

Gold(I) Complexes with the *nido*-Diphosphino Ligand [7,8-(Ph₂P)₂-7,8-C₂B₉H₁₀][−]. Preparation of the First Metallocarborane Complex of this Ligand. Crystal Structures of [Au{(PPh₂)₂C₂B₉H₁₀}(PPh₃)]·CH₂Cl₂ and [Au₂{(PPh₂)₂C₂B₉H₁₀}₂{(PPh₂)₂(CH₂)₃}]·3Me₂CO

Olga Crespo,[†] M. Concepción Gimeno,[†] Peter G. Jones,[‡] and Antonio Laguna^{*,†}

Departamento de Química Inorgánica, Instituto de Ciencia de Materiales de Aragón, Universidad de Zaragoza-CSIC, 50009 Zaragoza, Spain, and Institut für Anorganische und Analytische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany

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The reaction of [AuCl(PR₃)] with [1,2-(Ph₂P)₂-1,2-C₂B₁₀H₁₀] in refluxing ethanol proceeds with partial degradation (removal of a boron atom adjacent to carbon) of the *closo* species to give [Au{(PPh₂)₂C₂B₉H₁₀}(PR₃)] [PR₃ = PPh₃ (**1**), PPh₂Me (**2**), PPh₂(4-Me-C₆H₄) (**3**), P(4-Me-C₆H₄)₃ (**4**), P(4-OMe-C₆H₄)₃ (**5**)]. Similarly, the treatment of [Au₂Cl₂(μ-P-P)] with [1,2-(Ph₂P)₂-1,2-C₂B₁₀H₁₀] under the same conditions leads to the complexes [Au₂{(PPh₂)₂C₂B₉H₁₀}₂(μ-P-P)] [P-P = dppe = 1,2-bis(diphenylphosphino)ethane (**6**), dppp = 1,3-bis(diphenylphosphino)propane (**7**)], where the dppe or dppp ligands bridge two gold *nido*-diphosphine units. The reaction of **1** with NaH leads to removal of one proton, and further reaction with [Au(PPh₃)(tht)]ClO₄ gives the novel metallocarborane compound [Au₂{(PPh₂)₂C₂B₉H₉}(PPh₃)₂] (**8**). The structure of complexes **1** and **7** have been established by X-ray diffraction. [Au{(PPh₂)₂C₂B₉H₁₀}(PPh₃)] (**1**) (dichloromethane solvate) crystallizes in the monoclinic space group *P*2₁/*c*, with *a* = 17.326(3) Å, *b* = 20.688(3) Å, *c* = 13.442(2) Å, β = 104.710(12)°, *Z* = 4, and *T* = −100 °C. [Au₂{(PPh₂)₂C₂B₉H₁₀}₂(μ-dppp)] (**7**) (acetone solvate) is triclinic, space group *P*1, *a* = 13.432(3) Å, *b* = 18.888(3) Å, *c* = 20.021(3) Å, α = 78.56(2)°, β = 72.02(2)°, γ = 73.31(2)°, *Z* = 2, and *T* = −100 °C. In both complexes the gold atom exhibits trigonal planar geometry with the 7,8-bis(diphenylphosphino)-7,8-dicarba-*nido*-undecaborate(1−) acting as a chelating ligand.

Introduction

1,2-Dicarba-*closo*-dodecaborane (*o*-carborane) forms a great variety of derivatives as a consequence of the high resistance of the *o*-carborane cage to chemical attack.¹ Despite their cost, such properties make them attractive for various specialized applications. These include the incorporation of high concentrations of boron atoms in tumor-seeking drugs for boron neutron capture therapy (BNCT),^{2–4} the synthesis of high temperature polymers,⁵ the production of ceramics related to boron carbide,⁶ or the synthesis of derivatives with nonlinear optical (NLO) properties.⁷ Furthermore, the ability to form *nido* anionic species by treatment with nucleophiles has led to their use in coordination chemistry with potential applications as novel catalysts,⁸ radiochemical drugs,⁹ etc.

During our studies using *o*-carborane derivatives as ligands, we have reported the synthesis of unusual higher-coordinated

gold(I) complexes with the 1,2-bis(diphenylphosphino)-*o*-carborane ligand,^{10–12} and also of related species for silver(I).¹³ *o*-Carborane is an interesting backbone because its electron-withdrawing power, large size, and extensive electron delocalization confer an unusual stability on the molecule. These factors, together with the great tendency of the 1,2-bis(diphenylphosphino)-*o*-carborane ligand to act as a chelating ligand, have allowed us to prepare novel complexes with *o*-carborane derivatives as ligands. However, very little work has been done on the partially degraded anionic ligand [7,8-(Ph₂P)₂-7,8-C₂B₉H₁₀][−], and the only reported complexes are [Cu{(PPh₂)₂C₂B₉H₁₀}(PPh₃)], (NMe₄)[Cu{(PPh₂)₂C₂B₉H₁₀}Cl-(PPh₃)], and [Cu{(PPh₂)₂C₂B₉H₁₀}(PPh₃)(Me₂CO)].¹⁴ This ligand has several coordination modes: first *exo-nido*-coordination to the phosphorus atoms, and second η⁵-coordination, after deprotonation, to the open pentagonal C₂B₃ face, thus forming metallocarboranes. No examples combining both types of coordination have been reported thus far.

Here we describe the synthesis of three-coordinate gold(I) complexes with [7,8-(Ph₂P)₂-7,8-C₂B₉H₁₀][−] and the preparation of the first metallocarborane complex of this ligand.

Results and Discussion

It has been reported that the treatment of 1,2-bis(diphenylphosphino)-*o*-carborane with alkoxides does not produce the expected *nido* species, leading instead to the 7,8-dicarba-*nido*-

[†] Universidad de Zaragoza-CSIC.

[‡] Technische Universität Braunschweig.

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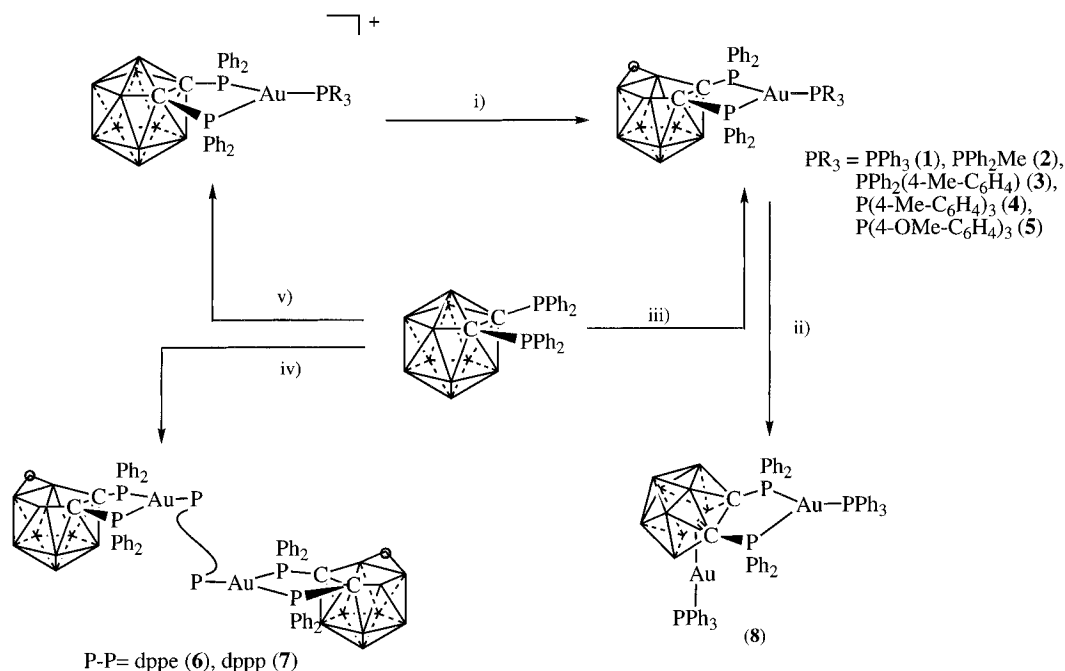
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Scheme 1^a

^a Key: (i) EtOH/ Δ ; (ii) [Au₂Cl₂(μ -P-P)], EtOH/ Δ ; (iii) [AuCl(PR₃)], EtOH/ Δ ; (iv) NaH, [Au(PPh₃)(tht)]ClO₄; (v) [AuCl(PR₃)], CH₂Cl₂.

undecaborate(1-) anion by C-P cleavage, and also that the diphosphine is stable in refluxing ethanol.¹⁴ However complexation to a metal centre makes the partial degradation of the carborane moiety easier.¹⁴ We have prepared gold(I) complexes with the ligand [(Ph₂P)₂C₂B₉H₁₀]⁻ using this method. Thus the reaction of [AuCl(PPh₃)] with [(Ph₂P)₂C₂B₁₀H₁₀] in refluxing ethanol proceeds with removal of one of the boron atoms adjacent to carbon and gives the neutral complexes [Au{(PPh₂)₂C₂B₉H₁₀}(PR₃)] [PR₃ = PPh₃ (1), PPh₂Me (2), PPh₂(4-Me-C₆H₄) (3), P(4-Me-C₆H₄)₃ (4), P(4-OMe-C₆H₄)₃ (5)].

The first step in these processes must be the coordination of the gold atom to the diphosphine, followed by the partial degradation of the carborane moiety. To prove this we have carried out the reaction with [Au{(PPh₂)₂C₂B₁₀H₁₀}(PR₃)]ClO₄,¹⁰ which contains the *closo* diphosphine, in refluxing ethanol, also obtaining the neutral *nido* species (see Scheme 1).

In the IR spectra of these complexes the absorptions arising from the B-H stretching modes of the *o*-carborane nucleus appear at lower energy than the complexes that contain the *closo* carboranes.¹⁰ Teixidor *et al.* have observed¹⁵ that the position of the B-H bands in the IR spectrum is highly significant when studying 1,2-dicarba-*closo*-dodecaborane or 7,8-dicarba-*nido*-undecaborate derivatives; with few exceptions it seems that if $\nu(\text{B-H})$ is < 2550 cm⁻¹, the cluster is partially degraded, but if $\nu(\text{B-H})$ is > 2550 cm⁻¹, the cluster is a *closo* compound. This is consistent with the values found in our complexes.

Their ³¹P{¹H} NMR spectra show AB₂ systems, except for PR₃ = PPh₂Me which presents an AX₂ system. The AB₂ systems show eight transitions and simulated spectra agree with experimental findings. Chemical shifts and coupling constants are very similar to those found in the spectra of the analogous *closo* derivatives. In the ¹H NMR spectra a multiplet around -2 ppm appears; this corresponds to the proton of the eliminated boron atom, which possesses a marked mobility in solution. The ¹¹B NMR spectra show the characteristic pattern of the 7,8-dicarba-*nido*-undecaborate(1-) derivatives, which usually present five resonances in a ratio 2:3:2:1:1.¹⁶⁻¹⁸ In our complexes only

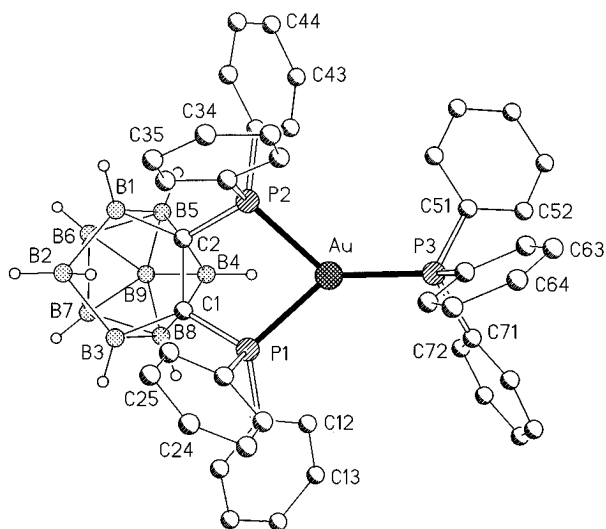


Figure 1. Molecular structure of **1** with the atom-numbering scheme. Phenyl hydrogen atoms are omitted for clarity; radii are arbitrary.

four signals appear, one of which has a shoulder. The range of the resonances is from *ca.* -5 to -38 ppm; the high-field signals are assigned to the boron atoms with higher coordination number.

The positive-ion fast atom bombardment (FAB) mass spectra of these derivatives show the molecular peak [M]⁺ at *m/z* = 961 (**1**, 69%), 899 (**2**, 100%), 975 (**3**, 100%), 1003 (**4**, 100%) and 1051 (**5**, 47%). Other characterization data for **1-8** are given in Table 1.

The crystal structure of complex **1** has been determined by X-ray diffraction, and is shown in Figure 1. Atom coordinates are given in Table 2 and selected bond lengths and angles in Table 3. The gold atom displays trigonal planar coordination by the three phosphorus atoms, and is only 0.076(1) Å out of the phosphorus plane. The major deviation from ideal geometry

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Table 1. Analytical, Conductivity, and NMR Data and $\nu(\text{B-H})$ Frequencies for Complexes **1–8**

complex	yield (%)	anal. (%) ^a		Λ_{M}^b	³¹ P{ ¹ H} ^c		¹ H ^c		$\nu(\text{B-H})/\text{cm}^{-1}$
		C	H		δ	$J(\text{PP})$	δ	$J(\text{PP})$	
[Au{(PPh ₂) ₂ C ₂ B ₉ H ₁₀ }(PPh ₃)] (1)	76	54.55 (55.0)	4.50 (4.7)	1	45.8 63.1	130.6			2536
[Au{(PPh ₂) ₂ C ₂ B ₉ H ₁₀ }(PPh ₂ Me)] (2)	71	51.95 (52.1)	4.75 (4.8)	0	26.2 (t) 63.4 (d)	134.8	2.21(d)	8.79	2521
[Au{(PPh ₂) ₂ C ₂ B ₉ H ₁₀ }{PPh ₂ (4-Me-C ₆ H ₄)}] (3)	74	55.2 (55.45)	4.6 (4.85)	0	44.8 62.8	132.1	2.42 (s)		2524
[Au{(PPh ₂) ₂ C ₂ B ₉ H ₁₀ }{P(4-Me-C ₆ H ₄) ₃ }] (4)	71	56.15 (56.25)	5.05 (5.1)	0	43.2 62.6	132.4	2.43 (s)		2521
[Au{(PPh ₂) ₂ C ₂ B ₉ H ₁₀ }{P(4-OMe-C ₆ H ₄) ₃ }] (5)	68	53.5 (53.7)	4.75 (4.9)	1	41.3 62.4	133.0	3.8 (s)		2587
[Au ₂ {(PPh ₂) ₂ C ₂ B ₉ H ₁₀ }] ₂ (μ -dppe)] (6)	81	52.1 (52.2)	4.35 (4.7)	1	63.7 (d) 42.8 (t)	131.23	2.4 (m)		2529
[Au ₂ {(PPh ₂) ₂ C ₂ B ₉ H ₁₀ }] ₂ (μ -dppp)] (7)	94	52.35 (52.45)	4.8 (4.8)	2	63.4 (d) 38.0 (t)	131.35	2.1 (m) 4.8 (m)		2531
[Au ₂ {(PPh ₂) ₂ C ₂ B ₉ H ₁₀ }(PPh ₃) ₂] (8)	75	52.6 (52.45)	4.2 (4.25)	4	46.6 (t) 66.0 (d) 41.0 (m)	128.7			2526

^a Calculated values in parentheses. ^b In acetone, $\Omega^{-1} \text{ cm}^2 \text{ mol}^{-1}$. ^c Recorded in CDCl₃, values in ppm, coupling constants in Hz, with t = triplet, d = doublet, s = singlet, and m = multiplet.

Table 2. Atomic Coordinates [$\times 10^4$] and Equivalent Isotropic Displacement Parameters [$\text{\AA}^2 \times 10^3$] for compound **1**^a

	x	y	z	U(eq)		x	y	z	U(eq)
Au	7305.6(1)	4974.7(1)	6814.5(1)	23.2(1)	C(34)	8093(4)	6345(3)	10689(5)	45.9(15)
P(1)	6595.9(7)	4185.2(6)	7531.4(10)	20.6(3)	C(35)	7335(4)	6116(3)	10620(4)	42.5(14)
P(2)	6552.1(7)	5745.9(6)	7508.1(10)	20.4(3)	C(36)	6851(3)	5928(2)	9690(4)	29.5(12)
P(3)	8323.0(7)	5014.7(6)	6019.6(10)	24.3(3)	C(41)	6267(3)	6524(2)	6908(4)	23.8(11)
C(1)	5655(3)	4565(2)	7586(4)	19.3(10)	C(42)	6089(3)	6567(2)	5836(4)	29.8(12)
C(2)	5625(3)	5339(2)	7566(4)	20.3(10)	C(43)	5874(3)	7149(3)	5362(5)	38.5(13)
B(1)	5001(3)	5636(3)	8161(5)	23.9(12)	C(44)	5846(3)	7698(2)	5931(5)	39.7(14)
B(2)	4513(4)	4948(3)	8607(5)	27.5(12)	C(45)	6021(4)	7661(2)	6991(5)	42.6(15)
B(3)	5051(3)	4265(3)	8219(4)	22.7(12)	C(46)	6228(3)	7078(2)	7482(4)	31.8(12)
B(4)	5214(3)	4933(3)	6437(4)	23.6(12)	C(51)	8307(3)	5700(2)	5169(4)	30.4(12)
B(5)	4734(3)	5619(3)	6780(4)	24.7(12)	C(52)	8651(3)	5686(3)	4341(4)	40.4(14)
B(6)	4058(3)	5367(3)	7472(4)	23.7(12)	C(53)	8616(4)	6216(3)	3719(5)	56(2)
B(7)	4076(3)	4488(3)	7497(4)	26.4(13)	C(54)	8233(4)	6772(3)	3904(5)	60(2)
B(8)	4781(3)	4242(3)	6842(4)	24.6(13)	C(55)	7885(4)	6794(3)	4703(6)	55(2)
B(9)	4187(3)	4911(3)	6368(4)	24.8(12)	C(56)	7920(3)	6262(3)	5336(5)	43.7(15)
C(11)	6370(3)	3406(2)	6918(4)	25.3(11)	C(61)	9268(3)	5059(2)	6977(4)	30.4(11)
C(12)	6475(4)	3320(3)	5938(5)	43.9(14)	C(62)	9947(3)	5335(3)	6789(4)	34.3(12)
C(13)	6314(4)	2719(3)	5467(6)	64(2)	C(63)	10665(3)	5325(3)	7530(5)	42.2(14)
C(14)	6064(4)	2214(3)	5964(6)	56(2)	C(64)	10715(4)	5036(3)	8466(5)	50(2)
C(15)	5965(3)	2301(3)	6936(6)	48(2)	C(65)	10048(4)	4763(3)	8676(5)	51(2)
C(16)	6112(3)	2888(2)	7405(4)	36.1(13)	C(66)	9326(3)	4775(3)	7934(4)	41.7(14)
C(21)	7125(3)	3975(2)	8837(4)	24.9(11)	C(71)	8439(3)	4319(2)	5239(4)	28.3(11)
C(22)	7774(3)	3563(3)	8979(5)	48(2)	C(72)	7820(3)	4162(3)	4391(4)	40.5(14)
C(23)	8201(4)	3396(3)	9958(5)	59(2)	C(73)	7860(3)	3620(3)	3816(4)	41.2(14)
C(24)	7997(4)	3640(3)	10801(5)	50(2)	C(74)	8515(3)	3220(3)	4072(5)	43.2(14)
C(25)	7371(3)	4063(3)	10679(4)	41.7(14)	C(75)	9133(4)	3371(3)	4905(5)	49(2)
C(26)	6934(3)	4231(3)	9697(4)	31.8(12)	C(76)	9096(3)	3914(3)	5483(4)	37.2(13)
C(31)	7121(3)	5952(2)	8802(4)	22.1(10)	C(99)	10262(7)	2141(4)	8040(8)	114(3)
C(32)	7896(3)	6169(3)	8889(4)	39.0(14)	Cl(1)	10418(2)	1943(2)	6899(3)	132.8(11)
C(33)	8372(3)	6365(3)	9818(5)	47(2)	Cl(2)	9867.7(14)	2915.4(11)	8103(2)	89.7(7)

^a $U(\text{eq})$ is defined as one-third of the trace of the orthogonalized U_{ij} tensor.

is associated with the bite angle of the diphosphine, P(1)–Au–P(2) 84.91(4)°. This angle is even narrower than that in the *closo* species [Au{(PPh₂)₂C₂B₁₀H₁₀}(PPh₃)]ClO₄ [90.2(1)°].¹⁰ The bond to the monodentate phosphine, 2.2831(13) Å, is appreciably shorter than those to the diphosphine [2.3896(13) and 2.3952(12) Å]; a similar pattern was observed in [Au{(PPh₂)₂C₂B₁₀H₁₀}(PPh₃)]ClO₄ [2.318, 2.405, 2.417(1) Å]. Broadly similar values were found for other three coordinate complexes such as [Au(PPh₃)₃]⁺ (three independent investigations)^{19–21} [2.345–2.408 Å], or [Au(dppf)(PPh₃)]ClO₄ [2.343–(2)–2.409(2) Å].²² The P–C bond lengths are 1.827(5) and 1.832(5) Å, somewhat shorter than those in the *closo* species,

1.883(5) and 1.870(5) Å, possibly because of the negative charge of the carborane moiety. The chelate ring displays an envelope conformation, with the gold atom 0.85 Å out of the plane of the other four atoms. The central boron atom of the C₂B₃ open face is bonded to two hydrogen atoms, with B–H 1.08(3) and 1.09(3) Å.

The reaction of [Au₂Cl₂(μ -P-P)] with 2 equiv of [(Ph₂P)₂C₂B₁₀H₁₀], under the same conditions of refluxing ethanol, proceeds in a similar manner, and the complexes [Au₂{(PPh₂)₂C₂B₉H₁₀}]₂(μ -P-P) [P-P = 1,2-bis(diphenylphosphino)ethane, dppe (**6**), 1,3-bis(diphenylphosphino)propane, dppp (**7**)] are isolated. Complexes **6** and **7** are yellow solids stable to air and moisture and behave as non-conductors in acetone solution.

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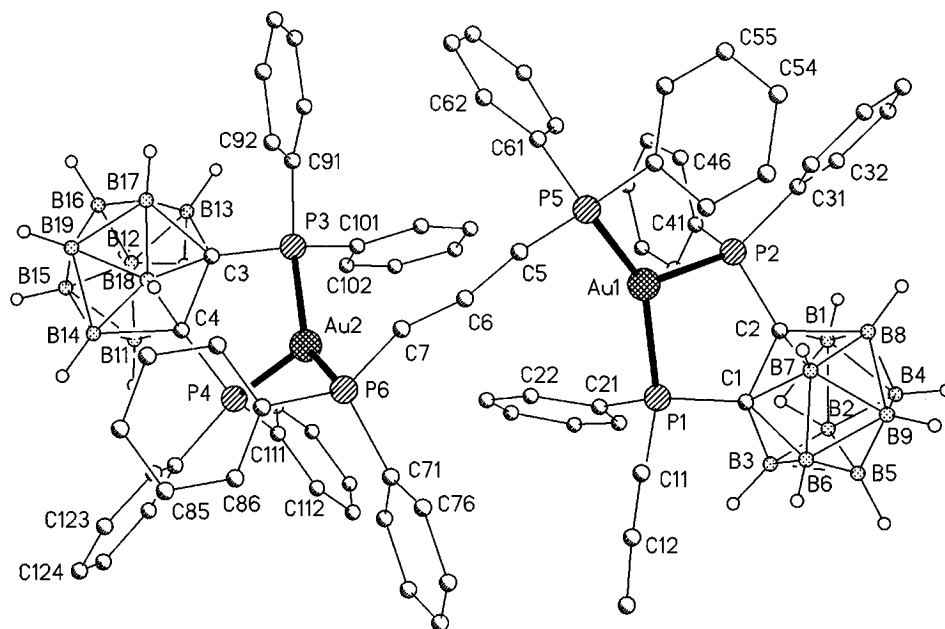


Figure 2. Molecular structure of **7** with the atom-labeling scheme. Phenyl hydrogen atoms are omitted for clarity; radii are arbitrary.

Table 3. Selected Bond Lengths (Å) and Angles (deg) for Compound **1**

Au—P(3)	2.2831(13)	Au—P(1)	2.3896(13)
Au—P(2)	2.3952(12)	P(1)—C(11)	1.808(5)
P(1)—C(21)	1.814(5)	P(1)—C(1)	1.827(5)
P(2)—C(41)	1.812(5)	P(2)—C(31)	1.819(5)
P(2)—C(2)	1.832(5)	P(3)—C(61)	1.809(5)
P(3)—C(51)	1.818(5)	P(3)—C(71)	1.822(5)
P(3)—Au—P(1)	138.67(4)	P(3)—Au—P(2)	136.05(4)
P(1)—Au—P(2)	84.91(4)	C(11)—P(1)—C(21)	103.0(2)
C(11)—P(1)—C(1)	108.1(2)	C(21)—P(1)—C(1)	107.5(2)
C(11)—P(1)—Au	120.0(2)	C(21)—P(1)—Au	111.6(2)
C(1)—P(1)—Au	106.1(2)	C(41)—P(2)—C(31)	103.7(2)
C(41)—P(2)—C(2)	106.5(2)	C(31)—P(2)—C(2)	109.6(2)
C(41)—P(2)—Au	122.2(2)	C(31)—P(2)—Au	108.2(2)
C(2)—P(2)—Au	106.3(2)	C(61)—P(3)—C(51)	105.9(2)
C(61)—P(3)—C(71)	103.7(2)	C(51)—P(3)—C(71)	103.9(2)
C(61)—P(3)—Au	109.6(2)	C(51)—P(3)—Au	115.6(2)
C(71)—P(3)—Au	116.9(2)		

In the IR spectra, apart from the typical bands arising from the diphosphine ligands, the $\nu(\text{B—H})$ absorptions appear at 2529 (s, br) and 2531 (s, br) cm^{-1} , respectively.

The partially degraded nature of the carborane in the complexes can be seen in the ^1H NMR spectra, which show a pattern similar to that discussed above for complexes **1–5**. The $^{31}\text{P}\{^1\text{H}\}$ NMR spectra correspond to AX_2 systems, and therefore a triplet and a doublet appear for the two different phosphorus environments.

In the positive-ion mass spectra (FAB+) the molecular peak appears only for complex **7** at $m/z = 1809$, and then with only 5% intensity; however, the cations arising from the loss of one negative ligand, $[(\text{Ph}_2\text{P})_2\text{C}_2\text{B}_9\text{H}_{10}]^-$, are present in both cases with high intensity.

The structure of complex **7** was confirmed by an X-ray diffraction study, and the molecule is shown in Figure 2. Positional parameters are collected in Table 4 and selected bond lengths and angles in Table 5. The molecule has two gold atoms, each one bonded to the two phosphorus atoms of the diphosphine $[(\text{Ph}_2\text{P})_2\text{C}_2\text{B}_9\text{H}_{10}]^-$ and to one of the phosphorus atoms of the other diphosphine; thus both display a trigonal planar coordination. The major distortion arises from the restricted bite of the *nido* diphosphines, 86.06(10) and 84.35-(9)°, values of the same order as found in complex **1**. The gold atoms are located 0.023(2) and 0.127(2) Å out of the planes

formed by their three bonding partners. The proton initially belonging to the eliminated boron atom is bonded to the central boron atom of the C_2B_3 open face.

In a similar manner to that observed for complex **1**, the Au—P distances to the phosphorus atoms of the $[(\text{Ph}_2\text{P})_2\text{C}_2\text{B}_9\text{H}_{10}]^-$ are longer, 2.429(3), 2.365(3), 2.374(3) and 2.443(3) Å, than to the phosphorus atom of the bridging diphosphine, 2.293(3) and 2.288(3) Å, consistent with the principle that the shortest bond is opposite the narrowest angle. The chelate rings again display an envelope conformation, with the gold atoms 0.81 and 0.96 Å out of the plane of the other four atoms. The propyl backbone of the dppp is extended, with torsion angles -174 and -175° about C(5)—C(6) and C(6)—C(7) respectively.

The treatment of complex **1** with the strong base NaH deprotonates the cage affording the complex $\text{Na}[\text{Au}\{(\text{PPh}_2)_2\text{C}_2\text{B}_9\text{H}_9\}(\text{PPh}_3)]$, which was not isolated. Further reaction with $[\text{Au}(\text{PPh}_3)(\text{tht})]\text{ClO}_4$ gives the complex $[\text{Au}_2\{(\text{PPh}_2)_2\text{C}_2\text{B}_9\text{H}_9\}(\text{PPh}_3)_2]$ (**8**), where one AuPP_3^+ fragment has an *exo-nido* coordination to the phosphorus atoms, and the other has an η^5 -coordination to the open C_2B_3 face. Compound **8** is a yellow air- and moisture-stable solid, nonconducting in acetone solution. The IR spectrum shows bands similar to those of the starting material, with the $\nu(\text{B—H})$ absorption at 2526 (s, br) cm^{-1} . In the ^1H NMR spectrum the signal at ca. -2 ppm has disappeared, and in the $^{31}\text{P}\{^1\text{H}\}$ spectrum there are three different phosphorus environments. The *exo-nido*-phosphorus atoms present an AX_2 system, thus appearing as a triplet (PPh_3) and a doublet for the diphosphine phosphorus; the phosphorus atoms of the AuPP_3^+ group coordinated to the carborane open face appear as a broad multiplet, as a consequence of the coupling with the boron nuclei. As far as we are aware this is the first example of a complex combining coordination to the *exo*-heteroatoms and to the carborane moiety, thus forming the first metallocarborane complex of this ligand.

The mass spectrum also confirms the proposed stoichiometry, with the molecular peak appearing at $m/z = 1420$ (44%), and other fragments corresponding to the loss of AuPP_3^+ or the association $[\text{M} + \text{AuPP}_3]^+$ are present.

Experimental Section

Instrumentation. Infrared spectra were recorded in the range 4000–200 cm^{-1} on a Perkin-Elmer 883 spectrophotometer using Nujol mulls between polyethylene sheets. Conductivities were measured in

Table 4. Atomic Coordinates [$\times 10^4$] and Equivalent Isotropic Displacement Parameters [$\text{\AA}^2 \times 10^3$] for Compound 7

	x	y	z	U(eq)		x	y	z	U(eq)
Au(1)	1999.6(3)	1377.9(2)	2137.5(2)	28.1(1)	C(56)	5067(9)	19(6)	1065(6)	44(3)
Au(2)	3537.7(4)	3821.3(2)	3286.1(2)	27.6(1)	C(61)	4497(7)	1701(5)	1214(5)	23(2)
P(1)	304(2)	1726(2)	2937(2)	31.1(7)	C(62)	5548(8)	1748(6)	1126(6)	36(3)
P(2)	1022(2)	1338(2)	1312.2(15)	29.7(7)	C(63)	6054(10)	2184(6)	546(7)	51(3)
P(3)	4108(2)	4431.1(14)	2120.9(14)	24.9(7)	C(64)	5509(9)	2573(6)	77(6)	45(3)
P(4)	2796(2)	5070.7(15)	3600.8(15)	28.3(7)	C(65)	4479(10)	2540(6)	140(6)	50(3)
P(5)	3822(2)	1098.5(14)	1943.7(14)	23.4(6)	C(66)	3963(9)	2106(6)	725(6)	38(3)
P(6)	3710(2)	2715.6(14)	4000.1(14)	22.2(6)	C(71)	2581(11)	2432(9)	4677(7)	31(7)
C(1)	-629(8)	1341(6)	2681(6)	31(3)	C(72)	1679(13)	2988(7)	4850(8)	34(6)
C(2)	-286(8)	1188(6)	1875(5)	28(3)	C(73)	831(10)	2858(8)	5434(8)	54(7)
C(3)	4319(8)	5312(5)	2225(5)	26(2)	C(74)	885(11)	2171(9)	5846(7)	48(7)
C(4)	3693(8)	5625(5)	2965(5)	22(2)	C(75)	1787(13)	1615(7)	5674(8)	53(7)
C(5)	4331(8)	1104(5)	2694(5)	25(2)	C(76)	2635(11)	1745(8)	5089(8)	35(6)
C(6)	3839(8)	1844(6)	2999(5)	29(3)	C(71')	2430(11)	2595(10)	4584(8)	20(6)
C(7)	4359(9)	1881(6)	3572(6)	29(3)	C(72')	1515(15)	3077(9)	4459(8)	54(7)
B(1)	-1347(10)	1320(7)	1589(7)	32(3)	C(73')	519(12)	3027(10)	4926(11)	99(11)
B(2)	-2520(12)	1528(8)	2352(8)	41(4)	C(74')	436(12)	2496(12)	5519(10)	73(9)
B(3)	-1910(11)	1553(7)	3029(7)	36(3)	C(75')	1350(15)	2013(10)	5644(8)	66(8)
B(4)	-2067(11)	631(7)	2111(7)	40(4)	C(76')	2347(12)	2063(9)	5177(9)	53(7)
B(5)	-2406(12)	791(8)	3017(8)	47(4)	C(81)	4577(8)	2741(5)	4524(5)	28(2)
B(6)	-1210(11)	656(7)	3258(8)	38(4)	C(82)	5630(9)	2795(7)	4166(7)	58(4)
B(7)	-150(12)	436(8)	2523(7)	41(4)	C(83)	6291(10)	2875(8)	4534(9)	82(5)
B(8)	-660(10)	395(7)	1824(7)	32(3)	C(84)	5944(11)	2901(8)	5252(8)	77(4)
B(9)	-1293(11)	72(8)	2697(7)	40(4)	C(85)	4911(12)	2858(7)	5602(7)	60(4)
B(11)	3457(10)	6517(7)	2916(7)	31(3)	C(86)	4230(10)	2788(6)	5235(6)	45(3)
B(12)	4017(10)	6860(7)	2005(7)	31(3)	C(91)	5294(8)	4069(5)	1483(5)	29(2)
B(13)	4530(10)	5992(7)	1591(7)	32(3)	C(92)	6154(8)	3582(5)	1690(6)	34(3)
B(14)	4503(11)	5995(7)	3281(7)	37(3)	C(93)	7115(9)	3362(6)	1210(6)	47(3)
B(15)	4710(11)	6762(8)	2650(7)	40(4)	C(94)	7252(9)	3633(6)	504(6)	42(3)
B(16)	5400(10)	6441(7)	1803(7)	34(3)	C(95)	6439(9)	4112(6)	272(6)	40(3)
B(17)	5604(11)	5468(7)	1958(7)	36(3)	C(96)	5460(8)	4321(6)	754(5)	36(3)
B(18)	5027(10)	5183(7)	2849(7)	31(3)	C(101)	3074(8)	4593(5)	1668(5)	27(2)
B(19)	5709(11)	5921(7)	2599(7)	33(3)	C(102)	2383(8)	5267(6)	1579(5)	33(3)
C(11)	85(8)	1467(6)	3883(6)	37(3)	C(103)	1595(9)	5334(7)	1264(6)	47(3)
C(12)	-793(11)	1788(7)	4366(6)	65(4)	C(104)	1472(10)	4734(6)	1041(6)	48(3)
C(13)	-945(11)	1546(8)	5082(6)	68(4)	C(105)	2148(10)	4064(7)	1103(6)	52(3)
C(14)	-205(10)	1004(7)	5327(7)	60(4)	C(106)	2949(9)	3998(6)	1411(6)	45(3)
C(15)	696(11)	715(7)	4834(6)	58(4)	C(111)	1431(9)	5477(5)	3553(5)	29(2)
C(16)	855(9)	927(7)	4121(6)	44(3)	C(112)	629(9)	5157(6)	4025(6)	42(3)
C(21)	-123(8)	2728(6)	2831(5)	34(3)	C(113)	-417(9)	5400(7)	3991(7)	54(3)
C(22)	564(10)	3134(6)	2902(6)	50(3)	C(114)	-675(11)	5967(7)	3489(7)	62(4)
C(23)	330(12)	3897(7)	2813(8)	73(4)	C(115)	133(9)	6290(7)	3015(7)	55(3)
C(24)	-586(11)	4258(8)	2633(7)	70(4)	C(116)	1176(9)	6051(6)	3045(6)	39(3)
C(25)	-1291(11)	3887(6)	2572(6)	58(3)	C(121)	2797(8)	5219(5)	4461(5)	28(2)
C(26)	-1072(9)	3126(6)	2676(6)	48(3)	C(122)	3473(9)	4714(6)	4818(5)	42(3)
C(31)	1504(8)	648(6)	712(5)	37(3)	C(123)	3510(10)	4821(7)	5472(6)	49(3)
C(32)	1078(9)	698(7)	161(6)	52(3)	C(124)	2861(9)	5420(6)	5784(6)	48(3)
C(33)	1437(10)	159(8)	-293(7)	65(4)	C(125)	2165(9)	5914(6)	5445(6)	40(3)
C(34)	2213(10)	-443(7)	-169(7)	58(4)	C(126)	2143(8)	5819(6)	4790(5)	30(2)
C(35)	2646(9)	-503(7)	386(7)	48(3)	O(1)	1734(9)	7072(7)	1375(6)	94(4)
C(36)	2277(8)	41(6)	837(5)	35(3)	C(200)	697(15)	8093(10)	830(10)	98(6)
C(41)	823(8)	2206(7)	734(6)	38(3)	C(201)	1160(13)	8114(9)	2017(9)	80(5)
C(42)	5(9)	2792(6)	957(6)	36(3)	C(202)	1225(13)	7720(9)	1430(9)	77(5)
C(43)	-49(10)	3469(6)	552(7)	51(3)	O(2)	3661(25)	-822(19)	4881(18)	333(15)
C(44)	712(10)	3585(8)	-80(7)	62(4)	C(204)	5108(24)	-739(16)	3970(15)	186(11)
C(45)	1509(11)	2996(8)	-307(7)	69(4)	C(205)	3373(27)	-312(19)	4066(19)	211(13)
C(46)	1599(9)	2318(7)	91(6)	53(3)	C(206)	3951(30)	-131(21)	4434(20)	221(14)
C(51)	4359(8)	170(5)	1708(5)	26(2)	O(3)	2718(27)	1383(20)	7794(18)	348(16)
C(52)	3934(9)	-405(6)	2170(6)	37(3)	C(207)	2650(34)	2149(24)	7073(24)	273(18)
C(53)	4248(10)	-1103(6)	1990(7)	54(3)	C(208)	1560(32)	2182(21)	7798(21)	239(16)
C(54)	4959(10)	-1262(6)	1351(6)	50(3)	C(209)	2551(38)	2389(25)	7769(25)	277(18)
C(55)	5370(10)	-695(6)	897(7)	51(3)					

^a U(eq) is defined as one-third of the trace of the orthogonalized U_{ij} tensor.

ca. 5×10^{-4} mol dm⁻³ solutions with a Philips 9509 conductimeter. C and H analyses were carried out with a Perkin-Elmer 240C microanalyzer. Mass spectra were recorded on a VG autospec, with the FAB technique, using nitrobenzyl alcohol as matrix. NMR spectra were recorded on a Varian Unity 300 spectrometer and a Bruker AR X 300 spectrometer, in CDCl₃. Chemical shifts are cited relative to SiMe₄ (¹H), 85% H₃PO₄ (external, ³¹P), CFCl₃ (external, ¹⁹F) and BF₃OEt₂ (external, ¹¹B). The yields, melting points, analyses, conductivities and NMR data for the new complexes are listed in Table 1. The yields correspond to the crude products, they can be recrystallized from CH₂-Cl₂/n-hexane.

Materials. [1,2-(PPh₂)₂-1,2-C₂B₁₀H₁₀] was prepared following literature procedures.²³ Complexes [AuClPR₃],²⁴ [Au(PPh₃)(tht)]ClO₄,²⁵ and [Au₂Cl₂(μ-P-P)] [P-P = dppe, dppp]²⁶ were also prepared following literature procedures. Synthesis of [Au{(PPh₂)₂C₂B₁₀H₁₀}(PR₃)]ClO₄ was carried out as previously described by us.¹⁰

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Table 5. Selected Bond Lengths (Å) and Angles (deg) for Compound 7

Au(1)–P(5)	2.293(3)	Au(1)–P(1)	2.365(3)
Au(1)–P(2)	2.429(3)	Au(2)–P(6)	2.288(3)
Au(2)–P(3)	2.374(3)	Au(2)–P(4)	2.443(3)
P(1)–C(11)	1.810(11)	P(1)–C(21)	1.822(11)
P(1)–C(1)	1.835(11)	P(2)–C(31)	1.809(11)
P(2)–C(41)	1.821(12)	P(2)–C(2)	1.828(10)
P(3)–C(91)	1.782(11)	P(3)–C(101)	1.817(11)
P(3)–C(3)	1.818(10)	P(4)–C(121)	1.799(11)
P(4)–C(111)	1.823(11)	P(4)–C(4)	1.826(10)
P(5)–C(51)	1.815(10)	P(5)–C(61)	1.816(10)
P(5)–C(5)	1.835(10)		
P(5)–Au(1)–P(1)	147.36(10)	P(5)–Au(1)–P(2)	126.55(10)
P(1)–Au(1)–P(2)	86.06(10)	P(6)–Au(2)–P(3)	144.80(10)
P(6)–Au(2)–P(4)	129.81(10)	P(3)–Au(2)–P(4)	84.35(9)
C(11)–P(1)–C(21)	104.3(5)	C(11)–P(1)–C(1)	106.6(5)
C(21)–P(1)–C(1)	108.0(5)	C(11)–P(1)–Au(1)	121.4(3)
C(21)–P(1)–Au(1)	109.8(3)	C(1)–P(1)–Au(1)	106.2(3)
C(31)–P(2)–C(41)	103.8(5)	C(31)–P(2)–C(2)	106.4(5)
C(41)–P(2)–C(2)	108.3(5)	C(31)–P(2)–Au(1)	120.9(3)
C(41)–P(2)–Au(1)	112.4(4)	C(2)–P(2)–Au(1)	104.5(3)
C(91)–P(3)–C(101)	103.4(5)	C(91)–P(3)–C(3)	104.1(5)
C(101)–P(3)–C(3)	109.3(4)	C(91)–P(3)–Au(2)	123.3(3)
C(101)–P(3)–Au(2)	110.3(3)	C(3)–P(3)–Au(2)	105.8(3)
C(121)–P(4)–C(111)	104.1(4)	C(121)–P(4)–C(4)	105.6(5)
C(111)–P(4)–C(4)	108.7(4)	C(121)–P(4)–Au(2)	116.8(3)
C(111)–P(4)–Au(2)	116.8(3)	C(4)–P(4)–Au(2)	104.1(3)
C(51)–P(5)–C(61)	105.9(4)	C(51)–P(5)–C(5)	104.4(4)
C(61)–P(5)–C(5)	105.9(5)	C(51)–P(5)–Au(1)	110.1(3)
C(61)–P(5)–Au(1)	112.9(3)	C(5)–P(5)–Au(1)	116.8(3)

Safety Note. *Caution!* Perchlorate salts of metal complexes with organic ligands are potentially explosive. Only small amounts of material should be prepared, and these should be handled with great caution.

Synthesis of [Au{(PPh₂)₂C₂B₉H₁₀}(PR₃)]. (a) To a suspension of [AuCl(PR₃)] [0.1 mmol; 0.049 g (PR₃ = PPh₃), 0.043 g (PR₃ = PPh₂Me), 0.050 g (PR₃ = PPh₂(4-Me-C₆H₄)), 0.053 g (PR₃ = P(4-Me-C₆H₄)₃), 0.058 g (PR₃ = P(4-OMe-C₆H₄)₃)] in ethanol (30 mL) was added [(PPh₂)₂C₂B₉H₁₀] (0.1 mmol, 0.051 g). The mixture was refluxed for 30 min. The complexes, as yellow solids, were collected by filtration and washed with 10 mL of ethanol. (b) These complexes can be also synthesized by refluxing a suspension of [Au{(PPh₂)₂C₂B₉H₁₀}(PR₃)ClO₄] [0.1 mmol; 0.107 g (PR₃ = PPh₃), 0.100 g (PR₃ = PPh₂Me), 0.108 g (PR₃ = PPh₂(4-Me-C₆H₄)), 0.111 g (PR₃ = P(4-Me-C₆H₄)₃), 0.116 g (PR₃ = P(4-OMe-C₆H₄)₃)] in ethanol (30 mL). The complexes were isolated by filtration. ¹¹B NMR data, δ : (1) –7.7 (s, br, 2B), –12.7 [d, 3B, *J*(BH) 103.6 Hz], –16.4 (s, br, 2B), –29.3 (m, 1B), –34.7 [d, 1B, *J*(BH) 133.2 Hz]; (2) –7.9 (s, br, 2B), –13.7 [d, 3B, *J*(BH) 101.2 Hz], –17.8 (s, br, 2B), –29.6 (m, 1B), –34.5 [d, 1B, *J*(BH) 128.4 Hz]; (3) –8.1 (s, br, 2B), –12.8 [d, 3B, *J*(BH) 103.8 Hz], –16.6 (s, br, 2B), –29.8 (m, 1B), –34.2 [d, 1B, *J*(BH) 134.1 Hz]; (5) –7.8 (s, br, 2B), –12.5 (m, 3B), –17.8 (s, br, 2B), –29.7 (m, 1B), –34.3 [d, 1B, *J*(BH) 130.2 Hz].

Synthesis of [Au₂{(PPh₂)₂C₂B₉H₁₀}₂(μ -P-P)]. As previously described, to a suspension of [Au₂Cl₂(μ -P-P)] [0.1 mmol; 0.084 g, (P-P = dppe), 0.087 g, (P-P = dppp)] in ethanol (30 mL) was added [(PPh₂)₂C₂B₉H₁₀] (0.2 mmol, 0.102 g). The mixture was refluxed for an hour and the complexes separated by filtration and washed with 10 mL of ethanol.

Synthesis of [Au₂{(PPh₂)₂C₂B₉H₁₀}₂(PPh₃)₂]. To a two-necked round bottom flask (100 mL) fitted with a dinitrogen inlet/outlet, containing deoxygenated dry THF (40 mL), were added [Au{(PPh₂)₂C₂B₉H₁₀}(PPh₃)] (0.1 mmol, 0.097 g) and excess NaH. The mixture was stirred for an hour and the excess NaH filtered off. The solution was treated with [Au(PPh₃)(tht)]ClO₄ and stirred for another hour. Upon addition of *n*-hexane (15 mL), complex **9** was obtained as a yellow solid.

Crystal Structure Determinations of Complexes 1 and 7. Crystals were mounted in inert oil on glass fibers. Data were collected using monochromated MoK α radiation (λ = 0.710 73 Å). Diffractometer type: Stoe-STADI-4 (1) and Siemens R3 (7), both with Siemens low temperature attachment. Scan type ω/θ (1) and ω (7). Cell constants

Table 6. Details of X-ray Structure Analyses for Compounds 1 and 7

	1·CH ₂ Cl ₂	7·3Me ₂ CO
formula	C ₄₅ H ₄₇ Au ₃ B ₉ Cl ₂ P ₃	C ₈₈ H ₁₀₄ Au ₂ B ₁₈ O ₃ P ₆
<i>M_r</i>	1045.89	1984.04
cryst habit	yellow tablet	yellow tablet
cryst size (mm)	0.4 × 0.4 × 0.2	0.65 × 0.5 × 0.25
space group	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> 1
temp (°C)	–130	–100
cell constants		
<i>a</i> (Å)	17.326(3)	13.432(3)
<i>b</i> (Å)	20.688(3)	18.888(3)
<i>c</i> (Å)	13.442(2)	20.021(3)
α (deg)	90	78.56(2)
β (deg)	104.710(12)	72.02(2)
γ (deg)	120	75.31(2)
<i>V</i> (Å ³)	4660.2	4633.5
<i>Z</i>	4	2
<i>D_x</i> (Mg m ^{–3})	1.491	1.422
<i>F</i> (000)	2080	1988
μ (mm ^{–1})	3.4	3.3
transm factors	0.56–0.98	0.90–0.95
2 θ _{max} (deg)	50	45
no. of reflcns		
measd	9399	12863
indep	8224	11742
<i>R</i> _{int}	0.094	0.050
<i>R</i> (<i>F</i> ² , all reflcns)	0.095	0.112
<i>R</i> (<i>F</i> , <i>F</i> > 4 σ (<i>F</i>))	0.036	0.053
no. of params	508	898
no. of restraints	489	729
<i>S</i>	1.09	1.08
max. Δ/σ	0.001	0.008
max. $\Delta\rho$ (e Å ^{–3})	0.8	1.4

were refined from $\pm\omega$ angles (1) or setting angles (7) of *ca.* 60 reflections in the 2 θ range 20–23° (1) and 10–25° (7). Absorption corrections were applied on the basis of Ψ -scans.

Structures were solved by the heavy atom method and refined anisotropically (except B, H, disordered or solvent C/O) on *F*² using the program SHELXL-93.²⁷

Special Details for 1. The structure is pseudosymmetric, the gold atom lying at *y* = *ca.* 0.5. This causes reflections with *h* + *l* odd to be weak. These reflections were therefore collected again slowly, and are thus the main contributors to *R*_{int}, which is correspondingly high. All the hydrogen atoms of the carborane moiety were clearly identified in difference syntheses. The B–H on the open carborane face were refined freely (although heavily restrained with SADI). Other hydrogens atoms were included using a riding model. The dichloromethane molecule is well-defined.

Special Details for 7. The structure contains three molecules of acetone, of which however only the first is well defined; the other two are distorted and probably severely disordered over two alternative orientations. The phenyl ring C(71)–C(76) is disordered over two positions. The hydrogen atoms of the open face of the carborane were identified with difficulty from difference syntheses and refined as above; their positions should be regarded as tentative. The pattern of H atoms is however the same as in other analogues. Further details are given in Table 6.

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Supporting Information Available: Tables of crystal data, data collection, and solution and refinement parameters, atomic parameters, bond distances and angles, and anisotropic thermal parameters, and hydrogen atom parameters for 1 and 7 and a thermal ellipsoid plot for 1 (22 pages). Ordering information is given on any current masthead page.

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(27) Sheldrick, G. M. *SHELXL-93. A program for Crystal Structure Refinement*; University of Göttingen: Göttingen, Germany, 1993.