product was chromatographed on a 35 × 1 cm column of silica gel with 1% ethyl acetate in hexane, yielding 100 mg (65%) of methyl nonanoate as a colorless liquid.

Acknowledgment. This work was supported by a Whitaker Foundation Grant of Research Corporation.

Registry No. NIS, 516-12-1; nonanal, 124-19-6; methyl nonanoate, 1731-84-6; dodecanal, 112-54-9; methyl dodecanoate, 111-82-0; tetradecanal, 124-25-4; methyl tetradecanoate, 124-10-7; cyclohexanecarbaldehyde, 2043-61-0; methyl cyclohexanecarboxylate, 4630-82-4; 2-phenylethanal, 122-78-1; benzeneacetic acid methyl ester, 101-41-7; 5-hexenal, 764-59-0; methyl hexenoate, 2396-80-7; benzaldehyde, 100-52-7; methyl benzoate, 93-58-3; 3-pyridinecarbaldehyde, 500-22-1; methyl 3-pyridinecarboxylate, 93-60-7; 3-nitrobenzaldehyde, 99-61-6; methyl 3-nitrobenzoate, 618-95-1; *trans*-cinnamaldehyde, 14371-10-9; *trans*-cinnamic acid methyl ester, 1754-62-7; 2-methylbenzaldehyde, 529-20-4; methyl 2-methylbenzoate, 89-71-4.

A Highly Stereoselective and Iterative Approach to Isoprenoid Chains: Synthesis of Homogeraniol, Homofarnesol, and Homogeranylgeraniol

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We recently described a highly stereoselective synthesis of trisubstituted homoallylic alcohols based on the Ni(0)-catalyzed coupling of Grignard reagents with 5-alkyl-2,3-dihydrofurans[1] first reported by Wenkert and coworkers.[2] The reaction is easy to do on a substantial scale and generally gives good yields. In this note we show how the procedure can be applied in an iterative sense to the synthesis of the isoprenoids homogeraniol (**5**),[3] homofarnesol (**8**), and homogeranylgeraniol (**11**). Each turn of the cycle requires the alkylation of 5-lithio-2,3-dihydrofuran (**2**) with a homoallylic iodide followed by the Ni(0)-catalyzed coupling with methylmagnesium bromide as shown in Scheme I. The resultant homoallylic alcohol can then be converted to the corresponding iodide and the cycle repeated.

All of the reagents used in the scheme are comparatively cheap and readily available. The Ni(0) catalyst, of which only 2 mol % is required, is generated in situ by the reaction of MeMgBr with $[Ph_3P]_2NiCl_2$, giving the thermally unstable $[Ph_3P]_2NiMe_2$ which loses ethane to give the active dark red catalyst. The yields at every stage are uniformly good. However, the salient feature of the sequence is the very high stereoselectivity of the three coupling steps. Analysis of the alcohols **5**, **8** and **11** by high-field ^1H and ^{13}C NMR and capillary gas chromatography indicated a purity of ≥97% in each case.

In conclusion this sequence compares favorably with analogous iterative isoprenoid syntheses[4] by virtue of its economy, experimental ease, and high stereoselectivity. The various homoprenols should prove useful as precursors to other higher terpenoids via coupling to appropriate C_4 components.

Experimental Section

2,3-Dihydrofuran (**1**) was obtained from Aldrich and freshly distilled. 1-Iodo-4-methylpent-3-ene (**3**) was prepared from the corresponding bromide by a Finkelstein reaction in the usual way, using NaI in acetone, and then freshly distilled. Benzene, Et_2O, and THF were freshly distilled from Na wire. ^1H (270 MHz) and ^{13}C (67.5 MHz) NMR spectra were recorded with a JEOL GX 270 spectrometer in the solvent specified with Me_4Si as an internal standard. Coupling constants (J) are reported in hertz. All IR spectra were recorded as thin films. Capillary gas chromatography was performed on a Packard 436 machine using a Chrompack 220 μ CP WAX 52 column. The purity of compounds **4**, **6**, **7**, **9**, and **10** was shown to be >94% by ^1H NMR and capillary GC.

5-(4-Methylpent-3-enyl)-2,3-dihydrofuran (4). A solution of *tert*-butyllithium in pentanes (81.2 mL, 130 mmol) was added dropwise to a solution of 2,3-dihydrofuran (8.4 g, 120 mmol) in dry tetrahydrofuran (33 mL) cooled to −50 °C under argon. The resulting yellow suspension was allowed to warm to 0 °C and was stirred for a further 30 min. The mixture was then cooled to −30 °C and a solution of 1-iodo-4-methylpent-3-ene (**3**) (20.0 g, 95 mmol) in dry tetrahydrofuran (40 mL) was added. The mixture was allowed to warm to room temperature and was stirred for 18 h. The white suspension so obtained was poured into a solution of saturated ammonium hydroxide (10 mL) in saturated ammonium chloride (90 mL) and the organic products were extracted with ether. The combined extracts were dried briefly ($MgSO_4$) and evaporated to leave a yellow oil. Bulb-to-bulb distillation gave the desired dihydrofuran **4** (14.0 g, 97%) as a colorless oil: bp 90 °C (bath)/15 mmHg; IR 2980 s, 2920 s, 2860 s, 1670 s, 1450 s, 1380 s, 1180 s, 1165 s, 1010 s, 930 s cm^{-1}; ^1H NMR (acetone-d_6) δ 5.11 (m, 1 H, J = 1.2, 6.0), 4.55 (m, 1 H), 4.20 (t, 2 H, J = 9.4), 2.52 (tdd, 2 H, J = 1.9, 1.9, 9.4), 2.17–1.99 (m, 4 H), 1.63 (s, 3 H), 1.59 (s, 3 H); ^{13}C NMR (acetone-d_6) δ 159.8 (s), 132.7 (s), 125.0 (d), 94.6 (d), 70.5 (t), 31.0 (t), 29.2 (t), 26.4 (t), 26.2 (q), 18.1 (q); MS m/z 152 (M^+, 76), 135 (38), 111 (21), 109 (23), 108 (23), 97 (16), 95 (24), 93 (18), 84 (62), 83 (25), 67 (100); high resolution EIMS m/z 152.1201 ($C_{10}H_{16}O$ = 152.1198).

(3*E*)-4,8-Dimethylnona-3,7-dien-1-ol (Homogeraniol) (5). A solution of methylmagnesium bromide in ether (69 mL, 0.2 mol) was added to a stirred suspension of bis(triphenylphosphine)nickel dichloride (2.25 g, 3.45 mmol) in dry benzene[5] (200 mL) under dry nitrogen. The resulting red solution was stirred at room temperature for 20 min, and a solution of 5-(4-methylpent-3-enyl)-2,3-dihydrofuran (**4**) (10.5 g, 69 mmol) in benzene (100 mL) was then added. The mixture was heated to reflux for 40 min, cooled to room temperature, and poured into saturated ammonium chloride solution (200 mL) with vigorous stirring. The mixture was stirred until decolorized and the organic material was extracted with ether. The combined extracts were dried ($MgSO_4$) and evaporated to leave a yellow oil. Column chromatography on silica gel (ether/petroleum ether, 1:4) and distillation gave homogeraniol (**5**) (10.3 g, 89%) as a colorless oil: bp 135 °C (bath)/15 mmHg; IR 3600–3100 m, 2980 s, 2930 s, 1670 w, 1460 s, 1380 s, 1060 s cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.14–5.05 (m, 2 H), 3.61 (t, 2 H, J = 6.5), 2.28 (dt, 2 H, J = 6.5, 7.1), 2.13–2.01 (m, 4 H + OH), 1.68, 1.64, 1.60 (s, 3 H each); ^{13}C NMR ($CDCl_3$) δ 138.9 (s), 131.1 (s), 124.2 (d), 119.9 (d), 62.4 (t), 39.8 (t), 31.5 (t), 26.6 (t), 25.7 (q), 17.7 (q), 16.2 (q). The IR and ^1H NMR spectra are in agreement with data reported by Leopold.[6]

(3*E*)-1-Iodo-4,8-dimethylnona-3,7-diene (6). Methanesulfonyl chloride (2.15 mL, 28 mmol) was added dropwise to a

(1) Wadman, S.; Whitby, R.; Yeates, C.; Kocieński, P.; Cooper, K. *J. Chem. Soc., Chem. Commun.* **1987**, 241. Kocieński, P.; Dixon, N. J.; Wadman, S. *Tetrahedron Lett.* **1988**, *29*, 2353. Kocieński, P.; Wadman, S.; Cooper, K. *Tetrahedron Lett.* **1988**, *29*, 2357. Kocieński, P.; Love, C.; Whitby, R.; Roberts, D. A. *Tetrahedron Lett.* **1988**, *29*, 2867.

(2) Wenkert, E.; Michelotti, E. L.; Swindell, C. S.; Tingoli, M. *J. Org. Chem.* **1984**, *49*, 4894.

(3) Kobayashi, M.; Valente, L. F.; Negishi, E. *Synthesis* **1980**, 1034.

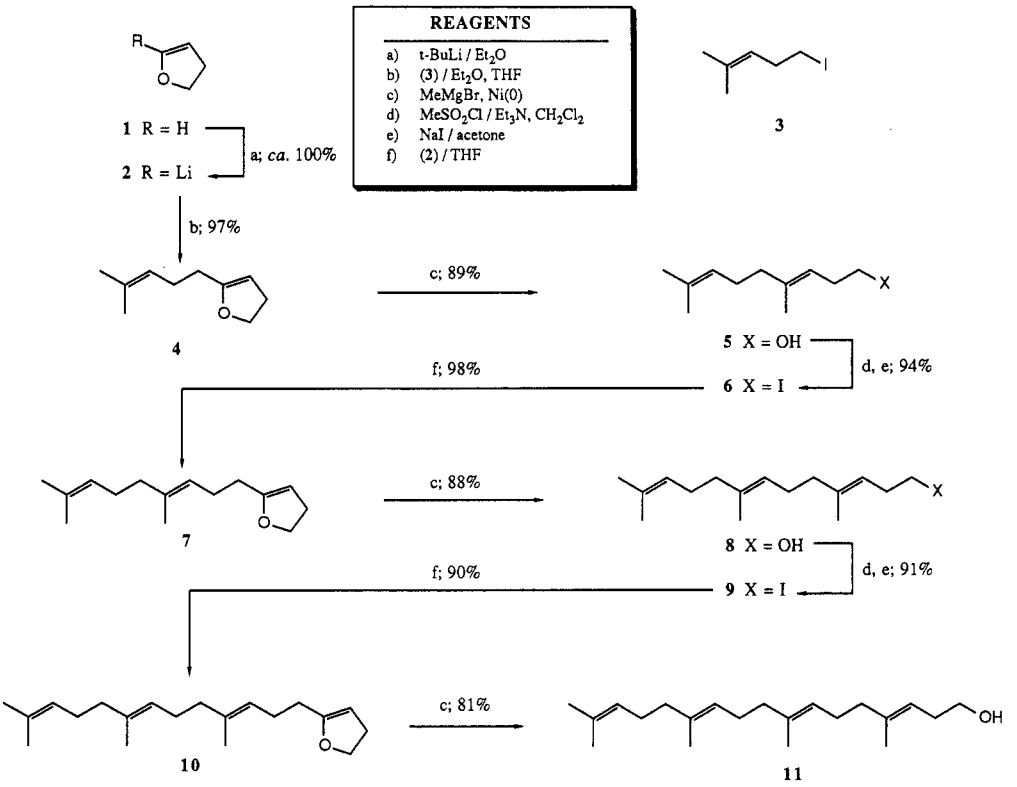
(4) Carboalumination (review): Negishi, E.; Takahashi, T. *Synthesis* **1988**, *1*. Carbocupration (review): Normant, J. F.; Alexakis, A. *Synthesis* **1981**, 841. Alkylcuprate coupling with alkenyl iodides: Corey, E. J.; Katzenellenbogen, J. A.; Posner, G. H. *J. Am. Chem. Soc.* **1967**, *89*, 4245. Alkylcuprate coupling with enol phosphates: Sum, F. W.; Weiler, L. *J. Am. Chem. Soc.* **1979**, *101*, 4401.

(5) Benzene can be replaced by toluene without incident.

(6) Leopold, E. L. *Org. Synth.* **1986**, *64*, 164.

0022-3263/89/1954-1215$01.50/0

Scheme I



solution of homogeraniol (**5**) (4.25 g, 25.3 mmol) and triethylamine (7.7 mL, 56 mmol) in dry dichloromethane (25.0 mL) stirred at 0 °C under nitrogen. After addition was complete, the mixture was stirred for 30 min and a white precipitate was formed. 3-(Dimethylamino)propylamine (1.1 mL, 13 mmol) was then added dropwise and the mixture was stirred at room temperature for 5 min before being poured into water. The mixture was extracted with ether and the combined extracts were concentrated to a yellow oil, which was taken up in acetone (150 mL). Sodium iodide (30.3 g, 202 mmol) was added and the mixture was heated to reflux. A white solid precipitated. After 2 h at reflux the solvent was removed in vacuo and the solid residue was partitioned between light petroleum ether and water. The organic solution was dried ($MgSO_4$) and evaporated to leave a yellow oil. Filtration through silica, eluting with light petroleum ether gave **6** (6.61 g, 94%) as a colorless oil: IR 2960 s, 2920 s, 2850 s, 1660 w, 1440 s, 1250 s, 1160 s, 770 s, 700 s cm^{-1}; ^1H NMR (acetone-d_6) δ 5.15 (m, 2 H), 3.19 (t, 2 H, J = 7.2), 2.57 (dt, 2 H, J = 6.9, 7.2), 2.15–1.97 (m, 4 H), 1.66 (s, 3 H), 1.61, 1.53 (s, 3 H each); ^{13}C NMR (acetone-d_6) δ 138.7 (s), 132.2 (s), 125.3 (d), 124.5 (d), 40.7 (t), 33.4 (t), 27.6 (t), 26.3 (q), 18.2 (q), 16.7 (q), 7.3 (t).

5-[(3*E*)-4,8-Dimethylnona-3,7-dienyl]-2,3-dihydrofuran (7). This was prepared in 98% yield from **6** (18 mmol) as described for **4**. IR 2970 s, 2920 s, 2860 s, 1745 w, 1665 s, 1460 s, 1385 s, 1370 s, 1180 m, 1170 m, 1010 s cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.05 (m, 2 H), 4.54 (br s, 1 H), 4.20 (t, 2 H, J = 9.3), 2.53 (tdd, 2 H, J = 1.9, 1.9, 9.3), 2.18–1.86 (m, 8 H), 1.61 (s, 3 H), 1.54 (s, 6 H); ^{13}C NMR ($CDCl_3$) δ 159.2 (s), 135.2 (s), 131.7 (s), 124.5 (d), 124.1 (d), 94.0 (d), 69.9 (t), 39.8 (t), 39.6 (t), 30.1 (t), 26.7 (t), 26.5 (t), 17.8 (q), 16.2 (q), 16.1 (q).

Like all higher molecular weight 5-alkyl-2,3-dihydrofurans, this compound could not be distilled without suffering deterioration. The endocyclic double bond rearranges to the exocyclic position on heating or treatment with very mild acid.[7] The dihydrofurans are best used immediately after preparation, but if they must be stored they should be stabilized by adding ca. 1% triethylamine and kept at −20 °C.

(3*E*,7*E*)-4,8,12-Trimethyltrideca-3,7,11-trien-1-ol (Homofarnesol) (8). This was prepared in 88% yield from **7** (16 mmol) as described above for homogeraniol (**5**): IR 3700–3100 s, 2960 s, 2920 s, 1750 w, 1670 w, 1440 s, 1385 s, 1375 s, 1050 s, 790 s cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.09 (m, 3 H), 3.60 (t, 2 H, J = 6.5), 2.28 (dt, 2 H, J = 6.5, 7.3), 2.16–1.91 (m, 8 H), 1.68 (s, 3 H), 1.64 (s, 3 H), 1.60 (s, 6 H); ^{13}C NMR ($CDCl_3$) δ 138.9 (s), 135.3 (s), 131.3 (s), 124.4 (d), 124.0 (d), 119.9 (d), 62.4 (t), 39.8 (t), 39.4 (t), 31.5 (t), 26.8 (t), 26.5 (t), 25.7 (q), 17.7 (q), 16.2 (q), 16.1 (q); MS m/z 236 (M^+, 2.5), 136 (16), 123 (14), 107 (9), 93 (12), 81 (37), 69 (100); high resolution EIMS m/z 236.2147 ($C_{16}H_{28}O$ = 236.2140).

(3*E*,7*E*)-1-Iodo-4,8,12-trimethyltrideca-3,7,11-triene (9). This was prepared in 91% yield from homofarnesol (**8**) (5 mmol) as described for **6**: IR 2960 s, 2920 s, 1850 s, 1660 w, 1440 s, 1380 s, 1370 s, 1250 s, 1160 s cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.12 (m, 3 H), 3.12 (t, 2 H, J = 7.4), 2.59 (dt, 2 H, J = 7.0, 7.5), 2.17–1.95 (m, 8 H), 1.69 (s, 3 H), 1.61 (3 superimposed s, 9 H); ^{13}C NMR ($CDCl_3$) δ 138.1 (s), 135.2 (s), 131.3 (s), 124.4 (d), 124.0 (d), 123.0 (d), 39.8 (t), 39.7 (t), 32.5 (t), 26.8 (t), 26.4 (t), 25.7 (q), 17.8 (q), 16.3 (q), 16.1 (q), 6.1 (t).

5-[(3*E*,7*E*)-4,8,12-Trimethyltrideca-3,7,11-trienyl]-2,3-dihydrofuran (10). This was prepared from iodide **9** (3 mmol) in 90% yield as described above for **7**: IR 2970 s, 2920 s, 2860 s, 1750 w, 1665 s, 1450 s, 1385 s, 1370 s, 1180 m, 1170 m, 1010 s cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.05 (m, 3 H), 4.51 (br s, 1 H), 4.23 (t, 2 H, J = 9.3), 2.53 (tdd, J = 1.9, 1.9, 9.3), 2.18–1.86 (m, 12 H), 1.61 (s, 3 H), 1.54 (3 superimposed s, 9 H); ^{13}C NMR ($CDCl_3$) δ 158.7 (s), 135.7 (s), 135.0 (s), 131.3 (s), 124.5 (d), 124.2 (d), 123.6 (d), 93.8 (d), 69.8 (t), 39.8 (t), 39.7 (t), 30.1 (t), 28.2 (t), 26.8 (t), 26.6 (t), 25.7 (q), 25.3 (t), 17.8 (q), 16.1 (q, $2CH_3$).

(3*E*,7*E*,11*E*)-4,8,12,16-Tetramethylheptadeca-3,7,11,15-tetraen-1-ol (Homogeranylgeraniol) (11). This was prepared from **10** (2.4 mmol) in 81% yield as described above for **5**: IR 3600–3100 m, 2960 s, 2920 s, 2850 s, 1665 w, 1440 m, 1380 m, 1040 s cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.17–5.06 (m, 4 H), 3.61 (t, 2 H, J = 6.5), 2.28 (dt, 2 H, J = 6.5, 7.0), 2.16–1.90 (m, 12 H), 1.68 (s, 3 H), 1.65 (s, 3 H), 1.60 (3 superimposed s, 9 H); ^{13}C NMR ($CDCl_3$) δ 138.9 (s), 135.3 (s), 135.0 (s), 131.3 (s), 124.4 (d), 124.2 (d), 124.0 (d), 119.9 (d), 62.4 (t), 39.9 (t), 39.8 (t), 39.7 (t), 31.5 (t), 26.8 (t), 26.7 (t), 26.5 (t), 25.8 (q), 17.7 (q), 16.3 (q), 16.1 (q), 16.0 (q); MS m/z 304 (M^+, 13), 235 (19), 204 (29), 189 (15), 167 (11), 161 (15), 149 (24), 136 (45), 123 (44), 107 (29), 95 (33), 81 (73), 69 (100); high resolution EIMS m/z 304.2765 ($C_{21}H_{36}O$ = 304.2766).

Acknowledgment. We thank the Science and Engineering Research Council of Great Britain for a CASE

(7) Taskinen, E. *Tetrahedron* **1978**, *34*, 433 and references therein.

studentship and Pfizer Central Research for financial support.

Registry No. 1, 1191-99-7; 2, 75213-94-4; 3, 43161-11-1; 3 (bromide), 2270-59-9; 4, 118495-28-6; 5, 459-88-1; 5 (X = OMs), 118495-32-2; 6, 22339-13-5; 7, 118495-29-7; 8, 459-89-2; 8 (X = OMs), 118495-33-3; 9, 113219-28-6; 10, 118495-30-0; 11, 118495-31-1; 1,1-bis(methylamino)propylamine, 118495-34-4.

Diels–Alder Reactions of Cycloalkenones. 15. Synthesis of *cis*- and *trans*-Δ^6-4a-Methyl-1-octalones[1]

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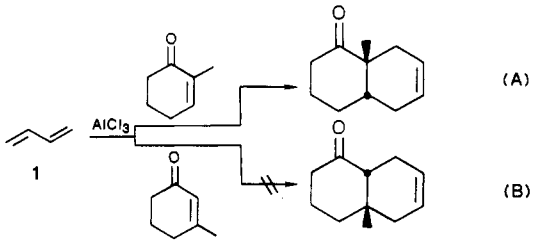
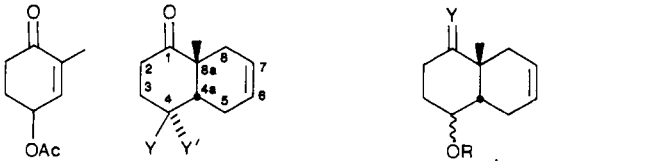
Angularly methylated octalones are useful intermediates for terpene or steroid synthesis. The high-yielding, Lewis acid catalyzed cycloaddition of 2-methyl-2-cyclohexenones and 1,3-butadienes, as illustrated for the simplest case in equation A,[3] yields nowadays easy access to such octalones in the cis isomer form. The lack of reactivity of 3-methyl-2-cyclohexenone toward 1,3-butadiene (**1**) in thermal or acid-induced Diels–Alder reactions (equation B)[4] precludes the ready preparation of *cis*-octalones (or *trans*-octalones, i.e., after equilibration) in which the angular methyl group is in a 1,3-positional relationship with the keto function. The following study was undertaken in order to overcome the obstacle and introduce full flexibility into the Diels–Alder reaction scheme.

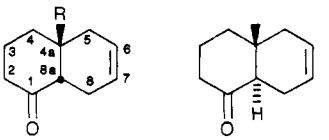


Cycloaddition of 1,3-butadiene (**1**) with 4-acetoxy-2-methyl-2-cyclohexenone (**2**) (prepared in 57% yield from 2-methyl-2-cyclohexenone on C(4)-bromination with *N*-bromosuccinimide and subsequent bromide displacement with potassium acetate) in degassed (i.e., oxygen-free) toluene solution under the influence of aluminum chloride at 40 °C led to a 1.3:1 mixture of ketoacetates **3a** and **3b** in 60% yield. It is noteworthy that the diastereofacial selectivity of the reaction was the same as that between 2,4-dimethyl-2-cyclohexenone and 1,3-butadiene (**1**),[5] in accord with the hypothesis of the cycloaddition being governed by stereoelectronic and conformational factors.[5]



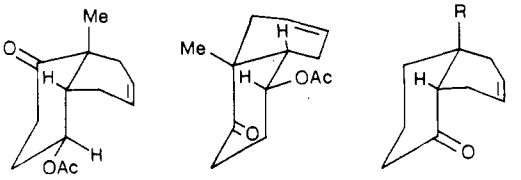
2 **3a**: Y = OAc, Y′ = H; **b**: Y = H, Y′ = OAc **4a**: Y = NNHSO$_2$C$_6$H$_4$Me-*p*, R = Ac; **b**: Y = H$_2$, R = Ac; **c**: Y = H$_2$, R = H

Conversion of the **3a–3b** ketoacetate mixture into its *p*-tosylhydrazone derivatives (**4a**), reduction of the latter with catecholborane,[6] base-induced hydrolysis of the resultant acetates **4b**, and Jones oxidation of alcohols **4c** furnished octalone **5a** in 36% overall yield. Base-catalyzed isomerization of the latter gave quantitatively a 4:1 trans–cis isomer mixture (ketones **6**[7] and **5a**).



5a, R = Me; **b**, R = H **6**

The gross structure and relative configuration of ketones **3** were determined by ^{13}C NMR spectroscopy and carbon shift comparison with models **3** (Y = Me, Y′ = H)[5] and **3** (Y = H, Y′ = Me).[5] The latter shift correlation revealed ketoacetates **3a** and **3b** to possess predominantly the solution conformations **7** and **8**, respectively. Gross structure and conformational analysis of ketone **5a** depended on the interpretation of its ^{13}C NMR data and carbon shift analysis of octalone **5b**.[6b] Conformational analysis of the latter ketone, in turn, required correlation of its carbon shifts with those of the conformationally biased ketones 2α,6-dimethyl- and 2β,6-dimethyl-**5b**.[5] These analyses showed compounds **5** to prefer conformation **9** in solution. Finally, the ^{1}H chemical shifts of the angular methyl groups of ketones **5a** (1.11 ppm) and **6** (0.79 ppm) are consistent with the assigned configurations.[8]



7 **8** **9**

The above, short preparation makes methyloctalones **5a** and **6** readily available for natural product synthesis. In this connection it is of interest that the ethylene ketal of methyloctalone **6**, acquired by a multistep preparation,[9,10] has been utilized recently for the synthesis of the sesquiterpenes (±)-β-costol,[11] (±)-β-costal,[11] (±)-β-arctiol,[11]

(1) For the previous paper, see: Angell, E. C.; Fringuelli, F.; Guo, M.; Minuti, L.; Taticchi, A.; Wenkert, E. *J. Org. Chem.* **1988**, *53*, 4325.
(2) (a) University of California. (b) Università di Perugia.
(3) Fringuelli, F.; Pizzo, F.; Taticchi, A.; Halls, T. D. J.; Wenkert, E. *J. Org. Chem.* **1982**, *47*, 5056.
(4) Fringuelli, F.; Pizzo, F.; Taticchi, A.; Wenkert, E. *J. Org. Chem.* **1983**, *48*, 2802.
(5) Angell, E. C.; Fringuelli, F.; Pizzo, F.; Porter, B.; Taticchi, A.; Wenkert, E. *J. Org. Chem.* **1986**, *51*, 2642.
(6) (a) Kabalka, G. W.; Hutchins, R.; Natale, N. R.; Yang, D. T. C.; Broach, V. *Org. Synth.* **1979**, *59*, 42. (b) Angell, E. C.; Fringuelli, F.; Pizzo, F.; Taticchi, A.; Wenkert, E. *J. Org. Chem.* **1988**, *53*, 1424.
(7) Whereas ketone **6** has not been described previously in pure form, it has been reported as a minor constituent in a four-component mixture of products of the acid-catalyzed cyclization of 2-(3-butenyl)-3-methyl-2-cyclohexenone (Cooper, J. L.; Harding, K. E. *Tetrahedron Lett.* **1977**, 3321).
(8) van der Gen, A.; Wiedhaup, K.; Swoboda, J. J.; Dunathan, H. C.; Johnson, W. S. *J. Am. Chem. Soc.* **1973**, *95*, 2656 and references therein.
(9) Torii, S.; Inokuchi, T.; Yamafuji, T. *Bull. Chem. Soc. Jpn.* **1979**, *52*, 2640.
(10) Garratt, P. J.; Porter, J. R. *J. Org. Chem.* **1986**, *51*, 5450.
(11) Torii, S.; Inokuchi, T. *Bull. Chem. Soc. Jpn.* **1980**, *53*, 2642.

0022-3263/89/1954-1217$01.50/0