trometer at 250 MHz. Infrared spectra were recorded on a Perkin-Elmer 983 spectrometer. Elemental analyses were performed by Desert Analytics, Tucson, AZ.

Tetrahydrofuran was purified by distillation from $LiAlH_4$. Tetracyanoethylene (3) was purchased from Aldrich and was purified by two recrystallizations from chlorobenzene followed by two sublimations through a layer of charcoal (125 °C, 0.5 mmHg).

1,1-Dimethoxy-2-bromoethylene (1) was obtained by dehydrobromination of 1,1-dibromo-2,2-dimethoxyethane according to a literature procedure.[10] Dibromo acetal was synthesized by bromination of 2-bromo-1,1-dimethoxyethane with bromine and calcium carbonate in dry diethyl ether.

1,1-Diethoxy-2-bromoethylene (2) was prepared according to a similar procedure as for 1,1-dimethoxy-2-bromoethylene (1).

Methyl 2,2,3,3-Tetracyanocyclopropanecarboxylate (6). TCNE (2.56 g, 20 mmol) was dissolved in 30 mL of tetrahydrofuran. The solution under nitrogen was cooled to −10 °C in dry ice–ethanol–water. Freshly distilled 1,1-dimethoxy-2-bromoethylene (4.18 g, 25 mmol) was added slowly with stirring, and the reaction mixture was stirred for 1 h. Solvent and methyl bromide were then evaporated on a rotary evaporator. The crude product obtained was washed with hexane and purified by column chromatography (column size 4.0 cm/40 cm diameter/height; adsorbent silica gel, 70–230 mesh, 60 Å; eluent ethyl acetate/*n*-hexane, 40/60, v/v). Yield: 3.52 g (88%). Mp = 175–176 °C dec. IR (KBr): 3051, 2261, 1752 cm^{-1}. ^1H NMR (acetone-d_6): δ 3.93 (s, $COOCH_3$), 4.55 (s, CHCOO-). Anal. Calcd for $C_9H_4N_4O_2$: C, 53.89; H, 2.00; N, 27.94. Found: C, 53.66; H, 1.95; N, 27.86.

Ethyl 2,2,3,3-tetracyanocyclopropanecarboxylate (7) was synthesized according to a similar procedure as for cyclopropane 6 from TCNE and 1,1-diethoxy-2-bromoethylene. Yield: 3.62 g (85%). Mp = 158–159 °C dec. IR (KBr): 3057, 2263, 1753 cm^{-1}. ^1H NMR (acetone-d_6): δ 1.33 (t, CH_3), 4.38 (q, $-CH_2-$), 4.52 (s, CHCOO-). ^{13}C-NMR (acetone-d_6): δ 14.2 (s, CH_3), 22.3 (s, >CH<), 38.5 (s, >C<), 64.5 (s, $-OCH_2-$), 108.6 (s, cis-CN), 110.8 (s, trans-CN), 161.7 (s, -COO-). Anal. Calcd for $C_{10}H_6N_4O_2$: C, 56.08; H, 2.82; N, 26.16. Found: C, 56.03; H, 2.67; N, 26.14.

2-(Bromomethylidene)-1,3-dioxolane (8) was obtained by dehydrobromination of 2-(dibromomethyl)-1,3-dioxolane as described by McElvain.[11] 2-(Dibromomethyl)-1,3-dioxolane was prepared by an alcohol exchange between 1,1-dibromo-2,2-diethoxyethane and glycol.[10]

2-(Bromoethylidene)-1,3-dioxolane (9) was synthesized according to a procedure similar to that for 2-(bromomethylidene)-1,3-dioxolane (8) from 2-(β,β-dibromoethyl)-1,3-dioxolane, which was obtained from propionaldehyde by two successive bromination and alcohol exchange reactions. The total yield of the reactions was 66%. Bp = 59–60 °C/0.5 mmHg. This compound crystallized in the refrigerator, approximate mp 14 °C. IR (neat): 2968, 1644 cm^{-1}. ^1H NMR ($CDCl_3$): δ 2.10 (s, CH_3), 4.32 (m, $-CH_2CH_2-$). Anal. Calcd for $C_5H_7BrO_2$: C, 33.52; H, 3.91; Br, 44.69. Found: C, 33.39; H, 3.88; Br, 44.48.

Bromoethyl 2,2,3,3-Tetracyanocyclopropanecarboxylate (10). TCNE (3.84 g, 30 mmol) was dissolved in 30 mL of tetrahydrofuran. The solution was cooled in an ice bath under a nitrogen atmosphere. 2-(Bromomethylidene)-1,3-dioxolane (6.6 g, 40 mmol) was added slowly with stirring under nitrogen, and the reaction mixture was stirred for 2 h. The homopolymer of 2-(bromomethylidene)-1,3-dioxolane was separated by filtration, and the resulting filtrate was concentrated by rotary evaporator. The obtained crude product was washed with hexane and purified by column chromatography. After washing with cold ether, the white crystals were dried under vacuum. Yield: 6.32 g (72%). Mp 152–153 °C. IR (KBr): 3051, 2265, 1746 cm^{-1}. ^1H NMR (acetone-d_6): δ 3.72 (t, CH_2Br), 4.67 (t, $-COOCH_2-$). ^{13}C-NMR (acetone-d_6): δ 22.5 (s, >CH-), 29.2 (s, $-CH_2Br$), 38.2 (s, >C<), 67.9 (s, $-OCH_2-$), 108.5 (s, cis-CN), 110.7 (s, trans-CN), 161.9 (s, -COO-). Anal. Calcd for $C_{10}H_5BrN_4O_2$: C, 40.96; H, 1.70; Br, 27.30; N, 19.11. Found: C, 41.05; H, 1.65; Br, 27.18; N, 19.16.

Bromoethyl 2,2,3,3-tetracyano-3-methylcyclopropanecarboxylate (11) was synthesized according to a procedure similar to that of bromoethyl 2,2,3,3-tetracyanocyclopropanecarboxylate (10) from TCNE (3.84 g, 30 mmol) and 2-(bromoethylidene)-1,3-dioxolane (7.17 g, 40 mmol). Yield: 8.3 g (90%). Mp = 179–180 °C dec. IR (KBr): 2964, 2257, 1745 cm^{-1}. ^1H NMR (DMSO-d_6): δ 1.77 (s, CH_3), 3.71 (t, CH_2Br), 4.54 (t, $-COOCH_2-$). ^{13}C-NMR (acetone-d_6): δ 16.4 (s, CH_3), 29.2 (s, CH_2Br), 43.8 (s, >C<), 68.2 (s, $-OCH_2$), 109.1 (s, cis-CN), 110.2 (s, trans-CN), 163.3 (s, -COO-). Anal. Calcd for $C_{11}H_7BrN_4O_2$: C, 42.99; H, 2.28; Br, 26.05; N, 18.24. Found: C, 43.12; H, 2.26; Br, 26.00; N, 18.14.

Acknowledgment. We are deeply indebted to the Office of Naval Research for financial support of this work and to Dr. Anne Padias for helpful discussions.

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Isobatzellines A, B, C, and D. Cytotoxic and Antifungal Pyrroloquinoline Alkaloids from the Marine Sponge *Batzella* sp.

Hao H. Sun,*,† Shinichi Sakemi,‡ Neal Burres,§ and Peter McCarthy

Division of Biomedical Marine Research, Harbor Branch Oceanographic Institution, Inc., Ft. Pierce, Florida 34946

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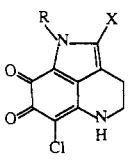
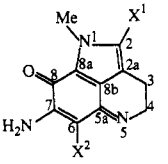
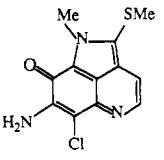
Recently we reported the isolation of three highly functionalized pyrroloquinoline alkaloids, batzellines A (1), B (2), and C (3), from the deep water Caribbean sponge







1 R = Me, X = SMe
2 R = H, X = SMe
3 R = Me, X = H

4 X^1 = SMe, X^2 = Cl
5 X^1 = SMe, X^2 = H
6 X^1 = H, X^2 = Cl
8 X^1 = H, X^2 = H

7

Batzella sp. (family Esperiopsidae, order Poecilosclerida). The structure of batzelline A was secured by X-ray analysis.[1] Further search for bioactive agents from *Batzella* has resulted in the discovery of four structurally related pyrroloquinolines, which we have named isobatzellines A (4), B (5), C (6), and D (7). The isobatzellines were found to exhibit in vitro cytotoxicity against P388 leukemia cell and moderate antifungal activity against *Candida albicans*.[2] The structures of the isobatzellines are described here and are based on spectral comparison with batzelline A and chemical interconversion.

Deep water sponge samples collected off the Grand Bahama Islands in June, 1984, and November, 1987, were frozen immediately and subsequently extracted with methanol–chloroform (2:1). Solvent partitioning of the extracts and centrifugal countercurrent chromatography of the resulting fractions yielded isobatzellines A (4), B (5), C (6), and D (7). The molecular formula of isobatzelline A (4) was determined to be $C_{12}H_{12}N_3OSCl$ by high-resolution FAB mass spectrometry. The ^1H and ^{13}C NMR data of 4, which are summarized in Table I, are similar to those of batzelline A. The presence of *S*-methyl, *N*-methyl, *N*-methylene, and an allylic methylene group including eight nonprotonated sp^2 carbons in 4 is consistent with the structural features of 1, but 4 contains one more nitrogen

† Present address: Sterling Drug Inc., 25 Great Valley Parkway, Malvern, PA 19355.
‡ Present address: Pfizer Central Research, Nagoya, 5-2 Taketoyo, Chita, Aichi 470-23, Japan.
§ Present address: Abbott Laboratories, Abbott Park, IL 60064.

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Table I. ^{13}C and ^{1}H NMR Assignments of Isobatzellines A (4), B (5), C (6), and D (7)[a]

	4		5		6		7	
atom	^{13}C, δ	^{1}H, δ (m, J (Hz))	^{13}C, δ	^{1}H, δ (m, J (Hz))	^{13}C, δ	^{1}H, δ (m, J (Hz))	^{13}C, δ	^{1}H, δ (m, J (Hz))
2	137.1		136.4		132.4	7.13 (s)	138.6	
2a	121.6		122.8		120.3		118.8	
3	19.3	2.99 (t, 7.3)	19.6	2.84 (t, 7.5)	19.2	2.96 (t, 7.5)	112.0	7.64 (d, 5.8)
4	43.8	3.95 (t, 7.3)	43.6	3.89 (t, 7.5)	44.3	3.91 (t, 7.5)	141.4	8.34 (d, 5.8)
5a	154.0		158.7[b]		155.1[b]		146.4[b]	
6	94.1		88.0	5.69 (s)	93.9		103.9	
7	152.5		157.3[b]		153.5[b]		143.8[b]	
8	165.8		168.8		166.8		163.3	
8a	124.6		125.9[c]		124.1[c]		125.5[c]	
8b	124.3		124.2[c]		122.8[c]		116.5[c]	
1-Me	33.9	3.93 (s)	33.7	3.96 (s)	36.6	3.93 (s)	34.5	4.22 (s)
2-SMe	18.5	2.39 (s)	18.5	2.33 (s)			17.5	2.79 (s)
7-NH_2								6.44 (s)

[a] 4–6, recorded in 1:1 $CDCl_3$–CD_3OD; 7, recorded in $(CD_3)_2SO$. [b,c] Interchangeable within column.

and one less oxygen atom. Reductive dechlorination and desulfurization by catalytic hydrogenation yielded the product 8, having a molecular formula of $C_{11}H_{11}N_3O$. This led to the supposition that one of the *o*-quinone oxygens in the pyrroloquinoline ring of 1 is replaced by a nitrogen in 4. Treatment of 4 with sodium nitrite in acidic solution resulted in the formation of 1 via the diazonium salt.[3]

Isobatzelline A (4) was noted to convert to isobatzelline D (7) on an EM HPTLC NH_2 $F_{254}S$ plate within several hours. Isobatzelline D (7) analyzed for $C_{12}H_{10}N_3OSCl$ by high-resolution EIMS. The ^{1}H and ^{13}C NMR spectra of (7) (Table I) revealed the absence of two methylenes and the presence of two olefinic CH carbons in addition to eight nonprotonated sp^2 carbons in 7, which suggested that 4 aromatized to 7. This conversion was also achieved by refluxing 4 in dioxane with 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ).[4] The 2D HETCOR NMR spectrum of 7 showed that a D_2O exchangeable NH_2 resonance observed at δ 6.44, not observable in either the ^{1}H NMR spectrum of 4 or 8, presumably due to tautomerism,[5] was long-range coupled to two carbon resonances observed at δ 163.3 and 103.9, assigned respectively to the carbonyl carbon and the chlorine-bearing olefinic carbon. These data indicated that the amino group was most likely attached to C7, and, therefore, isobatzellines A and D are represented by structures 4 and 7, respectively.

High-resolution EI mass spectrometry established the molecular formulas of isobatzellines B (5) and C (6) as $C_{12}H_{13}N_3OS$ and $C_{11}H_{10}N_3OCl$, respectively. The absence of the chlorine in 5 and the *S*-methyl in 6 was evident from their respective ^{1}H and ^{13}C NMR spectra (Table I). On hydrogenation, both compounds were reduced to 8, thereby establishing the structures of 5 and 6.

The isobatzellines possess the same pyrrolo[4,3,2-*de*] quinoline ring system as the batzellines but contain an aminoiminoquinone moiety[6,7] that is different from the aminoquinone moiety in the batzellines. It is interesting to note that only the isobatzellines, not the batzellines, are cytotoxic and moderately antifungal.

Experimental Section

Spectra were determined at 360 MHz for ^{1}H NMR and 90 MHz for ^{13}C NMR. A Parr hydrogenation apparatus was used for catalytic hydrogenation.

Isolation of the Isobatzellines. The sponge *Batzella* sp. was collected on June 3, 1984, by using the Johnson-Sea-Link II manned submersible between Freeport and West End in the Grand Bahamas Islands at a depth of 120 m. A taxonomic voucher specimen is deposited at Harbor Branch Oceanographic Institution, Inc., Indian River Coastal Zone Museum, catalog number 003:00050. The sample (68 g) was stored frozen and then extracted with 2:1 MeOH–$CHCl_3$ (200 mL × 4). The extract was concentrated to an aqueous suspension in vacuo, followed by addition of water (150 mL) and extraction with ethyl acetate (100 mL × 4). The resulting aqueous layer was lyophilized and extracted with MeOH (200 mL). The methanol extract (2.7 g) was further partitioned using 13:7:8 $CHCl_3$–MeOH–H_2O. Centrifugal countercurrent chromatography of the lower layer using 2:7:6:3 heptane–$CHCl_3$–MeOH–H_2O (lower phase stationary) yielded isobatzellines A (4, 12 mg, 0.018% of the wet weight sponge), B (5, 19 mg, 0.028%), and C (6, 14 mg, 0.021%). Subsequent centrifugal countercurrent chromatography of the stationary phase using 4:7:4:3 heptane–EtOAc–MeOH–H_2O (lower phase stationary) afforded isobatzelline D (7, 17 mg, 0.025%). All isobatzellines were obtained as brown solids, which stayed as solids at 250 °C but released gaseous material above 200 °C.

A second collection (817 g) of *Batzella* sp. was made on November 14, 1987, from Mangrove Island off West End, Grand Bahamas Islands, at a depth of 130 m, by using the Johnson-Sea-Link II manned submersible (voucher specimen catalog number 003:00051). A portion of the sponge (740 g) was also extracted with 2:1 MeOH–$CHCl_3$ (1 L × 3). Removal of solvents in vacuo gave 42 g of concentrated extract, which was partitioned by using 15:2:15 $CHCl_3$–MeOH–H_2O (1.6 L). Repeated centrifugal countercurrent chromatography of a portion (3.7 g) of the upper layer (32 g) yielded 4 (70 mg, 0.082%), 5 (20 mg, 0.024%), 6 (49 mg, 0.058%), and 7 (31 mg, 0.037%).

Isobatzelline A (4): a brown solid; HRFABMS MH^+ 282.0482 (calcd for $C_{12}H_{13}ON_3{}^{35}ClS$, Δ mmu 1.4); LREIMS 283/281 (42/100), 266/264 (29/53), 248 (15), 233 (23), 221 (13), 205 (21), 191 (6), 180 (6), and 161 (5); UV (MeOH) 262 (ε 15 500), 342 (9000), and 430 nm (3100); IR (KBr) 3460, 3340, 2930, 1640, 1580, 1560, 1435, 1400, 1380, 1350, 1295, 1070, and 965 cm^{-1}; ^{1}H and ^{13}C NMR, Table I.

Isobatzelline B (5): a reddish brown solid; HREIMS M^+ 247.0780 (calcd for $C_{12}H_{13}ON_3S$, Δ mmu 0.1); LREIMS 247 (100), 231 (24), 214 (15), 199 (35), 187 (13), 174 (10), and 149 (10); UV (MeOH) 264 (ε 14 200) 362 (7000), and 402 (5000); IR (KBr) 3430, 3080, 2930, 1655, 1600, 1525, 1390, 1300, 1250, 1140, 1050, 956, and 845 cm^{-1}; ^{1}H and ^{13}C NMR, Table I.

Isobatzelline C (6): a greenish brown solid; HREIMS M^+ 235.0510 (calcd for $C_{11}H_{10}ON_3{}^{35}Cl$, Δ mmu 0.2); LREIMS 237/235

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(2) IC_{50} against P388 (μg/mL): 4, 0.42; 5, 2.6; 6, 12.6; 7, 20. MIC against *Candida albicans* (μg/mL): 4, 3.1; 5, 25; 6, 50; 7, 25.

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(36/100), 222 (8), 208 (27), 201 (84), 173 (34), 145 (45), and 129 (27); UV (MeOH) 244 (ε 9700), 344 (5900), 394 (2600); IR (KBr) 3360, 3050, 1670, 1600, 1420, 1340, 1320, 1200, 1135, 835, 804, and 720 cm^{-1}; ^1H and ^{13}C NMR, Table I.

Isobatzelline D (7): a reddish brown solid; HREIMS M^+ 279.0230 (calcd for $C_{12}H_{10}N_3OS^{35}Cl$, Δ mmu 0.3); LREIMS 281/279 (38/100), 266/264 (25/72), 246 (18), 232 (23), 220 (11), 196 (11), 186 (7), 173 (9), 159 (6); IR (KBr) 3450, 3310, 2930, 1640, 1560, 1545, 1485, 1440, 1400, 1340, 1320, 1290, 1250, 1145, 1080, and 960 cm^{-1}; UV (MeOH) 239 (ε 33 900), 263 (25 000), and 439 nm (25 200); ^1H and ^{13}C NMR, Table I.

Catalytic Hydrogenation of 4 to Product 8. A suspension of 25 mg of isobatzelline A (4) and 10 mg of 10% Pd/C in 10 mL of MeOH was agitated under 30 psig hydrogen atmosphere at room temperature for 16 h. After removal of the catalyst and the solvent, the reddish brown solid was separated on a Hibar NH_2 HPLC column to give 6 mg (34%) of 8, as a greenish brown solid: mp dec >200 °C; HRFABMS MH^+ 202.0994 (calcd for $C_{11}H_{12}ON_3$, Δ 1.4 mmu); LREIMS 201 (100), 174 (34), 145 (16), 119 (11), 105 (29), 91 (39), and 77 (17); UV (MeOH) 244 (ε 14 200), 346 (8500), and 392 nm (4000); IR (KBr) 3400, 2930, 1660, 1605, 1430, 1360, 1350, 1320, 1255, 1205, 1100, 835, and 800 cm^{-1}; ^1H NMR (1:2 $CDCl_3$-CD_3OD) δ 2.86 (2 H, t, J = 7.6 Hz), 3.82 (2 H, t, J = 7.6 Hz), 3.94 (3 H, s), 5.63 (1 H, s), and 7.00 (1 H, s); ^{13}C NMR (CD_3OD) δ 19.5, 36.5, 43.9, 87.9, 119.8, 123.9, 125.1, 131.9, 158.0, 159.6, and 169.6.

Conversion of 4 to 1. An aqueous $NaNO_2$ solution (100 mg in 5 mL) was added dropwise into an ice-chilled solution containing 20 mg of 4 in glacial acetic acid (2 mL) and dioxane (1 mL). After stirring at 0 °C for 2 h, 3 mL of 1 N HCl was added, and the solution was stirred at room temperature overnight and then extracted with $CHCl_3$-MeOH (1:1, 10 mL × 2). The extract was fractionated on a Superco LC-NH_2 cartridge with 1% MeOH-$CHCl_3$ and subsequently purified on a HPLC LiChrosorb-NH_2 column with 3% MeOH-$CHCl_3$ to give 1 (4 mg, 20%), along with 4 (2 mg).

Conversion of 4 to 7. A solution of 10 mg of 4 in 5 mL of dioxane was stirred with 10 mg of DDQ at room temperature overnight. The resulting product was purified by HPLC (LiChrosorb NH_2, 3% MeOH-$CHCl_3$) to give 6 mg (60%) of 7.

Conversion of 5 and 6 to 8. Isobatzellines B (5, 15 mg) and C (6, 30 mg) were reduced to 8 (8 mg (65%) and 19 mg (74%), respectively) under the conditions already described above for the conversion of 4 to 8.

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Supplementary Material Available: ^{13}C NMR spectra for compounds 4-8 (5 pages). Ordering information is given on any current masthead page.

Transalkylation Reactions of 4,4'-(1-Methylethylidene)bisphenol with Diphenyl Ether

Zhi Yuan Wang, Nurit Berard, Irena Wisniewska, and Allan S. Hay*

Chemistry Department, McGill University, 801 Sherbrooke St. W., Montreal, Quebec, Canada H3A 2K6

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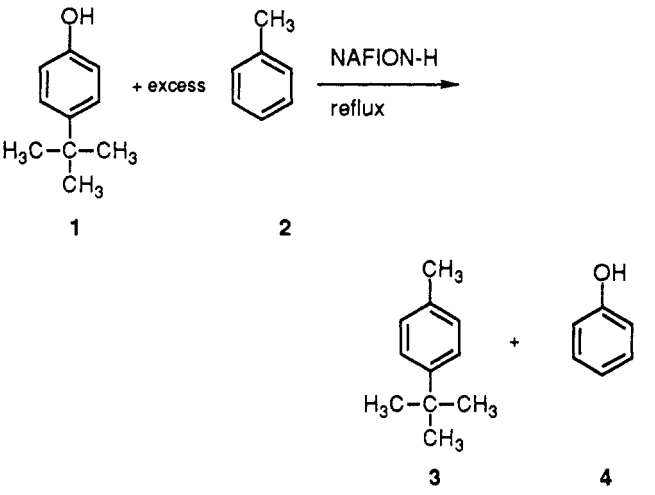
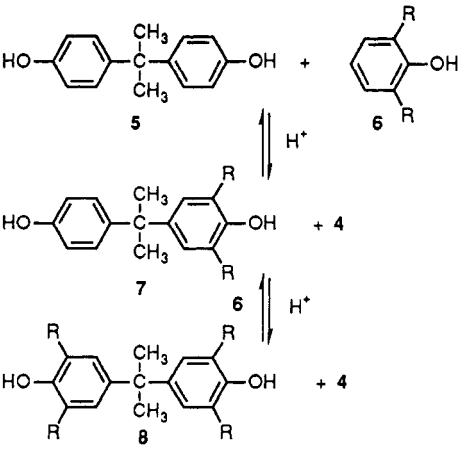
Olah and others[1-3] have recently described the use of Nafion-H resins as catalysts in the de-*tert*-butylation of aromatic compounds. Phenol and biphenols containing *tert*-butyl groups, in the presence of Nafion, undergo an almost quantitative transalkylation reaction with toluene, or biphenyl, to yield the unsubstituted phenol or biphenol along with 4-*tert*-butyltoluene or 4-*tert*-butylbiphenyl as coproducts. For example, 4-*tert*-butylphenol (1) and toluene (2) yield phenol (4; 96%) and 4-*tert*-butyltoluene (3; 96%) as products (Scheme I).

Transalkylation reactions between phenols and bisphenols have been reported previously.[4-6] Mark[5] found that bisphenols such as 4,4'-(1-methylethylidene)bisphenol

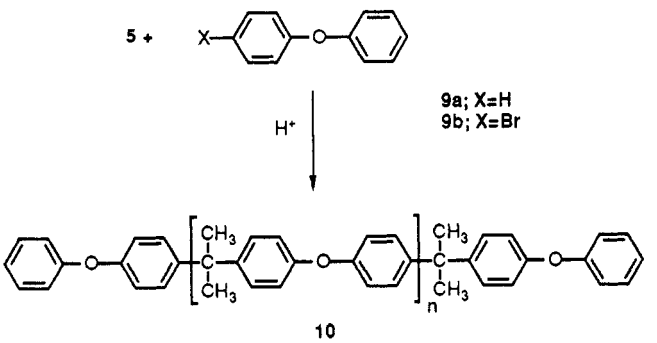
Scheme I



Scheme II



Scheme III



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