difference spectra were obtained as described by Hall and Sanders[14] using a pulse width of 8 μs (90°), a 10-s delay for NOE buildup, a 10-s delay for relaxation, two dummy scans and eight transients per cycle per frequency, and 16-32 cycles. IR spectra were recorded of neat thin films of analyte on NaCl plates on a Perkin-Elmer 727B spectrophotometer. Elemental analyses were performed by Galbraith Laboratories, Knoxville, TN. FAB-HRMS spectra were obtained at the Regional MS Facility located at MIT on a Finnigan MAT 8200 spectrometer.

9-(5′-*O*-(*tert*-Butyldimethylsilyl)-2′,3′-dideoxy-3′-iodo-β-D-*erythro*-pentofuranosyl)adenine (2) and 9-(5′-*O*-(*tert*-Butyldimethylsilyl)-2′,3′-dideoxy-3′-iodo-β-D-*threo*-pentofuranosyl)adenine (3). A solution of iodine (125 mg, 0.49 mmol), triphenylphosphine (215 mg, 0.81 mmol), and imidazole (37 mg, 0.54 mmol) in 2.0 mL of toluene-acetonitrile (9:1) was added to a solution of 5′-*O*-(*tert*-butyldimethylsilyl)-2′-deoxyadenosine[7] (1, 100 mg, 0.27 mmol) in 2 mL of toluene-acetonitrile (1:9) at ~40 °C under argon. After stirring for 12 h at 60 °C, the resulting suspension of a white solid in a colorless solution was evaporated to dryness and chromatographed ($CHCl_3$/EtOH, 96:4) to give **2**, 14 mg (11%), and **3**, 43 mg (33%).

For **2**: TLC ($CHCl_3$/EtOH, 96:4) R_f = 0.20; mp 142-144 °C; ^1H NMR δ 8.32 (1 H, s, H-8 or H-2), 8.28 (1 H, s, H-2 or H-8), 6.33 (1 H, dd, J = 6.7, 2.4 Hz, H-1′), 5.75 (2 H, s, NH_2), 4.46 (1 H, ddd, J = 10.1, 8.4, 6.8 Hz, H-3′), 4.33 (1 H, dψt, J = 8.5, 2.4, 2.4 Hz, H-4′), 4.04 (1 H, dd, J = 11.8, 2.5 Hz, H-5′a), 3.95 (1 H, dd, J = 11.8, 2.4 Hz, H-5′b), 3.01 (1 H, ddd, J = 13.7, 6.8, 2.4 Hz, H-2′β), 2.87 (1 H, ddd, J = 13.7, 10.1, 6.7 Hz, H-2′α), 0.91 (9 H, s, t-Bu), 0.11 (6 H, s, Me_2Si); FAB-HRMS (m/z) calcd for $C_{16}H_{26}IN_5O_2Si$ + H 476.0979, found 476.0977.

For **3**: TLC ($CHCl_3$/EtOH, 96:4) R_f = 0.14; mp 148-149 °C; ^1H NMR δ 8.32 (1 H, s, H-8 or H-2), 8.31 (1 H, s, H-2 or H-8), 6.36 (1 H, dd, J = 7.2, 4.5 Hz, H-1′), 5.66 (2 H, s, NH_2), 4.51 (1 H, ddd, J = 6.8, 4.9, 4.8 Hz, H-3′), 4.05 (1 H, dd, J = 10.7, 4.7 Hz, H-5′a), 3.86 (1 H, dd, J = 10.7, 4.9 Hz, H-5′b), 3.64 (1 H, dd, J = 4.9, 4.8 Hz, H-4′), 3.25 (1 H, ddd, J = 14.7, 7.4, 6.9 Hz, H-2′α), 2.98 (1 H, dψt, J = 14.7, 4.6, 4.6 Hz, H-2′β), 0.89 (9 H, s, t-Bu), 0.11 (6 H, s, Me_2Si). Anal. Calcd for $C_{16}H_{26}IN_5O_2Si$: C, 40.42; H, 5.51; N, 14.73. Found: C, 40.42; H, 5.43; N, 14.47.

9-(5′-*O*-(*tert*-Butyldimethylsilyl)-2′,3′-dideoxy-3′-cyano-β-D-*erythro*-pentofuranosyl)adenine (5) and 9-(5′-*O*-(*tert*-Butyldimethylsilyl)-2′,3′-dideoxy-3′-cyano-β-D-*threo*-pentofuranosyl)adenine (6). A solution of dried iodo compound **2** (30 mg, 0.063 mmol), tributyltin chloride (4.1 mg, 0.013 mmol), 2,2′-azobisisobutyronitrile (AIBN, 2.1 mg, 0.013 mg), and sodium cyanoborohydride (7.8 mg, 0.12 mmol) in 0.7 mL *tert*-butyl isocyanide was deoxygenated by repeated evacuation and admission of argon, then warmed to 87 °C for 1.5 h. The solvent was removed in vacuo, and the residue was chromatographed (EtOAc/EtOH, 94:6) to give **5** (8.5 mg, 36%) and **6** (7.4 mg, 32%).

Reaction of iodo isomer **3** (134 mg, 0.28 mmol) under identical conditions provided **5** (33 mg, 31%) and **6** (30 mg, 29%).

For **5**: TLC (EtOAc/EtOH, 94:6) R_f = 0.64; mp 135-136 °C; IR 2240 cm^{-1} (weak); ^1H NMR δ 8.30 (1 H, s, H-8 or H-2), 8.04 (1 H, s, H-2 or H-8), 6.32 (1 H, dd, J = 7.2, 2.6 Hz, H-1′), 5.59 (2 H, s, NH_2), 4.34 (1 H, m, H-4′), 4.09 (1 H, dd, J = 7.2, 14.6 Hz, H-5′a), 3.98 (1 H, dd, J = 3.5, 14.6, H-5′b), 3.86 (1 H, m, H-3′), 3.07 (1 H, ddd, J = 10.5, 7.9, 2.6 Hz, H-2′β), 2.84 (1 H, ddd, J = 13.6, 10.6, 7.2 Hz, H-2′α), 0.88 (9 H, s, t-Bu), 0.04 (6 H, s, Me_2Si); FAB-HRMS (m/z) calcd for $C_{17}H_{26}N_6O_2Si$ + H 375.1965, found 375.1943.

For **6**: TLC (EtOAc/EtOH, 94:6) R_f = 0.76; IR (neat) 2251 cm^{-1}; ^1H NMR δ 8.36 (1 H, s, H-8 or H-2), 8.30 (1 H, s, H-2 or H-8), 6.35 (1 H, dd, J = 6.4, 3.2 Hz, H-1′), 5.52 (2 H, s, NH_2), 4.35 (1 H, dψt, J = 8.0, 2.9 Hz, H-4′), 4.05 (1 H, dd, J = 11.5, 3.0 Hz, H-5′a), 3.81 (1 H, dd, J = 11.5, 3.0 Hz, H-5′b), 3.64 (1 H, m, H-3′), 2.84 (2 H, m, H-2′α and H-2′β), 0.91 (9 H, s, t-Bu), 0.09 (6 H, s, Me_2Si); FAB-HRMS (m/z) calcd for $C_{17}H_{26}N_6O_2Si$ + H 375.1965, found 375.1947.

9-(5′-*O*-(*tert*-Butyldimethylsilyl)-2′,3′-dideoxy-3′-formyl-β-D-*erythro*-pentofuranosyl)adenine (4). To a solution of dry **5** (15 mg, 0.04 mmol) in 0.4 mL of THF at -78 °C was added diisobutylaluminum hydride (17 mg, 0.12 mmol), and the mixture was stirred at -78 °C for 1 h and then at 25 °C for 1.5 h. Acetic acid (0.8 mL of a 0.5 M aqueous solution) was added, and the reaction was stirred for 3 h. The mixture was extracted with chloroform and chromatographed ($CHCl_3$/EtOH, 92:8) to give **4**: 6.5 mg (43%); TLC (dioxane/CH_2Cl_2, 1:2) R_f = 0.25; mp 108-109 °C; IR (neat) 1725 cm^{-1}; ^1H NMR ($CDCl_3$) δ 9.80 (1 H, d, J = 1.6 Hz, C*H*O), 8.31 (1 H, s, H-8 or H-2), 8.14 (1 H, s, H-2 or H-8), 6.29 (1 H, dd, J = 3.8, 6.7 Hz, H-1′), 5.68 (2 H, s, NH_2), 4.38 (1 H, ddd, J = 3.5, 4.6, 7.1 Hz, H-4′), 3.96 (1 H, dd, J = 4.6, 10.9 Hz, H-5′a), 3.83 (1 H, dd, J = 3.5, 10.9 Hz, H-5′b), 3.64 (1 H, m, H-3′), 2.87 (1 H, m, H-2′α), 2.77 (1 H, m, H-2′β), 0.88 (9 H, s, t-Bu), 0.21 (6 H, s, Me_2Si); FAB-HRMS (m/z) calcd for $C_{17}H_{27}N_5O_3Si$ + H 378.1962, found 378.1935.

9-(2′,3′-Dideoxy-3′-formyl-β-D-*erythro*-pentofuranosyl)adenine (7). A solution of **4** (5 mg, 0.013 mmol), tetrabutylammonium fluoride (100 mg, 0.38 mmol), and KF (20 mg, 0.34 mmol) in 1.5 mL of THF/H_2O/MeOH (1:1:1) was stirred at 40 °C for 30 h. The solvent was evaporated, and the residue was chromatographed ($CHCl_3$/EtOH, 92:8) to give **7**: 2.0 mg (60%); mp 210-212 °C; IR 1750 cm^{-1}; ^1H NMR (Me_2SO-d_6) δ 9.73 (1 H, s, C*H*O), 8.36 (1 H, s, H-8 or H-2), 8.14 (1 H, s, H-2 or H-8), 7.32 (2 H, s, NH_2), 6.23 (1 H, t, J = 6.0 Hz, H-1′), 5.30 (1 H, br s, OH), 4.29 (1 H, m, H-4′), 3.90 (1 H, m, H-3′), 3.65 (2 H, m, H-5′), 2.70 (2 H, m, H-2′α,β); FAB-HRMS (m/z) calcd for $C_{11}H_{13}N_5O_3$ + H 264.1097, found 264.1068.

9-(3′-Cyano-2′,3′-dideoxy-β-D-*erythro*-pentofuranosyl)adenine (8). Protected nitrile **5** (8.4 mg, 0.22 mmol) was treated exactly as for **7** above, to give **8**: 4 mg (67%); mp 194-195 °C; TLC ($CHCl_3$/EtOH, 94:6) R_f = 0.20; IR 2240 cm^{-1} (weak); ^1H NMR (Me_2SO-d_6) δ 8.32 (1 H, s, H-8 or H-2), 8.14 (1 H, s, H-2 or H-8), 7.12 (2 H, s, NH_2), 6.36 (1 H, dd, J = 3.9, 6.6 Hz, H-1′), 5.30 (1 H, t, J = 5.6 Hz, OH), 4.25 (1 H, dψt, J = 4.1, 8.5 Hz, H-4′), 3.91 (1 H, m, H-3′), 3.60 (2 H, m, H-5′), 3.15 (1 H, m, H-2′α), 2.92 (1 H, m, H-2′β); FAB-HRMS (m/z) calcd for $C_{11}H_{12}N_6O_2$ + H 261.1100, found 261.1060.

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Annelation Reaction via Tandem Michael-Claisen Condensations. 3. Synthesis of the Hydrindan System

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We have recently reported a new 4C + 2C annelation reaction based on the tandem Michael-Claisen condensation of 3-(phenylthio)-1-(trimethylsiloxy)-1-methoxy-1,3-butadiene (**1**) with a number of α,β-unsaturated ketones.[1,2] The reaction is formally similar to the Diels-Alder reaction but offers the advantage that even relatively unreactive dienophiles can undergo the annelation reaction. The usefulness of the reaction was demonstrated by the construction of the 9-methyldecalin system[1] and the syntheses of two sesquiterpenes, aristolone and fukinone.[2]

In trying to extend the reaction to the synthesis of the hydrindan system by the condensation of **1** with cyclopent-2-enone (**3**) according to Scheme I, the yield of the Michael adduct **4** was found to be poor.[1] Because of the presence of the hydrindan structure in many natural

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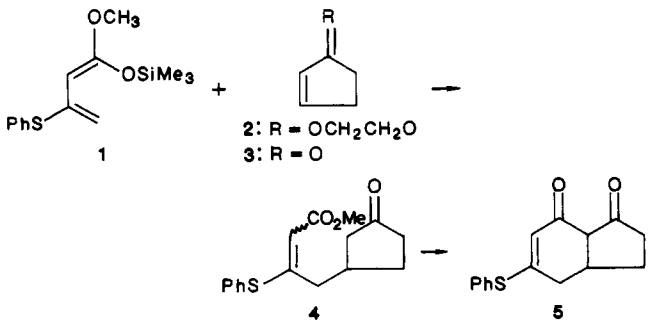
0022-3263/89/1954-3242$01.50/0

Scheme I

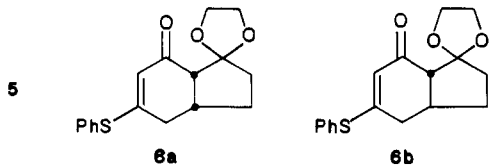


products,[3-5] we decided to examine the reaction in greater detail.

The poor yield of 4 was apparently due to the polymerization of cyclopentenone under the acidic reaction conditions of using $TiCl_4$ as the Lewis acid. We first attempted to solve the problem by using a mixed $TiCl_4$–$Ti(O\text{-}i\text{-}Pr)_4$ catalyst. It was found that when the ratio of $TiCl_4$ to $Ti(O\text{-}i\text{-}Pr)_4$ was 2:1, the polymerization of cyclopentenone remained a severe side reaction, but when the ratio was 1:1, the Michael reaction did not proceed completely and considerable amount of cyclopentenone was recovered.

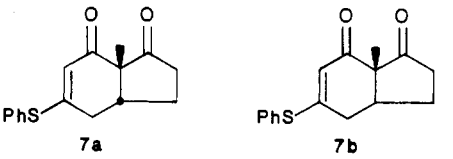
Recently, Noyori and co-workers described the use of trimethylsilyl triflate as the catalyst in the reaction of enol silyl ethers with acetals and ketals.[6] Ketals of α,β-unsaturated ketones may react in a Michael fashion under these reaction conditions and avoid the problem of polymerization. Indeed, when cyclopent-2-enone ethylene ketal (2) was reacted with 1 at −82 °C with 10 mol % of trimethylsilyl triflate, the adduct 4 was formed in 74% yield as a mixture of *E* and *Z* isomers.

The Michael adduct 4 was smoothly cyclized with LiSPh as previously described[1] to give the bicyclic compound **5**. As expected, compound **5** exists as a mixture of the keto and enol tautomers in $CDCl_3$. On the other hand, it exists mainly in the keto form in CD_3OD. When compound **5** was treated with ethylene glycol and a catalytic amount of *p*-toluenesulfonic acid under Dean–Stark conditions, the ethylene ketals **6a** and **6b** were obtained in 86% yield.

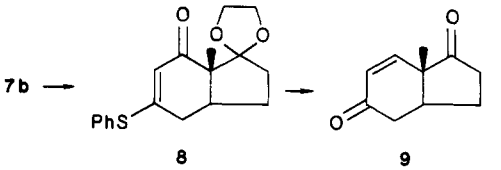


The two ketals, in a ratio of 12:1, are stereoisomers at the ring junction and were separable by careful column chromatography. The ratio reflects the relative thermodynamic stability of the two isomers since the pure minor isomer **6b**, on treatment with a catalytic amount of sodium methoxide in methanol, gave a mixture of **6a** and **6b** in 12:1 ratio again. The proton NMR spectrum of **6b** showed a sharp doublet at 2.69 ppm. Decoupling experiments established this to be the proton next to the carbonyl group at the ring junction. Its coupling constant (J = 12.3 Hz) suggests that **6b** has the trans stereochemistry. It follows that **6a** is the cis isomer.

We also examined the preparation of the important 8-methylhydrindan system. In the base-catalyzed cyclization of **4**, if the reaction mixture was quenched with alkyl halide instead of water, alkylated product should result. Accordingly, when the reaction mixture was quenched with iodomethane, a mixture of stereoisomers, **7a** and **7b**, were formed in a 3:1 ratio with 87% yield. The two isomers could be separated by column chromatography.



The stereochemistry about the ring junction in compounds **7a** and **7b** was established by chemical correlation. The minor isomer **7b** was converted to the ethylene ketal 8 under standard conditions. Reduction of 8 with lithium aluminum hydride followed by acid hydrolysis gave the known *trans*-enedione **9**. Compound **7b** thus has the trans stereochemistry at the ring junction.



In conclusion, we have demonstrated that the tandem Michael–Claisen condensation can be used effectively to construct highly functionalized hydrindan systems. The reaction gave stereoselectively the *cis*-hydrindans. We are exploring the use of this approach to the synthesis of natural products.

Experimental Section

Melting and boiling points are uncorrected. Infrared spectra were obtained from films on NaCl plates for liquids and from solutions in 0.1-mm cells or as a KBr pellet for solids on a Perkin-Elmer 297 spectrophotometer. The ^1H NMR spectra were recorded on Varian XL-200, T-60, and T-60A instruments and are reported in δ units with Me_4Si as internal standard; the abbreviations s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and br = broad are used throughout. Mass spectra were obtained on a Du Pont 492B machine operating at 70 eV. Column chromatography was performed on silica gel 60 (Merck).

3-(Phenylthio)-1-(trimethylsiloxy)-1-methoxy-1,3-butadiene (1). This compound was prepared according to published procedure.[1]

Methyl 3-(Phenylthio)-4-(3-oxocyclopentyl)but-2-enoate (4). To a well-stirred solution of 1 (14 g, 50 mmol) and 1,4-dioxaspiro[4.4]non-6-ene (**2**, 5.05 g, 40 mmol) in 500 mL of dry CH_2Cl_2 under nitrogen at −82 °C was added trimethylsilyl trifluoromethanesulfonate (0.97 mL, 5 mmol), and stirring was continued for 30 h. The reaction mixture was quenched at −82 °C with 20 mL of water. The organic layer was separated and washed twice with two 50-mL portions of water. The organic layer was dried (Na_2SO_4), and the solvent was evaporated under vacuum. The crude product was purified by column chromatography (eluant, 20% ethyl acetate–hexane) to give (*E*)-4 and (*Z*)-4 in 74% yield. Compound (*E*)-4 was crystallized from 5% ethyl acetate–hexane as white crystalline prisms (mp 69–71 °C). The spectral data of (*E*)-4 and (*Z*)-4 were identical in all respects with those reported previously.[1]

5-(Phenylthio)-2,3,3a,7a-tetrahydro-1*H*-indene-1,7(4*H*)-dione (5). To a well-stirred solution of (*E*)-4 (1.16 g, 4 mmol) in 20 mL of THF under nitrogen at room temperature was added potassium *tert*-butoxide (470 mg, 4 mmol). After 2 h, the solvent was removed under vacuum and the viscous mass was treated with

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5 mL of saturated aqueous NH_4Cl solution followed by extraction with CH_2Cl_2. The extract was dried (Na_2SO_4), and the solvent was removed. The crude product was column chromatographed (eluant, 50% ethyl acetate–hexane) to give **5** (mp 135–137 °C) in 72% yield: IR (KBr) 2930, 1722, 1615, 1555 cm^{-1}; ^1H NMR (CD_3OD) δ 7.02 (s, 5 H), 4.92 (br, 1 H), 2.73–1.4 (m, 8 H); MS m/z (relative intensity) 258 (100), 230 (34), 213 (36), 202 (43), 176 (33), 149 (50), 109 (76); exact mass calcd for $C_{15}H_{14}O_2S$ 258.071, obsd 258.068.

Compound **5** can also be obtained from (*Z*)-**4** by using lithium thiophenoxide as the reagent. To a well-stirred solution of thiophenol (1.54 mL, 15 mmol) in 20 mL of THF at 0 °C under nitrogen was added 6 mL of 2.5 M *n*-BuLi (15 mmol) followed by (*Z*)-**4** (1.5 mmol) in 5 mL of THF, and then the solution was refluxed for 20 h. The solvent was removed, and the residue was dissolved in CH_2Cl_2 and washed twice with 8% aqueous sodium hydroxide. The organic extract was dried (Na_2SO_4), and the solvent was removed. The residue was purified as above to give **5** in 69% yield.

Ketalization of Compound 5. To a well-stirred solution of 5-(phenylthio)-2,3,3a,7a-tetrahydro-1*H*-indene-1,7(4*H*)-dione (**5**, 1.03 g, 4 mmol) in 70 mL of benzene were added a catalytic amount of *p*-toluenesulfonic acid and ethylene glycol (2.48 g, 40 mmol). The reaction mixture was refluxed on a Dean–Stark apparatus. The reaction was followed by thin-layer chromatography using 30% ethyl acetate–hexane as eluant. At the end of the reaction, the reaction mixture was diluted with 100 mL of ether and washed with 20 mL of saturated aqueous $NaHCO_3$ solution. The organic layer was separated and dried (Na_2SO_4). The solvent was evaporated under vacuum, and the crude reaction mixture was purified by column chromatography using 20% ethyl acetate–hexane as an eluant to give **6a** and **6b** in a ratio of 12:1 with 86% yield. *cis*-**6a**: IR (film) 2940, 1646, 1580 cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.52–7.35 (m, 5 H), 5.53 (d, J = 2.0 Hz, 1 H), 3.97–4.16 (m, 1 H), 3.69–3.92 (m, 3 H), 2.55–2.89 (m, 3 H), 2.34–2.48 (m, 1 H), 1.79–2.16 (m, 3 H), 1.56–1.72 (m, 1 H); MS m/z (relative intensity) 302 (1), 259 (21), 149 (6), 99 (50), 77 (27), 74 (37), 67 (32), 59 (44), 43 (48), 31 (100). *trans*-**6b**: IR (film) 2960, 1656, 1560 cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.56–7.24 (m, 5 H), 5.45 (d, J = 2.0 Hz, 1 H), 4.23–4.42 (m, 1 H), 3.82–4.15 (m, 3 H), 2.69 (d, J = 12.3 Hz, 1 H), 1.29–2.48 (m, 7 H); MS m/z (relative intensity) 302 (2), 259 (46), 165 (11), 110 (11), 109 (32), 99 (86), 86 (46), 65 (56), 31 (57), 28 (100).

Methylhydrindans 7a and 7b. To a well-stirred solution of **4** (1.74 g, 6 mmol) in 30 mL of dry THF at 0 °C under nitrogen was added potassium *tert*-butoxide (0.71 g, 6.3 mmol), and stirring was continued for another 2 h. Then, iodomethane (0.75 mL, 12 mmol) was added, and stirring was continued for another 10 h. At the end of the reaction, the solvent was removed under vacuum, and the crude reaction mixture was dissolved in 100 mL of ether and washed with 10 mL of water. The organic phase was dried (Na_2SO_4), and the solvent was evaporated. The crude product was purified by column chromatography (eluant, 40% ethyl acetate–hexane) to give in 87% combined yield the *cis*-**7a** and the *trans*-**7b** in a ratio of 3:1, respectively. The cis compound was crystallized from 15% ethyl acetate–hexane to give white crystalline prisms (mp 86–88 °C). The trans compound was also crystallized from 25% ethyl acetate–hexane as white crystalline prisms (mp 131–133 °C). *cis*-**7a**: IR (KBr) 2880, 1745, 1635, 1580 cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.40–7.60 (m, 5 H), 5.48 (d, J = 2.0 Hz, 1 H), 2.96 (ddd, J = 2.0 Hz, 5.7 Hz, 1 H), 1.80–2.62 (m, 6 H), 1.30 (s, 3 H); MS m/z (relative intensity) 272 (44), 176 (64), 148 (53), 110 (24), 109 (22), 91 (28), 67 (100), 39 (64); exact mass calcd for $C_{16}H_{16}O_2S$ 272.087, obsd 272.093. *trans*-**7b**: IR (KBr) 1740, 1660, 1555, 1275 cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.42–7.61 (m, 5 H), 5.44 (s, 1 H), 2.03–2.77 (m, 6 H), 1.73–1.94 (m, 1 H), 1.20 (s, 3 H); MS m/z (relative intensity) 272 (35), 176 (61), 148 (42), 110 (36), 109 (36), 91 (43), 77 (73), 67 (100), 39 (63), 28 (44); exact mass calcd for $C_{16}H_{16}O_2S$ 272.087, obsd 272.086.

Enedione 9. To a well-stirred solution of *trans*-**7b** (1.09 g, 4 mmol) in 70 mL of benzene was added a catalytic amount of *p*-toluenesulfonic acid and ethylene glycol (0.50 g, 8 mmol). The reaction mixture was refluxed on a Dean–Stark apparatus. After 4 h, once again ethylene glycol (0.50 g, 8 mmol) was added, and reflux was continued for another 3 h. Then the solvent was removed under vacuum, and the residue was dissolved in 100 mL of ether. The ether extract was washed with 10 mL of saturated aqueous $NaHCO_3$, and the extracts were dried (Na_2SO_4). The solvent was evaporated, and the crude product was dissolved in 60 mL of dry ether under nitrogen. To the stirred solution was added lithium aluminum hydride (46 mg, 4.8 mmol). After 30 min, once again lithium aluminum hydride (46 mg, 4.8 mmol) was added, and the solution was refluxed for 1 h. Then, the excess lithium aluminum hydride was destroyed by dropwise addition of ethyl acetate. The reaction mixture was diluted with 100 mL of ether, washed with 10% aqueous HCl, and then dried (Na_2SO_4), and the solvent was evaporated under vacuum. The crude product was treated with 20% HCl–CH_3OH, and then the solution was stirred for 24 h. The mixture was evaporated and extracted with ether. The ether solution was dried (Na_2SO_4) and evaporated to give **9** in 61% yield after purification by column chromatography (eluent, 30% ethyl acetate–hexane). The physical data of **9** are in agreement with the reported values.[7]

Acknowledgment. Financial support from Merck Frosst Canada, NSERC, and FCAR are gratefully acknowledged. C.V.C.P. thanks McGill University for the award of the Bindra Fellowship.

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