with the sample obtained in the earlier experiment. Lastly, elution with 20% ethyl acetate–hexane gave the unsaturated compound (40 mg, 14%), which was found to be identical with the sample described in the earlier experiment.

Flash Vacuum Pyrolysis of Hexacyclo[7.4.2.0^{1,9}.0^{3,7}.0^{4,14}.0^{6,15}]pentadecane-2,8-dione (4). Hexacyclic propellane 4 (2 g, 8.92 mmol) was slowly sublimed (120 °C (1 torr)) through a quartz tube (1.5 × 30 cm) packed with quartz chips, connected to a vacuum line, and provided with a collection flask and a liquid nitrogen trap. The quartz tube was heated with nichrome wire wound around it and was insulated with asbestos padding. The temperature was controlled by a Variac and was measured by a Chromel–Alumel thermocouple on a Keithley digital multimeter. The quartz tube was preheated and equilibrated at 550 °C. The solid condensate collected in the receiver was dissolved in benzene and charged on a silica gel (30 g) column. Elution with benzene removed minor impurities. Further elution of the column with 10% ethyl acetate–benzene gave 11 (1.4 g, 70%), which was recrystallized from dichloromethane–hexane: mp 158 °C; UV (MeOH) λ_{max} 224 nm (ϵ 11 035); IR (KBr) 1710, 1630 cm^{-1} (cyclopentenone); ^{1}H NMR ($CDCl_3$, 100 MHz) δ 6.9 (s, 2 H), 3.4 (s, 4 H), 2.4–1.0 (m, 10 H); ^{13}C NMR ($CDCl_3$, 25.0 MHz) δ 209.0 (s), 160.3 (d), 148.2 (s), 55.7 (d), 47.8 (d), 31.9 (t), 20.0 (t), 25.6 (t). Anal. Calcd for $C_{15}H_{16}O_2$: C, 78.92; H, 7.06. Found: C, 79.10; H, 7.11.

Photocyclization of Tetracyclo[9.2.1.1^{3,6}.0^{4,13}]pentadeca-6(15),11(14)-diene-5,12-dione (11). A solution of 11 (228 mg, 1 mmol) in 100 mL of ethyl acetate was purged with nitrogen and irradiated with a Hanovia 450-W medium-pressure mercury vapor lamp in a quartz immersion well with use of Pyrex filter for 20 min. The TLC and IR spectrum of the product after removal of solvent were found to be identical with those of hexacyclic dione 4.

Addition of Grignard Reagent to Tetracyclo[9.2.1.1^{3,6}.0^{4,13}]pentadeca-6(15),11(14)-diene-5,12-dione (11). To the methylmagnesium iodide solution prepared from magnesium (125 mg, 5.1 mmol) and methyl iodide (2 mL) in dry ether (25 mL) was added bis-enone 11 (700 mg, 3.07 mmol) in 15 mL of THF through an addition funnel, and the reaction mixture was stirred at room temperature for 3 h. Then the reaction mixture was carefully quenched with saturated ammonium chloride solution and extracted with ether (2 × 25 mL). The organic layer was washed with brine and dried. Removal of solvent gave 12 (640 mg, 85%). Analytically pure 12 was obtained by recrystallization from hexane–carbon tetrachloride: mp 158–159 °C; IR (KBr) 3400, 3050, 1650 cm^{-1}; ^{1}H NMR ($CDCl_3$, 100 MHz) δ 5.36 (s, 1 H), 5.12 (s, 1 H), 3.3–2.8 (m, 6 H), 2.2–1.6 (9 H), 1.4 (s, 3 H); ^{13}C NMR ($CDCl_3$, 25.0 MHz) δ 145.6, 144.5, 136.7, 133.2, 118.1, 95.4, 61.0, 59.6, 48.0, 47.1, 36.2, 29.7, 29.2, 28.8, 27.6, 25.0. Anal. Calcd for $C_{16}H_{20}O_2$: C, 78.65; H, 8.25. Found: C, 78.55; H, 8.35.

Crystal Data for 6a. The crystals belong to the space group $C2/c$ with a = 20.845 (4), Å, b = 6.168 (2) Å, c = 23.320 (3) Å, and β = 90.27 (1)°. The intensity data (2419 reflections) were collected on a CAD4F-11M diffractometer with Mo Kα radiation (0.7107 Å) by using the $\omega/2\theta$ scan technique. The structure was solved by direct methods. Full-matrix refinement of scale factor, positional, and anisotropic thermal parameters (isotropic for H atoms) gave R = 0.063 for 1400 reflections with $|F_o| > 3\sigma|F_o|$.[8]

Crystal Data for 8. The crystals belong to the space group *Pbca* with a = 9.354 (1) Å, b = 11.050 (2) Å, c = 23.761 (4) Å. The intensity data (2146 reflections) were collected on a CAD4F-11M diffractometer with Mo Kα radiation (0.7107 Å) by using the $\omega/2\theta$ scan technique. The structure was solved by direct methods. Full-matrix refinement of scale factor, positional, and anisotropic thermal parameters (isotropic for H atoms) gave R = 0.047 for 1158 reflections with $|F_o| > 3\sigma|F_o|$.[8]

Acknowledgment. K.R.R. and K.S.R. thank CSIR for financial support in the form of JRF. Research at the University of Hyderabad was supported under UGC Special Assistance Programme in Organic Chemistry.

(8) Further details about X-ray crystal structures can be obtained from the NCL group.

Dioxa-1,3-bishomopentaprismane: Synthesis and Transformations

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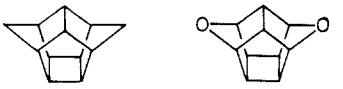
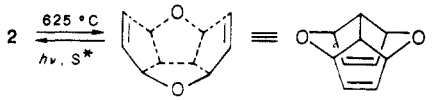
Heterocaged systems have received attention in recent years from synthetic as well as mechanistic considerations.[1] The main motivation for these studies has been the desire to compare the reactivity pattern of carbon caged compounds with their heterologues. We have been investigating the chemistry of bishomopentaprismane (1) and its derivatives[2] and in this context became interested in its dioxa-analogue 2. Herein, we report the synthesis of hexacyclic diether 2 and describe its transformations to some new and structurally related polycyclic diethers.



1 2

Readily available[3] hexacyclic keto ether 3 on treatment with aqueous alkali furnished the hexacyclic carboxylic acid 4 in good yield through Haller–Bauer cleavage and sequential intramolecular displacement of two chlorine atoms with the carboxylate oxygen.[3,4] Hunsdieker reaction on 4 proceeded readily to the bromo-compound 5 (85%). Reductive dehalogenation of 5 with Li–THF–*t*-BuOH[5] proceeded as expected to furnish the dioxabishomopentaprismane 2, mp 271–273 °C, in 70% yield, Scheme I. The structure and C_{2v} symmetry of 2 were clearly revealed through its three-line ^{13}C NMR spectrum with diagnostic resonances at δ 83.5, 56.4, and 41.9.

In view of the previous interesting observations[2,6] with 1 and its derivatives, the dioxa-analogue 2 was also subjected to thermally induced cycloreversion reaction. Flash vacuum pyrolysis (FVP) of 2 through a quartz column at 625 °C led to facile and regioselective fragmentation of the cyclobutane ring, and dioxatetraquinane 6 was obtained in 70% yield. The structure of 6 followed from its spectral characteristics (vide Experimental Section) and in particular through its ^{13}C NMR resonances at δ 134.7, 88.7, and 53.0. On irradiation with UV light, the proximate



6

double bonds of 6 underwent photocycloaddition back to 2. The dioxatetraquinane 6 appeared to be a good source of novel pentacyclic diethers related to heterodiamantanes via transannular bonding of its cyclopentane double bonds.

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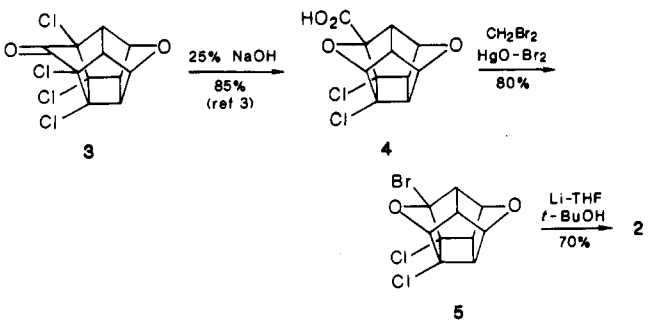
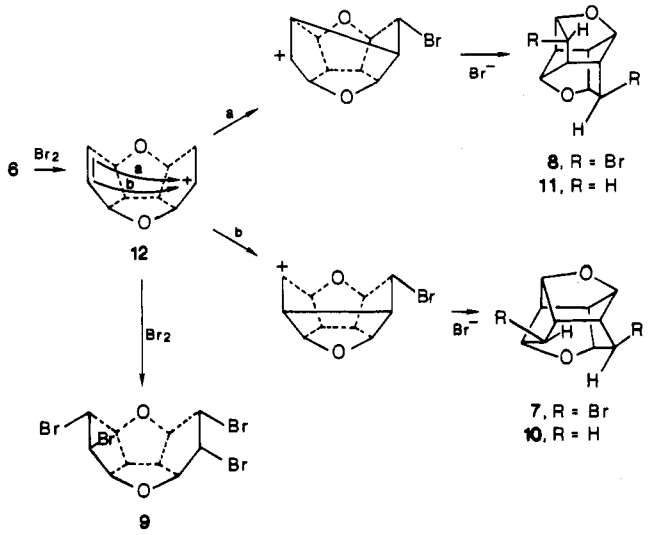
0022-3263/87/1952-0460$01.50/0

Scheme I



Scheme II



Addition of an equimolar amount of bromine to **6** in CCl_4 led to the isolation of two dibromides **7** (35%) and **8** (25%) and a minor tetrabromide **9** (12%). The pentacyclic structures **7** and **8** were deduced from their ^1H and ^{13}C NMR parameters, which showed the presence of symmetry elements and the absence of olefinic carbons and protons. Reductive debromination of **7** and **8** with tri-*n*-butylstannane furnished the C_{10}-pentacyclic diethers **10** and **11**, respectively. The pentacyclic diether **11** was found identical with the compound synthesized recently by Ammann et al.[1e] While **11** is derived from **6** transannular bonding "a" in the intermediate carbonium ion **12**, the other pentacyclic diether **10** originates through bonding "b", Scheme II. The stereochemistry of bromine substituents in **7** and **8** follows from the known propensity of these spheroidal systems to react from the convex face.

In summary, we have outlined useful synthetic entry into the dioxa-1,3-bishomopentaprismane (**2**) and structurally related cage compounds **10** and **11**.

Experimental Section[7]

2-Bromo-6,7-dichlorohexahydro-2,6,3,5-ethanediylidene-2*H*,3*H*-1,4-dioxacyclopenta[*cd*]pentalene (5). In a three-necked round-bottomed flask fitted with N_2 inlet and condenser were placed acid **4** (1 g, 3.3 mmol) and CH_2Br_2 (20 mL). The contents were refluxed for 15 min, and bromine (0.8 g, 5 mmol) in CH_2Br_2 (10 mL) was added dropwise for 15 min. The reaction mixture was refluxed for 3 h, after which CH_2Br_2 was removed in vacuo, to give a brown solid residue. This was extracted with ethyl acetate (3 × 75 mL). The organic extract was washed with water and dried. Removal of solvent gave a viscous liquid, which was charged on a silica gel (10 g) column. Elution of the column with 15% ethyl acetate–hexane gave hexacyclic bromo-compound **5** (950 mg, 85%). Recrystallization from CH_2Cl_2–hexane furnished colorless crystals: mp 136–138 °C; IR (KBr) ν_{max} 3030, 1040, 790 cm^{-1}; ^1H NMR (100 MHz, $CDCl_3$) δ 4.9–5.1 (2 H, m), 4.6–4.8 (1 H, m), 3.2–3.4 (2 H, m), 3.1–3.2 (2 H, m); ^{13}C NMR (25 MHz, $CDCl_3$) δ 99.3, 89.1, 81.8, 79.5, 75.8, 74.0, 63.5, 55.8, 50.9, 50.2. Anal. Calcd for $C_{10}H_7O_2BrCl_2$: C, 38.74; H, 2.27. Found: C, 39.00; H, 2.14.

Hexahydro-2,6,3,5-ethanediylidene-2*H*,3*H*-1,4-dioxacyclopenta[*cd*]pentalene (2). In a three-necked round-bottomed flask equipped with N_2 inlet and condenser was placed 500 mg (1.61 mmol) of **5** in 20 mL of dry THF and 5 mL of *tert*-butyl alcohol. The reaction mixture was slowly stirred, and Li chips (90 mg) were added in small portions over a period of 10 min. The reaction mixture was refluxed for 45 min and cooled to room temperature. The unreacted Li metal was filtered off and destroyed separately. The filtrate was concentrated and diluted with ether (3 × 25 mL). The ethereal solution was washed with 3% HCl, water, and brine and dried. Removal of solvent furnished a crude product, which was charged on a silica gel (10 g) column. Elution with 20% ethyl acetate–hexane furnished dioxabishomopentaprismane **2** (180 mg, 70%), which was recrystallized from CH_2Cl_2–hexane: mp 271–273 °C; IR (KBr) ν_{max} 3000, 1020, 920 cm^{-1}; ^1H NMR (100 MHz, $CDCl_3$) δ 4.6–4.8 (4 H, m), 3.8–4.0 (2 H, m), 3.7–3.8 (4 H, m); ^{13}C NMR (25 MHz, $CDCl_3$) δ 83.5, 56.4, 41.9. Anal. Calcd for $C_{10}H_{10}O_2$: C, 74.05; H, 6.22. Found: C, 74.03; H, 6.26.

5,12-Dioxatetracyclo[7.2.1.0^{4,11}.0^{6,10}]dodeca-2,7-diene (6). The dioxabishomopentaprismane **2** (250 mg, 1.54 mmol) was slowly sublimed (140 °C (4 torr)), through a quartz tube filled with quartz chips and preheated and equilibrated to 625 °C (±10 °C). The solid residue collected in the receiver flask was carefully chromatographed over alumina (5 g). Elution with 20% ethyl acetate–hexane gave tetracyclic diether **6** (170 mg, 70%), which was recrystallized from CH_2Cl_2–hexane: mp 136–137 °C; IR (KBr) ν_{max} 3075, 1350, 1070 cm^{-1}; ^1H NMR (100 MHz, $CDCl_3$) δ 5.7 (4 H, s), 4.9–5.1 (4 H, m), 3.6–3.8 (2 H, m); ^{13}C NMR (25 MHz, $CDCl_3$) δ 134.7, 88.7, 53.0. Anal. Calcd for $C_{10}H_{10}O_2$: C, 74.05; H, 6.22. Found: C, 74.02; H, 6.23.

Addition of Bromine to Tetracyclic Diether 6. To an ice-cold magnetically stirred solution of **6** (100 mg, 0.62 mmol) in CCl_4 (10 mL) was added bromine (0.36 mL, ca. 0.62 mmol) in 5 mL of CCl_4 over a period of 10 min. The reaction mixture was allowed to warm up and was stirred at room temperature for another 10 min. Solvent was removed under reduced pressure, and crude product was charged over a silica gel (5 g) column. Elution with 5% ethyl acetate–hexane furnished pentacyclic dibromide **7** (70 mg, 35%) which was crystallized from CH_2Cl_2–hexane: mp 152 °C; IR (KBr) ν_{max} 3000, 1090, 720 cm^{-1}; ^1H NMR ($CDCl_3$, 100 MHz) δ 2.6–2.8 (2 H, s), 3.1–3.3 (2 H, m), 4.04 (2 H, s), 4.7–4.9 (2 H, m), 5.2–5.3 (2 H, m); ^{13}C NMR ($CDCl_3$, 25 MHz) δ 89.3, 86.9, 55.3, 53.8, 51.0. Anal. Calcd for $C_{10}H_{10}O_2Br_2$: C, 37.30; H, 3.13. Found: C, 37.24; H, 3.10. Continued elution with the same solvent system afforded the pentacyclic dibromide **8** (50 mg, 25%), which was recrystallized from CH_2Cl_2–hexane: mp 144 °C; IR (KBr) ν_{max} 3000, 1090, 720 cm^{-1}; ^1H NMR (100 MHz, $CDCl_3$) δ 2.5–2.7 (2 H, br s), 3.1–3.3 (2 H, br s), 4.2 (2 H, s), 4.5–4.7 (4 H, br s), 5.0–5.2 (4 H, br s); ^{13}C NMR (25 MHz, $CDCl_3$) δ 84.7, 84.4, 54.4, 53.6, 48.7. Anal. Calcd for $C_{10}H_{10}O_2Br_2$: C, 37.30; H, 3.13. Found: C, 37.28; H, 3.10.

Finally, elution with 10% ethyl acetate–hexane gave tetrabromide **9** (35 mg, 12%). Recrystallization from CH_2Cl_2–hexane gave the crystalline compound: mp 147–148 °C; IR (KBr) ν_{max} 2925, 1070, 720 cm^{-1}; ^1H NMR (100 MHz, $CDCl_3$) δ 3.2–5.1 (series of m); ^{13}C NMR (25 MHz, $CDCl_3$) δ 95.2, 95.1, 90.4, 87.4, 59.0, 56.9, 56.3, 55.4, 54.1, 52.9. Anal. Calcd for $C_{10}H_{10}O_2Br_4$: C, 24.92; H, 2.09. Found: C, 25.37; H, 2.04.

Hexahydro-2,3,5-ethanylylidene-2*H*,3*H*-1,4-dioxacyclopenta[*cd*]pentalene (10). Dibromide **7** (45 mg, 0.14 mmol) in dry toluene was placed in a three-necked round-bottomed flask under N_2, and tri-*n*-butylstannane (90 mg, 0.30 mmol) along with a catalytic amount of AIBN was added and the mixture refluxed for 2 h. The reaction mixture was diluted with ethyl acetate and washed successively with saturated aqueous potassium fluoride, 10% HCl, and brine. Drying and removal of solvent gave a residue, which was charged on a silica gel (5 g) column and eluted

(7) For general write-up on Experimental Section, see: Mehta, G.; Rao, K. S. *J. Org. Chem.* **1985**, *50*, 5537.

with 10% ethyl acetate–hexane to give **10** (14 mg, 65%). Sublimation at 100 °C (8 torr) gave analytically pure sample **10**: mp 246 °C; IR (KBr) ν_{max} 2950, 1090 cm^{-1}; ^{1}H NMR (100 MHz, $CDCl_3$) δ 1.7–1.9 (4 H, m), 2.2–2.3 (2 H, br s), 2.9–3.0 (2 H, br s), 4.4–4.8 (4 H, m); ^{13}C NMR (25 MHz, $CDCl_3$) δ 86.5, 81.4, 56.9, 42.0, 36.2. Anal. Calcd for $C_{10}H_{12}O_2$: C, 73.14; H, 7.36. Found: C, 73.10; H, 7.35.

Hexahydro-3,2,5-ethanylylidene-2*H*,3*H*-1,4-dioxacyclopenta[*cd*]pentalene (11). Pentacyclic bromide **8** (10 mg, 0.30 mmol) in 5 mL of dry toluene was placed in a three-necked round-bottomed flask under N_2. Tri-*n*-butylstannane (20 mg, 0.06 mmol) along with a catalytic amount of AIBN was added, and the reaction mixture was refluxed for 2 h. The reaction was worked up as described above. Sublimation of the residue at 80 °C (10 torr) furnished 3 mg of analytical sample **11**: mp 208 °C (lit.[1e] mp 213 °C); IR (KBr) ν_{max} 2900, 1090 cm^{-1}; ^{1}H NMR (100 MHz, $CDCl_3$) δ 1.2–2.2 (6 H, m), 2.9–3.1 (2 H, m), 4.2–4.4 (2 H, m), 4.4–4.6 (2 H, m); ^{13}C NMR (25 MHz, $CDCl_3$) δ 85.4, 78.8, 54.8, 38.2, 36.7.

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2-Bornyllithium. Preparation, Characterization, and Use in Synthesis

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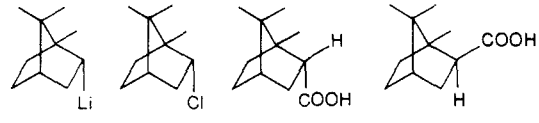
The use of chiral, nonracemic components derived from inexpensive naturally occurring materials to construct new molecules with high degrees of enantiomeric purity is an appealing synthetic strategy. The advantages of the use of such "chiral building blocks" have been discussed in depth.[1] We were interested in using an organometallic derivative of the 2-bornyl system in order to pursue the synthesis of some chiral organosilicon hydrides by this approach. In this note we describe our work with 2-bornyllithium (**1**).

Perusal of the literature reveals that the Grignard reagent derived from 2-bornyl chloride (**2**) does not serve as a well-behaved synthetic intermediate. Treatment of 2-bornyl chloride with magnesium followed by carbonation is reported to lead to variable yields of mixtures of the diastereomeric boranecarboxylic (endo) and isobornanecarboxylic (exo) acids **3** and **4**, respectively, along with various hydrocarbon byproducts.[2] In addition, bornyl-



1 2 3 4

magnesium chloride tends to act as a reducing agent toward ketones instead of adding to them.[3] In view of these reports of low synthetic yields, variable degrees of diastereomeric purity, and the undesirable reducing properties of the 2-bornyl Grignard reagent, we decide to investigate the possibility of using the previously unreported 2-bornyllithium reagent in its place.

Results and Discussion

Initial attempts to prepare the organolithium reagent of bornyl chloride (prepared by hydrochlorination of α- or β-pinene) were run under argon at low temperatures (−30 to −45 °C) in diethyl ether by using lithium wire containing 1% sodium. The yields of bornyllithium in these initial reactions were estimated by GC analysis of the products obtained from the addition of reaction aliquots to dimethyldiethoxysilane. The yields of the desired dimethylbornylethoxysilane were very low, ranging from from 0 to 8%. The major products of the reaction were bornane and unreacted bornyl chloride. Considerable improvements in the yield of bornyllithium were obtained by using a very finely dispersed lithium sand containing 3–4% sodium and by carefully drying the bornyl chloride just prior to use. The bornyl chloride was dried by addition of benzene and then subsequent removal of the benzene by distillation at atmospheric pressure. This caused any residual water to be lost as an azeotrope. Small traces of remaining benzene did not pose any problems in later steps.

The diastereomeric purity of the bornyllithium produced from bornyl chloride (which was prepurified by preparative GC) was determined by carbonation with solid carbon dioxide of an aliquot of the reagent prepared in diethyl ether, a technique used before to establish the configuration of organometallic reagents.[4] The ^{1}H NMR spectrum of the resulting carboxylic acid product was consistent in all respects to a literature spectrum of bornanecarboxylic acid (**3**).[5] No evidence for the formation of isobornanecarboxylic acid (**4**) was present. The presence of any **4** would have been detected in the NMR spectrum by a triplet due to the α-methine hydrogen of **4** at δ 2.34.[6] The α-methine hydrogen in **3** appears as a distorted triplet farther downfield at δ 2.70. Additional evidence of the diastereomeric purity of product **3** came following methylation of the acid and analysis of the methyl ester by GC. Only a single methyl derivative was observed.

Another significant improvement in both the yield and ease of preparation of bornyllithium was attained through the use of refluxing anhydrous pentane as the reaction solvent. The yields, as determined by the Gilman double titration procedure using 1,2-dibromoethane to analyze for residual base,[7] ranged from 45 to 61% after reaction times of 6–12 h. These yields are believed to represent somewhat lower concentration values of bornyllithium than were actually present, since relatively large values were obtained for residual base content in the Gilman titration procedure. Residual base content is usually high only with organolithium reagents prepared in ethereal solvents where solvent cleavage can occur to give lithium alkoxides. The implication is that 1,2-dibromoethane is not reacting quantitatively with the organolithium reagent to give nonbasic products.

This belief was reinforced by the finding that both GC and isolated yields of the products derived from the reactions of bornyllithium solutions and either chlorodimethylsilane (1 equiv) or dichloromethylsilane (0.5 equiv) were often higher than the yields of bornyllithium given

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