Synthesis of Mono-*B*-alkylcyclotriborazanes. Structural Characterization of 2-(PhC_2H_4)$B_3N_3H_{11}$

Andrew E. Wille, Patrick J. Carroll, and Larry G. Sneddon*

Department of Chemistry, University of Pennsylvania,
Philadelphia, Pennsylvania 19104-6323

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Introduction

While the chemistry of borazine, $B_3N_3H_6$, has been extensively developed, that of its hydrogenated derivative cyclotriborazane, $B_3N_3H_{12}$, has received much less attention.[1] This is in part because of the saturated tetrahedral bonding configurations of the ring borons and nitrogens in cyclotriborazane which render them relatively inert, thereby making the synthesis of functionalized rings difficult. While a range of nitrogen-substituted trialkylcyclotriborazanes have been produced via reactions such as the polymerization of alkylamine−borane complexes or alkylamine/THF·BH_3 mixtures,[2] the syntheses of unsymmetrical boron-substituted cyclotriborazanes, such as mono- or dialkyl derivatives, have not been reported. In this note we report the first syntheses of mono-*B*-alkylcyclotriborazanes, along with a structural characterization of 2-(PhC_2H_4)-$B_3N_3H_{11}$.

Experimental Section

All manipulations were carried out by using standard high-vacuum or inert-atmosphere techniques as described by Shriver.[3]

Materials. 2-(PhC_2H_4)$B_3N_3H_5$ and 2-(C_2H_5)$B_3N_3H_5$ were prepared as previously reported.[4] Methylene chloride was dried by distillation over P_2O_5 and freshly distilled from molecular sieves before use. Pentane, glyme, and diethyl ether were dried with sodium benzophenone and freshly distilled before use. Sodium borohydride was purchased from Aldrich and used as received. HCl gas was purchased from Matheson and used as received.

Physical Measurements. ^1H NMR spectra at 200.1 MHz and ^{11}B NMR spectra at 64.2 MHz were obtained on a Bruker AF-200 spectrometer, equipped with the appropriate decoupling accessories. ^1H NMR spectra at 500.1 MHz and ^{11}B NMR spectra at 160.5 MHz were obtained on a Bruker AM-500 spectrometer. All ^{11}B chemical shifts are referenced to external $BF_3·O(C_2H_5)_2$ (0.0 ppm) with a negative sign indicating an upfield shift. All ^1H chemical shifts were measured relative to internal residual protons in the lock solvents and are referenced to Me_4Si (0.0 ppm). High- and low-resolution mass spectra were obtained on a VG-ZAB-E high-resolution mass spectrometer. Infrared spectra were recorded on a Perkin-Elmer 1430 infrared spectrophotometer. Melting points were obtained on a standard melting point apparatus and are uncorrected. Elemental analyses were performed at Robertson Microlit Laboratories, Inc., Madison, NJ.

Synthesis of 2-(PhC_2H_4)$B_3N_3H_{11}$. To a 100 mL, two-neck round-bottom flask fitted with a vacuum stopcock were added 0.32 g (1.73 mmol) of 2-(PhC_2H_4)$B_3N_3H_5$ and 25 mL of diethyl ether. The solution was degassed and then cooled to −78 °C, and 6.9 mmol of HCl was condensed into the flask. The solution was stirred for 3 h at −78 °C and then slowly warmed to room temperature. The volatiles were vacuum-evaporated to leave a white solid, which was dissolved in 50 mL of glyme. The solution was frozen at −196 °C, and 0.32 g (8.46 mmol) of $NaBH_4$ was added via a sidearm. The solution was warmed to room temperature and stirred overnight. The volatiles were vacuum-evaporated to leave a white solid, which was then washed with three 10 mL portions of a dilute aqueous solution of NH_4OH, followed by three 10 mL portions of distilled water. The remaining solid was dissolved in CH_2Cl_2, and the solution was filtered. Removal of the volatiles in vacuo and recrystallization of the solid from CH_2Cl_2/pentane at −78 °C gave 0.11 g (0.58 mmol, 33.5% yield) of 2-(PhC_2H_4)$B_3N_3H_{11}$ as a white solid; mp 123−25 °C. ^{11}B NMR (160.5 MHz; CD_2Cl_2), δ (multiplicity, assignment, *J* in Hz): −6.4 (d, B2, J_{BH} 103), −12.2 (t, B4,6, J_{BH} 102). $^1H\{^{11}B\}$ NMR (500.1 MHz; CD_2Cl_2), δ (multiplicity, assignment, *J* in Hz): 7.25 (t, phenyl, J_{HH} 7), 7.21 (d, phenyl, J_{HH} 7), 7.13 (t, phenyl, J_{HH} 7), 2.55 (t, CH_2, J_{HH} 8), 2.33 (br, 3NH), 2.25 (br, 1BH), 2.15 (br, 4BH), 1.99 (br, 3NH), 0.67 (m, CH_2). IR (KBr, cm^{-1}): 3300 (s), 3250 (s), 3090 (w), 3060 (w), 3015 (w), 2920 (w), 2880 (w), 2350 (s), 2320 (s), 1585 (w), 1555 (m), 1490 (w), 1450 (w), 1255 (s), 1230 (s), 1215 (m), 1195 (s), 1165 (m), 1135 (m), 1115 (s), 1070 (m), 1030 (m), 960 (w), 910 (w), 750 (m), 705 (m), 505 (w). Exact mass: calcd for $^{11}B_3{}^{14}N_3{}^{12}C_8{}^1H_{19}$ (M − H), 190.1858; found, 190.1852. Anal. Calcd: C, 50.39; H, 10.57; N, 22.04; B, 17.01. Found: C, 49.51; H, 10.20; N, 19.91; B, 15.90.

Table 1. Data Collection and Structure Refinement Information for 2-(PhC_2H_4)$B_3N_3H_{11}$

empirical formula	$B_3C_8H_{20}N_3$
fw	190.70
crystal class	monoclinic
space group	$P2_1/c$ (No. 14)
Z	4
a, Å	13.282(2)
b, Å	4.537(1)
c, Å	20.229(2)
β, deg	107.51(1)
V, Å^3	1162.5(6)
μ, cm^{-1}	4.47
crystal size, mm	0.75 × 0.30 × 0.12
D_{calc}, g/cm^3	1.090
radiation (λ, Å)	Cu Kα (1.541 84)
θ range, deg	2.0−65.0
scan mode	$\omega-2\theta$
hk*l* collected	+15,−5,±23
no. of reflns measd	2368
no. of unique reflns	1981
no. of reflns used in refinement ($F^2 > 3.0\sigma$)	1424
no. of parameters	208
data/parameter ratio	6.8
R_1	0.048
R_2	0.070

Synthesis of 2-(C_2H_5)$B_3N_3H_{11}$. To a 100 mL, two-neck round-bottom flask fitted with a vacuum stopcock were added 0.26 g (2.40 mmol) of 2-(C_2H_5)$B_3N_3H_5$ and 20 mL of diethyl ether. The solution was then cooled to −78 °C, and 9.7 mmol of gaseous HCl was condensed into the flask. The solution was stirred for 3 h at −78 °C and then slowly warmed to room temperature. The volatiles were vacuum-evaporated, and the resulting white solid was redissolved in 20 mL of glyme. The solution was frozen at −196 °C, and 0.32 g (8.46 mmol) of $NaBH_4$ was added via a sidearm. The solution was warmed to room temperature and stirred overnight. The volatiles were removed in vacuo to leave a white solid, which was then washed with three 10 mL portions of a dilute aqueous solution of NH_4OH, followed by three 10 mL portions of distilled water. The remaining solid was dissolved in CH_2Cl_2, and the solution was filtered. Vacuum evaporation of the volatiles and recrystallization of the resulting solid from CH_2Cl_2/pentane at −78 °C gave 0.09 g (0.78 mmol, 32.5% yield) of 2-(C_2H_5)-$B_3N_3H_{11}$ as a white solid; mp 134 °C dec. ^{11}B NMR (64.2 MHz; C_6D_6), δ (multiplicity, assignment, *J* in Hz): −6.4 (d, B2, J_{BH} 103), −12.2 (t, B4,6, J_{BH} 104). $^1H\{^{11}B\}$ NMR (200.1 MHz; C_6D_6), δ (multiplicity, assignment, *J* in Hz): 2.28 (br, 2BH), 2.19 (br, 3BH), 1.36 (br, 3NH), 0.90 (br, 3NH), 0.78 (t, CH_3, J_{HH} 8), 0.07 (m, CH_2). IR (KBr, cm^{-1}):

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(2) See, for example: (a) Bissot, T. C.; Parry, R. W. *J. Am. Chem. Soc.* **1955**, *77*, 3481−3482. (b) Brown, M. P.; Heseltine, R. W.; Sutcliffe, L. H. *J. Chem. Soc. A* **1968**, 612−616. (c) Narula, C. K.; Janik, J. F.; Duesler, E. N.; Paine, R. T.; Schaeffer, R. *Inorg. Chem.* **1986**, *25*, 3346−3349.

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Table 2. Refined Positional Parameters for 2-$(PhC_2H_4)B_3N_3H_{11}$

atom	x	y	z	B_{eq},[a] Å^2
B2	0.7089(2)	0.4419(5)	0.8765(1)	3.70(4)
B4	0.6058(2)	0.3779(5)	0.9713(1)	3.80(4)
B6	0.4967(2)	0.3857(6)	0.8363(1)	4.06(5)
N1	0.6044(1)	0.3208(4)	0.82190(7)	3.92(3)
N3	0.7074(1)	0.3081(4)	0.94865(7)	3.82(3)
N5	0.5075(1)	0.2646(4)	0.91159(7)	3.95(3)
C7	0.8123(1)	0.3530(5)	0.85583(9)	4.26(4)
C8	0.9154(1)	0.4545(5)	0.9085(1)	4.89(5)
C9	1.0135(1)	0.4183(5)	0.88658(9)	4.26(4)
C10	1.0260(2)	0.5780(6)	0.8315(1)	5.97(6)
C11	1.1170(2)	0.5467(7)	0.8114(1)	6.97(6)
C12	1.1952(2)	0.3586(7)	0.8460(1)	6.65(6)
C13	1.1840(2)	0.2007(7)	0.9004(1)	6.44(6)
C14	1.0938(2)	0.2285(6)	0.9207(1)	5.28(5)
H1a	0.609(2)	0.100(6)	0.815(1)	7.3(6)*
H1e	0.601(1)	0.405(5)	0.7814(9)	5.5(5)*
H2a	0.698(1)	0.697(5)	0.8811(9)	5.7(5)*
H3a	0.723(1)	0.112(5)	0.947(1)	6.5(5)*
H3e	0.774(1)	0.377(5)	0.9836(9)	5.6(5)*
H4a	0.600(1)	0.632(4)	0.9763(8)	4.1(4)*
H4e	0.612(1)	0.258(4)	1.0214(7)	3.5(4)*
H5a	0.502(2)	0.065(7)	0.911(1)	10.2(7)*
H5e	0.445(1)	0.315(4)	0.9216(8)	4.9(4)*
H6a	0.483(1)	0.639(5)	0.8360(9)	5.2(5)*
H6e	0.427(1)	0.265(4)	0.7940(8)	4.3(4)*
H7a	0.805(1)	0.448(5)	0.8137(9)	5.9(5)*
H7b	0.814(1)	0.134(4)	0.8462(9)	5.0(4)*
H8a	0.928(2)	0.360(5)	0.953(1)	7.0(5)*
H8b	0.906(2)	0.675(7)	0.917(1)	10.7(8)*
H10	0.968(2)	0.719(5)	0.802(1)	6.9(5)*
H11	1.125(2)	0.671(6)	0.768(1)	8.1(6)*
H12	1.256(1)	0.312(5)	0.827(1)	7.3(6)*
H13	1.240(1)	0.091(5)	0.924(1)	7.2(6)*
H14	1.083(2)	0.112(6)	0.961(1)	7.4(6)*

[a] Starred values indicate that atoms were refined isotropically. $B_{eq} = (4/3)[\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + \beta_{12}ab \cos \gamma + \beta_{13}ac \cos \beta + \beta_{23}bc \cos \alpha]$.

3320 (m), 3300 (s), 3250 (m), 2940 (w), 2910 (w), 2860 (m), 2810 (w), 2350 (s), 2310 (s), 1590 (w), 1565 (w), 1555 (m), 1280 (m), 1255 (m), 1240 (m), 1220 (s), 1200 (s), 1150 (m), 1120 (s), 1050 (m), 1030 (m), 995 (w), 950 (w), 935 (w), 905 (w), 825 (w), 790 (w). Exact mass: calcd for $^{11}B_3{}^{14}N_3{}^{12}C_2{}^{1}H_{15}$ (M − H), 114.1545; found, 114.1540. Anal. Calcd: C, 20.96; H, 14.07; N, 36.67; B, 28.30. Found: C, 21.16; H, 14.19; N, 36.16; B, 27.53.

Crystallography of 2-$(PhC_2H_4)B_3N_3H_{11}$. Single crystals were grown from CH_2Cl_2/hexane solutions maintained at 0 °C.

Collection and Refinement of the Data. X-ray intensity data were collected on an Enraf-Nonius CAD4 diffractometer employing graphite-monochromated Cu Kα radiation and using the $\omega - 2\theta$ scan technique. Three standard reflections measured every 3500 s of X-ray exposure showed an intensity decay of 0.1% over the course of data collection. A linear decay correction was applied. The intensity data were corrected for Lorentz and polarization effects but not for absorption.

Solution and Refinement of the Structure. X-ray data were processed, and the structure was solved and refined using the Enraf-Nonius MolEn[5] package on a DEC MicroVAX 3100 computer. The structure was solved by direct methods (SIR88).[6] Refinement was by full-matrix least-squares techniques based on F to minimize the quantity $\sum w(|F_o| - |F_c|)^2$ with $w = 1/\sigma^2(F)$. Non-hydrogen atoms were refined anisotropically, and hydrogen atoms were refined isotropically.

Results and Discussion

The parent cyclotriborazane, $B_3N_3H_{12}$, was first synthesized by Dahl and Schaeffer[7] by the reaction of borazine with HCl, followed by $NaBH_4$.[8]

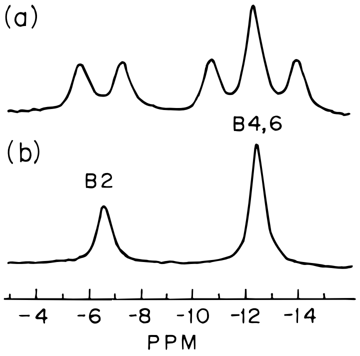

Figure 1. ^{11}B NMR spectra of 2-$(C_2H_5)B_3N_3H_{11}$: (a) proton coupled; (b) proton decoupled.

$$B_3N_3H_6 + 3HCl \rightarrow B_3N_3H_6{\cdot}3HCl \qquad (1)$$

$$2B_3N_3H_6{\cdot}3HCl + 6NaBH_4 \rightarrow 2B_3N_3H_{12} + 6NaCl + 3B_2H_6 \qquad (2)$$

Gaines and Schaeffer[9] subsequently showed that a similar reaction sequence could be used to achieve the synthesis of the nitrogen-substituted trialkylcyclotriborazane 1,3,5-$Me_3B_3N_3H_9$ starting from 1,3,5-$Me_3B_3N_3H_3$.

The use of the Dahl and Schaeffer method for the synthesis of unsymmetrical boron-substituted cyclotriborazanes, such as the mono-*B*-alkylcyclotriborazanes reported herein, had not previously been possible because methods for making the corresponding substituted-borazines were not available. However, with our recent discovery[4] of a high-yield rhodium-catalyzed route for the synthesis of B-alkylated borazines, 2-$R_xB_3N_3H_{6-x}$ ($x = 1-3$), a range of such substituted borazines can now be efficiently prepared, including the monoalkyl borazines shown in eq 3.

$$B_3N_3H_6 + RHC{=}CH_2 \xrightarrow{RhH(CO)(PPh_3)_3} \begin{array}{c}2\text{-}R'B_3N_3H_5\\ R' = CH_2CH_2R\end{array} \qquad (3)$$

We now report that the Dahl and Schaeffer method can be used to convert these substituted borazines to the corresponding mono-*B*-alkylcyclotriborazanes, 2-$RB_3N_3H_{11}$, as shown in eqs 4 and 5.

$$2\text{-}RB_3N_3H_5 + 3HCl \rightarrow 2\text{-}RB_3N_3H_5{\cdot}3HCl \qquad (4)$$

$$2(2\text{-}RB_3N_3H_5{\cdot}3HCl) + 6NaBH_4 \rightarrow 2(2\text{-}RB_3N_3H_5) + 6NaCl + 3B_2H_6 \qquad (5)$$

Typical reactions with 2-$(C_2H_5)B_3N_3H_5$ and 2-(PhC_2H_4)-$B_3N_3H_5$ gave the corresponding mono-*B*-alkylcyclotriborazanes, 2-$(C_2H_5)B_3N_3H_{11}$ and 2-$(PhC_2H_4)B_3N_3H_{11}$, in yields of 30−40% for the recrystallized products. The compounds are isolated as crystalline white solids and, like the parent $B_3N_3H_{12}$, are both air and water stable; however, unlike the parent, they are very soluble in organic solvents such as benzene.

The compositions and purities of the products were established by elemental analyses and exact mass determinations, and their NMR data support their mono-*B*-alkyl formulations.

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Table 3. Selected Bond Distances (Å) and Angles (deg) for 2-(PhC_2H_4)$B_3N_3H_{11}$

B2−N1	1.589(2)	N1−H1a	1.02(3)	B2−N3	1.587(3)
N1−H1e	0.89(2)	B2−C7	1.603(3)	N3−H3a	0.92(2)
B2−H2a	1.17(2)	N3−H3e	1.00(2)	B4−N3	1.583(3)
N5−H5a	0.91(3)	B4−N5	1.574(2)	N5−H5e	0.94(2)
B4−H4a	1.16(2)	B4−H4e	1.13(2)	B6−N1	1.571(3)
B6−N5	1.584(3)	B6−H6a	1.16(2)	B6−H6e	1.19(2)
N1−B2−N3	104.9(1)	B6−N1−H1e	107(1)	N1−B2−C7	111.7(2)
H1a−N1−H1e	106(2)	N1−B2−H2a	107.2(8)	B2−N3−B4	116.0(1)
N3−B2−C7	113.1(1)	B2−N3−H3a	106(1)	N3−B2−H2a	105.6(9)
B2−N3−H3e	106(1)	C7−B2−H2a	114(1)	B4−N3−H3a	115(1)
N3−B4−N5	107.1(1)	B4−N3−H3e	113(1)	N3−B4−H4a	107.8(9)
H3a−N3−H3e	100(2)	N3−B4−H4e	109.0(8)	B4−N5−B6	116.0(2)
N5−B4−H4a	109.4(7)	B4−N5−H5a	113(1)	N5−B4−H4e	110.4(7)
B4−N5−H5e	110(1)	H4a−B4−H4e	113(1)	B6−N5−H5a	110(2)
N1−B6−N5	106.8(1)	B6−N5−H5e	107(1)	N1−B6−H6a	109(1)
H5a−N5−H5e	100(2)	N1−B6−H6e	109.7(9)	B2−C7−C8	113.6(2)
N5−B6−H6a	108.5(9)	B2−C7−H7a	105(1)	N5−B6−H6e	110.8(9)
B2−C7−H7b	112(1)	H6a−B6−H6e	112(12)	B2−N1−B6	117.4(1)
B2−N1−H1a	111(1)	B2−N1−H1e	106(1)	B6−N1−H1a	108(1)

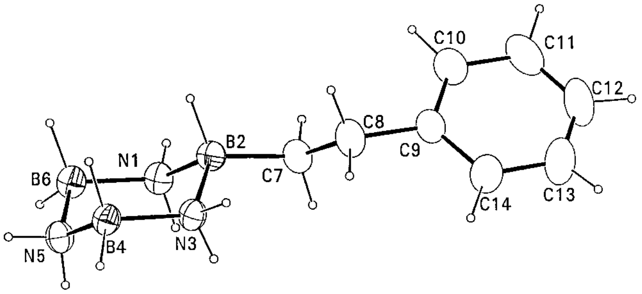

Figure 2. ORTEP drawing of the molecular structure of 2-(PhC_2H_4)-$B_3N_3H_{11}$.

Thus, as shown in Figure 1, the ^{11}B NMR spectrum of 2-(C_2H_5)-$B_3N_3H_{11}$ consists of one triplet and one doublet in a 2:1 ratio, in agreement with two equivalent BH_2 groups and one BH group in the molecule. Likewise, the observed chemical shifts and coupling constants fall in the expected regions for this ring system.[2c,8,9]

The structure of 2-(PhC_2H_4)$B_3N_3H_{11}$ was confirmed by a single-crystal X-ray study. As shown in the ORTEP drawing, Figure 2, the B_3N_3 ring adopts a chair conformation with the alkyl group attached to B2 in an equatorial position. The X-ray-determined structures of the parent $B_3N_3H_{12}$,[10] the hexa-*N*-methylcyclotriborazane 1,1,3,3,5,5-$Me_6B_3N_3H_6$,[11] and the tri-*N*-methylcyclotriborazanes 1e,3e,5e-$Me_3B_3N_3H_9$[2c] and 1e,3e,5a-$Me_3B_3N_3H_9$[2c] have also shown chair conformations for the B_3N_3 ring. All of the B−N bond lengths are essentially identical in 2-(PhC_2H_4)$B_3N_3H_{11}$, varying only from 1.571(3) to 1.589(2) Å (1.581 Å average). The B−N−B ring angles (116.5° average) are greater than the N−B−N angles (106.3° average). These values are comparable to those found in the parent cyclotriborazane (averages B−N 1.576 Å, B−N−B 115.9°, N−B−N 107.3°)[10] and the two isomeric compounds 1e,3e,5e- and 1e,-3e,5a-$Me_3B_3N_3H_9$ (averages for 1e,3e,5e B−N 1.565 Å, B−N−B 116.5°, N−B−N 108.9°; averages for 1e,3e,5a B−N 1.578 Å, B−N−B 114.7°, N−B−N 108.5°).[2c]

Crabtree recently proposed[12] that the high melting points observed for boron-nitrogen compounds relative to their hydrocarbon analogs[13] (e.g. H_3BNH_3, 104 °C; relative to C_2H_6, −181 °C) may result from intermolecular dihydrogen bonds between BH and NH hydrogens in these molecules. In 2-(PhC_2H_4)$B_3N_3H_{11}$, the closest normalized[14] intermolecular interactions are, indeed, found between B−H and N−H hydrogens with, as shown in Table 4, the H---H distances and bond angles in at least three cases in the ranges identified by Crabtree for dihydrogen bonds (bond distances 1.7−2.2 Å; bond angles B−H−H(N) 90−171° and (B)H−H−N 117−171°). Thus, the combined effect of the intermolecular BH−HN interactions in both 2-(PhC_2H_4)$B_3N_3H_{11}$ (mp 123−125 °C) and 2-(C_2H_5)$B_3N_3H_{11}$ (mp 134 °C dec) may also account for their higher melting points relative to those of their hydrocarbon counterparts (PhC_2H_4)C_6H_{11} (<25 °C)[15] and (C_2H_5)C_6H_{11} (−111.3 °C).[16]

Table 4. Normalized[14] Intermolecular Bond Distances and Angles for 2-(PhC_2H_4)$B_3N_3H_{11}$

intermolecular bond	(B)H----H(N), Å	(B)H--H--N, deg	(N)H--H--B, deg
B6(H6e)--(H1e)N1	2.07	152	140
B2(H2a)--(H3a)N3	2.17	146	143
B4(H4a)--(H5e)N5	2.18	134	104
B2(H2a)--(H1a)N1	2.30	141	137

The reaction conditions and/or reactants in the catalyzed reaction in eq 3 can be varied to selectively produce, in addition to mono-*B*-alkylborazines, di- and tri-*B*-alkylborazines, as well as mixed alkylborazines. This suggests that a variety of new substituted cyclotriborazanes should now be readily accessible using the Dahl and Schaeffer method and thus enable more extensive investigations of the chemistry and applications of this class of ring compounds.

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Supporting Information Available: Tables listing refined thermal parameters, bond distances, and bond angles (3 pages). Ordering information is given on any current masthead page.

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