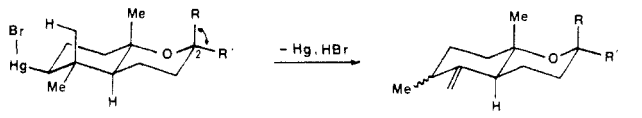
and 4.81 in the ^1H NMR spectrum (cf. CH_3 doublet at δ 0.90 and C=CH_2 at δ 4.78 and 4.45 in muzigadial, **8**),[8] absorptions at 3080, 1790, 1640, and 885 cm^{-1} characteristic of an exomethylene unit in the IR spectrum, and an M + H^+ peak at *m/e* 209 in the isobutane CI mass spectrum] in 34% yield. Presumably rearrangement of **3g** results, perhaps via a reductive elimination of mercury metal, in relief of steric crowding of the three 1,3-diaxial methyl groups. Careful examination of the ^1H NMR spectra of crude product mixtures at the organomercury bromide stage for the cyclizations described here and elsewhere[4] revealed that in only one other case were olefin resonances in the δ 4.5–4.8 region observable. This was in the crude products **9** and **9′** (broad singlets at δ 4.70 and 4.78; *m/e*



3g R,R′ = Me, Me
9,**9′** R,R′ = Me, CH_2CO_2Me
11,**11′** R,R′ = Me, CN
12,**12′** R,R′ = Me, H

7 R,R′ = Me, Me
10,**10′** R,R′ = Me, CH_2CO_2Me

284 (M + NH_4^+) and 267 (M + H^+) in the ammonia CI mass spectrum) where strain relief might again serve as the driving force for conversion to rearranged **10** and **10′**. In this case these products were not isolated. Finally, no evidence for rearrangement could be observed in the crude products **11**/**11′** and **12**/**12′** in which the 1,3-interactions can be alleviated more easily by a bending away of the C_2-axial methyl group with concomitant reduction in the R–C_2–R′ internal angle.

Experimental Section[9]

General Procedure for Preparation of Organomercury Bromides 3. A dry nitromethane solution of mercuric trifluoroacetate (0.45 M) was added to a stirred solution of diene **1** in nitromethane (0.3 M) at room temperature[10] under nitrogen. After 20 min, excess saturated aqueous potassium bromide was added and the resulting heterogeneous mixture was stirred efficiently at room temperature for 14 h. The mixture was extracted with methylene chloride, dried ($MgSO_4$), and concentrated to afford crude **3** as a brown oil. Short column chromatography (hexanes–EtOAc elution) afforded **3b**, **3e**, **3f**, and **3g** in the yields listed in Table I and with the following spectral properties. **3b**: ^1H NMR ($CDCl_3$) δ 1.05 (s, 3 H), 1.16 (s, 3 H), 1.41 (s, 3 H), 1.4–2.3 (m, 7 H), 2.5–2.9 (m, 3 H); IR ($CHCl_3$) 1715 cm^{-1}; CI mass spectrum (NH_3, pos), appropriate clusters at *m/e* 494 (for ^{79}Br and ^{202}Hg, M + NH_4^+), 477 (M + H^+), 450 (M + NH_4^+ - CO_2), 433 (M + H^+ - CO_2), 292 (M + NH_4^+ - Hg), 275 (M + H^+ - Hg); CI mass spectrum (NH_3, neg), *m/e* 555 (M + Br^-), 511 (M + Br^- - CO_2), 281 ($HgBr^-$). **3e**: ^1H NMR ($CDCl_3$) δ 1.08 (s, 3 H), 1.18 (s, 6 H), 1.4–2.5 (m, 7 H), 2.17 (s, 3 H), 2.75 (m, 1 H, CHHg), 3.68 (s, 3 H); IR (neat) 1710, 1620 cm^{-1}; CI mass spectrum (NH_3, pos), appropriate clusters at *m/e* 550 (for ^{79}Br and ^{202}Hg, M + NH_4^+), 533 (M + H^+), 348 (M + NH_4^+ - Hg), 331 (M + H^+ - Hg); CI mass spectrum (NH_3, neg), *m/e* 611 (M + Br^-), 281 ($HgBr^-$). **3f**: ^1H NMR ($CDCl_3$) δ 1.04 (s, 3 H), 1.11 (s, 3 H), 1.22 (s, 3 H), 1.35–2.3 (m, 7 H), 2.81 (m, 1 H, CHHg), 2.95 (br s, 2 H, CH_2C=O), 3.66 (s, 3 H), 4.62 (m, 1 H, HC=C); IR ($CHCl_3$) 1740, 1685 cm^{-1}; CI mass spectrum (NH_3, pos), appropriate clusters at *m/e* 550 (for ^{79}Br and ^{202}Hg, M + NH_4^+), 533 (M + H^+), 348 (M + NH_4^+ - Hg), 331 (M + H^+ - Hg); CI mass spectrum (NH_3, neg), *m/e* 611 (M + Br^-), 567 (M + Br^- - CO_2), 531 (M - H^+), 329 (M - H^+ - Hg), 281 ($HgBr^-$). **3g**: see ref 4.

General Procedure for Reduction of 3 to 5. The organomercury bromide **3** in 1:1 methylene chloride–95% ethanol (0.1 to 0.3 M) was purged with argon and treated dropwise with 2.5 molar equiv of a caustic sodium borohydride solution (4.4 M $NaBH_4$ in 14 M NaOH). After ~1 h at room temperature ether was added and the mixture was washed with water and brine, dried ($MgSO_4$), and concentrated to afford a yellow oil. Short column chromatography provided the reduced products **5** in the yields listed in Table I. Spectral data for **5f–h** follow. **5f**: ^1H NMR ($CDCl_3$) δ 0.81 (s, 3 H), 0.91 (s, 3 H), 1.17 (s, 3 H), 1.3–2.0 (m, 9 H), 2.95 (br s, 2 H, CH_2C=O), 3.66 (s, 3 H), 4.59 (m, 1 H); IR (neat) 1745, 1680 cm^{-1}; EI mass spectrum (relative intensity) *m/e* 252 (41, M^+), 219 (39), 167 (25), 145 (29), 136 (25), 129 (53), 109 (100), 97 (27), 81 (49), 69 (15), 55 (23), 41 (38). An analytical sample was prepared by PGC (SE-30). Anal. Calcd for $C_{15}H_{24}O_3$: C, 71.39; H, 9.59. Found: C, 71.48, H, 9.72.

5g: ^1H NMR ($CDCl_3$) δ 0.76 (s, 3 H), 0.89 (s, 3 H), 1.16 (s, 3 H), 1.24 (s, 3 H), 1.27 (s, 3 H), 1.3–1.9 (m, 11 H); IR (neat) 2950, 2880, 1478, 1382, 1146, 1115, 1057, 992, 844 cm^{-1}; EI mass spectrum, (relative intensity), *m/e* 210 (7, M^+), 195 (57), 109 (71), 69 (100); exact mass calcd for $C_{14}H_{26}O$ 210.1980, found 210.1981.

5h. One pure diastereomer was isolated (8%) as the least polar component [^1H NMR ($CDCl_3$) δ 0.80 (s, 3 H), 0.95 (s, 3 H), 1.16 (s, 3 H), 1.24 (d, *J* = 6.5 Hz, 3 H), 1.10–2.25 (m, 11 H), 2.56 (m, 1 H, CHC=O), 3.64 (s, 3 H), 4.16 (dq, *J* = 3, 6.5 Hz, 1 H); IR (neat) 1724, 1436, 1374, 1020, 989, 795, 783 cm^{-1}; EI mass spectrum (relative intensity), 268 (1, M^+), 253 (5), 224 (20), 209 (23), 183 (57), 151 (33), 138 (34), 123 (100), 109 (51), 100 (16), 95 (44), 87 (55), 81 (42), 79 (31), 69 (56), 67 (31), 59 (18), 55 (55), 43 (68), 41 (58); exact mass calcd for $C_{16}H_{28}O_3$ 268.2038, found 268.2057] followed by a mixture of three additional diastereomers (12%).

Rearranged Olefin 7. Short column chromatography on silica gel (5:1 hexanes–EtOAc) of the crude reaction mixture from the preparation of **3g** gave a faster running olefin **7** (34%): ^1H NMR ($CDCl_3$) δ 0.96 (d, *J* = 6 Hz, 3 H), 1.23 (s, 9 H), 1.5–2.1 (m, 10 H), 4.71 (br s, 1 H), 4.81 (br s, 1 H); IR (neat) 3080, 3000–2820, 1790, 1642, 1460, 1381, 1317, 1258, 1146, 990, 885 cm^{-1}; CI mass spectrum (isobutane, pos), *m/e* 209 (M + H^+).

Acknowledgment. This research was supported by Grant No. CA 24056 awarded by the National Cancer Institute of the National Institutes of Health. The NMR instrument was purchased through NSF Departmental Instrumentation Grant No. CHE 76-05167.

Registry No. **1a**, 459-85-8; **1b**, 5579-63-5; **1c**, 3796-70-1; **1d**, 3879-26-3; **1e**, 51933-45-0; **1f**, 56523-17-2; **1g**, 71041-60-6; **1h**, 77984-75-9; **1i**, 105-87-3; **3a**, 78003-79-9; **3b**, 77984-76-0; **3c**, 77984-77-1; **3d**, 77984-78-2; **3e**, 77984-79-3; **3f**, 77984-80-6; **3g**, 77984-81-7; **3h**, 77984-82-8; **5a**, 37531-07-0; **5b**, 78038-67-2; **5c**, 18444-96-7; **5d**, 6136-74-9; **5e**, 66901-68-6; **5f**, 77984-83-9; **5g**, 77984-84-0; **5h**, 77984-85-1; **7**, 77984-86-2; 2,2-dimethyl-6-methylenecyclohexanemethanol acetate, 77984-87-3; 2,6,6-trimethylcyclohex-2-enemethanol acetate, 69842-11-1.

(8) Kubo, I.; Miura, I.; Pettei, M. J.; Lee, Y.-W.; Pilkiewicz, F.; Nakanishi, K. *Tetrahedron Lett.* **1977**, 4553.
(9) See ref 4 for general experimental procedure.
(10) In the case of **1a** at least the cyclization proceeds rapidly at -10 °C.

Synthesis of Chloro Lactones by Reaction of Unsaturated Acids with Chloramine T

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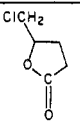
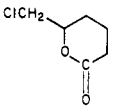
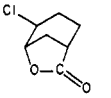
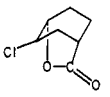
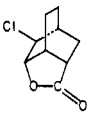
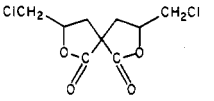
The synthesis and synthetic utility of halo lactones have recently been reviewed by Dowle and Davies.[1] The halolactonization of unsaturated carboxylic acids was first described at the beginning of the century. However, this reaction is of interest nowadays. Whereas bromo- and iodolactonization are generally well documented, there are

(1) Dowle, M. D.; Davies, D. I. *Chem. Soc. Rev.* **1979**, *8*, 171.

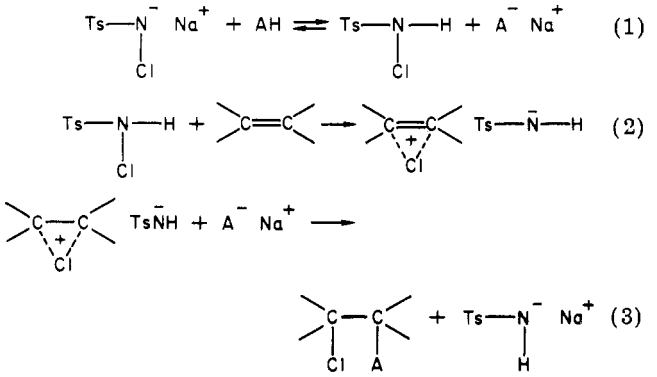
0022-3263/81/1946-3552$01.25/0

Table I. Preparation of Chloro Lactones by Reaction of Chloramine T with Unsaturated Acids

unsaturated acids	reaction time[a]	product	no.	% yield[b,c]	mp or bp (torr), °C
4-pentenoic acid (2)	2		3	67 (63)	64 (0.01)
5-hexenoic acid (4)	3		5	65 (61)	88 (0.01)
3-cyclohexenoic acid (6)	2		7	24 (21)	129
			8	41 (37)	86
bicyclo[2.2.2]oct-5-ene-2-carboxylic acid (9)	3		10	74 (67)	97
diallylmalonic acid (11)	5		12	82 (77)	115 (0.01)

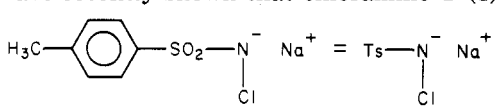
[a] Reaction time in hours at 80 °C in benzene or chlorobenzene. [b] Yield determined by GLC analysis vs. an internal standard (dodecane, tetradecane, hexadecane). [c] The yield of product isolated by distillation, column chromatography, or recrystallization is given in parentheses. [d] Compound 12 is a mixture (50/50) of the two diastereoisomers.

Scheme I



relatively few examples of the conversion of unsaturated carboxylic acids into chloro lactones.[2-6] The typical process of preparing halo lactones is the addition of halogen to the salts of unsaturated acids. However, dihalo compounds are generally formed as byproducts as indicated recently by Garratt.[7]

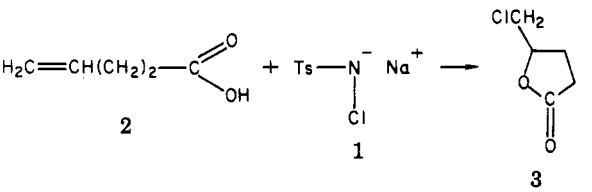
We have recently shown that chloramine T (1) in an



1

organic acid medium reacts with the olefin to give the corresponding chloroacyloxy compound[8] (Scheme I). The same reaction with an unsaturated acid such as allylacetic acid produces the expected γ-(chloromethyl)-γ-butyrolactone (3) (Scheme II) with a poor yield (5%) because the acidity of the unsaturated acid is too weak to protonate the chloramine T (eq 1). By using an equimolecular amount of a strong acid with a conjugate anion of low nucleophilicity such as methanesulfonic acid, we succeeded in preparing chloro lactone 3 in a fairly good yield. The corresponding chloro lactones are also prepared from other unsaturated acids (Table I).

Scheme II



The reactions were typically run by adding an equimolecular amount of methanesulfonic acid to a mixture of chloramine T (1) and unsaturated acid in benzene or chlorobenzene. The very exothermic reaction was controlled, and the decay of positive chlorine was checked by iodometric analysis. Filtration and solvent evaporation afforded the crude product which was further purified by distillation or chromatography.

Experimental Section

General Methods. Melting points were determined on a Reichert melting point apparatus and are uncorrected. ^1H NMR spectra were recorded with a Perkin-Elmer R 24 spectrometer using tetramethylsilane (Me_4Si) as an internal standard. ^{13}C NMR spectra were obtained on a Brücker WP 80 spectrometer. Chemical shifts were reported in parts per million in relation to Me_4Si as an internal standard. Mass spectra were performed on

(2) Van Tamelen, E. E.; Shamma, M. *J. Am. Chem. Soc.* **1954**, *76*, 2315.
(3) Berti, G. *Gazz. Chim. Ital.* **1951**, *81*, 305.
(4) Berti, G. *Tetrahedron* **1958**, *4*, 393.
(5) Reppe, W.; Schlichling, O.; Klager, K.; Toepel, T. *Justus Liebigs Ann. Chem.* **1948**, *560*, 1.
(6) Kurkov, V. P. U.S. Patent 4 031 115, 1977.
(7) Garratt, D. G.; Rayn, M. D.; Beaulieu, P. L. *J. Org. Chem.* **1980**, *45*, 839.
(8) Damin, B.; Garapon, J.; Sillion, B. *Tetrahedron Lett.* **1980**, 1709.

a VG Micromass 7070 F instrument operating at an ionizing potential of 70 eV. GLC was performed by using a steel column packed with Chromosorb PAW 80-100. Infrared spectra (IR) were recorded on a Perkin-Elmer 377 spectrophotometer.

General Procedure for Preparation of Chloro Lactones. To a suspension of 105 mmol of anhydrous chloramine T[9] in 80 mL of anhydrous benzene or chlorobenzene were added dropwise 100 mmol of unsaturated acid and 105 mmol of methanesulfonic acid simultaneously. The reaction was very exothermic, and the addition of reagents was adjusted to a rate so as to keep the temperature below 50 °C. At the end of the addition, the mixture was heated to 80 °C until the complete consumption of chloramine T (checked by iodometric analysis). After the mixture returned to room temperature and was filtrated, the solvent was removed by rotary evaporation. He chloro lactone was separated by direct distillation or purified by column chromatography on silica with methylene chloride as solvent and then distilled.

3: IR (neat ν_{max} 1775 (C=O) 1180 (CO) cm^{-1}; ^1H NMR ($CDCl_3$) δ 4.83 (complex m, 1 H), 3.77 (d, 2 H), 2.51 (complex m, 4 H); ^{13}C NMR ($CDCl_3$) δ 24.86, 28.36, 46.69, 78.72, 177.10; mass spectrum, *m/e* (relative intensity) 136 ($^{37}ClM^+$, 0.8), 134 ($^{35}ClM^+$, 2.6), 85 ($M^+ - CH_2Cl$, base peak), 55 (16.7), 49 (11.4), 41 (13.5), 39 (17).

Anal. Calcd for $C_5H_7ClO_2$: C, 44.63; H, 5.24; Cl, 26.35. Found: C, 44.43; H, 5.17; Cl, 26.22.

5: IR (neat) ν_{max} 1740 (C=O) 1170 (CO) cm^{-1}; ^1H NMR ($CDCl_3$) δ 4.50 (complex m, 1 H), 3.73 (d, 2 H), 2.40 (m, 4 H), 1.83 (m, 2 H); ^{13}C NMR ($CDCl_3$) δ 18.10, 25.21, 29.51, 46.41, 79.12, 171.25; mass spectrum, *m/e* (relative intensity) 150 ($^{37}ClM^+$, 0.6), 148 ($^{35}ClM^+$, 1.9), 99 ($M^+ - CH_2Cl$, 98.9), 71 (42), 55 (60.9) 42 (base peak), 41 (75.9), 39 (54.8).

Anal. Calcd for $C_6H_9ClO_2$: C, 48.50; H, 6.10; Cl, 23.86. Found: C, 48.74; H, 6.36; Cl, 23.56.

7: IR (KBr) ν_{max} 1787 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 4.77 (br t, 1 H), 4.33 (br t, 1 H), 2.80–1.77 (complex m, 7 H); ^{13}C NMR ($CDCl_3$) δ 22.40, 27.90, 31.91, 38.27, 53.57, 79.06, 177.84; mass spectrum, *m/e* (relative intensity) 162 ($^{37}ClM^+$, 2.4), 160 ($^{35}ClM^+$, 7.1), 124 ($M^+ - HCl$, 39.6), 120 (28.0), 118 (base peak), 97 (27.3), 83 (41.4), 81 (46.1), 80 (34.8), 42 (68.3), 41 (51.4).

Anal. Calcd for $C_7H_9ClO_2$: C, 52.35; H, 5.65; Cl, 22.10. Found: C, 52.24; H, 5.92; Cl, 22.12.

8: IR (KBr) ν_{max} 1775 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 4.85 (d, 1 H), 4.19–3.97 (complex m, 1 H), 2.75–1.55 (complex m, 7 H); ^{13}C NMR ($CDCl_3$) δ 26.41, 30.02, 37.13, 37.41, 57.12, 81.59, 177.67; mass spectrum, *m/e* (relative intensity) 162 ($^{37}ClM^+$, 1.5), 160 ($^{35}ClM^+$, 6.2), 124 (44.5), 120 (34.4), 118 (base peak), 97 (32), 83 (44.5), 81 (53.4), 80 (37.4), 70 (30.7), 42 (67.6), 41 (48).

Anal. Calcd for $C_7H_9ClO_2$: C, 52.35; H, 5.65; Cl, 22.10. Found: C, 52.30; H, 5.56; Cl, 22.18.

10: IR (KBr) ν_{max} 1788 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 4.53 (m, 1 H), 4.10 (m, 1 H), 2.53 (complex m, 3 H), 2.33–1.2 (complex m, 6 H); ^{13}C NMR ($CDCl_3$) δ 14.84, 19.36, 27.73, 30.94, 34.60, 35.92, 59.70, 83.71, 179.84; mass spectrum, *m/e* (relative intensity) 188 ($^{37}ClM^+$, 5.4), 186 ($^{35}ClM^+$, 13.9), 158 ($M^+ - CO$, 16.9), 151 ($M^+ - Cl$, 21.2), 150 (95.4), 131 (22.3), 123 (87.6), 122 (30.6), 107 (34.2), 95 (49.8), 91 (48.6), 80 (51.7), 79 (base peak), 78 (75.7), 77 (42), 70 (59.7), 67 (47.7), 65 (20.4), 41 (28.5).

Anal. Calcd for $C_9H_{11}ClO_2$: C, 57.61; H, 6.45, Cl, 18.89. Found: C, 57.23; H, 6.11; Cl, 18.77.

12: IR (neat) ν_{max} 1780 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.50–4.60 (complex m, 2 H), 4.00–3.80 (complex m, 4 H), 3.20–1.30 (complex m, 4 H); ^{13}C NMR ($CDCl_3$) δ 35.52, 36.78, 44.40, 44.69, 45.03, 45.38, 52.42, 76.49, 76.89, 77.46, 77.80, 173.03; mass spectrum, *m/e* (relative intensity) 254 ($^{37}ClM^+$, 0.4), 252 ($^{35}ClM^+$, 0.6), 208 ($M^+ - CO_2$, 11.0), 205 ($M^+ - CH_2Cl$, 34.1), 203 (base peak) 161 (25.5), 159 (80.7), 137 (30.5), 97 (56.6), 67 (76.2), 65 (20), 43 (21.8), 41 (52.4).

Anal. Calcd for $C_9H_{10}Cl_2O_4$: C, 42.71; H, 3.98; Cl, 28.02. Found: C, 42.77; H, 3.98; Cl, 27.96.

Registry No. 1, 127-65-1; 2, 591-80-0; 3, 39928-72-8; 4, 1577-22-6; 5, 77944-06-0; 6, 4771-80-6; 7, 20893-19-0; 8, 77924-81-3; 9, 40610-12-6; 10, 77924-82-4; 11, 4372-31-0; 12, 77944-07-1.

(9) Sharpless, K. B.; Hori, T.; Truesdale, L. K.; Dietrich, C. O. *J. Am. Chem. Soc.* **1976**, *98*, 269.

Acid-Catalyzed Annulation. Simple and Highly Stereospecific Synthesis of *cis*-5,10-Dimethyl-1(9)-octal-2-one

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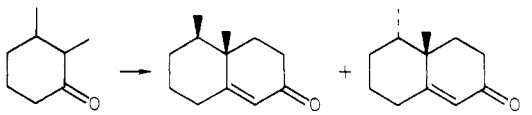
Received March 12, 1981

The problem of stereochemical control with respect to vicinally disposed *cis*-dimethyl groups of **1** which characterizes the eremophilane–valencane family of terpenes has been studied[1] by several groups of workers. Our interest in **1** resulted from a project directed toward the synthesis of certain eremophilane-type sesquiterpenoids. Direct Robinson annulation of 2,3-dimethylcyclohexanone with methyl vinyl ketone in the presence of base affords **1** and **2** in a nonstereospecific manner (3:2 trans/cis) and in poor



1 **2**

yield (15%).[2,3] Octalone **1** has also been obtained by dehydration of the purified aldol intermediate[4] resulting from ordinary annulation at −15 °C but in low yield.

A higher degree of stereospecifity (9:1, cis/trans) was observed in an alternative approach to (**1**) by reaction of the enolate of 2,3-dimethyl-6-[(*n*-butylthio)methylene]-cyclohexanone[2] and ethyl 3-bromopropionate, the corresponding keto esters being precursors to the *cis*- and *trans*-dimethyloctalones. A conjugate addition–annulation reaction[5] involving 1,4 conjugate addition to 2-methyl-2-cyclohexenone and alkylation of the resulting regiospecificly generated enolate with 3-(trimethylsilyl)-3-buten-2-one followed by subsequent cyclization has also been reported to afford **1** and **2** in high stereospecificity (97:3 cis/trans) as determined by VPC analysis.

These alternate syntheses of **1** involve multistep conversions of 2,3-dimethylcyclohexanone and 2-methyl-2-cyclohexenone followed by base-catalyzed cyclization to **1** and **2**. Although the overall yields of **1** are good (30–54%) and the realized stereospecificity is ≥90%, the overall pathways are six to seven steps in length and would require a substantial synthetic effort for the preparation of large quantities of **1**.

We report herein a simple and highly stereospecific synthesis of **1** which can be obtained via an acid-catalyzed[6,7] Robinson annulation reaction. When 2,3-dimethylcyclohexanone and methyl vinyl ketone in the presence of sulfuric acid in benzene were allowed to react

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