mospheric pressure and temperature until 1 mol of gas had been absorbed. The solution was filtered and concentrated in vacuo, and the residue was distilled: bp 105 °C (2 mmHg); 0.89 g, 98%; IR (film) 1727 (C=O), 1206, 1140 cm^{-1} (CO); NMR ($CDCl_3$) δ 1.26 (s, 6 H, 2 CH_3), 1.4–1.8 (m, 4 H, CH_2CH_2), 2.2 (m, 2 H, CH_2CO).

B. By Baeyer–Villiger Oxidation of 2,2-Dimethylcyclopentanone. 2,2-Dimethylcyclopentanone was synthesized from 2-methylcyclohexanone.[3] Trifluoroperacetic acid was prepared by mixation of 85% hydrogen peroxide (1.0 mL) and trifluoroacetic anhydride (6.14 g, 0.030 mol) at 5–10 °C. After 90 min at 0 °C 2,2-dimethylcyclopentanone (3.36 g, 0.030 mol) was added portionwise with shaking and ice-cooling during 40 min. The mixture was kept at 0 °C overnight and then diluted with dichloromethane (50 mL) and poured into an excess of cold aqueous potassium carbonate. The layers were separated, and the aqueous layer was extracted twice with dichloromethane. The combined organic extracts were dried (Na_2SO_4), filtered, and concentrated at room temperature in vacuo. The residual lactone distilled at 105 °C (2 mmHg) (3.8 g, 99%); it was identical (IR and GC comparison) with the product prepared as in **A** above.

C. From 2-Methyl-3-buten-2-ol. The lactone was also prepared from ethyl cyanoacetate by free-radical addition to 3-buten-2-ol followed by alkaline hydrolysis and lactonization.[4] The product (60% overall yield) distilled at 105–107 °C (3–4 mmHg) and was identical with that prepared by procedures **A** and **B**.

Dihydrocatalpalactone (5). A. From 3-Bromophthalide. To diisopropylamine (0.395 g, 0.0039 mol) in dry tetrahydrofuran (10 mL) was added *n*-butyllithium (0.0039 mol) gradually, with stirring at −78 °C. The mixture was allowed to reach room temperature and stirred thereat for 30 min. It was then recooled to −78 °C and δ,δ-dimethyl-δ-valerolactone (0.5 g, 0.0039 mol) in dry tetrahydrofuran (10 mL) was added all at once. The solution was allowed to reach room temperature, then recooled to −78 °C, and treated with a solution of 3-bromophthalide (0.83 g, 0.0039 mol) in dry tetrahydrofuran (30 mL) with stirring. The red solution was allowed to arrive at room temperature, refluxed gently for 17.5 h, and then poured onto crushed ice and dilute HCl. The product was isolated with ether. It was chromatographed on silica gel (30 g) with elution with benzene. Evaporation of the eluates yielded dihydrocatalpalactone (0.081 g, 8%), which separated from methanol in plates, mp 154–155 °C (lit.[1,2] mp 153–154 °C). The IR spectrum was identical with the published spectra.[1,2]

B. From 3-[(*p*-Toluenesulfonyl)oxy]phthalide. To 2,2,6,6-tetramethylpiperidine (1.55 g, 0.011 mol) in dry tetrahydrofuran (5 mL) was added *n*-butyllithium (0.011 mol) at −78 °C, with stirring. The mixture was allowed to reach room temperature during 30 min, then recooled to −78 °C, and δ,δ-dimethyl-δ-valerolactone (1.20 g, 0.0094 mol) in dry tetrahydrofuran (10 mL) added. The cooling bath was removed and the mixture stirred for 30 min and then recooled to −78 °C. A solution of 3-[(*p*-toluenesulfonyl)oxy]phthalide (2.85 g, 0.0094 mol) in dry tetrahydrofuran (20 mL) was added all at once. After 1 h of stirring at −78 °C the mixture was poured onto crushed ice and dilute HCl and the product isolated with ether. The extracts were washed with brine, dried (Na_2SO_4), and concentrated in vacuo. A thick, orange syrup (2.73 g) remained; it was chromatographed on silica gel (60 g) with dichloromethane elution. Evaporation of the eluates afforded a very pale yellow syrup (1.90 g, 78%) which solidified readily. A small portion separated from methanol in plates, mp 154–155 °C, identical with the product from A above: IR (film) 1757 (C=O), 1704 (C=O), 1443, 1267, 1193 (CO), 1106 (CO) cm^{-1}; NMR ($CDCl_3$) δ 1.43 (s, 6 H, 2 CH_3), 1.2–1.9 (m, 4 H, CH_2CH_2), 3.0 (m, 1 H, CHCO), 6.36 (d, 1 H, CHO), 7.4–8.0 (m, 4 H, Ar H); mass spectrum, *m/e* 260 (M^+). Anal. Calcd for $C_{15}H_{16}O_4$: C, 69.21; H, 6.20. Found: C, 69.21; H, 6.20.

Catalpalactone (1). Lithium 2,2,6,6-tetramethylpiperidide (0.0019 mol) was prepared as described above at −78 °C and allowed to reach room temperature with stirring during 2 h. The solution was then recooled to −78 °C and a solution of dihydrocatalpalactone (0.325 g, 0.001 25 mol) and hexamethylphosphoric triamide (0.34 g, 0.0019 mol) in dry tetrahydrofuran (10 mL) was added rapidly, with stirring. After it had reached ambient temperature the mixture was treated with diphenyl diselenide (0.59 g, 0.0019 mol) in dry tetrahydrofuran (10 mL) rapidly. Reaction was allowed to proceed at room temperature for 30 min and then the whole was poured onto a mixture of crushed ice and dilute HCl. The product, isolated by 3-fold ether extraction, was a gum (0.94 g) which was chromatographed on silica gel (20 g) with elution in order by hexanes–chloroform (9:1, leading to unreacted diselenide, 0.45 g), 1:1 (leading to unreacted dihydrocatalpalactone, 0.25 g), and finally with chloroform to yield 2′-(phenylseleno)-dihydrocatalpalactone (7; 0.078 g, 15%; 63% based on unrecovered catalpalactone) used without further purification. This product (0.085 g, 0.2 mmol) was mixed with glacial acetic acid (2 drops), tetrahydrofuran (2 mL), and 30% hydrogen peroxide (0.3 mL), stirred at 0 °C for 30 min, and then poured into ice-cold aqueous sodium bicarbonate; the product was isolated with ether. In vacuo concentration of the dried extracts afforded a semisolid product, which was chromatographed on silica gel (3.5 g) and eluted with hexanes-chloroform (1:1). Evaporation yielded catalpalactone (1) which separated from methanol in plates, mp 106–107 °C (0.06 g, 100%), undepressed by admixture with an authentic sample (mp 105–106 °C). The IR, NMR, and UV spectra were identified with those of natural catalpalactone:[1,2] IR (film) 1760 (C=O), 1742 (C=O), 1447, 1267, 1107 cm^{-1}; NMR ($CDCl_3$) δ 1.3 (s, 3 H, CH_3), 1.45 (s, 3 H, CH_3), 2.5 (m, 2 H, CH_2), 6.4 (s, 1 H, CHO), 6.6–6.8 (t, J = 5 Hz, 1 H, =CH), 7.4–7.9 (m, 4 H, Ar H); UV λ_{max} (EtOH) 275 (log ϵ 3.20), 282 nm (3.20); mass spectrum, *m/e* 258 (M^+). Anal. Calcd for $C_{15}H_{14}O_4$: C, 69.75; H, 5.46. Found: C, 69.61; H, 5.51.

Acknowledgment. We thank Dr. G. Agnès (Istituto Guido Donegani, Novara, Italy) for a gift of 5,5-dimethyl-2-pentenolactone, and Dr. N. Nagakura (Kobe Women's College of Pharmacy, Japan) for an authentic sample of catalpalactone. Mr. J. N. Herron we thank for the NMR spectra. The NMR spectrometer was purchased with funding provided by the National Science Foundation.

Registry No. 1, 1585-68-8; 2, 2610-95-9; 3, 4541-32-6; 4, 6940-49-4; 5, 1585-50-8; 6, 82027-09-6; 7, 82027-10-9; 5,5-dimethyl-2-pentenolactone, 19895-34-2; 2-methyl-3-buten-2-ol, 115-18-4.

A Partial Homo-Favorskii Rearrangement in the Diterpene Series

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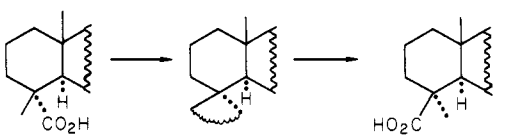
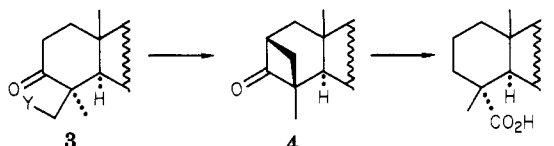
The inversion of a quaternary carbon site without scission of bonds attached thereto is an inherently difficult task. It was shown some time ago in the resin acid field that such a problem [e.g., the conversion of compounds of the dehydroabietic acid type (1) into those of the callistric acid form (2)] can be solved by functional group exchange via a ring formation–cleavage reaction sequence:[1]



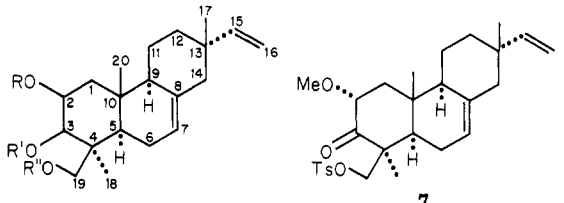
1 2

It was felt that a conceptually similar transformation could be effected on a quaternary carbon system flanked by two functional groups (e.g., 3), disposed toward each other in such a manner as to invite ready ring (4) formation[2,3] and subsequent ring opening (e.g., 4 → 5):[2,4]



3 4 5

The latter scheme was tested with a derivative (7) of virescenol A (6a).[5] Alkylation of its benzylidene derivative (6b)[6] with methyl iodide and sodium hydride gave a methyl ether (6c), whose hydrolysis with aqueous acid yielded diol 6d. Treatment of the latter with *p*-toluenesulfonyl chloride in pyridine produced tosylate 6e, whose Collins oxidation afforded keto tosylate 7, i.e., a product of type 3.

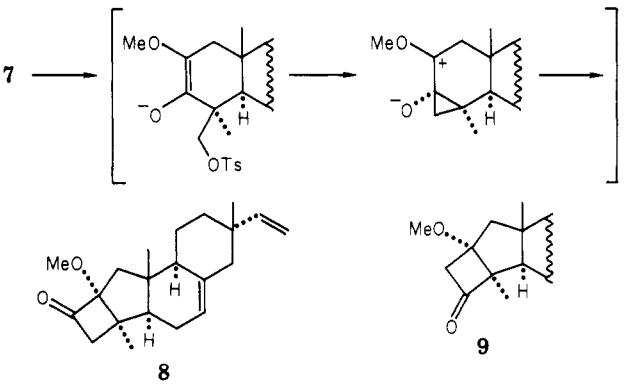


7

6a, R = R′ = R″ = H
b, R = H, R′ + R″ = CHC_6H_5
c, R = Me, R′ + R″ = CHC_6H_5
d, R = Me, R′ = R″ = H
e, R = Me, R′ = H, R″ = Ts

Exposure of the sulfonate 7 to potassium *tert*-butoxide in benzene soluton led to a cyclobutanone, but not of structure type 4. Its ^1H NMR spectrum revealed the presence of an α-ketomethylene unit, characteristic of intermediates of homo-Favorskii reactions accompanied by skeletal rearrangement.[2] By analogy, the cyclobutanone was the product of tosylate solvolysis of the enolate of 7 (vide infra) and hence could be formulated as structure 8. The alteration of the desired pathway, i.e., an ordinary, intramolecular displacement, may have been helped by the methoxy group participation in the stabilization of the intermediate cyclopropylcarbinyl cation and, most importantly, by the steric resistance to the boat cyclohexane formation of type 4 due to the unfavorable "stem-to-stern" interaction of the angular methyl group and the newly formed ring carbon.[7]

Treatment of ketone 8 with acid converted it into an isomer (9). This transformation is precedented.[8,9]



8 9

Experimental Section

Infrared spectra of chloroform solutions were determined on a Beckman Acculab 5 spectrophotometer in ^1H NMR spectra on JEOL INM-C-60 HL and Varian EM-360 spectrometers. Melting points were recorded on a Kofler micro hotstage and are uncorrected. Column chromatography was performed with 0.063–0.200 mm mesh Merck silica gel adsorbant. All organic extracts were dried over sodium sulfate.

3,19-Di-*O*-benzylidene-2α-methoxyisopimaradiene (6c). A solution of 1.40 g of acetal **6b** and 0.32 mL of methyl iodide in 10 mL of tetrahydrofuran was added to a stirring suspension of 225 mg of sodium hydride in 5 mL of tetrahydrofuran under nitrogen at 50 °C and the stirring continued for 45 min. The excess hydride was decomposed with water and the mixture extracted with chloroform. The extract was washed, dried, and evaporated under vacuum. The residue (1.35 g) was chromatographed. Elution with 50:1 benzene–ethyl acetate yielded 120 mg of starting material and 1.10 g of product, whose crystallization from ether gave crystalline ether **6c**: mp 114–116 °C; NMR δ 0.86, 1.03, 1.40 (s, 3 each, Me), 3.51 (s, 3, OMe), 3.58 (d, 1, J = 10 Hz, H-3), 4.01 (4-line AB, 2, J = 11 Hz, OCH_2), 4.03 (m, 1, H-2), 5.38 (m, 1, H-7), 5.83 (s, 1, OCH), 7.2–7.7 (m, 5, aromatic H's).

Anal. Calcd for $C_{28}H_{40}O_3$: C, 79.20; H, 9.50. Found: C, 79.08; H, 9.61.

2-*O*-Methylvirescenol A (6d). A methanolic 0.1 N sulfuric acid solution (10 mL) was added to a solution of 800 mg of acetal **6c** in 10 mL of chloroform and the mixture refluxed for 5 h. Water (50 mL) was added and the mixture extracted with chloroform. The extract was washed with sodium bicarbonate solution and water, dried, and evaporated. Chromatography of the residue (750 mg) and elution with 4:1 benzene–ether led to 180 mg of starting compound and 500 mg of semisolid diol: NMR δ 0.86, 0.86, 1.26 (s, 3 each, Me), 3.36 (d, 1, J = 10 Hz, H-3), 3.42 (m, 1, H-2), 3.46 (s, 3, OMe), 3.78 (4-line AB, 2, J = 11 Hz, OCH_2), 5.40 (m, 1, H-7).

Anal. Calcd for $C_{21}H_{34}O_3$: C, 75.40; H, 10.25. Found: C, 75.50; H, 10.16.

2-*O*-Methyl-19-*O*-(*p*-toluenesulfonyl)virescenol A (6e). A solution of 300 mg of diol **6d** and 180 mg of *p*-toluenesulfonyl chloride in 5 mL of pyridine was left at room temperature for 18 h. It was poured into ice water and extracted with chloroform. The extract was washed with water, dried, and evaporated. Chromtography of the residue (400 mg) and elution with 10:1 benzene–ethyl acetate yielded 85 mg of starting material (in late fractions) and 200 mg of semisolid sulfonate **6e**: NMR δ 0.86, 0.90, 1.06 (s, 3 each, Me), 2.46 (s, 3, aromatic Me), 3.0–3.2 (m, 2, H-2, H-3), 3.33 (s, 3, OMe), 4.19 (4-line AB, 2, J = 11 Hz, OCH_2), 5.40 (m, 1, H-7), 7.40, 7.80 (d, 2 each, J = 9 Hz, aromatic H's).

Anal. Calcd for $C_{28}H_{40}O_5S$: C, 68.83; H, 8.25. Found: C, 68.91; H, 8.15.

2α-Methoxy-3-oxo-19-(*p*-toluenesulfonoxy)isopimaradiene (7). A solution of 100 mg of sulfonate **6e** in 5 mL of methylene chloride was added to a stirring solution of 200 mg of chromic anhydride and 0.25 mL of pyridine in 10 mL of dichloromethane and stirring continued for 1 h. The mixture was poured into ice water and extracted with chloroform. The extract was washed with water, dried, and evaporated. Chromatography of the residue (900 mg) and elution with 50:1 benzene–ethyl acetate gave 80 mg of product, whose crystallization from ether yielded ketone 7: mp

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90–92 °C; IR 1716 (s, C=O) cm^{-1}; NMR δ 0.86, 1.13, 1.20 (s, 3 each, Me), 2.46 (s, 3, aromatic Me), 3.40 (s, 3, OMe), 4.16 (dd, 1, J = 14, 6 Hz, H-2), 4.36 (4-line AB, 2, J = 11 Hz, OCH_2), 7.40, 7.80 (d, 2 each, J = 9 Hz, aromatic Hs).

Anal. Calcd for $C_{28}H_{38}O_5S$: C, 69.11; H, 7.87. Found: C, 69.16; H, 7.76.

Ketone 8. A solution of 80 mg of ketone **7** in 10 mL of benzene was added over a 15-min period to a stirring suspension of 48 mg of potassium *tert*-butoxide in 5 mL of benzene under nitrogen at 60 °C and the mixture stirred at this temperature for an additional 30 min. It was poured into ice water and extracted with chloroform. The extract was washed with water, dried, and evaporated. Chromatography of the residue (60 mg) over neutral alumina and elution with 50:1 benzene–ethyl acetate yielded 48 mg of semisolid ketone **8**: IR 1768 (s, C=O) cm^{-1}; NMR δ 0.90, 1.10, 1.26 (s, 3 each, Me), 2.65 (4-line AB, 2, J = 18 Hz, $COCH_2$), 3.43 (s, 3, OMe), 5.48 (m, 1, H-7).

Anal. Calcd for $C_{21}H_{30}O_2$: C, 80.21; H, 9.62. Found: C, 80.32; H, 9.56.

Treatment of 70 mg of the ketone **8** with 2 mL of 2 N sodium deuteroxide in deuterium oxide and 1 mL of dioxane at 70 °C under nitrogen for 27 h, followed by the usual workup, yielded 60 mg of dideuterio **8**, whose ^1H NMR spectrum had lost its two-proton multiplet at 2.65 ppm.[10]

Ketone 9. A mixture of 40 mg of ketone **8** and 200 mg of silica gel in 5 mL of benzene was stirred at room temperature for 6 h and then filtered. Evaporation of the filtrate gave 40 mg of residue, whose crystallization from ether yielded crystalline ketone **9**: mp 86–88 °C; IR 1768 (s, C=O) cm^{-1}; NMR δ 0.83, 0.87, 1.26 (s, 3 each, Me), 2.87 (4-line AB, 2, J = 18 Hz, $COCH_2$), 3.50 (s, 3, OMe), 5.48 (m, 1, H-7).

Anal. Calcd for $C_{21}H_{30}O_2$: C, 80.21; H, 9.62. Found: C, 80.15; H, 9.71.

Deuterium exchange on ketone **9** by the procedure used for the cyclobutanone **8** (vide supra) yielded a dideuterio derivative whose ^1H NMR spectrum showed the loss of the two-proton multiplet at 2.87 ppm.[10]

Acknowledgment. The work in Perugia was supported by the CNR (Rome).

Registry No. 6b, 63089-05-4; **6c**, 82064-68-4; **6d**, 82064-69-5; **6e**, 82064-70-8; **7**, 82064-71-9; **8**, 82064-72-0; **9**, 82064-73-1; methyl iodide, 74-88-4.

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Ring-Opening Reactions of Electrophilic Cyclopropanes

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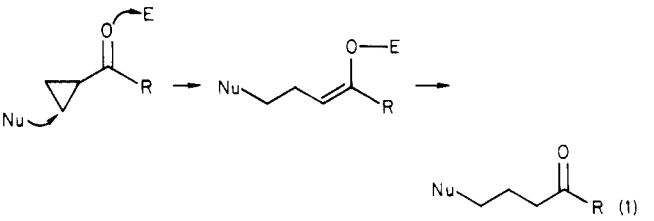
Recent developments in organoheteroatom (e.g., aluminum, silicon, sulfur, and selenium) chemistry have involved reagents in which a hard acid is bound to a soft base. Application of the hard and soft acids and bases principle (HSAB)[1] predicts that the weak hard–soft interaction in the reagent should facilitate reaction pathways involving complementary hard–hard/soft–soft interactions between reagent and substrate. The use of reagents combining nucleophiles with potent oxygenophiles (hard acids) has led to several extremely mild procedures involving the formal addition of a nucleophile to an electron-deficient carbon center. Examples of these reagents include Me_3SiI, Me_2AlSPh, and $MeS–SiMe_3$ in which the silicon and aluminum atoms are hard acids and the iodine and sulfur atoms are soft bases. Transformations such as epoxide ring openings (Me_3SiI,[2] Me_3SiCN[3]), nucleophilic acyl substitutions (Me_2AlSPh,[4] $Me_2AlSePh$[5]), and dealkylation of esters (Me_3SiI,[2] $AlBr_3/PhSH$,[7] $Me_3SiCl/NaI/CH_3CN$[8]), acetals (Me_3SiI[2]), and methyl and benzyl ethers (Me_3SiSR[9]) illustrate successful applications of these reagents. The above reactions suggested the possibility of adding various nucleophiles to cyclopropanes containing an electron-withdrawing substituent under very mild reaction conditions (eq 1). The well-known parallel between



(1)

cyclopropane and olefin chemistry[10] would also suggest a soft β-carbon in a cyclopropyl carbonyl compound in analogy with α,β-unsaturated carbonyl compounds. We have examined nine reagent combinations and three functional group substituents to explore the scope of this homologous Michael[11] addition procedure.

The addition of nucleophiles to cyclopropanes conjugated with electron-withdrawing substituents is well precedented[11] and represents a reactivity umpolung procedure[12] that has been actively investigated in recent years. The ring cleavage of electron-deficient cyclopropanes can be effected under nucleophilic conditions[11] or assisted by the presence of powerful electrophiles. The nucleophilic ring-opening reactions are generally limited to diactivated and highly strained monoactivated cyclopropanes unless very powerful nucleophiles are employed.[13] Lewis and Brønsted acids have been utilized in electrophilicly assisted ring-opening reactions of cyclopropyl ketones but often require vigorous reaction conditions that may result in poor regioselective cleavage.[14] Recent reports describing cleavage of cyclopropyl ketones with trimethylsilyl iodide[15] and acetyl methanesulfonate[16] under mild conditions

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