solvent was evaporated at reduced pressure, and the residue was treated with H_2O, extracted with ethyl acetate or diethyl ether, and dried (Na_2SO_4). Evaporation of the solvent gave the crude product, which was further purified by column chromatography.

Indol-2-yldiphenylmethanol (5a): separated by column chromatography ($CHCl_3$ as eluate); granule (from hexane); mp 136–137 °C (lit.[11] mp 136–139.5 °C); ^1H NMR ($CDCl_3$) δ 3.20 (s, 1 H, OH), 6.03 (d, J = 2 Hz, indole-3H), 7.01–7.47 (m, 14 H), 8.28 (bs, 1 H, NH); ^{13}C NMR ($CDCl_3$) δ 77.2, 103.4, 111.0, 119.8, 120.7, 122.2, 127.1, 127.6, 128.0, 128.2, 136.0, 142.6, 145.1.

Indol-2-ylphenylmethanol (5b): separated by column chromatography ($CHCl_3$ as eluate); oil (lit.[8a] oil); ^1H NMR ($CDCl_3$) δ 5.75 (s, 1 H, OH), 5.95 (s, 1 H, CH), 6.17 (s, 1 H, indole-3H), 7.04–7.28 (m, 8 H), 7.49 (d, J = 7.6 Hz, 1 H), 8.21 (bs, 1 H, NH); ^{13}C NMR ($CDCl_3$) δ 70.6, 100.8, 111.0, 119.8, 120.5, 122.0, 126.5, 127.8, 127.9, 128.5, 136.1, 140.1, 141.5.

Indol-2-yl-*p*-tolylmethanol (5c): separated by column chromatography ($CHCl_3$–CH_3OH, 19.9:0.1); oil; ^1H NMR ($CDCl_3$) δ 2.29 (s, 3 H), 3.19 (bs, 1 H, OH), 5.74 (s, 1 H, CH), 6.17 (d, J = 2 Hz, 1 H, indole-3H), 6.97–7.19 (m, 7 H), 7.50 (d, J = 8 Hz, 1 H), 8.29 (s, 1 H, NH); ^{13}C NMR ($CDCl_3$) δ 21.1 (CH_3), 70.5, 100.7, 111.0, 119.7, 120.5, 121.9, 126.5, 128.0, 129.2, 136.1, 137.8, 138.6, 140.3; mass spectrum, m/z (relative intensity) 237 (M^+, 64), 220 (70), 204 (100), 130 (24), 120 (38), 119 (60), 91 (82), 79 (20), 65 (31); MS (HR) calcd for $C_{16}H_{15}NO$ 237.1153, found 237.1146.

2-(Phenylthio)indole (5d): separated by column chromatography ($CHCl_3$ as eluate); needles; mp 64–66 °C; ^1H NMR ($CDCl_3$) δ 6.84 (d, J = 2 Hz, 1 H, indole-3H), 7.01–7.75 (m, 9 H), 7.99 (bs, 1 H, NH); ^{13}C NMR ($CDCl_3$) δ 110.9, 111.5, 120.2, 120.7, 123.2, 126.1, 127.4, 128.4, 129.0, 129.1, 136.6, 137.7; mass spectrum, m/z (relative intensity) 225 (40), 117 (3), 110 (24), 77 (11), 65 (7), 51 (4), 39 (6); MS (HR) calcd for $C_{14}H_{11}NS$ 225.0612, found 225.0615.

2-Methylindole (5e): separated by column chromatography ($CHCl_3$ as eluate); mp 58–60 °C (lit.[9a] mp 58–60 °C); ^1H NMR ($CDCl_3$) δ 2.41 (s, 3 H, CH_3), 6.20 (d, J = 3 Hz, indole-3H), 7.07–7.52 (m, 4 H), 7.85 (br, 1 H, NH); ^{13}C NMR ($CDCl_3$) δ 13.7, 100.3, 110.2, 118.9, 119.5, 120.8, 129.0, 135.0, 136.7.

Indol-2-yl(4-methoxyphenyl)methanol (5f): separated by column chromatography ($CHCl_3$–CH_3OH, 19.9:0.1); mp 96–98 °C (lit.[8a] mp 98–100 °C); ^1H NMR ($CDCl_3$) δ 3.01 (bs, 1 H, OH), 3.72 (s, 3 H, CH_3), 5.75 (s, 1 H, CH), 6.18 (d, J = 2 Hz, indole-3H), 6.78 (d, J = 8 Hz, 2 H), 7.05–7.51 (m, 6 H), 8.28 (bs, 1 H, NH); ^{13}C NMR ($CDCl_3$) δ 55.2, 70.3, 100.6, 111.0, 119.1, 119.8, 120.5, 121.9, 127.9, 128.0, 133.8, 136.1, 140.4, 159.0.

Registry No. 3, 5379-79-3; **4a**, 126594-12-5; **4b**, 126754-44-7; **4c**, 126754-45-8; **4d**, 126754-46-9; **4e**, 126754-47-0; **4f**, 126754-48-1; **5a**, 20538-21-0; **5b**, 40900-00-3; **5c**, 126754-49-2; **5d**, 120517-31-9; **5e**, 95-20-5; **5f**, 40900-01-4; $(C_6H_5)_2CO$, 119-61-9; C_6H_5CHO, 100-52-7; 4-$CH_3C_6H_4CHO$, 104-87-0; $(C_6H_5S)_2$, 882-33-7; 4-$CH_3OC_6H_4CHO$, 123-11-5; indole, 120-72-9; gramine, 87-52-5.

Supplementary Material Available: ^{13}C NMR spectra for compounds **4d**, **4e**, **5c**, **5d**, and **5f** (5 pages). Ordering information is given on any current masthead page.

(11) Dolby, L. J.; Lord, P. D. *J. Org. Chem.* **1969**, *34*, 2988.

Single-Step Removal of the Allyl Ether Protecting Group with $(Ph_3P)_4RhH$ and Trifluoroacetic Acid

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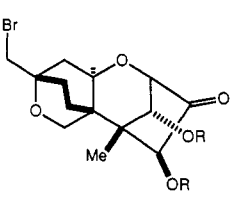
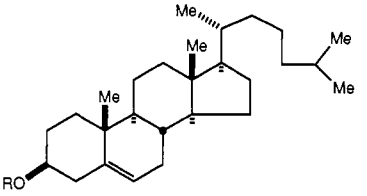
During a study directed toward the synthesis of the highly functionalized trichothecene anguidine and its congeners,[2] we were confronted with the problem of removal of the allyl protecting groups in tetracycle **1a**. A number of procedures for this transformation have been reported,[3] but only the method of Corey and Suggs[3c] was deemed mild enough for our substrate. This procedure effects isomerization of the double bond of the allyl group to the vinyl ether with Wilkinson's catalyst [$(Ph_3P)_3RhCl$] in refluxing ethanol in the presence of diazabicyclooctane (DABCO). The base prevents premature liberation of propionaldehyde, the byproduct of hydrolysis which undergoes decarbonylation in the presence of the catalyst and thereby generates a less active catalyst.[3c] Although isomerization of the double bonds of **1a** proved successful, acidic hydrolysis of the vinyl ethers or ozonolysis followed by deformylation (K_2CO_3/MeOH) gave complex mixtures of products.



1a, R = $CH_2CH{=}CH_2$
b, R = H



2a, R = $CH_2CH{=}CH_2$
b, R = $CH_2C(CH_3){=}CH_2$
c, R = $CH_2CH{=}CHCH_3$ (E + Z)
d, R = $CH_2CH{=}C(CH_3)_2$
e, R = H

A recent report by Sundberg[4] demonstrated that a tertiary allylic amine can be deprotected with 25 mol % of hydridotetrakis(triphenylphosphine)rhodium[5,6] [$(Ph_3P)_4RhH$] in the presence of trifluoroacetic acid in refluxing ethanol. When this procedure was applied to bis allyl ether **1a**, (cat. 25 mol %; 100 equiv of TFA), the diol **1b** was isolated directly in 72% yield.

To explore the reactivity of this catalytic system, we prepared the series of allyl cholesteryl ethers **2a–d** by the Williamson procedure (KH, THF, reflux; allylic halide). Each deprotection was conducted in refluxing ethanol for 30 min using equal amounts of substrate and trifluoroacetic acid and only 3 mol % of catalyst. Table I reveals the relative reactivity of the four allyl ethers under these conditions. Clearly, the allyl ether **2a** is cleaved faster than the mono-methyl-substituted allyl ethers **2b** and **2c**, and

Table I. Isomerization of Cholesteryl Allyl Ethers[a]

ether	isolated yields, % (ether/**2e**)
2a	0/98
2b	66/16
2c[b]	80/9
2d	93/4

[a] Conditions: see Experimental Section. In the absence of catalyst and the presence of TFA, a <4% yield of cholesterol was obtained in 6.5 h. [b] Mixture of E/Z (~5:1) in starting material and recovered **2c**.

(1) Recipient of a Dox Fellowship, 1987–1988. Taken in part from the Ph.D. Thesis of S.B.S., Yale University, 1989.
(2) Ziegler, F. E.; Sobolov, S. B. *J. Am. Chem. Soc.* **1990**, *112*, 2749.
(3) (a) Price, C. C.; Snyder, W. H. *J. Am. Chem. Soc.* **1961**, *83*, 1773. (b) Boss, R.; Scheffold, R. *Angew. Chem., Int. Ed. Engl.* **1976**, *15*, 558. (c) Corey, E. J.; Suggs, W. J. *J. Org. Chem.* **1973**, *38*, 3224. (d) Kariyone, K.; Yazawa, H. *Tetrahedron Lett.* **1970**, 2885.
(4) Sundberg, R. J.; Hamilton, G. S.; Laurino, J. P. *J. Org. Chem.* **1988**, *53*, 976.
(5) Ahmed, N.; Robinson, S. D.; Uttley, M. F. *J. Chem. Soc., Dalton Trans.* **1972**, 843.
(6) For the isomerization of *N*-acylallylamines with this catalyst, see: Stille, J. K.; Becker, Y. *J. Org. Chem.* **1980**, *45*, 2139.

0022-3263/90/1955-3691$02.50/0

much more rapidly than the disubstituted prenyl ether **2d**. This difference in relative rates suggests the use of these moieties as selective protecting groups.[7]

The catalyst in the absence of trifluoroacetic acid failed to effect isomerization. However, when 5 mol % of the catalyst was treated with 5 mol % of trifluoroacetic acid in ethanol, effervescence occurred as the mixture changed from a yellow suspension to an orange solution.[8] Addition of 100 mol% of allyl ether **2a** resulted in isomerization to a mixture (~1:1) of vinyl ethers upon heating at reflux. Application of Wilkinson's catalyst (5 mol %, with DABCO) under similar conditions affected isomerization at half the rate of the new system. As in the case of Wilkinson's catalyst, the activity of the $(Ph_3P)_4RhH$/TFA catalyst is reduced when the isomerization is conducted in the presence of propionaldehyde (100 mol %). In addition, "old" ethanol that may have undergone partial oxidation should be avoided as solvent.

Experimental Section

General Methods. All reactions were performed in oven-dried glassware under N_2. Tetrahydrofuran (THF) was distilled from sodium/benzophenone ketyl under nitrogen prior to use. Absolute (200 proof) ethanol was used as received. Cholesterol (Fisher) was recrystallized twice from absolute ethanol and dried under vacuum to give material melting at 145.5–146.5 °C. Potassium hydride (Aldrich) was cautiously washed with dry hexane (CaH_2), diluted with dry THF, and the suspension titrated with dry 2-butanol. The liberated hydrogen was measured volumetrically (buret). All other reagents and solvents were used as received.

The following equipment, instrumentation, and services were employed. IR spectra were obtained by using a Nicolet 5-SX FT spectrometer; ^1H NMR spectra were recorded on a Bruker WM 250-MHz spectrometer with spectra referenced to $CHCl_3$ (δ 7.27); low-resolution mass spectra (EI) were obtained on a Hewlett-Packard 5989 instrument; high-resolution spectra were recorded on a Kratos MS-80 RFA instrument; elemental analyses were performed by Atlantic Microlabs, Inc., Atlanta, GA; melting points (uncorrected) were measured by Fisher-Johns apparatus.

Allyl Cholesteryl Ether (2a). An oven-dried, 100-mL round-bottomed flask containing a magnetic stirring bar and serum cap was charged with a suspension of KH in oil (2.08 g, 10.9 mmol), which was then washed (hexane) free of oil. The apparatus was attached to a nitrogen line via a syringe needle. Dry THF (15 mL) was added, and the suspension was stirred rapidly at room temperature as a solution of cholesterol (1.00 g, 2.59 mmol) in THF (15 mL) was added via syringe over 1 min (mild effervescence). When gas evolution had ceased (5 min), allyl bromide (0.94 mL, 10.9 mmol) was added to the solution via syringe, a nitrogen-flushed reflux condenser was attached to the reaction flask, and the mixture was heated at reflux for 10 h. The cooled mixture was cautiously decomposed with 2-propanol and concentrated in vacuo. The pasty residue was partitioned between water and ether, and the aqueous portion was extracted with ether (2×). The combined organic portions were washed with water and brine and were dried over anhydrous K_2CO_3. Filtration and concentration gave crude product (1.07 g, 97%) as a pale yellow oil. Recrystallization from absolute ethanol yielded allyl cholesteryl ether as a white, crystalline solid (734 mg, 66.5%): mp 76–77 °C; ^1H NMR ($CDCl_3$) δ 5.95 (1 H, ddt, J = 17.3, 10.3, 5.6 Hz, H_2C=C*H*CH_2), 5.36 (1 H, br d, J = 5.4 Hz, C_6-H), 5.29 (1 H, dd, J = 17.3, 1.6 Hz, H*H*C=$CHCH_2$), 5.17 (1 H, br d, J = 10.3 Hz, *H*HC=$CHCH_2$), 4.04 (2 H, d, J = 5.6 Hz, CH_2O), 3.23 (1 H, m, R_2CHOR), 2.40 (1 H, m), 2.23 (1 H, m), 2.08–1.76 (5 H, m), 1.68–0.97 (21 H, m), 1.03 (3 H, s), 0.96 (3 H, d, J = 7.1 Hz), 0.89 (6 H, 2 overlapping d's, J = 7.2 Hz), 0.71 (3 H, s); IR (neat melt on NaCl) 2949, 2937, 2867, 1648, 1467, 1437, 1379, 1373, 1361, 1338, 1139, 1098, 1074, 1016, 980, 916, 840, 799 cm^{-1}; LRMS, m/z 426 (M^+, 21.1), 371 (29.5), 370 (100.0), 369 (38.6), 368 (35.3), 355 (37.8), 353 (21.1); HRMS calcd for $C_{30}H_{50}O$ 426.3864, found 426.3848. Anal. Calcd for $C_{30}H_{50}O$: C, 84.44; H, 11.81. Found: C, 84.35; H, 11.76.

Cholesteryl β-Methallyl Ether (2b). This ether was prepared in 99% crude yield by the above procedure, utilizing cholesterol (1.00 g, 2.59 mmol), KH in oil (2.08 g, 10.9 mmol), and methallyl chloride (1.06 mL, 10.9 mmol) in THF (35 mL). Recrystallization from absolute ethanol yielded the ether as a white, crystalline solid (877 mg, 77%): mp 77.5–78.5 °C; ^1H NMR ($CDCl_3$) δ 5.35 (1 H, br d, J = 5.1 Hz, C_6-H), 4.98 (1 H, br s, H*H*C=C), 4.87 (1 H, br s, *H*HC=C), 3.93 (2 H, br s, CH_2O), 3.20 (1 H, m, R_2CHOR), 2.39 (1 H, m), 2.23 (1 H, m), 2.09–1.66 (5 H, m), 1.77 (3 H, s, CH_3C=C), 1.65–0.97 (21 H, m), 1.04 (3 H, s), 0.93 (3 H, d, J = 7.1 Hz), 0.89 (6 H, 2 overlapping d's, J = 6.9 Hz), 0.70 (3 H, s); IR (neat melt on NaCl) 2949, 2931, 2867, 1654, 1467, 1432, 1379, 1373, 1367, 1338, 1191, 1121, 1103, 1027, 893, 840, 799, 734 cm^{-1}; LRMS, m/z 440 (M^+, 9.7), 371 (28.1), 370 (100.0), 369 (29.4), 368 (18.3), 355 (33.6), 329 (14.1), 55 (47.9); HRMS calcd for $C_{31}H_{52}O$ 440.4021, found 440.4032. Anal. Calcd for $C_{31}H_{52}O$: C, 84.48; H, 11.89. Found: C, 84.34; H, 11.83.

Cholesteryl γ-Methallyl Ether (2c). The ether was prepared in 99% crude yield (vide supra) from cholesterol (1.00 g, 2.59 mmol), KH in oil (2.08 g, 10.9 mmol), and a commercial mixture of (*E*)- and (*Z*)-2-butenyl bromide and 3-bromo-1-butene (7:1:1) (1.24 mL, 10.9 mmol) in THF (35 mL). Recrystallization (2×) from absolute ethanol yielded a 6:1 mixture of (*E*)- and (*Z*)-cholesteryl γ-methallyl ethers as a white, crystalline solid (457 mg, 40.1%): mp 57.5–60.5 °C; ^1H NMR ($CDCl_3$, *E* isomer) δ 5.82–5.54 (2 H, m, RHC=CHR'), 5.37 (1 H, br d, J = 5.3 Hz, C_6-H), 3.95 (2 H, d, J = 6.0 Hz, CH_2O), 3.21 (1 H, m, R_2CHOR), 2.39 (1 H, m), 2.21 (1 H, m), 2.10–1.80 (5 H, m), 1.71 (3 H, d, J = 6.7 Hz, C*H*$_3$HC=C), 1.70–0.96 (21 H, m), 1.03 (3 H, s), 0.93 (3 H, d, J = 6.5 Hz), 0.88 (6 H, 2 overlapping d's, J = 7.1 Hz), 0.70 (3 H, s); [*Z*-isomer spectrum similar to that of *E* isomer, except 3.95 (2 H, d, J = 6.0 Hz, CH_2O) resonance replaced by 4.10 (d, 2 H, J = 5.5 Hz, CH_2O)]; IR (neat melt on NaCl) 2937, 2902, 2867, 2849, 1672, 1467, 1443, 1379, 1361, 1344, 1133, 1098, 1068, 1021, 963, 840, 799 cm^{-1}; LRMS, m/z 440 (M^+, 21.0), 371 (25.1), 370 (74.6), 355 (25.9), 55 (100.0); HRMS calcd for $C_{31}H_{52}O$ 440.4021, found 440.4012. Anal. Calcd for $C_{31}H_{52}O$: C, 84.48; H, 11.89. Found: C, 84.36; H, 11.86.

Cholesteryl γ,γ-Dimethylallyl Ether (2d). The ether was prepared (vide supra) from cholesterol (1.00 g, 2.59 mmol), KH in oil (2.08 g, 10.9 mmol KH), and γ,γ-dimethylallyl bromide (1.26 mL, 10.9 mmol) in THF (35 mL). Recrystallization from absolute ethanol (15 mL) yielded the ether as a white, crystalline solid (940 mg, 80%): mp 60–61 °C; ^1H NMR ($CDCl_3$) δ 5.38 (2 H, m, olefins), 4.02 (2 H, d, J = 6.9 Hz, CH_2O), 321 (1 H, m, R_2CHOR), 2.40 (1 H, m), 2.23 (1 H, m), 2.08–1.79 (5 H, m), 1.75 (3 H, s, CH_3C=C), 1.69 (3 H, s, CH_3C=C), 1.69–0.97 (21 H, m), 1.02 (3 H, s), 0.93 (3 H, d, J = 6.8 Hz), 0.88 (6 H, 2 overlapping d's, J = 7.1 Hz), 0.70 (3 H, s); IR (neat melt on NaCl) 2937, 2908, 2867, 2844, 1672, 1467, 1379, 1367, 1197, 1086, 1021, 834, 799 cm^{-1}; LRMS, m/z 454 (M^+, 17.5), 439 (11.3), 371 (32.1), 370 (100.0), 369 (16.1), 367 (10.6), 355 (27.8), 69 (56.9); HRMS calcd for $C_{32}H_{54}O$ 454.4177, found 454.4182. Anal. Calcd for $C_{32}H_{54}O$: C, 84.51; H, 11.97. Found: C, 84.41; H, 11.94.

Deprotection of Allyl Cholesteryl Ether. To a solution of allyl cholesteryl ether **2a** (100 mg, 0.234 mmol) in absolute ethanol at 50 °C were added $(Ph_3P)_4RhH$ (6.9 mg, 0.006 mmol) and F_3CCO_2H (18.5 μL, 0.234 mmol). The solution was successively heated at reflux for 30 min, cooled to room temperature, and concentrated in vacuo. The residual yellow-orange gum was purified by flash chromatography (Et_2O), to provide 89.5 mg (98%) of cholesterol (**2e**) (TLC, ^1H NMR).

Acknowledgment. This work was supported by a grant from the National Cancer Institute, NIH (CA-39976). S.B.S. expresses her gratitude for the receipt of a Dox Fellowship (Yale).

Registry No. **1a**, 126786-73-0; **1b**, 126786-74-1; **2a**, 25092-65-3; **2b**, 126724-30-9; *E*-**2c**, 126724-31-0; *Z*-**2c**, 126724-32-1; **2d**, 126724-33-2; **2e**, 57-88-5; $(Ph_3P)_4RhH$, 18284-36-1; F_3CCO_2H,

(7) (a) Gent, P. A.; Gigg, R.; Conant, R. *J. Chem. Soc. Perkin Trans. 1* **1972**, 1535; **1973**, 1858. (b) Gent, P. A.; Gigg, R. *J. Chem. Soc., Chem. Commun.* **1974**, 277.

(8) $(Ph_3P)_4RhH$ has been found to react with carbon acids to form the cationic species $(Ph_3P)_3Rh^+$: Siedle, A. R.; Newmark, R. A.; Howells, R. D. *Inorg. Chem.* **1988**, *27*, 2473.

76-05-1; allyl bromide, 106-95-6; methallyl chloride, 1458-98-6; (*E*)-2-butenyl bromide, 29576-14-5; (*Z*)-2-butenyl bromide, 39616-19-8; 3-bromo-1-butene, 22037-73-6; γ,γ-dimethylallyl bromide, 870-63-3.

Efficient, Trimethylsilyl Triflate Mediated Conversion of Diels-Alder Adducts of 1-Methoxy-3-[(trimethylsilyl)oxy]-1,3-butadiene (Danishefsky's Diene) to Cyclohexenones[1a]

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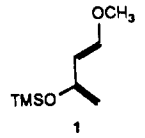
Since its introduction in 1974,[2] 1-methoxy-3-[(trimethylsilyl)oxy]-1,3-butadiene (Danishefsky's diene, 1) has been successfully employed in a wide variety of synthetic efforts.[3-6] The foremost synthetic application of 1 continues to be the Diels-Alder synthesis of cyclohexenones.



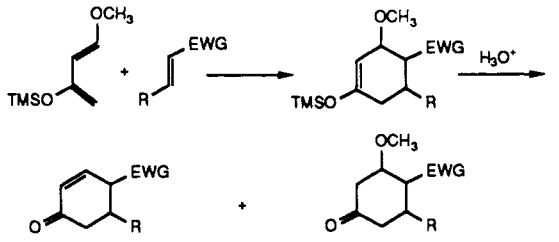
1

Heretofore, the conversion of Diels-Alder adducts of Danishefsky's diene to cyclohexenones has been accomplished simply by treatment with aqueous acid. However, this method sometimes suffers from the formation of a methoxy ketone as the sole product or admixed with the desired enone (Scheme I).[7]

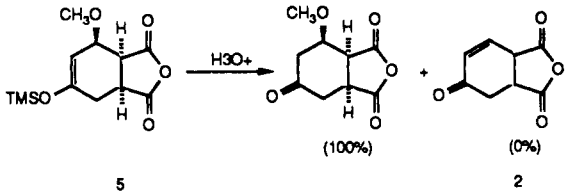
The enone and methoxy ketone products appear to arise via competing hydrolysis pathways. Hydrolysis to enone can occur via stepwise or concerted formal elimination of methanol. Enone formation thus competes with simple hydrolysis of the silyl enol ether, which affords β-methoxy ketone. Danishefsky has shown that the methoxy ketones generally are not converted to enones under acid hydrolysis conditions.[4c,8] Accordingly, the enone and methoxy ketone probably arise through competitive C- and O-protonation of adduct.

Danishefsky has further shown that the amount of methoxy ketone formed in the hydrolysis is dependent upon the concentration of the acid and on specific structural features of the adducts.[4c] Dilute acid (4:1 THF/0.5 N HCl) was found to minimize methoxy ketone formation in many cases, whereas more concentrated acid led to a

Scheme I



Scheme II



5 2

Scheme III

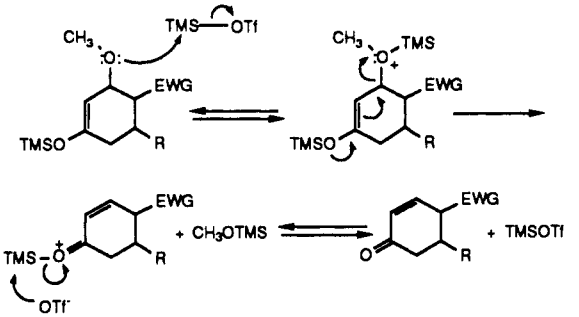
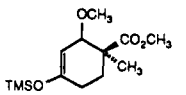
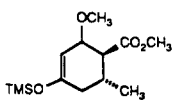
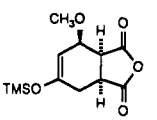


Table I. Comparison of Product Yields for Acid Hydrolysis and Catalytic TMSOTf

	0.005 N HCl[a]		TMSOTf	
	% methoxy ketone	% enone	% methoxy ketone	% enone
 3[b]		65	0	95
 4[b]	20	10[c]	0	93[d]
 5[e]	100	0	0	98

[a] Reference 4c. [b] Exo endo mixture. [c] 1:1 mixture of 8 and 9. [d] 27.5:1 mixture of 8 and 9. [e] Reference 9.

(1) (a) This material taken from the Ph.D. Thesis of the author. (b) Present address: Department of Chemistry, United States Air Force Academy, Colorado Springs, CO 80840.

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(6) See, for example: (a) Kobayashi, S.; Kerwin, J. F., Jr.; Danishefsky, S. *J. Am. Chem. Soc.* **1982**, *104*, 358-359. (b) Uang, B. J.; Quallich, G.; Harvey, D. F.; Danishefsky, S. *J. Org. Chem.* **1984**, *49*, 393-395. (c) Uang, B. J.; Quallich, G.; Danishefsky, S. *J. Am. Chem. Soc.* **1984**, *106*, 2453-2455. (d) Harvey, D. F.; Pearson, W. H.; Danishefsky, S. *J. Am. Chem. Soc.* **1984**, *106*, 2455-2458.

(7) For examples, see refs 4 and 5.

(8) Methanol can be eliminated by repeated exposure to neutral alumina; see ref 4c.

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