dithiane, 6007-26-7; 2-vinyl-1,3-dioxolane, 3984-22-3; copper(II) trifluoromethanesulfonate, 34946-82-2; rhodium(II) trifluoroacetate, 72654-51-4; ethyl diazoacetate, 623-73-4; dimethyl diazomalonate, 6773-29-1; acrolein dimethyl acetal, 6044-68-4; methyltriphenylphosphonium bromide, 1779-49-3; 1,2-ethanedithiol, 540-63-6; 2,3-butanedione, 431-03-8; dimethylformamide, 68-12-2.

Supplementary Material Available: Mass spectral data for reaction products and ^{1}H NMR spectral data for **8-12** (6 pages). Ordering information is given on any current masthead page.

Photolysis of Tri-1-naphthylboron Does Not Give Naphthylborene

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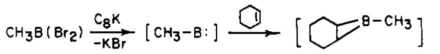
Received November 15, 1983

Irradiation of tri-1-naphthylboron in cyclohexene does not give evidence for formation of naphthylborene. No *cis*-1,2-cyclohexanediol is observed after oxidative workup, and only a low yield of 1,1′-binaphthyl is obtained. These findings are in contrast to the results of Ramsey and Anjo, which were used to support naphthylborene formation.

In recent years there has been a resurgence of interest in the chemical and physical properties of reactive intermediates. In part this renewal has been driven by the development of new spectroscopic techniques that permit the direct monitoring of formerly elusive transient species. In particular, our understanding of hypovalent intermediates such as carbenes, nitrenes, and silylenes has benefited from EPR and laser spectrophotometric investigation.[1] In contrast, the corresponding boron-centered hypovalent species (R–B), alternately called either a borylene,[2] boryne[3] or a borene,[4] remains much less studied and is incompletely understood. Indeed, there is some doubt that an authentic example of this intermediate has ever been prepared (vide infra).

There have been two general routes explored for the preparation of borenes. The first is based upon the α-elimination of two groups from a boron atom and is illustrated in eq 1 by the results of van der Kerk and co-workers[5] who examined the reaction of methylboron dibromide with potassium on graphite in the presence of cyclohexene. A related reaction of phenylboron dibromide in the presence of diphenylacetylene also was used to support the intermediacy of a borene[6] but proved to be unreliable.[4] In neither case was the key boracyclopropane actually isolated or characterized as such.



(1)

Using a similar approach, Eisch investigated the reaction of phenylboron dichloride with bis(trimethylsilyl)mercury.[4] In the absence of a trapping olefin, the presumed intermediate phenylborene underwent polymerization in competition with its eventual insertion into carbon–hydrogen bonds. Attempts to intercept the borene with diphenylacetylene failed due to reaction of the acetylene with presumed intermediates formed before the borene is generated. This observation, and others, led Eisch to warn that "...detection of borenes, generated from organoboron dibromides....and reducing agents, by acetylenes cannot give reliable evidence." However, van der Kerk and coworkers[7] argue that these complications do not compromise their findings with cyclohexene.

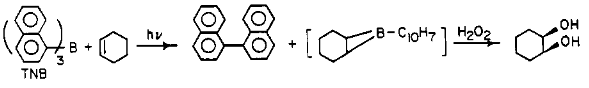
The second general route for the preparation of hypovalent boron compounds is based on the photochemical elimination of a biaryl from a triarylboron or a tetraarylborate anion. This approach is illustrated in eq 2 and 3.

$$Ar_3B \xrightarrow{h\nu} Ar{-}B{:} + Ar{-}Ar \qquad (2)$$

$$Ar_4B^- \xrightarrow{h\nu} Ar_2B^- + Ar{-}Ar \qquad (3)$$

It has been known for some time that irradiation of sodium tetraphenylborate in hydroxylic solvents gives aryl coupling products.[8] More recently, Eisch[9] has shown that in ether solvents the diphenylborate can be trapped with diphenylacetylene to give eventually products analogous to the carbene reaction products.

Similarly, early investigation of the photochemistry of triphenylboron in methanol solution revealed the formation of biphenyl in low yield.[10] More recently, Ramsey and Anjo[3] reported on the photochemistry of tri-1-naphthylboron (TNB) in hydrocarbon solvents. They claim that the photolysis of TNB in cyclohexene solution followed by oxidation of the reaction mixture gives *cis*-1,2-cyclohexanediol in 40% yield and 1,1′-binaphthyl, eq 4. These observations led them to suggest that relatively



(4)

efficient formation of 1-naphthylborene (1-NB) occurs on photolysis of TNB. This conclusion encouraged us to begin an investigation of the properties of 1-NB by pulsed laser

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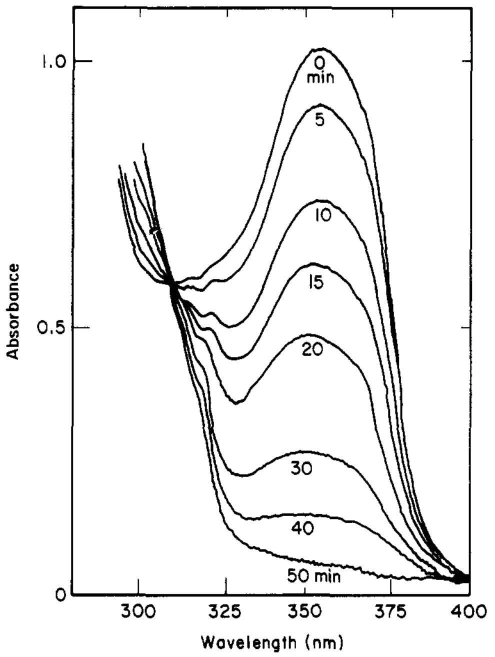


Figure 1. Absorption spectrum of trinaphthylboron in cyclohexene solution recorded during irradiation through a uranium glass filter.

spectroscopy. Unfortunately, we are unable to reproduce the critical experiments supporting the formation of 1-NB and must conclude that its formation is not a significant contribution to the photochemistry of TNB.

Results

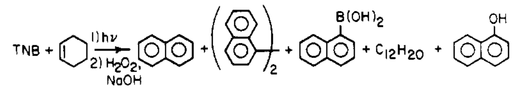
The key result used to support formation of 1-NB from TNB is the detection of *cis*-1,2-cyclohexanediol after oxidation of the mixture of photolysis products.[3] We searched for this diol in reactions run under a range of conditions without success.

Irradiation of TNB in oxygen-free cyclohexene (2 mL, 1.0×10^{-2} M) with light from a 450-W Hanovia Hg arc filtered through a uranium glass sleeve to remove wavelengths shorter than 350 nm results in consumption of the borane. The progress of this photolysis is conveniently followed by monitoring the absorption spectrum of TNB as the irradiation proceeds. In cyclohexene solution the borane exhibits an absorption band with a maximum at ca. 352 nm. The intensity of this band decreases monotonically during the irradiation, and an apparent isosbestic point at ca. 309 nm is evident in the spectra, Figure 1. The rate of consumption of TNB revealed by the changes in its absorption is, as expected, approximately zeroth order and under these conditions is complete in ca. 45 min.[11] These results are confirmed by monitoring the photolysis of TNB by HPLC and are not changed when the photolysis is performed through a Pyrex filter.

The products formed from the photolysis of TNB were examined by using several analytical and spectroscopic techniques. In the previous report,[3] it is claimed that oxidation and hydrolysis of the reaction mixture in basic aqueous dilute hydrogen peroxide by standard procedures gives *cis*-1,2-cyclohexanediol. We attempted to reproduce this observation. A 0.25-mL portion of a basic (0.2 M NaOH) aqueous ethanol (80%) solution of hydrogen peroxide (0.2 M) was added to a 1.0-mL portion of the photolysis solution described above. This mixture was stirred vigorously for 10 min at room temperature, and then the excess hydrogen peroxide was reduced by addition of sodium metabisulfite. The aqueous phase was saturated with NaCl, the pH was adjusted to achieve neutrality, and the organic layer was removed and dried.

Analysis of the organic layer by gas chromatography using a 5% Carbowax 20 M glass column does not reveal any cis diol. Similar experiments involving extraction of the organic components of the photolysis solution with chloroform and concentration give no cis diol. Control experiments, in which cis diol is added to cyclohexene and carried through the workup procedure, reveal a limit for its detectability of less than 2%, assuming formation of 1 equiv of diol from each equivalent of TNB reacted.

The products of the photoreaction of TNB after the oxidative workup were identified by using gas and high-pressure liquid chromatography to be naphthalene (80%), 1-naphthol (92%), and 1,1′-binaphthyl (4.4%). All yields are based on reacted TNB and are calculated from the assumption that 1 equiv of TNB will give 3 equiv of naphthalene-containing products. We presume that naphthylboronic acid accounts for the balance of the naphthalene from TNB.[12] In addition to the naphthalene-containing products, we observe four hydrocarbons confirmed to be cyclohexene dimers by mass spectrometric analysis. These results are summarized in eq 5.



(5)

The pathway previously suggested to lead to 1-NB should yield 1 equiv of binaphthyl for each equivalent of borene formed. In the earlier report it is suggested that the low yield of binaphthyl, compared to the estimated yield of 1-NB, is a consequence of its secondary reaction with naphthyl radical.[3] We examined this possibility using two independent techniques.

If binaphthyl is consumed in a secondary reaction after it is formed, or if its formation is a consequence of secondary photolysis of a primary product, then its yield should be a nonlinear function of the TNB consumed in the irradiation. A plot of the yield of binaphthyl (determined by HPLC) against the change in absorbance at 350 nm due to the TNB shows that it is constant and independent of the extent of conversion of TNB.

The second test of the susceptibility of binaphthyl to radical initiated decomposition directly probed its stability in cyclohexene solution under conditions designed to mimic the photolysis of TNB. Irradiation of deoxygenated cyclohexene solutions of *tert*-butyl peroxy-1-naphthoate (1.1×10^{-2} M) or benzoyl peroxide (7.0×10^{-3} M) in the presence of binaphthyl (3×10^{-3} M) until all of the peroxide is consumed does not cause a significant change in the concentration of binaphthyl.

Similarly, in an experiment designed to probe the source of binaphthyl in the photolysis of TNB, irradiation of *tert*-butyl peroxy-1-naphthoate in deoxygenated cyclohexene does not give a detectable yield of binaphthyl. This observation rules out, not unexpectedly, a bimolecular radical coupling path to binaphthyl.

These observations indicate that the measured yield of binaphthyl is its actual yield. Thus, if we accept that 1-NB

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[11] In a private communication, Professor Ramsey informed us that they photolyzed samples of TNB until the fluorescence had largely disappeared. Using this criterion they report that irradiation times ranged from 3 to 10 days. We have found that some primary product from photolysis of TNB fluoresces and that irradiation of optically dense solutions of TNB sometimes results in formation of a "polymeric" film on the walls of the reaction vessel, thus greatly slowing the rate of reaction.

[12] Naphthylboronic acid is stable to the oxidative hydrolysis conditions employed.[3]

and binaphthyl must be formed in a 1:1 ratio, then the *maximum* yield of 1-NB from photolysis of TNB is 5%.

If, as previously claimed,[3] the yield of *cis*-1,2-cyclohexanediol from photolysis of TNB and oxidation is 40%, then the yield of the precurser boracyclopropane must be at least as great, eq 4. We searched unsuccessfully by ^{11}B NMR spectroscopy for conclusive evidence of the existence of the boracyclopropane.

The proton-decoupled ^{11}B NMR spectrum of TNB in cyclohexene shows one absorption at δ -30.8 ($BF_3{\cdot}Et_2O$, external standard). The irradiation of a 1.2×10^{-2} M solution of TNB in deoxygenated cyclohexane was followed both by optical and ^{11}B NMR spectroscopy. At 50% conversion (as indicated by the UV spectrum), the ^{11}B NMR spectrum showed two absorptions, the original band at δ -30.8 and a new broader band at δ -46.0. Continued irradiation to 90% conversion results in the further decrease of the TNB band and the continued growth of the resonance at δ -46.0.

Since no authentic boracyclopropanes have been prepared, the chemical shift of boron in these compounds is unknown. The magnitude of the ^{11}B chemical shift is dominated by paramagnetic shielding, which makes simple estimation of the shifts from models difficult. However, comparison with the chemical shift[13] of *B*-phenylboracyclohexane (δ -77.5) and *B*-phenylboracyclopentane (δ -84.5) leads to an estimated chemical shift for the *B*-naphthylboracyclopropane of less than -70 ppm. No absorption is detected in this region of the ^{11}B NMR spectrum from photolysis of TNB. This result parallels our inability to detect the diol and further weakens the support for formation of 1-NB from photolysis of TNB.

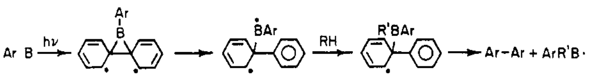
Discussion

The previous report by Ramsey and Anjo[3] that naphthyl borene is formed in high yield from photolysis of tri-1-naphthylboron was supported by two observations. We are unable to reproduce either of these. We cannot detect any of the cis diol, and we find that binaphthyl is stable to the reaction conditions. In addition, analysis of the ^{11}B NMR spectrum offers no support for the previous scheme. We are therefore forced to conclude that formation of 1-NB does not occur in greater than 5% yield from photolysis of TNB and that there is no compelling evidence for its formation even in this low yield.

The photochemistry of TNB appears to be dominated by simple carbon–boron bond homolysis. This path, which has been recognized in these materials for some time,[14] is illustrated in eq 6 and accounts easily for the formation of naphthalene, naphthol, and naphthylboronic acid.

$$Ar_3B \xrightarrow{h\nu} Ar_2B\cdot + Ar\cdot \xrightarrow{RH} Ar_2BH + ArH \qquad (6)$$

The origin of the low yield of binaphthyl is less clear. We note that electrochemical oxidation of the tetraphenylborate anion[15] leads to low yields of biphenyl without formation of free phenyl radicals. Similarly, photolysis of tetraaryl borates leads to biaryls in moderate yield.[8,9] In all cases, it appears that biaryl formation is intramolecular, monophotonic, and involves bond formation between the two carbons formerly bound to boron. These findings all point to the involvement of a bridged intermediate, but not necessarily to generation of a borene. One path illustrating this hypothesis is shown in eq 7.



(7)

Irradiation of TNB leads to some (ca. 5%) 1,1′ bridging, giving a boracyclopropyl diyl which in turn undergoes rearrangement and rearomatization to give a second diradical. This diradical may interact with solvent (cyclohexene) to abstract a hydrogen or add to the double bond (R' = H or $C_6H_{10}\cdot$). Finally, rearomatization with loss of $ArR'B\cdot$ gives the observed biaryl. The only evidence supporting this scheme is the formation of biaryl, but it does account for this observation without postulating a borene.

In sum, there is no evidence to support generation of a borene in the photolysis of triarylboranes. The photochemistry of these compounds appears to be dominated by simple carbon–boron bond homolysis. We are continuing our efforts to form and detect hypovalent boron intermediates that are analogous to carbenes.

Experimental Section

General Methods. ^1H NMR spectra were recorded on a Varian EM-390 spectrometer with Me_4Si as internal standard. ^{11}B NMR spectra were recorded on a Varian XL-100 spectrometer with $BF_3{\cdot}OEt_2$ as external standard. UV spectra were obtained with a Perkin-Elmer 552 spectrophotometer. Gas chromatographic analyses were performed with a Varian 2700 all-glass instrument equipped with flame ionization detectors. The chromatograms were quantitated with a Hewlett-Packard 3390A integrator. High pressure liquid chromatography was carried out on a Perkin-Elmer series 2 chromatograph. GC/MS analysis was performed on a VG 7070EH spectrometer. Elemental analyses were done by the Analytical Laboratory, Department of Chemistry, University of Illinois.

Materials. Trinaphthylboron was prepared according to the procedure of Lyle, DeWitt, and Pattison,[16] except that THF rather than ether was used in the Grignard reaction: mp 202–203 °C (lit.[16] mp 207–208 °C). Cyclohexene used for the photolysis was purified by first passing it through a column of activity 1 basic alumina and then distilling it from $LiAlH_4$. The cyclohexene was stored under N_2 in the dark. *cis*-1,2-Cyclohexanediol was prepared by the procedure of Wiberg and Saegebarth:[17] mp 96–97 °C (lit.[17] mp 97.5–99 °C). 1,1′-Binaphthyl was prepared by the method of Sakellarios and Kyrimis.[18] *tert*-Butyl peroxynaphthoate was obtained by using the synthesis described by Bartlett and Hiatt:[19] mp 51–52 °C (lit.[20] mp 52 °C). All compounds gave satisfactory elemental analyses.

Attempted Detection of Cis Diol from Photolysis of TNB. A solution of TNB (1×10^{-2} M, 1 mL) in cyclohexene was deoxygenated by purging with dry N_2 for 5 min or by freeze–pump–thaw degassing and then irradiated with the output of a Hanovia 450-W Hg lamp which was filtered through a uranium-glass or Pyrex filter sleeve. The progress of the reaction was monitored by withdrawing an aliquot of the irradiated solution and measuring the absorbance at 350 nm due to TNB (Figure 1). When the TNB had been consumed (ca. 45 min), the photolysis was stopped and worked up according to the standard procedure.[21] A 0.25-mL portion of a basic (0.2 M NaOH) aqueous ethanol (80%) solution of H_2O_2 (0.2 M) was added to the photolysis solution. The mixture was stirred vigorously for 10 min at room temperature, the excess H_2O_2 was reduced with $Na_2S_2O_5$, and the pH of the solution was adjusted to ca. 7. The water layer was saturated with NaCl and the organic phase was separated

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from chromatographic analysis.

Gas chromatography was performed with a 6 ft × $^1/_4$ in. Carbowax 20 M on Chromosorb W column. The column temperature was programmed from 110 to 180 °C over a period of 9 min, and the flow rate was 30 mL/min. Under these conditions *cis*-cyclohexane-1,2-diol has a rentention time of 6.8 min. The analysis of the photolysis solution showed no peak at this retention time.

Control experiments showed that the diol survives the workup procedure and that it is not bound to or decomposed by any of the photolysis products. A limit for detectability of cis diol of 2% was established by adding an amount of cis diol corresponding to this yield to cyclohexene, and then subjecting this solution to the oxidative workup described above.

Binaphthyl, naphthol, and the cyclohexene dimers were also resolved by gas chromatography. The binaphthyl and naphthol were identified and quantified by reference to authentic samples. The cyclohexene dimers all showed molecular ions at m/e 162 in the mass spectrum.

High-pressure liquid chromatographic analysis on a C 8/10 column eluting with 28.5% H_2O/71.5% CH_3CN confirmed the identity of the binaphthyl and showed that its formation was not a consequence of thermolysis of some intermediate in the injection port of the gas chromatograph.

Survival of 1,1′-Binaphthyl in Cyclohexene Solution in the Presence of Radicals. A solution of binaphthyl (9.2×10^{-4} M) and *tert*-butyl peroxynaphthoate (1.1×10^{-2} M) in cyclohexene was prepared and deoxygenated. The solution was irradiated through Vycor glass until all of the peroxide was reacted. Gas chromatographic analysis of the reaction mixture after the irradiation showed the concentration of binaphthyl to be 9.1×10^{-4} M.

Acknowledgment. Financial support of this work by the National Science Foundation is gratefully acknowledged.

Registry No. TNB, 6962-78-3; 1-NB, 62981-86-6.

Kinetics of the Thermal Decomposition of Benzenesulfinyl Azide[1]

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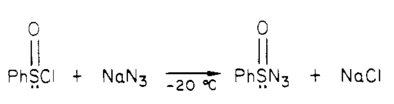
Received July 25, 1983

Kinetic studies show that the thermal decomposition of benzenesulfinyl azide (2) follows first-order kinetics in the presence of a variety of substrates. The typical rate constant at 20 °C was 3×10^{-3} s^{-1}. In acetonitrile, $\Delta H^{\ddagger} = 19$ kcal/mol and $\Delta S^{\ddagger} = -4$ eu. These results are consistent with a postulated dipolar sulfinylnitrene intermediate. Thermal decomposition of 2 in the presence of strong nucleophiles, however, was not first order. Thiols and amines caused significant decreases in the rate.

A limited kinetic study of the thermal decomposition of arenesulfinyl azides has been reported as part of a paper on their reactions with sulfoxides.[2] In that report, rate constants were determined only at 0 °C by using cumbersome apparatus at constant atmospheric pressure. The scope of added substrates was limited to sulfoxides. The present report describes results obtained at different temperatures and at constant volume for the reaction of benzenesulfinyl azide (2) with a wide variety of substrates.

Results

Solutions of 0.2 M benzenesulfinyl azide (2) were prepared at −20 °C (±5 °C) from benzenesulfinyl chloride (1)



$$\mathrm{Ph\overset{O}{\overset{\|}{S}}Cl} + \mathrm{NaN_3} \xrightarrow{-20\ ^\circ\mathrm{C}} \mathrm{Ph\overset{O}{\overset{\|}{S}}N_3} + \mathrm{NaCl}$$

1 2

in either 1,2-dimethoxyethane (DME) or acetonitrile. Kinetic runs were recorded as increases in pressure at constant volume and temperature by means of a pressure transducer.

Table I. Kinetics of Benzenesulfinyl Azide Decomposition in the Presence of Various Substrates in DME at 20.0 °C

added substrate	mole ratio of azide to substrate[a]	average rate constants,[b] s^{-1} × 10^3	number of runs
		2.62 ± 0.08	5
H_2O	1:2	2.65 ± 0.03	1
H_2O	1:10	2.88 ± 0.03	1
CH_3OH	1:2	2.50 ± 0.08	6
CH_3OH	1:10	2.70 ± 0.17	5
$CH_2{=}CHCH_2OH$	1:2	2.47 ± 0.05	2
$CH_2{=}CHCH_2OH$	1:10	2.67 ± 0.05	3
$CH_3CH_2CH_2CH_2OH$	1:2	2.47 ± 0.07	3
$CH_3CH_2CH_2CH_2OH$	1:10	2.55 ± 0.07	3
$HC{\equiv}CCH_2OH$	1:2	2.48 ± 0.03	4
$CH_2{=}CHCHOHCH_3$	1:2	3.27 ± 0.03	3
$CH_2{=}CHCH_2CO_2H$	1:2	2.37 ± 0.10	5
$CH_3C({=}O)CH_3$	1:2	2.58 ± 0.03	2
$CH_3C({=}O)CH_3$	1:10	2.57 ± 0.03	1
$CH_3S({=}O)CH_3$	1:2	2.47 ± 0.02	1

[a] Initial concentrations of the azide range between 0.2 and 0.3 M. [b] ± standard deviations.

Table I lists the first-order rate constants for the decomposition of 2 at 20 °C in DME alone and with 2 and 10 equiv of various added substrates. Good first-order plots were obtained to three half-lives in most cases. A nonlinear least-squares curve-fitting program was used to calculate the rate constants from eq 1, where P_∞ and P_0

$$\ln((P_\infty - P_0)/(P_\infty - P_x)) = kt \qquad (1)$$

are the pressure related recorder response at infinite and zero time, respectively; P_x is the response at different times during the reaction. The standard deviations for each

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(1) Abstracted in part from the Masters Thesis of C.N.A., California State University, Long Beach, CA, 1978.

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