

Synthesis and Structural Characterization of Carbons-Adjacent Stannacarboranes of the C₂B₁₀ System

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Summary: The chemistry of p-block metallacarboranes of the C₂B₁₀ systems is largely unexplored in comparison with that of s-, d-, and f-block metallacarboranes. This article reports several carbons-adjacent stannacarboranes of the C₂B₁₀ system and their chemical properties for the first time. Reaction of SnCl₂ with [μ -1,2-[o-C₆H₄(CH₂)₂]-1,2-C₂B₁₀H₁₀]₂Na₄(THF)₆]_n gave the Lewis base free stannacarborane [μ -1,2-[o-C₆H₄(CH₂)₂]-1,2-C₂B₁₀H₁₀]₂Sn (**1**). Recrystallization of **1** from MeCN, THF, and DME afforded the corresponding Lewis base coordinated stannacarboranes [μ -1,2-[o-C₆H₄(CH₂)₂]-1,2-C₂B₁₀H₁₀]-Sn(MeCN) (**2**), [μ -1,2-[o-C₆H₄(CH₂)₂]-1,2-C₂B₁₀H₁₀]-Sn(THF)·THF (**3**·THF), and [μ -1,2-[o-C₆H₄(CH₂)₂]-1,2-C₂B₁₀H₁₀]-Sn(DME) (**4**), respectively. They were fully characterized by various spectroscopic data and elemental analyses. Complexes **2**–**4** were further confirmed by single-crystal X-ray analyses.

Introduction

The incorporation of p-block elements in the carborane cages has been documented.¹ A number of stannacarboranes with or without Lewis base coordination have been prepared and structurally characterized, in which the carboranyl ligands are either the C₂B₄^{1–11} or C₂B₉^{1,2,12–15} systems. In sharp contrast, the chemistry of p-block metallacarboranes of the large C₂B₁₀ systems has been virtually unexplored, although the 13-vertex metallacarboranes of s-, d- and f-block elements are well-known.^{1,16} The only examples of the supericos-

ahedral p-block metallacarboranes are the very recently reported carbons-apart stannacarboranes of the C₂B₁₀ system.¹⁷

We have recently developed a methodology to prepare carbons-adjacent carborane anions of the C₂B₁₀ system by linking the cage carbon atoms together via a short bridge.^{18–20} The alkali-metal salts of the dianion [μ -1,2-[o-C₆H₄(CH₂)₂]-1,2-C₂B₁₀H₁₀]₂M₄(THF)₆]_n are useful synthons for the production of carbons-adjacent lanthanacarboranes.²¹ We have extended our research to include p-block elements and report herein the synthesis, structural characterization, and Lewis acid properties of carbons-adjacent stannacarboranes of the C₂B₁₀ system.

Experimental Section

General Procedures. All experiments were performed under an atmosphere of dry dinitrogen with the rigid exclusion of air and moisture using standard Schlenk or cannula techniques, or in a glovebox. THF and *n*-hexane were freshly distilled from sodium benzophenone ketyl immediately prior to use. CH₃CN and CH₂Cl₂ were freshly distilled from CaH₂ and P₂O₅, respectively, immediately prior to use. [μ -1,2-[o-C₆H₄(CH₂)₂]-1,2-C₂B₁₀H₁₀]₂Na₄(THF)₆]_n was prepared according to the literature method.¹⁹ All other chemicals were purchased from either Aldrich or Acros Chemical Co. and used as received unless otherwise noted. Infrared spectra were obtained from KBr pellets prepared in the glovebox on a Perkin-Elmer 1600 Fourier transform spectrometer. ¹H and ¹³C NMR spectra were recorded on a Bruker DPX 300 spectrometer at 300.13 and 75.47 MHz, respectively. ¹¹B and ¹¹⁹Sn NMR spectra were recorded on a Varian Inova 400 spectrometer at 128.32 and 149.11 MHz, respectively. All chemical shifts are reported in δ units with references to the residual protons of the deuterated solvents for proton and carbon chemical shifts, to external BF₃·OEt₂ (0.0 ppm) for boron chemical shifts, and to external Me₄Sn (0.0 ppm) for tin

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Table 1. Crystal Data and Summary of Data Collection and Refinement Details for 2–4

	2	3·THF	4
formula	C ₁₂ H ₂₁ B ₁₀ NSn	C ₁₈ H ₃₄ B ₁₀ O ₂ Sn	C ₁₄ H ₂₈ B ₁₀ O ₂ Sn
cryst size, mm	0.40 × 0.35 × 0.20	0.35 × 0.25 × 0.20	0.63 × 0.18 × 0.13
fw	406.1	509.2	455.2
cryst syst	monoclinic	monoclinic	monoclinic
space group	<i>Pn</i>	<i>P2₁/n</i>	<i>P2₁/n</i>
<i>a</i> , Å	7.161(1)	10.298(2)	10.807(1)
<i>b</i> , Å	9.514(2)	14.631(3)	16.965(1)
<i>c</i> , Å	13.892(3)	16.924(3)	12.397(1)
β, deg	97.32(3)	105.80(3)	105.54(1)
<i>V</i> , Å ³	938.7(3)	2453.6(9)	2189.9(2)
<i>Z</i>	2	4	4
<i>D</i> _{calcd} , Mg/m ³	1.437	1.379	1.381
radiation (λ, Å)		Mo Kα (0.710 73)	
θ range, deg	2.14–25.65	1.87–25.67	2.09–28.03
μ, mm ⁻¹	1.352	1.055	1.173
<i>F</i> (000)	400	1032	912
no. of obsd rflns	1786	3822	5276
no. of params refined	219	282	244
goodness of fit	1.021	1.105	0.909
R1	0.034	0.047	0.048
wR2	0.087	0.141	0.107

chemical shifts. Elemental analyses were performed by MEDAC Ltd., Brunel University, Middlesex, U.K.

Preparation of {μ-1,2-[o-C₆H₄(CH₂)₂]-1,2-C₂B₁₀H₁₀}Sn (1). To a clear colorless THF (20 mL) solution of anhydrous SnCl₂ (189 mg, 1.00 mmol) was slowly added a THF (25 mL) solution of [{μ-1,2-[o-C₆H₄(CH₂)₂]-1,2-C₂B₁₀H₁₀}₂Na₄(THF)₆]_{*n*} (508 mg, 0.50 mmol) at -78 °C with stirring. The reaction mixture was slowly warmed to room temperature and then stirred overnight. After removal of the dark precipitate, the clear pale yellow solution was concentrated to give a yellow solid, which was extracted with CH₂Cl₂ (8 mL × 3). The CH₂-Cl₂ solutions were combined and concentrated to about 10 mL. **1** was isolated as pale yellow crystals, after the solution stood at 20 °C for 1 week (171 mg, 42%). ¹H NMR (CD₂Cl₂): δ 7.29 (m, 4H, aryl H), 4.23 (d, 2H, *J* = 15.9 Hz, C₆H₄(CH₂)₂), 3.46 (d, 2H, *J* = 15.9 Hz, C₆H₄(CH₂)₂). ¹³C NMR (CD₂Cl₂): δ 135.5, 128.8, 126.8 (aryl C), 50.8 (C₆H₄(CH₂)₂); cage carbons were not observed. ¹¹B NMR (CD₂Cl₂): δ 9.25 (2), 7.28 (4), 3.75 (1), -0.63 (3). ¹¹⁹Sn NMR (CD₂Cl₂): δ -363.8. IR (KBr, cm⁻¹): ν 3006 (w), 2952 (w), 2529 (s), 2395 (m), 1596 (m), 1448 (w), 1260 (m), 1011 (m). Anal. Calcd for C₁₀B₁₀H₁₈Sn: C, 32.90; H, 4.97. Found: C, 33.28; H, 5.29.

Preparation of {μ-1,2-[o-C₆H₄(CH₂)₂]-1,2-C₂B₁₀H₁₀}Sn (MeCN) (2). Recrystallization of **1** (203 mg, 0.50 mmol) from MeCN (10 mL) at 20 °C afforded **2** as pale yellow crystals (181 mg, 89%). ¹H NMR (CD₂Cl₂): δ 7.29 (m, 4H, aryl H), 4.23 (d, 2H, *J* = 15.9 Hz, C₆H₄(CH₂)₂), 3.45 (d, 2H, *J* = 15.9 Hz, C₆H₄(CH₂)₂), 1.60 (s, 3H, CH₃CN). ¹³C NMR (CD₂Cl₂): δ 135.5, 128.8, 126.9 (aryl C), 120.5 (CH₃CN), 50.8 (C₆H₄(CH₂)₂), 1.3 (CH₃CN); cage carbons were not observed. ¹¹B NMR (CD₂Cl₂): δ 8.85 (2), 6.77 (4), 3.22 (1), -0.56 (1), -1.69 (2). ¹¹⁹Sn NMR (CD₂Cl₂): δ -364.2. IR (KBr, cm⁻¹): ν 3010 (w), 2959 (m), 2529 (s), 2395 (s), 2307 (m), 1608 (m), 1260 (s), 1098 (s), 1024 (s), 803 (s). Anal. Calcd for C₁₂H₂₁B₁₀NSn: C, 35.49; H, 5.21; N, 3.45. Found: C, 35.23; H, 5.01; N, 3.65.

Preparation of {μ-1,2-[o-C₆H₄(CH₂)₂]-1,2-C₂B₁₀H₁₀}Sn (THF)·THF (3·THF). Recrystallization of **1** (203 mg, 0.50 mmol) from THF (10 mL) at 20 °C afforded **3·THF** as pale yellow crystals (219 mg, 86%). ¹H NMR (CD₂Cl₂): δ 7.29 (m, 4H, aryl H), 4.21 (d, 2H, *J* = 15.9 Hz, C₆H₄(CH₂)₂), 3.69 (m, 8H, THF), 3.47 (d, 2H, *J* = 15.9 Hz, C₆H₄(CH₂)₂), 1.83 (m, 8H, THF). ¹³C NMR (CD₂Cl₂): δ 135.5, 128.7, 126.8 (aryl C), 68.4 (THF), 50.9 (C₆H₄(CH₂)₂), 26.1 (THF); cage carbons were not observed. ¹¹B NMR (CD₂Cl₂): δ 8.93 (2), 7.03 (2), 6.55 (2), 3.38 (1), -1.14 (3). ¹¹⁹Sn NMR (CD₂Cl₂): δ -364.4. IR (KBr, cm⁻¹): ν 3020 (w), 2959 (m), 2529 (s), 2395 (m), 1622 (m), 1253 (s), 1098 (m), 1025 (s), 803 (s). Anal. Calcd for C₁₆B₁₀H₃₀O_{1.5}Sn (3 + 0.5 THF): C, 40.61; H, 6.39. Found: C, 40.57; H, 6.03.

Preparation of {μ-1,2-[o-C₆H₄(CH₂)₂]-1,2-C₂B₁₀H₁₀}Sn (DME) (4). Recrystallization of **1** (203 mg, 0.50 mmol) from DME (10 mL) afforded **4** as pale yellow crystals (209 mg, 92%). ¹H NMR (CD₂Cl₂): δ 7.26 (m, 4H, aryl H), 4.17 (d, 2H, *J* = 15.9 Hz, C₆H₄(CH₂)₂), 3.51 (s, 4H, DME), 3.49 (d, 2H, *J* = 15.9 Hz, C₆H₄(CH₂)₂), 3.35 (s, 6H, DME). ¹³C NMR (CD₂Cl₂): δ 128.5, 126.8 (aryl C), 72.1 (DME), 59.1 (DME), 50.9 (C₆H₄(CH₂)₂); cage carbons were not observed. ¹¹B NMR (CD₂Cl₂): δ 8.67 (2), 6.83 (2), 6.20 (2), 2.59 (1), -2.01 (3). ¹¹⁹Sn NMR (CD₂Cl₂): δ -380.5. IR (KBr, cm⁻¹): ν 3010 (w), 2958 (m), 2542 (s), 2395 (s), 1608 (s), 1447 (m), 1260 (m), 1018 (s), 803 (s). Anal. Calcd for C₁₄H₂₈B₁₀O₂Sn: C, 36.94; H, 6.20. Found: C, 36.72; H, 6.02.

X-ray Structure Determination. All single crystals were immersed in Paratone-N oil and sealed under N₂ in thin-walled glass capillaries. Data were collected at 293 K on a Bruker SMART 1000 CCD diffractometer using Mo Kα radiation. An empirical absorption correction was applied using the SADABS program.²² All structures were solved by direct methods and subsequent Fourier difference techniques and refined anisotropically for all non-hydrogen atoms by full-matrix least squares calculations on *F*² using the SHELXTL program package.²³ For the noncentrosymmetric structure **2**, the Flack parameter *x* = 0.45(8) after refinement.²⁴ Most of the carborane hydrogen atoms were located from difference Fourier syntheses. All other hydrogen atoms were geometrically fixed using the riding model. Crystal data and details of data collection and structure refinements are given in Table 1. Further details are included in the Supporting Information.

Results and Discussion

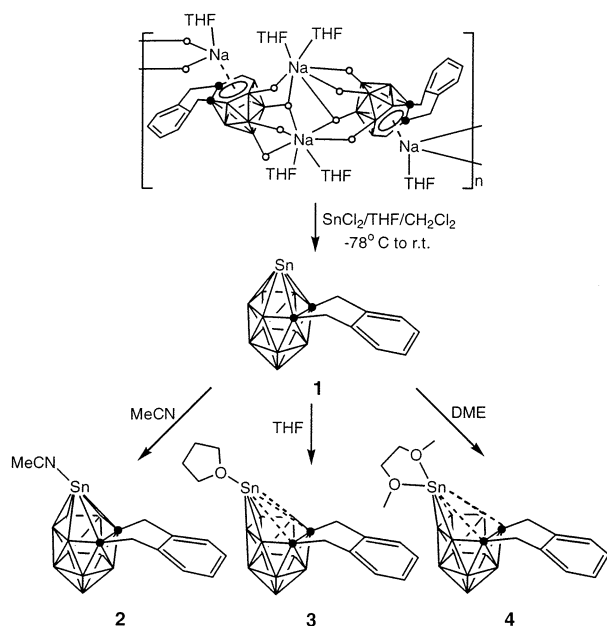
Synthesis. Salt metathesis is a general method for the production of metallacarboranes. Treatment of SnCl₂ with 0.5 equiv of [{μ-1,2-[o-C₆H₄(CH₂)₂]-1,2-C₂B₁₀H₁₀}₂Na₄(THF)₆]_{*n*} in THF in the temperature range -78 to 0 °C gave, after recrystallization from CH₂-Cl₂, an unsolvated carbons-adjacent stannacarborane of the C₂B₁₀ system, {μ-1,2-[o-C₆H₄(CH₂)₂]-1,2-C₂B₁₀H₁₀}Sn, in 42% isolated yield. Temperature control is very important to this reaction. Otherwise, a redox reaction

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Scheme 1



occurred leading to the formation of the neutral cage carbons-linked *o*-carborane μ -1,2-[*o*- $\text{C}_6\text{H}_4(\text{CH}_2)_2$]-1,2- $\text{C}_2\text{B}_{10}\text{H}_{10}$ and tin metal, since $\{\mu$ -1,2-[*o*- $\text{C}_6\text{H}_4(\text{CH}_2)_2$]-1,2- $\text{C}_2\text{B}_{10}\text{H}_{10}\}^{2-}$ is known to be a strong reducing agent.²¹ This process can be closely monitored by ^{11}B NMR spectroscopy.

Complex **1** is a Lewis acid which can accept lone pairs from various donor solvents to form Lewis acid–base adducts. Recrystallization of **1** from MeCN, THF, and DME afforded the corresponding Lewis base coordinated stannacarboranes $\{\mu$ -1,2-[*o*- $\text{C}_6\text{H}_4(\text{CH}_2)_2$]-1,2- $\text{C}_2\text{B}_{10}\text{H}_{10}\}$ -Sn(MeCN) (**2**), $\{\mu$ -1,2-[*o*- $\text{C}_6\text{H}_4(\text{CH}_2)_2$]-1,2- $\text{C}_2\text{B}_{10}\text{H}_{10}\}$ -Sn(THF) (**3**), and $\{\mu$ -1,2-[*o*- $\text{C}_6\text{H}_4(\text{CH}_2)_2$]-1,2- $\text{C}_2\text{B}_{10}\text{H}_{10}\}$ -Sn(DME) (**4**), respectively. Scheme 1 outlines the transformations mentioned above.

Complexes **1–4** are sensitive to moisture and air. The ^{11}B NMR data indicate that they slowly decompose into the neutral carborane μ -1,2-[*o*- $\text{C}_6\text{H}_4(\text{CH}_2)_2$]-1,2- $\text{C}_2\text{B}_{10}\text{H}_{10}$ and tin metal upon heating in solution.

The composition and the carborane-to-solvent ratio of complexes **1–4** are supported by the ^1H and ^{13}C NMR spectroscopic data. Their ^{11}B NMR spectra are very similar and exhibit a 2:2:2:1:3 splitting pattern. The solid-state IR spectra all show a strong broad absorption at $\sim 2530\text{ cm}^{-1}$. The ^{119}Sn chemical shifts in CD_2Cl_2 are -363.8 , -364.2 , -364.4 , and -380.5 ppm for **1–4**, respectively. This trend is consistent with the increasing donor ability of the solvents going from CH_2Cl_2 through MeCN and THF to DME, although the differences are not very significant. These measured data can be compared to the value of -431 ppm reported for $(\eta^6\text{-C}_2\text{B}_{10}\text{H}_{12})\text{Sn}$.¹⁷

Crystal Structures. The solid-state structures of **2–4** have been confirmed by single-crystal X-ray analyses and are shown in Figures 1–3, respectively. They all adopt monomeric structures, and **3** shows one THF of solvation. The selected bond distances are compiled in Table 2.

The geometries of the carboranyl ligands in **2–4** are very similar. The bond distances and angles are very close to those in $\{\mu$ -1,2-[*o*- $\text{C}_6\text{H}_4(\text{CH}_2)_2$]-1,2- $\text{C}_2\text{B}_{10}\text{H}_{10}\}^{2-}$

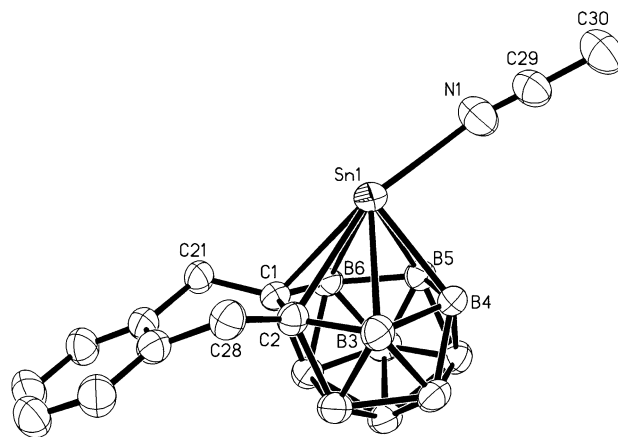


Figure 1. Molecular structure of $\{\mu$ -1,2-[*o*- $\text{C}_6\text{H}_4(\text{CH}_2)_2$]-1,2- $\text{C}_2\text{B}_{10}\text{H}_{10}\}$ Sn(MeCN) (**2**).

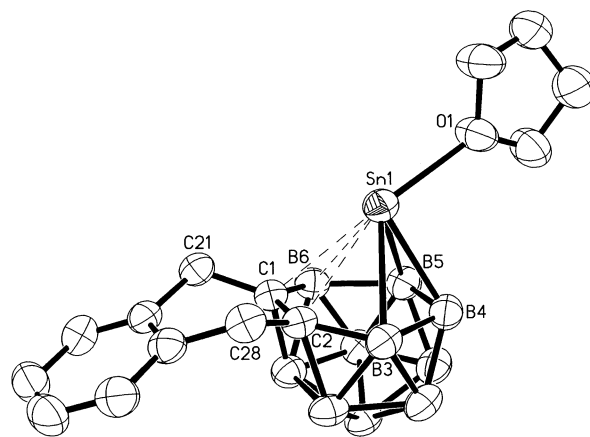


Figure 2. Molecular structure of $\{\mu$ -1,2-[*o*- $\text{C}_6\text{H}_4(\text{CH}_2)_2$]-1,2- $\text{C}_2\text{B}_{10}\text{H}_{10}\}$ Sn(THF) (**3**).

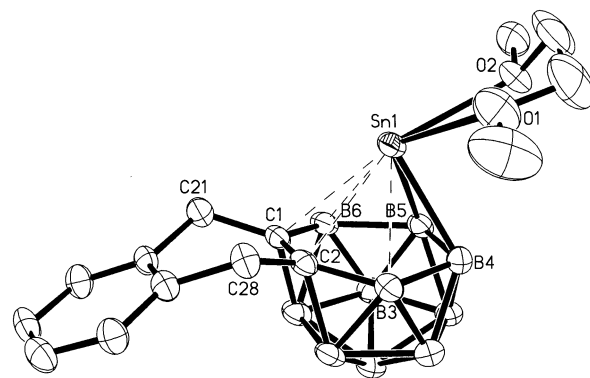


Figure 3. Molecular structure of $\{\mu$ -1,2-[*o*- $\text{C}_6\text{H}_4(\text{CH}_2)_2$]-1,2- $\text{C}_2\text{B}_{10}\text{H}_{10}\}$ Sn(DME) (**4**).

$\text{Na}_4(\text{THF})_6\}_n$.¹⁹ These structures show an increased slip distortion of tin toward the boron side of the C_2B_4 bonding face on complexation with the stronger base,⁷ which is consistent with the ^{119}Sn NMR data. It is reasonable to suggest that the unsolvated stannacarborane **1** may adopt a more symmetric structure, like the carbons-apart $(\eta^6\text{-Me}_2\text{C}_2\text{B}_{10}\text{H}_{10})\text{Sn}$.¹⁷

The Sn–cage atom distances (see Table 2) fall in the range $2.4\text{--}2.8\text{ \AA}$, which is normally observed in stannacarboranes.^{1b} To make a comparison between carbons-apart and carbons-adjacent stannacarboranes of the C_2B_{10} systems, the value of the longest Sn–cage atom distance of 2.68 \AA in $(\eta^6\text{-Me}_2\text{C}_2\text{B}_{10}\text{H}_{10})\text{Sn}$ is taken as a

Table 2. Selected Bond Lengths^a

	2	3 ·THF	4
Sn(1)–C(1)	2.668(6)	2.745(5)	2.745(5)
Sn(1)–C(2)	2.680(6)	2.705(5)	2.692(5)
Sn(1)–B(3)	2.676(7)	2.658(5)	2.697(5)
Sn(1)–B(4)	2.431(7)	2.407(5)	2.455(5)
Sn(1)–B(5)	2.450(8)	2.506(5)	2.456(5)
Sn(1)–B(6)	2.680(9)	2.795(5)	2.756(5)
Sn(1)–X(N,O)	2.623(8)	2.439(3)	2.716(5) 2.660(5)

^a All distances are in Å.

cutoff point.¹⁷ Dashed lines are drawn in Figures 1–3 when the Sn–cage atom distances are longer than 2.68 Å. One might suggest that the apical tin atom could be considered as η^6 bonded to the C₂B₄ face of the carborane fragment in **2**, whereas the tin is η^3 and η^2 bonded to the carborane in **3** and **4**, respectively; these notions are supported by the ¹¹⁹Sn NMR data. Similar results have also been observed in the C₂B₄ and C₂B₉ systems.¹ These results show that the Lewis acid properties of the tin atom in stannacarboranes of the C₂B₄, C₂B₉, and C₂B₁₀ systems are very similar.

Conclusion

Several carbons-adjacent stannacarboranes of the C₂B₁₀ system have been prepared and structurally characterized. They represent the first examples of carbons-adjacent p-block metallacarboranes of the C₂B₁₀ system. The interactions between the tin atom and carborane are very diverse and dependent upon the basicity of the coordinated bases. A stronger base leads to an increased slip distortion of the tin from the center of the C₂B₄ bonding face of the carboranyl ligand.

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Supporting Information Available: Tables of crystallographic data and data collection details, atomic coordinates, bond distances and angles, anisotropic thermal parameters, and hydrogen atom coordinates and figures giving atom-numbering schemes for complexes **2–4**; data are also available as CIF files. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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