spectra were determined on a Perkin-Elmer 257 spectrometer. ^1H and ^{13}C NMR spectra were measured at 90 and 400 MHz and 20.15, 50.31, and 100.62 MHz, respectively, in pyridine-d_5, $CDCl_3$, or C_6D_6 solution with Me_4Si as an internal standard. Assignments of ^{13}C chemical shifts were made with the aid of off-resonance, APT-type, and noise-decoupled ^{13}C NMR spectra.

Mass spectra were obtained by electron impact on a Hitachi Perkin-Elmer RMU-6MG instrument.

Isolation of Teucroxide (1). Dried and finely powdered *T. chamaedrys* L. (aerials parts, 2.2. kg), collected near Cimelos del Pinor (Guadalajara, Spain), were extracted with acetone as previously described.[2g] The most polar chromatographic diterpenoid, eluted from a silica gel column with $CHCl_2$–MeOH (6:1), was teucroxide (1): 1062 mg, mp 183–184 °C (from AcOEt); $[\alpha]^{22}_D$ −29.2° (*c* 0.545, pyridine); IR (KBr) 3460, 3360, 3290, 3150, 3130, 3120, 2960, 2910, 2890, 1760, 1600, 1508, 1470, 1360, 1325, 1315, 1190, 1165, 1065, 1025, 975, 960, 880, 835, 820, 740, 720, 620, 600 cm^{-1}; UV (EtOH) λ_{max} 210 nm (log ϵ 3.60), furan ring; ^1H NMR (pyridine-d_5), see Table I; ^{13}C NMR (pyridine-d_5), see Table II; mass spectrum (75 eV, direct inlet), *m/z* (relative intensity) 378 (M^+, 3) 360 (36), 347 (48), 342 (3), 329 (6), 311 (5), 301 (4), 286 (7), 283 (13), 266 (14), 179 (38), 178 (21), 161 (26), 145 (21), 133 (26), 108 (37), 105 (38), 95 (100, base peak), 94 (62), 91 (50), 81 (90), 79 (40), 77 (43), 69 (32), 67 (31), 65 (32), 55 (53), 53 (52), 43 (98). Anal. Calcd for $C_{20}H_{26}O_7$: C, 63.48; H, 6.93. Found: C, 63.21; H, 6.96.

Compound 2. A solution of 750 mg of teucroxide (1) in 20 mL of pyridine and 5 mL of acetic anhydride was allowed to stand overnight at room temperature. The reaction mixture was poured into ice–water and extracted with $CHCl_3$. A workup in the usual manner yielded, after purification by column chromatography, 990 mg of 2: mp 115–118 °C (from MeOH); $[\alpha]^{19}_D$ −20.4° (*c* 0.90, $CHCl_3$); IR (KBr) 3170, 3140, 3020, 2970, 2940, 2890, 1760, 1740, 1720, 1600, 1505, 1440, 1370, 1240, 1190, 1160, 1035, 1025, 985, 930, 910, 880, 860, 810, 740, 730 cm^{-1}; UV (EtOH) λ_{max} 215 nm (log ϵ 3.49), furan ring; ^1H NMR ($CDCl_3$ and C_6D_6), see Table I; ^{13}C NMR ($CDCl_3$), see Table II; mass spectrum (75 eV, direct inlet), *m/z* (relative intensity) 504 (M^+, 3) 444 (12), 431 (46), 402 (16), 384 (28), 371 (4), 342 (10), 329 (28), 324 (12), 308 (15), 290 (60), 283 (33), 275 (12), 268 (21), 248 (41), 174 (73), 145 (40), 143 (36), 119 (38), 95 (94), 91 (52), 81 (85), 43 (100, base peak). Anal. Calcd for $C_{26}H_{33}O_{10}$: C, 61.89; H, 6.39. Found: C, 61.76; H, 6.47.

Acknowledgment. We thank Dr. J. Borja, Botany Department, Faculty of Pharmacy, Madrid, for collection and botanical classification of the plant material. One of us (M.C.G.-A.) thanks the Spanish CSIC for a fellowship. This work was supported in part by the Comisiõn Asesora de Investigacioñ Cientifica y Technica (Grant No. 11/81), Spain, and in part by the National Research Council (CNR), Italy.

Registry No. 1, 87587-49-3; 2, 87587-50-6.

Synthesis of 2-Methyl-3-hydroxy-4*H*-pyran-4-one and 4-Hydroxy-5-methyl-2*H*-furan-3-one from Carbohydrates

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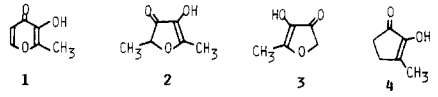
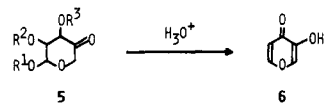
Received June 21, 1983

Cyclic α-diketones, such as 2-methyl-3-hydroxy-4*H*-pyran-4-one (**1**, maltol),[1] 2,5-dimethyl-4-hydroxy-2*H*-furan-3-one (**2**, furaneol),[2,3] 4-hydroxy-5-methyl-2*H*-furan-one (**3**), and 2-hydroxy-3-methylcyclopent-2-en-1-one (**4**, cyclotene)[4] have been known to be important key flavors in a variety of foods, and much effort has been devoted to their synthesis.



Although some routes of the synthesis of **1**–**3** have been developed so far by utilizing carbohydrates as starting compounds,[5] the yields or availability of the starting carbohydrates are not always satisfactory. Thus, it seems worthwhile to develop new methods of synthesizing α-diketones from the most easily available carbohydrates. This paper describes the synthesis of **1** from D-glucose and the synthesis of **3** from D-xylose and from D-xylitol.

Synthesis of 1 from D-Glucose. The structure of **1** is characterized by a methyl group on C-2 and a hydroxyl group on C-3 of the 4*H*-pyran-4-one skeleton. The construction of the 3-hydroxy-4*H*-pyran-4-one skeleton from glucose is expected to be accomplished by the oxidation of only one hydroxyl group of glucose at the C-4 position, since it has already been found by us[1] and other groups[1,5] that the 1,2,3-trihydroxy-4-ketotetrahydropyran skeleton **5** can be transformed to the 3-hydroxy-4*H*-pyran-4-one skeleton **6** by treatment of **5** with aqueous acid (eq 1).



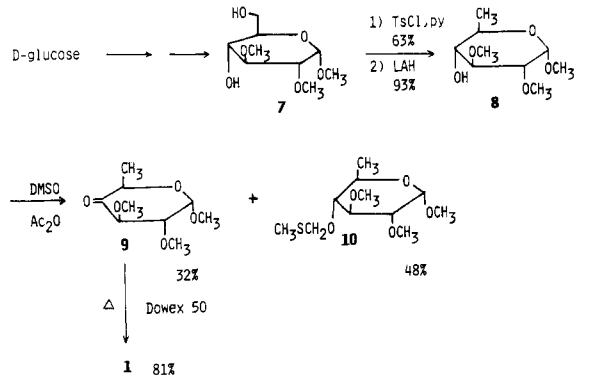
(1)

5 **6**

R^1, R^2, R^3 = alkyl, hydrogen, benzoyl, or acyl group

The methyl group on C-2 of **1** may be easily formed by the reductive removal of a hydroxyl group on C-6 of glucose. In fact, the synthesis of **1** from glucose was achieved by the procedures shown in Scheme I.

Scheme I



Methyl 2,3-di-*O*-methyl-α-D-glucopyranoside (**7**), easily prepared from D-glucose by the reported method,[6] was tosylated to give methyl 2,3-di-*O*-methyl-6-*O*-tosyl-α-D-glucopyranoside, and the tosylate was subsequently re-

(1) (a) Shono, T.; Matsumura, Y. *Tetrahedron Lett.* **1976**, 1363. (b) Torii, S.; Tanaka, H.; Anoda, T.; Shimizu, Y. *Chem. Let.* **1976**, 495. (c) Weeks, P. D.; Brennan, T. M.; Brannegan, D. P.; Kuhla, D. E.; Elliott, M. L.; Watson, H. A.; Wlodecki, B.; Breitenback, R. *J. Org. Chem.* **1980**, 45, 1109, and references cited therein.

(2) (a) Buchi, G.; Demole, E.; Thomas, A. F. *J. Org. Chem.* **1973**, *38*, 123. (b) Henry, D. W.; Silverstein, R. M. *Ibid.* **1966**, *31*, 2391.

(3) Re, L.; Maurer, B.; Ohloff, G. *Helv. Chim. Acta* **1973**, *56*, 1882.

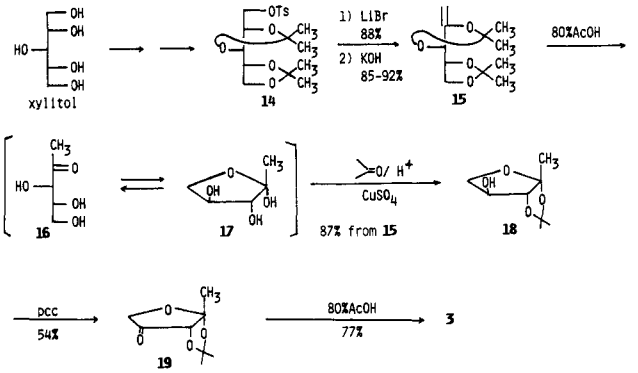
(4) (a) Leir, C. M. *J. Org. Chem.* **1970**, *35*, 3203. (b) Sato, K.; Inoue, S.; Kitagawa, T.; Takahashi, T. *Ibid.* **1973**, *38*, 551. (c) Shono, T.; Matsumura, Y.; Hamaguchi, H. *J. Chem. Soc., Chem. Commun.* **1977**, 712.

(5) (a) For the synthesis of 1, see: Lichtenthaler, F. W.; Heidel, P. *Angew. Chem.* **1969**, *81*, 998. Chawla, R. K.; McGonigal, W. E. *J. Org. Chem.* **1974**, *39*, 3281. (b) For the synthesis of compounds **2** and **3**, see: Mills, F. D.; Hodge, J. E. *Carbohydr. Res.* **1976**, *51*, 9. Hicks, K. B.; Feather, M. S. *J. Agric. Food Chem.* **1975**, *23*, 957.

(6) Edington, R. A.; Hirst, E. L.; Percival, E. E. *J. Chem. Soc.* **1955**, 2281.

0022-3263/83/1948-5126$01.50/0

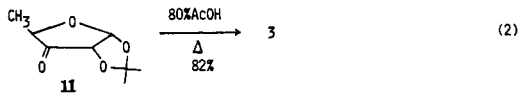
Scheme II



duced to afford the alcohol 8. Although the hydroxyl group on C-4 of 8 was hardly oxidizable with MnO_2, CrO_3, or pyridinium chlorochromate (pcc), the oxidation of 8 with Me_2SO–Ac_2O gave a mixture of 9 (32%) and 10 (48%). After the isolation of 9 from the mixture, refluxing the aqueous solution of 9 in the presence of Dowex 50 for 25 h afforded 1 in 81% yield.

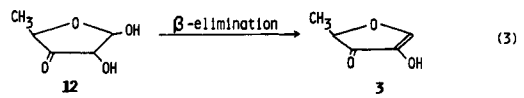
Synthesis of 3 from D-Xylose. On the basis of the similarity of the structure of 3 with that of furanose, D-xylose was used as a starting sugar for the synthesis of 3.

The hitherto know synthesis of the key compound 11 from D-xylose involves oxidation of the intermediate 5-deoxy-1,2-*O*-(1-methylethylidene)-α-D-xylofuranose to 11 by using RuO_4 as an oxidizing agent (75% yield),[7] whereas the oxidation was also achieved by using pcc in our study (83% yield). The treatment of 11 with 80% acetic acid for 0.5 h was found to afford 3 in 82% yield (eq 2).



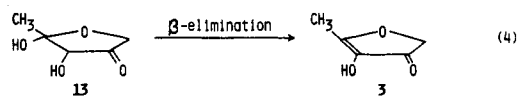
(2)

Synthesis of 3 from Xylitol. The conversion of 11 to 3 presumably proceeds through the intermediary formation of 12, followed by the β-elimination of a hydroxyl group of 12 (eq 3).



(3)

The synthesis of 3 may also be possible by β-elimination of an hydroxyl group of 13 (eq 4). Thus, the synthesis of



(4)

the intermediate 13 or its derivatives from easily available sugars is the next problem for the synthesis of 3.

Then, xylitol was converted to 1,2:3,4-bis-*O*-(1-methylethylidene)-5-*O*-tosylxylitol (14) according to a known method.[8] The replacement of the tosyl group of 14 with bromide ion and subsequent dehydrobromination gave the vinyl ether 15, which was deprotected in aqueous acetic acid to yield a mixture of 16 and 17. Treatment of the mixture with acetone in the presence of acid and subsequent oxidation of 18 with pcc gave the expected ketone 19.

Similarly to the transformation of 11 to 3 in eq 2, the conversion to 19 to 3 was accomplished by treatment of 19 with acid, the yield being 77% (Scheme II).

The route utilizing xylitol as a starting sugar is more practical for the synthesis of 3 than that starting from xylose, since in Scheme II the reductive transformation of a terminal hydroxymethyl group to a methyl group and the oxidation of a 2-hydroxy group to a carbonyl group were achieved by the intramolecular oxidation–reduction process without using any oxidizing and reducing agents.

Experimental Section

General Procedures. Infrared spectra were recorded on Hitachi 215 spectrometer. Proton nuclear magnetic resonance spectra were measured on Varian Associates EM-390 spectrometer, with chemical shifts given in parts per milion (δ) downfield from tetramethylsilane as an internal standard. Mass spectra were obtained on JEOL IMS-DX300 instrument. Elemental analyses were determined by the Center for Instrumental Analysis of Kyoto University. Melting points were determined on a Yanako melting points apparatus and were uncorrected. Column chromatography was performed on silica gel 70–230 mesh obtained from Merk. Tetrahydrofuran and ether were distilled under nitrogen from lithium aluminium hydride. Methylene chloride was distilled from calcium chloride. Pyridine was distilled from potassium hydroxide. Me_2SO and DMF were dried with calcium hydride.

Methyl 2,3-di-*O*-methyl-6-*O*-tosyl-α-D-glucopyranoside was prepared according to Harrison et al.[9] in 63% yield and had the following properties: oil; IR (neat) 3450, 1590, 1360, 1180, 1060, 980 cm^{-1}; ^1H NMR (CCl_4) δ 2.40 (d, 3 H), 2.70–4.00 (m, 6 H), 3.33 (s, 3 H), 3.40 (s, 3 H), 3.53 (s, 3 H), 4.20 (d, J = 9 Hz, 2 H), 4.67 (d, J = 4 Hz, 1H), 7.30 (d, J = 9 Hz, 2 H), 7.70 (d, J = 9 Hz, 2 H). Anal. Calcd for $C_{16}H_{24}O_8S$: C, 51.06; H, 6.43; S, 8.52. Found: C, 50.93; H, 6.48; S, 8.70.

Methyl 6-Deoxy-2,3-di-*O*-methyl-α-D-glucopyranoside (8). The reduction of methyl 2,3-di-*O*-methyl-6-*O*-tosyl-α-D-glucopyranoside (7.850 g, 20.9 mmol) with $LiAlH_4$ (1.5 g, 39 mmol) in 100 mL of dry ether and 5 mL of tetrahydrofuran yielded 8 (4.010 g, 19.5 mmol): yield 93%; oil; bp 102–105 °C (1.6 mm); IR (neat) 3450, 1200, 1160, 1060 cm^{-1}; ^1H NMR (CCl_4) δ 1.13 (d, J = 6 Hz, 3 H), 2.67 (br m, 1 H), 2.6–3.67 (m, 4 H), 3.30 (s, 3 H), 3.33 (s, 3 H), 3.50 (s, 3 H), 4.55 (d, J = 4 Hz, 1 H). Anal. Calcd for $C_9H_{18}O_5$: C, 52.41; H, 8.80. Found: C, 52.25; H, 9.02.

Oxidation of 8. Acetic anhydride (4 mL) was added into the solution of 8 (1.020 g, 4.95 mmol) in 6 mL of dry Me_2SO. The starting material was completely consumed when the solution was stirred at room temperature overnight. After the solvent was removed in vacuo, the products (9 and 10) were isolated by distillation. **9**: yield 32%; oil; bp 97–102 °C (1.6 mm); IR (neat) 1730, 1160, 1100, 1060 cm^{-1}; ^1H NMR (CCl_4) δ 1.20 (d, J = 5 Hz, 3 H), 3.20–4.27 (m. 3 H), 3.50 (s, 6 H), 3.53 (s, 3 H) 4.70 (d, J = 4 Hz, 1 H). Anal. Calcd for $C_9H_{16}O_5$: C, 55.54; H, 7.46. Found: C, 55.48; H, 7.49. **10**: yield 45%; oil; bp 118–121 °C (1.6 mm); IR (neat) 2910, 1150, 1060 cm^{-1}; ^1H NMR (CCl_4) δ 1.17 (d, J = 6 Hz, 3 H), 2.10 (s, 3 H), 2.80–3.65 (m, 4 H), 3.30 (s, 3 H), 3.36 (s, 3 H), 3.47 (s, 3 H), 4.50 (d, J = 4 Hz, 1 H), 4.73 (s, 2 H). Anal. Calcd for $C_{11}H_{22}O_5S$: C, 49.61; H, 8.33; S, 12.07. Found: C, 49.72; H, 8.47; S, 11.77.

Preparation of Maltol from 9. Reflux of the aqueous solution of 9 (0.28 g, 1.37 mmol) in 4 mL of water with Dowex 50 W for 25 h gave maltol (0.14 g, 1.12 mmol) in 81% yield.

Synthesis of 5-Deoxy-1,2-*O*-(1-methylethylidene)-D-*glycero*-D-*glycero*-3-pentofuranosulose (11). A solution of 5-deoxy-1,2-*O*-(1-methylethylidene)-α-D-xylofuranose[7] (2.40 g, 0.014 mol) in CH_2Cl_2 (10 mL) was added dropwise into a flask containing pcc (4.5 g) and CH_2Cl_2 (30 mL). After the solution was refluxed for 8 h, the usual workup and purification (silica gel column, THF/hexane, 1:4) gave 11 (1.965 g, 0.0116 mol) in 83% yield: mp 53–54 °C (lit.[7] mp 39–41 °C); IR (KBr) 2980, 1770, 1445, 1380, 1220, 1160, 1095, 1025, 995, 870 cm^{-1}; ^1H NMR ($CDCl_3$)

(7) Tronchet, J. M. J.; Graf, R.; Gurny, R. *Helv. Chim. Acta* **1972**, *55*, 613.

(8) Hann, R. M.; Ness, A. T.; Hudson, C. S. *J. Am. Chem. Soc.* **1944**, *66*, 73.

(9) Harrison, J. M.; Inch, T. D.; Lewis, G. J. *J. Chem. Soc., Perkin Trans 1*, **1975**, 1892.

δ 1.30 (d, J = 6 Hz, 3 H), 1.36 (s, 3 H), 1.50 (s, 3 H), 4.39 (q, J = 6 Hz, 1 H), 4.30 (d, J = 4 Hz, 1 H), 6.03 (d, J = 4 Hz, 1 H). Anal. Calcd for $C_8H_{12}O_4$: C, 55.80; H, 7.03. Found: C, 55.67; H, 7.21.

Synthesis of 3 from 11. A solution of **11** (250 mg, 1.45 mmol) in 80% aqueous AcOH (25 mL) was refluxed for 0.5 h. The solvent was evaporated in vacuo to afford a yellow solid. Pure product (**3**) was obtained in 82% yield by recrystallization of the crude yellow solid (Et_2O/pentane, 1:4), mp 124–127 °C (lit.[3] mp 124–125 °C).

Synthesis of 1,2:3,4-Bis-*O*-(1-methylethylidene)-5-bromo-5-deoxyxylitol. Into a solution of 1,2:3,4-bis-*O*-(1-methylethylidene)-5-tosylxylitol[8] (38.5 g, 0.1 mol) in 250 mL of DMF was added lithium bromide (21.0 g, 0.2 mol). After the solution was stirred at 90 °C for 2 h, it was cooled to room temperature, poured into ice–water, and extracted with ether. The extract was washed with brine, dried over $MgSO_4$, and filtered, and the filtrate was evaporated. The residue was distilled under reduced pressure to give 1,2:3,4-bis-*O*-(1-methylethylidene)-5-bromo-5-deoxyxylitol (26.0 g, 0.088 mol) in 88% yield: bp 97.5–102 °C (2–3 mm); IR (neat) 2980, 2935, 2890, 1455, 1380, 1250, 1220, 1150, 1060, 990, 960, 890, 845, 800 cm^{-1}; ^1H NMR (CCl_4) δ 1.33 (br s, 12 H), 3.40 (d, J = 5 Hz, 2 H), 3.7–4.4 (m, 5 H). Anal. Calcd for $C_{11}H_{19}BrO_4$: C, 44.76; H, 6.49; Br, 27.07. Found: C, 44.91; H, 6.58; Br, 27.41.

Synthesis of 1,2:3,4-Bis-*O*-(1-methylethylidene)-5-deoxy-4,5-didehydroxylitol (15). Into a distillation flask were charged 1,2:3,4-bis-*O*-(1-methylethylidene)-5-bromo-5-deoxyxylitol (23.6 g, 0.80 mol) and pulverized KOH (54 g, 0.96 mol), and the mixture was heated under reduced pressure to distill crude vinyl ether. Fractional distillation gave **15**: bp 101–105 °C (15 mm); yield 14.5–15.8 g (85–92%); IR (neat) 2990, 2940, 2880, 1680, 1380, 1220, 1150, 1060 cm^{-1}; ^1H NMR (CCl_4) δ 1.25 (s, 3 H), 1.33 (s, 6 H), 1.43 (s, 3 H), 3.50–4.33 (m, 5 H), 4.53 (m, 1 H); mass spectrum, m/e 214.

Synthesis of 1-Deoxy-2,3-*O*-(1-methylethylidene)-α-L-*threo*-2-pentosulose-(2,5) (18). After a solution of **15** (10.75 g, 0.05 mol) in acetic acid/H_2O (4:1, 50 mL) was stirred at room temperature for 12 h, the solvent was removed under reduced pressure to afford a colorless syrup. Into a solution of this syrup in acetone (50 mL) were added anhydrous $CuSO_4$ (15 g) and concentrated H_2SO_4 (0.2 mL). The reaction mixture was stirred for 24 h and filtered, and the filtrate was neutralized with $Ca(OH)_2$ (10 g). After the precipitate was filtered, the solution was evaporated to give a syrup. Pure alcohol (**18**) was isolated from the syrup by column chromatography (THF/hexane, 1:4; silica gel) in 87% yield: mp 77–78 °C; IR (KBr) 3400, 2980, 2940, 1460, 1440, 1380, 1308, 1300, 1250, 1240, 1208, 1180, 1155, 1100, 1060, 1000, 985, 925, 900, 870, 845 cm^{-1}; ^1H NMR (CDCl)3) δ 1.36 (s, 3 H), 1.48 (s, 3 H), 1.70 (s, 3 H), 2.53 (br s, 1 H), 3.80–4.30 (m, 4 H). Anal. Calcd for $C_8H_{14}O_4$: C, 55.16; H, 8.10. Found: C, 55.04; H, 8.19.

Oxidation of 18. Pcc (3.2 g, 14.8 mmol) was gradually added into a solution of **18** (0.87 g, 5.0 mmol) in CH_2Cl_2 (150 mL). After the solution was refluxed for 6 h, the usual workup and subsequent purification by silica gel column chromatography (THF/hexane, 1:4) gave **19** in 54% yield: oil; IR (neat) 2980, 2840, 1770, 1200, 1100, 1050, 980, 900, 850 cm^{-1}; ^1H NMR (CCl_4) δ 1.40 (s, 6 H), 1.63 (s, 3 H), 3.89 (s, 1 H), 3.92 (d, J = 18 Hz, 1 H), 4.33 (d, J = 18 Hz, 1 H). Anal. Calcd for $C_8H_{12}O_4$: C, 55.80; H, 7.03. Found: C, 56.10; H, 7.18.

Synthesis of 3 from 19. A solution of **19** (250 mg, 1.45 mmol) in 80% aqueous acetic acid (25 mL) was refluxed for 0.5 h. The solvent was evaporated in vacuo to afford a yellow solid. Pure product **3** was obtained in 77% yield by recrystallization of the crude solid (Et_2O/pentane, 1:4): mp 125–127 °C; IR (KBr) 3180, 1680, 1615, 1450, 1400, 1360, 1305, 1195, 1140, 995, 920, 700 cm^{-1}; ^1H NMR ($CDCl_3$–CCl_4) δ 2.27 (s, 3 H), 4.47 (s, 2 H), 6.30 (br, 1 H).

Registry No. 1, 118-71-8; **3**, 19322-27-1; **7**, 14048-30-7; **8**, 62853-52-5; **9**, 69500-61-4; **10**, 87597-65-7; **11**, 32453-67-1; **12**, 87597-66-8; **14**, 87678-03-3; **15**, 87597-67-9; **16**, 60299-43-6; **17**, 5077-24-7; **18**, 87597-68-0; **19**, 87597-69-1; methyl 2,3-di-*O*-methyl-6-*O*-tosyl-α-D-glucopyranoside, 25019-43-6; 1,2:3,4-bis-*O*-(1-methylethylidene)-5-bromo-5-deoxyxylitol, 87597-70-4.

Chemistry of Oxaziridines. 6.[1] Hydroxylation of Anisole by 2-Sulfonyloxaziridines

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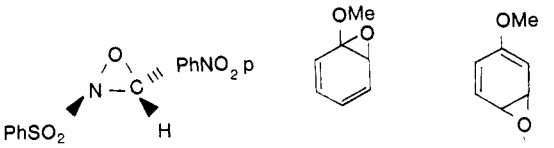
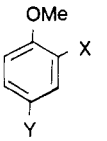
Department of Chemistry, Drexel University, Philadelphia, Pennsylvania 19104

Received March 1, 1983

Exploration of the oxygen-transfer reactions of 2-sulfonyloxaziridines, **1**, is of importance not only because of synthetic and mechanistic considerations[2] but also as models for biochemical monooxygenases.[3,4] Although controversial,[5] an oxaziridine intermediate has been proposed by Orf and Dolphin[6] and extended by Rastetter et al.[7] for hydroxylations mediated by the flavin-dependent monooxygenases.



1 **2**



3, X = OH, Y = H

4, X = H, Y = OH

The oxygen-transfer processes in the photolysis of heteroaromatic *N*-oxides are considered to be one of the better model systems for the monooxygenases.[8-11] This biomimetic system epoxidizes olefins, oxidizes sulfides to sulfoxides, and hydroxylates aromatic hydrocarbons.[10] Enzymatic oxidations of aromatic hydrocarbons frequently

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