compounds such as amines[2,4] and pyrazolines.[6]

Experimental Section

Melting points are uncorrected. IR spectra were recorded with a Hitachi 26-50 spectrometer, ^1H NMR spectra with a Varian EM 360A spectrometer, and ^{13}C NMR with a JEOL JNM FX100 spectrometer (solvent, deuteriochloroform; tetramethylsilane as internal standard), and UV spectra with a Shimazu UV 365 spectrometer. GLC analysis was performed by using a Hitachi 164 gas chromatograph with a FID detector. Cyclic voltammograms of substrates were obtained in 0.1 M n-Bu_4NClO_4/CH_3CN solution (vs. SCE; scan rate, 200 mV/s; Hokuto Denko Ltd., Potentiostat/galvanostat HA-301). The light source was a Xenon lamp ($\lambda > 460$ nm) through a glass filter (Y-48, HOYA Co.). Irradiations were carried out through a 2-mm slit while passing oxygen.

Diphenylisobenzofuran (DPBF, Tokyo Kasei) was used as received. Its purity was confirmed by the lack of a carbonyl absorption in the IR spectrum, and GLC analysis showed that no trace of *o*-dibenzoylbenzene (DBB) was present. 2-Methyl-2-pentene (2-MP, Wako Pure Chemicals) was distilled from sodium hydride to remove alcoholic impurities, and its purity was confirmed by GLC analysis.

Pyran **1** was prepared as follows: 200 mL of ethanol solution of 4-(formylmethylidene)-2,6-diphenyl-4*H*-pyran[14] (1.8 mmol) and *N,N*-dimethylhydrazine (18 mmol) was refluxed for 30 min under an nitrogen atmosphere. The deposited precipitates after cooling were collected and recrystallized from ethanol: 84% yield; mp 279–280 °C; UV λ_{max} 406 nm (log ϵ 4.53); ^1H NMR δ 2.93 (s, 6 H), 5.83 (d, 1 H), 6.41 (d, 1 H), 6.79 (d, 1 H), 7.42–8.0 (m, 11 H); ^{13}C NMR δ 43.27 (q), 101.51 (d), 108.03 (d), 112.19 (d), 150.3 (s), 151.56 (s). Anal. Calcd for $C_{21}H_{20}N_{20}$: C, 79.71; H, 6.37; N, 8.85. Found: C, 79.73; H, 6.34; N, 8.75.

Quenching of Photosensitized Oxygenation of Diethyl Sulfide. A solution of diethyl sulfide (6.3×10^{-2} M) and a sensitizer (6.3×10^{-5} M) in the absence or presence of the quencher (6.3×10^{-3} M) was saturated with oxygen and the system closed and irradiated. At regular intervals, the yield of diethyl sulfoxide was measured by GLC (4 mm × 1 m glass tube, 10% PEG-20M on Uniport HP; column temperature, 150 °C). The results obtained are shown in Figure 1.

Photosensitized Oxygenation of DPBF. A stock solution of RB (2 mL, 4×10^{-4} M) in methanol and 2 mL of freshly prepared DPBF with pyran **1** (variable concentrations of DPBF to constant concentrations of 1) in methanol–benzene (3:2) were mixed in Pyrex reaction tubes. After irradiation for a constant time (1 min) under constant oxygen bubbling, the internal standard, 9,10-diphenylanthracene (DPA), was added, and the reaction mixture was immediately analyzed by GLC (4 mm × 1 m glass column, 10%-SF 96 on Chromosorb; column temperature, 220 °C). The yields of DBB produced were determined by the comparison of the peak areas with that of the internal standard (DPA).

Photosensitized Oxygenation of 2-MP. A stock solution of RB (2.5 mL, 4×10^{-4} M) in methanol, 2.5 mL of freshly prepared 2-MP, and 2.5 mL of pyran **1** (variable concentrations of 2-MP to constant concentrations of 1) in methanol–benzene (3:2) were mixed in Pyrex reaction tubes. After irradiation for 20 min under a constant oxygen flow, the reaction mixture was reduced with an excess of dimethyl sulfide, allowed to stand for 6 h, and the internal standard (cyclohexanone) was added and the reaction mixture analyzed by GLC (4 mm × 2 m glass column, 10% Carbowax 20M on Celite 545; column temperature, 80 °C). The yields of alcohols[6] were determined by the comparison of the peak areas with that of the internal standard.

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Registry No. 1, 104549-50-0; DPBF, 5471-63-6; 2-MP, 625-27-4; $^1O_2$, 7782-44-7.

(14) Reynolds, G. A.; Vanallan, J. A. *J. Org. Chem.* **1969**, *34*, 2734.

On the Reaction of α-Methylene-β-peroxy Lactones with Olefins

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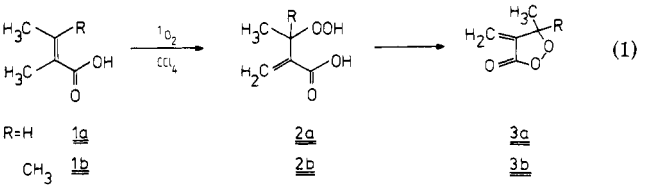
In a previous paper[1] we reported the synthesis of the novel α-methylene-β-peroxy lactone **3a** via photooxygenation of tiglic acid (**1a**) and subsequent treatment of the initially formed β-hydroperoxy acid **2a** with catalytic amounts of mineral acid. Here we report the synthesis of derivative **3b**, another member of this new class of substances, and the reaction of such β-peroxy lactones with olefins.

Trimethylacrylic acid (**1b**)[2] was allowed to react with singlet oxygen, generated by tetraphenylporphine (TPP) photosensitization in chlorinated solvents. The initially formed hydroperoxy acid **2b** was acidic enough to catalyze its own cyclization to the α-methylene-β-peroxy lactone **3b** (eq 1). However, **2b** could be detected by means of

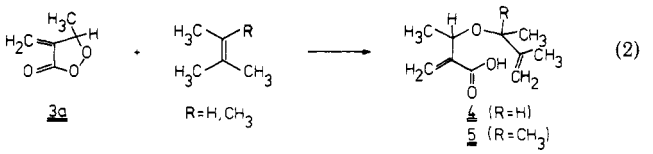


(1)

^1H NMR and IR spectra, taken directly after completion of the photooxygenation. Characteristic for the structure of **2b** is its carbonyl absorption at 1696 cm^{-1}, which on standing becomes replaced by the 1780-cm^{-1} C=O absorption of the β-peroxy lactone **3b**.

3b was isolated in high yield (>90%) by Kugelrohr distillation. The reaction of **1b** with singlet oxygen, therefore, shows the same surprising regioselectivity observed already for the (*Z*)- and (*E*)-2-methyl-2-butene carboxylic acids.[1] For **1b** the degree of regio control was even higher as for its methyl ester, where an 88% preference for hydrogen abstraction from the methyl group proximate to the carbonyl functionality was observed.[3]

We were interested in using the α,β-unsaturated carbonyl moiety of the α-methylene-β-peroxy lactones **3a,b** in Diels–Alder reactions with electron-rich olefins as a potential route to vinylic peroxides. Although the novel β-peroxy lactone **3a** reacted readily with olefins such as 2,3-dimethyl-2-butene or 2-methyl-2-butene at 0 °C, instead of the desired Diels–Alder adducts, the ene-type products **4** and **5** were generated quantitatively (eq 2).



(2)

Formally, this transformation can be considered as a $[(\sigma^2 + \pi^2) + \sigma^2]$-ene reaction, in which the peroxy lactone **3a** serves as the σ^2-enophile. The reaction of phthaloyl peroxide with olefins serves as precedent,[4] leading to the phthalate half-esters as ene-type products. Astounding is the fact that for the unsymmetrical 2-methyl-2-butene, the ene reaction with **3a** proceeded perfectly regioselectively.

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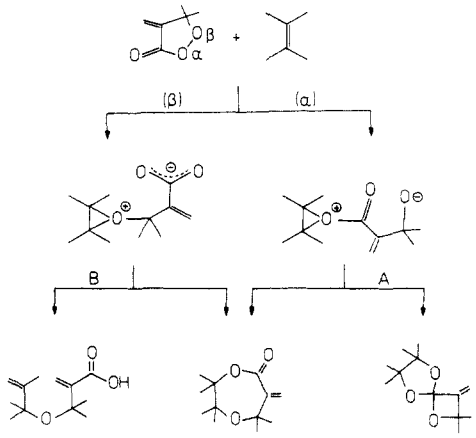
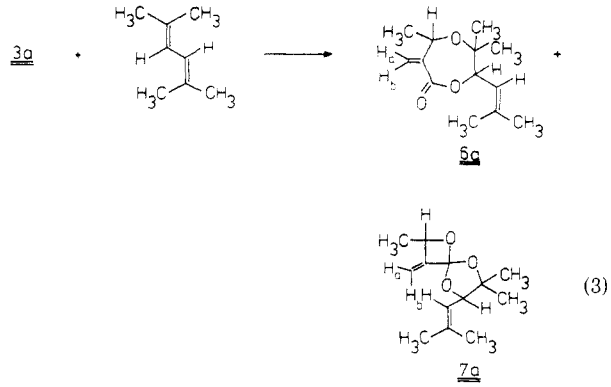
Scheme I



Of no little surprise was the chemical behavior of **3a** toward 2,5-dimethyl-2,4-hexadiene. The lactone **6a** and the orthoester **7a** were formed quantitatively in 28:72 ratio (eq 3). On separation by means of preparative HPLC, the



(3)

lactone **6a** (minor product) eluted first. Structural assignment rests on the following spectral data: (a) the carbonyl absorption at 1726 cm^{-1} is typical for α,β-unsaturated esters; (b) the two α-methylenic protons appear at quite similar chemical shifts ($\Delta\delta$ = 0.24 ppm), of which only one hydrogen shows a small coupling (J = 1.1 Hz) to the methinyl proton 7-H; (c) the singlet carbon resonance at δ 169.65 is typical for α,β-unsaturated esters.[5]

The orthoester **7a** eluted as second fraction (major product). The following spectral data speak for the suggested 1,4,6-trioxaspiro[4.3]octane structure: (a) no carbonyl absorption in the IR spectrum; (b) the two olefinic protons of the methylene group of the oxetane ring appear at significantly distinct chemical shifts ($\Delta\delta$ = 0.45 ppm) and exhibit coupling ($J_{6,2}$ = 1.8 Hz; $J_{6',2}$ = 2.9 Hz) to the methinyl proton 2-H, which is typical for exo-methylenes of four-membered rings; (c) the low intensity singlet carbon resonance at δ 120.11 is characteristic for orthoesters.[6] Also these products are similar to those produced between phthaloyl peroxide and stilbene.[7] The ^{1}H NMR (400 MHz) spectrum of the crude product mixture immediately after reaction revealed that no ene-type products were formed between **3a** and the diene.

To rationalize these experimental facts of the reaction between the α-methylene-β-peroxy lactone **3a** and olefins, we postulate the mechanism in Scheme I, with the zwitterionic species A and B as key intermediates. The zwitterion B, arising from electrophilic attack of the β-oxygen on the electron-rich alkenes, leads in the case of tetramethyl- and trimethylethenes under abstraction of an allylic hydrogen to the ene-type products **4** and **5** (eq 2). The zwitterion A, which would result from electrophilic attack by the α-oxygen on the simple alkenes, is less likely formed since it should be less stable than the zwitterion B. In the case of the diene, allylic stabilization of zwitterion B directs preferential attack at the internal carbon, affording the lactone **6a** as minor product rather than allylic hydrogen abstraction. In view of the additional allylic stabilization in the case of the diene, as major pathway the zwitterion A is formed via α-oxygen attack. Collapse at the internal carbon produces again the lactone **6a**. Alternatively, first attack at the carbonyl carbon and subsequent reaction at the internal carbon generates the orthoester **7a** as major product (eq 3).[7]

Experimental Section

IR spectra were obtained on a Perkin-Elmer Model 1420. ^{1}H NMR spectra were run on Hitachi-Perkin-Elmer R-24B (60 MHz), Varian EM-390 (90 MHz), and Bruker WM-400 (400 MHz) spectrometers and the ^{13}C NMR spectra (100 MHz) also on the latter. Chemical shifts (ppm) are given relative to tetramethylsilane for protons and deuteriochloroform for carbons. Mass spectra (70 eV) were measured on a Varian MAT CH-7. Combustion analysis for elemental composition were run in the Institute of Inorganic Chemistry, University of Würzburg. HPLC was performed on a Knauer preparative HPLC system using a 4-mm SiO_2 column.

4-Methylene-5,5-dimethyl-1,2-dioxolan-3-one (3b). A sample of 600 mg (5 mmol) of 2,3-dimethyl-2-butenoic acid **1b** in 20 mL of tetrachloromethane and 10 mL of dichloromethane (6×10^{-4} M in TPP) was irradiated at 0 °C for 12 h with a 150-W sodium lamp (Philips G/98/2) under continuous purging of dry oxygen. After rotoevaporation of the solvent, the residue was Kugelrohr distilled, yielding 610 mg (91%) of **3b** as colorless oil: bp 44–47 °C (0.1 torr); IR (film) 2991, 2918, 2875, 1780, 1670, 1460, 1408, 1315, 1190, 1099, 955, 849 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.56 (s, 6 H), 5.70 (d, J = 0.7 Hz, 1 H), 6.27 (d, J = 0.7 Hz, 1 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 25.47 (q), 87.62 (s), 121.71 (d), 142.23 (s), 169.15 (s). Anal. Calcd for $C_6H_8O_3$ (128.1): C, 56.25; H, 6.29. Found: C, 56.67; H, 6.72.

General Procedure for the Reaction of Olefins with 3a. To a solution of 300 mg (2.6 mmol) of **3a** in 5 mL of tetrachloromethane at 0 °C was added a solution of 26 mmol of the olefin in 5 mL of tetrachloromethane over a period of 10 min. After the solution was allowed to stir for 1 h at 0 °C and an additional 24 h at room temperature, the solvent was removed by rotoevaporation and the residue distilled under reduced pressure. The crude products were purified via column chromatography or preparative HPLC.

3,5,5,6-Tetramethyl-2-methylene-4-oxahept-6-enoic Acid (4). Following the general procedure, from the reaction of 300 mg (2.6 mmol) of **3a** and 2.18 g (26 mmol) of 2,3-dimethyl-2-butene was obtained 410 mg (80%) of **4** by distillation at 130–140 °C (0.1 torr) as colorless oil, which crystallized in colorless needles: mp 80–82 °C (1:2 petroleum ether (30–50)–ether); IR (film) 3400, 3002, 1702, 1388, 1280, 1175, 1091, 1056, 980, 918 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.21 (d, J = 7.0 Hz, 3 H), 1.27 (s, 6 H), 1.71 (mc, 3 H), 4.39 (q, J = 7.0 Hz, 1 H), 4.96 (mc, 2 H), 6.07 (d, J = 1.1 Hz, 1 H), 6.33 (d, J = 1.1 Hz, 1 H), 10.73 (s, 1 H, COO*H*); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 18.93 (q), 23.99 (q), 25.47 (q), 27.02 (q), 66.86 (d), 78.58 (s), 112.21 (t), 126.29 (t), 145.21 (s), 148.32 (s), 170.73 (s); MS (70 eV), m/e (relative intensity) 198 (M^+, 0.03), 154 ($M^+ - CO_2$, 0.6), 99 (38), 85 (100), 83 (78), 82 (20), 81 (42), 55 (76), 43 (93). Anal. Calcd for $C_{11}H_{18}O_3$ (198.2): C, 66.67; H, 9.10. Found: C, 66.63; H, 9.28.

(1) Adam, W.; Griesbeck, A. *Angew. Chem., Int. Ed. Engl.* **1985**, *12*, 1070.
(2) Normant, H.; Maitte, P. *Bull. Soc. Chim.* Fr. **1956**, 1439.
(3) Orfanopoulos, M.; Foote, C. S. *Tetrahedron Lett.* **1985**, *26*, 5991.
(4) Greene, F. D.; Rees, W. W. *J. Am. Chem. Soc.* **1958**, *80*, 3432.
(5) Adam, W.; Griesbeck, A. *Synthesis*, in press.
(6) Kalinowski, H. O.; Berger, S.; Braun, S. *^{13}C-NMR-Spektroskopie*; Thieme Verlag: Berlin, 1984.
(7) Greene, F. D. *J. Am. Chem. Soc.* **1955**, *78*, 2250.

3,5,6-Trimethyl-2-methylene-4-oxahept-6-enoic Acid (5). Following the general procedure, from the reaction of 300 mg (2.6 mmol) of **3a** and 1.82 g (26 mmol) of 2-methyl-2-butene was obtained 390 mg (82%) of **5** as colorless oil: bp 120–125 °C (0.08 torr); IR (film) 3420, 3001, 2952, 1702, 1639, 1441, 1378, 1282, 1096, 971, 909 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 1.22 (d, J = 6.4 Hz, 3 H), 1.27 (d, J = 7.2 Hz, 3 H), 1.62 (s, 3 H), 3.93 (q, J = 7.2 Hz, 1 H), 4.31 (q, J = 6.4 Hz, 1 H), 4.78 (s, 1 H), 4.88 (s, 1 H), 6.02 (s, 1 H), 6.35 (s, 1 H), 11.16 (s, 1 H, COO*H*); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 17.07 (q), 20.03 (q), 21.23 (q), 70.24 (d), 77.28 (d), 111.95 (t), 126.60 (t), 143.46 (s), 146.46 (s), 171.71 (s). Anal. Calcd for $C_{10}H_{16}O_3$ (184.2): C, 65.22; H, 8.69. Found: C, 65.39; H, 8.78.

Reaction of 3a with 2,5-Dimethyl-2,4-hexadiene. Following the general procedure, from the reaction of 300 mg (2.6 mmol) of **3a** with a fivefold excess of 2,5-dimethyl-2,4-hexadiene was isolated by Kugelrohr distillation at 158–170 °C (0.1 torr) 410 mg (70%) of colorless oil consisting of a 28:72 mixture of **6** and **7**. Separation by means of preparative HPLC afforded from a 100-mg mixture 21 mg of **6** (t_r 3.7 min) and 52 mg of **7** (t_r 5.6 min), using 92:8 petroleum ether (40–80)–ethyl acetate as eluant.

2,2,7-Trimethyl-3-(2-methyl-1-propenyl)-6-methylene-1,4-dioxacycloheptan-5-one (6a): IR (film) 2991, 2938, 2860, 1726, 1450, 1388, 1267, 1168, 1088, 1052, 981 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 1.04 (s, 3 H, 2-CH_3), 1.26 (s, 3 H, 2-CH_3), 1.32 (d, J = 5.9 Hz, 3 H, 7-CH_3), 1.62 (d, J = 1.9 Hz, 3 H, propenyl CH_3), 1.70 (d, J = 1.3 Hz, 3 H, propenyl CH_3), 4.34 (dq, ^3J = 5.9 Hz, ^4J = 1.1 Hz, 1 H, 7-H), 4.87 (d, ^3J = 9.6 Hz, 1 H, 3-H), 5.18 (dqq, ^3J = 9.6 Hz, ^4J = 1.9 and 1.3 Hz, 1 H, propenyl H), 5.68 (d, ^4J = 1.1 Hz; 1 H, methylene H), 5.92 (s, 1 H, methylene H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 17.56 (q) 18.93 (q), 25.85 (q), 27.08 (q), 29.78 (q), 64.54 (d), 77.59 (s), 84.20 (d), 120.59 (d), 122.46 (t), 138.53 (s), 145.46 (s), 169.65 (s); MS (70 eV); m/e (relative intensity) 222 (M^+ – 2, 0.1), 193 (3), 150 (15), 107 (24), 95 (17), 91 (18), 43 (100). Anal. Calcd for $C_{13}H_{20}O_3$ (224.1): C, 69.61; H, 8.99. Found: C, 69.38; H, 9.21.

2,6,6-Trimethyl-7-(2-methyl-1-propenyl)-3-methylene-1,5,8-trioxaspiro[3.4]octane (7a): IR (film) 2996, 2938, 1.8 1600, 1552, 1448, 1372, 1281, 1182, 1088, 1040, 982 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 1.08 (s, 3 H, 6-CH_3), 1.16 (s, 3 H, 6-CH_3), 1.31 (d, J = 6.3 Hz, 3 H, 2-CH_3), 1.63 (d, J = 1.4 Hz, 3 H, propenyl CH_3), 1.72 (d, J = 1.4 Hz, 3 H, propenyl CH_3), 4.54 (ddq, ^3J = 6.3 Hz, ^4J = 1.8 and 2.9 Hz, 1 H, 2-H), 4.97 (d, ^3J = 8.8 Hz, 1 H, 7-H), 5.23 (ddd, ^3J = 8.8 Hz, ^4J = 1.4 and 1.2 Hz, 1 H, propenyl H), 5.35 (d, ^4J = 1.8 Hz, 1 H, methylene H), 5.79 (d, ^4J = 2.9 Hz, 1 H, methylene H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 19.19 (q), 20.75 (q), 22.32 (q), 23.78 (q), 25.97 (q), 65.76 (d), 77.40 (s), 77.79 (d), 119.14 (d), 120.11 (s), 120.18 (t), 139.61 (s), 149.70 (s); MS (70 eV), m/e (relative intensity) 224 (M^+, 0.5), 163 (20), 121 (14), 95 (23), 55 (24), 43 (100), 41 (32). Anal. Calcd for $C_{13}H_{20}O_3$ (224.1): C, 69.61; H, 8.99. Found: C, 69.85; H, 9.14.

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C-Prenylation of Phenols Promoted by Aluminum Oxide Surfaces

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C-Alkenylation of phenols are traditionally carried out in acidic[1] or basic media.[2-4] Other processes, such as those utilizing unusual Friedel–Crafts conditions,[5] platinum catalysis,[6] and silicon chemistry[7] were developed more recently. Attempts to C-alkylate phenols containing strongly electron-withdrawing substituents with allylic halides have met with only limited success.[5] These reactions produced mainly *O*-alkylated phenols and only very minor amounts of the C-alkylated isomers.

A large number of natural products of mixed acetate–mevalonate origin contain prenyl substituents and in connection with a synthetic project in this area we had occasion to examine the prenylation of phenols containing electron-withdrawing appendages and found that C-alkylation with either prenyl chloride or bromide can be accomplished with acceptable yields, in two-phase systems. The solid phases examined consisted of a mixture of aluminum oxide and barium oxide or simply commercial aluminum oxide used for thin-layer chromatography. The reactions were found to proceed slowly at room temperature, and the conditions are sufficiently mild that even the sensitive phenol **15** could be prenylated in satisfactory yield. No α,α-dimethallyl-substituted phenols were detected, and in three cases minor amounts of isomeric, C-substituted γ,γ-dimethallyl derivatives were formed. Two of these are derived from cyclohexadienone intermediates not containing the β-hydroxy α,β-unsaturated aldehyde chelate and the third from an intermediate with disrupted α-pyrone ring. We have prepared nine dimethallyl-substituted phenols employing this method and the results are summarized in Table I. The solid phases can be reused provided solvents have been removed by heating in vacuo at approximately 80 °C. Raising the temperature of the reaction medium caused darkening of the solutions and yields were lower.

Experimental Section

The following spectrometers were used: IR, Perkin-Elmer 397; NMR, Varian T-60, JEOL FXQ, Bruker wm 250; UV, Hitachi Perkin-Elmer 200; MS, Varian MAT-44, Finnigan MAT 8200. Melting points were determined on a Reichert hot stage microscope and are uncorrected.

Method A. The phenol (1.0 mmol) in 5 mL of THF was added to a slurry of 5–10 g of Al_2O_3 (basic, or neutral Type T or E for thin layer chromatography, Merck) in ether. The solvent was evaporated and 1–5 equiv of the allylic halide in ether–hexane (30–100%) or CH_2Cl_2 was added. After stirring for 1–5 days, Al_2O_3 was filtered off and washed with 1% HOAc–EtOAc. The combined organic layer was evaporated and the product purified by chromatography or crystallization.

Method B. The phenol (1.0 mmol) in 5 mL of THF was added to a slurry of 30% BaO–Al_2O_3 (3–5 equiv by weight as a fine powder (10 μm) in dry ether. The solvent was evaporated and 1–5 equiv of the allylic halide in ether–hexane (30–100%) or CH_2Cl_2 was added. After 1–6 days the solid was filtered off and washed with 1% HOAc–EtOAc. The combined organic layers were evaporated and purified by chromatography or crystallization.

(1) Molyneux, R. J.; Jurd, L. *Tetrahedron* **1970**, *26*, 4743.
(2) Jain, A. C.; Lal, P.; Seshadri, T. R. *Indian J. Chem.* **1969**, *7*, 1072.
(3) Jain, A. C.; Sharma, B. N. *J. Org. Chem.* **1974**, *39*, 2215.
(4) Canonica, L.; Rindone, B.; Santaniello, E.; Scolastico, C. *Tetrahedron* **1972**, *28*, 4395.
(5) Bigi, F.; Casiraghi, G.; Casnati, G.; Sartori, G. *Synthesis* **1981**, 310.
(6) De Renzi, A.; Panunzi, A.; Saporito, A.; Vitagliano, A. *J. Chem. Soc., Perkin Trans.* **1983**, 993.
(7) Sarma, D. N.; Baruah, P.; Pandey, U. C.; Sharma, R. P. *Tetrahedron Lett.* **1984**, *25*, 5581.
(8) Steck, W. *Can. J. Chem.* **1971**, *49*, 1197.
(9) Bajwa, B. S.; Khanna, P. L.; Seshadri, T. R. *Indian J. Chem.* **1971**, *9*, 1322.
(10) Kyogoku, K.; Hatayama, K.; Yokomori, S.; Tanaka, I. *Agric. Biol. Chem.* **1975**, *39*, 133.
(11) Mori and Sato (Mori, K.; Sato, K. *Tetrahedron* **1982**, *38*, 1221) failed to alkylate phenol 17 with geranyl bromide.

0022-3263/86/1951-4481$01.50/0