

SYNTHESIS OF BIS(DIPHENYLPHOSPHINIO)OCTAHYDRO-*closo*-DECABORATES $L_2B_{10}H_8$ ($L = Ph_2PH$) AND 1-(DIPHENYLPHOSPHINIO)-NONAHYDRO-*closo*-DECABORATES(1-) $[1-LB_{10}H_9]^-$ ($L = Ph_2PH, Ph_2P(OH)$) BY THE PALLADIUM-CATALYZED REACTION OF Ph_2PH WITH 1-DIAZONONAHYDRO-*closo*-DECABORATE(1-) $[1-N_2B_{10}H_9]^-$

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Dedicated to Dr Stanislav Heřmánek on the occasion of his 70th birthday in recognition of his outstanding contributions to the areas of borane and NMR spectroscopy. He has also devoted his energy to the utilization of chemistry for the protection and welfare of mankind. It has been a privilege to collaborate with him.

(PPh_3)₂PdCl₂ and CuI, used in low concentration as catalysts, favour the replacement of the diazo group of $[1-N_2B_{10}H_9]^-$ by Ph_2PH . Owing to its instability $[1-(Ph_2PH)B_{10}H_9]^-$ could not be isolated in a pure form and was obtained as a mixture with $[1-Ph_2P(OH)B_{10}H_9]^-$, but pure samples of $[1-Ph_2P(OH)B_{10}H_9]^-$ were easily obtainable. (PPh_3)₂PdCl₂ also acted as a reactant, the reduction of Pd(II) to Pd(0) allowing the formation of the neutral substituted boranes 1,6-(Ph_2PH)₂B₁₀H₈ and 1,10-(Ph_2PH)₂B₁₀H₈. These two isomers were separated by chromatography. All the products were characterized by ¹¹B and ³¹P NMR and mass spectrometry techniques.

Key words: Phosphines; Boranes; Hydroborate anions; 1-Diazononahydro-*closo*-decaborate(1-); 1-Phosphinononahydro-*closo*-decaborate(1-); Palladium.

Lipophilic anions can be obtained by attaching appropriate organic groups to the cage of a *closo* hydroborate anions. Alkylated polyhedral borane anions are of particular interest for boron neutron capture therapy^{1,2}. Other types of application can be envisaged for lipophilic hydroborate derivatives. We have recently prepared monoanions containing various amino groups bound either to the $[B_{12}H_{12}]^{2-}$ (ref.³) or the $[B_{10}H_{10}]^{2-}$ (ref.⁴) with the aim of

obtaining extractants of radioactive cations (Cs^+ , Sr^{2+} , Eu^{3+}). The reaction of $[(CH_3)_4N][N_2B_{10}H_9]$ with diphenylphosphine described here has been investigated in order to obtain $[phosphine-B_{10}H_9]^-$ anions. $[Amine-B_{10}H_9]^-$ anions have been successfully prepared by the analogous reaction using primary or secondary amines⁴. In contrast, the reaction took place only sparingly when tertiary amines were used, apparently due to sterical reasons, and the decomposition reaction of $[1-N_2B_{10}H_9]^-$ leading to the formation of $[B_{20}H_{18}]^{2-}$ prevailed. The choice of diphenylphosphine over other phosphine derivatives was motivated by better reactivities of secondary species, bulkiness of phenyl substituents, and also by practical reasons: the phosphine is liquid in the 120–150 °C temperature range with a low volatility and a low toxicity. The phosphine was used in excess in order to act not only as a reactant, but also as a solvent, because all the other effective solvents that we have tested reacted with the starting materials.

Anions containing phosphorus were obtained in reasonable yield provided that $(PPh_3)_2PdCl_2$ and CuI , acting as catalysts, were added to the reaction mixture. The addition of palladium catalyst in low concentration also caused the formation of neutral phosphine derivatives $(Ph_2PH)_2B_{10}H_8$. The complexity of the reaction mixture obtained was increased by the presence of $[1-Ph_2P(OH)B_{10}H_9]^-$ due to side reactions.

EXPERIMENTAL

General

All syntheses were carried out under argon using standard inert atmosphere techniques. $(NH_4)_2B_{10}H_{10}$ was provided by Katchem Ltd., Prague. Ph_2PH (purity >95%) was purchased from Fluka and no phosphine oxide was detected as an impurity by 1H NMR. However, in order to remove traces of oxide, several samples were tentatively distilled from $LiAlH_4$ under reduced pressure prior to use, but without producing any effect on the presence of $[1-Ph_2P(OH)B_{10}H_9]^-$ in the final product. $[(CH_3)_4N][N_2B_{10}H_9]$ was prepared according to published methods^{4–9}.

Physical Measurements

NMR spectra were obtained on a Bruker WF-300 spectrometer. ^{11}B NMR spectra were recorded at 96.29 MHz (external reference $Et_2O \cdot BF_3$, positive values downfield) and ^{31}P NMR spectra at 146.2 MHz (external reference 85% H_3PO_4). IR spectra were recorded on a Nicolet Magna 550 FT spectrometer using KBr pressed discs. Mass spectrometry measurements were performed in the Mass Spectrometry Laboratory, Central Analytical Service of the CNRS, Solaize, France. A hybrid mass spectrometer ZAB-2-SEQ (Micros) was used for the analyses by the SIMS technique (caesium beams). Other measurements were performed using the Electro Spray (ES) method on a VG-platform micromass spectrometer. Samples were intro-

duced in the spectrometer as acetonitrile solutions. A B70 Setaram apparatus was used for TGA measurements.

Chromatography

TLC was performed on laboratory made DEAE cellulose (refs.^{3,10} and references therein) with 2 M aqueous solution of NH_4NO_3 as eluent. The spots were detected by spraying with a solution of PdCl_2 (0.5% in 5% HCl). For the separation of products, silicagel gel 70–230 mesh (Aldrich) was used to fill glass columns.

Synthesis in Absence of Catalyst

A suspension of $\text{NMe}_4[\text{N}_2\text{B}_{10}\text{H}_9]$ (1.5 g, 6.85 mmol) in diphenylphosphine (10 ml), was stirred at 120 °C for 15 h. Toluene (50 ml) was added to the reaction mixture in order to dissolve the excess of phosphine and precipitate the ionic species. The mixture was filtered and the solid was washed with toluene (2×20 ml) and dried under vacuum. The solid (1.7 g) still contained $[(\text{CH}_3)_4\text{N}][\text{N}_2\text{B}_{10}\text{H}_9]$ as indicated by TLC (spot at R_F 0.45) and IR (strong absorption at $2\,240\text{ cm}^{-1}$). The ^{11}B NMR analysis of the product in acetonitrile showed that both $[\text{1-Ph}_2\text{P}(\text{OH})\text{B}_{10}\text{H}_9]^-$ and $[\text{1-(Ph}_2\text{PH})\text{B}_{10}\text{H}_9]^-$ were present in low concentration (NMR data reported in Table I). After heating for 38 h the reaction was still incomplete, but after a 78 h reaction time the diazo derivative had completely disappeared. A solid (2.15 g) was obtained as described above and its composition determined by ^{11}B NMR in acetonitrile was: $[(\text{CH}_3)_4\text{N}]_2[\text{B}_{10}\text{H}_{10}]$ 70% (δ , ppm: -0.3, d, 2B; -29.33, d, 8B) (ref.⁴ and references therein), $[(\text{CH}_3)_4\text{N}]_2[\text{B}_{20}\text{H}_{18}]$ 2% (δ , ppm: 30.55, d, 2B; 15.97, s, 2B; -6.84, d, 2B; -12.35, d, 4B; -15.86, d, 4B; -19.29, d, 4B; -25.33, d, 2B) (ref.⁴ and references therein), $[(\text{CH}_3)_4\text{N}][\text{1-(Ph}_2\text{PH})\text{B}_{10}\text{H}_9]$ 11% and $[(\text{CH}_3)_4\text{N}][\text{1-Ph}_2\text{P}(\text{OH})\text{B}_{10}\text{H}_9]$ 17%. The solid product was dissolved in water and passed on C20 H Duolite resin charged with protons. When triethylammonium chloride was added to the acidic solution, a precipitate containing only

TABLE I

^{11}B NMR data for the products formed by the reaction of $[\text{1-N}_2\text{B}_{10}\text{H}_9]^-$ with Ph_2PH catalyzed by $(\text{PPh}_3)_2\text{PdCl}_2$ and CuI at 150 °C

Compound	Chemical shift (ppm) (multiplicity, relative intensity, coupling)
$\text{NMe}_4[\text{Ph}_2\text{PHB}_{10}\text{H}_9]^a$	+15.55 d, 1 B (B-P); -1.41 d, 1 B; -22.35 d, 4 B; -24.07 d, 4 B
$\text{NMe}_4[\text{Ph}_2\text{P}(\text{OH})\text{B}_{10}\text{H}_9]^a$	+9.24 broad, 1 B (B-P); +1.40 d, 1 B; -26.35 d, 4 B; -27.88 d, 4 B
$1,10\text{-(Ph}_2\text{PH)}_2\text{B}_{10}\text{H}_8^b$	+1.58 d, 2 B (B-P); -19.17 d, 8 B, $J(\text{B,H}) = 136$ Hz
$1,6\text{-(Ph}_2\text{PH)}_2\text{B}_{10}\text{H}_8^{b,c}$	+16.4 d, B_{10} , $J(\text{B,H}) = 148$ Hz; -8.16 d, B_1 , $J(\text{B,P}) = 189$ Hz; -18.92 d, B_8 ; -20.20 d, (B_2, B_3); -21.10 d, (B_4, B_5); -23.33 d, (B_7, B_9), $J(\text{B,H}) = 123$ Hz; -28.00 d, B_6 , $J(\text{B,P}) = 123$ Hz

^a In CH_3CN . ^b In CD_2Cl_2 . ^c The chemical shift values are consistent with those reported for $(1,6\text{-PhMe}_2\text{P})_2\text{B}_{10}\text{H}_9$ (ref.¹⁸): +11.7 d, B_{10} ; -4.5 d, B_1 ; -21.9 d, B_8 ; -22.5 d (B_2, B_3); -23.6 d (B_4, B_5); -25.3 d (B_7, B_9); -26.1 d, B_6 .

$[(C_2H_5)_3NH][1-(Ph_2PH)B_{10}H_9]$ and $[(C_2H_5)_3NH][1-Ph_2P(OH)B_{10}H_9]$ appeared. A solid mixture of these two salts (0.2 g) was recovered by filtration.

When the same mixture of starting materials was heated at 150 °C $[(CH_3)_4N][N_2B_{10}H_9]$ was completely consumed after 15 h. The composition of the product formed (^{11}B NMR analysis) was: $[1-(Ph_2PH)B_{10}H_9]^-$ 11%, $[1-Ph_2P(OH)B_{10}H_9]^-$ 15%, $[B_{10}H_{10}]^{2-}$ 20% and $[B_{20}H_{18}]^{2-}$ 54%.

Reaction in Presence of $(PPh_3)_2PdCl_2$ and CuI

Several attempts showed that the addition of catalyst was ineffective at 120 °C. In a typical reaction performed at 150 °C, a suspension of $[(CH_3)_4N][N_2B_{10}H_9]$ (1.5 g, 6.85 mmol), $(PPh_3)_2PdCl_2$ (100 mg, 0.23 mmol), CuI (100 mg, 0.52 mmol) in diphenylphosphine (10 ml), was heated for 15 h under stirring. After cooling down at ambient temperature, an excess of toluene was added to the reaction mixture. The toluene layer was kept for further separation of the neutral boranes $L_2B_{10}H_8$ that it contained. The solid was collected by filtration and washed with toluene (2×25 ml). Green solid (2 g) were recovered and $[(CH_3)_4N][B_{10}H_{10}]^-$ m/z 193.2 (calculated 192), $[(Ph_2PH)B_{10}H_9]^-$ m/z 302.2 (calculated 303) and $[(Ph_3P)B_{10}H_9]^-$ m/z 379.2 (calculated 379) were detected by FAB(-) mass spectrometry. An electrospray analysis showed that $[(Ph_2POH)B_{10}H_9]^-$ m/z 318.4 (calculated 319) was also present. ^{11}B NMR analysis revealed that only $[(Ph_2PH)B_{10}H_9]^-$ and $[(Ph_2POH)B_{10}H_9]^-$ were present in notable concentration. After recrystallization from hot ethanol, 1.82 g of product were recovered (total yield 75%), TLC analysis showed only one spot at R_f 0. The product was passed on DEAE cellulose using 2 M aqueous solution of NH_4NO_3 as mobile phase. Only $[(Ph_2POH)B_{10}H_9]^-$ was eluted and was quantitatively precipitated in a pure form by addition of Bu_4NCl . $[(Ph_2PH)B_{10}H_9]^-$ apparently decomposed as indicated by a change of colour at the top of the column which turned from white to grey. The NMR data reported for $[(Ph_2POH)B_{10}H_9]^-$ in Table I have been obtained from the pure product. Those for $[(Ph_2PH)B_{10}H_9]^-$ have been obtained by analysis of mixtures of $[(Ph_2PH)B_{10}H_9]^-$ and $[(Ph_2POH)B_{10}H_9]^-$.

The toluene layer was concentrated to a volume of 15 ml then chromatographed on silica gel column (2×30 cm) using toluene then toluene-dichloromethane (1 : 1 v/v) as eluent. The first fraction (100 ml) contained diphenylphosphine in excess, the second (200 ml) mainly $1,10-(Ph_2PH)_2B_{10}H_8$ (not pure) ($\approx 20\%$ of neutral $L_2B_{10}H_8$), and the later pure

TABLE II
 $^{31}P\{^1H\}$ NMR data for the products formed by the reaction of $[1-N_2B_{10}H_9]^-$ with Ph_2PH catalyzed by $(PPh_3)_2PdCl_2$ and CuI at 150 °C

Compound ^a	Chemical shift (ppm) (multiplicity)
$NMe_4[Ph_2PHB_{10}H_9]$	+2.84 broad, m
$NMe_4[Ph_2P(OH)B_{10}H_9]$	-4.99 broad, m
$(Ph_2PH)_2B_{10}H_8$	+0.50 broad, m; -5.61 sharp, m, $J(B,P) = 187$ Hz

^a In CD_3CN .

1,6-(Ph₂PH)₂B₁₀H₈ (80% of neutral boranes). The overall yield in neutral boranes did not exceed 5%. For NMR data see Tables I and II.

RESULTS AND DISCUSSION

The reaction of [(CH₃)₄N][N₂B₁₀H₉] with Ph₂PH in absence of catalyst was very slow at 120 °C: the IR absorption due to the diazo function disappeared only after a 72 h reaction time. The reaction mixture contained [1-(Ph₂PH)B₁₀H₉]⁻ (11% yield), [1-Ph₂P(OH)B₁₀H₉]⁻ (17% yield), [B₁₀H₁₀]²⁻ (70%) formed simultaneously with small amounts of boric acid and [B₂₀H₁₈]²⁻ (2%), the presence of which is due to thermal degradation of [1-N₂B₁₀H₉]⁻. When the reaction temperature was increased to 150 °C, [1-N₂B₁₀H₉]⁻ had completely disappeared after 15 h. The yield of phosphine derivatives had not changed, but [B₂₀H₁₈]²⁻ had become the main product instead of [B₁₀H₁₀]²⁻. The addition of (PPh₃)₂PdCl₂ and CuI to the reaction mixture proved to be very effective at 150 °C, [1-(Ph₂PH)B₁₀H₉]⁻ and [1-Ph₂P(OH)B₁₀H₉]⁻ (Fig. 1) were obtained after recrystallization from ethanol in 30 and 45% yields, respectively. The presence of the neutral borane derivatives (Ph₂PH)₂B₁₀H₈ in low concentration (ca 170 mg, 5% yield) was due to the use of (PPh₃)₂PdCl₂ as catalyst. The reaction of (Me₂PPh)₂PdCl₂ with Li₂[B₁₀H₁₀] resulting in 1,10-, 1,6- and 2,7-isomers of (Me₂PPh)₂B₁₀H₈ has been reported¹¹. The ionic species [(Me₂PPh)B₁₀H₉]⁻, which were proba-

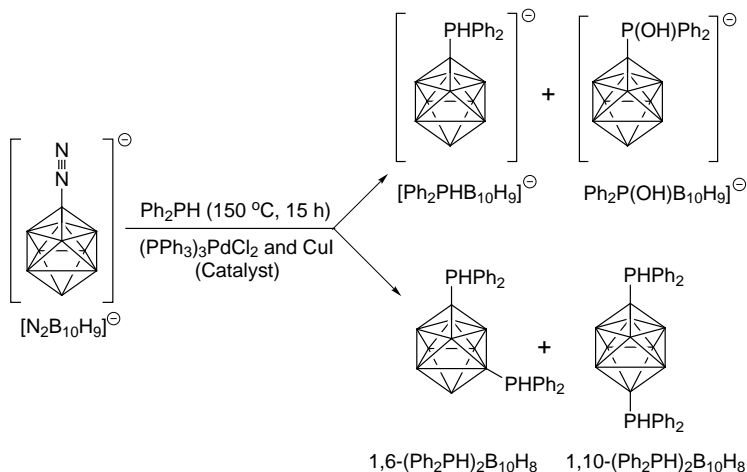


FIG. 1

Products formed by the reaction of $[1-N_2B_{10}H_9]^-$ with Ph_2PH catalyzed by $(PPh_3)_3PdCl_2$ and CuI at 150 °C

bly intermediates in this reaction, were not detected. The reaction was made possible by the reduction of Pd(II) to Pd(0). The same reduction occurred in our case to produce the conversion of $[1-(Ph_2PH)B_{10}H_9]^-$ to $(Ph_2PH)_2B_{10}H_8$, but to a limited extent due to the small amount of catalyst used.

1,6-(Ph₂PH)₂B₁₀H₈ and 1,10-(Ph₂PH)₂B₁₀H₈

Neutral boranes were separated on a silica gel column. 1,10- (20% of neutral boranes) and 1,6-(Ph_2PH)₂B₁₀H₈ (80%) isomers were characterised by FAB(-) and electrospray(-) mass spectrometry (m/z 486.3 and 487.4, respectively, calculated 488), by ¹¹B NMR (Table I) and ³¹P NMR (Table II). Additional analysis by COSY ¹¹B-¹¹B was used to elucidate the structure of the isomer 1,6-(Ph_2PH)₂B₁₀H₈ (Fig. 2).

³¹P NMR spectra (¹H decoupled) distinguish between apical and equatorial boron substituted¹¹. In the 1,6-isomer spectrum (Fig. 3), while both signals are quartets at +0.50 and -5.61 ppm, the apical signals are sharp and easily recognizable; the equatorial signals are broader.

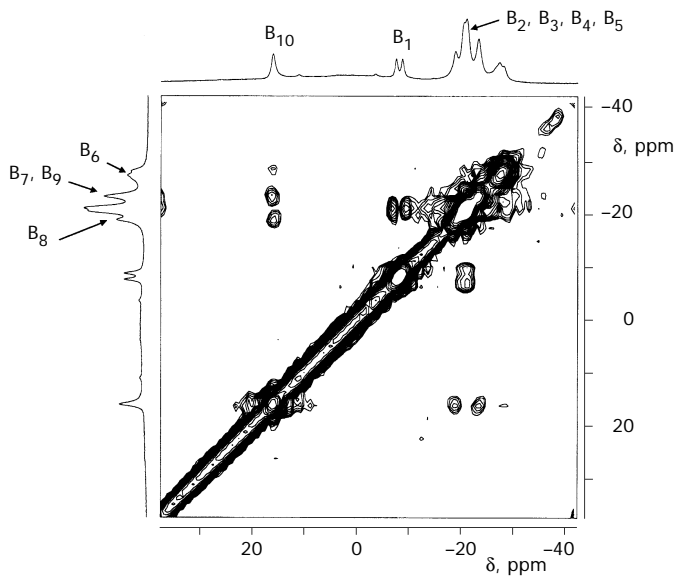


FIG. 2
COSY ¹¹B-¹¹B spectrum of isomer 1,6-(Ph_2PH)₂B₁₀H₈

$[1-(\text{Ph}_2\text{PH})\text{B}_{10}\text{H}_9]^-$ and $[1-\text{Ph}_2\text{P}(\text{OH})\text{B}_{10}\text{H}_9]^-$

The solid product obtained by addition of toluene to the reaction mixture was analysed by FAB(-) mass spectrometry. Three anions were detected: $[\text{B}_{10}\text{H}_{10}]^{2-}$ (m/z 193.2, calculated 192 for $\{(\text{CH}_3)_4\text{N}[\text{B}_{10}\text{H}_{10}]\}^-$), $[(\text{Ph}_2\text{PH})\text{B}_{10}\text{H}_9]^-$ (m/z 302.2, calculated 303) and $[(\text{PPh}_3)\text{B}_{10}\text{H}_9]^-$ (m/z 379.2, calculated 379). An electrospray(-) analysis confirmed the presence of these anions and showed that $[\text{Ph}_2\text{P}(\text{OH})\text{B}_{10}\text{H}_9]^-$ was also present (m/z 318.4, calculated 319). $[(\text{PPh}_3)\text{B}_{10}\text{H}_9]^-$ formed in low concentration from PPh_3 that $(\text{PPh}_3)_2\text{PdCl}_2$ contained and could not be detected by ^{11}B NMR analysis. A mixture of $[(\text{Ph}_2\text{PH})\text{B}_{10}\text{H}_9]^-$ and $[\text{Ph}_2\text{P}(\text{OH})\text{B}_{10}\text{H}_9]^-$ free of $[\text{B}_{10}\text{H}_{10}]^{2-}$ was obtained by recrystallization from hot ethanol. The mixture of anions was passed on a column containing DEAE-cellulose, pure $[\text{Ph}_2\text{P}(\text{OH})\text{B}_{10}\text{H}_9]^-$ was recovered whereas $[(\text{Ph}_2\text{PH})\text{B}_{10}\text{H}_9]^-$ was not eluted and probably decomposed. The NMR data are reported in Tables I and II. The resonances have been unambiguously assigned to the products by analysis of the COSY ^{11}B - ^{11}B s pectrum of the crude product and the ^{11}B and ^{31}P NMR spectra of the various fractions.

In spite of several attempts, we have never succeeded in obtaining $[1-(\text{Ph}_2\text{PH})\text{B}_{10}\text{H}_9]^-$ free of $[1-\text{Ph}_2\text{P}(\text{OH})\text{B}_{10}\text{H}_9]^-$. The formation of this

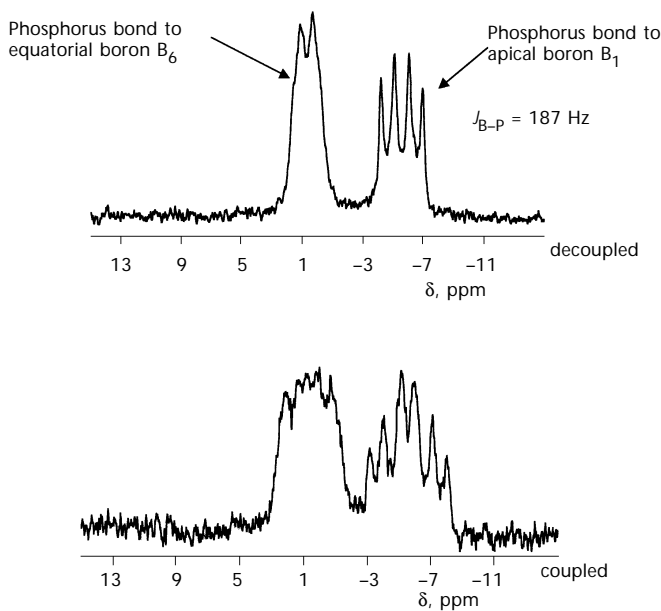


FIG. 3
 ^{31}P and $^{31}\text{P}\{^1\text{H}\}$ spectrum of isomer 1,6- $(\text{Ph}_2\text{PH})_2\text{B}_{10}\text{H}_8$ in CD_3CN

hydroxy derivative could be due to the reaction of $[1-N_2B_{10}H_9]^-$ with $Ph_2P(OH)$ (the tautomeric form¹²⁻¹⁷ of $Ph_2(H)P=O$) or to the degradation of $[1-(Ph_2PH)B_{10}H_9]^-$ in contact with traces of oxygen or humidity during the reaction. However the most probable cause of the formation of the hydroxy derivative was our failure to dry completely $[(CH_3)_4N][N_2B_{10}H_9]$. According to the IR spectrum of this product and TGA measurements, the presence of a very low water amount was suspected. However, we were limited in the possibility to dry it: when the product was heated at 90 °C for 8 h, we observed its partial decomposition to $[B_{20}H_{18}]^{2-}$.

Influence of the Catalyst

The catalyst obviously favoured the formation of $[phosphine-B_{10}H_9]^-$ anions and generated the neutral boranes, but other points deserve to be mentioned.

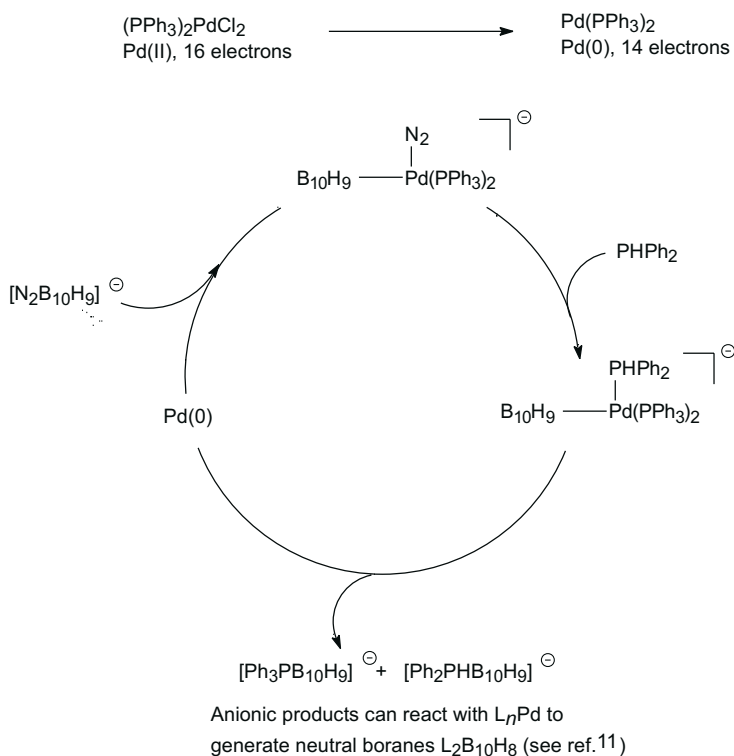


FIG. 4

Possible mechanism of formation of anionic $[phosphine-B_{10}H_9]^-$ and neutral $L_2B_{10}H_8$

A) $[\text{B}_{20}\text{H}_{18}]^{2-}$ did not form at 150 °C by thermal decomposition of $[\text{1-N}_2\text{B}_{10}\text{H}_9]^-$ because of the presence of the palladium catalyst. This indicates that the catalyst interacts with $[\text{1-N}_2\text{B}_{10}\text{H}_9]^-$.

B) $[(\text{PPh}_3)\text{B}_{10}\text{H}_9]^-$ is formed by the exchange of PPh_3 and N_2 groups between the catalyst and the B_{10}H_9 moiety.

Based on recent work on the utilization of *trans*- $(\text{PPh}_3)_2\text{PdCl}_2$ as catalyst in the reaction of $[\text{B}_{12}\text{H}_{11}\text{I}]^{2-}$ with Grignard reagents², it can be assumed that an oxidative addition on Pd leads to $\text{B}_{10}\text{H}_9\text{-Pd-N}_2$ and that $[(\text{Ph}_3\text{P})\text{B}_{10}\text{H}_9]^-$ is generated by a reductive elimination. $[\text{1-(Ph}_2\text{PH)}\text{B}_{10}\text{H}_9]^-$ could be obtained by the same process provided that Ph_2PH exchanges for diazo leaving ligands bound to palladium. Possible mechanism based on such interpretation is shown in Fig 4.

The formation of $1,6\text{-(Ph}_2\text{PH)}_2\text{B}_{10}\text{H}_8$ and $1,10\text{-(Ph}_2\text{PH)}_2\text{B}_{10}\text{H}_8$ from $[\text{1-(Ph}_2\text{PH)}\text{B}_{10}\text{H}_9]^-$ could proceed through a PdLBH four-centered intermediate¹¹ ($\text{L} = \text{Ph}_2\text{PH}$).

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