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(4) Et_4NF had a similar effect in acetone. However, neither Et_4NOAc nor Et_4NF had much effect on the reaction in *tert*-butyl alcohol (TBA), whereas the tetraethylammonium salts of chelating diacids such as *o*-phthalic, camphoric, and *cis*-1,2-cyclohexane dicarboxylic acid did substantially improve the reaction even in TBA. $C_6H_5PO_3(Et_4N)_2$ and $(Et_4N)_2CO_3$ also had good effects on the reaction in TBA.

(5) The use of acetone in place of *tert*-butyl alcohol as solvent dramatically increases the beneficial effect of weak bases, such as Et_4NOAc, on these reactions (see also ref 4).

(6) (a) V. Van Rheenen, R. C. Kelly and P. Y. Cha, *Tetrahedron Lett.*, 1973 (1976). (b) We are grateful to a referee for pointing out this patent which describes the use of TBHP in buffered (slightly alkaline) aqueous solution for the osmium catalyzed hydroxylation of allyl alcohol to glycerol [M. N. Sheng and W. A. Mameniskis, U.S. Patent 4 049 724 (1977)].

(7) A number of other osmium complexes were tried and proved to be equally active as catalysts. For example, in the oxidation of (*E*)-4-octene, OsO_3 (pyridine)$_2$, $K_2O_2Os(OCH_3)_4$, and the imido complex OsO_3 (*N-tert*-butyl) all gave yields of diol comparable to that realized with OsO_4 as catalyst. These nonvolatile solids can simply be weighed out (0.2% based on olefin) and added to the reactions in place of the portion of OsO_4 stock solution.

(8) (a) K. Akashi and K. B. Sharpless, unpublished results; (b) K. B. Sharpless, A. O. Chong, and K. Oshima. *J. Org. Chem.*, **41**, 177 (1976).

(9) More hydrophobic olefins may oil out of the acetone solution due to the additional water in the 70% *tert*-butyl hydroperoxide. This only slows the initial rate and has no adverse effect on the reaction.

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(11) In the present work we did not encounter any problems in using sodium bisulfite ($NaHSO_3$) as the reagent to reduce the excess *tert*-butyl hydroperoxide (TBHP). However, in other work[12] we have found that use of $NaHSO_3$ can have a deleterious effect on the isolated yields. The problem was especially serious when the product to be isolated contained either epoxide or allylic alcohol moieties. For more detailed discussion of this problem and for alternative means of dealing with the excess TBHP, see ref 12.

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A Synthesis of α-Azido Nitriles

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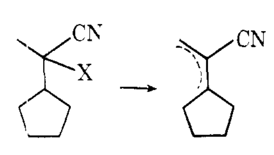
As part of our program to study the chemistry of nitriles bearing photoactive functionality in the α position,[1] we required a general synthesis of α-azidonitriles **4**. This intriguing synthon was first prepared by Moore[2] in the photochemical rearrangement of 2,3-diazido-1,4-quinones. Although the scope of this rearrangement process has not been determined, we were interested in devising other approaches which would utilize ketones **1** as starting materials.

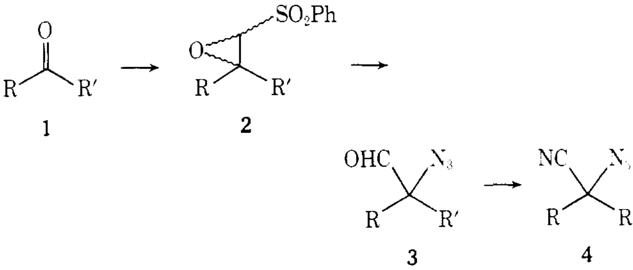
Our initial foray in this area focused on the substitution of α-iodo or α-mesyloxynitriles by azide ion. Although aware of the difficulties which beset such a reaction, we were prodded into exploring this reaction by a report[3] that tertiary α-bromo ketones underwent just such a substitution with azide ion in 82–88% yield. Our efforts to utilize **5** in such a reaction led exclusively to the expected α,β-unsaturated nitriles[4] **6**.



5a, X = I
b, X = OMs

6

An alternate route involving the ring opening of an epoxide ultimately proved successful in this connection. A modified Darzens condensation of ketones **1** with chloromethyl phenyl sulfone[5] provided the α,β-epoxy sulfones[6] **2**. Contrary to a report by Durst,[7] the ring opening of **2** with sodium azide in dimethylformamide provided the α-azidoaldehydes **3** in good yields. Conversion of **3** to the α-azidonitriles **4** was then accomplished by dehydration of the oximes derived from the aldehydes **3**. We have applied this sequence to the synthesis of α-azidonitriles **4** from aryl alkyl and dialkyl ketones **1** in overall yields of 23 to 59% as shown in Table I. We have also explored the direct conversion of **3** to **4** using reagents such as hydroxylamine *O*-sulfonic acid but found that this latter procedure offered certain disadvantages. For example, the reaction of 20-azido-6β-methoxy-3α,5α-cyclopregnane-20-carboxyaldehyde (**3g**) with hydroxylamine *O*-sulfonic acid converted not only the aldehyde to the nitrile but also effected the ring opening of the isocyclopropyl ether to give 20-azido-3β-hydroxy-5-pregnene-20-carbonitrile. We are presently engaged in studying the photochemistry of α-azidoaldehydes **3** and α-azidonitriles **4**.



Experimental Section

Infrared spectra were determined on a Perkin-Elmer infracord spectrophotometer. The abbreviation TF denotes thin film. NMR spectra were determined on a Varian EM390 spectrometer. Mass spectra were determined on a Varian MAT CH5 mass spectrometer. Melting points were determined using a Thomas-Hoover apparatus and are uncorrected. Elemental analyses were performed by Atlantic Microlabs, Atlanta, Ga. Samples for elemental analysis were prepared by recrystallization or by chromatography on Merck silica gel F254 preparative layer plates followed by drying under high vacuum at 25 °C for 6–10 h.

The following is a typical experimental procedure.

1,1-Undecamethylene-2-(benzenesulfonyl)-1,2-epoxyethane (2c). The procedure of Tarares[6] was repeated using 1.05 g (5.5 mmol, 1.1. equiv) of chloromethyl phenyl sulfone[5] and 910 mg (5.0 mmol) of cyclododecanone to afford 1.69 g of solid which was recrystallized to furnish 1.15 g (68%) of the α,β-epoxy sulfone **2c**: mp 102–103 °C; IR (KBr) 7.55 and 8.69 μm; NMR ($CDCl_3$) δ 1.17–2.36 (m, 22, CH_2), 3.72 (s, 1, $CHSO_2Ph$), and 7.50–8.02 (m, 5, aromatic H); mass spectrum (70 eV) *m/e* (rel intensity) 250 (4), 196 (10), 185 (68), 177 (8, $M^+ - (PhSO_2 + H_2O)$), 94 (100), and 77 (31). The loss of *m/e* 159 was characteristic of all α,β-epoxy sulfones.

An analytical sample was prepared from two recrystallizations from dichloromethane–ether. Anal. Calcd for $C_{19}H_{28}O_3S$: C, 67.82; H, 8.39. Found: C, 67.83; H, 8.39.

1-Azidocyclododecane-1-carboxaldehyde (3c). To 890 mg (13.7 mmol, 4 equiv) of sodium azide in 10 mL of anhydrous dimethylformamide under a nitrogen atmosphere was added 1.15 g (3.4 mmol) of α,β-epoxy sulfone **2c** in 10 mL of dimethylformamide. The mixture was stirred for 18 h at 73 °C. This crude product was diluted with 60 mL of 30% dichloromethane–ether and washed with 50 mL of water. The aqueous layer was extracted with 60 mL of 30% dichloromethane–ether. The combined organic layers were washed with 50 mL of water and 50 mL of brine and dried over anhydrous $MgSO_4$. Evaporation of the solvent afforded 821 mg (100%) of **3c**: IR (TF) 4.76 and 5.80 μm; NMR ($CDCl_3$) δ 1.26–1.90 (m, 22, CH_2) and 9.48 (s, 1, CHO); mass spectrum (70 eV) *m/e* (rel intensity) 181 (50), 138 (29), 124 (44), 95 (44), 81 (39), and 69 (45).

Anal. Calcd for $C_{13}H_{23}N_3O$: C, 65.78; H, 9.77. Found: C, 65.99; H, 9.82.

1-Azidocyclododecane-1-carbonitrile (4c). To 530 mg (7.68 mmol, 3 equiv) of hydroxylamine hydrochloride and 307 mg (7.68

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Table I. Synthesis of α-Azidonitriles 4

	Ketone 1			Base used in condensation	Isolated Yields, %					
4	R	R′	Registry no.	**1** → **2** (equiv)	2	Registry no.	3	Registry no.	4	Registry no.
a	$-(CH_2)_5-$		108-94-1	KO-*t*-Bu (1.1)	97	28937-60-2	52	65516-42-9	67	65545-20-2
b	$-(CH_2)_7-$		502-49-8	KO-*t*-Bu (1.5)	*a*[b]	65516-36-1	63[d]	65516-43-0	66	65516-49-6
c	$-(CH_2)_{11}$		830-13-7	KO-*t*-Bu (1.1)	68	65516-37-2	100	65516-44-1	87	65516-50-9
d	CH_3	Ph	98-86-2	KO-*t*-Bu (1.5)	*a*[b]	65516-38-3	68	65516-45-2	60	65516-51-0
e	CH_2CH_2Ph	CH_2CH_2Ph	5396-91-8	KO-*t*-Bu (1.1)	71	65516-39-4	87	65516-46-3	63	65516-52-1
f	c-C_5H_9	CH_3	6004-60-0	KO-*t*-Bu (1.5)	79[b]	65516-40-7	86[d]	65516-47-4	74	65516-53-2
g	6β-Methoxy-3α,5α-cyclopregnan-20-one		32249-55-1	LiN(*i*-Pr)$_2$ (2)	49	65516-41-8	78	65516-48-5	59[c]	65516-54-3

[a] α,β-Epoxy sulfone **2** was unstable to preparative layer chromatography and crude **2** was converted directly to **3**. [b] Used 1.5 equiv of chloromethyl phenyl sulfone. [c] Used 2 equiv of hydroxylamine hydrochloride and sodium hydroxide. [d] Reaction temperature was 45–50 °C.

mmol, 3 equiv) of sodium hydroxide in 7 mL of water was added 607 mg (2.6 mmol) of the α-azidoaldehyde **3c** in 7 mL of THF. This mixture was stirred at 63 °C for 13 h. The crude product was diluted with 60 mL of 30% dichloromethane–ether and washed with 40 mL of water. The aqueous layer was extracted with 60 mL of 30% dichloromethane–ether. The combined organic layers were washed with 40 mL of water and 40 mL of brine and dried over anhydrous $MgSO_4$. Evaporation of the solvents afforded 666 mg of a light yellow solid. To this crude product and 650 mg (6.40 mmol, 2.5 equiv) of triethylamine in 20 mL of dichloromethane at 0 °C was slowly added 325 mg (2.82 mmol, 1.1 equiv) of methanesulfonyl chloride. This solution was stirred at 25 °C for 1 h. The reaction was poured into 50 mL of cold water and extracted with 50 mL of ether. The aqueous layer was reextracted with 50 mL of 30% dichloromethane–ether. The combined organic layers were washed with 50 mL of saturated sodium bicarbonate solution and 50 mL of brine and dried over anhydrous $MgSO_4$. Evaporation of the solvent afforded 598 mg of a yellow solid which was chromatographed on two 20 × 20 cm (2 mm thick) Merck silica gel F254 preparative layer plates in 2:1 dichloromethane–hexane. A band (R_f 0.61) was eluted to afford 521 mg (87%) of **4c**: mp 46–47 °C; IR (KBr) 4.69 and 4.75 μm; NMR ($CDCl_3$) δ 1.20–2.13 (m, 22, CH_2); mass spectrum (70 eV) *m/e* (rel intensity) 192 (50), 163 (25), 149 (34), 135 (41), 121 (33), 80 (50), and 55 (100).

Anal. Calcd for $C_{13}H_{22}N_4$: C, 66.63; H, 9.46. Found: C, 66.63; H, 9.48.

Spectral Data for α,β-Epoxy Sulfones 2. 2a: IR ($CHCl_3$) 7.61 and 8.60 μm; NMR ($CDCl_3$) δ 1.37–2.36 (m, 10, CH_2), 3.74 (s, 1, $CHSO_2Ph$), and 7.39–8.10 (m, 5, aromatic H); mass spectrum (70 eV) *m/e* (rel intensity) 190 (7), 143 (30), 111 (95), 93 (63), 82 (9), 77 (41), and 76 (100).

Anal. Calcd For $C_{13}H_{16}O_3S$: C, 61.89; H, 6.39. Found: C, 61.84; H, 6.41.

2e: IR(TF) 7.55 and 8.52 μm; NMR ($CDCl_3$) δ 1.80–3.16 (m, 8, CH_2), 3.81 (s, 1, $CHSO_2Ph$), 6.97–7.38 (m, 10, aromatic H), and 7.50–8.03 (m, 5, aromatic H); mass spectrum (70 eV) *m/e* (rel intensity) 251 (7), 250 (13), 233 (8), 91 (100), and 77 (13).

Anal. Calcd for $C_{24}H_{24}O_3S$: C, 73.45; H, 6.16. Found: C, 73.20; H, 6.21.

2f: IR (TF) 7.61 and 8.60 μm; NMR ($CDCl_3$) δ 1.79 (s, 3, CH_3), 3.78 (s, 1, $CHSO_2Ph$), and 7.40–8.02 (m, 5, aromatic H); mass spectrum (70 eV) *m/e* (rel intensity) 143 (19), 125 (44), 107 (24), 94 (40), 77 (19), and 67 (77).

Anal. Calcd for $C_{14}H_{18}O_3S$: C, 63.14; H, 6.81. Found: C, 62.91; H, 6.88.

20-Benzenesulfonylmethyl-20,22-epoxy-6β-methoxy-3α,5α-cyclopregnane (2g). To a solution of 101 mg (1.0 mmol, 2 equiv) of diisopropylamine in 0.5 mL of THF under a nitrogen atmosphere at −78 °C was added 0.45 mL of 2.23 M (1 mmol, 2 equiv) *n*-butyllithium. The solution was stirred for 10 min at −78 °C and then warmed to 25 °C. To this diisopropylamide solution, 191 mg (1 mmol, 2 equiv) of chloromethyl phenyl sulfone[5] was added in 0.5 mL of THF. The reaction was stirred for 15 min and 165 mg (0.5 mmol) of 6β-methoxy-3α,5α-cyclopregnan-20-one[9] (**1g**) was added in 0.5 mL of THF. The reaction was stirred at 25 °C for 48 h, diluted with 30 mL of ether, washed two times with 10 mL of water and 10 mL of brine, and dried over anhydrous $MgSO_4$. Evaporation of the solvent afforded 220 mg of a brown oil. The product was chromatographed on two 20 × 20 cm (2 mm thick) Merck silica gel F254 preparative layer plates in 40:1 benzene–ether. After two developments, a band (R_f 0.44) was eluted to afford 0.12 g (49%) of **2g:** mp 50–67 °C; IR ($CHCl_3$) 7.57 and 8.61 μm; NMR ($CDCl_3$) δ 0.79 and 1.01 (two s, 6, C-18 and C-19 angular CH_3), 1.88 (s, 3, C-21 CH_3), 2.73 (t, *J* = 3 Hz, 1, C-6α H), 3.30 (s, 3, OCH_3), 3.68 (s, 1, $CHSO_2Ph$), and 7.42–8.01 (m, 5, aromatic H); mass spectrum (70 eV) *m/e* (rel intensity) 484 (52, M^+), 343 (71), 311 (76), 288 (66), 199 (21), 159 (46), and 90 (100).

Anal. Calcd for $C_{29}H_{40}O_4S$: C, 71.87; H, 8.32. Found: C, 71.64; H, 8.40.

Spectral Data for α-Azidoaldehydes 3. 3a:[8] IR (TF) 4.76 and 6.13 μm; NMR ($CDCl_3$) δ 1.10–2.19 (m, 10, CH_2), and 9.42 (s, 1, CHO); mass spectrum (70 eV) *m/e* (rel intensity) 125 (1), 124 (3), 111 (1), 110 (1), 97 (6), 96 (66), 81 (5), 55 (100), and 54 (20).

3b: IR (TF) 4.75 and 5.78 μm; NMR ($CDCl_3$) 1.43–2.09 (m, 14, CH_2), and 9.44 (s, 1, CHO); mass spectrum (70 eV) *m/e* (rel intensity) 124 (30), 82 (22), 81 (42), 78 (20), and 55 (100).

Anal. Calcd for $C_9H_{15}N_3O$: C, 59.64; H, 8.34. Found: C, 59.75; H, 8.38.

3d: IR (TF) 4.76 and 5.75 μm; NMR ($CDCl_3$) δ 1.77 (s, 3, CH_3), 7.36 (s, 5, aromatic H), and 9.41 (s, 1, CHO); mass spectrum (70 eV) *m/e* (rel intensity) 147 (9), 119 (7), 118 (58), 77 (100), and 51 (25).

Anal. Calcd for $C_9H_9N_3O$: C, 61.70; H, 5.18. Found: C, 61.76; H, 5.25.

3e: IR (TF) 4.74 and 5.75 μm; NMR ($CDCl_3$) δ 1.84–2.99 (m, 8, CH_2), 7.02–7.41 (m, 10, aromatic H), and 9.58 (s, 1, CHO); mass spectrum (70 eV) *m/e* (rel intensity) 236 (5), 132 (4), 105 (100), and 91 (34).

Anal. Calcd for $C_{18}H_{19}N_3O$: C, 73.69; H, 6.53. Found: C, 73.66; H, 6.56.

3f: IR (TF) 4.75 and 5.78 μm; NMR ($CDCl_3$) δ 1.40 (s, 3, CH_3), 9.49 (s, 1, CHO); mass spectrum (70 eV) *m/e* (rel intensity) 139 (1), 111 (4), 95 (2), 70 (8), and 69 (98).

Anal. Calcd for $C_8H_{13}N_3O$: C, 57.46; H, 7.84. Found: C, 57.28; H, 7.88.

3g: IR (TF) 4.75 and 5.78 μm; NMR ($CDCl_3$) δ 0.84 and 1.01 (two s, 6, C-18 and C-19 angular CH_3), 1.45 (s, 3, C-21 CH_3), 2.74 (t, *J* = 3 Hz, 1, C-6α H), 3.30 (s, 3, OCH_3), and 9.52 (s, 1, CHO); mass spectrum (70 eV) *m/e* (rel intensity) 385 (10, M^+), 330 (35), 328 (100), 255 (78), and 121 (20).

An analytical sample was prepared from three recrystallizations from hexane, mp 85–86.5 °C.

Anal. Calcd for $C_{23}H_{35}N_3O_2$: C, 71.65; H, 9.15. Found: C, 71.61; H, 9.16.

Spectral Data for α-Azidonitriles 4. 4a:[8] IR (TF) 4.57 and 4.74 μm; NMR ($CDCl_3$) δ 1.09–2.28 (m, 10, CH_2); mass spectrum (70 eV) *m/e* (rel intensity) 150 (9, M^+), 108 (51), 93 (23), 82 (11), 83 (100), 54 (30), and 42 (78).

4b:[8] IR (TF) 4.74 μm; NMR ($CDCl_3$) δ 1.40–2.25 (m, 14, CH_2); mass spectrum (70 eV) *m/e* (rel intensity) 136 (24), 121 (19), 109 (25), 107 (33), and 93 (36).

4d: IR (TF) 4.70 μm; NMR ($CDCl_3$) δ 1.90 (s, 3, CH_3) and 7.32–7.66 (m, 5, aromatic H); mass spectrum (70 eV) *m/e* (rel intensity) 172 (6, M^+), 131 (11), 130 (100), 103 (41), and 77 (48).

Anal. Calcd for $C_9H_8N_4$: C, 62.77; H, 4.68. Found: C, 62.56; H, 4.71.

4e: IR ($CHCl_3$) 4.69 (sh) and 4.76 μm; NMR ($CDCl_3$) δ 1.93–3.02 (m, 8, CH_2), 7.09–7.50 (m, 10, aromatic H), 9.60 (s, 1, CHO); mass spectrum (70 eV) *m/e* (rel intensity) 262 (9), 248 (1), 171 (15), 158 (52), 105 (49), 91 (100), and 77 (12).

Anal. Calcd for $C_{18}H_{18}N_4$: C, 74.45; H, 6.25. Found: C, 74.29; H, 6.30.

4f: IR (TF) 4.72 (sh) and 4.78 μm; NMR ($CDCl_3$) δ 1.60 (s, 3, CH_3); mass spectrum (70 eV) *m/e* (rel intensity) 122 (13), 94 (16), 69 (100), 67 (71), and 53 (31).

Anal. Calcd for $C_8H_{12}N_4$: C, 58.21; H, 7.37. Found: C, 58.38; H, 7.42.

4g: mp 64–66 °C; IR (TF) 4.76 μm; NMR ($CDCl_3$) δ 0.94 and 1.01 (two s, 6, C-18 and C-19 angular CH_3), 1.69 (s, 3, C-21 CH_3), 2.75 (t, J = 3 Hz, 1, C-6α H), and 3.30 (s, 3, OCH_3); mass spectrum (70 eV) *m/e* (rel intensity) 382 (50, M^+), 368 (52), 351 (81), 328 (100), 159 (19), and 119 (24).

Anal. Calcd for $C_{23}H_{34}N_4O$: C, 72.21; H, 8.96. Found: C, 72.20; H, 8.97.

2-Cyclopentyl-2-iodopropanenitrile (5a). To 486 mg (3.0 mmol, 1.5 equiv) of iodine monochloride at −10 °C under a nitrogen atmosphere was added 474 mg (2.0 mmol) of *N-tert*-butyldimethylsilylcyclopentylmethylketenimine[10] in 2 mL of anhydrous THF. This dark brown solution was stirred for 1 h at 25 °C, diluted with 25 mL of ether, and washed with 25 mL of water. The aqueous layer was reextracted with 25 mL of ether, and the combined organic layers were washed with two 25-mL portions of a saturated sodium thiosulfate solution, with two 25-mL portions of water, and with 25 mL of brine and dried over anhydrous $MgSO_4$. Evaporation of the solvent afforded 542 mg of a brown oil which was chromatographed on a 20 × 20 (2 mm thick) Merck silica gel F254 preparative layer plate in 5:1 hexane–ether. A band (R_f 0.57) was eluted to afford 182 mg (37%) of **5a:**[8] IR (TF) 4.50 μm; NMR ($CDCl_3$) δ 2.25 (s, 3, CH_3); mass spectrum (70 eV) (rel intensity) 127 (3), 122 (93), 105 (11), 95 (63), 80 (21), and 67 (100).

2-Cyclopentyl-2-hydroxypropanenitrile Mesylate (5b). The procedure of Crossland[11] was repeated using 139 mg (1.0 mmol) of 2-cyclopentyl-2-hydroxypropanenitrile, 126 mg (1.1 mmol, 1.1 equiv) of methanesulfonyl chloride, and 111 mg (1.1 mmol, 1.1 equiv) of triethylamine in 3 mL of anhydrous dichloromethane at 0 °C to afford 195 mg (90%) of **5b:** IR ($CHCl_3$) 7.32 and 8.41 μm; NMR ($CDCl_3$) δ 1.94 (s, 3, CH_3) and 3.16 (s, 3, SO_2CH_3); mass spectrum (70 eV) *m/e* (rel intensity) 138 (11), 122 (62), 95 (86), 79 (18), and 69 (100).

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Registry No.—**5a,** 65516-55-4; **5b,** 65516-56-5; chloromethyl phenyl sulfone, 7205-98-3; *N-tert*-butyldimethylsilylcyclopentylmethylketenimine 65516-57-6; 2-cyclopentyl-2-hydroxypropanenitrile, 65516-58-7.

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Alternative Route to Three of the Four Terpenoid Components of the Boll Weevil Sex Pheromone

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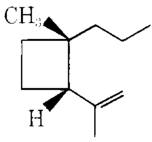
The ecological imbalance and environmental pollution due to insectiside residues has stimulated a great interest in the synthesis of pheromones, since they may provide a generally nontoxic method of biological control of insect populations.[1] A pheromone complex emitted by live male boll weevils (*Anthonomus grandis* Boheman) comprising the four terpenoid compounds (+)-*cis*-2-isopropenyl-1-methylcyclobutaneethanol (**1**), (*Z*)-3,3-dimethyl-$\Delta^{1,\beta}$-cyclohexaneethanol (**2**), (*Z*)-3,3-dimethyl-$\Delta^{1,\alpha}$-cyclohexaneacetaldehyde (**3**), and (*E*)-3,3-dimethyl-$\Delta^{1,\alpha}$-cyclohexaneacetaldehyde (**4**) were identified and first synthesized by Tumlinson et al.[2] We would like to report a simple sequence of reactions which afford the synthesis of alcohol **2** and aldehydes **3** and **4** in high yield from readily available starting materials.



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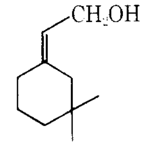

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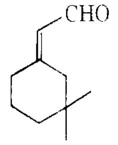

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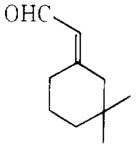

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Scheme I shows the synthesis of three cyclohexyl constituents of the boll weevil pheromone. 3,3-Dimethylcyclohexanone (**6**), utilized in previous syntheses,[3-5] was prepared from commercially available 3-methyl-2-cyclohexen-1-one by conjugate addition.[6] Reaction of ketone **6** with triethyl orthoformate in anhydrous ethanol and a catalytic amount of *p*-toluenesulfonic acid afforded 3,3-dimethylcyclohexanone diethyl ketal (**7**) in 87% yield. Treatment of ketal **7** with ethyl vinyl ether in a 10% $ZnCl_2$–ethyl acetate solution[7] gave the acetal **8** in 94% yield. Hydrolysis of compound **8** with glacial acetic acid, sodium acetate, and water afforded the isomeric aldehydes **3** and **4**, in 84% yield.[8] Thus, aldehydes **3** and **4** were prepared in 69% overall yield from starting material **6**. Reduction of a mixture of aldehydes 3 and 4 with $NaBH_4$ in

Scheme I