o-(α-Benzotriazolylalkyl)phenols: Novel Precursors for the Preparation of 1,1-Bis(2-hydroxyaryl)alkanes

Alan R. Katritzky,* Zhongxing Zhang, Hengyuan Lang, and Xiangfu Lan

Center for Heterocyclic Compounds, Department of Chemistry, University of Florida, Gainesville, Florida 32611-7200

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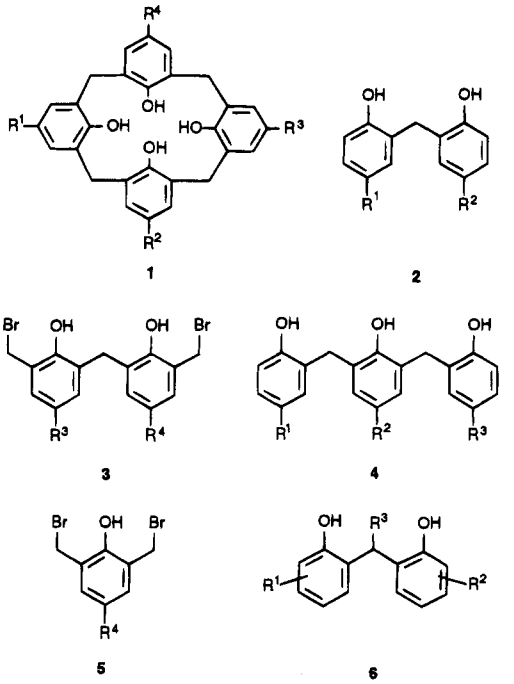
Reactions of *o*-(α-benzotriazolylalkyl)phenols with a variety of substituted phenols and naphthols in the presence of a base provide efficient access to symmetrical and more importantly to unsymmetrical 1,1-bis(2-hydroxyaryl)alkanes.

Introduction

Calixarenes, a versatile class of macrocyclic compounds, have received increasing attention in the last 20 years as illustrated by the recent publication of two monographs in this area.[1,2] The synthesis, characterization, and study of conformations and properties of calixarenes as well as their potential applications in industry are among the topics intensively investigated. Calixarenes represent acceptor molecules for metal cations of widely varying size and can also act as enzyme mimics.[1–5]

Symmetrical calixarenes can be synthesized by a one-pot reaction of phenol with formaldehyde. Neutral, base-induced, or acid-catalyzed conditions can be used. Multistep processes are usually required for the synthesis of unsymmetrical calixarenes. Unsymmetrical calixarenes having at least three different substituents in the phenolic units or a substituent in the *meta* position of a phenolic unit are inherently chiral and are of particular interest for study of their properties, of their stereochemistry, and their potential use as chiral molecular acceptors. Two general procedures for the synthesis of calix[4]arenes of type **1** are available.[1,2] A [2 + 2] convergent process involves cyclization of 4,4′-disubstituted 2,2′-methylenebisphenols **2** with bis(bromomethyl)-*o*,*o*′-methylenebisphenols **3**. A [3 + 1] process involves cyclization of a trimer **4** with the corresponding bis(bromomethyl)ated phenol **5**.

Obviously, 1,1-bis(2-hydroxyaryl)methanes (or *o*,*o*′-methylenebisphenols) are important precursors to calixarenes. Preparation of *o*,*o*′-methylenebisphenols **6** has been previously effected by condensing *o*-(hydroxymethyl)phenol with an excess of phenols at high temperature (160–170 °C) in alkali.[6] Novolak resins are formed as byproducts in the reaction. An alternate method[7,8] for the preparation of *o*,*o*′-methylenebisphenols **6** is to react



2-hydroxybenzaldehyde first with a Grignard reagent and then with a phenol. These routes suffer from multistage procedures or the relatively difficult preparation of *o*-(hydroxymethyl)phenols: direct condensations of phenols and formaldehyde depend strongly on the reaction conditions and starting phenols and often lead to complicated mixtures of symmetrical *o*,*o*′-methylenebisphenols, 2,6-bis(hydroxymethyl)phenols, and oligomers.

Other specialized methods for the preparation of this type of compounds include the reaction of 2-bromo-6-(hydroxymethyl)-4-phenylphenol with a phenol in the presence of concentrated hydrochloric acid,[3] reaction of 2-bromo-6-(bromomethyl)-4-phenylphenol with a 4-*tert*-butylphenol,[3] reaction of a phenol with the quarternary salt of a Mannich base in the presence of KOH,[10] and treatment of a phenol with propionaldehyde diethyl acetal.[11]

We now report a general, yet simple procedure for the synthesis of 1,1-bis(hydroxyaryl)alkanes from the corresponding phenols. We previously reported the use of benzotriazole as a synthetic auxiliary[12] and showed that

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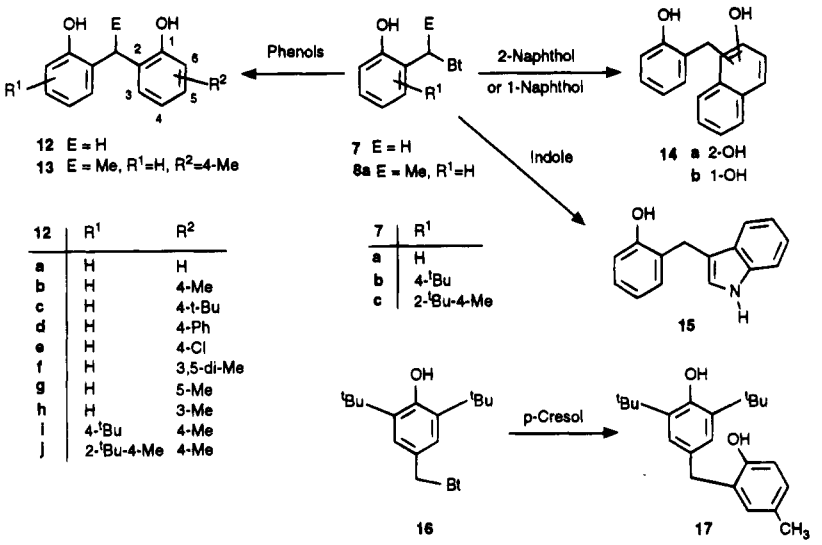
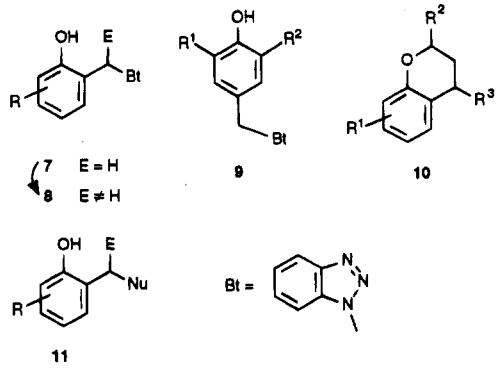
Scheme 1



phenols readily condense with 1-(hydroxymethyl)benzotriazole to give *o*-(benzotriazolylmethyl)phenols **7** (2,6-disubstituted phenols give *p*-(benzotriazolylmethyl)-phenols **9**).[13] These derivatives can be elaborated at the benzylic carbon via lithiation and the anion can be quenched with various electrophiles to give **8**. We have shown that the parent **7** as well as the derived *o*-(benzotriazolylalkyl)phenols **8** are precursors to *o*-quinone methides: they are trapped by ethyl vinyl ether and 1-vinyl-2-pyrrolidinone to give chroman derivatives **10**,[14] and they react with Grignard reagents or $LiAlH_4$ to give *o*-alkylphenols **11** (Nu = alkyl or H, respectively).[13] More recently, we found that they react with a variety of nucleophiles including thiols, alcohols, amines, amides, and active methylene compounds through Michael additions to give *o*-substituted phenols **11** (Nu = N, O, S, and C nucleophiles).[15] We now report that compounds **7** and **8** also react readily with phenols in the presence of a base to form the 1,1-bis(2-hydroxyaryl)alkanes.



Results and Discussion

o-(Benzotriazolylmethyl)phenols **7** were reacted with phenols to afford *o,o'*-methylenebisphenols **12** in moderate to good yields. Thus, heating a mixture of *o*-(benzotriazol-1-ylmethyl)phenol (**7a**) with phenol in *t*-BuOH in the presence of 2 equiv of *t*-BuONa under reflux gave exclusively the *ortho*-substituted product **12a** in 40% yield. No product from the *para* alkylation was isolated or detected by NMR. The reaction presumably involves the initial elimination of sodium benzotriazole to give the *o*-quinone methide which is attacked by sodium phenoxide; the relatively low yield can be attributed to the high reactivity of the *o*-quinone methide leading to the formation of byproducts. The phenomenon of exclusive *ortho* substitution of a phenol under basic conditions is recognized and has been studied for decades.[1] The byproduct, benzotriazole, was easily removed during column chromatography.

Since the precursors to calixarenes should have two open *ortho* positions, we examined examples with substituents in the *para* or the *meta* positions. Methyl, *tert*-butyl, or phenyl substituents in the phenol component under similar conditions all gave the expected *o,o'*-methylenebisphenols **12** in 39–57% yields. A weak electron-withdrawing chloro group at the *para* position also reacted with **7a** to afford **12e** in 27% yield, the low yield being due to the low reactivity of the ring.

When symmetrical 3,5-dimethylphenol reacted with **7a**, one product **12f** was obtained in 55% yield. However, use of mono-*meta*-substituted phenol gave a mixture of two isomers. Thus, heating a mixture of **8a** and *m*-cresol in *i*-PrOH/*i*-PrONa for 40 h afforded a mixture of compounds **12g** and **12h** in a total yield of 51% with a ratio of *ca.* 1:1 according to the NMR spectra. Steric hindrance does not seem to be an important factor in the regiochemical selectivity.

The methodology was extended to naphthols. Thus, compound **7a** reacted with 2-naphthol at the 1-position and with 1-naphthol at the 2-position to form products **14a–b**, in 65% and 58% yields, respectively. The reactions of *o*-(benzotriazol-1-ylmethyl)phenol possessing a ring substituent were also investigated. Heating a mixture of **7b** with *p*-cresol under the above-described conditions afforded compound **12i** in 62% yield. **12j** was similarly obtained in 53% yield.

Reactions of *o*-(α-benzotriazolylalkyl)phenols **8a**, substituted in the methylene group and available from **7a** via lithiation and quenching with electrophiles,[13] were also examined with phenols. Thus, heating a mixture of **8a** and 4-methylphenol in BuOH in the presence of

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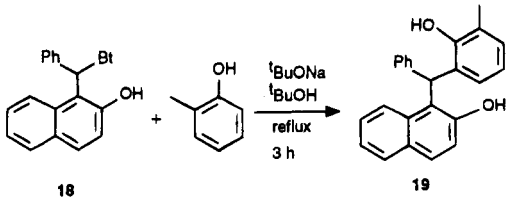
Table 1. Preparation of Compounds 12a–j, 13, 14a,b, 15, and 19

						lit. mp (°C) or calcd/found		
prodt	react.	time (h)	yield (%)	mp (°C)	purif[a]	C	H	N
12a	**7a**	72	40	117–18	10:1		118–119.5[16]	
12b	**7a**	40	57	98–99	10:1		96–97[6]	
12c	**7a**	40	39	89–90	10:1		91–92[6]	
12d	**7a**	40	40	144–45	10:1		143–46[6]	
12e	**7a**	40	27	122–23	10:1	66.53/66.69	4.72/4.70	
12f[f]	**7a**	40	55	139–40	10:1		139–40[6]	
12g (12h)	**7a**	40	51	97–99	10:1		132–33 (**12g**), 114–15 (**12h**)[6]	
12i	**7b**	40	62	94.5–5.5	10:1	79.96/79.76	8.20/8.34	
12j	**7c**	38	53	116–117	10:1	80.24/80.34	8.51/8.58	
13	**8a**	4	43	oil	8:1	78.92/78.73	7.06/7.20	
14a	**7a**	40	65	164–65	10:1	81.58/81.71	5.64/5.70	
14b	**7a**	32	56	120–21	8:1	81.58/81.31	5.64/5.62	
15	**7a**	36	58	114–15	10:1	80.69/80.90	5.87/5.83	6.27/6.28
19	**18**	3	59	158–59	10:1	84.68/84.97	5.92/6.21	

[a] Eluent for chromatography, ratio of hexane:ethyl acetate.

Scheme 2



t-BuOK gave the expected **13** in 43% yield. When 4-(benzotriazol-1-ylmethyl)-2,6-di-*tert*-butylphenol (**16**) was reacted with *p*-cresol, compound **17** was formed in 37% yield.

We have found that *o*-(benzotriazol-1-ylmethyl)phenol (**7a**) also reacts with other electron-rich aromatic compounds to give diarylmethanes. Nevertheless, the reaction conditions required are considerably different from those used in the reactions with phenols. Reaction of **7a** with indole occurred in neutral rather than basic or acidic conditions. Thus, a neat mixture of compound **7a** and indole in a closed vial was heated in an oil bath at 150 °C for 35 h to give the desired product **15** in 55% yield. When a mixture of compound **7a** and indole was heated in *i*-PrOH in the presence of *i*-PrONa, compound **7a** was decomposed and indole was completely recovered. When a neat mixture of **7a** and indole was heated with a catalytic amount of *p*-TsOH, no clean product was observed according to TLC and NMR; rather, a tar resulted.

o-(Benzotriazolylalkyl)naphthols **18**, obtained by condensation of the appropriate naphthol with an aldehyde and benzotriazole,[13] reacted with phenols in the presence of a base to give diarylmethanes or diarylalkanes. Compound **19** was prepared in this way in 59% yield.

The structures of **12–15**, **17**, and **19** were confirmed by their ^1H and ^{13}C NMR spectral data and elemental analysis or high-resolution mass spectroscopic data. The data for the known compounds are in agreement with those reported in the literature. The assignments of groups R^1, R^2, and $-CH_2-$ are given in Tables 2 and 3, and NMR spectra of these methylenebisphenols clearly showed the disappearance of the characteristic benzotriazolyl signals. The methylene (in compounds **12**, **13–15**, **17**, and **19**) and methine (in compound **13**) signals in both ^1H and ^{13}C NMR spectra shifted upfield as compared to those of their precursors due to the loss of the electron-withdrawing benzotriazole group (see Tables 2 and 3).

In summary, a new route has been developed for the preparation of 1,1-bis(2-hydroxyaryl)alkanes as well as *o,p*-methylenebisphenols. *o*-(Benzotriazolylalkyl)phenols also react with indole to give diarylmethanes. Compared to the previous methods, our method uses readily available starting materials and mild reaction conditions. This provides a novel and efficient approach to both symmetrical and, more importantly, unsymmetrical 1,1-bis(2-hydroxyaryl)alkanes.

Experimental Section

Melting points were determined with a Kofler hot apparatus and are uncorrected. ^1H and ^{13}C NMR spectra were recorded on a Varian 300-MHz spectrometer in $CDCl_3$ using TMS as an internal reference for ^1H spectra and $CDCl_3$ for ^{13}C NMR spectra. Elemental analyses were performed on a Carlo Erba-1106 instrument under the supervision of Dr. D. Powell. High-resolution mass measurements were recorded on an AEI MS-30 mass spectrometer. Column chromatography was carried out on MCB silica gel (230–400 mesh).

The benzotriazole adducts **7a–c**, **16**, and **18** were prepared according to the previously described method.

***o*-[α-(Benzotriazol-1-yl)ethyl]phenol (8a).** This novel compound was prepared according to the literature procedure[13,15] by quenching with methyl iodide and purified by column chromatography using hexane and ethyl acetate (8:1) as eluent: yield 35%; mp 170–171 °C; ^1H NMR δ 2.03 (d, 3 H, J = 7.1 Hz), 6.48 (q, 1 H, J = 7.1 Hz), 6.77 (t, 1 H, J = 7.8 Hz), 6.87 (d, 1 H, J = 7.8 Hz), 7.10 (t, 1 H, J = 7.8 Hz), 7.12 (t, 1 H, J = 8.3 Hz), 7.36 (t, 1 H, J = 7.8 Hz), 7.46 (t, 1 H, J = 7.8 Hz), 7.60 (d, 1 H, J = 8.3 Hz), 8.03 (d, 1 H, J = 8.3 Hz), 9.92 (s br, 1 H); ^{13}C NMR δ 19.8, 51.9, 110.7, 115.4, 119.1, 119.3, 123.8, 126.5, 126.6, 127.0, 129.0, 132.4, 145.3, 154.3. Anal. Found: C, 70.22; H, 5.49; N, 17.63. $C_{14}H_{13}N_3O$ requires: C, 70.28; H, 5.48; N, 17.56.

Preparation of 1,1-Bis(hydroxyaryl)alkanes 12a–j and 14a–b. General Procedure. A mixture of benzotriazolyl derivative (5 mmol), phenol (10 mmol), and *i*-PrONa (10 mmol; for **12a,d**: *t*-BuONa) in *i*-PrOH (50 mL; for **12a,d**: *t*-BuOH) was heated under reflux for the time given in Table 1. The solvent was evaporated, to the residue was added dilute HCl (1 N, 50 mL), and the solution was extracted with $CHCl_3$ (3 × 60 mL). The combined extracts were dried and the solvent evaporated. The residue was chromatographed with eluents of a mixture of hexane/ethyl acetate to give the desired product. The yields, melting points, chromatography eluents used, literature data, and elemental analyses or high-resolution mass spectroscopic data of the compounds prepared are summarized in Table 1.

2-[α-(*o*-Hydroxyphenyl)ethyl]-4-methylphenol (13). A mixture of **8a** (1.0 g, 4.1 mmol), *p*-cresol (0.6 g, 5.6 mmol), and *t*-BuONa (10 mmol) in *t*-BuOH (50 mL) was heated under reflux for 4 h and the solvent removed under reduced pressure. To the residue was added diluted HCl (1 N) to make the

Table 2. ^{1}H NMR Spectra of Compounds 12a–j, 13, 14a,b, 15, and 19

compd	R^1	R^2	$-CH_2-$ or (CH)	others
12a			3.89 (s, 2H)	6.8–6.9 (m, 4H), 7.03 (td, 2H, J = 7.4, 1.6 Hz), 7.21 (dd, 2H, J = 7.4, 1.6 Hz), 9.05 (s br, 2H)
12b		2.22 (s, 3H)	3.88 (s, 2H)	6.68 (d, 1H, J = 8.1 Hz), 6.8–6.9 (m, 3H), 7.01 (d, 1H, J = 7.3 Hz), 7.06 (s, 1H), 7.27 (d, 1H, J = 7.3 Hz), 7.77 (s br, 2H)
12c		1.26 (s, 9H)	3.91 (s, 2H)	6.75 (d, 1H, J = 8.5 Hz), 6.78 (d, 1H, J = 8.1 Hz), 6.86 (t, 1H, J = 8.1 Hz), 7.0–7.1 (m, 2H), 7.26–7.30 (m, 2H), 8.15 (s 2H)
12d			3.94 (s, 2H)	6.70 (t, 1H, J = 7.4 Hz), 6.84 (d, 1H, J = 8.1 Hz), 6.92 (d, 1H, J = 8.1 Hz), 6.97 (t, 1H, J = 7.4 Hz), 7.09 (d, 1H, J = 7.4 Hz), 7.2–7.4 (m, 5H), 7.43 (d, 2H, J = 7.9 Hz), 9.21 (s br, 2H)
12e			3.86 (s, 2H)	6.69 (d, 1H, J = 8.6 Hz), 6.79 (d, 1H, J = 7.6 Hz), 6.90 (t, 1H, J = 6.9 Hz), 6.98 (d, 1H, J = 8.6 Hz), 7.08 (t, 1H, J = 7.5 Hz), 7.2–7.3 (m, 2H)
12f		2.11 (s, 3H), 2.19 (s, 3H)	3.83 (s, 2H)	6.46 (s, 1H), 6.54 (s, 1H), 6.58 (t, 1H, J = 7.2 Hz), 6.68 (d, 1H, J = 6.7 Hz), 6.78 (d, 1H, J = 7.9 Hz), 6.92 (t, 1H, J = 7.3 Hz), 8.99 (s br, 2H)
12g (12h)[a]		2.18 (s, 3H), 2.41 (s, 3H)	3.87 (s, 2H), 3.96 (s, 2H)	6.60 (s, 1H), 6.67 (d, 1H, J = 7.6 Hz), 6.7–6.9 (m, 5H), 6.94 (t, 1H, J = 7.6 Hz), 7.04 (t, 1H J = 7.4 Hz), 7.06 (t, 1H, J = 7.4 Hz), 7.13 (d, 1H, J = 7.4 Hz), 7.26 (t, 2H, J = 7.4 Hz)
12i	1.28 (s, 9H)	2.24 (s, 3H)	3.87 (s, 2H)	6.7–6.9 (m, 3H), 7.1–7.2 (m, 2H), 7.30 (d, 1H, J = 7.0 Hz)
12j	1.38 (s, 9H), 2.23 (s, 3H)	2.25 (s, 3H)	3.84 (s, 2H)	5.86 (s, 1H), 6.59 (s, 1H), 6.61 (d, 1H, J = 5.7 Hz), 6.86 (d, 1H, J = 8.2 Hz), 6.95 (m, 1H), 7.07 (s, 1H)
13		2.23 (s, 3H)	4.66 (q, 1H, J = 7.1 Hz)	1.65 (d, 3H, J = 7.1 Hz), 6.66 (d, 1H, J = 7.6 Hz), 6.7–6.8 (m, 2H), 6.91 (t, 1H, J = 7.6 Hz), 7.02 (td, 1H, J = 7.7, 1.9 Hz), 7.09 (s, 1H), 7.31 Hz (d, 2H, J = 7.6 Hz)
14a			4.35 (s, 2H)	6.55 (t, 1H, J = 7.2 Hz), 6.80 (d, 1H, J = 7.4 Hz), 6.85–6.95 (m, 2H), 7.2–7.4 (m, 3H), 7.59 (d, 1H, J = 8.9 Hz), 7.68 (d, 1H, J = 8.0 Hz), 7.87 (d, 1H, J = 8.4), 9.35 (s, br, 2H)
14b			4.06 (s, 2H)	6.62 (s br, 1H), 6.66 (dd, 1H, J = 8.0, 1.2 Hz), 6.86 (td, 1H, J = 7.4, 1.2 Hz), 6.99 (td, 1H, J = 7.0, 1.2 Hz), 7.31 (dd, 1H, J = 7.4, 1.2 Hz), 7.37–7.42 (m, 4H), 7.7–7.8 (m, 1H), 7.79 (s br, 1H), 8.15–8.19 (m, 1H)
15			3.97 (s, 2H)	5.04 (s br, 1H), 6.6–6.7 (m, 2H), 6.78 (t, 1H, J = 7.3 Hz), 6.9–7.2 (m, 5H), 7.43 (d, 1H, J = 7.7 Hz), 7.67 (s br, 1H)
19		2.24 (s, 3H)	6.51 (s, 1H)	4.92 (s br, 1H), 5.58 (s br, 1H), 6.8–6.9 (m, 2H), 7.08 (t, 2H, J = 7.1 Hz), 7.24–7.38 (m, 6H), 7.42 (t, 1H, J = 7.0 Hz), 7.77 (dd, 2H, J = 7.8, 3.3 Hz) 8.00 (d, 1H, J = 8.5 Hz)

[a] A mixture of **12g** and **12h**, ratio 1:1.

Table 3. ^{13}C NMR Spectra of Compounds 12a–j, 13, 14a,b, 15, and 19

compd	R^1	R^2	$-CH_2-$ or (CH)	others
12a			30.8	115.7, 120.2, 127.2, 127.4, 130.3, 153.4
12b		20.5	30.9	115.7, 115.9, 121.5, 126.6, 127.1, 128.0, 128.4, 130.7, 130.8, 131.2, 149.8, 152.3
12c		31.5, 34.0	31.4	115.4, 116.0, 121.5, 124.9, 126.3, 127.3, 127.5, 127.9, 130.7, 144.3, 149.8, 152.3
12d		*a*	29.8	115.0, 115.5, 119.1, 125.2, 126.0, 126.1, 126.8, 127.0, 127.6, 128.4, 128.8, 130.2, 131.5, 140.7, 154.5, 154.6
12e			30.7	115.8, 117.2, 121.8, 125.9, 126.1, 127.8, 128.3, 128.6, 130.3, 130.8, 151.2, 152.0
12f		19.2, 20.6	24.9	113.3, 114.4, 118.6, 121.6, 121.8, 126.0, 126.5, 128.3, 135.4, 137.6, 154.7, 154.8
12g (12h)[b]		20.4, 20.9	27.3, 30.6	113.4, 115.9, 116.1, 116.5, 120.7, 121.5, 122.3, 123.7, 123.9, 124.8, 125.6, 127.2, 127.3, 127.9, 130.5, 130.7, 131.4, 138.1, 138.9, 152.0, 152.3, 152.4, 153.4
12i	34.0[c]	20.5	31.5	115.4, 115.8, 124.9, 126.3, 126.9, 127.5, 128.4, 130.6, 131.2, 144.1, 150.1, 150.2
12j	20.8, 29.9, 34.5	20.5	31.1	115.3, 126.2, 126.5, 127.4, 128.2, 128.9, 129.3, 131.0, 131.3, 136.7, 149.8, 150.0
13		20.7	19.5	19.5, 115.6, 115.8, 121.4, 126.9, 127.3, 127.5, 127.8, 130.7, 131.2, 131.7, 149.7, 152.2
14a			24.0	114.9, 118.0, 118.2, 119.1, 122.3, 123.3, 126.0, 126.5, 127.1, 127.6, 128.2, 128.5, 129.2, 133.7, 152.5, 154.6
14b			31.0	115.3, 120.5, 120.7, 121.5, 121.8, 125.1, 125.4, 125.7, 126.9, 127.5, 127.9, 128.4, 130.7, 133.6, 148.2, 151.7
15			26.8	111.2, 113.3, 115.9, 119.1, 119.5, 120.8, 122.3, 122.4, 126.5, 127.1, 127.8, 130.5, 136.6, 154.2
19		16.0	43.5	117.8, 119.6, 121.2, 122.7, 123.4, 124.5, 127.0, 127.3, 127.4, 128.8, 129.2, 129.7, 130.0, 130.3, 133.1, 140.6, 152.3, 153.3

[a] The peaks are overlapped and indistinguishable from others. [b] A mixture of **12g** and **12h**, ratio 1:1. [c] Another peak is overlapped and indistinguishable from the methylene signal.

solution slightly acidic (pH = 6). The solution was then extracted with diethyl ether (3 × 60 mL), washed with water (2 × 50 mL), and dried with $MgSO_4$. The solvent was evaporated in vacuo and the residue chromatographed with hexane/ethyl acetate. The preparative details and physical data are given in Table 1.

3-[(*o*-Hydroxyphenyl)methyl]indole (15). A mixture of *o*-(benzotriazol-1-ylmethyl)phenol (**7a**) (0.23 g, 1.0 mmol) and indole (0.12 g, 1.0 mmol) was heated in a screw-top vial in an oil bath at 150 °C for 36 h. This mixture was separated by column chromatography with hexane and EtOAc as eluents. The preparative details and physical data are given in Table 1.

2,6-Di-*tert*-butyl-4-[(2′-hydroxy-5′-methylphenyl)methyl]phenol (17). A mixture of 2,6-di-*tert*-butyl-4-(benzotriazol-1-ylmethyl)phenol (**16**) (2.02 g, 6 mmol), *p*-cresol (0.65 g, 6 mmol), and *i*-PrONa (10 mmol) in *i*-PrOH (50 mL) was heated under reflux for 40 h and the solvent removed under reduced pressure. Diluted HCl (1 N) was added to the residue to make the solution slightly acidic (pH = 6). The solution was then extracted with diethyl ether (3 × 60 mL), washed with water (2 × 50 mL), and dried with $MgSO_4$. The solvent was evaporated in vacuo and the residue chromatographed with hexane/ethyl acetate (20:1) to give 0.54 g of oil: yield 27%; ^{1}H NMR δ 1.39 (s, 18 H), 2.24 (s, 3 H), 3.86 (s, 2 H), 4.77 (s, 1 H),

5.07 (s, 1 H), 6.66 (d, 1 H, J = 7.9 Hz), 6.87–6.92 (m, 2 H), 7.04 (s, 1 H); ^{13}C NMR δ 20.5, 30.2, 34.3, 36.5, 115.6, 125.1, 127.0, 128.0, 129.8, 130.0, 131.3, 136.1, 151.6, 152.3. Anal. Found: C, 80.94; H, 9.28. $C_{22}H_{30}NO_2$ requires: C, 81.11; H, 9.28.

1-[Phenyl(2-hydroxy-3-methylphenyl)methyl]-2-naphthol (19). A mixture of **18** (1.75 g, 5.0 mmol), *o*-cresol (0.64 g, 6.0 mmol), and *t*-BuONa (10 mmol) in *t*-BuOH (50 mL) was heated under reflux for 3 h and the solvent removed under reduced pressure. Diluted HCl (1 N) was added to the residue to make the solution slightly acidic (pH = 6). The solution was then extracted with diethyl ether (3 × 60 mL), washed with water (2 × 50 mL), and dried with $MgSO_4$. The solvent was evaporated in vacuo and the residue chromatographed with hexane/ethyl acetate. The preparative details and physical data are given in Table 1.