for $C_{12}H_{26}O_3Si_2$: C, 52.51; H, 9.55. Found: C, 52.28; H, 9.63.

1,3,5,6-Tetramethoxy-8-methylanthraquinone (Erythrolaccin Tetramethyl Ether, 35). A suspension of 3-chloro-5,7-dimethoxy-1,4-naphthoquinone[30] (**34**; 379 mg, 1.50 mmol) and pentadienes (**33a,b**; 549 mg, 2.00 mmol) in dry benzene (12 mL) was refluxed for 21 days (an extra portion of diene (1.00 mmol) was added after 6 days). The residue was heated at 110 °C for 4 h, hydrolyzed, and methylated in the usual way. Purification of the crude product by chromatography (chloroform) and crystallization from methanol gave the tetramethyl ether **35**: 243 mg (47%); mp 155–156 °C (lit.[19] mp 159 °C); mass spectrum, *m/e* 342 (M^+). This substance had physical and spectroscopic characteristics very close to the published data.

2-Chloro-6-hydroxy-5-methoxy-8-methyl-1,4-naphthoquinone (36). Pentadienes **33a,b** (686 mg, 2.50 mmol) were added to a suspension of 2,6-dichlorobenzoquinone (**6**; 354 mg, 2.00 mmol) in ether (30 mL) at −120 °C. After being stirred for 15 min, the mixture was allowed to warm to room temperature (2 h). The solvent was evaporated and the residue refluxed in methanol (20 mL) for 30 min. Naphthoquinone **36** was isolated by chromatography (benzene) and crystallization from CCl_4: 217 mg (43%); mp 154.0–155.5 °C; λ_{max} 218 nm (log ϵ 4.27), 275 (3.82), 298 (sh, 3.65), 420 (3.16); ν_{max} (KBr) 3300 (OH), 1650 (C=O), 1618 and 1587 (C=C) cm^{-1}; NMR ($CDCl_3$) δ 2.69 (3 H, s, 8-CH_3), 3.91 (3 H, s, 5-OCH_3), 6.60 (1 H, br s, 6-OH), 6.81 (1 H, s, 7-H), 7.07 (1 H, s, 3-H); mass spectrum, *m/e* 254/252 (M^+). This substance is somewhat unstable and correct analyses could not be obtained.

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Acknowledgment. We thank Professor V. Snieckus, Dr. A. Stoessl, and Professor R. H. Thomson for samples. Financial support from the Natural Sciences and Engineering Research Council of Canada and the Ministère de l'éducation du Québec as well as the award of NSERC and MEQ scholarships to C.B. is gratefully acknowledged.

Registry No. **1**, 61838-70-8; **2**, 63446-78-6; **5**, 615-93-0; **6**, 697-91-6; **7**, 69119-29-5; **8**, 76665-65-1; **8** methyl ether, 76665-66-2; **9**, 76665-67-3; **10**, 76665-68-4; **11**, 1010-60-2; **12**, 18855-92-0; **13**, 62993-89-9; **14**, 52431-62-6; **15**, 605-32-3; **15** methyl ether, 3274-20-2; **16**, 17241-40-6; **16** acetate, 17241-41-7; **17**, 17241-42-8; **18**, 569-10-8; **18** diacetate, 75312-34-4; **19**, 6866-87-1; **20**, 34425-59-7; **21**, 76665-69-5; **22**, 518-73-0; **23**, 61539-61-5; **24**, 71241-94-6; **24** diacetate, 76665-70-8; **25**, 71241-95-7; **26**, 74272-66-5; **27**, 22225-67-8; **27** diacetate, 22225-68-9; **28**, 76665-71-9; **29**, 76665-72-0; **30**, 6290-50-2; **31**, 76665-73-1; **32**, 76665-74-2; **33a,b**, 76665-75-3; **34**, 57165-99-8; **35**, 801-96-7; **36**, 76665-76-7.

Oxidation of 1,5-Naphthalenediol and Its Methyl Ether: Preparation of Juglone Methyl Ether Monoacetal[1]

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Thallium trinitrate (TTN) oxidizes 1,5-naphthalenediol (**1a**) and its monomethyl ether (**1b**) in good yield to juglone (**2a**) and its methyl ether (**2b**), respectively. Oxidation of **1b** with TTN in a mixture of ethylene glycol and trimethyl orthoformate gave **2c** (the 4-monoacetal of **2b**), which is a promising intermediate in the synthesis of daunomycinone (**3a**) and some of its analogues. This is the first direct oxidation of a para-unsubstituted phenol to a monoacetal of a quinone.

For some years the synthesis of daunomycinone (**3a**; see Chart I), which is convertible into the important anticancer (and cardiotoxic) antibiotic adriamycin (**3b**), has been studied by many research groups.[2] We have been interested in devising a DCAB regioselective route to **3a** and some of its analogues, e.g., **3c** and the tricyclic compound **4**.[3] A logical starting point is the readily available diol **1a**. Two approaches are being examined: in the first the methyl ether **1b**[4,5] is acylated at C_2 via a photo-Fries rearrangement, and then the oxygen at C_4 is introduced;[6] in the second, which is described in this paper, the oxygen at C_4 is introduced in **1** to give a compound in which further substituents on the quinone ring can be introduced regioselectively.

As **2b**, the methyl ether of juglone, does not undergo addition at the double bond regioselectively,[7] we originally planned to reduce β-dihydrojuglone (**5**) regioselectively at C_4[8] and later acylate at C_2. However, in our hands the reduction of **5** was not selective and the route was aban-

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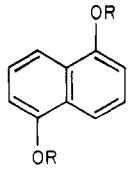
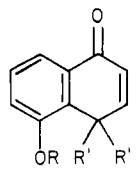
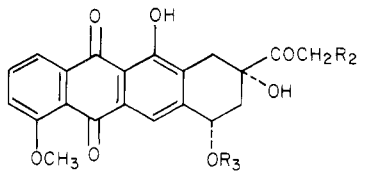
Chart I



1a, R = R′ = H
b, R = H; R′ = CH_3
c, R = R′ = CH_3



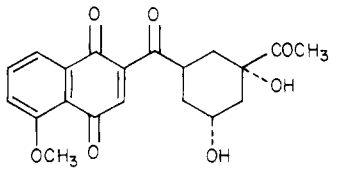
2a, R = H; R′R′ = =O
b, R = CH_3; R′R′ = =O
c, R = CH_3; R′R′ = -OCH_2CH_2O-
d, R = CH_3; R′ = OCH_3
e, R = H; R′R′ = -OCH_2CH_2O-



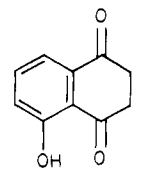
3a, R_1 = OH; R_2 = H; R_3 = H

b, R_1 = R_2 = OH; R_3 =

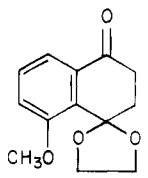
c, R_1 = R_2 = R_3 = H



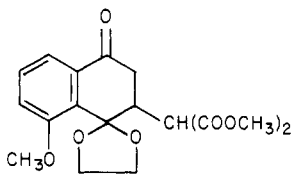
4



5



6



7

doned. During this work we investigated the preparation of **2a** and **2b** in detail.

Preparation of 2a and 2b. Juglone, which has been known for about 100 years, is an important compound (cf. ref 9) and has until recently only been available in poor yield. Despite a claim[10] that the diol **1a** was oxidized in 30–40% yield to **2a** by dichromate, later workers have consistently obtained 15–20%.[11] Both juglone and its methyl ether have been made indirectly from **1a**, through the 4-amino compound which is then oxidized to the quinone[12,13a,b] and also through a Diels–Alder addition of benzoquinone and 3-methoxy-α-pyrone.[13c] Teubner[14] claimed that the oxidation of **1a** with Fremy's salt gave a 49% yield of **2a** and 50% of the *o*-quinone.

Since our work began there have been several reports of the preparation of **2a** and **2b**. Griffiths and his co-workers[15] found that the oxidation of **1a** and **1b** with singlet oxygen gave **2a** (70%) and **2b** (43%), respectively. Oxidation of **1a** with peracetic acid gave **2a** (45–50%).[16] Jackson and Swenton[17] reported that **1c** can be converted into **2b** in 65% yield by two anodic oxidations each of which was followed by treatment with acid. Rapoport and co-workers[18] described a three-step conversion of **1b** into **2b** in good yield. Finally Heinzman and Grunwell report the oxidation of 1,5-diacetoxynaphthalene to 2-bromo-5-acetoxy-1,4-naphthoquinone.[19]

We tried a series of different conditions for the oxidation of **1a** and **1b** using chromate and other reagents.[20] Only marginal improvements (yields up to 30%) resulted from our work with chromate. Most of the other reagents gave juglone, but again the yields were, with the exception of thallium trinitrate (TTN),[22] poor. Starting with the conditions developed for the oxidation of phenols to quinones,[22] we studied in detail the oxidation of **1a** and **1b** with TTN and obtained good results, with yields usually about 60%. Solid supports[23] (Celite, Florisil, and molecular sieves) were also tried; of these, Celite was the most satisfactory, leading to a 72% yield of **1b**. In summary, oxidation with TTN gives a satisfactory one-step preparation of **2a** and **2b** from **1a** and **1b**.

Monoacetal of Juglone Methyl Ether. Monoacetals of quinones, which are useful reagents in synthetic work,[24] are made by selective hydrolysis of the diacetals produced electrolytically from hydroquinone ethers,[25] by electrolysis of *p*-diacetoxybenzene,[26] and by oxidation of *p*-alkoxyphenols.[22,27] McKillop and Taylor[22] suggested that the monoacetals are the last intermediates on the route by which TTN oxidizes a phenol to a quinone but were only able to isolate these acetals when they treated a para-substituted phenol with TTN in a mixture of methanol and trimethyl orthoformate.

We tried these conditions for the oxidation of **1b**. The NMR (with signals for the two acetal methoxyls at δ 3.1

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and an AB quartet for the vinyl hydrogens centered at δ 6.5), IR, and mass spectra of the crude product showed it was impure acetal **2d**. The material reacted with cyanoacetic ester[28] but not with isoprene.[30] Unfortunately, the oxidation of **1b** in methanol/methyl orthoformate never worked cleanly again, giving mainly **2b** with some **2d**. The oxidation was repeated by using ethylene and propylene glycols in place of methanol; the reaction with dry ethylene glycol was studied in detail. The crude material from the reaction always consisted of a mixture of **2b** and **2c**, which could be separated by medium-pressure liquid chromatography on silica gel. However, a more convenient method of purification was to mix the crude reaction mixture with a solution of sodium bisulfite.[31] This led to the isolation of pure **2c** in yields of about 33%. The structure of **2c** follows from its composition and spectra. The methylene protons in the acetal appear as a AA'BB' pattern centered at δ 4.3. Hydrolysis of **2c** gives the quinone **2b**.

The isolation of **2c** supports the ideas put forward by McKillop and Taylor[22] for the mechanism of the conversion of a phenol to a quinone by oxidation with TTN. Presumably the naphthalene monoacetal is easier to isolate than one in the benzene series because the increase in resonance energy in going from the acetal to the quinone is less in the naphthalene than in the benzene series.

The monoacetal **2c** should be a useful intermediate for the synthesis of compounds related to **3** and **4** (cf. ref 24c). Methyl malonate adds smoothly to **2c** to give **7**,[28] thus providing a way of introducing carbon at C_3. Isoprene failed to add.[30] Catalytic hydrogenation of **2c** with rhodium (to avoid hydrogenolysis[32]) gave, in good yield, the dihydro compound **6**, which is a suitable substrate for acylation at C_2.

In attempts to extend the oxidation to make other quinone monoacetals, we found that oxidation of 2,6-dimethylphenol and 1-naphthol with TTN in a mixture of ethylene glycol and trimethyl orthoformate gave only small yields of the corresponding monoacetals. With the diol **1a** the reaction took a different course, and **2e** was not a product. The oxidation of **1b** to **2c** appears to be a special case; possibly the methoxyl group at C_5 provides sufficient steric hindrance to the developing carbonyl at C_4 to slow down the cleavage of the acetal. By contrast, the phenolic group in the monoacetal **2e** could serve as an intramolecular catalyst for cleavage of the acetal (cf. ref 3), hence the difficulty in making this material.

Experimental Section

General Methods. Melting points (uncorrected) were obtained in capillary tubes by using a Mel-Temp apparatus. NMR spectra were recorded on either a Varian Associates A-60D or T-60. All chemical shifts are reported in parts per million (δ) downfield from Me_4Si as the internal standard. IR spectra were recorded on either a Perkin-Elmer 137 or a Beckman Acculab 4 grating spectrophotometer. UV spectra were determined on a Cary 14 recording spectrophotometer or a Hewlett-Packard 8450. Mass spectra were determined on a AEI MS-50 mass spectrometer. Elemental analyses were performed by Micro-Tech Laboratories, Inc.

Preparative TLC utilized Analtech 2013 silica gel of 1-mm thickness. Analytical TLC was done on Eastman 13181 silica gel. Column chromatography used Merck No. 9385 silica gel (400–230 mesh) or basic alumina.

5-Hydroxy-1,4-naphthoquinone (2a). A solution of 1,5-naphthalenediol (**1a**; 0.215 g, 1.34 mmol) in methanol (20 mL) was added dropwise over 10 min to a solution of thallium(III) nitrate trihydrate (1.097 g, 2.47 mmol) in methanol (20 mL) at 0 °C. The mixture was stirred for 10 min more and then filtered. The filtrate was passed through Celite, and the solvent was removed to give dark brown crystals (0.360 g). Chloroform was added to this solid, and the solution was filtered. Evaporation of the chloroform gave juglone (**2a**) as an orange solid [mp 150–151 °C (lit.[33] mp 153–154 °C); 0.137 g (64%)] identified by NMR and IR spectral comparison with an authentic sample.

5-Methoxy-1,4-naphthoquinone (2b). A solution of 5-methoxy-1-naphthol (**2a**; 0.137 g, 0.79 mmol) in methanol (10 mL) was added dropwise over 10 min to thallium trinitrate trihydrate (0.676 g, 1.5 mmol) in methanol (10 mL, 0 °C). The solution was stirred for 20 min and filtered. The filtrate was partitioned between CH_2Cl_2 and saturated brine. The organic phase was dried (Na_2SO_4) and then passed through a short alumina column. The yellow eluent was evaporated to give the naphthoquinone **2b** as orange crystals: mp 183–185 °C (lit.[13b] mp 189 °C); 77 mg (54%); IR ($CHCl_3$) ν_{max} 1655, 1585 cm^{-1}; NMR ($CDCl_3$) δ 4.0 (s, 3 H), 6.8 (s, 2 H), 7.2–7.7 (m, 3 H); UV (CH_3CN) λ 390 nm (ε 2800), 244 (13 600).

Oxidations with TTN/Celite Reagent. (a) Preparation of Reagent. A solution of TTN (9.18 g, dried over P_2O_5 and KOH in vacuo for 3 h) in methanol/trimethyl orthoformate (1:1, 50 mL) was stirred vigorously with acid-washed Celite (24.8 g) for 30 min. The solvent was removed in vacuo at 60–70 °C for 1.5 h. The resulting powder was used soon (within 2–3 days) for best results.

(b) Oxidation of 1b. The TTN/Celite reagent (1.69 g, 1.06 mmol of Tl^{3+}) was added in one portion to a solution of 5-methoxy-1-naphthol (**1b**; 0.092 g, 0.53 mmol) in methylene chloride (40 mL) at 0 °C. The mixture was stirred vigorously for 45 min and then filtered. The filtrate was passed through a short alumina column and evaporated to give yellow crystals of 5-methoxy-1,4-naphthoquinone [**2b**; mp 188–189 °C; 0.071 g (72%)] which was identified by comparison of its spectra and melting point with those of an authentic sample.

(c) Oxidation of 1-Naphthol. 1-Naphthol (0.4 g, 270 mmol) was oxidized by a similar procedure to 1,4-naphthoquinone [mp 120–121 °C (lit. mp 121 °C); 0.195 g (44%)], identified by comparison of its IR and NMR spectra with those of an authentic sample.

(d) Oxidation of 1a. Silica gel (approximately 2.5 g) was added to the filtrate obtained after oxidizing 1,5-naphthalenediol (**1a**) for 30 min by the procedure described in b. The solvent was evaporated and the silica gel placed on a silica gel column. 5-Hydroxy-1,4-naphthoquinone (**2a**; 0.057 g, 12%), identified by spectral comparison, was eluted in petroleum ether/ethyl acetate (85:15).

1,4-Dihydro-4,4-(ethylenedioxy)-5-methoxy-1-oxonaphthalene (2c).[34] Solutions of 5-methoxy-1-naphthol (**2a**; 1.11 g, 6.4 mmol) in trimethyl orthoformate–ethylene glycol (30 mL, 1:1) and thallium(III) nitrate trihydrate (5.69 g, 12.8 mmol) in TMOF/glycol (30 mL) were added simultaneously to stirred TMOF/glycol (40 mL) at −40 °C. The stirring was continued at −40 °C for 5 h. The solution was warmed to room temperature and filtered. Cold saturated sodium bisulfite was added to the filtrate, and this mixture was extracted with ether (4 × 50 mL).[31] The combined extracts were washed thoroughly with saturated sodium chloride and dried (Na_2SO_4). The solution was then reduced under vacuum to approximately 10 mL, and the precipitate was collected. The precipitate in dichloromethane was passed through a short alumina column, and the solvent was removed to give the acetal **2c** as yellow crystals (500 mg, 33%), which on crystallization from petroleum ether/ethyl acetate gave light yellow crystals: mp 152–154 °C; UV (CH_3CN) λ 322 nm (ε 2100), 281 (2700), 215 (16 000); UV (cyclohexane) λ 327 nm (ε

(28) Parker and Kang[29] have reported the addition of malonate to the 1-monoacetal of **2b**.

(29) Parker, K. A.; Kang, S.-K. *J. Org. Chem.* **1980**, *45*, 1218.

(30) Farina and his workers[26] have reported Diels–Alder reactions of monoacetals of benzoquinone. They used dienes that are more reactive than isoprene.

(31) Alternatively, an organic extract of the crude reaction mixture can be shaken with sodium hydrosulfite.

(32) Roy, S. K.; Wheeler, D. M. S. *J. Chem. Soc.* **1963**, 2155.

(33) Bernthsen, A.; Semper, A. *Chem. Ber.* **1885**, *18*, 203.

(34) In this experiment the ethylene glycol should be dried over anhydrous $CuSO_4$ for 2 days, distilled under vacuum, and kept over molecular sieves. TTN should be dried over P_2O_5 and KOH in vacuo for 3–12 h.

2300), 276 (3000), 223 (14 100); IR ($CHCl_3$) λ_{max} 1680, 1644, 1590, 1315, 1110 cm^{-1}; NMR ($CDCl_3$) δ 3.92 (s, 3 H), 4.17–4.47 (m, 4 H), 6.19, 6.36, 6.65, 6.82 (AB q, J = 10 Hz, 2 H), 7.07–7.85 (m, 3 H); mass spectrum, m/e (relative intensity) 232.073 55 (M^+, 100), 204 (20), 187 (50), 76 (80), calcd for $C_{13}H_{12}O_4$ 232.073 39. Anal. Calcd for $C_{13}H_{12}O_4$: C, 67.24; H, 5.17. Found: C, 67.26; H, 5.30.

The monoacetal failed to react with isoprene even in the presence of boron trifluoride etherate.

Hydrolysis of Monoacetal (2c). A solution of the monoacetal (**2c**; 0.043 g, 0.18 mmol) in tetrahydrofuran (10 mL) and 1 N hydrochloric acid (10 mL) was stirred at 60 °C for 12 h. The mixture was poured into saturated sodium hydrogen carbonate which was then extracted with ether several times. The combined ethereal extracts were dried and evaporated to give red crystals (0.047 g) which were dissolved in methylene chloride. This solution was passed through a short column of alumina by using methylene chloride as the eluent. Evaporation of the yellow eluate gave yellow crystals of 5-methoxy-1,4-naphthoquinone [**2b**; mp 188–189 °C; 0.030 g (86%)], identified by spectral comparison with an authentic sample.

4-(Ethylenedioxy)-5-methoxy-1-oxo-1,2,3,4-tetrahydronaphthalene (6). A mixture of the quinone acetal **2c** (1.21 g, 5.2 mmol) in ethyl acetate (50 mL) with 5% Rh/C (62 mg)[32] was shaken with H_2 (1 atm) for 1 h and was then filtered. The filtrate was evaporated to give yellow-brown crystals (1.26 g) that yielded from cyclohexane yellow crystals of **6**: mp 96–96.5 °C; 942 mg (77%); IR ($CHCl_3$) ν_{max} 1700, 1590 cm^{-1}; NMR ($CDCl_3$) δ 2.1–2.9 (m, 4 H), 3.8 (s, 3 H), 4.0–4.3 (m, 4 H), 7.0–7.8 (m, 3 H); UV (cyclohexane) λ 306 nm (ϵ 1640), 247 (3430), 238 (3510), 218 (12 270); mass spectrum, m/e (relative intensity) 234.088 90 (M^+, 60), 206 (25), 189 (10), 178 (100), calcd for $C_{13}H_{14}O_4$ 234.089 20.

Oxidation of 1b with TTN in Methanol/Trimethyl Orthoformate. A solution of 5-methoxy-1-naphthol (**1b**; 0.5 g, 2.9 mmol) in methanol/trimethyl orthoformate (1:1, 30 mL) was added dropwise to a stirred solution of TTN (2.5 g, 5.78 mmol) in methanol/trimethyl orthoformate (20 mL) at −78 °C. The stirred mixture was then allowed to warm to room temperature. Petroleum ether was added, and the solution was filtered. The filtrate was passed through a short alumina column which was eluted with petroleum ether. Evaporation of the petroleum ether solutions gave greenish brown crystals [mp 172–175 °C; 0.68 g (63%)] believed to be compound **2d** on the basis of the spectroscopic properties: IR ($CHCl_3$) ν_{max} 1670, 1630, 1570 cm^{-1}; NMR ($CDCl_3$) δ 3.1 (s, 6 H), 3.9 (s, 3 H), 6.5 (AB q, 2 H), 6.8–7.8 (m, 3 H); mass spectrum, m/e (relative intensity) 234 (M^+, 1.45), 203 (100), 188 (43).

Compound **2d** reacts with ethyl cyanoacetate but not isoprene. Attempts to repeat the preparation of **2d** were not successful.

Reaction of 2c with Methyl Malonate. The quinone monoacetal (**2c**; 0.136 g, 0.59 mmol) in dry methanol (10 mL) was added dropwise to a solution of methyl malonate (0.085 g, 0.64 mmol) and a catalytic amount of sodium methoxide in methanol (10 mL). The mixture was stirred overnight at room temperature and was then partitioned between ether (25 mL) and saturated ammonium chloride (50 mL). The aqueous layer was extracted twice more with ether. The combined ethereal extracts were dried and evaporated in vacuo to give oily white crystals (0.233 g). These crystallized from petroleum ether/ethyl acetate to give the 1,4-adduct **7** as colorless crystals: mp 131–133 °C; 0.173 g (80%); NMR ($CDCl_3$) δ 2.7–4.3 (overlapping m with s at 3.63 and 3.80, 17 H), 6.9–7.6 (m, 3 H); IR ($CHCl_3$) ν_{max} 1755, 1735, 1690 cm^{-1}; mass spectrum, m/e (relative intensity) 364.1157 (57), 305 (32), 233 (40), 178 (100), calcd for $C_{18}H_{20}O_8$ 364.1158.

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Registry No. **1a**, 83-56-7; **1b**, 3588-80-5; **2a**, 481-39-0; **2b**, 4923-61-9; **2c**, 74555-12-7; **2d**, 74555-13-8; **6**, 76741-85-0; **7**, 76741-86-1; TTN, 13746-98-0; 1-naphthol, 90-15-3; 1,4-naphthoquinone, 130-15-4; methyl malonate, 108-59-8.

(35) Meyers, A. I.; Slade, J.; Smith, R. K.; Mihelich, E. D.; Hershenson, F. M.; Liang, C. D. *J. Org. Chem.* **1979**, *44*, 2247.

Aromatization of Arene 1,2-Oxides. 1-(Trimethylsilyl)benzene 1,2-Oxide

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Aromatization of 1-(trimethylsilyl)benzene 1,2-oxide (1) affords a mixture of *o*-(trimethylsilyl)phenol and phenol, the ratio of which is pH dependent. Aromatization of [5-^{2}H]-1-(trimethylsilyl)benzene 1,2-oxide gave the following results. (1) At pH 1.1 or 7 all the deuterium label in *o*-(trimethylsilyl)phenol was para to the hydroxyl group. (2) At pH 1.1 the phenol formed was exclusively [4-^{2}H]phenol, but at pH 7 it was 70–75% [4-^{2}H]phenol and 25–30% [3-^{2}H]phenol. The pathway of the aromatization reaction is discussed.

Our interest in the pathway of aromatization of arene 1,2-oxides derives from their possible role as intermediates in the ortho hydroxylation of aromatic substrates in biological systems.[2] Many arene 1,2-oxides, although not involved in normal metabolism, are nonetheless of interest for further understanding of the effect of the 1-substituent in determining the course of the aromatization reaction. Interest in the influence of the 1-trimethylsilyl substituent in determining the regiospecificity of oxirane ring-opening of arene 1,2-oxides follows from the observations that, although cations β to silicon are stabilized by σ–π hyperconjugation and cations α to silicon are destabilized, acid-catalyzed reactions of (trimethylsilyl)oxiranes normally proceed by cleavage of the C–O bond adjacent to the trimethylsilyl substituent.[3] In a preliminary report[4] we described the preparation of 1-(trimethylsilyl)benzene oxide–oxepin (**1**) and the 2-methyl (**2**) and 4-methyl (**3**)

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