Preparation of Carbocations. Freshly distilled FSO_3H was dissolved in an appropriate amount of SO_2ClF as solvent at Dry Ice–acetone temperature (ca. −78°). To this was slowly added with vigorous stirring a cold solution of appropriate precursor dissolved in SO_2ClF, to give an approximately 15–20% solution of the ion. Except for the protonated ketones **1**-OH and **2**-OH, both secondary and tertiary dihydrodibenzotropylium and dibenzotropylium ions generally gave deep-red colored solutions.

Proton and Carbon-13 NMR Spectroscopy. Both proton and carbon-13 NMR spectra were obtained as previously reported.[21]

Acknowledgment. Support of our work by grants from the National Science Foundation and the National Institutes of Health is gratefully acknowledged.

Registry No.—**1**-H, 55090-25-0; **1**-CH_3, 55090-26-1; **1**-CH_2CH_3, 55090-27-2; **1**-C_3H_5, 55124-05-5; **1**-C_6H_5, 30880-08-1; **1**-OH, 55090-28-3; **2**-H, 55090-18-1; **2**-CH_3, 55090-19-2; **2**-CH_2CH_3, 55090-20-5; **2**-C_3H_5, 55090-21-6; **2**-C_6H_5, 55090-22-7; **2**-OH, 55090-23-8; **2**-Cl, 55090-24-9; **3**-H, 10354-00-4; **3** (R = CH_3), 10354-00-4; **3** (R = CH_2CH_3), 18259-45-5; **3** (R = C_3H_5), 55124-06-6; **3** (R = C_6H_5), 55090-29-4; **3** (R = OH), 55090-30-7; **4**-H, 1210-34-0; **4** (R = CH_3), 15323-25-8; **4** (R = CH_2CH_3), 55090-31-8; **4** (R = C_3H_5), 3241-97-2; 4 (R = C_6H_5), 55090-32-9; 4 (R = OH), 55090-33-0; **7**, 13099-45-1; FSO_3H, 7789-21-1.

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Delocalized Carbanions. V.[1] A Tetraanion from the Lithium Reduction of *cis,cis*-1,2,3,4-Tetraphenylbutadiene

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The reduction of *cis,cis*-1,2,3,4-tetraphenylbutadiene with lithium in tetrahydrofuran yields 1,2,3,4-tetralithio-1,2,3,4-tetraphenylbutane, which upon hydrolysis with D_2O gives *dl*- and *meso*-1,2,3,4-tetradeuterio-1,2,3,4-tetraphenylbutane in yields up to 86%. The progress of the reduction was followed through the radical anion and dianion stages to the tetraanion by EPR and uv-visible spectroscopy. No trianion radical intermediate was detected. The tetraanion is apparently stable for weeks in the presence of excess lithium, but gradually cyclizes to give 3-benzyl-1,2,-diphenylindene upon hydrolysis. This amazing stability is attributed to a cyclic reaction scheme which regenerates tetraanion from partially protonated species. In ether, reduction with Li for 4 hr, Na for 24 hr, or K for 50 hr yielded *cis*- and *trans*-1,2,3,4-tetraphenyl-2-butene in 4:1, 5:1, and 1.4:1 ratios, respectively. Reduction for 24 hr with Li or Na for 20 days yielded 1,4-dihydro-1,2,3-triphenylnaphthalene. Reduction for 4 days with Li yielded 9,14-dihydro-9-phenyldibenz[*a,c*]anthracene.

Brook, Tai, and Gilman[2] have reported the reduction of *cis,cis*-1,2,3,4-tetraphenylbutadiene (**1**) with lithium metal, followed be ethanolysis to yield *dl*- and *meso*-1,2,3,4-tetraphenylbutanes.[3] The intriguing possibility of a 1,2,3,4-tetralithio-1,2,3,4-tetraphenylbutane (**2**) intermediate in this reaction prompted the present investigation. Although West and coworkers[4] have prepared several polylithium derivatives of acetylenes containing four or more lithium atoms, the tetralithium compound (**2**) differs from these compounds in that each carbon–lithium bond is benzylic in nature, and may give rise to delocalized carbon–metal bonding. Since delocalization greatly affects the physical and chemical properties of organometallic compounds, the preparation of **2** and the investigation of its properties were of considerable interest.

Our case for the intermediacy of **2** in the reduction of **1** rests primarily upon obtaining *dl*- and *meso*-1,2,3,4-tetradeuterio-1,2,3,4-tetraphenylbutanes (**3a**-d_4 and **3b**-d_4) in up to 86% yield when **1** is reduced with lithium in tetrahydrofuran (THF) and hydrolyzed with D_2O. In addition to the detection of **2** and the investigation of the reaction sequence leading to it, preparatively useful reductions of **1** with lithium, sodium, and potassium are here reported.

Reduction of *cis,cis*-1,2,3,4-Tetraphenylbutadiene (1) with Li in THF. When a solution of **1** in THF was treated with a large excess of lithium under argon, reduc-

Table I
Reaction Products vs. Time

Run	Reaction time, hr	Butadienes	Butanes[a]		Butanes[b]	Indene (5)
			Cis (**4a**)	Trans (**4b**)	*dl* (**3a**) and meso (**3b**)	
1	1.3	3	39	7	49	
	4.0		21	9	58	
	7.0		19	9	61	
	22		8	2	74	5
	46		20	7	58	5
	78		19	7	54	10
	123		32		5	35
2	0.083	95	5			
	0.50	7	93			
	1.67		17		83	
	6.0		13		87	
3[c]	4.25		5		95	
	22		7		93	1
	168		8		76	15
	264		16		67	17
	432		25		56	19

[a] Individual butenes are estimated from the NMR spectra of the crude reduction products. [b] NMR spectra of the crude products gave an invariant 1.2:1 rough estimate of the *dl* (**3a**) to meso (**3b**) isomer ratio. [c] Hydrolyzed with D_2O. Yields are percentages of the volatile material only.

tion occurred with accompanying color changes from colorless to bluish-purple, then to a dark brown-black with the precipitation of a black solid. After removal of the excess lithium, hydrolysis yielded *cis*- (**4a**)[5] and *trans*-1,2,3,4-tetraphenyl-2-butene (**4b**), *dl*- (**3a**) and *meso*-1,2,3,4-tetraphenylbutane[3] (**3b**), 3-benzyl-1,2-diphenylindene[6] (**5**), and hydrogen in varying amounts depending upon the reaction time. The general classes of compounds (butenes, butanes, and indene) were separated and quantified by GLC, but separation of the configurational isomers within the groups was not accomplished by this method. Fortunately, the *cis*- and *trans*-2-butenes and *dl* and meso butanes were easily separated by fractional crystallization. The products separated by GLC and crystallization were identified by comparing their retention times, spectroscopic properties, and melting points with those of authentic samples. In addition to the products listed, some of the GLC analyses indicated the presence of two additional unidentified products which did not amount to more than 8% of the total products.

Typical product ratios in the reduction are shown as a function of time in Table I. In runs 1 and 2 the yields are actual yields obtained by GLC analysis with an internal standard, whereas in run 3 the yields are reported as percentages of the volatile fraction of the product only. In other reductions (run 1 and others not reported), it was ascertained that even with very long reaction times, the volatile material accounted for 70–90% of the product, so absolute yields do not differ greatly from the reported yields. The butadiene recovered in run 2 was a mixture of 1 and the trans,trans isomer.[7] Estimation of the yields of cis and trans butenes **4a** and **4b** was accomplished using a combination of GLC analysis and NMR integration to estimate isomer ratios. The same technique was more difficult to apply to the butane isomers **3a** and **3b** because the aliphatic proton signals in these compounds overlap; however, the ratio of *dl* (**3a**) and meso (**3b**) isomers appeared to remain invariant at about 1.2:1. The poor reproducibility of the yields with time probably results from agitation and metal surface differences in the heterogeneous reactions.

Table II
Deuterium Analyses[a] of the Butane Fractions from the D_2O-Hydrolyzed Reduction (Run 3)

Time, hr	d_1	d_2	d_3	d_4
4.25	1	2	6	90
22.0	0	2	9	88
168	1	5	19	74
264	0	16	61	24
432	1	44	41	14

[a] These results are accurate to approximately ±3%.

When reduction times exceeded 12 hr, hydrogen was evolved upon hydrolysis. A 24-hr reduction mixture, for example, liberated 14 ml of gas when hydrolyzed with D_2O (after removal of excess lithium), and titration of the hydrolyzed mixture with acid indicated that 4.38 g-atoms of lithium had reacted per mole of starting diene. Mass spectrometric analysis of the gas evolved showed it to be 2% H_2, 97% HD, and 1% D_2. The preponderance of HD indicates that lithium hydride is the source of the gas upon hydrolysis.

Having shown that approximately 4 g-atoms of lithium was consumed during the reduction of 1, the case for the production of a tetraanion rests upon establishing that four deuterium atoms were incorporated into the hydrolyzed product. In run 3 (Table I), aliquots of the reduction mixture were hydrolyzed with D_2O and the butane fractions were separated by GLC and analyzed for deuterium content by mass spectrometry,[8] the results being shown in Table II. The intervention of a tetralithio species was inferred from the high (95%) yield of 1,2,3,4-tetraphenylbutanes in the 4.25-hr reduction mixture and the fact that 90% of it was tetradeuterated (**3a**-d_4 and **3b**-d_4). Although alternative mechanisms for the production of tetradeuteriobutanes may be envisioned, these involve either abstraction of protons or hydrogen atoms from the solvent or disproportionation of odd-electron species, neither of which is consistent with the 86% overall yield of tetradeuteriobutanes. Since **3b** does not undergo H–D exchange when treated with LiOD in D_2O, the only reasonable source of tetradeuteriobutanes would seem to be the deuterolysis of a tetralithio species. It is not clear at this point, however, whether 1,2,3,4-tetralithio-1,2,3,4-tetraphenylbutane (**2**) is actually the intermediate, since migration of anionic sites

Table III
Relative Uv, Visible, and EPR Absorptions

Reaction time, hr	650 nm	550 nm	425 nm[a]	305 nm	EPR
0.17	1.0	2.0	1.0	50	Strong
2.0	0.058[b]	*c*	1.0[d]	*e*	Medium
4.0	0.0	0.0	1.0	*e*	Weak
20.0	0.0	0.0	1.0	*e*	Very weak
20.0[f]	1.6	2.8	1.0	1.0	Strong

[a] There is a 340-nm shoulder on each of these peaks. [b] λ_{max} was 685 nm. [c] The 550-nm peak was masked by the 425-nm absorption and appeared as a slight shoulder on the tail of this peak. [d] λ_{max} was 435 nm. [e] A minimum between the 425-nm band and end absorption occurs at 265 nm. The intensity of this minimum increased from 0.2 at 2.0 hr to 0.5 at 4.0 hr, and then remained at 0.5 in the 20.0-hr spectrum. [f] With 3 molar equiv of *cis,cis*-1,2,3,4-tetraphenylbutadiene added.

within a molecule are not uncommon. Similar analysis of the butene fraction of the product (**4a**-d_2 and **4b**-d_2) showed it to be almost exclusively dideuterated, indicating that its precursor is indeed the dianion of **1**.

Evidence for the 1,2,3,4 disposition of anionic sites was obtained from the NMR spectra of the tetradeuteriobutanes. The *dl* (**3a**-d_4) and meso (**3b**-d_4) butane isomers[9] from the same 4.25-hr reduction were separated by fractional crystallization and examined by NMR spectroscopy. The *dl* isomer (**3a**-d_4) showed two broad (3-Hz half-width) singlets from the aliphatic hydrogens at δ 2.83 and 3.12 ppm and a complex multiplet at δ 6.65–7.2 ppm from the aromatic hydrogens. The meso isomer **3b**-d_4 showed broad (3.5 Hz) singlets at δ 2.53 and 2.76 ppm and a complex multiplet at δ 6.5–7.3 ppm. Integration of the aromatic and aliphatic proton signals in both *dl* (**3a**-d_4) and meso (**3b**-d_4) isomers gave the proper 10:1 ratio, indicating that no ring deuteration occurred. The NMR spectra show conclusively that the products were actually the 1,2,3,4-tetradeuterio-1,2,3,4-tetraphenylbutanes **3a**-d_4 and **3b**-d_4. In order to explain the 1:1 double singlet nature of the aliphatic proton spectra, it would be useful to first consider what the spectrum of a 1,2,3,4-tetraphenylbutane-*2,3*-d_2 (PhCH$_2$CDPhCDPhCH$_2$Ph) would look like. Since the 2 and 3 carbons are chiral, the methylene protons would be magnetically nonequivalent and would give rise to an AB quartet. If we now randomly substitute deuterium for one of the A or B hydrogens in each methylene, J_{AB} would not be observed and two singlets are expected, exactly as found in the spectra of **3a**-d_4 and **3b**-d_4. The singlets are broadened owing to coupling with deuterium. If the hydrogens were 1,3 to each other, a similar argument would predict three peaks, two peaks of ½ proton intensity each for H-1 and a singlet of unit intensity for H-3. A 1,2 and 1,1 disposition of the hydrogens is eliminated by the absence of coupling. Thus, one infers from the 1,2,3,4 deuterium positions in the product that the anionic sites had a similar disposition in **2**.

The progress of the reduction was also followed qualitatively by uv–visible and EPR spectroscopy, with the results being tabulated in Table III. The intensities of the absorptions in the electronic spectrum are presented relative to the intensity of the dianion–tetraanion band at 425 nm, which is arbitrarily assigned a value of 1.0. Doran and Waack[7] reported absorptions at 585 and 435 nm for the tetraphenylbutadiene radical anion and dianion, respectively. We assign the two bands at 550 and 650 nm (intensity ratio about 2:1) to the radical anion because they rose and then fell proportionately during the early phase of the reaction as expected of the radical anion spectrum. The band at approximately 305 nm is attributed to tetraphenylbutadienes.[7] It was disturbing not to find a separate band directly attributable to the tetraanion **2**. A small red shift was observed between the 2.0-hr sample (435 nm, in agreement with the data of Doran and Waack[7] for the dianion) and those with longer reduction times (425 nm). This is in the expected direction since there is less conjugation in the tetraanion than the dianion, but the shift is close to the limit of detectability because of the extreme broadness of the 425-nm band. Another indication of change, however, was an increase in the minimum at 265 nm between the 425-nm band and end absorption from a relative intensity of 0.2 to 0.5.

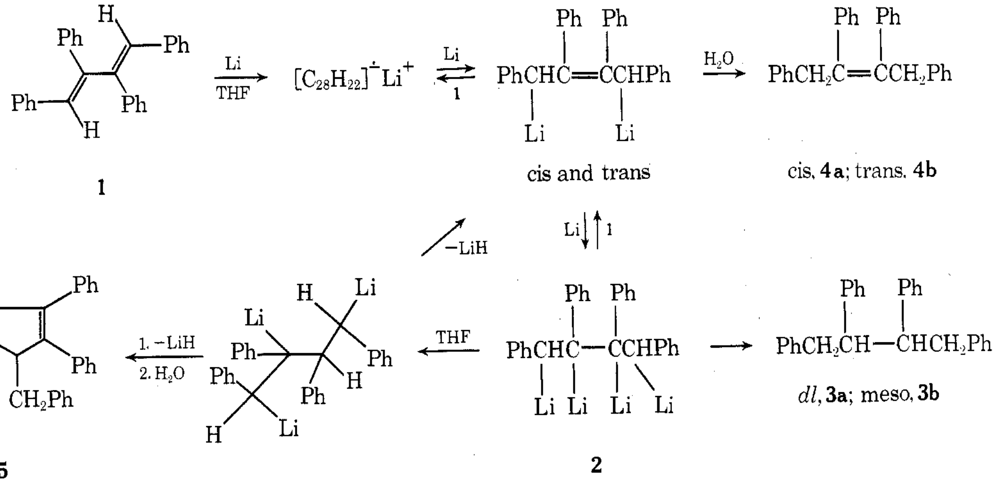
The EPR spectrum consisted of a single line about 7 G in width[7] which was most intense in the 0.17-hr sample, and whose intensity decreased progressively with longer reaction times. The progressive decay of the EPR signal after the radical anion stage suggests that the trianion radical is unstable and either disproportionates or is rapidly reduced to tetraanion. Reversibility of the reduction process was demonstrated by adding 3 molar equiv of tetraphenylbutadiene (**1**) to the reaction mixture after reduction for 20 hr, whereupon the EPR signal returned and the uv–visible spectrum showed the same bands (although in different intensities) as the 0.17-hr sample. This ready reversibility of the reduction illustrates the strong electron-donating property of the tetraanion.

The stability of **2** in THF was surprising. Even after 168 hr at room temperature an overall yield of 56% of tetradeuteriobutanes was obtained (Tables I and II), which implies that a similar amount of tetraanion survived the extended reaction time. In comparison, Gillman and McNinch[10] found the initial half-life of benzyllithium in THF to be approximately 5 hr, although the long-term decomposition rate decreased to 0.44%/hr. Thus, it appears as though **2** has roughly the same, or perhaps even greater, stability in THF than benzyllithium. Recall, however, that lithium hydride was one of the products in the reductions with long reaction times. The apparent stability of **2** and the production of lithium hydride may both be explained by a cyclic process in which **2** is regenerated from its partially protonated analogs by splitting out LiH and reverting back to dianion as shown in Scheme I. Evidence for this lies in the fact that LiH is formed in reaction mixture even when the only organic products are butenes and butanes.

Another source of lithium hydride in the system is ring closure to **5** under long reaction times. Although the precursor of **5** is not known, its appearance so late in the reduction scheme suggests that it may not be the tetraanion. Perhaps a more likely candidate would be the trianion formed upon monoprotonating **2** at the more basic 2 position. This would preclude delocalization of charge into the 2-phenyl ring and make it more susceptible to nucleophilic aromatic substitution. A suggested reduction scheme which is in accord with the experimental data is presented in Scheme I.

Variation of Reducing Metal and Solvent. A cursory investigation of the effects of cation solvation on the reduction of **1** was carried out by varying the solvent and reducing metal. When the reduction of **1** is carried out with sodium in THF, very little butane is obtained. Treatment of the diene **1** with excess sodium for 18 hr yielded 37% butadienes, 62% butenes, and only 1% butanes (identified by GLC retention times). Thus, it is apparent that the tetraanion forms far less readily when sodium is the reducing agent, and may not form at all. The small amount of butane formed could have arisen by electron transfer from the

Scheme I



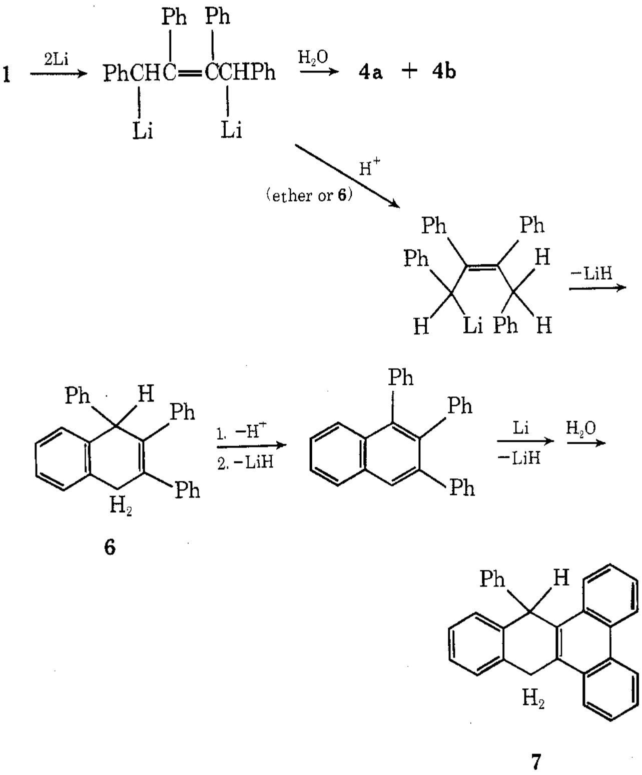
dianion to tetraphenylbutene during hydrolysis. An analogous reaction was reported by Levin, Jogur-Grodzinski, and Szwarc,[11] who obtained bibenzyl from the methanolysis of the diphenylacetylene dianion. Alternatively, the butane could result from protonation of the dianion by THF, followed by further reduction. The greater reducing ability of lithium compared to sodium is undoubtedly due to the greater energy of solvation of the organolithium compounds in THF.

In ether the reduction of 1 with Li for 4 hr, with Na for 24 hr, or with K for 50 hr yields *cis*- and *trans*-1,2,3,4-tetraphenyl-2-butene (**4a** and **4b**, respectively). Apparently only the lower melting (78°) isomer has been described in the literature,[5] and no assignment of configuration is made. We tentatively assign the cis geometry to the 78° melting isomer because it is thermodynamically less stable than the 194° isomer (equilibrium ratio of the 194° isomer and the 78° isomer is 1.93:1), and because the phenyl protons of the 2- and 3-phenyl rings appear as singlets in the NMR spectrum similar to those of *cis*-stilbene, whereas the pattern from the corresponding protons in the 194° isomer are a complex multiplet similar to *trans*-stilbene. Unfortunately, attempts to confirm this assignment by stereospecific reduction to the known *dl* and meso butanes (**3a** and **3b**) by hydrogenation and hydroboration were unsuccessful. The ratio of cis:trans isomers of 4 obtained in the reduction of 1 appeared to be metal dependent. Typical ratios are: Li, from 4:1 to 2:1, with the higher ratio being obtained with shorter reduction times; Na, 5:1, K, 1.4:1. Hydrolysis of the reduction mixtures with D_2O yielded **4a**-d_2 and **4b**-d_2 as expected from a dianion intermediate.

When the reductions of 1 were carried out with lithium in ether for 24 hr, 1,4-dihydro-1,2,3-triphenylnaphthalene[12] (**6**) was obtained in addition to the butenes. **6** was also obtained in better yield by treating 1 in ether with excess sodium for 20 days, followed by hydrolysis. When 1 was treated with lithium metal in ether for 4 days, further cyclization occurred, yielding nearly pure 9,14-dihydro-9-phenyldibenz[*a*,*c*]anthracene (**7**) upon hydrolysis. The structure of the product was confirmed by dehydrogenation to 9-phenyldibenz[*a*,*c*]anthracene.[12]

It is interesting that the first ring closure in ether yields a six-membered ring, whereas in THF a five-membered ring was obtained. In both cases, the direction of ring closure may be determined by the position of monoprotonation of the polyanions. Earlier we mentioned that monoprotonation of the tetraanion at the more basic secondary site could direct ring closure to the phenyl ring attached to the same carbon. In a similar manner, protonation of the dianion might be expected to occur at the 1 position, yielding the allylic monoanion in which the 1-phenyl is now most susceptible to nucleophilic aromatic substitution. The source of the proton may be solvent, or the cyclization product **6**. A suggested reduction scheme consistent with the products obtained is presented in Scheme II.

Scheme II



Experimental Section

Melting points are uncorrected. The nuclear magnetic resonance spectra were recorded on either Varian A-60 or HA-100 spectrometers. The ultraviolet–visible spectra were obtained using a Cary 14 spectrophotometer, and the electron paramagnetic resonance spectra were recorded using 100-kHz modulation on a Varian V-4560 spectrometer equipped with a 12-in. magnet.

Reduction of 1,2,3,4-Tetraphenylbutadiene (1) with Lithium in Tetrahydrofuran (THF). The preparative reactions were carried out under a positive pressure argon atmosphere. The THF solvent used was reagent grade dried over KOH pellets and freshly distilled from the radical anion of anthracene before each experiment. In a typical reduction, 3.0 g (8.4 mol) of **1** in 50 ml of THF was stirred at 22° with 0.07 g of Li metal cut into four pieces 2 × 4 × 4 mm. Periodically samples of the solution were withdrawn by syringe and run into degassed H_2O or D_2O. Each sample was extracted with CH_2Cl_2, dried over Na_2SO_4, and evaporated. The samples were analyzed by NMR and GLC at 265° using an F & M Model 500 chromatograph with a 2-ft column packed with Apiezon L on Chromosorb P. The GLC peaks were identified by trapping the exiting materials in capillaries and running their ir and mass spectra. These were compared with the retention times and spectra of authentic samples.

In run 3 of Table I, the tetradeuterated *dl* (mp 82–85°, lit.[3] mp 89–90° undeuterated) and *meso*-1,2,3,4-tetraphenylbutane-d_4 (mp 179–180°, lit.[3] mp 183–185° undeuterated) from the 4.25-hr reduction sample were separated by fractional crystallization from ethanol.

The reductions in which the uv–visible and EPR spectra were monitored were carried out under vacuum in a sealed glass system having a 1.0-mm absorption cell and an EPR sample tube attached. In the run leading to the data in Table III, 0.025 g of **1** in 8.0 ml of THF was treated with a 1 × 5 × 10 mm piece of lithium in a reaction compartment. Periodically the solution was decanted from the metal into an adjacent compartment from which it was transferred to either the absorption or EPR cell to record its spectra. For the uv–visible spectra the samples had to be diluted considerably. After the solution had contacted the metal for 20 hr it was decanted into the adjacent compartment, 0.075 g of **1** was added via a break-seal tube, and the spectra were again recorded. The solution was then allowed to react with the metal again for another 18 hr, after which the metal was removed and H_2O was added via another break-seal tube. GLC analysis of the product showed it to consist of 83% tetraphenylbutanes and 17% tetraphenylbutenes.

Attempted H–D Exchange in *meso*-1,2,3,4-Tetraphenylbutane (3b). A 26-mg sample of **3b** in 2 ml of THF was stirred with 4 ml of 1.2 *M* LiOD in D_2O for 1.5 hr at 22°, then heated on a steam bath for 5 min. The recovered **3b** showed no detectible H–D exchange when analyzed by mass spectrometry.

Preparation of *cis*- (4a) and *trans*-1,2,3,4-Tetraphenylbutene (4b). Reductions of **1** with Li in ether for 4 hr, with Na for 24 hr, and with K for 50 hr were carried out using about a threefold excess of metal. The reactions were hydrolyzed by removing the solution from the metal via syringe and introducing it into degassed H_2O. Extraction with CH_2Cl_2, drying over Na_2SO_4, and evaporation yielded crude products which were analyzed by NMR for the relative amounts of high- and low-melting 1,2,3,4-tetraphenyl-2-butenes. The isomers were separated by fractional crystallization from ethanol.

Isomer A, mp 194–195°. Anal. Calcd for $C_{28}H_{24}$: C, 93.29; H, 6.71. Found: C, 93.05; H, 6.83. NMR ($DCCl_3$) δ 3.60 (s, 4), 6.9–7.4 ppm (m, 20).

Isomer B, mp 77–78° (lit.[5] mp 80°). Anal. Found: C, 93.11; H, 6.71. NMR ($DCCl_3$) δ 4.03 (s, 4), 7.03 (s, 10), 7.25 (s, 10).

Equilibration of *cis*- and *trans*-1,2,3,4-Tetraphenyl-2-butene. In separate experiments, isomers A and B were heated at 280° in an evacuated sealed tube for 17 hr. NMR analysis of the product in both cases yielded a ratio A:B of 1.93:1.

Preparation of 1,4-Dihydro-1,2,3-triphenylnaphthalene (6). The title compound was the predominant product along with butenes **4a** and **4b** when **1** was reduced with Li in ether for 24 hr. The products were readily separated by fractional crystallization from CH_2Cl_2–ethanol. From 2.0 g of **1** was obtained 0.5 g of pure **6**, mp 168–169.5° (lit.[12] mp 165°).

6 was also obtained when 3.0 g of **1** was treated with excess Na in ether for 20 days, followed by hydrolysis. Crystallization from CH_2Cl_2–ethanol yielded 2.3 g, mp 153–161°. Two more recrystallizations from the same solvent gave 1.3 g of **6**, mp 168–169.5°.

Preparation of 9,14-Dihydro-9-phenyldibenz[a,c]anthracene (7). The reduction of **1** with excess Li in ether for 4 days followed by hydrolysis, extraction with CH_2Cl_2, and evaporation of the solvent yielded crude **7**. The NMR spectrum of this crude product showed no detectable impurities. Recrystallization from CH_2Cl_2–ethanol yielded **7** melting at 192–194° (lit.[12] mp 192°).

Dehydrogenation of 7. The method of Bergmann and Zwecker[12] was used. A mixture of 0.40 g of **7** and 0.40 g of Se was heated for 21 hr at 290–320°. The crude product was extracted with $CHCl_3$ and decolorized with Norit, ethanol was added, and the solution was concentrated until brown crystals began to form. Cooling the solution yielded 0.120 g of 9-phenyldibenzo[*a,c*]anthracene, mp 225–230° (lit.[12] mp 227°).

Registry No.—1, 1608-10-2; **2,** 55255-17-9; **3a,** 35341-52-7; **3b,** 5271-40-9; **4a,** 55255-18-0; **4b,** 55255-19-1; **5,** 55255-20-4; **6,** 55255-21-5; **7,** 55255-22-6; lithium, 7439-93-2.

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(8) We are indebted to Professor F. W. McLafferty for the mass spectral analyses.

(9) The terms *dl* and meso are no longer strictly correct when applied to **3a**-d_4 and **3b**-d_4 because two new chiral centers are created by the introduction of deuterium on the 1 and 4 carbons. This complication is ignored in the discussion since the properties of **3a**-d_4 and **3b**-d_4 roughly correspond to those of the undeuterated *dl* and meso butanes, respectively.

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