the solution for 48 h at 60 °C. The usual workup gave a crude product (150 mg), which was chromatographed on a silica gel column. Elution with ethyl acetate–benzene (20:1, v/v) gave **4** (145 mg) as reddish orange yellow needles: mp 149–150 °C; IR (KBr) ν 3400, 2950, 1650, 1618, 1603, 1500, 1275, 1030, 905, 820, 610 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.12 (d, J = 1.54 Hz, C_2Me, 3 H), 2.28 (s, 3 H, C_3Me), 6.63 (m, 1 H, H-3), 6.72 (d, 1 H, J = 2.63 Hz, H-5), 6.84 (d, 1 H, J = 7.62 Hz, H-5′), 7.20 (dd, 1 H, J = 7.62 Hz, J = 1.81 Hz, H-6′), 7.30 (d, 1 H, J = 1.81 Hz, H-2′), 7.55 (br, 1 H, ArOH, exchangeable with D_2O); MS, m/e 228.0801 (M^+), calcd for $C_{14}H_{12}O_3$ m/e 228.0787. Anal. Found: C, 73.50; H, 5.14. Calcd for $C_{14}H_{12}O_3$: C, 73.67; H, 5.30.

Reductive Acetylation of 4. The quinoid compound **4** (200 mg) in acetic anhydride (10 mL) was added with zinc powder (100 mg) and anhydrous sodium acetate (100 mg). The mixture was stirred at 60 °C for 12 h. The reaction mixture was poured into ice–water (500 mL) and extracted with CH_2Cl_2 (100 mL × 3). The extracts were dried over Na_2SO_4 and concentrated. The product **5** (90 mg) was isolated by silica gel chromatography eluted with benzene–ethyl acetate (20:1, v/v): viscous oil; ^1H NMR ($CDCl_3$) δ 2.05 (s, 3 H, ArO*Ac*), 2.15 (s, 6 H, ArO*Ac*), 2.23 (s, 3 H, Ar*Me*), 2.28 (s, 3 H, Ar*Me*), 6.88–7.00 (m, 3 H), 7.15 (d, 1 H, J = 1.80 Hz), 7.20 (d, 1 H, J = 1.80 Hz); IR (neat) 2950, 1760, 1610, 1600, 1500, 1475, 1440, 1380 cm^{-1}. Anal. Found: C, 67.21; H, 5.55. Calcd for $C_{20}H_{20}O_6$: C, 67.40; H, 5.66.

Oxidation of 2-Naphthol. 2-Naphthol (290 mg, 2.0 mmol) was dissolved in 10 mL of absolute ethanol and was added to the ethanol solution (80 mL) of cupric chloride (270 mg, 2.0 mmol) under bubbling oxygen. The reaction solution was stirred for 60 °C for 24 h under bubbling oxygen through the solution, and then the solvent was removed under reduced pressure. The usual workup gave a crude product (382 mg), which was chromatographed over silica gel. The first eluate was concentrated in vacuo to give colorless needles, **6** (107 mg). The last eluate was concentrated and cooled to give 263 mg (65%) of orange yellow crystals, **7**: mp 107–108 °C; IR (KBr) 2990, 1685, 1660, 1615, 1595, 1580, 1250, 1215, 1050, 780, 730, 700 cm^{-1}; ^1H NMR ($CDCl_3$); δ 1.52 (t, 3 H, CH_2CH_3), 4.15 (q, 2 H, CH_2CH_3), 6.20 (s, 1 H, H-3), 7.60–8.20 (m, 4 H, H-5,6,7,8); MS, m/e 202.0625, calcd for $C_{12}H_{10}O_3$ m/e 202.0630; MS, m/e 174 (M^+ – CO), 158 (M^+ – OEt + H).

1-Chloro-2-naphthol (**7**): mp 68–69 °C (lit.[14] 70 °C; IR (KBr) ν 3300, 2925, 1625, 1600, 1500, 1430, 1350, 1000, 810 cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.85 (s, 1 H, exchangeable with D_2O), 7.15–8.10 (m, 6 H); this compound was positive to a Beilstein test;[15] MS, m/e 178 (M^+).

Preparation of 4-Methoxy-1,2-naphthoquinone (10). 2-Naphthol (288 mg, 2.0 mmol) and anhydrous cupric chloride (270 mg, 2.0 mmol) in absolute ethanol (50 mL) were used for the synthesis of **10**. The reaction solution was stirred at 60 °C for 35 h under bubbling oxygen through the solution. The reaction mixture was condensed on a rotary evaporator under the reduced pressure. The usual workup gave a crude product (310 mg), which was purified by silica gel chromatography (benzene–ethyl acetate, 10:1) to yield orange yellow crystals (226 mg): mp 152 °C; IR (KBr) 2950, 1682, 1650, 1610, 1598, 1580, 1450, 1250, 730 cm^{-1}; ^1H NMR ($CDCl_3$) δ 388 (s, 3 H), 6.15 (s, 1 H), 7.60–8.20 (m, 4 H); MS, m/e 188 (M^+), 160 (M^+ – CO), 158 (M^+ – OMe + H). Anal. Found: C, 69.98; H, 4.16. Calcd for $C_{11}H_8O_3$: C, 70.20; H, 4.28.

Oxidation of 2,6-Dimethylphenol. 2,6-Dimethylphenol (610 mg, 5.0 mmol) in absolute ethanol (20 mL) and anhydrous cupric chloride (670 mg, 5.0 mmol) in absolute ethanol (80 mL) were added in the same method described for the oxidation of thymol. The reaction solution was stirred at 60 °C for 24 h under bubbling oxygen through the solution. The usual workup and the following silica gel chromatography gave 200 mg of yellow crystals of **8** and 310 mg of reddish brown crystals of **9**.

2,6-Dimethyl-1,4-benzoquinone (**8**): mp 69–70 °C (lit.[16] 72–73 °C); IR (KBr) ν 2980, 1660, 1620, 1445, 1390, 1298, 1185, 920 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.15 (s, 6 H), 7.70 (s, 2 H); ^{13}C NMR ($CDCl_3$) δ 187.55 (C-1), 186.87 (C-4), 145.31 (C-2), 132.89 (C-3), 15.35 (CH_3).

3,3′,5,5′-Tetramethyl-4,4′-diphenoquinone (9): mp 205–207 °C (lit.[17] mp 205.5–208 °C); ^1H NMR ($CDCl_3$) δ 6.41 (s, 4 H), 1.92 (s, 12 H); ^{13}C NMR ($CDCl_3$) δ 187.21 (C-4 and C-4′), 139.10 (C-3 and C-3′), 135.67 (C-1 and C-1′), 129.56 (C-2 and C-2′), 17.07 (4 × CH_3).

3,5,3′,5′-Tetra-*tert*-butyl-4,4′-diphenoquinone (11). 2,6-Ditetra-*tert*-butylphenol (1.018 g, 5.0 mmol) in absolute ethanol (10 mL) and anhydrous cupric chloride (0.672 g, 5.0 mmol) in absolute ethanol were added. The reaction mixture was stirred at 60 °C for 24 h under bubbling oxygen through the solution. The reaction mixture was condensed on a rotary evaporator under reduced pressure. The usual workup and the following silica gel chromatography gave 680 mg (66.8%) of reddish brown crystals of **11**: mp 239–242 °C (lit.[17] mp 242.5–244 °C); IR (KBr) 2950, 1640, 1605, 1570, 1360, 1100, 900 cm^{-1}.

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Registry No. **1a**, 98268-32-7; **1b**, 98268-33-8; **2**, 490-91-5; **3**, 98268-34-9; **4**, 98268-35-0; **5**, 98268-36-1; **6**, 7473-19-0; **7**, 633-99-8; **8**, 527-61-7; **9**, 4906-22-3; **10**, 18916-57-9; **11**, 2455-14-3; thymol, 89-83-8; *o*-cresol, 95-48-7; 2-naphthol, 135-19-3; 2,6-dimethylphenol, 576-26-1; 2,6-di-*tert*-butylphenol, 128-39-2; cupric chloride, 7447-39-4.

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(18) Solvent effect on the oxidation of *o*-cresol (yields of **4**, % are as follows: MeOH (80), EtOH (85), BuOH (34), *i*-PrOH (18). In the solvents acetonitrile, DMF, acetone, and pyridine, **4** was not produced.

Selective Permanganate Oxidation of *cis*- vs. *trans*-2,5-Dihydro-2,5-dimethoxyfuran

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2,5-Dihydro-2,5-dimethoxyfuran, prepared by electrolytic methoxylation of furan[1,2] or by the action of methanolic chlorine or bromine[3,4] on furan is a mixture of cis (**1**) and trans (**2**) isomers, which, on a preparative scale, are difficultly separable by very efficient vacuum fractionation[3,5] (2.5 °C boiling point difference). 2,5-Dihydro-2,5-dimethoxyfuran is most often used in synthesis as a masked 1,4-dialdehyde; therefore, the mixture of stereoisomers is of no great consequence since the stereochemistry of the 2,5-substituents disappears upon hydrolysis.[6,7] For example, the cis 3,4-diols (mixture of **3** and **4**) made from the mixture of cis/trans isomers **1** and **2** upon hydroylsis give *meso*-tartaraldehyde.[7] We needed to synthesize some stereochemically defined model furan compounds derived from **1** and **2** and therefore explored these reactions further.

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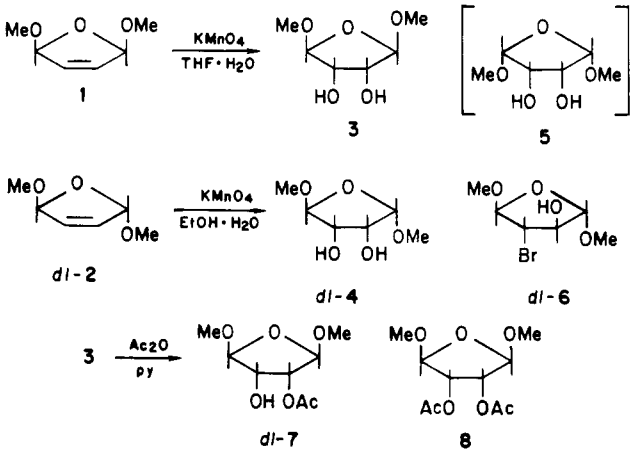
Both 1 and 2 are oxidized by permanganate at comparable rates under various conditions[1,2,5] (primarily aqueous acetone and aqueous ethanol) to give a mixture of the cis diols 3 and 4. However, we have found a dramatically faster rate of permanganate oxidation of 1 (cis) vs. 2 (trans) in aqueous tetrahydrofuran (THF) solvent. This large rate difference permits oxidation of the commercially available 70:30 mixture of 1 and 2 to give exclusively diol 3 from the cis isomer 1 and almost complete recovery of unreacted trans isomer 2.



The structures of 1, 2, 3 and 4 have been established by ^{13}C NMR and analysis of the proton ^{13}C satellite coupling constants.[8] It has been proven[5] that diol 3 (cis-2,5-dimethoxy groups trans to cis-3,4-dihydroxy groups) is the sole product of the permanganate oxidation of pure 1. Evidence that only one isomer was formed was obtained by NMR shift experiments (EuFOD), which revealed no peak separations. We were unable to detect the presence of the all cis diol isomer 5, which, to our knowledge, has not been reported. Oxidation of the recovered trans starting material 2 with permanganate in aqueous ethanol gave *dl-trans*-2,5-dimethoxy-*cis*-3,4-dihydroxyfuran (4).[5,7] With hypobromous acid, 2 was converted to the bromohydrin derivative 6, whose structure is completely analogous to that of the previously established all trans configuration of the 4-bromo-2,5-diethoxy-3-hydroxyfuran homologue.[8]

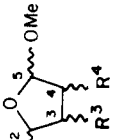
To further substantiate the structural assignment of 3, its monoacetate 7 was prepared. This derivative no longer has the meso configuration of 3, and the NMR signals for all four ring protons and their coupling constants were discernible. Each signal was unambiguously identified by NOE experiments. The NMR data for these compounds are given in Table I.

We have no clear rationalization for the difference in this permanganate oxidation when carried out in aqueous THF vs. aqueous ethanol or aqueous acetone. We observed that in homogeneous anhydrous conditions in THF in the presence of 18-crown-6 under phase-transfer conditions[10] the same selectivity was observed as in aqueous THF.

Experimental Section

Proton magnetic resonance spectra (^1H NMR) were taken at either 100 MHz (Varian XL-100) or at 300 MHz (Nicolet NMC) in Fourier transform mode. Chemical shifts are reported in δ downfield from tetramethylsilane (Me_4Si) as internal standard in $CDCl_3$ solvent. Coupling constants (J) are in hertz (Hz) and

Table I. ^1H NMR Data of Substituted Tetrahydrofurans[a,b]



3, *r*-3,*cis*-4-dihydroxy-*trans*-2,*trans*-5-dimethoxy-
4, *dl*-*r*-3,*cis*-4-dihydroxy-*cis*-2,*trans*-5-dimethoxy-
6, *dl*-*trans*-4-bromo-*r*-3-hydroxy-*trans*-2,*cis*-5-dimethoxy-
7, *cis*-4-acetoxy-*trans*-2,*trans*-5-dimethoxy-*r*-3-hydroxy-
8, *r*-3,*cis*-4-diacetoxy-*trans*-2,*trans*-5-dimethoxy-
9, *r*-3,*cis*-4-dibenzoyloxy-*trans*-2,*trans*-5-dimethoxy-

compd	chem shift, δ							coupling constants (J), Hz
	H-3	H-4	H-2	H-5	2-OCH_3	5-OCH_3	others	
3	——— 4.21 (s)	———	——— 4.95 (s)	———	——— 3.44 (s)	———		
4	4.29 (m)	3.90 (m)	4.96 (d)	4.98 (s)	3.53 (s)	3.39 (s)		$J_{2,3} = 4.92$, $J_{3,OH} = 9.11$, $J_{4,OH} = 8.10$, $J_{3,4} = 4.80$
6	3.93 (m)	4.25 (dm)	5.23 (d)	5.05 (d)	3.48 (s)	3.49 (s)		$J_{2,3} = 2$, $J_{3,4} = 2.5$, $J_{4,5} = 1.65$, $J_{4,OH} = 9.11$
7	5.09 (dd)	4.42 (dd)	5.03 (d)	4.98 (d)	3.46 (s)	3.43 (s)	2.16 (s, 3-OAc)	$J_{2,3} = 1.93$, $J_{3,4} = 4.95$, $J_{4,5} = 2.59$
8	——— 4.98 (d)	———	——— 5.21 (d)	———	——— 3.37 (s)	———	2.01 (s, 3,5-$(OAc)_2$)	$J_{2,3} = 0.8$, $J_{3,4} = 0.8$
9	——— 5.66 (s)	———	——— 5.30 (s)	———	——— 3.53 (s)	———	7.94 (d, ortho H), 7.53 (t, para H), 7.36 (t, meta H)	$J_{ortho,meta} = 8$, $J_{meta,para} = 8$

[a] See Experimental Section for NMR conditions, abbreviations, and notations. [b] See ref 12 for nomenclature.

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splitting patterns are abbreviated as follows: s, singlet; d, doublet; m, multiplet; dd, doublet of doublets; dm, doublet of multiplets. Shift experiments used Resolve-Al EuFOD [Aldrich Chemical Co., tris(6,6,7,7,8,8,8-heptafluoro-2,2-dimethyl-3,5-octanedionato)europium]. Infrared spectra (IR) were taken on a Perkin-Elmer Model 237B grating spectrophotometer; only major band positions (cm^{-1}) are reported. Reactions were routinely followed by thin-layer chromatography (TLC) using Analtech PGBE silica gel GF plates (250 μm) with ethyl acetate–cyclohexane (3:2) as developing solvent; visualization was achieved by spraying with 10% aqueous sulfuric acid followed by heating at 150 °C. Preparative TLC used the same type solvent and plates but with 1000 μm thickness. Uncorrected melting points were determined between cover glasses on an aluminum block microscope hot stage. Solvents were reagent grade and, when necessary, were purified according to standard procedures.[11] Where the simple cis/trans designation of stereochemistry is ambiguous, compounds are named according to the IUPAC system,[12] in which the cis or trans relationship of each substituent is related to the reference substituent designated by *r*.

2,5-Dihydro-2,5-dimethoxyfuran. Our sample of commercially available 2,5-dihydro-2,5-dimethoxyfuran (Aldrich Chemical Co.) was a 70:30 mixture of cis and trans isomers as determined by integration of the 300-MHz NMR spectrum of cis H-2 + H-5 signals vs. the trans H-2 + H-5 signals. The separation of methoxy signals was not sufficient to allow integration: ^{1}H NMR δ 6.07 (s, 2 H, H-3 + H-4), 5.92 (s, ~0.6 H, H-2 + H-5 trans), 5.63 (s, ~1.4 H, H-2 + H-5 cis), 3.44–3.42 (2 s, 6 H, OCH_3). The assignment of cis and trans isomers is unambiguous, based on direct comparison with the spectrum of the pure trans isomer (vide infra).[8]

***r*-3,*cis*-4-Dihydroxy-*trans*-2,*trans*-5-dimethoxytetrahydrofuran[2,5] (3).** To commercial 2,5-dihydro-2,5-dimethoxyfuran (1.0 g, 7.7 mmol, 70:30 **1**–**2**) in tetrahydrofuran (THF, 10 mL) at −10 °C under nitrogen was added dropwise with vigorous stirring $KMnO_4$ (1.22 g, 7.7 mmol; 50% theoretical excess for glycol formation) in H_2O (45 mL) at such a rate that the temperature did not exceed 5 °C. The resulting reaction mixture with the MnO_2 sludge was stirred at room temperature for 12 h; the MnO_2 was removed by filtration and washed with THF (3 × 30 mL). Solvents and unreacted starting material were removed by vacuum distillation [ultimately to 50 °C (0.1 mm of pressure)] leaving an oil, which was dissolved in ethyl acetate, dried ($MgSO_4$), and reconcentrated to give 0.61 g of **3** (48% overall yield, 69% yield of the cis diol based on the 0.70 g of the cis **1** present in the original mixture). This oil crystallized on triturating with ether at −15 °C: mp 34.0–35.5 °C, (lit. mp 36 °C,[3b] 34–36 °C[2]); IR 3425, 1450 cm^{-1}; ^{1}H NMR, cf. Table I. Anal. Calcd for $C_6H_{12}O_5$: C, 43.90; H, 7.37. Found: C, 43.60; H, 7.50. The solvent was removed from the original volatile fraction to give a residue (0.3 g), which was shown by NMR to be **2** uncontaminated with **1**.

Treatment of 10 g of 70:30 mixture of **1** and **2** with 8.1 g of $KMnO_4$ (the theoretical amount to react with both cis and trans isomers in the mixture) in THF–H_2O under the same conditions gave the crystalline diol **3**, 6.9 g (78% based on the 7 g of **1** in the starting mixture), and a recovery of 1.7 g (57%) of unreacted trans isomer **2**. However, oxidation in aqueous ethanol or methanol consumed both cis and trans isomers and gave an inseparable mixture of diols **3** and **4**.

***dl*-*r*-3,*cis*-4-Dihydroxy-*cis*-2,*trans*-5-dimethoxytetrahydrofuran.[5,7] (4).** The trans isomer **2**, recovered from the THF–H_2O oxidation, was oxidized with $KMnO_4$ in aqueous ethanol according to the literature[2,3a] to give **4**, mp 63–64 °C (lit. mp 60–62 °C) in 70% yield.

Monoacetylation of 3. A solution of 0.5 g (3 mmol) of **3**, 0.25 g (2.9 mmol) of acetic anhydride, and 0.4 g of pyridine in 10 mL of $CHCl_3$ was refluxed for 15 h. Volatiles were removed, and the residue was separated by preparative TLC to give three fractions: recovered starting material **3**, R_f 0.32, 140 mg (28%); monoacetate **7**, R_f 0.55, 300 mg (50%); diacetate **8**, R_f 0.77, 80 mg (11%), mp 93–94 °C (lit.[2,5] 97–98 °C).

Benzoylation of 3. A mixture of 1.0 g (6 mmol) of **3**, 3.44 g (24 mmol) of benzoyl chloride, and 4.0 g of pyridine in 30 mL $CHCl_3$ was refluxed for 14 h to give 2.0 g (88%) of white crystals of **9**; recrystallization from ethanol gave 1.8 g; mp 86–87 °C; IR (Nujol mull) 1725, 1750 cm^{-1} (aryl ester). Anal. Calcd for $C_{20}H_{20}O_7$: C, 69.51; H, 5.41. Found: C, 69.35; H, 5.38.

***dl*-*trans*-4-Bromo-*r*-3-hydroxy-*trans*-2,*cis*-5-dimethoxytetrahydrofuran (6).** At 0 °C a 0.4 M HOBr solution (25 mL, 10 mmol) was added to **2** (0.8 g, 6 mmol) and stirred for 12 h at 20 °C. The reaction mixture was neutralized ($NaHCO_3$), saturated with NaCl, and extracted with $CHCl_3$. The organic layer was dried ($MgSO_4$) and the solvent evaporated to leave a colorless syrup of **6**, 1.3 g (93%). Anal. Calcd for $C_6H_{11}BrO_4$: C, 31.73; H, 4.88. Found: C, 31.40; H, 5.10.

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Registry No. 1, 5143-07-7; 2, 98302-20-6; 3, 21462-16-8; 4, 98219-18-2; 6, 98219-22-8; 7, 98219-19-3; 8, 98219-20-6; 9, 98219-21-7; THF, 109-99-9.

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Nucleophilic Substitution Reaction of Alkyl Halide by Anion on a Macroporous Polymer Resin

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In recent years, it has been reported that nucleophilic substitutions of alkyl halides with anions are accelerated when the anionic species are impregnated on inorganic solid supports such as alumina and silica gel.[1,2] Subsequently, Quici and Regen have shown that alumina acts as a triphase catalyst.[3] Ando et al. have pointed out the importance of a trace of water in the system using alumina as a triphase catalyst[4] and have more recently shown that ultrasound accelerates the reaction.[5] On the other hand, many substitution reactions using reagents ionically bound to polymer supports such as basic anion-exchange resins have been well-known.[6] I wish to report that the substitution reaction is accelerated even by a simple impregnation of reagents on macroporous polymer resins such as Amberlite XAD-2, XAD-4, and XAD-7, which have no capability of ion-exchange and no function for phase-transfer catalysis.

$$KX + RY \xrightarrow{XAD} RX + KY$$

X: CN; AcO; I

R: $PhCH_2$; n-C_4H_9; n-C_8H_{17}

Y: Cl; Br; I

Results and Discussion

In a preliminary experiment, the reaction was performed with benzyl chloride and KCN impregnated onto several

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