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# Organometallic gold derivatives with the $[1-Si^{t}BuMe_{2}-1,2-C_{2}B_{10}H_{10}]$ -carboranyl ligand. Crystal structure of $[1-\{Au(PPh_{3})\}-2-Si^{t}BuMe_{2}-1,2-C_{2}B_{10}H_{10}]$

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**Abstract**—Reaction of 1-Li-2-Si'BuMe<sub>2</sub>-1,2-C<sub>2</sub>B<sub>10</sub>H<sub>10</sub> with [AuCIL] (L=PPh<sub>3</sub>, PPh<sub>2</sub>Me, AsPh<sub>3</sub>) in molar ratio 1:1 affords the organometallic gold complexes [1-AuL-2-Si'BuMe<sub>2</sub>-1,2-C<sub>2</sub>B<sub>10</sub>H<sub>10</sub>]. The complex PPN[Au{2-Si'BuMe<sub>2</sub>-1,2-C<sub>2</sub>B<sub>10</sub>H<sub>10</sub>]<sub>2</sub>] (PPN = N(PPh<sub>3</sub>)<sub>2</sub>) has also been synthesised by reaction of PPN[AuCl<sub>2</sub>] with 1-Li-2-Si'BuMe<sub>2</sub>-1,2-C<sub>2</sub>B<sub>10</sub>H<sub>10</sub> in molar ratio 1:2. The crystal structure of [1-{Au(PPh<sub>3</sub>)}-2-Si'BuMe<sub>2</sub>-1,2-C<sub>2</sub>B<sub>10</sub>H<sub>10</sub>] has been established by X-ray diffraction. © 1998 Elsevier Science Ltd. All rights reserved

Keywords: gold; carborane

### INTRODUCTION

The synthesis of carboranyl derivatives has been extensively studied because in some aspects they resemble organic compounds with electron-accepting groups [1–4]. During the past decade investigations in the field of C-substituted carboranes have involved the creation of new models to be used in theoretical investigations [5], the preparation of organic and organometallic carboranyl compounds for the production of polymeric materials [6–8] (and references cited therein) and biological and medical investigations [9]. These compounds have proved particularly useful in the boron neutron capture therapy (BNCT) for cancer [10–13].

It is known that *o*-carborane derivatives form unusually strong Au–C bonds. Nevertheless, examples of closo-carboranes  $\sigma$ -bonded to gold by 2c–2e  $\sigma$ bonds are relatively rare (as opposed to the most studied nido-carboranes interacting gold via cluster bonding) [14–18].

The first report was made by Mitchel and Stone

The treatment of  $1-Si'BuMe_2-1,2-C_2B_{10}H_{11}$  with "Bu leads to the lithiated derivative 1-Li-2-Si'

carborane) have been synthesised.

Li<sup>*n*</sup>Bu leads to the lithiated derivative 1-Li-2-Si<sup>*t*</sup> BuMe<sub>2</sub>-1,2-C<sub>2</sub>B<sub>10</sub>H<sub>10</sub> (1) which is not isolated; further reaction of 1 with the organochloro complexes [AuClL] affords the organometallic derivatives [1-

[19, 20], who prepared the complexes [Au(2-R-1,2-C<sub>2</sub> $B_{10}H_{10})$ (PPh<sub>3</sub>)] [R=H, Me, Ph]. More recently

species of stoichiometry  $[Au(2-R-1,2-C_2B_{10}H_{10})L]$ 

 $R = CH_2OCH_3$ , Ph;  $L = PPh_3$ , P(o-tol)<sub>3</sub>, PCy<sub>3</sub>, PEt<sub>3</sub>,

AsPh<sub>3</sub>] have been reported by Reid and Welch [21]

whereas in the work published by Hawthorne et al.

[22] dimetallated species of o-carborane and bis(o-

We have reported recently on the synthesis of the

first *m*-carborane derivatives bonded to gold by 2c-2e

 $\sigma$ -bond [23] as part of our studies with carborane

derivatives [24-30]. Our contribution this time is

related to some new organometallic gold(I) derivatives

with the carboranyl group [1-Si'BuMe<sub>2</sub>-1,2-

 $C_2B_{10}H_{10}$ ]<sup>-</sup>. This carboranyl derivative has been

described as a good precursor in the synthesis of

**RESULTS AND DISCUSSION** 

monosubstituted carborane derivatives [31].

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 $(AuL)-2-Si'BuMe_2-C_2B_{10}H_{10}$  [L=PPh<sub>3</sub> (2), PPh<sub>2</sub>Me (3) and AsPh<sub>3</sub> (4)] (Scheme 1).

Complexes 2–4 behave as nonconductors in acetone solutions. In their IR spectra the v(BH) absorptions appear at 2592 (br, vs), (2), 2664 (br, vs) (3) and 2566 (br, vs) (4) cm<sup>-1</sup>, respectively.

The positive liquid secondary-ion mass spectra (LSIMS) of complexes 2–4 show the molecular peaks at m/z = 716 (2, 50%), 597 (3, 5%) and 760 (4, 40%), although the most intense ones correspond to the fragments [AuL<sub>2</sub>]<sup>+</sup> [m/z 721 (2), 597 (3) and 809 (4)].

Compounds **2** and **3** exhibit only one signal in their  ${}^{31}P{}^{1}H{}$  NMR spectra downfield referred to the starting materials ( $\Delta\delta$  = 4.8 (**2**) and 5.5 (**3**) ppm).

In the <sup>1</sup>H NMR spectra of **2–4** a multiplet and a broad signal appear which correspond to the phenylic protons (7–7.8 ppm) and the BH protons (1–3 ppm) respectively. All of them exhibit two resonances due to the Si'BuMe<sub>2</sub> fragment at about 1.0 and 0.5 ppm. In addition to this pattern, compound **3** displays one doublet due to the methyl protons of the phosphine ligand.

Complex **2** has been characterised by X-ray diffraction studies and the structure is shown in Fig. 1. Selected bond lengths and angles are collected in Table 1. In the molecule the gold centre exhibits a distorted linear geometry,  $P-Au-C(1) = 171.3(12)^\circ$ , although there are no obvious reasons for this distortion, it may be possible that because the bulky nature of the Si'BuMe<sub>2</sub> group there is some steric hindrance. This distorted geometry is not observed in [Au(2-CH<sub>2</sub>O-CH<sub>3</sub>)-1,2-C<sub>2</sub>B<sub>10</sub>H<sub>10</sub>)(AsPh<sub>3</sub>)] [As-Au-C=177.05(2)°] [21].

The Au–P distance of 2.2718(13) Å is of the same order than those found in another *o*-carboranyl compounds such as  $[Au(2-H-1,2-C_2B_{10}H_{10})(PPh_3)]$ (2.271(2) Å) [22] or in the *m*-carboranyl derivative [23]  $[Au_2(\mu-1,7-C_2B_{10}H_{10})(PPh_3)_2]$  (2.265(2), 2.271(2) Å) and can also be compared with those found in complexes of the type  $[AuR(PPh_3)]$  ( $R = C_6F_5$ , 2.27(1) Å) [32].

The Au–C(1) distance, 2.050(4)Å, is of the same order as those obtained for the complexes commented below (2.054(7), 2.047(7)Å for the *m*-carborane derivative; 2.039(8) and 2.039(8) Å for the mono and disubstituted o-carborane derivatives respectively). It has been shown that the Au-C distances depend on the hybridisation state of the carbon atom. The distances found in the commented o-carboranyl and mcarboranyl complexes are clearly characteristic values of a carbon with sp<sup>2</sup> hybridisation [20], which range from 2.05 to 2.09 Å. These short Au-C distances in carborane derivatives support Stone's observations that the unusual stability of auracarboranes is attributed to the electron withdrawing influence of the cage. The fact that a similar stability is observed in gold-perfluoroalkyl compounds, with the Au-C distances only slightly longer, but in the range of an sp<sup>2</sup> hybridisation, is coherent with such a conclusion.

No organometallic derivative of this ligand has been reported as far as we know, but no appreciably distortion is expected for the Si'BuMe<sub>2</sub> group from one compound to another, thus the Si–C(2) distance 1.924(5) Å compares well with those found in  $1,2-(1-SiMe_2Bu-1,2-C_2B_{10}H_{10})_2$ -C<sub>6</sub>H<sub>4</sub> [1.951(4), 1.956 Å] [33] as well as the C(1)–C(2) distance 1.706(6) Å in complex **2**, 1.714(6), 1.707(7) Å in the related species.

Reaction of **1** with PPN[AuCl<sub>2</sub>] [PPN = N(PPh<sub>3</sub>)<sub>2</sub>] in molar ratio 2:1 affords a compound which contains two carboranyl groups: PPN[Au{2-Si'BuMe<sub>2</sub>-1,2- $C_2B_{10}H_{10}$ }] (5) (Figure 1).

Compound 5 behaves as 1:1 electrolyte in acetone solutions. In the IR spectrum of 5 the v(BH) absorption appears at 2632 cm<sup>-1</sup>. Its LSIMS (+) spectrum



Scheme. 1.



Fig. 1. The molecule of complex 2 in the crystal showing the atom-numbering scheme, radii are arbitrary. H atoms are omitted for clarity.

Au–C(1)	2.050(4)	Au–P	2.2718(13)	
Si-C(3)	1.859(5)	Si-C(4)	1.863(5)	
Si-C(5)	1.897(5)	Si-C(2)	1.924(5)	
P-C(11)	1.813(5)	P-C(31)	1.814(4)	
P-C(21)	1.822(4)	C(1)–C(2)	1.706(6)	
C(1)–Au–P	171.34(12)	C(3)-Si-C(4)	109.1(3)	
C(3)–Si–C(5)	109.2(3)	C(4)-Si-C(5)	109.9(2)	
C(3)-Si-C(2)	106.1(2)	C(4)-Si- $C(2)$	107.9(2)	
C(5)–Si–C(2)	114.6(2)	C(11)–P–C(31)	105.9(2)	
C(11)–P–C(21)	106.3(2)	C(31) - P - C(21)	104.4(2)	
C(11)–P–Au	116.7(2)	C(31)–P–Au	113.1(2)	
C(21)–P–Au	109.6(2)	C(2)-C(1)-Au	126.1(3)	
B(5)–C(1)–Au	117.7(3)	B(3)–C(1)–Au	118.7(3)	
B(6)-C(1)-Au	122.5(3)	B(4)-C(1)-Au	115.6(3)	
B(7)–C(2)–Si	119.1(3)	C(1)–C(2)–Si	121.9(3)	
B(11)–C(2)–Si	118.6(3)	B(3)-C(2)-Si	120.2(3)	
B(6)-C(2)-Si	118.7(3)	C(8)–C(5)–Si	111.4(3)	
C(7)–C(5)–Si	106.2(3)	C(6)–C(5)–Si	113.9(3)	
C(16)–C(11)–P	121.9(4)	C(12)–C(11)–P	118.9(4)	
C(26)–C(21)–P	118.2(4)	C(22)–C(21)–P	122.4(4)	
C(36)-C(31)-P	121.1(3)	C(32)–C(31)–P	119.4(4)	

Table 1. Selected bond lengths (Å) and angles (°) for **2** 

presents the molecular peak as the most intense at m/z=712. The <sup>1</sup>H NMR spectrum of **5** exhibits a similar pattern to those commented above for **2** and **4**.

## EXPERIMENTAL

Infrared spectra were recorded in the range from 4000 to  $200 \,\mathrm{cm}^{-1}$  on a Perkin-Elmer 883 spec-

trophotometer using Nujol mulls between polyethylene sheets. Conductivities were measured in ca.  $5 \times 10^{-4}$  mol dm<sup>-3</sup> solution with a Philips PW 9509 conductimeter. C and H analyses were carried out with a Perkin–Elmer 2400 microanalyser. Mass spectra were recorded on a VG Autospec, with the liquid secondary ion mass spectrometry (LSIMS +) technique using nitrobenzylalcohol as matrix. NMR spectra were recorded on a Varian Unity 300 and Bruker

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ARX 300 spectrometer in CDCl<sub>3</sub>. Chemical shifts are cited relative to 85% H<sub>3</sub>PO<sub>4</sub> (external, <sup>31</sup>P) and SiMe<sub>4</sub> (external, <sup>1</sup>H).

#### Starting materials

o-carborane was from Desxil Corporation and Si<sup>t</sup> BuMe<sub>2</sub>Cl was from Aldrich, both of them were used as given. Compounds 1-Si'BuMe<sub>2</sub>-1,2-C<sub>2</sub>B<sub>10</sub>H<sub>11</sub> [31], [AuCl(PPh<sub>3</sub>)], [AuCl(PPh<sub>2</sub>Me)], [AuCl(AsPh<sub>3</sub>)] [34] and PPN[AuCl<sub>2</sub>] [35] were synthesised according to the literature procedures.

# Preparation of $[1-(AuL)-2-St^*BuMe_2-1,2-C_2B_{10}H_{10}]$ [ $L=PPh_3$ (2), $PPh_2Me$ (3), $AsPh_3$ (4)]

To a solution of 1-Si'BuMe<sub>2</sub>-1,2-C<sub>2</sub>B<sub>10</sub>H<sub>11</sub> (77.4 mg, 0.3 mmol) in dried diethyl ether (30 cm<sup>3</sup>) at 0°C and under nitrogen atmosphere Li<sup>n</sup>Bu was added (0.3 mmol, 0.48 ml and 0.62 M). After stirring the mixture for 30 min [AuCIL] was added [0.3 mmol,  $L = PPh_3$ , 14.8 mg;  $L = PPh_2Me$ , 12.9 mg;  $L = AsPh_3$ , 15.9 mg]. The suspension was stirred for another 30 min and allowed to warm. After filtration of the suspension over celite and concentration to ca. 5 cm<sup>3</sup> complexes 2 (76%), 3 (48%) and 4 (57%) as white solids are obtained by addition of n-hexane (ca. 10 cm<sup>3</sup>). 2 Anal. (%) Found: C, 43.05; H, 5.35. Calc.  $C_{26}H_{40}AuB_{10}PSi:$  C, for 43.55; H, 5.6.  $\Lambda_M = 28 \,\Omega^{-1} \,\mathrm{cm}^2 \,\mathrm{mol}^{-1}$ . <sup>1</sup>H NMR: 0.25 (s, 6H, CH<sub>3</sub>), 0.88 (s, 9H, 'Bu), 7–7.6 (m, br, 15H, Ph).  ${}^{31}P{}^{1}H{}$ NMR: 38.4 (s). 3 Anal. (%) Found: C, 38.35; H, 5.6. Calc. for  $C_{21}H_{38}AuB_{10}PSi$ : C, 38.55; H, 5.85.  $\Lambda_M = 14 \,\Omega^{-1} \,\mathrm{cm}^2 \,\mathrm{mol}^{-1}$ . <sup>1</sup>H NMR: 0.35 (s, 6H, CH<sub>3</sub>), 0.99 (s, 9H, <sup>*t*</sup>Bu), 1.95 (d, J(PH) = 8.4 Hz, 3H, CH<sub>3</sub>), 7.2-7.6 (m, br, 10H, Ph). <sup>31</sup>P{<sup>1</sup>H} NMR: 22.8 (s). 4 Anal. (%) Found: C, 41.05; H, 5.3. Calc. for  $C_{26}H_{40}AsAuB_{10}Si:$ С, 41.35; 5.4. H.  $\Lambda_M = 9 \Omega^{-1} \text{ cm}^2 \text{ mol}^{-1}$ . <sup>1</sup>H NMR: 0.32 (s, 6H, Me), 0.95 (s, 9H, 'Bu), 7.0-7.51 (m, br, 15H, Ph).

# Preparation of $PPN[Au\{2-Si^{*}BuMe_{2}-1,2-C_{2}B_{10}H_{10}\}_{2}]$ (5)

To a solution of 1-Si'BuMe<sub>2</sub>-1,2-C<sub>2</sub>B<sub>10</sub>H<sub>11</sub> (77.4 mg, 0.3 mmol) in dry diethyl ether (30 cm<sup>3</sup>) at 0°C and under nitrogen atmosphere Li"Bu was added (0.3 mmol, 0.48 ml, 0.62 M). After stirring the mixture for 30 min PPN[AuCl<sub>2</sub>] was added [0.15 mmol, 12 mg]. The suspension was stirred for another 30 min and allowed to warm. After filtration of the suspension over celite and concentration to ca. 5 cm<sup>3</sup> complex **5** (68%) is afforded by addition of *n*-hexane (ca. 10 cm<sup>3</sup>). Anal. (%) Found: C, 50.65; H, 6.5; N, 1.15. Calc. for C<sub>52</sub>H<sub>80</sub>AuB<sub>20</sub>NP<sub>2</sub>Si<sub>2</sub>: C, 49.95; H, 6.45; N, 1.1.  $\Lambda_M = 134 \Omega^{-1}$  cm<sup>2</sup> mol<sup>-1</sup>. <sup>1</sup>H NMR: 0.39 (s, 12H, Me), 1.02 (s, 18H, 'Bu), 7.46–7.65 (m, br, 30H, Ph). <sup>31</sup>P {<sup>1</sup>H} NMR: 21.2 (s).

#### X-ray crystal structure determination of 2

Crystal Data: 2,  $C_{26}H_{40}AuB_{10}PSi$ , Mr = 716.71, triclinic, space group P1, a = 10.164(2), b = 12.557(3), c = 14.200(2) Å,  $\alpha = 115.680(10)$ ,  $\beta = 93.040(10)$ ,  $\gamma =$ 102.700(10), U=1570.5(5) Å<sup>3</sup>, Z=2,  $D_{c}=1.516$  Mg  $m^{-3}$ ,  $\lambda$  (Mo-K $\alpha$ ) = 0.71073 Å,  $\mu$  = 4.79 mm<sup>-1</sup>, F(000) = 708,  $T = -100^{\circ}$ C. Single crystals were obtained by a slow diffusion of *n*-hexane into a dichloromethane solution of complex 2. A colourless prism  $0.50 \times$  $0.40 \times 0.30 \text{ mm}$  was used to collect 5561 intensities to  $2\theta_{\rm max} = 50^{\circ}$  (Siemens P4 diffractometer, monochromated Mo-K $\alpha$  radiation) of which 5119 were independent ( $R_{int} = 0.032$ ). An absorption correction based on  $\Psi$ -scans was applied with transmission factors 0.599-0.863. Cell constants were refined from setting angles of 64 reflections in the range  $2\theta$  from 10 to 25°. The structure was solved by the heavy-atom method and subjected to full-matrix least-squares refinement on  $F^2$  (SHELXL-93); all the atoms were refined anisotropically, H atoms were included using a riding model. Refinement proceed to  $wR(F^2) = 0.074$ for 5117 reflections and 357 parameters, conventional  $R(F) = 0.029, S(F^2) = 1.022, \text{ maximum } \Delta \rho \ 2.4 \text{ e } \text{\AA}^{-3}.$ 

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