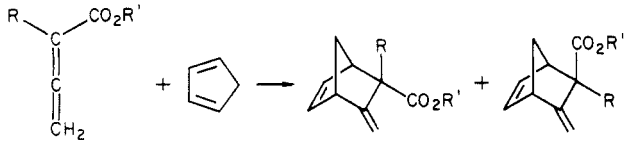
Table I. Endo/Exo Ratios for Diels-Alder Reactions of 2,3-Butadienoic Acid Derivatives (1–5) to Cyclopentadiene



dienophile endo exo

dienophile	R	R'	adduct endo	adduct exo	endo/exo ratio	reaction time, temp, and solvent	yield, %
1	H	H	1n	1x	68:32	12 h, 25 °C, Et_2O	86
2a	H	Me	2an	2ax	69:31[a]	reflux, benzene	85
2b	H	Et	2bn	2bx	64:36	6 h, 80 °C, benzene	90
2b + $AlCl_3$	H	Et			86:14	45 min, 25 °C, benzene	95
3a	Me	H	3an	3ax	40:60[b]		
3b	Me	Me	3bn	3bx	40:60	8 h, 80 °C, benzene	~80
3b + $AlCl_3$	Me	Me			76:24	45 min, 25 °C, benzene	90
4	n-C_5H_{11}	Me	4n	4x	40:60	12 h, 80 °C, benzene	50
5	$CH_2CH_2CH{=}CMe_2$	Me	5n	5x	48:52[c]	18 h, ~120 °C, toluene	50

[a] Reference 2k. [b] References 2f,g. [c] Endo epimer assumed to have the longer GC retention time on a glass capillary column.

evolution of hydrogen had ceased, the solution was heated to 90 °C, and homoallylic iodide **6** (10 g, 48 mmol) in 1,2-dimethoxyethane (25 mL) was added. After being refluxed for a further 20 h, the mixture was cooled and distributed between ether–water. The resulting organic phase was separated, dried ($MgSO_4$), and distilled, giving ethyl 2-acetyl-6-methylhept-5-enoate (7 g, 82%): 90-MHz ^1H NMR ($CDCl_3$) δ 1.26 (t, J = 7 Hz, 3 H, CH_2CH_3), 1.58 (s, 3 H, Me), 1.68 (s, 3 H, Me), 1.77–2.05 (m, 4 H), 2.22 (s, 3 H, COMe), 3.33–3.52 (m, 1 H), 4.18 (q, J = 7 Hz, 2 H, OCH_2), 4.94–5.2 (m, 1 H).

(iii) Preparation of the Pyrazolone. The ester (5.3 g, 25 mmol) in methanol was dropped into a solution of 80% hydrazine hydrate (1.6 g, 25 mmol) in methanol (30 mL) and stirred for 3 h at room temperature. The resulting white precipitate was filtered off and recrystallized from methanol: mp 218–220 °C; yield 3.5 g (83%); 90-MHz ^1H NMR (Me_2SO) δ 1.52 (s, 3 H), 1.63 (s, 3 H), 2.02 (s, 3 H), 2.16 (m, 4 H), 3.11–4.0 (br, 2 H), 5.11 (m, 1 H). Anal. Calcd for $C_{10}H_{16}N_2O$: C, 66.63; H, 8.95; N, 15.54. Found: C, 66.98; H, 9.23; N, 15.63.

(iv) Preparation of 5. A solution of thallium(III) nitrate (14 g, 36 mmol) in methanol (50 mL) was stirred into a suspension of 3 g (18 mmol) of the pyrazolone in methanol (25 mL) at room temperature. After 30 min the precipitated thallium salt was filtered off. The filtrate was poured onto water, extracted with $CHCl_3$, dried (Na_2SO_4), and distilled, giving **5**: 1.7 g (52%); IR ($CHCl_3$) 1708 (ester), 1930, 1960 cm^{-1} (allene); 90-MHz ^1H NMR (CCl_4) δ 1.59 (s, 3 H, Me), 1.67 (s, 3 H, Me), 2.0–2.24 (m, 4 H), 3.68 (s, 3 H, OMe), 4.88–5.22 (m, 3 H).

Cycloaddition Procedure. Commercial aluminum trichloride (Merck) (ca. 0.5–1 molar equiv with respect to **2b** and **3b**) was vigorously stirred into a solution of the allenic ester (9 mmol) over a period of 20 min, and cyclopentadiene (0.9 g, 1.5 equiv) was added. The reaction was followed by GC. After 45 min, ester **2b** could no longer be detected, and the reaction mixture was worked up by the usual procedure.

3-Methylenebicyclo[2.2.1]hept-5-ene-2-carboxylic acid (1n,x): 90-MHz ^1H NMR ($CDCl_3$) δ 1.33–2 (m, 4 H, 2 CH_2), 2.77–2.88 (dd, 1 H, *endo*-H), 3.16–3.33 (m, 4 H, bridgehead H's), 3.42–3.53 (dt, 1 H, *exo*-H), 5.0–5.22 (2 d, 4 H, 2 CH_2=), 6.18 (t, J = 2 Hz, 4 H, 2 CH=CH), 10.88–11.33 (br, 2 H, 2 CO_2H). The spectral properties of the ethyl esters **2bn,bx** have been described.[9]

Methyl 2-methyl-3-methylenebicyclo[2.2.1]hept-5-ene-2-carboxylate (3bn,bx) (uncatalyzed cycloaddition): 90-MHz ^1H NMR δ 1.24 (s, 3 H, *endo*-CH_3), 1.5 (s, 3 H, *exo*-CH_3), 1.58–1.70 (m, 2 CH_2), 2.85–3.33 (m, 4 H, bridgehead H's), 3.62 (s, 3 H, *endo*-OCH_3), 3.7 (s, 3 H, *exo*-OCH_3), 4.9–5.2 (m, 4 H, 2 CH_2=), 6.1–6.3 (m, 4 H, 2 CH=CH). The **3bn/3bx** ratio was 40:60 (^1H NMR, GC).

Methyl 2-*n*-pentyl-3-methylenebicyclo[2.2.1]hept-5-ene-2-carboxylate (4n,x): 90-MHz ^1H NMR ($CDCl_3$) δ 0.77–1.04 (m, 6 H, 2 CH_3), 1.04–1.52 (m, 16 H, 8 CH_2) 1.52–1.76 (m, 4 H, 2 bridge CH_2's), 3.0–3.36 (m, 4 H, bridgehead H's), 3.62 (s, 3 H, *endo*-OCH_3), 3.71 (s, 3 H, *exo*-OCH_3), 5.08 (d, 4 H, 2 CH_2=), 6.0–6.27 (m, 4 H, 2 CH=CH); IR ($CHCl_3$) 1710 cm^{-1}. For **4n**: GC/MS (t_R = 35.2 min), m/e (relative intensity) 234 (M^+, 19), 175 (23), 170 (34), 160 (34), 146 (34), 145 (36), 132 (42), 131 (29), 119 (45), 117 (100), 115 (30), 105 (34), 103 (46), 99 (39), 94 (48), 92 (47), 79 (21), 77 (43), 66 (97). For **4x**: GC/MS (t_R = 34.4 min) m/e (relative intensity) 234 (M^+, 34), 175 (29), 146 (36), 145 (39), 132 (46), 131 (34), 119 (34), 117 (97), 115 (29), 105 (36), 94 (46), 92 (24), 77 (34), 66 (100).

Methyl 3-Methylene-2-(4-methyl-3-pentenyl)bicyclo-[2.2.1]hept-5-ene-2-carboxylate (5n,x). For 5n: GC/MS (t_R = 31.6 min); mass spectrum, m/e (relative intensity) 246 (M^+, 13), 231 (7), 215 (15), 214 (11), 203 (10), 190 (13), 187 (37), 180 (100), 177 (21), 165 (42), 158 (48), 148 (99), 133 (76), 121 (52), 120 (56), 117 (51), 105 (72), 91 (52), 82 (13), 79 (43), 66 (99), 59 (18). **For 5x**: GC/MS (t_R = 31.4 min); mass spectrum, m/e (relative intensity) 246 (M^+, 16), 231 (4), 215 (11), 214 (16), 203 (9), 190 (26), 187 (19), 180 (100), 177 (16), 165 (29), 158 (44), 148 (93), 133 (31), 121 (29), 120 (24), 117 (26), 105 (44), 91 (34), 82 (21), 79 (24), 66 (49), 59 (16). Two additional $C_{16}H_{22}O_2$ isomers (GC/MS m/e 246 (M^+)) of unknown structure were also detected in ca. 20% yield.

Registry No. 1, 5732-10-5; **1n**, 24657-50-9; **1x**, 67903-38-2; **2a**, 18913-35-4; **2an**, 66241-97-2; **2ax**, 77965-89-0; **2b**, 14369-81-4; **2bn**, 77965-90-3; **2bx**, 78018-39-0; **3a**, 18913-36-5; **3an**, 52558-07-3; **3ax**, 32763-66-9; **3b**, 18913-37-6; **3bn**, 77965-91-4; **3bx**, 77965-92-5; **4**, 35895-74-0; **4n**, 77965-93-6; **4x**, 77965-94-7; **5**, 77965-95-8; **5n**, 77965-96-9; **5x**, 77965-97-0; **6**, 43161-11-1; cyclopentadiene, 542-92-7; cyclopropyldimethylmethanol, 930-39-2; ethyl acetoacetate, 141-97-9; ethyl 2-acetyl-6-methylhept-5-enoate, 42809-53-0; 5-methyl-4-(4-methylpent-3-enyl)-2,4-dihydro-3*H*-pyrazol-3-one, 77965-98-1.

(9) Hoffmann, H. M. R.; Ismail, Z. M.; Weber, A. *Tetrahedron Lett.* 1981, 22, 1953.

Internal Nucleophilic Termination in Mercuric Ion Initiated Diene Cyclizations

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Recent interest in the internal trapping by an appropriately placed nucleophilic functional group of a carbo-

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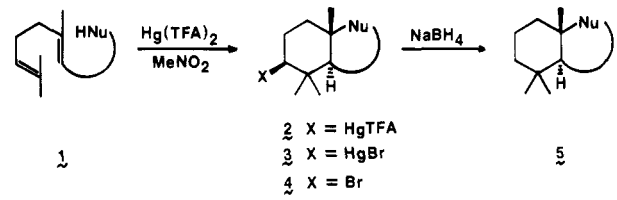
Table I. Conversion of Dienes 1 into Cyclized Products 5 via Organomercury Bromides 3

entry	substrate	product	% yield[a] X = HgBr	% yield[a] X = H
a	1a		3a	5a 88%
b	1b		3b 51%	5b 94%
c	1c		3c	5c 62%[b]
d	1d		3d	5d 65%[b]
e	1e		3e 59%	5e 98%
f	1f		3f 72%	5f 86%
g	1g		3g 41%	5g 98%
h	1h		3h	5h 20%
i	1i	+ SM + others (24%)		

[a] Yields refer to purified products for each step. If no yield is reported for 3, the crude organomercury bromides were reduced directly to 5 without purification. [b] See ref 3.

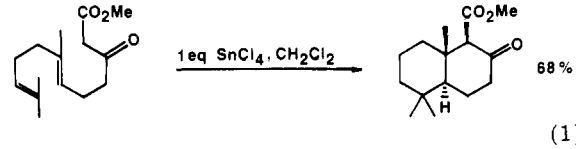
cation generated by cation-induced polyolefin cyclization is exemplified by reports from the Johnson laboratories.[2] In these instances primary alcohol and carboxylic acid groups[2a] and allyl-[2b] and propargylsilanes[2c] have served as terminators for cyclizations performed in the absence of external nucleophiles ($SnCl_4$ or CF_3COOH in CH_2Cl_2). We had occasion to compare the trapping ability of several internal oxygen containing functional groups in the mercuric ion initiated cyclization of simple 1,5-dienes of structure 1 and now describe our observations.

Dienes **1a–h** were treated with 1.1 equiv of mercuric trifluoroacetate in nitromethane at room temperature following the methodology of Semenovsky.[3] The results of these cyclizations are summarized in Table I. The crude organomercury trifluoroacetates **2** could be isolated if necessary but were usually converted directly to the mercury bromides **3** by ligand exchange with potassium bromide. In several instances these substances were isolated by chromatography on silica gel. We have previously described the conversion of the organomercury bromides **3a** and **3g** to the corresponding alkyl bromides **4a** and **4g**.[4] In this study the mercury atom in **3** was removed by sodium borohydride reduction to the hydrocarbon **5**.

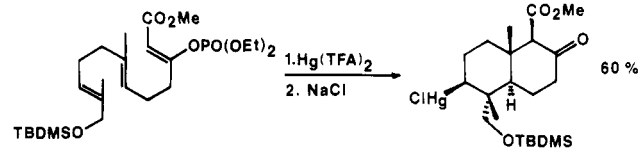


1 2 X = HgTFA 3 X = HgBr 4 X = Br 5

Carboxylic acids (Table I, entries a and b), ketones (entries c–f), and alcohols (entries h and i) are effective trapping nucleophiles whereas acetate esters (entry i) and the primary amide corresponding to acid **1a** (which gave a complex mixture of pruducts upon exposure to Hg(TFA)$_2$) are not. Both *E* and *Z* ketones **1c** and **1d** give bicyclic products,[3] but the *Z* alcohol **6** generates a host of products upon attempted cyclization. Ketones **1c–f** lead entirely to the endocyclic enol ethers **5c–f** even though conjugation to the ester is sacrificed in the last instance. In addition, cyclization occurs exclusively on oxygen rather than carbon even with the β-keto esters **1e** and **1f**. It is worth contrasting this result with the reactions reported by White[5] and Corey,[6] shown in eq 1 and 2, respectively, in which cyclization occurred only on carbon.[7]



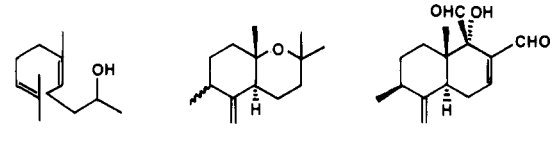
(1)



(2)

The relative ease of cyclization to six- vs. seven-membered ethers is seen in the yields of products **5g** and **5h**. The latter example demonstrates the greater propensity of hydroxyl in direct competition with carbalkoxy group to trap the intervening cation. No other monomeric materials were isolated from the reaction of **1h**.

The effective yield in the cyclization of tertiary alcohol **1g** to **3g** was anomolously low because that organomercury bromide was accompanied by a byproduct lacking mercury. Separation provided **3g** in 41% yield and a less polar olefin assigned structure **7** [on the basis of a methyl doublet at



6 7 8

δ 0.96 and olefinic resonances as broad singlets at δ 4.71

(1) Eastman Kodak Fellow, 1979–80.

(2) (a) Hughes, L. R.; Schmid, R.; Johnson, W. S. *Bioorg. Chem.* **1979**, *8*, 513. (b) Garst, M. E.; Cheung, Y.-F.; Johnson, W. S. *J. Am. Chem. Soc.* **1979**, *101*, 4404. (c) Schmid, R.; Huesmann, P. L.; Johnson, W. S. *Ibid.* **1980**, *102*, 5122.

(3) Kurbanov, M.; Semenovsky, A. V.; Smit, W. A.; Shmelev, L. V.; Kucherov, V. F. *Tetrahedron Lett.* **1972**, 2175.

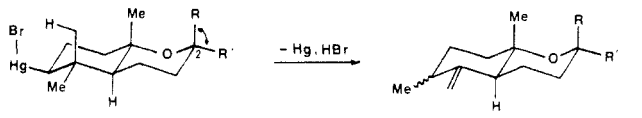
(4) Hoye, T. R.; Kurth, M. J. *J. Org. Chem.* **1979**, *44*, 3461.

(5) Skeean, R. W.; Trammell, G. L.; White, J. D. *Tetrahedron Lett.* **1976**, 525.

(6) Corey, E. J.; Tius, M. A.; Das, J. *J. Am. Chem. Soc.* **1980**, *102*, 1743.

(7) In an intermolecular version, acetylacetone has been reacted with ethylene and Hg(OAc)$_2$ to provide, after ligand exchange, 3-acetyl-5-chloromercuricpentan-2-one, the product of carbon alkylation, in 87% yield: Ichikawa, K.; Itoh, O.; Kawamura, T.; Fujiwara, M.; Ueno, T. *J. Org. Chem.* **1966**, *31*, 447.

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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Received November 5, 1980

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 © 1981 American Chemical Society