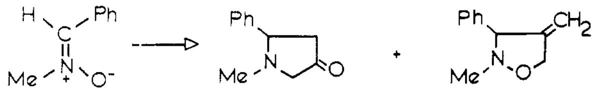
We have demonstrated that the product distribution reflects kinetic rather than thermodynamic factors by refluxing each of the products (i.e., **7**, **8**, and **9a**) separately in acetone and recovering each unchanged. Finally, Scheme I argues that nitrones not bearing an *N*-aryl substituent should afford only two products upon exposure to allene. Indeed, *C*-phenyl-*N*-methylnitrone undergoes cycloaddition with allene to afford pyrrolidinone **20** and isoxazolidine **21** in a 2:3 ratio, respectively. As expected from the absence of an *N*-aryl substituent in the starting nitrone, no benzazepinone (i.e., corresponding to **9a**) was formed. The structure of **20** follows from the presence of carbonyl absorption at 5.71 μm in the infrared and its ^1H NMR spectrum [($CDCl_3$, 100 MHz) δ 2.20 (s, 3), 1.8–3.0 (m, 3 besides Me), 3.50 (m, 2), and 7.24 (br s, 5)]. Isoxazolidine **21** shows no significant absorption in the 5–6-μm region of the infrared. Its ^1H NMR spectrum [($CDCl_3$, 100 MHz) δ 2.60 (s, 3), 3.85 (br s, 1), 4.50 (br s, 3), 4.93 (br s, 1), and 7.25 (s, 5)] is consistent with the structural assignment.



20 21

We have shown herein that the intermolecular reactions of nitrones with allene can result in the production of regioisomeric adducts, one of which (e.g., **18**) undergoes spontaneous further rearrangement. When an *N*-aryl substituent is present in the nitrone, the production of a benzazepinone (e.g., **9a**) can be anticipated as a major product.

Acknowledgment. We gratefully acknowledge the generous financial assistance provided by the National Institutes of Health (Institute of General Medical Sciences, GM 25303).

Registry No. 1b, 26505-49-7; **2a**, 463-49-0; 4 (R = Me), 34285-49-9; **7**, 5469-56-7; **8**, 71718-69-9; **9a**, 71718-70-2; **9b**, 71718-71-3; **10**, 71718-72-4; **11**, 71718-73-5; **12**, 19344-95-7; **13**, 69284-39-5; **14a**, 71718-74-6; **14b**, 71718-75-7; **15**, 1701-57-1; **16**, 71718-76-8; **17a**, 71718-77-9; **17b**, 71718-78-0; **20**, 23770-12-9; **21**, 71718-79-1; methyl acrylate, 96-33-3.

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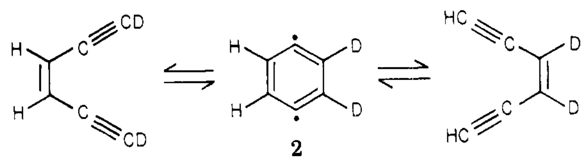
Evidence for the Formation of 1,3-Dehydrobenzene Diradicals by Silicon Shift in 1,4-Dehydrobenzenes at High Temperature

Summary: Evidence is presented that high temperature pyrolysis of three isomeric disubstituted 1,5-hexadiyn-3-enes proceeds by initial cyclization to 1,4-dehydrobenzene intermediates, and that these initially formed species are capable of rearranging to 1,3-dehydrobenzenes. Both types of intermediates appear to behave as diradicals at these temperatures.

Sir: Thermal rearrangement of 1,5-hexadiyn-3-ene (**1** and **3**, eq 1) proceeds through a 1,4-dehydrobenzene intermediate which we believe has diradical structure **2** at the temperatures required for rearrangement.[1,2] We now find that substituted systems also undergo cyclization to derivatives of **2**, and at higher temperatures than those required for rearrangement, chemistry involving interaction of the aryl radical centers with the substituents occurs. In one case formation of the observed product is most easily understood by postulating the intervention of 1,3-dehydrobenzene, or *m*-benzyne, intermediates. Furthermore, as in the 1,4 case, the 1,3 intermediates appear to behave as diradicals at these temperatures.



1 2 3 (1)

Our studies involve the synthesis (Chart I) and gas-phase flow pyrolysis of diynes **4**, **5**, and **6**. Both stereoisomers[3] of **4** and **5** were stable at temperatures substantially above those (ca. 150 °C) normally required for the $1 \rightleftharpoons 3$ rearrangement. At temperatures near 300 °C, thermal interconversion of **4a** and **5a** (Chart II) was observed, the equilibrium lying predominantly (99:1) on the side of **5a**. At 450 °C cis/trans isomerization set in, and irreversible conversion to new products was also observed. Four major products, along with several unidentified materials in minor amount, were formed (Chart II). The structures of **7** and **9** were assigned by independent synthesis, and **8** and **10** were assigned on the basis of mass spectroscopic and NMR data. Most importantly, only *m*-(trimethylsilyl)isobutenylbenzene (**7**) was formed; the ortho and para isomers were not observed. The quantitative product distributions are shown in Table I.

In the case of diyne **6**, the presence of two bulky substituents at the terminal positions made cyclization an even slower process. Cis/trans isomerization once again was observed near 450 °C but irreversible conversion to new products required temperatures near 530 °C. Three major products were formed in >95% total yield (Chart II). Two are isomeric isopropenyl(trimethylsilyl)toluenes: only one of the two possible 1,2,3 isomers (**11**) and one of the two possible 1,2,4 isomers (**12**).[4] The third product was once

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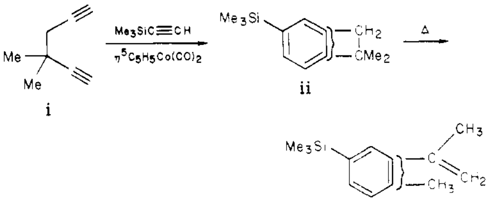
(1) Jones, R. R.; Bergman, R. G. *J. Am. Chem. Soc.* **1972**, *94*, 660.

(2) Bergman, R. G. *Acc. Chem. Res.* **1973**, *6*, 25.

(3) Application of the *E*/*Z* nomenclature to the hexenediynes discussed here requires that some of the compounds having the two alkynyl groups in cis relationship be termed the *E* isomer, and in other cases the *Z*. Because this makes discussion of our results somewhat clumsy, we have taken the liberty of reverting to the older (somewhat arbitrary) system, and will refer in all cases to those compounds with the two alkynyl groups in cis relationship as the cis isomer.

(4) The two pyrolysis products were determined to be isopropenyl(trimethylsilyl)toluenes on the basis of analytical and NMR spectral data.

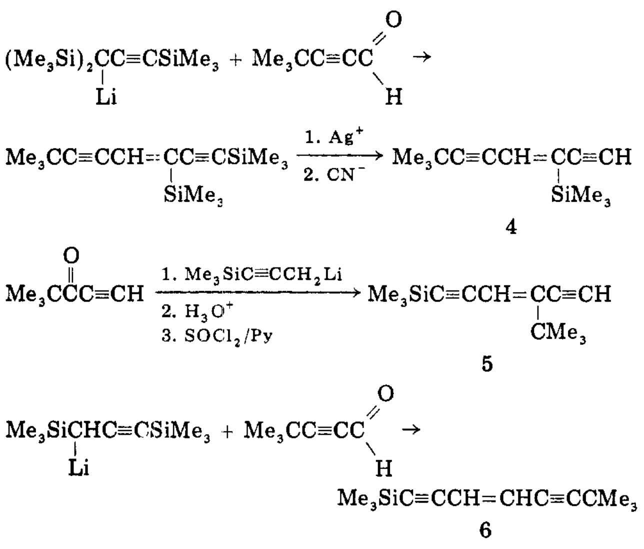


i ii 12

A mixture of 2,2-dimethyl-3- and 4-(trimethylsilyl)benzocyclobutenes (ii) were prepared from diyne i as shown above.[4a] Pyrolysis of this mixture gave 1-methyl-2-isopropenyl-4-(trimethylsilyl)benzene and 1-isopropenyl-2-methyl-4-(trimethylsilyl)benzene (**12**). This result allowed us to distinguish the set of compounds **11** from the set **12**. It also demonstrated that the two isomeric compounds present in **12** could be resolved gas chromatographically, and that compounds ii were unstable to the conditions used for pyrolysis of the diynes, rearranging only to **12**: Hillard, R. L.; Vollhardt, K. P. C. *J. Am. Chem. Soc.* **1977**, *99*, 4058.

0022-3263/79/1944-4215$01.00/0

Chart I



$(Me_3Si)_2\underset{Li}{C}C{\equiv}CSiMe_3 + Me_3CC{\equiv}CC(=O)H \rightarrow$

$Me_3CC{\equiv}CCH{=}\underset{SiMe_3}{C}C{\equiv}CSiMe_3 \xrightarrow[2.\ CN^-]{1.\ Ag^+} Me_3CC{\equiv}CCH{=}\underset{SiMe_3}{C}C{\equiv}CH$ **4**

$Me_3C\overset{O}{\overset{\|}{C}}C{\equiv}CH \xrightarrow[2.\ H_3O^+\quad 3.\ SOCl_2/Py]{1.\ Me_3SiC{\equiv}CCH_2Li} Me_3SiC{\equiv}CCH{=}\underset{CMe_3}{C}C{\equiv}CH$ **5**

$Me_3Si\underset{Li}{C}HC{\equiv}CSiMe_3 + Me_3CC{\equiv}CC(=O)H \rightarrow Me_3SiC{\equiv}CCH{=}CHC{\equiv}CCMe_3$ **6**

Chart II

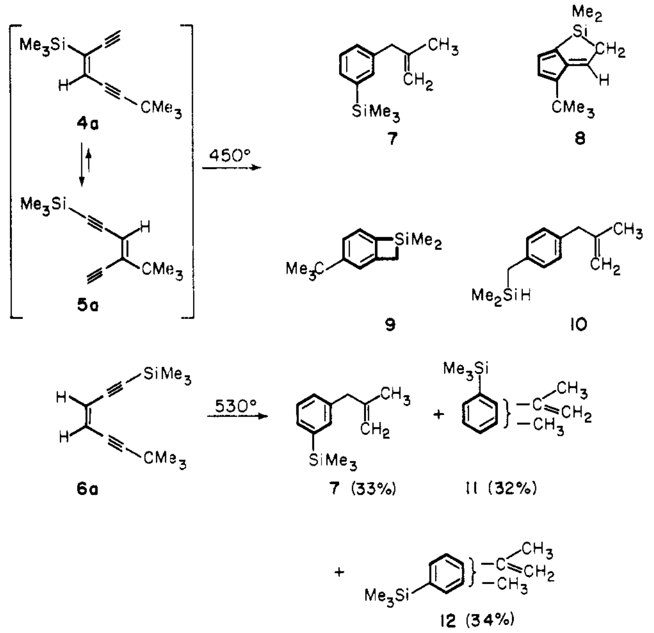


Table I. Products Formed Upon Gas-Phase Flow Pyrolysis[a] of 4, 5, and 6

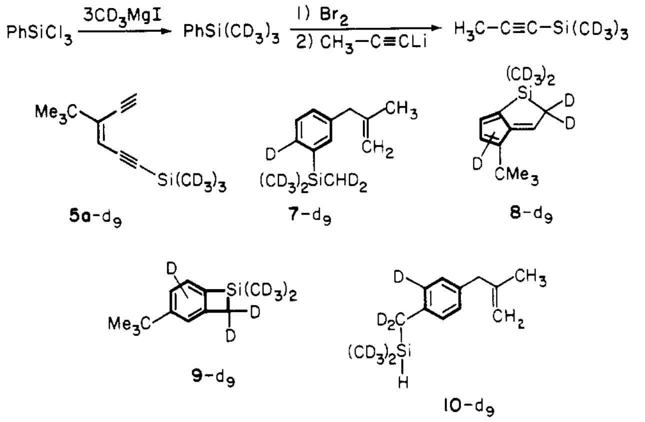
starting material	T, °C	product,[b] %			
		7	8	9	10
5a	454 ± 2	29.5	12.5	36.7	21.5
4a	454 ± 2	31.0	9.5	36.2	23.1
4b[c]	451 ± 1	30.7	10.4	36.7	22.2

[a] Approximately 10-mg samples were subjected to consecutive pyrolysis through a 35-cm quartz tube at atmospheric pressure, using N_2 carrier gas, with a contact time of ~2.2 min. [b] Product percentages are given as percent of total of 7 + 8 + 9 + 10. Absolute yields of these materials were ~70% in each case; an additional 10% of at least 12 minor products was also observed. [c] Cis isomer of 4a.

again *m*-isobutenyl(trimethylsilyl)benzene (7), still uncontaminated by ortho and para isomers.

In order to clarify the mechanism of these unusual reactions, we have examined the pyrolysis of **5a** labeled with deuterium in the trimethylsilyl group. This material (**5a**-d_9) was synthesized as before, using 1-(nonadeuteriotrimethylsilyl)propyne prepared as shown at the top of Chart III. **5a**-d_9 obtained in this way was pyrolyzed and the deuterated products were separated by preparative VPC. Using a combination of proton and ^{13}C NMR spectrometry, the position of label in each of the products was specified to the extent illustrated in Chart III.

Chart III



As might have been expected, the methylene groups adjacent to silicon in **8** and **9** arise from CD_3 groups, the missing deuteron having been transferred to the unsaturated ring in each case. In the case of **10**-d_9, the ^1H absorption due to the silicon-bound hydrogen was still fully present, but the nine-line splitting pattern observed in the proton case had been reduced to a broadened singlet. A proton originally present in one of the *tert*-butyl carbons was therefore now attached to the silicon, at minimum eight (but probably ten; vide infra) atoms away. Examination of the ^{13}C spectrum showed[5] that the deuterium originally bound to silicon methyl had been transferred to only the positions ortho to the $(CD_3)_2Si(H)CD_2$ group.

Most clear-cut, however, was the labeling observed in the *m*-(trimethylsilyl)isobutenylbenzene (**7**-d_9). The apparently "intact" Me_3Si group had in reality been broken into, its violation now made apparent by the presence of a single silylmethyl-bound proton in the NMR. The unsymmetrical structure of this compound allowed resolution of all the aromatic carbon signals in the ^{13}C NMR. Examination of the ^{13}C NMR spectrum of **7**-d_9 showed that deuterium was located *only* on the carbon *ortho* to silicon and *para* to the isobutenyl substituent.[5]

These data drastically restrict the possible mechanistic alternatives. Most important are the following conclusions: (a) the appearance of compounds of type **11** and **12** in the pyrolysis of **6a**, and their absence in the pyrolysis of **4** and **5**, imply that benzocyclobutenes are generated in the former pyrolysis but require that they are not formed in the latter (despite the formation of their silicon analogues in the pyrolysis of **4** and **5**); (b) **10** likely arises from a diradical which transfers a hydrogen atom between radical substituents located in para relationship to one another; (c) the labeling pattern in **7**-d_9 is most rationally accounted for by mechanisms in which the silicon substituent migrates from one position to an adjacent one on the aromatic ring, and the carbon substituent retains its location.

A mechanism for the rearrangement of **4** and **5**, consistent with these restrictions, is outlined in Chart IV. We believe the first intermediate is 1,4-dehydrobenzene A.

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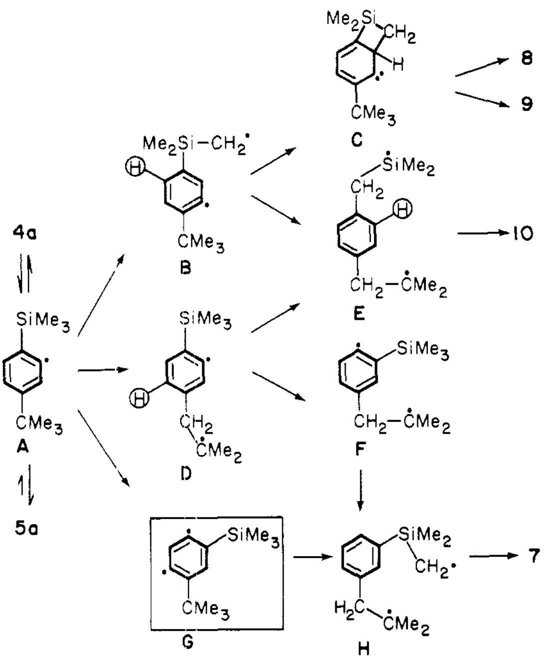
[5] The ring carbon chemical shifts in the ^{13}C NMR spectra of the aromatic pyrolysis products were assigned on the basis of careful comparison with the spectra of model compounds, including benzene, methallylbenzene, 4-(deuteriomethallyl)benzene, (trimethylsilyl)benzene, and *p*-(trimethylsilyl)methallylbenzene. Appearance of deuterium at a ring carbon caused a significantly reduced intensity in the ^{13}C NMR absorption due to that carbon.

Chart IV

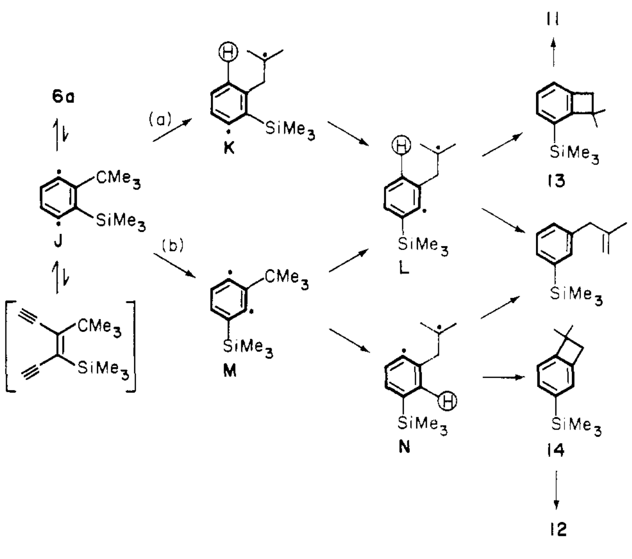


This may be further transformed by two paths involving hydrogen transfer to the aromatic radical centers from the methyl groups. Transfer from the silicon methyl leads to B; this may be followed by attack of the methylene carbon on the ring, which generates carbene C. 1,2 Hydrogen shift then leads to **9**, and 1,2-carbon shift leads to **8**. Alternatively, if the initial hydrogen transfer in A is from the *tert*-butyl group, a radical is formed which rapidly undergoes an aryl shift (a rearrangement with earlier precedent[6a,b]), leading to D. Hydrogen shift from the silicon methyl, followed by a similar aryl rearrangement (also with precedent[6c]), gives E (this diradical may also be available by attack on the *tert*-butyl group in B). We believe E to be the immediate precursor of **10** by long-range hydrogen transfer.[7]

The labeling data indicate strongly that the immediate precursor of **7** is diradical H. This could be formed by silicon shift in D (giving F) followed by hydrogen transfer from the silicon methyl group. Perhaps more interestingly, the order of these steps might be reversed. *This would form m-benzyne intermediate* G. Two hydrogen transfers and aryl shift at the carbon-substituted radical then lead to H.

Are *m*-benzyne intermediates involved in these reactions? The key to this question lies in the data obtained from diyne **6**. Because no products resulting from benzocyclobutenes are formed from **4** and **5**, a special route to these compounds must be available in **6**. The two most reasonable possibilities are outlined in Chart V. In path (a), hydrogen transfer in the initial *p*-benzyne intermediate J leads to K. Silicon shift then gives L, which can close to benzocyclobutene **13** by simple intramolecular radical recombination, a process not available in any of the intermediates formed from **4** and **5**. Hydrogen transfer in L leads to **7**. Importantly, however, this mechanism provides a route to only *one* benzocyclobutene. In alternative (b), initial silicon shift in J gives *m*-benzyne M. Diradical M is capable of *two* hydrogen shift/ring closure paths, leading to both L and N. These can give **13** and **14**, respectively, and ultimately **11** and **12**, as is observed. The data from this pyrolysis, therefore, are most consistent with path (b), leading to 1,3-dehydrobenzene intermediate M.

Chart V



Further work will be required to confirm our hypothesis that *m*-benzynes are generated from *p*-benzynes thermally at high temperature. If we are correct, however, it is interesting that both *m*- and *p*-benzynes appear to follow the same type of pattern in their chemistry, reactions characteristic of closed-ring compounds at low temperatures,[8,9] and transformations attributable to aromatic diradicals when the temperature is raised.[10]

Acknowledgment. We are grateful to the National Science Foundation (Grants CHE78-08706 and CHE77-26089) for financial support of this work, to Rudi Nunlist and Paul Becker for experimental assistance, and to Professor Ronald Breslow for first encouraging us to take seriously the possibility of 1,2-trimethylsilyl rearrangements in 1,4-dehydrobenzenes. We also acknowledge NSF Grant No. 23633 and NIH Grant No. RR00711 which support the Stanford University high-field NMR facility, where some of our proton NMR spectra were run.

Registry No. 4a, 71462-34-5; **4b**, 71462-35-6; **5a**, 71462-36-7; **5a**-d_9, 71462-37-8; **5b**, 71462-38-9; **6a**, 71462-39-0; **6b**, 71462-40-3; **7**, 71462-41-4; **7**-d_9, 71462-42-5; **8**, 71486-27-6; **8**-d_9, 71462-66-3; **9**, 71462-43-6; **9**-d_9, 71462-67-4; **10**, 71462-44-7; **10**-d_9, 71462-45-8; **11**, 71462-54-9; **12** (1,2,4-isomer), 71462-46-9; **12** (1,2,5-isomer), 71462-47-0; benzene, 71-43-2; (trimethylsilyl)benzene, 768-32-1; methallylbenzene, 3290-53-7; methallylbenzene-*4-d*, 71462-48-1; 4-(trimethylsilyl)methallylbenzene, 71462-49-2; $(Me_3Si)_2C(Li)C{\equiv}CSiMe_3$, 71462-50-5; $Me_3CC{\equiv}CC(O)H$, 2579-21-7; $Me_3CC{\equiv}CCH{=}C(SiMe_3)C{\equiv}CSiMe_3$, 71462-51-6; $Me_3CC(O)C{\equiv}CH$, 5891-25-8; $Me_3SiC{\equiv}CCH_2Li$, 23832-16-8; $Me_3SiCH(Li)C{\equiv}CSiMe_3$, 71500-57-7; $CH_3C{\equiv}CSi(CD_3)_3$, 71462-52-7; $CH_3C{\equiv}CLi$, 4529-04-8; $PhSi(CD_3)_3$, 71462-53-8; CD_3I, 865-50-9; $PhSiCl_3$, 98-13-5.

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[6] (a) Brunton, G.; Griller, D.; Barclay, L. R. C.; Ingold, K. U. *J. Am. Chem. Soc.* **1976**, *98*. 6803. (b) Brunton, G.; Gray, J. A.; Griller, D.; Barclay, L. R. C.; Ingold, K. U. *Ibid.* **1978**, *100*, 4197. (c) Wilt, J. W.; Chwang, W. K.; Dockus, C. F.; Tomiuk, N. M. *J. Am. Chem. Soc.* **1978**, *100*, 5534.

[7] Although it is not clear whether it is intramolecular, hydrogen transfer in a diradical involving an apparent ten-atom transition state has been observed by Tsuji, T.; Shibata, T.; Hienki, Y.; Nishida, S. *J. Am. Chem. Soc.* **1978**, *100*, 1806.

[8] (a) Breslow, R.; Napierski, J.; Clarke, T. C. *J. Am. Chem. Soc.* **1976**, *98*, 5703. (b) Breslow, R.; Khanna, P. L. *Tetrahedron Lett.* **1977**, 3429.

[9] Washburn, W. N.; Zahler, R.; Chen, I. *J. Am. Chem. Soc.* **1978**, *100*, 5863.

[10] Interestingly, recent theoretical calculations using the GVB method (which ought to provide improved reliability when applied to open-shell systems such as those under consideration here) predict diradical-like structures for the lowest-energy forms of *both m*- and *p*-benzyne; cf. Noell, J. O.; Newton, M. D. *J. Am. Chem. Soc.* **1979**, *101*, 51.

Supplementary Material Available: (I) Data and rationale for assignment of structure to (trimethylsilyl)isopropenyltoluenes **11** and **12** (includes ^{1}H and ^{13}C NMR, IR, and mass spectral data); (II) data and rationale for ring carbon ^{13}C NMR assignments for **7** and analysis of isotope labeling experiments (17 pages). Ordering information is given on any current masthead page.

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(11) (a) National Science Foundation Predoctoral Fellow, 1974–1977; (b) to whom inquiries should be addressed at the University of California, Berkeley.

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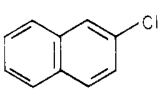
Received June 14, 1979

Generation of a 1,3-Dehydronaphthalene. Evidence for the Diyl Structure

Summary: The dichlorocarbene adduct of 2-bromoindene reacts with a solution of potassium *tert*-butoxide in tetrahydrofuran to yield derivatives of naphthalene which are consistent with the intermediacy of 1,3-dehydronaphthalene in the diradical form.

Sir: Although recent studies have shown that 1,4-dehydrobenzene may be generated and trapped in both the diradical[1] (1,4-benzenediyl) and bridged (butalene) forms,[2] only the bicyclic structure of 1,3-dehydrobenzene[3] (bicyclo[3.1.0]hexatriene) has been trapped. We now wish to report our evidence that 1,3-dehydronaphthalene can be generated and trapped as the diradical.

The starting material for the work, compound **1**, was prepared from indene, via 2-bromoindene, in ~10% yield (Scheme I).[4] Treatment of **1** with a solution of *t*-BuOK in THF at 0 °C for 1 h yielded products **2–11**.[6] When THF-d_8 is the solvent the same mixture of products is obtained, although in slightly different yields. Examination of the product mixture obtained from the deuterated solvent by GC–mass spectroscopy revealed that the chloronaphthalene is approximately 79% d_2, 13% d_1, and 8% d_0. The ^{13}C NMR spectrum helps to establish the location of the deuterium atoms, as in **2a**.[9]



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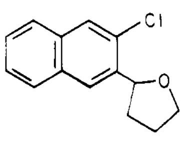

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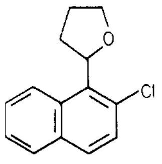

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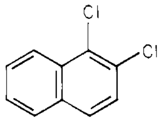

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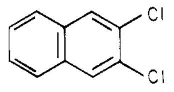

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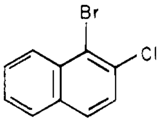

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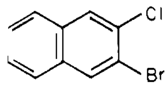

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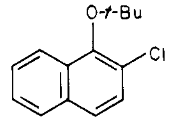

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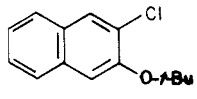

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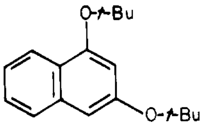

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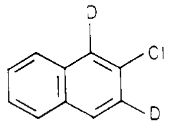

2a

Although it was not possible to establish the position

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(1) Jones, R. R.; Bergman, R. G. *J. Am. Chem. Soc.*, **1972**, *94*, 660.

(2) Breslow, R.; Napierski, J.; Clarke, T. C. *J. Am. Chem. Soc.*, **1975**, *97*, 6275.

(3) Washburn, W. N. *J. Am. Chem. Soc.*, **1975**, *97*, 1615. Washburn, W. N.; Zahler, R. *J. Am. Chem. Soc.*, **1976**, *98*, 7827, 7828. *Ibid.* **1977**, *99*, 2012. Washburn, W. N.; Zahler, R.; Chen, I. *Ibid.* **1978**, *100*, 5863. For earlier reference see: Berry, R. S.; Clardy, J.; Schafer, M. E. *Tetrahedron Lett.*, **1965**, 1011. McGriff, R. B. *Diss. Abstr. B* **1967**, *28*, 844. Bertorello, H. E.; Rossi, R. A.; de Rossi, R. H. *J. Org. Chem.* **1970**, *35*, 3332.

(4) A procedure similar to that reported by Joshi, Singh, and Pande[5] was used in the synthesis of **1**. The ^{1}H NMR spectrum of **1** proved to be surprisingly simple. What appears to be two singlets at δ 3.20 (1 H) and 3.71 (2 H) on expansion proved to be finely divided multiplets. The aromatic region is a four-proton complex multiplet around δ 7.02. In the decoupled ^{13}C NMR spectrum four quaternary carbons, two at 46.2 and 68.3 ppm and two aromatic at 138.1 and 142.6, could be detected easily. Of the remaining six signals two were at 45.9 and 50.0 ppm and the four aromatic ones at 123.6, 125.1, 126.7, and 128.0 ppm. The two signals at 45.9 and 50.0 ppm split into a triplet (J = ~2.2 ppm) and a doublet (J = ~2.2 ppm), respectively.

(5) Joshi, G. C.; Singh, N.; Pande, L. M. *Tetrahedron Lett.*, **1972**, 1461.

(6) The GC percentages (80% material balance) of compounds **2–11** are: **2**, 7%; **3**, 3.4%; **4**, 2.6%; **5** + **6**, 16%; **7** + **8**, 17%; **9**, 27%; **10**, 13%; **11**, 14%. Purification of the various products was achieved by TLC (silica gel, several solvents). Structures were assigned on the basis of the following data: Chloronaphthalene (**2**) was identified by comparison with a commercial sample (Aldrich Chemical Co. Inc.). A mixture of **3** and **4** exhibits NMR signals in CDCl$_3$ at δ 1.00–2.80 (m, 8 H), 3.70–4.12 (m, 2 H), 4.14–4.50 (m, 2 H), 5.20–5.40 (t, 1 H, J = 6 Hz), 5.60–5.80 (m, 1 H), 6.70–8.00 (m, 11 H), 8.25–8.50 (m, 1 H); IR (neat) 3060 (w), 2980 (s), 2870 (m), 1620 (w), 1590 (m), 1505 (m), 1460 (m), 1365 (m), 1325 (m), 1185 (m), 1165 (m), 1130 (m), 1070 (s), 1070 (s), 1050 (s), 805 (m), and 745 (s) cm^{-1}. Elemental composition was determined by high-resolution mass spectroscopy. These compounds were shown to be a 56:44 mixture by GC using a 250-ft SF-96 capillary column. Samples of **5** and **7** were prepared as described in the literature.[7,8] **6** and **8** were prepared by heating **1** in dimethyl sulfoxide at 40 °C. **6** has NMR signals in CDCl$_3$ at δ 7.34–7.50 (d of d, 2 H, J = 6 and 3 Hz), 7.56–7.70 (d of d, J = 6 and 3 Hz), and 7.86 (s, 24), whereas **8** has signals at δ 7.30–7.65 (AB quartet, 4 H, J = 9 Hz), 7.80 (s, 1 H), and 7.98 (s, 1 H). A sample of **9** was prepared from the corresponding chloronaphthol: NMR (CDCl$_3$) δ 1.47 (s, 9 H), 7.30–7.90 (m, 5 H), and 8.0–8.30 (m, 1 H). **10** has NMR signals at δ 1.47 (s, 9 H), 7.25–7.40 (m, 2 H), and 7.45 (s, 1 H). 1,3-Di-*tert*-butoxynaphthalene (**11**) exhibits the following spectral data: NMR (CDCl$_3$) δ 1.40 (s, 9 H), 1.48 (s, 9 H), 6.80–6.86 (d, 1 H, J = 3 Hz), 7.10–7.17 (d, 1 H, J = 3 Hz), 7.25–7.50 (m, 2 H), 7.55–7.75 (m, 1 H), and 8.05–8.23 (m, 1 H); IR (neat) 3060 (w), 2970 (s), 2940 (sh), 1622 (m), 1595 (m), 1575 (m), 1500 (m), 1390 (s), 1362 (s), 1280 (w), 1260 (w), 1230 (w), 1180 (s), 1160 (s), 1130 (s), 1080 (m), 995 (m), 905 (w), 880 (w), 840 (w), and 750 (m). Cleavage of this compound with 40% HBr in HOAc provided 1,3-naphthalenediol.

(7) Clemo, G. R.; Cockburn, J. G.; Spence, R. *J. Chem. Soc.*, **1931**, 1265.

(8) Wilson, N. K.; Zehr, R. D. *J. Org. Chem.*, **1978**, *43*, 1768.

(9) The studies of monosubstituted naphthalenes by Kitching, Bulpitt, Doddrell, and Adcock[10] provided a reference for the effects observed on deuteration of naphthalenes.