Multibridged [3_n]Cyclophanes, 1

Synthesis of [3_4](1,2,3,5)- and -(1,2,4,5)Cyclophanes

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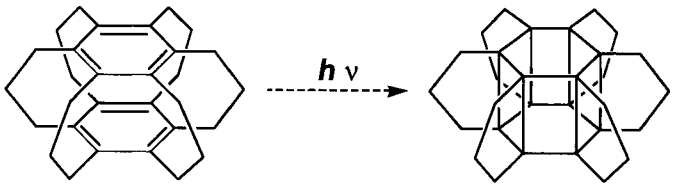
The acid-catalyzed cyclization of a pseudogeminally substituted acetyl group and a chloromethyl group of tri-bridged cyclophane **7**, followed by reduction of the carbonyl group afforded [3_4](1,2,3,5)cyclophane **3**. One-step TosMIC coupling reaction of tetrabromide **12** and subsequent reduction of the carbonyl groups provided an isomer of **3**, [3_4](1,2,4,5)cyclophane **4**.

[3_6](1,2,3,4,5,6)Cyclophane **1** is one of the final target molecules in the field of [3.3]cyclophane chemistry[1]. This compound is a promising precursor of propella[3_6]prismane **2**[2] since photochemical isomerization of **1** to **2** has been predicted based on molecular mechanics calculations (Scheme 1)[3]. Hexaprismane is a theoretically and structurally intriguing compound, but its synthesis has not been accomplished yet[4]. Therefore, if we succeeded in the synthesis of **1**, we would have a good chance of obtaining the first hexaprismane derivative **2**. The stereochemical features of **1** are also fascinating; six trimethylene chains are expected to undergo a correlated inversion process in solution (Scheme 5)[5].

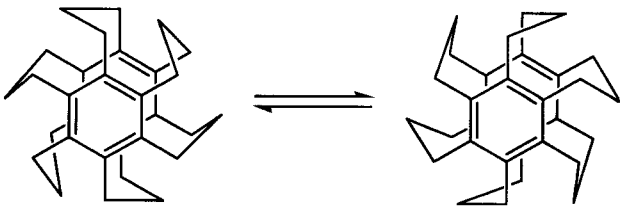
Scheme 1. Predicted photochemical isomerization of **1** to **2**



1 **2**

As a strategy for the synthesis of **1**, we chose the stepwise approach; stepwise introduction of additional trimethylene bridges into [3_3](1,3,5)cyclophane **5**[5], as a starting compound, might allow the preparation of the hexa-bridged cyclophane **1** by way of tetra- and penta-bridged cyclophanes, if we succeeded in developing an efficient method for introducing a trimethylene bridge. In our efforts along these lines, we have synthesized the first tetra-bridged [3.3]cyclophanes, [3_4](1,2,3,5)- and -(1,2,4,5)cyclophanes **3** and **4** (Scheme 3). We describe here their synthesis, conformation, and transannular π-π interaction.

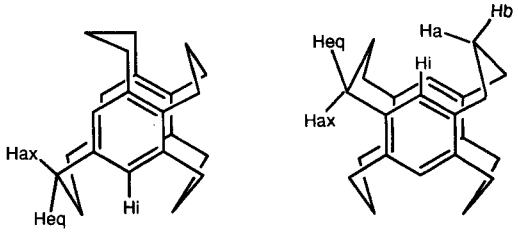
Scheme 2. Expected correlated inversion process of the six trimethylene bridges



1a **1b**

Acetylation of **5** with acetyl chloride in CH_2Cl_2 at −50°C in the presence of $AlCl_3$ afforded monoacetylated compound **6** (71%) with a small amount of a pseudogeminally diacetylated compound (9%). Taking advantage of the directing effect of the acetyl group of **6**[6], we were able to introduce a chloromethyl group into a pseudogeminal position ($ClCH_2OCH_3$, $AlCl_3$, room temp.). In this reaction, however, a mixture of the desired **7a** and methyl ether **7b**, which was

Scheme 3. Stable conformers of **3** and **4**



3 **4**

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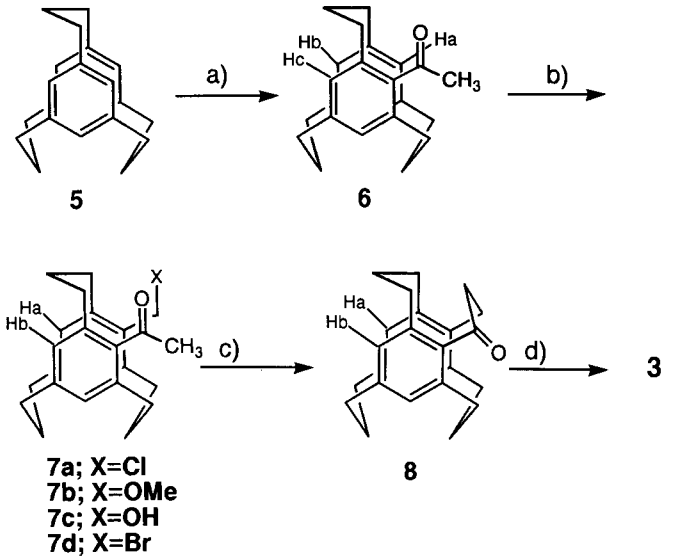
formed by the reaction of **7a** with in situ generated MeOH, was obtained. The chloride **7a** was relatively unstable and partially decomposed to alcohol **7c** during aqueous workup.

Scheme 4. Synthetic route to **3**



a) CH_3COCl, $AlCl_3$, CH_2Cl_2, −50°C, 71%. — b) $ClCH_2COCH_3$, $AlCl_3$, room temperature. — c) 30% HBr, CH_3COOH, reflux, 1 d, 53% based on **6**. — d) $NH_2NH_2 \cdot H_2O$, $NH_2NH_2 \cdot HCl$, KOH, $HO(CH_2)_2O(CH_2)_2OH$, 76%.

For the coupling reaction between a pseudogeminally substituted acetyl group and a chloromethyl group[7], we tried several attempts and finally found that an acid-catalyzed reaction effected the cyclization; refluxing of a mixture of **7a**, **7b**, and **7c** in 30% HBr in AcOH for a day yielded the desired tetra-bridged ketone **8** (yield 53% based on **6**). In this cycloalkylation reaction of an enol, HBr served as the catalyst and the bromination agent of **7b** and **7c** to furnish bromomethyl compound **7d**, which was more reactive than **7a**. The Wolff-Kishner reduction of the carbonyl group of **8** afforded [3$_4$]cyclophane **3** (76%).

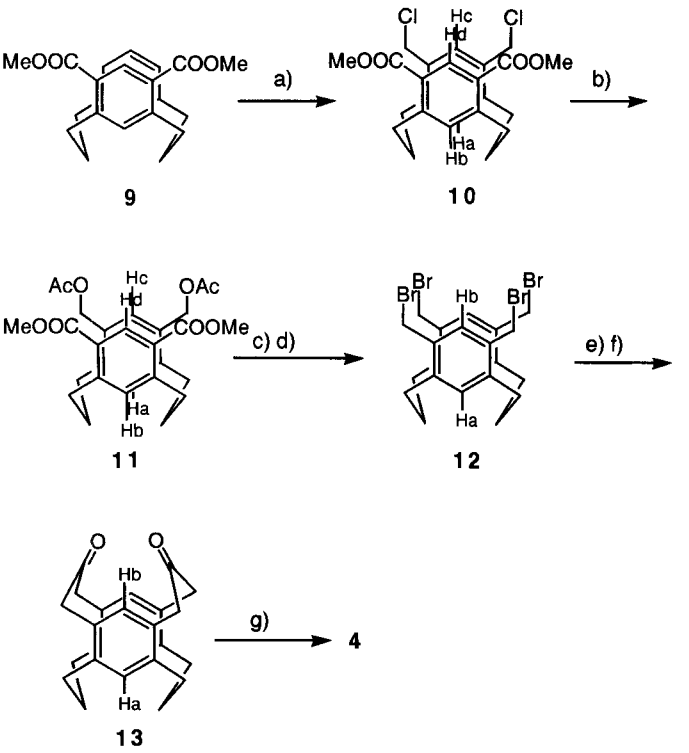
A similar directing effect of a methoxycarbonyl group of dimethyl [3.3]metacyclophane-5,7-dicarboxylate (**9**)[8] led to the formation of pseudogeminally substituted bis(chloromethyl) compound **10**[9,10]. Conversion of the chlorine atoms to the acetoxy groups, followed by $LiAlH_4$ reduction and bromination of the resultant tetraol with PBr_3 in $CHCl_3$ afforded tetrakis(bromomethyl)[3.3]metacyclophane **12**[10], a versatile precursor of various macrocyclic compounds, for instance of cyclic oligomers of [3.3]metacyclophane units[11]. *One-step TosMIC* (*p*-tolylsulfonylmethyl isocyanide) coupling reaction of **12** and subsequent acid hydrolysis afforded diketone **13** (24%)[12], which was converted into **4** by Wolf-Kishner reduction (97%).

A sharp singlet of the aromatic protons (H_i) at $\delta = 6.90$ and well-averaged spectra of the bridge protons of **4** (25°C; $CDCl_3$) suggested the mobile nature of this compound. Its NOE experiment at 25°C revealed the most stable conformation to be a boat-boat (Scheme 3). In compound **3**, a sharp singlet of the aromatic proton signal (H_i) at $\delta = 6.62$ (25°C; $CDCl_3$) as well as averaged benzylic proton signals (H_{eq} and H_{ax}) of the isolated trimethylene bridge in the 5-position suggested a rapid inversion of the trimethylene bridge[13].

The electronic spectra of **3** and **4** in CH_2Cl_2 showed the strong transannular π-π interaction; the λ_{max} value of **3** (321 nm; $\varepsilon = 60$) and **4** (331 nm; $\varepsilon = 89$) showed significant bathochromic shifts as compared with [3.3]paracyclophane[14] (296 nm; $\varepsilon = 42$) and the tri-bridged compound **5** (310 nm; $\varepsilon = 23$). This was further confirmed by the charge-transfer (CT) interaction with tetracyanoethylene (TCNE). Compounds **3** and **4** showed their CT bands at $\lambda = 630$ nm ($\varepsilon = 2670$) and 637 nm ($\varepsilon = 2670$) in CH_2Cl_2, respectively, whereas the corresponding complexes of [3.3]paracyclophane and **5** exhibited their CT bands at $\lambda = 601$ nm ($\varepsilon = 101$) and 585 nm ($\varepsilon = 706$). In the case of **4**, a 1:1 complex was isolated as purple needles[9]. These results indicated that the π-π interaction was considerably dependent on the number and substitution pattern of the bridges, and **4** showed the strongest CT interaction with TCNE among the [*m.n*]cyclophanes and multibridged cyclophanes.

The acid-catalyzed cyclization reaction described above is an especially efficient method for introducing a trimethylene bridge into a very crowded position since only the least

Scheme 5. Synthetic route to **4**



a) $ClCH_2OCH_3$, $AlCl_3$, +5°C to ca. room temp., 100%. — b) CH_3COOAg, CH_3COOH, reflux, 86%. — c) $LiAlH_3$, THF, reflux. — d) PBr_3, $CHCl_3$, reflux, 63% based on **11**. — e) TosMIC, nBu$_3$NI, NaOH, H_2O/CH_2Cl_2. — f) concd. HCl, CH_2Cl_2, room temp., 26% based on **12**. — g) $NH_2NH_2 \cdot H_2O$, $NH_2NH_2 \cdot HCl$, KOH, $HO(CH_2)_2O(CH_2)_2OH$, 97%.

bulky proton is needed for the reaction. The application of this method to the synthesis of [3$_5$](1,2,3,4,5)cyclophane is now in progress.

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Experimental

IR: Jasco IR-700, Perkin-Elmer 1640 FT-IR. – ^{1}H NMR: Hitachi R-20B, Jeol JNM-EX 270, and JNM-GSX 400; chemical shifts (δ values) relative to TMS (solvent $CDCl_3$). – MS: Shimadzu QP-1000EX and CONCEPT 1S (ionization energy 70 eV). – UV: Shimadzu UV-2100. – Column chromatography: Daiso gel IR-60 (40–63 μm). – TLC: Silica gel 60 F_{254} Merck for analytical purposes, silica gel 60 PF_{254} Merck for preparative purposes. – Elemental analyses: Service Center of the Elementary Analysis of Organic Compounds affiliated to the Faculty of Science, Kyushu University. – [3.3.3](1,3,5)Cyclophane (**5**)[5c] and dimethyl [3.3]metacyclophane-5,7-dicarboxylate (**9**)[8] were prepared according to the previously reported procedures.

5-Acetyl[3.3.3](1,3,5)cyclophane (**6**): $AlCl_3$ (3.40 g, 25.5 mmol) was dissolved in CH_2Cl_2 (200 ml) with stirring at room temp. Then acetyl chloride (3.50 ml, 49.2 mmol) was added, and the mixture was cooled to −50°C with a dry ice/acetone bath. To the mixture was added a solution of **5** (1.99 g, 7.20 mmol) in CH_2Cl_2 (70 ml) over a period of 10 min. After the mixture had been stirred for 1.5 h at −55 to −50°C, it was poured into a mixture of ice and concd. HCl. The organic portion was separated, washed with brine, dried with $MgSO_4$, and filtered. Removal of the filtrate afforded colorless crystalline solid, which was chromatographed on silica gel (60 g) with toluene to give recovered **5** (0.537 g, 27%, $R_f = 0.93$), desired **6** (1.186 g, 71% based on recovered **5**, $R_f = 0.47$), and the 5,14-diacetyl compound (0.164 g, 9%, $R_f = 0.00$).

6: Colorless plates from benzene, m.p. 166.5–167°C. – IR (KBr): $\tilde{\nu} = 1689$ cm^{-1} (C=O). – ^{1}H NMR: δ = 2.11–2.26 (m, 6H, $CH_2CH_2CH_2$), 2.29 (s, 3H, $COCH_3$), 2.58–2.78 (m, 12H, $CH_2CH_2CH_2$), 6.56 (s, 2H, ArH, H_b), 6.60 (s, 2H, ArH, H_c), 6.91 (s, 1H, ArH, H_a). – MS: $m/z = 318$ [M$^+$]. – $C_{23}H_{26}O$ (318.5): calcd. C 86.75, H 8.23; found C 86.38, H 8.20.

5,14-Diacetyl Compound: Colorless crystals from CH_2Cl_2/MeOH, m.p. 174–175°C. – IR (KBr): $\tilde{\nu} = 1675$ cm^{-1} (C=O). – ^{1}H NMR: δ = 2.21–2.40 (m, 6H, $CH_2CH_2CH_2$), 2.25 (s, 6H, $COCH_3$), 2.65–2.77 (m, 8H, $CH_2CH_2CH_2$), 3.02–3.13 (m, 4H, $CH_2CH_2CH_2$), 6.68 (s, 4H, ArH). – MS: $m/z = 360$ [M$^+$]. – $C_{25}H_{28}O_2$ (360.5): cacld. C 83.03, H 7.83; found C 83.08, H 7.78.

[3.3.3.3](1,2,3,5)Cyclophane (**3**): To a mixture of $AlCl_3$ (90.0 mg, 0.67 mmol) and $ClCH_2OCH_3$ (5.0 ml) was added dropwise a solution of **6** (200 mg, 0.63 mmol) in $ClCH_2OCH_3$ (2.0 ml) for 1 h at room temp. The reaction mixture was poured into ice/water (20 ml), stirred for 1 h, and extracted with CH_2Cl_2 (3 × 15 ml). The combined extracts were washed with water, dried with $MgSO_4$, and filtered. The filtrate was concentrated to dryness to give a mixture (210 mg) of desired chloride **7a** {R_f [silica gel, CH_2Cl_2/AcOEt (10:1)] = 0.78}, methyl ether **7b** ($R_f = 0.67$), and alcohol **7c** ($R_f = 0.28$), which was used in the next reaction without further purification.

7a: Colorless crystals from hexane/CH_2Cl_2, m.p. 123–124°C. – IR (KBr): $\tilde{\nu} = 1685$ cm^{-1} (C=O). – ^{1}H NMR: δ = 2.12–2.26 (m, 6H, $CH_2CH_2CH_2$), 2.21 (s, 3H, $COCH_3$), 2.64–2.85 (m, 10H, $CH_2CH_2CH_2$), 3.11–3.21 (m, 2H, $CH_2CH_2CH_2$), 5.09 (s, 2H, CH_2Cl), 6.60 (s, 2H, ArH, H_a), 6.68 (s, 2H, ArH, H_b). – MS: $m/z = 366$ [M$^+$]. – $C_{24}H_{27}ClO$ (366.9): calcd. C 78.56, H 7.42; found C 78.44, H 7.45.

7b: Colorless crystals. – IR (KBr): $\tilde{\nu} = 1685$ cm^{-1} (C=O), 1086 (C–O). – ^{1}H NMR: δ = 2.04–2.26 (m, 6H, $CH_2CH_2CH_2$), 2.19 (s, 3H, $COCH_3$), 2.62–2.86 (m, 10H, $CH_2CH_2CH_2$), 3.17 (s, 3H, CH_2OCH_3), 3.11–3.39 (m, 2H, $CH_2CH_2CH_2$), 4.77 (s, 2H, CH_2OCH_3), 6.56 (s, 2H, ArH, H_a), 6.66 (s, 2H, ArH, H_b). – MS: $m/z = 362$ [M$^+$].

7c: Colorless crystals. – IR (KBr): $\tilde{\nu} = 3442$ cm^{-1} (O–H), 1682 (C=O). – ^{1}H NMR: δ = 1.98–2.35 (m, 6H, $CH_2CH_2CH_2$), 2.19 (s, 3H, $COCH_3$), 2.50–3.37 (m, 12H, $CH_2CH_2CH_2$), 4.90 (s, 2H, CH_2OH), 6.59 (s, 2H, ArH, H_a), 6.67 (s, 2H, ArH, H_b). – MS: $m/z = 348$ [M$^+$].

To a solution of the crude mixture (100 mg) in AcOH (5 ml) was added 30% HBr in AcOH (10 ml) at room temp., and the mixture was refluxed for 1 d with stirring. After cooling, the mixture was poured into ice/water (20 ml), extracted with CH_2Cl_2 (3 × 20 ml), and the combined organic portions were washed with water, dried with $MgSO_4$, and filtered. Removal of the solvent and purification of the residue by preparative TLC (silica gel) with CH_2Cl_2 provided cyclic ketone **8** (47.5 mg, 53% based on **6**) as colorless needles, m.p. 295–297°C (sealed tube; hexane), R_f(silica gel; CH_2Cl_2) = 0.53. – IR (KBr): $\tilde{\nu} = 1675$ cm^{-1} (C=O). – ^{1}H NMR: δ = 2.13–2.38 (m, 6H, $CH_2CH_2CH_2$), 2.65–2.86 (m, 10H, $CH_2CH_2CH_2$), 2.95–3.06 (m, 2H, $CH_2CH_2CH_2$), 3.18 (t, $J = 7.4$ Hz, 2H, CH_2COAr), 3.35 (t, $J = 7.3$ Hz, 2H, CH_2Ar), 6.62 (s, 2H, ArH, H_a), 6.66 (s, 2H, ArH, H_b). – MS: $m/z = 330$ [M$^+$]. – $C_{24}H_{26}O$ (330.5): calcd. C 87.23, H 7.93; found C 87.03, H 7.92.

A mixture of **8** (420 mg, 1.27 mmol), 100% hydrazine hydrate (8 ml), hydrazine dihydrochloride (2.10 g), and diethylene glycol (50 ml) was heated at 130–140°C for 28 h with stirring. KOH (4.5 g) was added, and the temperature of the reaction mixture was gradually raised to ca. 220°C and then to the boiling point for 3.5 h. After cooling, the mixture was poured into dild. HCl solution, extracted with CH_2Cl_2 (200 ml), and the combined CH_2Cl_2 solutions were washed with water, dried with $MgSO_4$, and concentrated. The residue was passed through a short silica gel column with CH_2Cl_2 to afford **3** as colorless crystals (305 mg, 76%), m.p. 254.5–255°C (hexane), R_f(silica gel; CH_2Cl_2) = 0.94. – ^{1}H NMR: δ = 2.07–2.53 (m, 8H, $CH_2CH_2CH_2$), 2.68–2.77 (m, 8H, $CH_2CH_2CH_2$), 3.06–3.23 (m, 8H, $CH_2CH_2CH_2$), 6.62 (s, 4H, ArH, H_i). – MS: $m/z = 316$ [M$^+$]. – $C_{24}H_{28}$ (316.5): calcd. C 91.08, H 8.92; found C 90.82, H 8.87.

5,7,14,16-Tetrakis(bromomethyl)[3.3]metacyclophane (**12**): To an ice-cooled mixture of $AlCl_3$ (56.0 g, 420 mmol) and $ClCH_2OCH_3$ (50 ml) was added diester **9** (1.85 g, 5.25 mmol), dissolved in $ClCH_2OCH_3$ (30 ml), over a period of 20 min with vigorous stirring. During the addition, the temp. of the reaction mixture was kept below +5°C with an ice bath. After the addition, the resulting wine-red solution was stirred at +5°C for 1 h, then allowed to warm to room temp. Stirring was continued for an additional 2 h at room temp. The reaction mixture was carefully poured into ice (ca. 600 ml), stirred for 20 min, and extracted with CH_2Cl_2 (2 × 150 ml). The combined extracts were washed with brine, dried with $MgSO_4$, and filtered. Removal of the solvent afforded a yellow solid, which was purified by silica gel chromatography (50 g) with CH_2Cl_2 to provide bis(chloromethyl) compound **10** (2.36 g, 100%) as colorless crystals, m.p. 146–148°C (benzene/hexane), R_f(silica gel; CH_2Cl_2) = 0.64. – ^{1}H NMR: δ = 2.1–2.3 (m, 4H, $CH_2CH_2CH_2$), 2.8–3.0 (m, 4H, $CH_2CH_2CH_2$), 3.1–3.4 (m, 4H, $CH_2CH_2CH_2$), 3.83 (s, 6H, $COOCH_3$), 4.41 (s, 4H, CH_2Cl), 6.98 (s, 1H, ArH, H_a), 7.03 (s, 1H, ArH, H_b), 7.21 (s, 1H, ArH, H_c), 8.15 (s, 1H, ArH, H_d). –

MS: $m/z = 376$ [$M^+ - 2$ HCl]. – $C_{24}H_{26}Cl_2O_4$ (449.4): calcd. C 64.15, H 5.83; found C 64.07, H 5.76.

A mixture of **10** (2.36 g, 5.25 mmol), AgOAc (4.20 g, 31.5 mmol), and AcOH (100 ml) was refluxed for 17 h with stirring. After cooling, the mixture was filtered and the solid washed with CH_2Cl_2 (200 ml). The combined filtrate and washings were concentrated, water (150 ml) was added, and the mixture extracted with CH_2Cl_2 (2 × 100 ml). The combined extracts were washed successively with satd. aqueous $NaHCO_3$ solution and brine, dried with $MgSO_4$, filtered, and concentrated to afford a pale yellow solid. Purification of the crude product by silica gel chromatography (51 g) with CH_2Cl_2/AcOEt (10:1) afforded diacetate **11** (2.24 g, 86%) as colorless crystals, m.p. 127.5–129°C (benzene/hexane), R_f[silica gel; CH_2Cl_2/AcOEt (10:1)] = 0.56. – ^{1}H NMR: δ = 2.05 (s, 6H, $OCOCH_3$), 2.01–2.11 (m, 4H, $CH_2CH_2CH_2$), 2.85 (m, 4H, $CH_2CH_2CH_2$), 3.19 (m, 4H, $CH_2CH_2CH_2$), 3.86 (s, 6H, $COOCH_3$), 4.92 (s, 4H, CH_2OCOCH_3), 6.94 (s, 1H, ArH, H_a), 6.99 (s, 1H, ArH, H_b), 7.15 (s, 1H, ArH, H_c), 8.11 (s, 1H, ArH, H_d). – MS: $m/z = 437$ [M^+ – OAc]. – $C_{28}H_{32}O_8$ (496.6): calcd. C 67.73, H 6.50; found C 67.77, H 6.46.

To a refluxing mixture of $LiAlH_4$ (3.50 g, 92.2 mmol) and THF (200 ml) was added acetate **11** (2.05 g, 4.12 mmol), dissolved in THF (60 ml), over a period of 10 min with vigorous stirring. After the addition, the mixture was stirred for an additional 13 h at reflux. The oil bath was removed, and excess $LiAlH_4$ was decomposed by the careful addition of AcOEt (10 ml) and water (30 ml). The precipitate was filtered, and the filtrate was concentrated to dryness. The combined precipitate and concentrate were Soxhlet-extracted with EtOH for 6 h to afford the tetraol as colorless powder, which was used for the next reaction without further purification.

To a stirred mixture of the crude tetraol and $CHCl_3$ (150 ml) was added PBr_3 (6.00 ml, 63.8 mmol) in one portion at room temperature. After the addition, the mixture was refluxed for 12 h. The still warm solution was decanted, and the $CHCl_3$ solution was washed successively with satd. aqueous $NaHCO_3$ solution and brine, and dried with $MgSO_4$. This solution was directly passed through a silica gel column (34 g). The eluate was concentrated to dryness, and the residue was recrystallized from CH_2Cl_2/hexane to afford tetrabromide **12** (1.57 g, 63% based on **11**) as colorless prisms, m.p. 194.5–195°C, R_f(silica gel; CH_2Cl_2) = 0.88. – ^{1}H NMR: δ = 2.1–2.3 (m, 4H, $CH_2CH_2CH_2$), 2.8–3.1 (m, 8H, $CH_2CH_2CH_2$), 4.39 (s, 8H, CH_2Br), 6.99 (s, 2H, ArH, H_a), 7.04 (s, 2H, ArH, H_b). – MS: $m/z = 608$ [M^+]. – $C_{22}H_{24}Br_4$ (608.1): calcd. C 43.46, H 3.98; found C 43.58, H 3.99.

[3.3.3.3](1,2,4,5)Cyclophane (**4**): To a refluxing mixture of nBu_4NI (320 mg), NaOH (6 g), dissolved in water (10 ml), and CH_2Cl_2 (200 ml) was added dropwise a mixture of **12** (100 mg, 0.16 mmol) and TosMIC (128 mg, 0.66 mmol) in CH_2Cl_2 (50 ml) over a period of 3 h with vigorous stirring. After the addition, the mixture was stirred for an additional 2 h. After cooling, the mixture was washed with water (3 × 200 ml) and concentrated to a volume of ca. 10 ml. To the concentrate was added concd. HCl (15 ml), and the mixture was stirred at room temp. for 12 h. The mixture was washed with brine, dried with $MgSO_4$, and filtered. The filtrate was concentrated and the concentrate was purified by preparative TLC (silica gel; CH_2Cl_2; R_f = 0.21) to give diketone **13** as pale yellow crystals (13.3 mg, 24%). Recrystallization of the crude product from MeOH/CH_2Cl_2 afforded colorless plates, m.p. 293–293.5°C. – IR (KBr): $\tilde{\nu} = 1701$ cm^{-1} (C=O). – ^{1}H NMR: δ = 2.21–2.30 (m, 4H, $CH_2CH_2CH_2$), 2.75–2.80 (m, 4H, $CH_2CH_2CH_2$), 2.86–2.94 (m, 4H, $CH_2CH_2CH_2$), 3.64 (d, J = 15.1 Hz, 4H, CH_2COCH_2), 4.07 (d, J = 15.1 Hz, 4H, CH_2COCH_2), 7.02 (s, 2H, ArH, H_a), 7.06 (s, 2H, ArH, H_b). – MS: $m/z = 344$ [M^+]. – $C_{24}H_{24}O_2$ (344.5): calcd. 344.4524; found 344.1772 (MS).

A stirred mixture of **13** (45.7 mg, 0.13 mmol), 100% hydrazine hydrate (2 ml), hydrazine dihydrochloride (250 mg), and diethylene glycol (10 ml) was heated at 120°C for 2 h. Then KOH (2 g) was added, and the mixture was heated at 120°C for 2 h and then at 200°C for 3 h. After cooling, the mixture was poured into ice/water, acidified with concd. HCl, and extracted with CH_2Cl_2 (4 × 15 ml). The combined organic portions were washed with water, dried with $MgSO_4$, filtered, and concentrated to afford **4** (40.9 mg, 97%) as colorless crystals. Recrystallization from $ClCH_2CH_2Cl$ provided colorless plates, m.p. >300°C, R_f(silica gel; CH_2Cl_2) = 0.89. – ^{1}H NMR: δ = 2.23–2.27 (m, 8H, $CH_2CH_2CH_2$), 2.64–2.68 (t, J = 4.4 Hz, 8H, $CH_2CH_2CH_2$), 2.98–3.06 (m, 8H, $CH_2CH_2CH_2$), 6.90 (s, 4H, ArH, H_i). – MS: $m/z = 316$ [M^+]. – $C_{24}H_{28}$ (316.5): calcd. C 91.08, H 8.92; found C 90.91, H 8.90.

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