barner, R. M. Roberts, and H. Schmid, *Helv. Chim. Acta*, **45**, 1943 (1962).

(6) W. M. Lauer and T. A. Johnson, *J. Org. Chem.*, **28**, 2913 (1963).

SCHEME I



a, R = CH_3; R′ = H[2]
b, R = CH_3; R′ = $CO_2C_2H_5$[3]
c, R = CH_3; R′ = CH_3[4]
d, R = H; R′ = CH_3[5]
e, R = H; R′ = $CO_2C_2H_5$[6]

and **1e**, initially labeled with ^{14}C at $C_{3'}$, were heated, the isotopic carbon became equilibrated between $C_{3'}$ and C_3 as expected, since these carbons become equivalent in the intermediates **2d** and **2e**.[5,6] Similar evidence and further insight into the mechanism was given by experiments in which exchange of deuterium between O, $C_{3'}$, and C_3 in **1d** was monitored by means of nmr spectroscopy.[7]

Evidence of analogous rearrangements in an aliphatic (enolene) system was sought and found using the nmr-traced deuterium-labeling technique.[8] Exchange of deuterium between C_α, $C_{3'}$, and C_3 in homoallylic ketones was observed, as expected, on the basis of the mechanism outlined in Scheme II (*i.e.*, **4a** ⇄ **8a**). A demonstration of the *skeletal* rearrangement in the enolene system corresponding to the one responsible for the "abnormal Claisen rearrangement" is the subject of this report, *i.e.*, the isomerization of 3-ethyl-4-pentenophenone (**4b**) into 3-methyl-4-hexenophenone (**8b**).

3-Ethyl-4-pentenophenone was prepared by the addition of vinylmagnesium chloride to 2-pentenophenone.[9] The reported synthesis of 2-pentenophenone[10] was not satisfactory, but this compound was obtained in good yield by a Wittig synthesis from α-bromoacetophenone and propanal. Authentic 3-methyl-4-hexenophenone was prepared by the imine alkylation procedure[11] from acetophenone and 4-chloro-2-pentene.

Thermal rearrangement of neat 3-ethyl-4-pentenophenone was carried out at two temperatures, 192 ± 0.5° (refluxing decalin) and 217 ± 0.5° (refluxing naphthalene), and the progress of the isomerization was determined by capillary gas chromatography. The results are presented in Table I.

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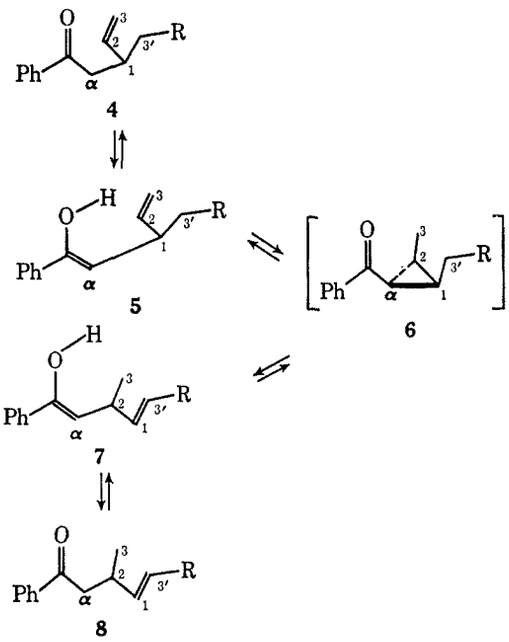
(8) R. M. Roberts, R. G. Landolt, R. N. Greene, and E. W. Heyer, *J. Amer. Chem. Soc.*, **89**, 1404 (1967).
(9) (a) A. J. Birch and M. Smith, *Proc. Chem. Soc.* (London), 356 (1962); (b) H. O. House, *J. Org. Chem.*, **28**, 348 (1963); (c) J. A. Marshall, *ibid.*, **31**, 667 (1966).
(10) E. Benary, *Ber.*, **64B**, 2543 (1931).
(11) G. Stork and S. R. Dowd, *J. Amer. Chem. Soc.*, **85**, 2178 (1963).

SCHEME II



a, R = H,D
b, R = CH_3

TABLE I

THERMAL REARRANGEMENT OF 3-ETHYL-4-PENTENOPHENONE (**4b**) INTO 3-METHYL-4-HEXENOPHENONE (**8b**)

	8b, %[a]		**4b** + **8b**, %[b]	
Time, hr	192°	217°	192°	217°
25	12 (3/97)	54 (7/93)	99	92
50	23 (3/97)		96	
53		81 (14/86)		82
75	33 (4/96)	91 (20/80)	96	66
100	42 (5/95)	94 (23/77)	92	73
173	60 (9/91)		92	

[a] Percentage of **8b** in the mixture of **8b** and **4b**, by capillary glpc (see Experimental Section). The figures in parentheses are the *cis/trans* proportions of **8b**. [b] Percentage of the total glpc eluents accounted for by **4b** and **8b**.

It is interesting to note that the equilibrium ratio of 3-methyl-4-hexenophenone and 3-ethyl-4-pentenophenone (94:6) is very similar to the equilibrium ratio of the analogous allylic phenols *o*-(α,γ-dimethylallyl)-*p*-cresol and *o*-(α-ethylallyl)-*p*-cresol (96:4).[4]

Experimental Section[12,13]

2-Pentenophenone[9] was prepared in 76% yield by the reaction of benzoylmethylene triphenylphosphorane (0.121 mol) and propanal (0.40 mol) in dry benzene, using the procedure of Ramirez and Dershowitz.[14] The product, bp 74–76° (15 μ), gave a single but shouldered glpc peak; the shoulder was probably due to *cis* and *trans* isomers. The nmr spectrum (neat) showed the presence of minor impurities, but was interpretable in terms of the

(12) A Beckman IR-5A instrument was used for ir analysis; Varian A-60 and HA-100 spectrometers were used for nmr measurements, with TMS as internal standard. Gas chromatographs employed for routine analysis and preparative work were Beckman GC-2A, Wilkens 600-D (Hy-Fi), and Wilkens A-700 (Autoprep). The analyses of the thermolysis mixtures were done on a Perkin-Elmer 900 capillary gas chromatograph equipped with a digital integrator.[13] The capillary column (200 ft × 0.02 in.) was coated with di-*n*-decyl phthalate. Capillary melting points are uncorrected. Microanalyses were performed by Galbraith Laboratories, Knoxville, Tenn.
(13) The authors express their appreciation to Mr. Bernard Young and Mr. Birt Allison of Cosden Oil and Chemical Co., Big Spring, Texas, for making the use of this instrument available to us.
(14) F. Ramirez and S. Dershowitz, *J. Org. Chem.*, **22**, 41 (1957).

desired structure: δ 0.9 (t, 3, CH_3), 2.0 (m, 2, CH_2), 6.7 (m, 2, CH=CH), 7.1, and 7.7 ppm (2 m, 5, C_6H_5).

3-Ethyl-4-pentenophenone.—2-Pentenophenone (0.092 mol), dry tetrahydrofuran (150 ml), and cupric acetate monohydrate (1.0 g) were stirred together under a nitrogen atmosphere at −70°, and 150 ml of a 2 *M* solution of vinylmagnesium chloride in tetrahydrofuran[15] was added over a 2-hr period. The reaction mixture was warmed to room temperature, stirred for an additional 3 hr, and hydrolyzed with saturated ammonium chloride solution (250 ml). The phases were separated and the aqueous one was extracted with ether; the organic phases were combined and concentrated. Subsequent vacuum distillation gave material, bp 88° (0.03 mm), which was rich in aromatic ketone, according to its ir spectrum, but which was shown to contain several minor impurities by glpc analysis. Careful column chromatography (twice) on silica gel followed by vacuum distillation gave 4.86 g of material, $n^{23}D$ 1.5193, 99% pure (glpc). The nmr spectrum (neat) was consistent with the structure of 3-ethyl-4-pentenophenone: δ 0.9 (t, 3, CH_3), 1.4 (m, 2, CH_2), 2.8 (m, 3, CH_2—CH), 4.9 (m, 2, $=CH_2$), 5.7 (m, 1, CH=), 7.3 and 7.9 ppm (2 m, 5, C_6H_5). The ir spectrum showed strong absorptions at 1690 cm^{-1} (C=O) and at 915 and 980 cm^{-1} (terminal vinyl stretching).

Anal. Calcd for $C_{13}H_{16}O$: C, 82.92; H, 8.58. Found: C, 82.94; H, 8.67.

The 2,4-dinitrophenylhydrazone of this ketone was prepared and recrystallized from ethanol–ethyl acetate to give fine red-orange needles, mp 148.5–150.5°.

Anal. Calcd for $C_{19}H_{20}N_4O_4$: C, 61.92; H, 5.47. Found: C, 61.75; H, 5.52.

3-Methyl-4-hexenophenone.—Acetophenone (240 g, 2.0 mol), cyclohexylamine (485 ml, 4.0 mol), and dry benzene (300 ml) were heated together in a flask fitted with a Dean–Stark tube for 100 hr, after which time 35 ml (97% of the theoretical amount) of water had been collected. Subsequent vacuum distillation gave 291 g (72.2%) of product, bp 125–135° (0.2 mm). Infrared analysis (strong absorption at 1640 cm^{-1}, C=N) showed this distillate to be the desired imine, uncontaminated by ketone.

Ethyl bromide (40 ml, 0.55 mol) was added dropwise to a stirred mixture of magnesium turnings (12.0 g, 0.50 g-atom) and dry tetrahydrofuran (160 ml) at room temperature over a 1-hr period. The resulting mixture was stirred overnight to complete the consumption of the magnesium.

The ethylmagnesium bromide solution was heated to gentle reflux, and the acetophenone cyclohexylimine (86.3 g, 0.43 mol) was added dropwise over a period of 45 min. The resulting reaction mixture was heated under reflux for 24 hr and then cooled to ambient temperature. 4-Chloro-2-pentene (44.0 g, 0.42 mol) was added dropwise, and the resulting mixture was heated to reflux for 2 hr, cooled to room temperature, and hydrolyzed with 10% hydrochloric acid (300 ml). The phases were separated and the aqueous one was extracted with ether; the combined organic phases were washed successively with 5% sodium bicarbonate and water, then dried over anhydrous calcium chloride, and concentrated in a rotary evaporator. Vacuum distillation followed by spinning-band fractionation gave 14.8 g (18.7%) of distillate, bp 80° (0.1 mm), $n^{26.5}D$ 1.5215, which was pure according to glpc analysis. The nmr spectrum (neat) was consistent with the structure of 3-methyl-4-hexenophenone: δ 1.0 (d, 3, CH—CH_3), 1.55 (d, 3, CH=CH—CH_3), 2.86 (m, 3, CH_2—CH), 5.5 (m, 2, CH=CH), 7.4 and 7.9 ppm (2 m, 5, C_6H_5). The ir spectrum showed strong absorptions at 1695 cm^{-1} (aromatic ketone) and 970 cm^{-1} (*trans* C=C), as well as the characteristic aromatic absorptions.

Anal. Calcd for $C_{13}H_{16}O$: C, 82.92; H, 8.58. Found: C, 82.69; H, 8.61.

The 2,4-dinitrophenylhydrazone was prepared; recrystallization from ethanol–ethyl acetate gave red-orange prisms, mp 128–129.5°.

Anal. Calcd for $C_{19}H_{20}N_4O_4$: C, 61.92; H, 5.47. Found: C, 61.65; H, 5.46.

Thermal Rearrangements.—The thermolyses were carried out at 192 ± 0.5° and 217 ± 0.5° using refluxing decalin (*cis–trans* mixture) and naphthalene, respectively, as constant-temperature baths. Samples for rearrangement experiments consisted of 50-μl portions of 3-ethyl-4-pentenophenone sealed in Pyrex tubes at a pressure ≤2 μ after degassing by alternately freezing (in liquid nitrogen) and thawing repeatedly.

(15) H. Normant, *Advan. Org. Chem.*, **2**, 37 (1960).

Experiments on glpc analysis of mixtures of authentic 3-ethyl-4-pentenophenone and 3-methyl-4-hexenophenone using conventional, packed columns showed that partial but not base-line separation could be achieved. Using the optimum glpc conditions so determined for analysis, preliminary thermolyses of 3-ethyl-4-pentenophenone were carried out. These showed that rearrangement into 3-methyl-4-pentenophenone occurred cleanly at 192°, but that equilibration was incomplete even after 173 hr. On the other hand, heating at 217° produced equilibration in *ca.* 50 hr, but gave rise to an appreciable amount of side products.

Capillary glpc analyses[12,13] provided more quantitative rearrangement data. Authentic 3-ethyl-4-pentenophenone (99.2% pure) had a retention time of 40.8 min under the conditions chosen for the analyses. Authentic 3-methyl-4-hexenophenone was found to consist of two major components, one comprised 85.7% of the material, with a retention time of 41.6 min, and the other 10.3%, with a retention time of 37.2 min. On the basis of other data, it was presumed that the major component was the *trans* isomer and the minor one was the *cis* isomer of 3-methyl-4-hexenophenone.

The data obtained at 192 ± 0.5° and 217 ± 0.5° are presented in Table I.

Registry No.—**4b**, 21779-18-0; **4b** (2,4-dinitrophenylhydrazone), 21779-19-1; **8b**, *cis*, 21779-20-4; **8b**, *cis* (2,4-dinitrophenylhydrazone), 21779-21-5; **8b**, *trans*, 21779-22-6; **8b**, *trans* (2,4-dinitrophenylhydrazone), 21779-23-7.

Keto Tosylates. I. Monotosylation of *cis*-3-(2-Hydroxyethyl)cyclopentanol

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In connection with our study of the reactions of keto tosylates and enol tosylates, we have synthesized 3-(2-tosyloxyethyl)cyclopentanone (**5**) utilizing a route that depended upon the tosylation of a specific group of a diol (see Scheme I). According to this synthetic scheme, norcamphor (**1**) underwent a Baeyer–Villiger reaction to give *cis*-3-hydroxycyclopentylacetic acid lactone (**2**) in a yield of 25% after distillation using *m*-chloroperbenzoic acid as the oxidizing agent.[1] The lactone **2** was reduced with lithium aluminum hydride into give *cis*-3-(2-hydroxyethyl)cyclopentanol (**3**) in 94% yield after distillation.[2] The diol **3** could be converted into the primary monotosylate, *cis*-3-(2-tosyloxyethyl)-cyclopentanol (**4**), in 59% yield when a solution of *p*-toluenesulfonyl chloride in pyridine was added slowly to a cold solution of the diol **3** in pyridine. The last step, the oxidation of **4** to 3-(2-tosyloxyethyl)cyclopentanone (**5**), went smoothly in a yield of 89% according to the procedure of Nelson.[3]

The success of the selective monotosylation of **3** to give **4** was found to depend critically upon the type of tosyl chloride addition. In contrast to previous mono-

(1) J. Meinwald and E. Frauenglass, *J. Amer. Chem. Soc.*, **82**, 5235 (1960). Meinwald reports a higher yield, but a cruder product, when using peracetic acid on norcamphor.

(2) A. Rassat and G. Ourisson [*Bull. Soc. Chim. Fr.*, 1133 (1959)] prepared **3** by this procedure, but they did not purify the product nor did they give a yield.

(3) N. A. Nelson and G. A. Mortimer, *J. Org. Chem.*, **22**, 1146 (1957).