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In situ formation of alkenyl- and alkynylboranes for Diels–Alder reactions by boron–silicon exchange with alkenyl- and alkynylsilanes

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Abstract

A variety of alkenylsilanes underwent transmetallation with BBr_3 to afford the corresponding alkenyldibromoboranes in solution. Similarly, the transmetallation of alkynylsilanes with BCl_3 afforded alkynyldichloroboranes. A number of the alkenyl- and alkynylboranes are novel and useful Diels–Alder dienophiles. An ionic mechanism for the transmetallation is proposed. © 1997 Elsevier Science S.A.

Keywords: Boron; Silicon; Diels–Alder reactions

1. Introduction

Alkenyl- and alkynylboranes are extremely useful synthetic intermediates in metal-catalyzed coupling reactions [1], addition reactions [2], and Diels–Alder reactions [3,4]. They have usually been synthesized by hydro- or haloboration of alkynes [5] (see also Ref. [6]), or by reactions of boranes with alkenyl or alkynyl lithium, mercury, zinc, zirconium, or tin reagents [7,8]. Alkenyl- and alkynyltributylstannanes are particularly versatile borane precursors via their boron–tin exchange reaction with haloboranes. This generally clean and quantitative transmetallation is suitable for the in situ formation and reaction of unstable boranes and allows the site- and stereospecific introduction of a boryl group into complex molecules [4,9,10]. However, this methodology suffers from the normal toxicity and atom-economy disadvantages of using tin reagents.

The corresponding silanes would be highly preferable precursors. Because of our interest in the Diels–Alder reactions of alkenyl- and alkynylboranes, we were excited by promising indications in the literature that boron–silicon exchange could be used to access these dienophiles. Several syntheses of arylboranes by electrophilic aromatic substitutions on aromatic silanes with haloboranes have been reported [11]. Similar reactions have been used to form the parent vinyldichloroborane and vinyldibromoborane from vinyltripropylsilane [12], and a recent report has used this process in the synthesis of *E*-alkenylboronic acid derivatives [13]. We report here the results of our studies of the formation of alkenyl- and alkynyldihaloboranes from the reaction of the corresponding silanes with BBr_3 or BCl_3. We also report our observations concerning the mechanism of this reaction, and on the utility of the exchange in forming novel alkenyl- and alkynylborane dienophiles for Diels–Alder reactions.

2. Results and discussion

As had been reported by Kaufmann and Mikhail [12], vinyltrimethylsilane reacts with BBr_3 to form vinyldibromoborane (**2**). Kaufmann and Mikhail had used methylene chloride as solvent, while we preferred hexanes because subsequent Diels–Alder reactions were usually cleaner. The transmetallation reaction is much slower in hexanes but proceeds rapidly and very cleanly (by NMR) at 75 °C. The isolation of **2** has been previously accomplished but is plagued by difficulties [8], and was not attempted here. The formation of **2** in solution was characterized by the appearance of broad resonances in the ^1H NMR at δ 6.60 (2 H) and 6.35 (1 H) identical with material formed from vinyltributyltin, and an ^{11}B NMR resonance at δ 55.4 (55.2 [14]). The

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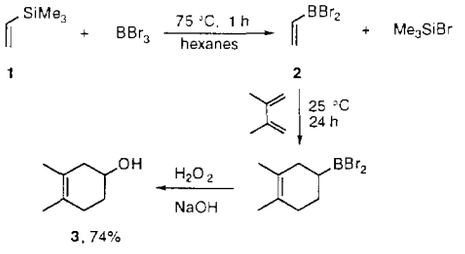
intermediate **2** was also characterized by its facile Diels–Alder reaction with 2,3-dimethylbutadiene. An oxidative work-up afforded the cyclohexenol **3** in 74% yield. This is the first example of a Diels–Alder reaction with **2**, and its high reactivity should make it a widely useful dienophile.

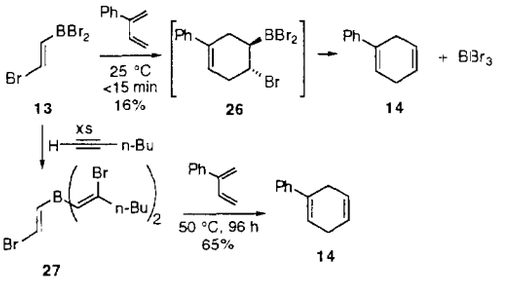


Our results with other alkenyl- and alkynylsilanes are summarized in Table 1. The reaction of the alkenylsilanes with BBr_3 proceeded smoothly, but BCl_3 reacted too sluggishly in hexanes to be useful. In contrast, the reactions of alkynylsilanes with BBr_3 were not successful at all, producing polymeric materials, while the transmetallations with BCl_3 went cleanly. Two of the alkenylboranes in Table 1 had been previously prepared by other methods—**5** from the corresponding tributylstannane [10] and **13** from bromoboration of acetylene with BBr_3 [15]—and these exhibited ^{1}H NMR spectra identical with previous observations.

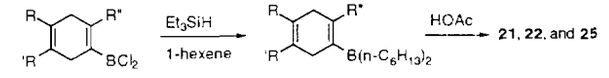
Attempts to form vinyldialkylboranes by the reaction of halodialkylboranes with vinylsilanes failed. For example, no vinylborane formation was observed in an attempted reaction of vinyltrimethylsilane (**1**) with *B*-bromo-9-BBN even after prolonged heating at 100 °C.

The transmetallations with cis- and trans-propenylsilanes **7** and **11** took a surprising course. The reaction of each with BBr_3 produced intermediates with indistinguishable ^{1}H NMR spectra. Based on an Hα–Hβ coupling constant of 17.5 Hz, the major isomer of the borane **8** produced was assigned for both cases as having trans stereochemistry. This stereochemistry was confirmed from Diels–Alder reactions of the intermediates, which produced equivalent mixtures of trans and cis adducts **9** and **10**, with the trans product **9** predominating. We have previously shown that the Diels–Alder reaction of *cis*-propenyldibromoborane is stereospecific, and no isomerization of the borane was observed [10]. The non-stereospecificity of the boron–silicon transmetallation here is in contrast to the stereospecificity of boron–tin exchange [10]. The mechanistic implications of this observation will be discussed below.

Although **13** appeared to be a highly reactive dienophile, the initial Diels–Alder adduct (**26**) would spontaneously eliminate BBr_3. The presence of large amounts of the strongly acidic BBr_3 would apparently lead to acid-mediated decomposition of the diene and/or product, resulting in very low yields. To avoid this problem, **13** was first treated with *n*-hexyne. The haloboration of *n*-hexyne by **13** results in the formation of **27**, which selectively reacts at the least-substituted double bond and cleanly forms **14** in good yield. The good reactivity of **27** and the facility of the elimination from the Diels–Alder adduct should make it attractive for use as an acetylene equivalent in Diels–Alder reactions.



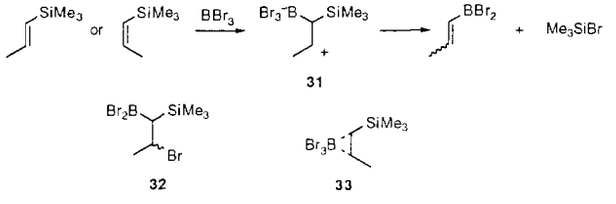
The structures of the alkynylboranes **20** and **24** were best confirmed by their Diels–Alder reactions. The intermediacy of the Diels–Alder adducts **28–30** was supported by the observation of new peaks in the ^{1}H NMR at δ 5.6 (1 H) and 3.0 (m, 4 H). The best work-up for these reactions involved the conversion of **28–30** to the corresponding dihexylboranes by the Matteson procedure [16] prior to protodeboronation which forms the observed isolated products. The structure and substitution pattern in **21**, **22**, and **25** was in each case confirmed by aromatization of the 1,4-cyclohexadienes using DDQ. A detailed examination of the Diels–Alder and other reactions of alkynyldichloroboranes will be presented in another paper.



28 R=Me, R'=H, R"=Ph
29 R=H, R'=Me, R"=Ph
30 R=H, R'=Me, R"=n-Bu

Several of the observations above suggest an ionic mechanism for the transmetallation, proceeding through a carbocation such as **31**. This would be parallel to the presumed mechanism for boron–silicon exchange on aryl groups. The intermediacy of the zwitterionic **31** would readily account for the much greater rate of transmetallation in methylene chloride over hexanes, and would be consistent with the general reactivity trend of: **15** > **7** ≈ **11** > **4** ≈ **1**. This would also explain

the formation of the same isomeric mixture of **8** from **7** and **11**, if **31** has sufficient lifetime to rotate about a single bond. The hyperconjugative effect of a β-trimethylsilyl substituent would be important in stabilizing the cation, particularly in the case of the parent vinylsilane. This hyperconjugation could hinder rotation in **31**, but the loss of stereochemistry in a mercury analog of **31** has been noted by Soderquist and Thompson [17]. Alternatively, the transmetallation could occur by an initial bromoboration to form **32** followed by elimination of Me_3SiBr. The elimination would be facilitated by the presence of the borane Lewis acids in solution. However, the haloboration of **7** and **11** would produce different diastereomers of **32**, and it would be unusual for these diastereomers to eliminate to form the same mixture of isomers of **8**. Suzuki and coworkers have proposed the intermediacy of a three-membered ring in the haloboration of acetylene [15]. An analogous mechanism for the transmetallation would involve **33**. This would not explain the much greater reactivity of **7** and **11** over **4**. Overall, our observations seem most consistent with the formation of **31** being the key step in the transmetallation.



The high reactivity and selectivity of vinylboranes as Diels–Alder dienophiles, combined with the versatility of the products, makes readily available a wide variety of structures that were previously difficult to obtain. The ability to use vinylsilanes as precursors to these dienophiles should greatly extend the practicality and utility of Diels–Alder reactions of vinylboranes.

3. Experimental section

All reactions were carried out in dried glassware under a positive pressure of nitrogen using standard syringe and septa techniques. The NMR spectra of aliquots of reaction mixtures containing boranes were taken as neat liquids in glass capillaries centered in NMR tubes, and were referenced approximately based on the internal trimethylsilyl bromide, trimethylsilyl chloride, or tributylstannyl bromide. Silanes **1** and **12**, isoprene, 2,3-dimethylbutadiene, 1-hexyne, and solutions of BCl_3 and BBr_3 in hexanes or methylene chloride were used as-purchased from Aldrich. The silanes **4** [17], **7** [18], **11** [18], **15** [18], **19** [19], and **23** [19] were prepared by literature procedures.

3.1. Formation of alkenyldibromoboranes 2, 5, 8, 13 and 16: general procedure

To the commercial 1.0 M solution of BBr_3 in hexanes or CH_2Cl_2 was added dropwise 1.2–1.6 equiv. of silanes **1**, **4**, **7**, **11**, **12** or **15**. The resulting homogeneous mixtures were heated to the temperatures indicated in Table 1 for the indicated times. The mixtures were then analyzed by NMR as described above with the results shown in Table 1.

3.2. Formation of alkynyldichloroboranes 20 and 24: general procedure

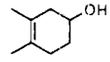
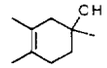
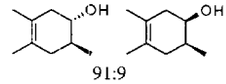
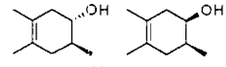
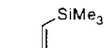
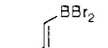
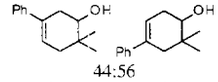
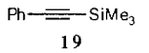
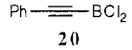
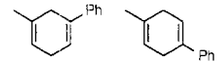
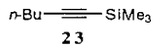
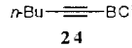
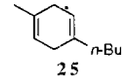
To the commercial 1.0 M solution of BCl_3 in hexanes or CH_2Cl_2 was added dropwise 1.05–1.1 equiv. of silanes **19** or **23**. The resulting homogeneous mixtures were stirred at 25 °C for the times indicated in Table 1. The mixtures were then analyzed by NMR as described above with the results shown in Table 1.

3.3. Diels–Alder reactions of 2, 5, 8, and 16

In a typical procedure, a solution of **2** was prepared by heating a mixture of 198 mg (2.0 mmol) of vinyltrimethylsilane and 1.4 ml (1.4 mmol) of 1.0 M BBr_3 in hexanes to 75 °C for 1 h. This solution was cooled to 0 °C and 80.6 mg (0.98 mmol) of 2,3-dimethylbutadiene was added. The reaction mixture was stirred at 25 °C for 24 h. There was then added successively 5 ml of THF, 3 ml of 3 N NaOH and 3 ml of 30% H_2O_2 at 0 °C, and the mixture was warmed to room temperature and stirred for 2 h. The resulting mixture was extracted with three 15 ml portions of 1:1 diethyl ether–petroleum ether, and the combined extracts were rinsed with 20 ml of brine, dried over anhydrous $MgSO_4$, and the solvent was removed on a rotary evaporator. The residue was flash chromatographed on a 3/4 in ×8 in silica gel column using 13% EtOAc–petroleum ether as eluent to afford 92 mg (74%) of the known [20] 3,4-dimethyl-3-cyclohexen-1-ol (**3**) as a colorless liquid: ^{1}H NMR δ 3.96–3.80 (m, 1 H), 2.30–2.12 (m, 1 H), 2.08–1.70 (m, 5 H), 1.65 (br s, 7 H); ^{3}C NMR δ 125.25 (C), 122.73 (C), 67.47 (CH), 40.61 (CH_2), 31.44 (CH_2), 29.67 (CH_2), 19.10 (CH_3), 18.56 (CH_3).

The Diels–Alder reactions of **5**, **8**, and **16** were carried out by similar procedures with the conditions and results shown in Table 1. Spectral properties for the known [10] 1,3,4-trimethyl-3-cyclohexen-1-ol (**6**): ^{1}H NMR δ 2.24–1.80 (m, 4 H), 1.78–1.34 (m, 9 H), 1.18 (s, 3 H); ^{13}C NMR δ 124.59 (C), 123.24 (C), 69.11 (C), 45.92 (CH_2), 35.63 (CH_2), 29.32 (CH_2), 28.36 (CH_3), 19.17 (CH_3), 18.55 (CH_3). Spectral properties for

Table 1
Formation and Diels–Alder reactions of alkenyl- or alkynyldihaloboranes

Silane	BX_3 Reaction conditions [a]	Alkenyl or alkynylborane	NMR key peaks	Diene Reaction conditions, yield	Product(s)
 1	BBr_3 75°C, 1 h hexanes	 2	^1H δ 6.60 (2H), 6.35 (1H); ^{11}B δ 55.4	2,3-dimethyl butadiene 25°C, 24 h 74% [b]	 3
 4	BBr_3 55°C, 32 h hexanes	 5	^1H δ 6.12 (br s), 6.48 (br s); k^{11}B δ 56.9	2,3-dimethyl 25°C, 9 h 72% [b]	 6
 7[c]	BBr_3 25°C, 22 h hexanes	 8[d]	^1H δ 1.90 (d, 3 H), 6.32 (d, J = 17.5 Hz, 1 H), 7.23 (m, 1 H); ^{11}B δ 56.9 [d]	2,3-dimethyl butadiene 25°C, 3 days 71% [b]	 91:9 9 10
 11[e]	BBr_3 25°C, 23 h hexanes	 8[d]		2,3-dimethyl butadiene 25°C, 4 days 74% [b]	 90:10 9 10
 12	BBr_3 25°C, 72 h hexanes or 25°C, 3.5 h CH_2Cl_2	 13	^1H δ 8.00 (d, J = 16 Hz), 7.10 (d, J = 16 Hz)	xs 1-hexyne, 25°C, 21 h; 2-phenyl-butadiene, 50 °C, 96 h 65%	 14
 15	BBr_3 25°C, 18 h hexanes	 16	^1H δ 6.25 (s, 1 H), 2.20 (s, 3H), 1.93 (s, 3 H)	2-phenyl butadiene 25°C, 47 h 22 % [b]	 44:56 17 18
 19	BCl_3 25°C, 55 h hexanes or 25°C, 3 h CH_2Cl_2	 20	^{11}B δ 42.8 (very br)	isoprene 55°C, 96 h hexanes 60% [f] 25°C, 15 min CH_2Cl_2 73% [f]	 79:21[g] 21 22
 23	BCl_3 25°C, 3.5 h CH_2Cl_2	 24	^1H δ 2.45 (t); ^{11}B δ 42.3	isoprene 25°C, 2.5 h 49% [f]	 25

[a] The reaction time is based on the disappearance of starting silanes in the ^1H NMR.
[b] Oxidative work-up using H_2O_2–NaOH.
[c] Contaminated with 10% of the trans isomer.
[d] The trans isomer appeared to predominate but the ratio of isomers could not be determined from the ^1H NMR. The spectra obtained starting from the cis and trans silanes **7** and **11** did not differ in any distinguishing way.
[e] Contaminated with 13% of the cis isomer.
[f] Work-up by treatment with excess 1-hexene–Et_3SiH then HOAc.
[g] The same mixture of regioisomers was obtained in both solvents.

trans-3,4,6-trimethyl-3-cyclohexen-1-ol (**9**): ^1H NMR δ 3.44 (m, 1 H), 2.30–1.50 (m, 12 H), 0.96 (d, 3 H); ^{13}C NMR δ 124.82 (C), 122.98 (C), 73.01 (CH), 40.32 (CH_2), 39.33 (CH_2), 35.97 (CH), 18.76 (CH_3), 18.40 (CH_3), 17.50 (CH_3). The previously synthesized [10] minor cis isomer (**10**) exhibited a characteristic ^1H NMR peak at δ 3.78. Characteristic spectral properties for 6,6-dimethyl-3-phenyl-3-cyclohexen-1-ol (**17**) and 6,6-dimethyl-4-phenyl-3-cyclohexen-1-ol (**18**): ^1H NMR δ 7.45–7.15 (m, 5 H), 5.95 (br s, 1 H, minor product), 5.78 (br s, 1 H, major product), 3.72–3.55 (m, 1 H), 2.65–1.55 (m, 5 H), 1.18–1.00 (m, 6 H).

3.4. 1-Phenyl-1,4-cyclohexadiene (*14*)

A mixture of 288 mg (1.6 mmol) of **12** and 1.5 ml (1.5 mmol) of 1.0 M BBr_3 in hexanes was stirred at 25 °C for 72 h and then 394 mg (4.8 mmol) of 1-hexyne was added at −78 °C. After warming to room temperature, the mixture was stirred for 21 h and 126 mg

(0.97 mmol) of 2-phenylbutadiene was added at 0 °C. The mixture was heated to 50 °C for 96 h, then cooled to 0 °C for the addition of 5 ml of THF and 3 ml of 3 N NaOH. The resulting mixture was extracted with three 15 ml portions of petroleum ether, and the combined extracts were dried over anhydrous $MgSO_4$. The solvent was removed on a rotary evaporator and the residue oil was filtered through a 1″ pad of silica gel, rinsing with 150 ml of petroleum ether. The solvent was removed from the filtrate on a rotary evaporator to afford 98.4 mg (65%) of the known [21] **14** as a white solid: ^{1}H NMR δ 7.60–7.20 (m, 5 H), 6.20 (br s, 1 H), 5.85 (m, 2 H), 3.15 (m, 2 H), 2.95 (m, 2 H); ^{13}C NMR δ 141.78 (C), 133.91 (C), 128.39 (CH), 127.01 (CH), 125.04 (CH), 124.49 (CH), 123.77 (CH), 121.84 (CH), 27.78 (CH_2), 27.12 (CH_2).

3.5. Diels–Alder reactions of 20 and 24

In a typical procedure, a solution of **24** was prepared from a mixture of 334 mg (2.2 mmol) of **23** and 2.1 ml (2.1 mmol) of 1.0 M BCl_3 in methylene chloride was stirred at 25 °C for 3.5 h and then 106 mg (1.6 mmol) of isoprene was added at −78 °C. The mixture was stirred at 25 °C for 2.5 h and then 0.52 g (6.2 mmol) of 1-hexene and 0.73 g (6.3 mmol) of triethylsilane were added at −78 °C. After the mixture was stirred at −78 °C for 30 min, it was warmed to room temperature and stirred for another 2 h, and all volatiles were removed under vacuum. The residue was dissolved in 5 ml of THF, 1.5 ml (27 mmol) of glacial acetic acid was added, and the mixture was stirred at room temperature for 2 h. A standard extraction and purification afforded 115 mg (49%) of **25** as a colorless liquid: ^{1}H NMR δ 5.40 (br s, 2 H), 2.55 (br s, 4 H), 1.95 (t, J = 8 Hz, 2 H), 1.65 (br s, 3 H), 1.3 (m, 4 H), 0.85 (t, J = 8 Hz, 3 H); ^{13}C NMR δ 135.27, 131.28, 118.87, 118.13, 36.84, 31.81, 30.18, 29.87, 22.88, 22.48, 13.89. Aromatization of **25** using DDQ afforded 4-*n*-butyltoluene: ^{1}H NMR δ 7.10 (s, 4 H), 2.62 (t, J = 9 Hz, 2 H), 2.35 (s, 3 H), 1.62 (m, 2 H), 1.40 (m, 2 H), 0.97 (t, J = 9 Hz, 3 H); ^{13}C NMR δ 139.81, 134.90, 128.90, 128.28, 35.22, 33.80, 22.37, 20.96, 13.95.

The Diels–Alder reactions of **20** were carried out by similar procedures with the conditions and results shown in Table 1. Characteristic spectral properties for **21** and **22**: ^{1}H NMR δ 7.52–7.20 (m, 5 H), 6.18 (m, 1 H, major), 5.80 (m, 1 H, minor), 5.60 and 5.54 (m, 1 H combined), 2.95 (m, 4 H), 1.82 and 1.78 (s, 6 H combined). Aromatization of the 79:21 mixture of **21** and **22** using DDQ afforded a 79:21 mixture of 3-phenyltoluene and 4-phenyltoluene: ^{1}H NMR δ 7.68–7.58 (m, 2 H), 7.56–7.15 (m, 7 H), 1.48 and 1.45 (s, 3 H, combined); ^{13}C NMR δ 141.37, 141.24, 138.3, 129.46, 128.66, 127.97, 127.16, 126.96, 124.26, 21.55 (Some carbon atoms of the minor regioisomer were not observed).

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