

Synthesis and Molecular Structure of Tetrahomodioxo *p*-Phenylcalix[4]arene Tetra Ester Derivative in 1,4-Alternate Conformation

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Reaction of tetrahomodioxo *p*-phenylcalix[4]arene with ethyl bromoacetate and potassium carbonate in acetone leads to the title tetra ester derivative, 7,13,21,27-tetra-phenyl-29,30,31,32-tetrakis(ethoxycarbonyl)methoxy-2,3,16,17-tetrahydro-3,17-dioxacalix[4]arene, its structure was determined by NMR spectra as 1,4-alternate conformation. The molecular structure has been solved by X-ray diffraction methods. The molecule has a conformation with pseudo center of symmetry. The benzene ring A is up, ring C is down. B and D rings are flat with respect to the plane of the macrocyclic ring.

Introduction

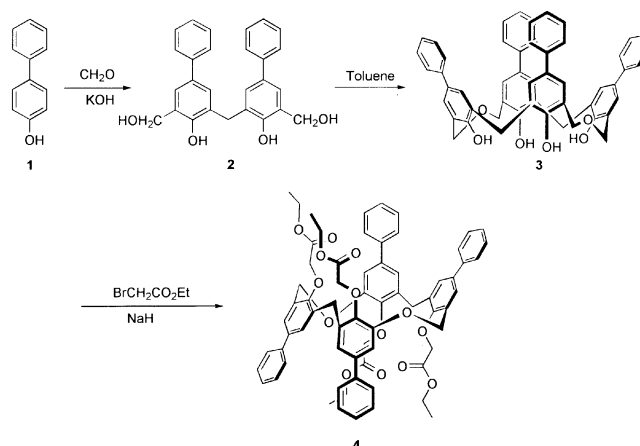
Calixarenes are synthetic macrocycle available in a variety of ring sizes and are of interest both as complexation hosts for ions and molecules and as frameworks for elaborating more complex structures and have received a great deal of attention in recent years.¹⁻³ In contrast to the calix[4]arenes, homooxacalix[4]arenes, containing extra oxygen in the macrocyclic ring, have received little attention mainly because they can only be synthesized in relatively small yield.^{4,6} Dihomooxa *p*-*t*-butylcalix[4]arene was obtained as a minor product (20% yield) from the direct base-catalyzed condensation reaction of *p*-*t*-butylphenol and formaldehyde⁴ and then the yield was enhanced up to 25%.⁵ Tetrahomodioxo *p*-*t*-butylcalix[4]arene was prepared by Gutsche in 44% yield from the dehydration of bishydroxymethylated dimer of *p*-*t*-butylphenol in xylene.⁶ Recently we reported the facile synthesis of bishomomonoxa- and tetrahomodioxo *p*-phenylcalix[4]arene by refluxing the bishydroxymethylated *p*-phenyl phenol in toluene.⁷ There have only been limited studies for the solution conformation and crystal structure of dihomooxa *p*-*t*-butylcalix[4]arene.⁸⁻¹¹ Recently Masci and coworkers¹² reported that the main conformation of tetrahomodioxo *p*-*t*-butylcalix[4]arene tetra methyl ether is 1,4-alternate based on the temperature dependent NMR spectral analysis, however, crystal structure of tetrahomodioxo derivative was not reported yet. In this paper we describe the synthesis and molecular structure determination of tetrahomodioxo *p*-phenylcalix[4]arene tetra ester derivative **4**.

Synthesis

When the mixture of *p*-phenyl phenol and 35% formaldehyde was stirred for 4 days at 40 °C in the presence of potassium hydroxide, the dimer diol **2** was prepared in 55% yield using the published procedures.^{13,14} Compound **2** (3.02 g, 7.32 mmole) was refluxed in toluene (180 mL) for 20 h to remove water in a Dean-Stark moisture trap to afford the tetrahomodioxacalix[4]arene **3** in 79% yield.⁷ When the homooxacalix[4]arene **3** was reacted with excess ethyl bro-

moacetate in acetone in the presence of K₂CO₃, 91% of compound **4** was isolated as crystalline solid. Compound **4** was also prepared with comparable yield from the treatment of compound **3** with ethyl bromoacetate in the presence of NaH in the mixture of THF and DMF.

The structure of compound **4** was confirmed by ¹H and ¹³C NMR spectra and elemental analysis. In the ¹H NMR spectrum, the methylenic protons from ArCH₂Ar and ArCH₂O bridges appear as two sets of AB quartet. ¹³C NMR spectrum shows single peak from carbonyl carbon, 8 peaks from aromatic carbons, one peak at 71.24 ppm from ArCH₂O bridge methylene carbons and one peak at 31.43 ppm from ArCH₂Ar bridge carbons. These spectral patterns are commensurated with cone or 1,4-alternate conformation in which two adjacent phenyl rings connected by methylene group are inverted. The position of the methylenic bridge carbons of ArCH₂Ar at 31.43 ppm indicates that these two adjacent benzene rings are in a syn orientation.¹⁵ The compound **4** can exist in five well defined conformation instead of the four conformation characteristic of calix[4]arene. They are symmetrical cone, partial cone, 1,2-alternates, 1,3-alternate, and 1,4-alternate forms as shown Figure 1. Partial cone, 1,2-alternate and 1,3-alternate conformer, in which



Scheme

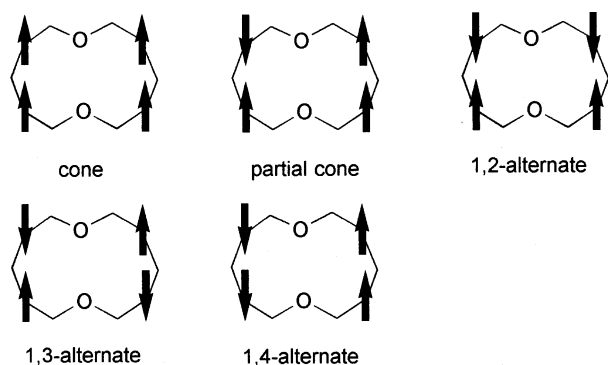


Figure 1. Conformations of tetrahomodioxacalix[4]arene.

two adjacent benzene rings are in an anti orientation, should show additional methylenic bridge carbon peak at around 37 ppm.

X-ray Structure Analysis. Colorless crystals were recrystallized from a mixture of dichloromethane and methanol. The crystal is unstable in the air and is sealed in the capillary tube. X-ray intensity data were on a Simens SMART¹⁶ diffractometer/CCD area detector at $-80\text{ }^{\circ}\text{C}$. Preliminary orientation matrix and cell constants were determined by a collection of 20 seconds and 15 frames, followed by spot integration and least-squares refinement. A hemisphere of data was collected using $0.3^{\circ}\omega$ scans at 20 seconds per frame. The 17330 reflections were integrated and refined using the program of SAINT.¹⁷ Data analysis and absorption correction were performed using the programs of Simens XPREP.¹⁸ All of the crystal data are summarized in Table 1.

The structure was solved by direct method and refined using the program SHELXL-97.¹⁹ The crystal structure has two dichloromethane solvent molecules. The final positional and thermal parameters of non hydrogen atoms are listed in Table 2.

Description of the Structure. The molecule has a conformation with pseudo center of symmetry. The selected molecular geometric parameters are listed in Table 3. These

Table 1. Crystal Data

molecular formula	$\text{C}_{70}\text{H}_{68}\text{O}_{14} \cdot 2\text{CH}_2\text{Cl}_2$
molecular weight	1303.1
temperature, K	193
space group	$P2_1/c$
$a = 20.626(3)$	$Z = 4$
$b = 11.454(3)$	$V = 6504(2)\text{ \AA}^3$
$c = 27.773(6)\text{ \AA}$	density (calc.) = 1.331 g/cm^3
$\beta = 97.59(1)^{\circ}$	crystal size : $0.53 \times 0.42 \times 0.49\text{ mm}$
Simens SMART diffractometer / CCD area detector	
scan type : ω	$\theta_{\text{max}} = 27^{\circ}$
no. of unique reflection	10943
no. of observed reflection	7282 $F_0 \ 4\sigma(F_0)$
GOF on F^2 1.101	μ 0.248 mm^{-1}
$R = 0.117$	$R_w = 0.167$
$\Delta\rho_{\text{max}} = 0.67\text{ e\AA}^{-3}$	$\Delta\rho_{\text{min}} = -0.95\text{ e\AA}^{-3}$

Table 2. Fractional Atomic Coordinates and Equivalent Isotropic Thermal Parameters for Nonhydrogen Atoms. The e. s. d.'s are in parentheses. $U_{\text{eq}} = 1/3 \sum U_{ij} a_i^* a_j^* a_i a_j (\text{\AA}^2)$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
C(A)	0.4096(3)	0.5948(6)	0.3183(2)	0.0365(15)
C(1A)	0.4282(3)	0.4664(5)	0.3935(2)	0.0330(14)
C(2A)	0.4175(3)	0.5774(5)	0.3724(2)	0.0323(14)
C(3A)	0.4088(3)	0.6713(5)	0.4019(2)	0.0347(15)
C(4A)	0.4091(3)	0.6598(5)	0.4521(2)	0.0347(15)
C(5A)	0.4174(3)	0.5469(5)	0.4713(2)	0.0329(14)
C(6A)	0.4257(3)	0.4497(5)	0.4431(2)	0.0314(14)
C(7A)	0.4300(3)	0.3289(5)	0.4656(2)	0.0340(15)
C(8A)	0.3995(3)	0.7626(5)	0.4832(2)	0.0379(16)
C(9A)	0.4217(4)	0.8739(6)	0.4713(3)	0.0460(17)
C(10A)	0.4120(4)	0.9703(7)	0.4997(3)	0.058(2)
C(11A)	0.3818(5)	0.9587(7)	0.5403(4)	0.065(2)
C(12A)	0.3613(5)	0.8504(8)	0.5529(3)	0.062(2)
C(13A)	0.3697(4)	0.7521(6)	0.5248(3)	0.0476(18)
C(14A)	0.4987(4)	0.3179(7)	0.3705(3)	0.0498(19)
C(15A)	0.5350(3)	0.3372(6)	0.3286(3)	0.0465(18)
C(16A)	0.6419(5)	0.3282(11)	0.3044(4)	0.091(3)
C(17A)	0.7086(5)	0.3171(11)	0.3267(4)	0.101(4)
C(1B)	0.3118(3)	0.2966(5)	0.4733(2)	0.0318(15)
C(2B)	0.3763(3)	0.3095(4)	0.4959(2)	0.0303(14)
C(3B)	0.3860(3)	0.3116(5)	0.5464(2)	0.0365(17)
C(4B)	0.3356(3)	0.3032(5)	0.5748(2)	0.0364(16)
C(5B)	0.2723(3)	0.2914(5)	0.5503(2)	0.0367(15)
C(6B)	0.2592(3)	0.2900(5)	0.4999(2)	0.0327(15)
C(7B)	0.1890(3)	0.2911(5)	0.4766(2)	0.0316(14)
C(8B)	0.3468(3)	0.3097(5)	0.6287(2)	0.0372(15)
C(9B)	0.4047(4)	0.3580(8)	0.6532(3)	0.063(2)
C(10B)	0.4152(5)	0.3600(9)	0.7036(3)	0.074(3)
C(11B)	0.3690(4)	0.3159(7)	0.7310(3)	0.060(2)
C(12B)	0.3124(4)	0.2745(8)	0.7075(3)	0.063(2)
C(13B)	0.3015(4)	0.2710(7)	0.6570(3)	0.056(2)
C(14B)	0.2670(3)	0.2167(5)	0.3950(2)	0.0363(16)
C(15B)	0.3109(4)	0.1294(6)	0.3747(3)	0.0519(19)
C(16B)	0.3078(6)	-0.0317(11)	0.3209(6)	0.160(7)
C(17B)	0.2718(5)	-0.1236(8)	0.3083(4)	0.090(3)
C(C)	0.0974(3)	0.1698(5)	0.4499(2)	0.0308(14)
C(1C)	0.0769(3)	0.3080(4)	0.3776(2)	0.0260(13)
C(2C)	0.0886(3)	0.1949(5)	0.3961(2)	0.0267(13)
C(3C)	0.0948(3)	0.1048(5)	0.3633(2)	0.0288(13)
C(4C)	0.0905(3)	0.1234(5)	0.3140(2)	0.0287(13)
C(5C)	0.0813(3)	0.2383(5)	0.2973(2)	0.0301(14)
C(6C)	0.0753(3)	0.3321(4)	0.3283(2)	0.0265(13)
C(7C)	0.0711(3)	0.4565(5)	0.3084(2)	0.0264(13)
C(8C)	0.0975(3)	0.0257(5)	0.2798(2)	0.0329(15)
C(9C)	0.0798(3)	-0.0876(6)	0.2912(3)	0.0440(17)
C(10C)	0.0885(4)	-0.1802(7)	0.2590(3)	0.057(2)
C(11C)	0.1132(4)	-0.1602(7)	0.2170(3)	0.059(2)
C(12C)	0.1291(4)	-0.0484(7)	0.2046(3)	0.057(2)
C(13C)	0.1222(4)	0.0444(6)	0.2364(3)	0.0459(18)
C(14C)	0.0091(3)	0.4511(6)	0.4078(2)	0.0373(16)
C(15C)	-0.0250(3)	0.4130(5)	0.4502(2)	0.0337(15)
C(16C)	-0.1275(4)	0.4115(8)	0.4796(4)	0.063(2)

Table 2. Continued

	<i>x</i>	<i>y</i>	<i>z</i>	<i>Ueq</i>
C(17C)	-0.1974(4)	0.4089(9)	0.4577(4)	0.089(3)
C(1D)	0.1909(3)	0.4837(4)	0.3003(2)	0.0274(14)
C(2D)	0.1256(3)	0.4807(4)	0.2779(2)	0.0266(13)
C(3D)	0.1138(3)	0.4981(5)	0.2284(2)	0.0295(15)
C(4D)	0.1628(3)	0.5180(4)	0.2003(2)	0.0277(14)
C(5D)	0.2274(3)	0.5148(5)	0.2230(2)	0.0332(14)
C(6D)	0.2423(3)	0.4961(5)	0.2728(2)	0.0292(14)
C(7D)	0.3132(3)	0.4847(5)	0.2937(2)	0.0335(15)
C(8D)	0.1496(3)	0.5401(5)	0.1467(2)	0.0306(14)
C(9D)	0.1954(3)	0.5083(6)	0.1163(2)	0.0394(16)
C(10D)	0.1829(4)	0.5270(6)	0.0669(2)	0.0425(17)
C(11D)	0.1244(4)	0.5774(6)	0.0468(3)	0.0431(18)
C(12D)	0.0792(4)	0.6112(5)	0.0765(2)	0.0399(17)
C(13D)	0.0909(3)	0.5915(5)	0.1257(2)	0.0361(15)
C(14D)	0.2381(3)	0.5507(5)	0.3787(2)	0.0350(15)
C(15D)	0.1957(4)	0.6480(6)	0.3921(3)	0.0474(18)
C(16D)	0.2015(5)	0.8212(11)	0.4386(7)	0.205(11)
C(17D)	0.2357(5)	0.9131(7)	0.4450(4)	0.076(3)
O(A)	0.34205(19)	0.5985(3)	0.29812(14)	0.0345(10)
O(1A)	0.4359(2)	0.3718(4)	0.36330(16)	0.0419(11)
O(2A)	0.5117(3)	0.3628(6)	0.28863(19)	0.0745(18)
O(3A)	0.5986(3)	0.3212(5)	0.34187(19)	0.0641(16)
O(1B)	0.30332(19)	0.3036(3)	0.42281(14)	0.0343(10)
O(2B)	0.3685(3)	0.1169(5)	0.3869(3)	0.083(2)
O(3B)	0.2749(3)	0.0643(5)	0.3413(2)	0.0787(19)
O(C)	0.16503(19)	0.1736(3)	0.47082(14)	0.0329(10)
O(1C)	0.07219(19)	0.3990(3)	0.41054(13)	0.0310(9)
O(2C)	0.0007(2)	0.3697(4)	0.48705(16)	0.0444(11)
O(3C)	-0.0888(2)	0.4380(4)	0.44101(18)	0.0484(12)
O(1D)	0.20161(19)	0.4637(3)	0.35014(13)	0.0302(9)
O(2D)	0.1398(3)	0.6668(5)	0.3761(3)	0.081(2)
O(3D)	0.2304(3)	0.7159(5)	0.4238(3)	0.092(2)
C(1S)	0.0654(3)	0.2845(6)	0.0799(3)	0.0509(19)
C(1)	0.1070(2)	0.2202(3)	0.13025(11)	0.1358(15)
C(2)	-0.01407(14)	0.3181(4)	0.08474(17)	0.1551(19)
C(2S)	0.4531(5)	0.4305(10)	0.1789(4)	0.095(3)
C(3)	0.4022(2)	0.5442(4)	0.18535(15)	0.1449(15)
C(4)	0.52001(19)	0.4687(4)	0.15486(17)	0.1559(16)

are mainly as expected for the type of bonds involved. The dihedral angles between the four benzene rings and the mean plane of the 20 atoms-membered macrocyclic ring are 88, 142, 86 and 151° for ring A to D, respectively. Successive dihedral angles between adjacent benzene rings are 87, 87, 85 and 84° for A/B to D/A, respectively. As shown in Figure 2, benzene ring A is up, ring C is down, B and D rings are flat with respect to the plane of the macrocyclic ring. Among the four ethyl ester groups, two ester groups bonded ring A and C point outwards, and other two groups attached ring B and D point inwards the macrocyclic ring, and these partly fill the cavity of the molecule and also could major part in flattened benzene units.

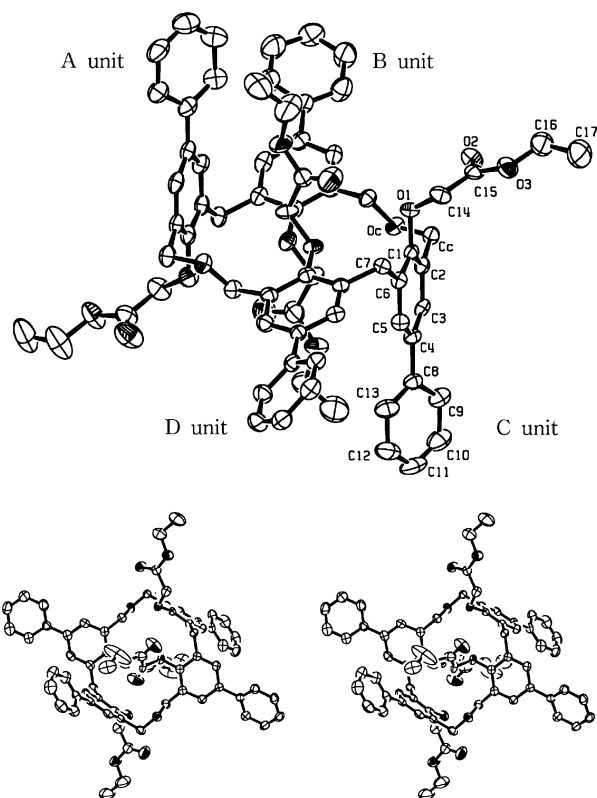


Figure 2. ORTEP²⁰ drawings showing the conformation and the atomic numbering scheme. From upper right, four units are designated A, B, C and D, clockwise.

Experimental Section

Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. Melting points of all compounds were taken in sealed and evacuated capillary tubes on an Mel-temp apparatus and were not corrected. IR spectra were determined on a Nicolet Impact 400 FT-IR spectrometer as KBr pellet. ¹H and ¹³C NMR spectra were recorded on Bruker AMX 500 instrument (OCRC, Seokang University). Chemical shifts are recorded as δ values in parts per million relative to TMS (δ 0.0) as an internal standard. TLC analyses were carried out on silica gel plates (absorbent thickness 250 μ m). Flash chromatography was carried out with E. Merck silica gel (230-400 mesh ASTM). Elution rate was 2 in/min.

3-(3-Hydroxymethyl-5-phenylsalicyl)-5-phenyl-2-hydroxybenzyl alcohol 3 was prepared in 55% yield following the published procedure.¹³

7,13,21,27-Tetra-phenyl-29,30,31,32-tetrahydroxy-2,3,16,17-tetrahydro-3,17-dioxacalix[4]arene 3 was prepared in 79% yield from bishydroxymethylated dimer of *p*-phenylphenol as described elsewhere⁷; mp 236-237 °C (lit⁷ 236-237 °C).

7,13,21,27-Tetra-phenyl-29,30,31,32-tetrakis(ethoxycarbonyl)methoxy-2,3,16,17-tetrahydro-3,17-dioxacalix[4]arene 4. K₂CO₃ method: A solution of compound **3** (1.00 g, 1.27 mmole) in acetone (130 mL) was treated with 700 mg (4 mole equivalent of **3**) of anhydrous K₂CO₃. To this sus-

Table 3. Selected Molecular Geometric Parameters

	A	B	C	D
Bond Length (Å)				
C(1)-O(1)	1.392(7)	1.392(7)	1.398(6)	1.392(6)
O(1)-C(14)	1.424(8)	1.412(7)	1.425(7)	1.425(7)
C(14)-C(15)	1.480(11)	1.507(10)	1.513(10)	1.495(9)
C(15)-O(2)	1.186(8)	1.200(9)	1.197(7)	1.199(8)
C(15)-O(3)	1.327(8)	1.338(9)	1.337(7)	1.314(8)
O(3)-C(16)	1.460(11)	1.445(11)	1.452(11)	1.429(10)
C(16)-C(17)	1.438(12)	1.309(13)	1.489(12)	1.267(13)
C(2A)-C(A)	1.503(9)	C(A)-O(A)	1.432(7)	O(A)-C(7D)
C(2C)-C(C)	1.510(8)	C(C)-O(C)	1.439(7)	O(C)-C(7B)
Bond Angle (°)				
C(2A)-C(A)-O(A)	111.4(5)	C(A)-O(A)-C(7D)	112.2(4)	
O(A)-C(7D)-C(6D)	109.0(5)	C(2C)-C(C)-O(C)	112.2(5)	
C(C)-O(C)-C(7B)	112.0(4)	O(C)-C(7B)-C(6B)	109.7(4)	
Torsion Angle (°)				
C(1)-O(1)-C(14)-C(15)	-111.6(7)	-100.9(7)	105.5(6)	85.7(7)
O(1)-C(14)-C(15)-O(2)	14.2(11)	-20.7(11)	17.0(9)	-11.7(11)
O(1)-C(14)-C(15)-O(3)	159.6(6)	-167.7(6)	-164.6(5)	171.1(6)
C(14)-C(15)-O(3)-C(16)	175.6(8)	-174.5(9)	-177.1(6)	174.2(10)
C(15)-O(3)-C(16)-C(17)	175.8(9)	148.0(12)	-162.3(6)	-141.1(14)
C(1A)-C(2A)-C(A)-O(A)	96.4(7)	C(2A)-C(A)-O(A)-C(7D)	77.7(7)	
C(A)-O(A)-C(7D)-C(6D)	179.4(5)	O(A)-C(7D)-C(6D)-C(1D)	-105.0(6)	
C(7D)-C(6D)-C(1D)-C(2D)	-172.0(5)	C(6D)-C(1D)-C(2D)-C(7C)	74.7(5)	
C(1D)-C(2D)-C(7C)-C(6C)	-66.7(6)	C(2D)-C(7C)-C(6C)-C(1C)	126.3(6)	
C(7C)-C(6C)-C(1C)-C(2C)	-172.3(5)	C(6C)-C(1C)-C(2C)-C(C)	173.6(5)	
C(1C)-C(2C)-C(C)-O(C)	-92.1(7)	C(2C)-C(C)-O(C)-C(7B)	75.5(6)	
C(C)-O(C)-C(7B)-C(6B)	178.5(5)	O(C)-C(7B)-C(6B)-C(1B)	96.5(7)	
C(7B)-C(6B)-C(1B)-C(2B)	172.2(5)	C(6B)-C(1B)-C(2B)-C(7A)	-173.5(5)	
C(1B)-C(2B)-C(7A)-C(6A)	70.8(7)	C(2B)-C(7A)-C(6A)-C(1A)	-130.2(6)	
C(7A)-C(6A)-C(1A)-C(2A)	173.4(5)	C(6A)-C(1A)-C(2A)-C(A)	-170.4(5)	

pension ethyl bromoacetate (1.5 mL) was added, then the reaction mixture was refluxed 24 hr. After removal of acetone by evaporation, the residue was acidified with dilute HCl, then extracted with chloroform. The organic layer was collected, washed with water, dried over anhydrous MgSO₄, and evaporated solvent to dryness to yield slightly colored solid which was recrystallized from CH₂Cl₂ and methanol to give 1.31 g (91%) of the product **4** as crystalline solid. **NaH method:** To heated suspension of compound **3** (1.00 g, 1.27 mmole) and NaH (818 mg, 60% oil dispersion, 4 mole equivalent per OH group) in dry THF (40 mL) and dry DMF (8 mL), a solution of ethyl bromoacetate (2.0 mL, 18 mmole) in dry THF (10 mL) was added dropwise un Ar and then the reaction mixture was refluxed for overnight. After small amount of ethanol was added to destroy the excess NaH, solvent was removed *in vacuo* and then the organic material was extracted with methylene chloride. The organic layer was washed with water, dried and evaporated to afford slightly brown colored residue, which was recrystallized from CH₂Cl₂ and methanol to produce 1.29 g (90%) of the product **4** as crystalline solid, mp 201-202 °C; IR (KBr) 1753 cm⁻¹; ¹H NMR (CDCl₃, 25 °C) 7.53-7.27 (m, 28, ArH),

5.19 (d, 2, CH₂, *J* = 13.9 Hz), 4.78 (d, 4, CH₂, *J* = 11.0 Hz), 4.55 (d, 4, CH₂, *J* = 11.0 Hz), 4.41 (d, 4, CH₂, *J* = 16.1 Hz), 3.97 (d, 4, CH₂, *J* = 16.1 Hz), 3.87 (q, 8, OCH₂, *J* = 7.2 Hz), 3.45 (d, 2, CH₂, *J* = 13.9 Hz), 0.93 (t, 12, CH₃, *J* = 7.2 Hz); ¹³C NMR (CDCl₃) δ 169.20 (C=O), 156.02, 140.66, 136.98, 135.22, 129.53, 129.18, 128.59, 126.93 (Ar), 71.24, 66.75, 60.39 (CH₂), 31.43 (ArCH₂Ar), 13.77 (CH₃); Anal. Calcd for C₇₀H₆₈O₁₄: C, 74.18; H, 6.06. Found: C, 74.13; H, 5.99.

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