reaction mixture was stirred for 72 h at 20–25 °C and then worked up by method B described above. An ^1H NMR spectral analysis of the reaction product showed the presence of a 3:7 ratio of methyl 1-*p*-tolylvinyl ether and the starting ester.

(c) Unmodified Agent Giving Aldol Condensation. Interaction of 100 mL of methylenating agent and 18 mmol of cyclohexanone gave, after 3 h at 25 °C and workup by method A, 60% of isolated 2-cyclohexylidenecyclohexanone, but no discernible methylene cyclohexane.

1,1-Dialuminohexanes (5–7) with Benzophenone. (a) Reagent 5. A solution of 18 mmol of **5** in 10 mL of toluene was mixed with a solution of 5.0 g (17.8 mmol) of benzophenone in 25 mL of anhydrous toluene. After 16 h of stirring at 25 °C, the reaction mixture was slowly and cautiously hydrolyzed (*gas evolution*) at 0 °C with 5 mL of 1 N aqueous HCl. The separated organic layer was washed with aqueous $NaHCO_3$, dried over anhydrous $MgSO_4$, and evaporated. Gas chromatographic analysis and mass spectral identification of the components showed that the ketone had been consumed, but only traces of the desired 1,1-diphenyl-1-heptene had been formed. The principal outcome of the reaction was reduction: diphenylmethanol was separated and identified.

In another similar reaction, conducted for 17 h at 25 °C and for 60 min at 110 °C, a 9% yield of 1,1-diphenyl-1-heptene was obtained, but again reduction dominated. This finding suggests that the reduction to diphenylmethanol (as its aluminum salt) may be reversible.

(b) Reagent 6. In a reaction conducted in an analogous manner, this reagent converted benzophenone into 1,1-diphenyl-1-heptene in a 71% yield. This product could easily be separated from reduction products by column chromatography on silica gel with hexane as the eluent, or even by simple distillation. This hydrocarbon was identified by spectral comparison with an authentic sample that was obtained by the partial reduction of known 1,1-diphenyl-1,6-heptadiene.[35]

(c) Reagent 7. In a similar manner, after 16 h at 25 °C, **7** converted benzophenone into 1,1-diphenyl-1-heptene in 55% yield.

(35) Eisch, J. J.; Merkley, J. H. *J. Am. Chem. Soc.* **1979**, *101*, 1148.

Notes

Base-Promoted Reaction of 5,6,7,8-Tetrachloro-5,8-dimethoxymethano-4a,5,8,8a-tetrahydro-1,4-naphthoquinone Epoxide[1]

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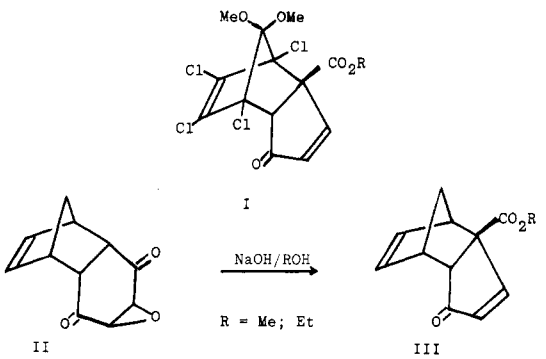
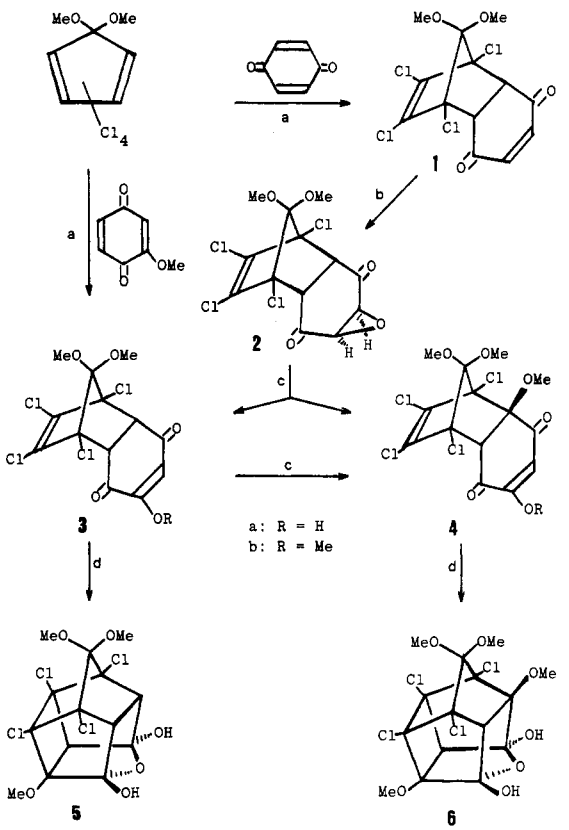
Received October 14, 1987

In connection with our program aimed at the synthesis of homocubane and triquinane derivatives, we choose the tricyclic compound I as the starting material. The preparation of I was suggested by the known reaction of tricyclic enedione epoxide II which, upon treatment with alcoholic sodium hydroxide, yielded the Favorskii ring contraction product III.[2] To this end, we undertook the investigation of the base-promoted reaction of the title compound **2**. However, we found that the enedione epoxide **2** exhibited chemical behavior different from that of epoxide II. In the present report, we describe the results of the study outlined in Scheme I.



Scheme I[a]



[a] (a) Benzene, reflux, ca. 2 days (**3b**, 98%); (b) acetone, ice-cold 20% aqueous $NaHCO_3$, 30% H_2O_2, 0 °C, 10 min (**2**, 95%); (c) NaOMe, MeOH, reflux, 2 h, then cold concentrated HCl (**3a**: 70%)/CH_2N_2, Et_2O (**3b**, 83%)//2 M aqueous NaOH, MeOH, overnight at room temperature, then cold concentrated HCl (**4a**, 70%)/CH_2N_2, Et_2O (**4b**, 88%); (d) acetone, light, 2 h (**5**, 73%; **6**, 81%).

(1) Studies on Cage Compounds. 2. 1: Chou, T.-C.; Chiou, J.-H. *J. Chin. Chem. Soc.* **1986**, *33*, 227.

(2) (a) Herz, W.; Iyer, V. S.; Nair, M. G. *J. Org. Chem.* **1975**, *40*, 3519. (b) Marchand, A. P.; Suri, S. C. *Ibid.* **1984**, *49*, 2041. (c) Klunder, A. J. H.; de Valk, W. C. G. M.; Verlaak, J. M. J.; Schellekens, J. W. M.; Noordik, J. H.; Parthasarathi, V.; Zwanenburg, B. *Tetrahedron* **1985**, *41*, 963.

0022-3263/88/1953-2835$01.50/0

The enedione epoxide **2** was prepared in quantitative yield from the readily accessible Diels–Alder adduct **1**[3] via epoxidation in acetone with alkaline hydrogen peroxide.[4] The epoxide ring in **2** was assigned the exo configuration based upon the more favored frontside attack on **1** by hydrogen peroxide anion and by analogy with the formation of II in which the stereochemistry of epoxide ring was well established.[5] Base-promoted reaction of epoxide **2** was performed by treatment with sodium methoxide or 2 M sodium hydroxide in methanol at refluxing or room temperature. The reaction thereby afforded an intractable mixture of two acidic products (**3a** and **4a**). The relative yields of these two products depended mainly upon the quantity of base used as revealed by ^{1}H NMR spectral analysis of the product mixtures via integration of the one-proton singlets at δ 6.38 and 6.18. Analysis of their spectral data, particularly ^{13}C NMR, revealed no indication of the presence of an α,β-unsaturated cyclopentenone moiety in either product. Epoxide **2** does not undergo base-promoted Favorskii ring contraction to give the anticipated I!

Subsequently, it was found that by using less than 2 molar equiv of base, the reaction could be optimized to afford predominantly **3a**. Characterization of **3a** was accomplished by analysis of its spectra and by characterizing its corresponding methyl enol ether **3b** obtained from methylation of **3a** with diazomethane. The structure of **3b** (and thus of **3a**) was established unequivocally by the fact that **3b** also could be synthesized via Diels–Alder cycloaddition of 1,1-dimethoxy-2,3,4,5-tetrachlorocyclopentadiene to 2-methoxy-1,4-benzoquinone[6] (see Experimental Section).

The base-promoted reaction of epoxide **2** using a large excess of base (more than 5 equiv) gave exclusively **4a**. It could also be obtained from **3a** by treatment with base under the same reaction conditions. Conventional spectral data (NMR, IR, and MS) and elemental composition of **4a** indicated that its formation involved only replacement of one of the two methine protons in **3a** by a methoxy group and the carbon skeleton of the molecule remained unchanged. However, structural assignment of **4a**, particularly concerning the location and stereochemistry of the second methoxy group, could not be ascertained from spectral information. Hence, a single-crystal X-ray diffraction analysis was performed on the corresponding methyl enol ether **4b**. Unambiguous proof of the structure of **4a** as depicted in Scheme I was thus obtained. Both **3b** and **4b** could be photocyclized (acetone, Pyrex filter) to hygroscopic cage compounds **5** and **6**, respectively.[7] The cis,endo configuration of the ring junction in **3** and **4** is thereby established.

In earlier studies of the base-promoted reactions of cyclic 2,3-epoxy ketones[8] and 2,3-epoxy 1,4-diketones[2,9] two major types of products that result via (i) a nucleophilic substitution–elimination process and (ii) Favorskii rearrangement have been reported. With bridged enedione epoxides such as II, the reactions uniformly afforded Favorskii ring contraction products, possibly because of relatively less steric interference with approach of the base from the exo side. In the present study, however, we observed that the epoxide **2** reacted by the substitution–elimination pathway to afford the product **3a**. Apparently, base is hindered from approaching the exo side of **2** by the methoxy group at the bridge and thus alternatively acts as a nucleophile to attack the epoxide ring.

The availability of **4b** is noteworthy. Although it is formally a Diels–Alder adduct of 1,1-dimethoxy-2,3,4,5-tetrachlorocyclopentadiene and 2,5-dimethoxy-1,4-benzoquinone, it cannot be prepared in this manner since 2,5-dimethoxy-1,4-benzoquinone is an extremely unreactive dienophile.

Experimental Section

Melting points are not corrected. In all cases, chemical shifts are reported in part per million (δ) downfield from Me_4Si and multiplicity is reported in the parentheses using the standard notation.

5,6,7,8-Tetrachloro-5,8-dimethoxymethano-4a,5,8,8a-tetrahydro-1,4-naphthoquinone Epoxide (2). To a stirring solution of **1** (10 g, 27 mmol) dissolved in acetone (200 mL) was added a freshly prepared ice-cold 20% aqueous $NaHCO_3$ solution (10 mL), followed immediately by a 30% hydrogen peroxide solution (40 mL). After being stirred for an additional 10 min at 0 °C, the reaction mixture was filtered and the solid residue was washed with water and cold acetone. Recrystallization from ethyl acetate yielded colorless crystalline epoxide **2** (10 g, 95%): mp 264–266 °C; IR (KBr) 3040, 1730, 1590, 1300–1090 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 4.23 (s, 2 H, CHO), 3.71 (s, 2 H, methine protons), 3.55 (s, 3 H, OCH_3), 3.45 (s, 3 H, OCH_3); MS, *m/e* (relative intensity) 355 (32), 353 (98), 351 (100), [M^+ – Cl], 317 (7), 315 (13), 255 (9), 253 (7), 209 (17), 207 (17), 181 (8), 179 (7), 107 (12), 59 (50).

Anal. Calcd for $C_{13}H_{10}Cl_4O_5$: C, 40.22; H, 2.60. Found: C, 40.23; H, 2.61.

2-Hydroxy-5,6,7,8-tetrachloro-5,8-dimethoxymethano-4a,5,8,8a-tetrahydro-1,4-naphthoquinone (3a). A solution of sodium methoxide in methanol was freshly prepared by dissolving sodium metal (0.5 g, 22 mg-atom) in dry methanol (100 mL). To this solution was added **2** (5 g, 13 mmol) in one portion. The reaction mixture was stirred and heated under reflux for 1 h. The resulting dark solution was then concentrated in vacuo. The viscous residue thereby obtained was diluted with water and extracted with ether. The aqueous layer was cooled and acidified with concentrated HCl and then extracted with ether. The ethereal solution was dried, filtered, and concentrated in vacuo to afford crude **3a** (3.5 g, 70%) as a viscous oil which was recrystallized from dichloromethane to give needles of **3a**: mp 106–108 °C; IR (KBr) 3500, 3360, 2540, 1680, 1625, 1600 cm^{-1}; ^{1}H NMR (acetone-d_6) δ 6.18 (s, 1 H, HC=), 3.98 (d, J = 8.4 Hz, 1 H, methine proton), 3.80 (d, J = 8.4 Hz, 1 H, methine proton), 3.80 (s, 3 H, OCH_3), 3.69 (s, 3 H, OCH_3); ^{13}C NMR (acetone-d_6) δ 190.2 (s), 188.5 (s), 160.5 (s), 129.9 (s), 128.2 (s), 116.2 (d), 111.3 (s), 77.7 (s), 55.2, 53.7, 52.6, 52.0.

Compound **3a** (3.5 g, 9 mmol) was converted to its methyl ether **3b** by reaction in anhydrous ether with diazomethane generated from (*p*-tosylsulfonyl)methylnitrosamide (4.0 g, 19 mmol). Recrystallization of crude product from dichloromethane–hexane afforded colorless crystals of **3b** (3 g, 83%): mp 206–207 °C; IR (KBr) 1690, 1665, 1600, 1450, 1360, 1185 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 6.00 (s, 1 H, HC=), 3.75 (s, 3 H, OCH_3), 3.64 (br s, 5 H, OCH_3 and methine protons), 3.59 (s, 3 H, OCH_3); ^{13}C NMR ($CDCl_3$) δ 190.0 (s), 186.2 (s), 162.6 (s), 129.8 (s), 128.4 (s), 114.5 (d), 111.1 (s), 77.7 (s), 56.4, 55.2, 54.2, 53.1, 52.1; MS, *m/e* (relative intensity) 371 (6), 369 (33), 367 (98), 365 (100), 331 (9), 329 (15), 317 (4), 315 (5), 305 (4), 303 (9), 301 (11), 279 (8), 257 (16), 255 (42), 253 (44), 209 (18), 207 (19), 69 (67), 59 (48).

Anal. Calcd for $C_{14}H_{12}Cl_4O_5$: C, 41.80; H, 3.01. Found: C, 41.79; H, 2.93.

(3) (a) McBee, E. T.; Diveley, W. R.; Burch, J. E. *J. Am. Chem. Soc.* **1955**, *77*, 385. (b) Marchand, A. P.; Chou, T.-C. *J. Chem. Soc., Perkin Trans. 1* **1973**, 1948.

(4) Alder, K.; Flock, F. H.; Beumling, H. *Chem. Ber.* **1960**, *93*, 1896.

(5) O'Brien, D. F.; Gates, J. W., Jr. *J. Org. Chem.* **1965**, *30*, 2593.

(6) This material was prepared from 2-hydroxy-3-methoxybenzaldehyde by oxidation with H_2O_2 to pyrogallol 1-monomethyl ether, followed by oxidation with $Na_2Cr_2O_7$–H_2SO_4. (a) Surrey, A. R. *Organic Syntheses*; Wiley: New York, 1955; Collect. Vol. III, p 759. (b) Vliet, E. B. *Organic Syntheses*; Wiley: New York, 1951; Collect. Vol. I, p 482.

(7) Cage compounds **5** and **6** could be dehydrated (*p*-TsOH/benzene/azeotropically refluxed) to give compounds which exhibited strong infrared absorption bands at 1690–1740 cm^{-1} but resumed to the hydrate forms upon recrystallization.

(8) Mouk, R. W.; Patel, K. M.; Reusch, W. *Tetrahedron* **1975**, *31*, 13.

(9) (a) Herz, W.; Nair, M. G. *J. Org. Chem.* **1969**, *34*, 4016. (b) Smith, W. B.; Marchand, A. P.; Suri, S. C.; Jin, P.-W. *Ibid.* **1986**, *51*, 3052.

5,6,7,8-Tetrachloro-2-methoxy-5,8-dimethoxymethano-4a,5,8,8a-tetrahydro-1,4-naphthoquinone (3b). A solution of 2-methoxy-*p*-benzoquinone (54 mg)[6] and 1,1-dimethoxy-2,3,4,5-tetrachlorocyclopentadiene (excess) in benzene (5 mL) was stirred and refluxed until the dienophile disappeared as indicated by TLC. The solvent was removed in vacuo and the residue was chromatographed on silica gel (1:5 ethyl acetate–hexane eluent), affording the Diels–Alder cycloadduct **3b** (154 mg, 98%). It was identical in melting point and spectral data with **3b** obtained from the reaction of epoxide **2** with base as described above.

2-Hydroxy-5,6,7,8-tetrachloro-4a-methoxy-5,8-dimethoxymethano-4a,5,8,8a-tetrahydro-1,4-naphthoquinone (4a). To a solution of epoxide **2** (3 g, 7.7 mmol) in methanol (15 mL) was added dropwise an aqueous solution of 2 M NaOH (15 mL) over a period of 30 min. The mixture was stirred at room temperature until the suspended epoxide totally disappeared. Another portion of 2 M NaOH solution (15 mL) was then added, and the brown reaction mixture was stirred overnight. The resulting precipitate was collected by filtration and washed with methanol (5 mL), affording colorless solids (salts of **4**) which were dissolved in water and acidified with dilute HCl solution to give pale yellow solids of **4a** (1.2 g, 37%). The filtrate was concentrated to remove most of methanol and extracted with dichloromethane. The organic layer was washed with water, dried, and concentrated to give an additional portion of oily **4a** (1.1 g, 35%). Recrystallization of crude product from ethyl acetate afforded pure colorless crystalline **4a** (2 g, 70%): mp 216–217 °C; IR (KBr) 3550, 3470, 2500, 1675, 1610, 1450, 1380, 1220, 1185 cm^{-1}; ^1H NMR (acetone-d_6) δ 10.4 (br s, =COH), 6.38 (s, 1 H, vinyl proton), 3.97 (s, 1 H, methine proton), 3.75 (s, 3 H, OCH_3), 3.65 (s, 3 H, OCH_3), 3.40 (s, 3 H, OCH_3); ^{13}C NMR (acetone-d_6) δ 189.7 (s), 188.4 (s), 161.6 (s), 131.4 (s), 129.9 (s), 117.7 (d) 112.5 (s), 85.8 (s), 80.6 (s), 77.4 (s), 59.0 (d), 54.0 (q), 52.4 (q), 51.6 (q); MS (12 eV), *m/e* (relative intensity) 420 (1), 418 (2), 416 (2) [M^+], 385 (18), 383 (56), 381 (57), 353 (4), 351 (10), 349 (11), 347 (6), 345 (10), 287 (18), 283 (16), 266 (14), 264 (23), 262 (18), 185 (11), 143 (8), 105 (100), 75 (8).

Anal. Calcd for $C_{14}H_{12}Cl_4O_6$: C, 40.20; H, 2.89; O, 22.97. Found: C, 40.16; H, 2.95; O, 23.26.

Compound **4a** (0.57 g, 1.4 mmol) was converted to **2,4a-dimethoxy-5,6,7,8-tetrachloro-5,8-dimethoxymethano-4a,5,8,8a-tetrahydro-1,4-naphthoquinone (4b)** in 88% yield by treatment with diazomethane as described above: mp 196–197 °C; IR (KBr) 1685, 1655, 1590, 1450, 1435, 1180, 1165 cm^{-1}; ^1H NMR ($CDCl_3$) δ 6.15 (s, 1 H, =CH), 3.83 (s, 1 H, methine proton), 3.81 (s, 3 H, OCH_3), 3.65 (s, 3 H, OCH_3), 3.56 (s, 3 H, OCH_3), 3.26 (s, 3 H, OCH_3); ^{13}C NMR ($CDCl_3$) 189.3 (s), 186.4 (s), 163.2 (s), 130.5 (s), 129.8 (s), 115.1 (d), 112.0 (s), 85.6 (s), 80.2 (s), 76.9 (s), 59.2 (d), 56.7 (q), 54.4 (q), 52.5 (q), 52.0 (q); MS (12 eV), *m/e* (relative intensity) 434 (5), 432 (11), 430 (9) [M^+], 399 (52), 397 (96), 395 (100), 287 (3), 285 (8), 283 (8), 266 (14), 264 (17), 262 (8), 111 (11), 109 (31), 105 (87), 85 (27), 83 (45).

Anal. Calcd for $C_{15}H_{14}Cl_4O_6$: C, 41.68; H, 3.27; Cl, 32.83. Found: C, 41.74; H, 3.27; Cl, 32.71.

Photocyclization of 3b. Preparation of 5. A solution of **3b** (10 g, 25 mmol) in acetone (500 mL) was irradiated for 2 h with a 450-W Hanovia medium-pressure Hg lamp (Pyrex filter). The reaction mixture was concentrated to leave a solid residue which was recrystallized from chloroform to give colorless crystalline **5** (7.3 g, 73%): mp 148–150 °C; IR (KBr) 3495, 3300, 1440, 1320, 1200 cm^{-1}; ^1H NMR ($CDCl_3$) δ 4.21 (br s, 2 H, disappeared upon addition of D_2O), 3.67 (s, 3 H, OCH_3), 3.62 (s, 3 H, OCH_3), 3.58 (s, 3 H, OCH_3), 3.39 (d, J = 2.5 Hz, 1 H), 3.25 (s, 1 H), 3.21 (d, J = 2.5 Hz, 1 H); ^{13}C NMR (DMSO-d_6) δ 108.7 (s), 106.9 (s), 106.8 (s), 88.2 (s), 78.7 (s), 76.5 (s), 74.4 (s), 73.7 (s), 59.0 (d), 58.9 (d), 55.5 (d), 53.8 (q), 51.0 (q), 50.9 (q); MS (75 eV), *m/e* (relative intensity) 404 (2), 402 (2), 400 (4) [$M^+ - H_2O$], 371 (3), 369 (33), 367 (98), 365 (100), 331 (8), 329 (11), 305 (3), 303 (11), 301 (15), 279 (3), 257 (6), 255 (15), 253 (17), 189 (4), 187 (13), 159 (5), 157 (6), 109 (7), 69 (10), 59 (21).

Anal. Calcd for $C_{14}H_{14}Cl_4O_6$: C, 40.01; H, 3.36; Cl, 33.77. Found: C, 39.79; H, 3.32; Cl, 34.13.

Photocyclization of 4b. Preparation of 6. Compound **4b** (0.33 g) was dissolved in acetone (10 mL) and irradiated for 2 h with a 450-W Hanovia medium-pressure Hg lamp (Pyrex filter). Removal of solvent left semisolids which were recrystallized from ethyl acetate to afford colorless crystals of **6** (0.15 g). Further chromatography of the residue on silica gel (1:3 ethyl acetate–hexane) gave additional product (0.13 g, total yield 81%): mp 180–182 °C; IR (KBr) 3400, 1460, 1450, 1305, 1235, 1200 cm^{-1}; ^1H NMR ($CDCl_3$) δ 4.14 and 4.08 (br s, both disappeared upon addition of D_2O), 3.68 (s, 3 H, OCH_3), 3.64 (s, 3 H, OCH_3), 3.62 (s, 3 H, OCH_3), 3.57 (s, 3 H, OCH_3), 3.55 (s, 1 H), 3.40 (s, 1 H); ^{13}C NMR (acetone-d_6) δ 108.8 (s), 106.3 (s), 106.2 (s), 90.5 (s), 89.1 (s), 78.5 (s), 78.2 (s), 74.6 (s), 70.7 (s), 57.8, 56.5, 54.3, 52.9, 50.9, and 50.5; MS (12 eV), *m/e* (relative intensity) 452 (3), 450 (4), 448 (2) [M^+], 434 (5), 432 (9), 430 (7) [$M^+ - H_2O$], 399 (32), 397 (98), 395 (100), 385 (11), 383 (28), 381 (29), 219 (10), 218 (16), 217 (21), 109 (21), 105 (36).

Anal. Calcd for $C_{15}H_{16}Cl_4O_7$: C, 40.01; H, 3.58; Cl, 31.52. Found: C, 40.11; H, 3.65; Cl, 31.26.

Acknowledgment. We thank the National Science Council of the Republic of China for financial support.

Registry No. 1, 50874-38-9; 2, 114301-84-7; 3a, 114301-85-8; 3b, 114301-86-9; 4a, 114301-87-0; 4b, 114301-88-1; 5, 114301-89-2; 6, 114324-37-7; 2-hydroxy-3-methoxybenzaldehyde, 148-53-8; pyrogallol 1-monomethyl ether, 934-00-9; 1,1-dimethoxy-2,3,4,5-tetrachlorocyclopentadiene, 2207-27-4; 2-methoxy-1,4-benzoquinone, 2880-58-2.

Supplementary Material Available: X-ray crystallographic analysis (crystal data and data collection parameters, structure drawings, bond lengths and angles) for **4b** (6 pages). Ordering information is given on any current masthead page.

Useful Route to Partially Saturated Isocoumarins. Biomimetic Syntheses of Mellein, Ramulosin, and Epiramulosin†

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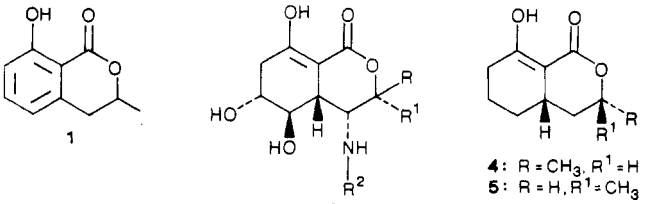
Received July 14, 1987

Many of the substituted 8-hydroxy-3-methyl-3,4-dihydroisocoumarins are naturally occurring substances of ascertained biological activity.[1] The parent system, mellein (**1**), is widespread in nature and was isolated from a number of different microorganisms.[2,3] One recent report indicates, however, its biological importance among the higher organisms as well.[4] More highly oxidized naturally occurring 8-hydroxy-3-methylisocoumarins have already been assessed as having considerable therapeutic value. Actinobolin (**2**)[5] and bactobolin (**3**),[6] for example, are potent broad-spectrum antibiotics and antitumor agents.[7,8] Ramulosin (**4**), the corresponding unsubstituted saturated system, was also identified in nature[9] and was later suggested to be biogenetically related to mellein.[10]



1

2: R = H, R^1 = CH_3, R^2 = L-alanyl
3: R = CH_3, R^1 = $CHCl_2$, R^2 = L-alanyl

4: R = CH_3, R^1 = H
5: R = H, R^1 = CH_3

Many different syntheses of mellein including preparation of natural (–)-mellein[11] have been reported to date.[12] As for ramulosin, only two very recent syntheses are available: one of (±)-ramulosin by Cordova and Snider[13]

† Dedicated to Prof. Ernest L. Eliel on the occasion of his 65th birthday.

0022-3263/88/1953-2837$01.50/0