ammonium chloride solution and then diluted with ethyl acetate (30 mL). The organic layer was separated and washed with 10% citric acid (10 mL×3), saturated aqueous sodium bicarbonate solution (10 mL×2), and brine (10 mL), successively, and then dried over anhydrous $MgSO_4$. After removal of the solvent, the resulting oily residue was purified by column chromatography (silica gel, hexane/ethyl acetate= 20-10/1), and recrystallized from ethyl acetate and hexane to give crystalline product. Reaction time and yield as well as physical, spectroscopic and elemental analysis data of products are summarized in Table 2.

Determination of the X-ray crystal structure of 4. Diffraction data were collected on an Enraf-Nomius CAD4 diffractometer with graphite-monochromated MoKα radiation ($\lambda(K\alpha_1)$=0.70926 Å) and structure was solved by direct method and refined anisotropically for non-H atoms. Crystal data and structure refinement are summarized in Table 3, and bond lengths and angles are listed in Table 4. Crystallographic diagram (Figure 3) was obtained using the program of ORTEP-PC version.

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Benzoyl Rearrangement in Synthesis of Asymmetrically Substituted Calix[4]arenes

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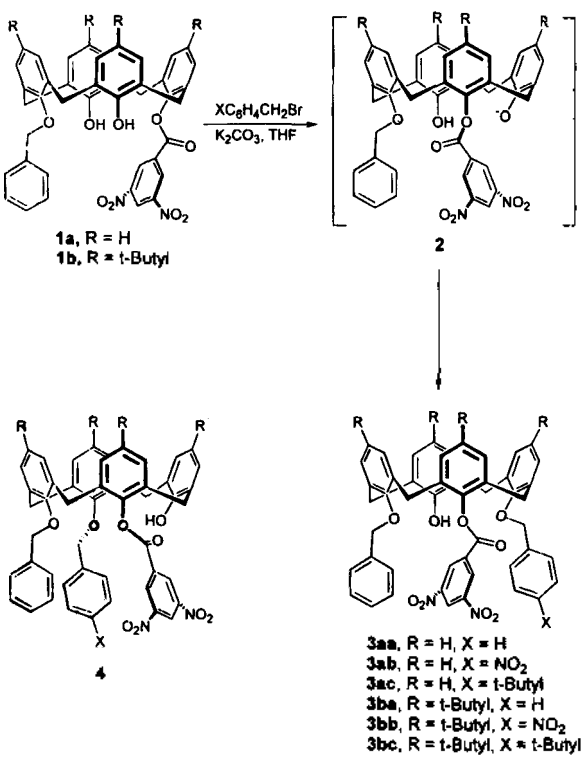
Calixarenes are cavity containing metacyclophanes which are currently utilized as a versatile host molecules.[1,2,3] One of the most important aspects about host-guest chemistry is molecular recognition.[4,5] Like chiral cyclodextrines, calixarenes are expected to have similar chiral recognition ability because molecular structure of calixarenes could allow the preparation of synthetic molecule with a chiral cavity.[6] If molecular asymmetry could be originated directly from the calixarene framework, the efficient chiral recognition would be expected.

Chiral calixarenes first have been prepared by attaching chiral residues to the tetramer.[7] Also the various asymmetric calix[4]arenes were synthesized by the direct introduction of the three or four different substituents at the upper rim[8,9] of calix[4]arenes as well as by the selective alkylation at the lower rim[10,11] of calix[4]arenes. In 1995, González *et al.*[12] reported that an intermolecular migration of sulfonyl groups in 1,3-bistriflate and 1,3-bismesylate derivatives of *p-tert*-butylcalix[4]arene took place in the presence of a palladium catalysts and chloride anion. Mar-

kovsky *et al.*[13] recently reported the phosphorotropic rearrangement in the synthesis of asymmetrically substituted calix[4]arene. Here we report for the first time benzoyl migration in calix[4]arene during the benzylation reaction in the presence of K_2CO_3. From this benzoyl migration, the four asymmetrically substituted calix[4]arenes were synthesized. By introducing three different benzene moieties at the lower rim of calix[4]arenes, not only the various chiral calix[4]arenes are obtained, but also ring inversion is inhibited. Since **1**[14,15] is easily available from the selective functionalization, this simple method could provide the efficient synthetic method for the chiral calix[4]arenes.

Refluxing **1a** in THF for 28 hours with benzyl bromide in the presence of K_2CO_3 surprisingly produced the dibenzylated calix[4]arene **3aa** in 80% yield, which could not be obtained by the direct benzylation reaction from **1a**. Two benzyl groups at **3aa** are located at the 2,4-position to the relative to the 3,5-dinitrobenzoyl group, which indicate that 3,5-dinitrobenzoyl group migrated to the adjacent OH group. **1a** could form an anion in the presence of base, even though the anion formed could not attack benzyl bromide at this stage presumably due to the neighboring large benzyl and benzoyl groups. But the anion can attack benzoyl ester near by to produce 1,2-disubstituted anion **2a**, which then attack benzyl bromide easily due to much favorable steric environment to produce 1,2,4-trisubstituted **3aa** as shown in Scheme 1. The 1,2-disubstituted intermediates **2a** could not be isolated, nor detected by TLC analysis. This suggests that the migration of benzoyl group is much slower than benzylation. Dibenzylation was confirmed by the hydrolysis of **3aa** which produced the



Scheme 1.

achiral 1,3-dibenzylated calix[4]arene. Benzoyl migration was only observed when benzyl bromides treated with K_2CO_3, suggesting that benzyl bromide happened to be a right alkylation agent for the benzoyl migration. To utilize this benzoyl migration for the preparation of chiral calix[4]arenes, **1a** were treated with *p*-nitrobenzyl bromide and *p-tert*-butylbenzyl bromide in the presence of K_2CO_3 under the same reaction conditions described above. It was turned out that the 3,5-dinitrobenzoyl group also migrated and then *para* substituted benzyl bromide reacted with **2a** to yield the chiral calix[4]arenes **3ab** and **3ac**, respectively. There was no difference between *p*-nitro and *p-tert*-butylbenzyl bromide toward the reactivity as well as selectivity, indicating that the benzoyl migration determined the rate and position of benzylation reaction. Also complete absence of **4** as a product and no detection of **2a** as a intermediate during and after the reaction strongly suggest that benzylation occurs almost simultaneously with benzoyl migration. *p-tert*-Butylcalix[4]arene derivative **1b** was also treated with the various benzyl bromide in the presence of K_2CO_3 in THF and obtained the products **3ba**, **3bb**, and **3bc** in high yield, suggesting that the benzoyl migration at the lower rim was occurred regardless of alkyl substituents at the upper rim of calix[4]arene.

The ^{1}H NMR spectrum of **3ab** showed the typical chiral calix[4]arene characteristics such as four pairs of doublets at 4.6-3.3 ppm for the eight methylene protons and very complicated aromatic signals. In order to confirm that the four asymmetrically substituted compounds **3ab**, **3ac**, **3bb**, and **3bc** consist of a pair of enantiomers, we measured their ^{1}H NMR spectra in the presence of chiral shift reagents as shown in Figure 1. It was found that Pirkle's reagent[16] ((S)-2,2,2-trifluoro-1-(9-anthryl)ethanol) is very effective. In all four asymmetric compounds peaks shifted slightly upfield and split into more complicated pattern due to doubling even at 25 °C.

Compounds **3aa**, **3ab**, **3ac**, **3ba**, **3bb**, and **3bc** exist in the cone conformation. They showed NMR data consistent with cone conformation. Particularly diagnostic were the chemical shifts for the methylene carbons of these derivatives which appeared four peaks at 30-32 ppm, charac-

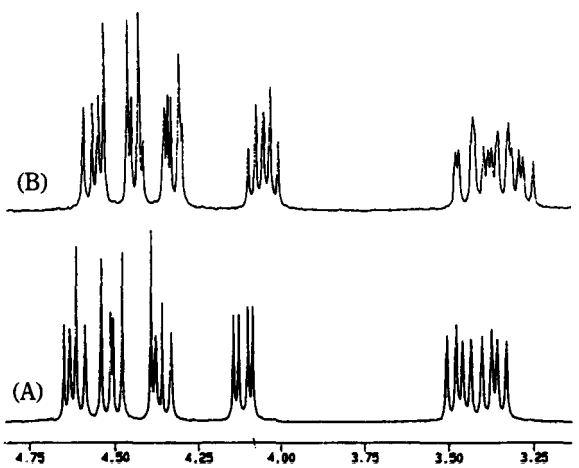


Figure 1. Partial ^{1}H NMR spectra of compound **3ab** in $CDCl_3$: (A) in the absence of Pirkle's reagent; (B) in the presence of Pirkle's reagent (2 equiv.).

teristic of *syn* oriented adjacent phenol ring.[17]

Experimental

Calix[4]arenes. **1a**[14] and **1b**[15] were synthesized by the reported procedures. **1a**: mp 267-270 °C **1b**: mp > 290 °C dec.

25-(3,5-Dinitrobenzoyloxy)-26,28-bisbenzyloxy-27-hydroxycalix[4]arene 3aa. A mixture of 0.84 g (1.19 mmol) of **1a**, 0.68 g (4.92 mmol) of K_2CO_3 and 3.0 mL (25.2 mmol) of benzyl bromide in 70 mL of THF was refluxed for 28 h. The solvents were evaporated and the residue was dissolved with 30 mL of $CHCl_3$ and washed with 0.1 N HCl. The organic layer was separated, dried over anhydrous Na_2SO_4, evaporated the solvents, and the residue triturated with methanol. Recrystallization from $CHCl_3$-MeOH gave 0.65 g (68%) of fine yellow crystalline. mp > 263 °C dec. ^1H NMR ($CDCl_3$) δ 9.43 (d, 2H, O_2NArH, J=2.2 Hz), 8.48 (t, 1H, O_2NArH ArH, J=2.2 Hz), 7.34 (d, 2H, ArH), 7.25-6.70 (m, 18H, ArH), 6.63 (t, 2H, ArH), 6.50 (s, 1H, OH), 4.47 and 4.38 (a pair of d, 4H, -OCH_2Ar, J=9.92 Hz), 4.53, 4.16, 3.45, and 3.42 (two pairs of d, 8H, $ArCH_2Ar$, J=13.64 Hz, and 12.90 Hz). ^{13}C NMR ($CDCl_3$) δ 163.71 (-CO_2-), 153.61, 152.48, 147.39, 146.34, 135.45, 135.34, 133.01, 132.16, 132.04, 131.59, 129.27, 129.23, 128.90, 128.75, 128.46, 128.24, 128.14, 127.98, 126.92, 124.67, 122.98, and 118.92 (Ar), 78.91 (-OCH_2Ar), 31.04, 30.27 ($ArCH_2Ar$). IR (KBr) 3458 cm^{-1} (OH), 1744 cm^{-1} (C=O), 1550 and 1340 cm^{-1} (NO_2).

25-(3,5-dinitrobenzoyloxy)-26-benzyloxy-27-hydroxy-28-(*p*-nitrobenzyloxy)calix[4]arene 3ab. A mixture of 0.32 g (0.452 mmol) of **1a**, 0.50 g (3.62 mmol) of K_2CO_3 and 1.90 g (8.79 mmol) of 4-nitrobenzyl bromide in 60 mL of THF was added and then refluxed for 31 h and followed the procedure described for **3aa** to give 0.26 g (68%) of **3ab**. mp > 252 °C dec. ^1H NMR ($CDCl_3$) δ 9.51 (d, 2H, O_2NArH, J=2.19 Hz), 8.58 (t, 1H, O_2NArH, J=2.19 Hz), 7.78 (d, 2H, ArH, J=8.19 Hz), 7.35 (t, 2H, ArH), 7.24 (d, 1H, ArH), 7.21-6.56 (m, 16H, ArH), 6.30 (s, 1H, OH), 4.62 and 4.51 (a pair of d, 2H, -OCH_2ArH, J=10.62 Hz), 4.48 and 4.36 (a pair of d, 2H, -OCH_2ArH, J=10.08 Hz), 4.59, 4.33, 4.10, 4.09, 3.46, 3.44, 3.36, and 3.33 (four pairs of d, 7H, $ArCH_2Ar$, J=13.41 Hz, 13.92 Hz, 13.02 Hz, 12.93 Hz, and 12.93 Hz). ^{13}C NMR ($CDCl_3$) δ 163.49 (-CO_2-), 153.41, 152.44, 151.91, 147.75, 147.66, 146.54, 142.34, 135.39, 135.29, 135.09, 133.38, 132.50, 132.42, 131.69, 131.33, 130.14, 129.45, 129.22, 129.08, 129.03, 128.84, 128.65, 128.60, 128.52, 128.34, 128.29, 127.35, 127.04, 125.16, 124.59, 123.12, 122.49, and 119.20 (Ar), 79.22 and 76.89 (-OCH_2Ar), 31.25, 30.84, 30.38, and 30.26 ($ArCH_2Ar$). IR (KBr) 3433 cm^{-1} (OH), 1730 cm^{-1} (-CO_2-), 1542 and 1344 cm^{-1} (-NO_2).

25-(3,5-Dinitrobenzoyloxy)-26-benzyloxy-27-hydroxy-28-(*p*-*tert*-butylbenzyloxy)calix[4]arene 3ac. A mixture of 0.30 g (0.424 mmol) of **1a**, 0.50 g (3.62 mmol) of K_2CO_3 and 1.67 mL (9.09 mmol) of *p*-*tert*-butylbenzyl bromide in 60 mL of THF was refluxed for 23 h and followed the procedure described for **3aa** to yield 0.25 g (69%) of light yellowish product. mp > 268 °C dec. ^1H NMR ($CDCl_3$) δ 9.52 (d, 2H, O_2NArH, J=2.13 Hz), 8.48 (t, 1H, O_2NArH, J=2.13 Hz), 7.34 (d, 2H, ArH, J=7.5 Hz), 7.25-6.57 (m, 19H, ArH), 6.53 (s, 1H, OH), 4.46 and 4.34 (a pair of d, 2H, -OCH_2Ar, J=10.11 Hz), 4.43 and 4.39 (a pair of d, 2H, -OCH_2Ar, J=10.26 Hz), 4.65, 4.49, 4.19, and 4.14 (four d, 4H, $ArCH_2Ar$, J=13.56 Hz, 13.48 Hz, 13.02 Hz, and 13.05 Hz), 3.50-3.40 (four d, 4H, $ArCH_2Ar$), 1.19 (s, 9H, *t*-Bu). ^{13}C NMR ($CDCl_3$) δ 163.88 (-CO_2-), 153.63, 152.88, 152.36, 151.33, 147.41, 146.72, 135.56, 135.45, 135.56, 133.27, 132.84, 132.52, 132.26, 132.19, 131.66, 129.34, 129.23, 129.14, 128.92, 128.89, 128.83, 128.60, 128.35, 128.31, 128.22, 127.71, 126.92, 124.93, 124.83, 124.47, 122.66, and 118.94 (Ar), 79.01 and 78.54 (-CH_2Ar), 34.40, 31.11, 31.06, 30.39, and 30.34 ($ArCH_2Ar$ and *t*-Bu). IR (KBr) 3448 cm^{-1} (OH), 1731 cm^{-1} (-CO_2-), 1542 and 1342 cm^{-1} (-NO_2).

5,11,17,23-Tetra-*tert*-butyl-25-(3,5-dinitrobenzoyloxy)-26,28-bisbenzyloxy-27-hydroxycalix[4]arene 3ba. A mixture of 0.84 g (1.19 mmol) of **1b**, 0.68 g (4.92 mmol) of K_2CO_3 and 3.0 mL (25.22 mmol) of benzyl bromide in 70 mL of THF was refluxed for 32 h and followed the procedure described for **3aa** to give 0.65 g (68%) of **3ba**. mp 310 °C dec. ^1H NMR ($CDCl_3$) δ 9.36 (d, 2H, O_2NArH, J=2.10 Hz), 8.43 (t, 1H, O_2NArH, J=2.10), 7.35 (s, 2H, ArH), 7.16 (s, 2H, ArH), 6.97-6.65 (m, 13H, ArH), 6.56 (s, 1H, OH), 4.50 and 4.36 (a pair of d, 4H, -OCH_2Ar, J=9.6 Hz), 4.56, 4.15, 3.41, and 3.40 (two pairs of d, 8H, $ArCH_2Ar$, J=13.2 Hz and 12.9 Hz), 1.42 (s, 9H, -$C(CH_3)_3$), 1.38 (s, 9H, -$C(CH_3)_3$), 0.88 (s, 18H, -$C(CH_3)_3$). ^{13}C NMR ($CDCl_3$) δ 164.00 (-CO_2-), 150.97, 150.20, 149.18, 147.38, 146.57, 145.32, 144.22, 141.31, 135.70, 134.83, 132.83, 132.18, 131.54, 131.29, 130.63, 129.18, 128.08, 128.03, 127.45, 125.89, 125.79, 125.29, 125.11, and 122.90 (Ar), 78.76 (-OCH_2Ar) 31.81, 31.61 ($ArCH_2Ar$), 34.43, 33.93, 33.85, 31.41, 30.99, and 30.61 (-$C(CH_3)_3$). IR (KBr) 3450 cm^{-1} (OH), 1740 cm^{-1} (-CO_2-), 1550 and 1340 cm^{-1} (NO_2).

5,11,17,23-Tetra-*tert*-butyl-25-(3,5-dinitrobenzoyloxy)-26-benzyloxy-27-hydroxy-28-(*p*-nitrobenzyloxy)calix[4]arene 3bb. A mixture of 0.33 g (0.354 mmol) of **1b**, 0.60 g (4.34 mmol) of K_2CO_3 and 1.53 g (7.08 mmol) of 4-nitrobenzyl bromide in 60 mL of THF was refluxed for 45 h and followed the procedure for **3aa** to yield 0.23 g (61%) of light yellow product **3bb**. mp 281-283 °C. ^1H NMR ($CDCl_3$) δ 9.45 (d, 2H, O_2NArH, J=2.13 Hz), 8.51 (t, 1H, O_2NArH, J=2.13 Hz), 7.77 and 7.13 (a pair of d, 4H, ArH from 4-nitrobenzyl, J=8.64 Hz), 7.36, 7.32, 7.18, 7.11, 6.83, 6.70, 6.65, and 6.59 (eight d, 8H, ArH, J=2.34 Hz), 7.07-6.90 (m, 5H, ArH from benzyl), 6.41 (s, 1H, OH), 4.67 and 4.54 (a pair of d, 2H, -OCH_2Ar, J= 10.59 Hz), 4.50 and 4.38 (a pair of d, 2H, -OCH_2Ar, J= 10.35 Hz), 4.65, 4.33, 4.06, 4.05, 4.06, 3.40, 3.32, and 3.29 (four pairs of d, 8H, $ArCH_2Ar$, J=13.17 Hz, 12.87 Hz, 13.38 Hz, and 12.84 Hz), 1.41, 1.37, 0.92, and 0.82 (four s, 36H, *t*-Bu). ^{13}C NMR ($CDCl_3$) δ 163.76 (-CO_2-), 150.76, 150.21, 149.52, 149.38, 147.67, 147.53, 147.21, 146.50, 144.07, 124.80, 141.62, 135.38, 134.71, 134.65, 132.67, 132.57, 131.73, 131.57, 131.29, 130.74, 130.05, 128.87, 128.37, 128.25, 128.18, 126.66, 126.05, 125.92, 125.86, 125.76, 125.46, 124.97, 123.02, 122.44, and 101.66 (Ar), 79.09, 76. 76 (-OCH_2Ar), 34.44, 33.93, 33.79, 31.77, 31.58, 30.98, 30. 93, 30.68, and 30.57 ($ArCH_2Ar$ and *t*-Bu). IR (KBr) 3467 cm^{-1} (OH), 2961 cm^{-1} (*t*-Bu), 1729 cm^{-1} (-CO_2-), 1545

and 1346 cm^{-1} ($-NO_2$).

5,11,17,23-Tetra-*tert*-butyl-25-(3,5-dinitrobenzoyloxy)-26-benzyloxy-27-hydroxy-28-(*p*-*tert*-butylbenzyloxy)calix[4]arene 3bc. A mixture of 0.30 g (0.322 mmol) of **1b**, 0.60 g (4.34 mmol) of K_2CO_3 and 1.18 mL (6.44 mmol) of *p*-*tert*-butylbenzyl bromide in 60 mL of THF was refluxed for 38 h and followed the procedure for **3aa** to yield 0.28 g (81%) of pale yellow product **3bc**. mp 270-272 °C. ^1H NMR ($CDCl_3$) δ 9.46 (d, 2H, O_2NArH), 8.41 (t, 1H, O_2NArH), 7.36 (s, 2H, ArH), 7.17, and 7.13 (two d, 2H, ArH, *J*=2.01 Hz), 7.04-6.87 (m, 9H, ArH), 6.79, 6.72, 6.67, and 6.64 (four d, 4H, ArH, *J*=2.01 Hz), 6.59 (s, 1H, OH), 4.48 and 4.36 (a pair of d, 2H, $-OCH_2Ar$, *J*=10.05 Hz), 4.47 and 4.41 (a pair of d, 2H, $-OCH_2Ar$, *J*=10.11 Hz), 4.69, 4.44, 4.18, and 4.10 (four d, 4H, $ArCH_2Ar$, *J*=13.38 Hz, 12.81 Hz, 12.84 Hz, and 12.87 Hz), 3.39 (m, 4H, $ArCH_2Ar$), 1.42, 1.38, 1.18, 0.90, and 0.86 (five s, 45H, *t*-Bu). ^{13}C NMR ($CDCl_3$) δ 164.13 ($-CO_2-$), 151.05, 150.96, 150.61, 149.96, 149.17, 147.28, 146.76, 146.29, 144.24, 141.28, 135.65, 134.93, 134.74, 132.89, 132.59, 132.26, 132.14, 131.60, 131.40, 131.23, 128.98, 128.69, 128.14, 128.08, 127.89, 127.06, 125.91, 125.78, 125.72, 125.49, 125.22, 125.04, 124.99, 124.77, and 122.59 (Ar), 78.84, 78.34 ($-OCH_2Ar$), 34.42, 34.34, 33.90, 33.86, 33.80, 31.80, 31.60, 31.53, 31.27, 31.10, 30.98, 30.69, and 30.58 (Ar CH_2Ar and *t*-Bu). IR (KBr) 3448 cm^{-1} (OH), 2961 cm^{-1} (*t*-Bu), 1732 cm^{-1} ($-CO_2-$), 1543 and 1342 cm^{-1} ($-NO_2$).

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