

dien-1-yl)methyl]trimethylsilane (**6g**; 3.0 g) in THF (60 mL) with H<sub>2</sub>O (4.2 mL), methanol (4.2 mL), and concentrated HCl (4.2 mL). After 24 h the product was worked up with ether by the general procedure. Mass spectrum, *m/e* (rel intensity) 152 (15, M<sup>+</sup>), 94 (17), 93 (100), 92 (51), 91 (60), 77 (30), 65 (12), 39 (11).

Anal. Calcd for C<sub>9</sub>H<sub>12</sub>O: C, 71.03; H, 7.95. Found: C, 71.21; H, 8.18.

**Bicyclo[4.3.0]nona-3,6-diene (7h)** was prepared by protodesilylation of 4,7-dihydro-1-(trimethylsilyl)indan (**6h**). Distillation of the crude product gave a 12:7:1 mixture of isomers which could be readily separated by gas chromatography (6 ft × 1/4 in., 10% DC 710, 60/80 Chromosorb W, 110 °C) to give bicyclo[4.2.0]nona-3,6-diene (**7h**) as the major product.

Anal. Calcd for C<sub>9</sub>H<sub>12</sub>: C, 89.94; H, 10.06. Found: C, 89.72; H, 9.93.

**4-Methylenecyclohex-1-ene (7j)**<sup>23</sup> was prepared by proto-

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desilylation of (1,4-cyclohexadien-1-yl-methyl)trimethylsilane (**6j**).<sup>6</sup> Distillation gave 4-methylenecyclohexene which was greater than 90% pure by gas chromatography (6 ft × 1/4 in., 10% DC 710, 60/80 Chromosorb W, 95 °C).

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**Registry No.** **3**, 71435-90-0; **5a**, 17988-20-4; **5b**, 71435-91-1; **5c**, 17961-78-3; **5d**, 71435-92-2; **5e**, 18412-15-2; **5f**, 71435-93-3; **5g**, 17998-93-5; **5h**, 18036-88-9; **5i**, 18053-75-3; **6a**, 71435-94-4; **6b**, 71435-95-5; **6c**, 71435-96-6; **6d**, 71435-97-7; **6e**, 71435-98-8; **6f**, 71435-99-9; **6g**, 71436-00-5; **6h**, 71436-01-6; **6j**, 55861-00-2; **7a**, 29648-66-6; **7c**, 16631-66-6; **7d**, 586-62-9; **7f**, 71436-02-7; **7g**, 71436-03-8; **7h**, 71436-04-9; **7j**, 13407-18-6; chlorotrimethylsilane, 75-77-4; 4-methoxybenzyl chloride, 824-94-2; 4-methoxy-3-methylbenzyl chloride, 60736-71-2;  $\alpha$ -phenethyl bromide, 585-71-7; 2-(4-methylphenyl)-2-propanol, 1197-01-9;  $\alpha,\alpha,4$ -trimethylbenzyl chloride, 7243-79-0; *m*-xylene dibromide, 626-15-3; *o*-toluic acid, 118-90-1; lithium diisopropylamide, 4111-54-0; [(4-methoxy-3-methyldihydrophenyl)-methyl]trimethylsilane, 71486-15-2.

## 1,1,2,2-Tetrabromonaphtho[*b*]cyclobutene: A New Source of Naphtho[*b*]cyclobutene-1,2-dione and Substituted Naphtho[*b*]cyclobutadienes

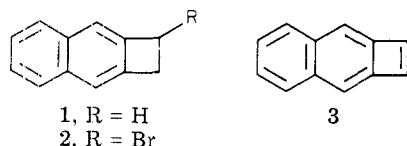
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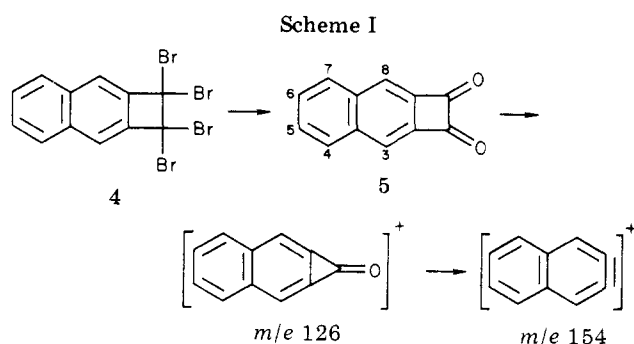
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Reaction of naphtho[*b*]cyclobutene (**1**) with excess *N*-bromosuccinimide gives 1,1,2,2-tetrabromonaphtho[*b*]cyclobutene (**4**), which is hydrolyzed by silver nitrate in aqueous acetonitrile to naphtho[*b*]cyclobutene-1,2-dione (**5**). Several reactions of dione **5** are described, including its peracetic acid oxidation, its lithium aluminum hydride reduction, and its reaction with methylmagnesium bromide to give the dimethyl diols **10**. Diols **10** and tetrabromide **4** may be used as precursors of the unstable 1,2-dimethylnaphtho[*b*]cyclobutadiene (**12**) and 1,2-dibromonaphtho[*b*]cyclobutadiene (**15**), respectively.

Some time ago we described the synthesis of 1-bromonaphtho[*b*]cyclobutene (**2**) from naphtho[*b*]cyclobutene (**1**), as well as the generation of naphtho[*b*]cyclobutadiene (**3**) from monobromide **2**.<sup>1</sup>



In this paper, we report the conversion of naphtho[*b*]cyclobutene (**1**) to 1,1,2,2-tetrabromonaphtho[*b*]cyclobutene (**4**) and describe some reactions of the latter, including a simple synthesis of naphtho[*b*]cyclobutene-1,2-dione (**5**). A synthesis of dione **5** by an unrelated pyrolytic route was reported in 1973 in preliminary form;<sup>2</sup> however, other than its electrochemical behavior,<sup>3</sup> no chemical or physical properties of **5** have yet been described.



### Results and Discussion

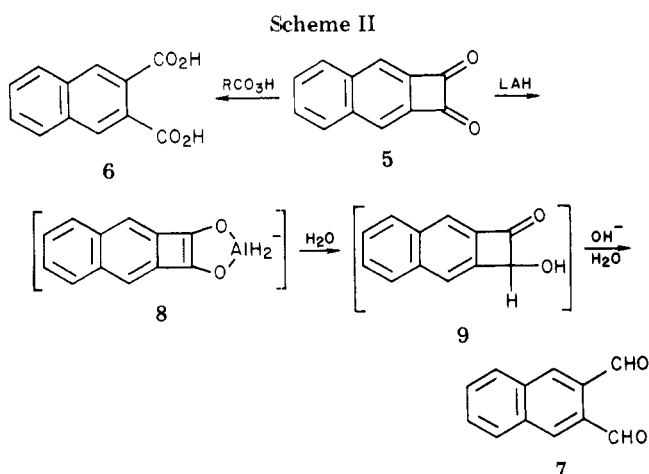
Partial free-radical bromination of naphtho[*b*]cyclobutene (**1**) leads to a very difficultly separable mixture of **1** and its mono- and dibromo derivatives.<sup>1</sup> In contrast, reaction of **1** with excess *N*-bromosuccinimide for several days affords a fair yield (38%) of the readily purified 1,1,2,2-tetrabromonaphtho[*b*]cyclobutene (**4**), mp 186–187 °C. Hydrolysis of tetrabromide **4** with silver trifluoroacetate in aqueous acetonitrile gave (46%) yellow needles of naphtho[*b*]cyclobutene-1,2-dione (**5**), mp 256.5–258 °C.

The mass spectrum of **5**, while also showing a fairly strong molecular ion (40%), shows two base peaks cor-

(1) Cava, M. P.; Hsu, A.-F. C. *J. Am. Chem. Soc.* **1972**, *94*, 6441.

(2) McOmie, J. F. W.; Perry, D. H. *J. Chem. Soc., Chem. Commun.* **1973**, 248.

(3) Rieke, E. D.; White, C. K.; Rhyne, L. D.; Gordon, M. S.; McOmie, J. W. F.; Hacker, N. P. *J. Am. Chem. Soc.* **1977**, *99*, 5387.

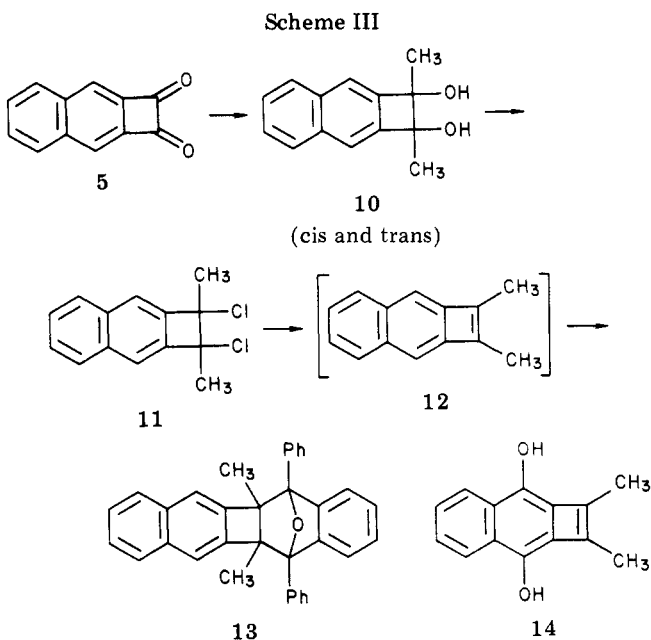


responding to the loss of one and two molecules of carbon monoxide, respectively. The NMR spectrum of **5** shows the highly deshielded C-3 and C-8 protons as a singlet at  $\delta$  8.80, while the  $\alpha$  and  $\beta$  protons of the more remote aromatic ring form an eight-line  $A_2B_2$  system centered at  $\delta$  8.31 and 7.90, respectively. The infrared spectrum of **5** (KBr) shows broad carbonyl absorption around  $5.8 \mu\text{m}$ ; its ultraviolet spectrum in chloroform is complex and includes six strong maxima in the 257–367-nm region.

Dione **5** was oxidized readily by peracetic acid to the expected naphthalene-2,3-dicarboxylic acid (**6**) product. On the other hand, LAH reduction of **5**, followed by aqueous hydrolysis, gave an unexpected product, naphthalene-2,3-dicarboxaldehyde (**7**). Formation of this dialdehyde may be rationalized by assuming the intermediacy of a cyclobutadienoid enediolate complex (i.e., **8**), followed by aqueous hydrolysis to an  $\alpha$ -hydroxycyclobutenone (**9**) and subsequent hydrolytic cleavage of **9** to **7**. Support for this mechanism may be found both in the observed ready electrochemical reduction of **5**<sup>3</sup> and in the known facile cleavage of benzocyclobuten-1-ol to *o*-tolu-aldehyde.<sup>4</sup>

Dione **5** reacted cleanly with methylmagnesium bromide in THF to give a separable mixture of dimethyl diols (**10**) in good yield (77%); the major isomer is tentatively assigned the *cis* stereochemistry on the basis of its higher melting point.<sup>5</sup> Reaction of the mixed diols **10** with thionyl chloride gave chlorides **11**, which were dehalogenated by zinc to liberate 1,2-dimethylnaphtho[b]cyclobutadiene (**12**). No definite product could be isolated from this reaction in the absence of a trapping agent. When the dechlorination was carried out in the presence of 1,3-diphenylisobenzofuran, however, hydrocarbon **12** was trapped in 44% yield to give a single crystalline adduct **13**. The high reactivity of **12** is in accord with the similar behavior of its 3,8-hydroxy derivative **14**, as reported earlier by Breslow and co-workers.<sup>6</sup>

Tetrabromide **4** reacted slowly with activated zinc in refluxing benzene to give, in low yield (8%), a yellow crystalline compound  $C_{24}H_{12}Br_2$  which was assigned structure **17**. The formation of **17** is assumed to involve the generation of 1,2-dibromonaphtho[b]cyclobutadiene (**15**), followed by *angular* dimerization of **15** to **16** and subsequent debromination of **16** to the fully aromatic dibromide **17**. The NMR spectrum of the yellow dibromide shows two one-proton singlets at  $\delta$  8.48 and 8.04



(H-3 and H-8).<sup>7</sup> The alternate linear dimeride structure **18** is incompatible with this observation because of its plane of symmetry. It should be noted, by contrast, that only *linear* dimerization of the unsubstituted naphtho[b]cyclobutadiene (**3**) has been observed.<sup>1</sup>

When the debromination of **4** was carried out in a similar manner in the presence of 1,3-diphenylisobenzofuran (DPIB), the cyclobutadiene **15** was effectively trapped, the initially formed adduct **19** being reduced in situ to give 5,12-diphenyldibenzo[*b,h*]biphenylene (**20**) in good yield (62%).<sup>8</sup> While the intermediate adduct **19** was not isolable in this reaction, it was obtained in fair yield (32%) by the dehydrohalogenation of 2,3-bis(dibromomethyl)naphthalene (**21**) in the presence of DPIB, a reaction which would appear to proceed via cyclobutadiene **15**. Confirmation of the structure of **19** was obtained by its reductive debromination by hydrazine and palladium to the endo adduct (**22**) of naphtho[b]cyclobutadiene (**3**) and DPIB.<sup>1</sup>

## Experimental Section

**General Procedures.** Melting points are uncorrected. Microanalyses were carried out by Midwest Microlab, Inc. Spectra were recorded on a Perkin-Elmer Model 137 IR spectrophotometer, a Perkin-Elmer Model 202 UV-visible spectrophotometer, a Varian A-60A NMR spectrometer, and a Perkin-Elmer Model 270B mass spectrometer. NMR spectra were run in  $CDCl_3$  ( $Me_4Si$  standard).

**1,1,2,2-Tetrabromonaphtho[b]cyclobutene (4).** To a solution of naphtho[b]cyclobutene (**1**, 5.16 g) in carbon tetrachloride (200 mL) was added *N*-bromosuccinimide (33.68 g, excess) and dibenzoyl peroxide (200 mg). The mixture was refluxed for 2 days and the reaction mixture was filtered to remove succinimide and excess *N*-bromosuccinimide. The filtrate was evaporated to dryness giving an oily brown residue which was chromatographed on alumina (NI), eluting with a benzene-hexane mixture (1:1). The eluant, upon evaporation to dryness, gave colorless needles which were recrystallized from benzene-hexane to give pure 1,1,2,2-tetrabromonaphtho[b]cyclobutene (**4**) (5.943 g, 37.8%), mp 186–187 °C.

Anal. Calcd for  $C_{12}H_6Br_4$ : C, 30.67; H, 1.29; Br, 68.04. Found: C, 30.74; H, 1.30; Br, 67.83.

(7) For NMR analyses of related systems, see: (a) Martin, R. H. *Tetrahedron* 1964, 20, 897. (b) Martin, R. H.; Van Trappen, J. P.; Defay, N. *Tetrahedron* 1964, 20, 2373.

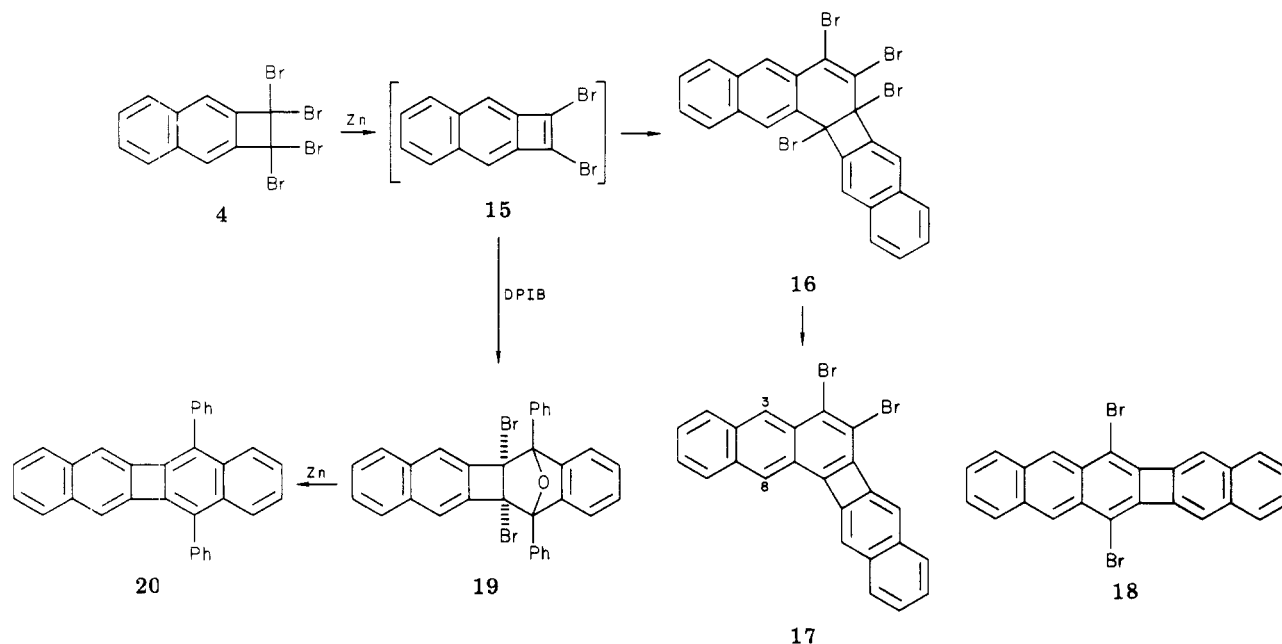
(8) For the exact analogy to the zinc reduction of **19** to **20** in the benzocyclobutene series, see: Cava, M. P.; Pohlke, R. *J. Org. Chem.* 1962, 27, 1564.

(4) Cava, M. P.; Muth, K. *J. Am. Chem. Soc.* 1960, 82, 652.

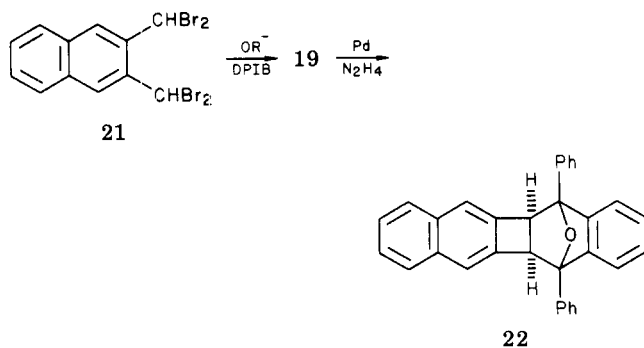
(5) Cava, M. P.; Pohl, R. J.; Mitchell, M. J. *J. Am. Chem. Soc.* 1963, 85, 2080.

(6) Breslow, R.; Grubbs, R.; Murahashi, S.-I. *J. Am. Chem. Soc.* 1970, 92, 4139.

Scheme IV



Scheme V



**Naphtho[b]cyclobutene-1,2-dione (5).** To a solution of 1,1,2,2-tetrabromonaphtho[b]cyclobutene (4, 600 mg) in acetonitrile (10 mL) and water (5 mL) was added silver trifluoroacetate (2 g), and the mixture was refluxed for 10 h in the dark with stirring. The mixture was filtered, the filtrate was extracted with methylene chloride, and the organic phase was washed three times with water (30 mL). The organic layer was separated and dried over anhydrous magnesium sulfate. Upon filtration and evaporation of the solvent, a yellow solid was obtained. Sublimation of this product at 0.5-mmHg pressure, 170 °C, gave yellow needles of naphtho[b]cyclobutene-1,2-dione (5, 99 mg, 46%): mp 256.5–258 °C;  $UV_{\max}^{CHCl_3}$  257 (log  $\epsilon$  4.18), 268 (4.84), 323 (3.29), 348 (3.65), 353 (3.87), 367 (3.46) nm;  $m/e$  (relative intensity) 182 (40), 181 (10), 154 (100), 153 (34), 126 (100), 127 (34).

Anal. Calcd for  $C_{12}H_6O_2$ : C, 79.11; H, 3.32. Found: C, 79.34; H, 3.32.

**Reaction of Dione 5 with Lithium Aluminum Hydride.** A solution of 5 (181 mg) in dry tetrahydrofuran (25 mL) was added dropwise to a tetrahydrofuran solution (25 mL) of lithium aluminum hydride (600 mg). Excess hydride was decomposed by slow addition of ethyl acetate and then water. The mixture was filtered and the filtrate was purified by chromatography on a silica gel plate developed with chloroform. The major product was isolated as white needles (110 mg, 60%, mp 125 °C) and proved to be naphthalene-2,3-dialdehyde (7), as determined by TLC, NMR, and mixture melting point.

**Oxidation of Dione 5.** To a solution of 5 (70 mg) in acetic acid (20 mL) was added dropwise hydrogen peroxide until the original yellow color disappeared. To the reaction mixture was then added a small amount of Pd/C (5%) in order to decompose excess peroxide. The mixture was filtered and the filtrate was concentrated to 5 mL. A white solid precipitated, which was filtered and recrystallized from chloroform, giving naphtha-

lene-2,3-dicarboxylic acid (6, 50 mg, 61%), mp 239–240 °C, identified by IR and by mixture melting point.

**trans-1,2-Dimethylnaphtho[b]cyclobutene-1,2-diol (10a) and cis-1,2-Dimethylnaphtho[b]cyclobutene-1,2-diol (10b).** To a solution of 3 M methylmagnesium bromide (50 mL) in tetrahydrofuran (20 mL) was added dropwise a solution of dione 5 (1.36 g) in 150 mL of dry tetrahydrofuran with stirring and external cooling. After the addition had been completed, the mixture was refluxed with stirring under nitrogen for 15 h. The reaction mixture was then decomposed with 600 mL of ice-water and extracted with ether. The ether layer, after drying over magnesium sulfate, was filtered and evaporated to dryness, giving a light yellow oily residue which was separated by preparative layer chromatography (silica), eluting with chloroform-methanol (9:1), to give the following two compounds.

**trans-1,2-Dimethylnaphtho[b]cyclobutene-1,2-diol (10a)** crystallized from methanol-benzene as white needles (0.11 g, 7%): mp 139–140.5 °C; NMR  $\delta$  1.48 (s, 2  $CH_3$ ).

Anal. Calcd for  $C_{14}H_{14}O_2$ : C, 78.50; H, 6.42. Found: C, 78.59; H, 6.55.

**cis-1,2-Dimethylnaphtho[b]cyclobutene-1,2-diol (10b)** crystallized from methanol-benzene as white needles (1.116 g, 70%): mp 190–191 °C; NMR  $\delta$  1.64 (s, 2  $CH_3$ ).

Anal. Calcd for  $C_{14}H_{14}O_2$ : C, 78.50; H, 6.42. Found: C, 78.75; H, 6.66.

**Adduct (13) of 1,2-Dimethylnaphtho[b]cyclobutadiene (12) with 1,3-Diphenylisobenzofuran.** A mixture of trans and cis diols 10 (188 mg) was added to thionyl chloride (20 mL). The mixture was refluxed for 2 h and then immediately stripped of the thionyl chloride, giving an oily residue. Hexane was added and the evaporation was repeated. The resulting crude chloride (11) was dissolved in dry benzene (20 mL), and excess activated zinc dust and 1,3-diphenylisobenzofuran (244 mg) were added. The mixture was refluxed for 10 h and filtered to remove zinc dust. The filtrate was evaporated to dryness, giving an oily residue which was purified by preparative layer chromatography on silica, using hexane as the developer, to give 13 as white plates (174 mg, 44%). Recrystallization from benzene and methanol gave an analytical sample: mp 215–216 °C; NMR  $\delta$  1.40 (s, 2  $CH_3$ ).

Anal. Calcd for  $C_{34}H_{26}O$ : C, 90.66; H, 5.77. Found: C, 90.53; H, 5.70.

**Debromination of 1,1,2,2-Tetrabromonaphtho[b]cyclobutene (4) with Zinc Dust.** 1,1,2,2-Tetrabromonaphtho[b]cyclobutene (4, 1.01 g) was dissolved in benzene (50 mL), and to this solution was added excess activated zinc dust (1.5 g). The mixture was refluxed with stirring for 15 h. The zinc dust was filtered and the filtrate was evaporated to dryness, giving a yellow residue. Preparative layer chromatography of this product on silica, developing with benzene, gave yellow needles of aromatized

compound 17, which crystallized from *N,N*-dimethylformamide: mp 338–340 °C; 40 mg, 8%; NMR  $\delta$  7.48 (m, 4 H), 7.68 (m, 4 H), 7.88 (m, 3 H), 8.04 (s, 1 H), 8.48 (s, 1 H); UV<sub>max</sub><sup>CHCl<sub>3</sub></sup> 315 (log  $\epsilon$  5.26), 330 (4.91), 340 (4.59), 365 (4.12), 362 (4.13), 384 (4.15), 408 (4.11), 432 (4.31) nm; *m/e* (relative intensity) 458 (50), 460 (100), 462 (50), 379 (5), 381 (5), 302 (5), 301 (26), 300 (84), 298 (34), 232 (8.6), 230 (17.2), 229 (8.6), 148 (9), 148.5 (5), 149 (26), 149.5 (8.6), 150 (43), 150.5 (9).

Anal. Calcd for C<sub>24</sub>H<sub>12</sub>Br<sub>2</sub>: C, 62.60; H, 2.65; Br, 34.75. Found: C, 62.42; H, 2.74; Br, 34.44.

**Reaction of 2,3-Bis(dibromomethyl)naphthalene (21) with 1,3-Diphenylisobenzofuran and Potassium *tert*-Butoxide.** A solution of the tetrabromide 21 (1.69 g) and 1,3-diphenylisobenzofuran (0.97 g) in tetrahydrofuran (45 mL) was added to 150 mL of a solution of potassium *tert*-butoxide in *tert*-butyl alcohol (1 M, 150 mL). After 4 days of stirring at room temperature under nitrogen, the mixture was diluted with water and extracted with chloroform. The chloroform layer was dried over magnesium sulfate and was evaporated to dryness. The residue was purified by silica preparative layer chromatography, developing with cyclohexane–benzene (4:1). The product 19, obtained in 32% yield (666 mg), crystallized from benzene–ethanol as white needles, mp 254.5–255.5 °C.

Anal. Calcd for C<sub>32</sub>H<sub>20</sub>Br<sub>2</sub>O: C, 66.32; H, 3.47; Br, 27.56. Found: C, 66.37; H, 3.47; Br 27.60.

**Reduction of Adduct (19).** To a solution of adduct 19 (89 mg) in benzene (3 mL) and absolute alcohol (3 mL) was added 5% palladium/carbon (150 mg) and hydrazine hydrate (99%, 8 mL). After 3 h of refluxing, the reaction mixture was filtered. Benzene was added to the filtrate and the solution was washed

with water. The benzene layer was separated, dried over magnesium sulfate, and evaporated. The residue crystallized from benzene and hexane to give the debrominated adduct 22 (46.4 mg, 80%), mp 198.5–200 °C; the melting point was undepressed on admixture with the endo adduct (22) of naphtho[*b*]cyclobutadiene (3) and 1,3-diphenylisobenzofuran.

**Debromination of 1,1,2,2-Tetrabromonaphtho[*b*]cyclobutene (4) with Zinc in the Presence of 1,3-Diphenylisobenzofuran.** To 1,1,2,2-tetrabromonaphtho[*b*]cyclobutene (4, 468 mg) and 1,3-diphenylisobenzofuran (42, 412 mg) in 40 mL of benzene was added activated zinc dust (2 g). The mixture was refluxed with stirring for 6 h and filtered. The filtrate was evaporated to dryness, giving an oily yellow residue which was purified by preparative layer chromatography on alumina (NI), developing with cyclohexane. Crystallization from benzene–hexane gave golden yellow needles of 5,12-diphenyldibenzo[*b,h*]biphenylene (20, 250 mg, 62%), mp 225–226 °C; the melting point was undepressed on admixture with material obtained by dehydration of the adducts of naphtho[*b*]cyclobutadiene and 1,3-diphenylisobenzofuran. NMR and infrared spectral data also showed both samples to be identical.

**Acknowledgment.** We thank the National Science Foundation for a grant in support of this research.

**Registry No.** 1, 6827-31-2; 3, 277-98-5; 4, 71382-94-0; 5, 41634-34-8; 6, 2169-87-1; 7, 7149-49-7; 10a, 71382-95-1; 10b, 71382-96-2; 11, 71382-97-3; 12, 71382-98-4; 13, 71382-99-5; 17, 71383-00-1; 19, 71411-05-7; 20, 38998-31-1; 21, 71383-01-2; 22, 38998-28-6; 42, 5471-63-6; methyl bromide, 74-83-9.

## Enones with Strained Double Bonds. 3. Cycloadducts from Bicyclo[3.3.1]non-1-en-3-one<sup>1</sup>

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Reaction of the bicyclic bromo ketone **2b** with triethylamine yields the bridgehead enone **1** which is rapidly trapped by any nucleophile present in the reaction solution. In the absence of competing nucleophiles, the enone **1** is trapped by a slower reaction with furan to form the Diels–Alder adducts **9** and **10**. If the reaction solution contains neither nucleophiles nor furan, the enone **1** undergoes a 2 + 2 cycloaddition reaction with itself to form the symmetrical dimers **14**, **15**, and **16**. These same dimers are produced in approximately the same proportions when the enone **1** is generated by a retro-Diels–Alder reaction of the adducts **9** and **10** in a pyrolysis tube.

Our initial efforts to prepare the bridgehead enone **1** (Scheme I) involved either dehydrochlorination<sup>2</sup> of the chloro ketone **2a** with a base or the thermal decomposition of a related structure **2** (X = S(O)Ph, Se(O)Ph, OAc). In each of these cases, we were unsuccessful either in isolating the enone **1** or in generating a sufficient concentration of the enone **1** so that it could be trapped in a cycloaddition reaction. In the high-temperature thermal decompositions, rearranged products derived from the enone were isolated. In all other cases, products resulting from the conjugate addition of some nucleophile to the enone were formed. It seemed likely that the use of relatively weak, sterically hindered bases (e.g., tertiary amines) in inert solvents might allow significant concentrations of the enone **1** to

exist. Although the previously studied  $\beta$ -chloro ketone **2a** failed to react with various tertiary amine bases,<sup>2</sup> we have now found that the known<sup>2a,3</sup>  $\beta$ -bromo ketone **2b** is satisfactorily dehydrobrominated by various tertiary amines. This paper describes the reactions of the enone **1** generated in solution by this procedure.

The previously described<sup>2-4</sup> ketol **2c** served as the starting material for our studies. Although reaction of this ketol **2c** with PBr<sub>3</sub> had been reported<sup>3</sup> to form a very unstable bromo ketone **2b**, we have independently prepared<sup>2a</sup> and characterized the bromo ketone **2b** and found it to be stable in the absence of impurities (especially acids). Consequently, the bromo ketone **2b** was readily obtained as a stable crystalline solid by reaction of the ketol **2c** with PBr<sub>3</sub> with suitable precautions to remove acidic impurities. Reaction of the bromo ketone **2b** with the amidine **3** (DBU) in PhH, a nonnucleophilic solvent,

(1) This research has been supported by Public Health Service Grant R01-GM-20197 from the National Institute of General Medical Science. The execution of this research was also assisted by Institutional Research Grants from the National Science Foundation for the purchase of a mass spectrometer and a Fourier transform NMR spectrometer.

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(4) W. D. K. Macrosson, J. Martin, W. Parker, and A. B. Penrose, *J. Chem. Soc. C*, 2323 (1968).