rate of meta-ring flipping in the latter was found to be 45 times faster with a $\Delta\Delta G^{\ddagger}$ of 2.6 kcal mol^{-1}. The most plausible explanation is the destabilization of the conformational ground state in **17** due to the $\pi-\pi$ repulsion of the stacking rings. The conformational ground states of *anti*-**13** and *syn*-**13** are expected to be similar to those of **15** and **17**, respectively. This would mean that *syn*-**13** is much less stable than *anti*-**13** due to the $\pi-\pi$ interaction. This is clearly consistent with the absence of *anti*-**13** → *syn*-**13** conversion in our dynamic NMR experiment and the change in conformational preference in going from *syn*-**9**/**11** to *anti*-**13**/**14** via desulfurization—a ring contraction reaction believed to involve ring-opening intermediate(s) thus resulting in the formation of the more stable anti [2.2]cyclophanes.

Experimental Section

All melting points were determined on a Synbron/Thermolyne MP-12615 melting point apparatus and were uncorrected. ^{1}H NMR spectra were determined in $CDCl_3$ on a JEOL FX90Q (90 MHz) Fourier transform spectrometer. All chemical shifts are reported in ppm downfield from tetramethylsilane as the internal standard. Infrared spectra were recorded on a Perkin-Elmer 1310 infrared spectrometer. UV/vis spectra were determined in dichloromethane on a Shimadzu UV 240 graphicord spectrometer. Mass spectra were determined on a VG Micromass 7305 mass spectrometer at 70 eV using electron impact. Relative intensities are given in parentheses. Microanalyses were performed by the Microanalytical Laboratory, Department of Chemistry, National University of Singapore.

***anti*- and *syn*-9-Methyl-2,11-dithia[3.3](1,4)naphthalenometacyclophanes (11).** A solution of 2,6-bis(mercaptomethyl)toluene[6a] (0.996 g, 5.25 mmol) and 1,4-bis(bromomethyl)naphthalene[14] (1.648 g, 5.25 mmol) in benzene (150 mL) was added dropwise over a period of 2 h into a well-stirred solution of KOH (0.943 g, 16.81 mmol) in nitrogen-purged 95% ethanol (500 mL). After the addition, the mixture was further stirred for 15 h and the bulk of the solvent was then removed under reduced pressure. The residue was extracted with dichloromethane. The organic layer was washed with water, dried, and evaporated. The crude product was chromatographed on silica gel, using benzene/hexane (1:3) as eluant. Eluted first was *anti*-**11**: 141 mg (8%); mp 240–241 °C; ^{1}H NMR, see Table I; IR (KBr) 1580, 1500, 1440, 1400, 1240, 1210, 1180, 1110, 1030, 995, 960, 940, 910, 840, 820, 780, 760, 740, 710, 695, 675 cm^{-1}; UV λ_{max} 235 (ϵ 50500), 295 (7300), 306 (9500), 319 (7100) nm; MS ($M^{\cdot+}$) m/z 336 (61), 187 (47), 185 (30), 184 (18), 154 (100), 148 (11), 115 (11). Anal. Calcd for $C_{21}CH_{20}S_2$: C, 74.95; H, 5.99. Found: C, 75.20; H, 5.93.

Eluted next was a mixture of the anti and syn isomers: 146 mg (8%).

Eluted last was *syn*-**11**: 53 mg (3%); mp 237–238 °C; ^{1}H NMR, see Table I; IR (KBr) 1585, 1500, 1430, 1400, 1230, 1175, 1070, 1030, 940, 910, 840, 760, 730, 710 cm^{-1}; UV λ_{max} 230 (ϵ 50100), 285 (7900), 294 (9700), 303 (8200) nm; MS ($M^{\cdot+}$) m/z 336 (55), 187 (39), 185 (26), 184 (16), 154 (100). Anal. Calcd for $C_{21}H_{20}S_2$: C, 74.95; H, 5.99. Found: C, 74.40; H, 5.79.

***anti*-[2.2](1,4)Naphthalenometacyclophane (13).** A solution of the dithiacyclophane **9**[10] (190 mg, 0.59 mmol) in trimethyl phosphite (10 mL) was irradiated on a Rayonet photochemical reactor (Model RPR-100) at 254 nm for 18 h. The solution was then added to a mixture of 1 H HCl (100 mL) and cyclohexane (100 mL) and stirred thoroughly for 1 h. The organic layer was separated, washed with water, dried, and evaporated. The crude product was chromatographed on silica gel, using hexane as eluant, to yield colorless crystals of the cyclophane *anti*-**13**: 0.13 g (42%); mp 155–156 °C (lit.[8] mp 155.5–156.5 °C); ^{1}H NMR, see Table I; IR (KBr) 1570, 1430, 1380, 1355, 1160, 1145, 1075, 1010, 940, 920, 790, 770, 760, 700, 625 cm^{-1}; UV λ_{max} 222 (ϵ 27900), 241 (33500), 305 (br; 3900) nm; MS ($M^{\cdot+}$) m/z 258 (93), 243 (64), 230 (18), 104 (100), 103 (28).

***anti*-8-Methyl[2.2](1,4)naphthalenometacyclophane (14).** A solution of *anti*-**11** (80 mg, 0.24 mmol) in benzene (3 mL) and trimethyl phosphite (6 mL) was subjected to photodesulfurization conditions as described for the preparation of *anti*-**13**. Colorless crystals of *anti*-**14** were obtained: 20 mg (31%); mp 196–198 °C; ^{1}H NMR, see Table I; IR (KBr) 1570, 1445, 1350, 1165, 1145, 890, 870, 800, 760, 710 cm^{-1}; UV λ_{max} 224 (sh; ϵ 21000), 241 (30300), 305 (3900) nm; MS ($M^{\cdot+}$) m/z 272 (75), 257 (33), 118 (100). Anal. Calcd for $C_{21}H_{20}$: C, 92.60; H, 7.40. Found: C, 93.12; H, 7.23.

The photodesulfurization was repeated with *syn*-**11** (40 mg, 0.12 mmol) to yield only *anti*-**14**: 20 mg (62%).

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Registry No. *syn*-**9**, 123487-52-5; *anti*-**11**, 123487-51-4; *syn*-**11**, 123538-43-2; *anti*-**13**, 116073-03-1; *anti*-**14**, 123487-53-6; 2,6-bis(mercaptomethyl)toluene, 41563-67-1; 1,4-bis(bromomethyl)naphthalene, 58791-49-4.

Stereochemical Studies of Simple Cyclooctyl Systems

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In an effort to utilize 1,5-cyclooctadiene (1,5-COD) as an inexpensive and readily available starting material for stereocontrolled synthesis, we have studied the functionalization of 1,5-cyclooctanedione, **1**, which can easily be prepared from 1,5-COD in three steps (Scheme I).[1,2] The selectivity of these reactions can be explained by the conformational preferences of the cyclooctyl system,[3] which are revealed by X-ray crystallographic analyses.

The X-ray crystal structure of 1,5-cyclooctanedione shows that in the solid state, the molecule exists in a boat–chair conformation.[4] The sp^2-hybridized carbon atoms of **1** are located at the flagpole positions of the eight-membered ring, thereby minimizing the strain which results from transannular interactions across the ring. If the bis(enolization) of **1** were to occur from the same conformation, high diastereoselectivity to produce a *E,E*-1,4-bis(enolate) would be expected. The most acidic protons are those which have the highest overlap of the C–H σ-bond with the carbonyl π-system, i.e. those nearly perpendicular to the plane of the C–O bond of the carbonyl at carbons 2, 4, 6, and 8. Enolization at positions 6 and 8 was considered unlikely since the strain involved with formation of a trans olefin in an eight-membered ring is on the order of 11 kcal/mol.[5]

In accord with these expectations, the bis(silyl ether) **2** was obtained in quantitative yield and as a single regio- and stereoisomer when 1,5-cyclooctanedione was subjected

(1) For the preparation of *cis*-1,5-cyclooctanediol from 1,5-COD, see: Knights, E. F.; Brown, H. C. *J. Am. Chem. Soc.* **1968**, *90*, 5280.

(2) The oxidation of *cis*-1,5-cyclooctanediol to 1,5-cyclooctanedione using PCC has recently been reported: Lyttle, M. H.; Streitwieser, A.; Miller, M. J. *J. Org. Chem.* **1989**, *54*, 2331.

(3) For a study of the diastereoselective transformations of cyclooctanones as well as other cyclic ketones, see: Still, W. C.; Galynker, I. *Tetrahedron* **1981**, *37*, 3981.

(4) Miller, R. W.; McPhail, A. T. *J. Chem. Soc., Perkin Trans. 2* **1979**, 1527.

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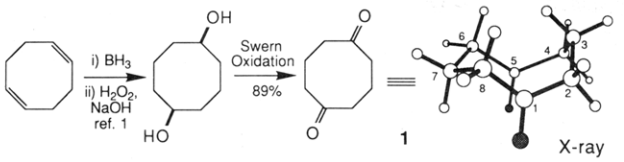
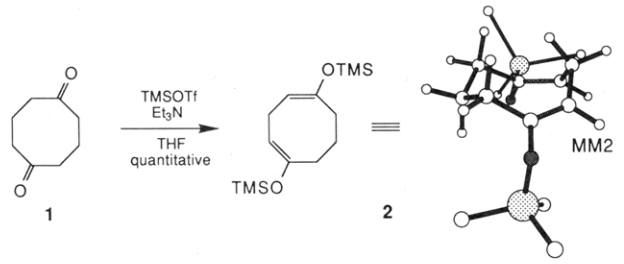
0022-3263/89/1954-5994$01.50/0

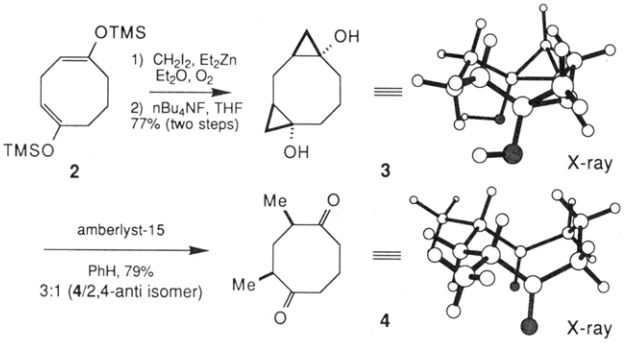
Scheme I



Scheme II



Scheme III



to the action of excess trimethylsilyl triflate in the presence of triethylamine (Scheme II). The preparation of **2** was also achieved in good yield through trapping of the bis(enolate) of **1** (generated at −78 °C with excess LDA) with trimethylsilyl chloride.

When compound **2** was treated with methyllithium in the presence of an electrophile (methyl iodide), the reaction did not proceed with high regio- or diastereoselectivity. This low selectivity was also observed when the dianion of **1** (generated by the treatment of **1** with LDA, KHMDS, or LHMDS) was treated with methyl iodide. The lack of selectivity in these reactions is presumably due to proton transfer, leading to enolate isomerization, between the monoalkylated intermediate and dialkylated product.

In order to avoid these problems, the cyclopropanation reaction shown in Scheme III was carried out. It was hoped that a cyclopropane derivative would impart some of the conformational rigidity present in the olefinic system, so that the anticipated monocyclopropyl intermediate would react in a stereoselective manner. When compound **2** was subjected to the modified Simmons–Smith[6] conditions shown in Scheme III and the crude reaction mixture was analyzed by ^1H and ^{13}C NMR spectroscopy, a single isomer was shown to predominate (>15:1). In practice, desilylation of the crude product was carried out to give the diol **3** as a single diastereomer in 77% yield after chromatography (mp = 128–130 °C). Compound **3** was shown to be the product of apparent peripheral addition[3] of the organozinc reagent (syn isomer) as determined by single-crystal X-ray diffractometry. Opening of the diol to produce *syn*-2,4-dimethyl-1,5-cyclooctanedione (**4**) was next attempted. The best results were obtained when **3** was treated with a catalytic amount of amberlyst-15 in benzene at room temperature, which produced **4** as a 3/1 mixture with the anti isomer in 79% yield. Apparently the ring-opening reaction is complicated by an accompanying epimerization process. Pure **4** could be obtained by HPLC separation (mp = 75–76 °C), and the structure was proven by X-ray crystallographic analysis.

Several interesting features are brought to light with the data provided by the X-ray analyses. For instance, the bis(cyclopropyl) diol exists in a splayed boat–chair conformation, reminiscent of the boat–chair conformation predicted for the bis(silyl enol) ether **2**. Four different boat–chair conformations that place the ketones at their preferred flagpole positions are available to the *syn*-2,4-dimethyl dione **4**. In agreement with the X-ray structure of **4**, molecular mechanics calculations predict that the conformation containing pseudoequatorial methyl groups in the chair locale is of the lowest energy.[7]

In this paper we have demonstrated that 1,5-cyclooctanedione can be functionalized selectively in the 2- and 4-positions. Since the conformational preferences of these cyclooctyl systems provide a simple explanation for the regio- and stereoselectivity of the reported transformations, further reactions of compounds related to and including **4** may also proceed in a predictable and selective manner.[8] The dione **1** appears to serve as a promising template for stereoselective addition reactions leading to cyclooctane-containing target molecules with multiple functionality.

Experimental Section

General. ^1H magnetic resonance spectra were obtained on a Bruker WM-250 (250 MHz) or a Bruker AM-500 (500 MHz) and are reported in parts per million with $CDCl_3$ (7.27) as an internal standard on a δ scale. Data are reported in the following sequence: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), coupling constant (Hz), integration, and assignment. ^1H decoupled ^{13}C NMR spectra were recorded on a Bruker AM-500 (125.8 MHz) instrument and are reported in parts per million with $CDCl_3$ (77.0) as an internal standard on a δ scale. Melting points were determined on a Mel-temp apparatus and are uncorrected. Infrared spectra were recorded on a Nicolet 5 PC FT-IR spectrometer (ν_{max} in cm^{-1}). Bands are characterized as strong (s), medium (m), or weak (w). All reactions were performed under nitrogen atmosphere unless otherwise noted and were monitored by analytical thin-layer chromatography. Flash chromatography was carried with use of E. Merck silica gel 60 (230–400 mesh) as described by Still.[9] Elemental analyses were performed by Atlantic Microlab, Inc., Norcross, GA. *cis*-1,5-Cyclooctanediol was purchased from the Aldrich Chemical Co. and was used as received.

1,5-Cyclooctanedione (1). To a flame-dried 500-mL round-bottom flask equipped with a magnetic stirbar and nitrogen line was added 50 mL of methylene chloride. The flask was cooled to −78 °C, and oxalyl chloride (3.8 mL, 43.6 mmol) was added; then DMSO (6.0 mL, 84.6 mmol) was added over about 5 min. After the mixture was stirred for 15 min at −78 °C, a solution of *cis*-1,5-cyclooctanediol (2.72 g, 18.9 mmol) in a mixture of 40 mL of methylene chloride and 10 mL of DMSO was added by syringe.

(6) Furukawa, J.; Kawabata, N.; Nishimura, J. *Tetrahedron* **1968**, *24*, 53.

(7) Calculations were performed using Macromodel on a Vax Station 3500. Multiconformer minimization was carried out using MM2 in the BDNR mode, using the default parameters to generate (and eliminate duplicate) conformations with the following exceptions: Dihedral angle resolution, 30° instead of 60°; iteration limit, 500 instead of 250. After minimization, further duplicate conformations were eliminated. For compound **4**, 780 initial conformations were generated, 218 remained after duplicate elimination, 15 remained after minimization and further duplicate elimination. The lowest energy conformation is virtually identical to that depicted in Scheme III. A similar process was used to generate and minimize the conformations of **2**, the lowest of which shown in Scheme II.

(8) For a related transformation, see: Mehta, G.; Kasibhatla, S. R. *J. Chem. Soc., Chem. Commun.* **1985**, 1464. See also ref 2 and 3.

(9) Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923.

The resultant cloudy solution was stirred for 70 min at −78 °C, triethylamine (27.0 mL, 193.7 mmol) was added, and the milky white solution was stirred for 30 min at that temperature. The reaction was allowed to warm gradually to ambient temperature; after 3 h water (100 mL) was added, and the separated organic layer washed sequentially with aqueous ammonium chloride (25 mL, saturated), aqueous sodium bicarbonate (25 mL, saturated), and brine (50 mL). The organic layer was dried over magnesium sulfate and filtered, and the solvent was removed under reduced pressure. Flash chromatography (20% hexanes in ethyl acetate) allowed isolation of 2.36 g (89%) of the dione at a white solid: mp = 65–66 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.53–2.47 (m, 8 H, C(O)CH_2), 2.17–2.10 (m, 4 H, C(O)CH$_2$CH_2); ^{13}C NMR ($CDCl_3$, 125.8 MHz) δ 213.0, 42.2, 22.1; IR ($CHCl_3$) 1711 (s).

1,5-Bis[(trimethylsilyl)oxy]-1,4-cyclooctadiene (2). To a solution of 1,5-cyclooctanedione (0.3025 g, 2.158 mmol) in 20 mL of THF at ambient temperature under nitrogen was added trimethylsilyl triflate (2.5 mL, 12.94 mmol), followed by triethylamine (3.60 mL, 25.83 mmol), both via syringe. After 2.5 h, most of the solvent was removed from the dark solution under reduced pressure, and the residue was diluted with 20 mL of hexanes and stirred vigorously for several minutes. After stirring was discontinued, the upper hexane layer was transferred into an Erlenmeyer flask, and the hexanes extraction process was repeated two more times on the residual oil. The combined hexane layers were dried over magnesium sulfate and then filtered through a short column of silica gel. Solvents were removed to leave 0.6088 g (99%) of a light yellow oil: ^{1}H NMR ($CDCl_3$, 250 MHz) δ 4.88 (t, J = 6.4, 2 H, C(OTMS)=CH), 2.61 (t, J = 6.4, 2 H, C(OTMS)=CHCH_2), 2.32–2.24 (m, 4 H, CH_2C(OTMS)=CH), 1.60–1.49 (m, 2 H, CH_2CH$_2$C(OTMS)=), 0.17 (s, 18 H, Si(CH_3)$_3$).

(1α,5α,7α,9α)-Tricyclo[7.1.0.0^{5,7}]decane-1,5-diol (3). A flask containing the bis(silyl enol) ether **2** (0.6088 g, 2.14 mmol) in 15 mL of ether was fitted with a magnetic stirbar, a reflux condenser, and a calcium chloride drying tube. No nitrogen line was attached. Diethylzinc (14.4 mL, 0.89 M, 12.8 mmol) was added via syringe, followed by methylene iodide (1.7 mL, 21.1 mmol). The mixture was gently refluxed under dry air for 3 h and then allowed to stir at room temperature overnight. The reaction was quenched by addition of saturated aqueous ammonium chloride (10 mL), and then the organic products were extracted with 60 mL of ether. The organic layer was washed with saturated aqueous sodium thiosulfate (5 mL) and brine (5 mL) and then dried over sodium sulfate. Solvent was removed to leave 0.696 g of an oil, which was taken up in 60 mL of THF and treated with n-Bu$_4$NF (7 mL, 1 M in THF, 7 mmol). After 1 h, solvent was removed under reduced pressure, and the residue was diluted with 100 mL of ethyl acetate. The organic layer was washed with saturated aqueous sodium bicarbonate (10 mL), and then the aqueous layer was back extracted with two portions of ethyl acetate (25 mL each). The combined organic layers were dried over sodium sulfate, and the solvent was removed to leave about 1 g of a crude oil. Flash chromatography (ethyl acetate) provided 0.277 g (77%) of the pure product as a white solid. X-ray quality crystals were grown from methylene chloride/ether/petroleum ether: mp 128–130 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 4.64 (s, 2 H, OH), 2.43 (dd, J = 15.3, 7.2, 2 H, CHHC(OH)(CH$_2$)CH), 2.22 (dt, J = 14.2, 3.3, 1 H, C(OH)(CH$_2$)CHCHH), 2.00 (dt, J = 14.9, 12.1, 1 H, CHHCH$_2$C(OH)(CH$_2$)CH), 1.45 (dt, J = 14.9, 7.2, 1 H, CHHCH$_2$C(OH)(CH$_2$)CH), 1.28–1.17 (m, 2 H, CH$_2$CH$_2$C(OH)(CH$_2$)CHCH$_2$), 1.09 (dd, J = 15.3, 12.1, 2 H, CH$_2$CHHC(OH)(CH$_2$)CH), 0.87 (dd, J = 10.3, 5.2, 2 H, C(OH)(CHH)CH), 0.2 (m, 1 H, C(OH)(CH$_2$)CHCHH), 0.16 (dd, J = 6.7, 5.2, 2 H, C(OH)(CHH)CH); ^{13}C NMR ($CDCl_3$, 125.8 MHz) δ 58.8, 38.3, 30.5, 27.5, 20.0, 18.6; IR ($CHCl_3$) 3395 (broad, s). Anal. Calcd for $C_{10}H_{16}O_2$: C, 71.39; H, 9.59. Found: C, 71.46; H, 9.62.

***syn*-2,4-Dimethyl-1,5-cyclooctanedione (4).** To a solution of the bis(cyclopropyl) diol (0.0212 g, 0.126 mmol) in 1 mL of benzene was added amberlyst-15 (0.020 g). The solution was stirred at room temperature under nitrogen overnight and then passed through a plug of $MgSO_4$. Solvent was removed, and the solid residue was purified using flash chromatography (5% ether in methylene chloride, then 20% ether in methylene chloride) to give 0.0167 g (79%) of the diketone as a 3/1 mixture of diastereomers favoring the *syn*-2,4-dimethyl compound. Data reported is for the HPLC purified major product. X-ray quality crystals were grown from ether/petroleum ether: mp 75–76 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.81–2.74 (dqd, J = 12.0, 6.8, 3.3, 2 H, C(O)CHCH$_3$CHH), 2.55–2.49 (ddd, J = 13.0, 13.0, 3.6, 2 H, C(O)CHH), 2.43–2.39 (ddd, J = 13.0, 5.7, 3.5, 2 H, C(O)CHH), 2.37–2.28 (m, 1 H, C(O)CHHCHH), 2.09–2.02 (m, 1 H, C(O)CHHCHH), 1.85–1.77 (dt, J = 14.0, 12.0, 1 H, C(O)CHCH$_3$CHH), 1.67–1.63 (dt, J = 14.0, 3.3, 1 H, C(O)CHCH$_3$CHH), 1.08 (d, J = 6.8, 6 H, C(O)CHCH_3CHH); ^{13}C NMR ($CDCl_3$, 125.8 MHz) δ 215.5, 45.7, 40.9, 39.7, 22.0, 18.3; IR ($CHCl_3$) 1713 (s).

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Registry No. 1, 1489-74-3; 2, 123540-61-4; 3, 123540-62-5; 3 (bis(trimethylsilyl) ether), 123540-64-7; *syn*-4, 123540-63-6; *anti*-4, 123540-65-8; *cis*-1,5-cyclooctanediol, 23418-82-8.

Supplementary Material Available: Protocols, fractional coordinates, bond distances, torsional angles, and anisotropic temperature factors for the X-ray crystallographic determinations of compounds 3 and 4 (9 pages). Ordering information is given on any current masthead page.

An Improved Procedure for the Conversion of Amines to Alcohols at Low Temperature

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One of the best methods for converting aliphatic amines to alcohols involves thermal rearrangement of the corresponding *N*-nitrosoamides (eq 1). This well-studied process[1-3] continues to find new and useful applications.[4-6] The normal experimental procedure for *n*-alkylamines entails isolating and heating the preformed *N*-nitrosoamide **1** for several hours in a nonpolar solvent such as hexane, whereupon transient diazoester **2** is produced. Smooth breakdown of **2** at 70–80 °C to the diazoalkane **3**[7] furnishes ester **4**, which can be saponified to **5**. We now report a milder procedure in which primary *n*-alkylamines are first converted to the corresponding trifluoro- or trichloroacetamides **6** and then nitrosated at 0 °C in acetic acid–acetic anhydride. Accelerated rearrangement to **3** in the presence of excess HOAc leads to acetates **7** (eq 2) without isolation of any intermediates. Besides being simple and

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