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SYNTHESIS AND MOLECULAR STRUCTURE OF (3-(PPH3)-3,3-(NO3)-3,1,2---ETC(U)
AUG 79 Z DEMIDOWICZ, R G TELLER N00014-76-C-0390
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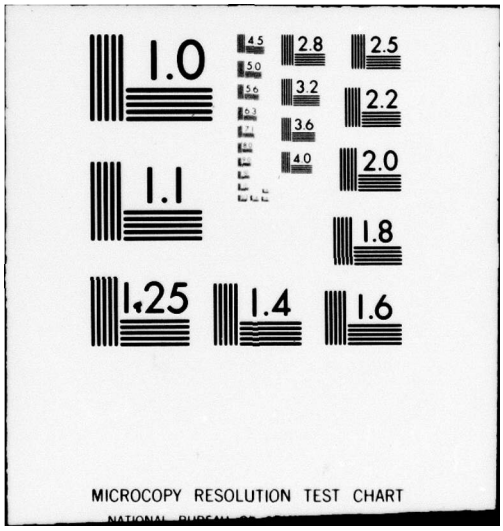
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TR-106

SYNTHESIS AND MOLECULAR STRUCTURE OF [3-(PPh_3)-3,3-(NO_3)-3,1,2-$RhC_2B_9H_{11}$], a Versatile Metallocarborane Reagent.

by

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Synthesis and Molecular Structure of $[3\text{-}(PPh_3)\text{-}3,3\text{-}(NO_3)\text{-}3,1,2\text{-}RhC_2B_9H_{11}]$, a Versatile Metallocarborane Reagent

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SUMMARY

The action of nitric acid or NO_2/N_2O_4 mixture on [3,3-$(PPh_3)_2$-3-H-3,1,2-$RhC_2B_9H_{11}$] affords the nitratorhodacarborane [3-(PPh_3)-3,3-(NO_3)-3,1,2-$RhC_2B_9H_{11}$](I) whose structure has been determined by X-ray crystallography, triclinic, space group P1, a = 12.592(5), b = 16.382(8), c = 17.059(6) Å, $\alpha = 75.74(4)^o$, $\beta = 105.34(3)^o$, $\gamma = 120.05(3)^o$, z = 2. The latter species has been shown to be a useful precursor in the synthesis of other rhodacarborane derivatives.

In continuation of our studies of the chemical properties of metallocarborane complexes[1-4] we have found that treatment of [3,3-$(PPh_3)_2$-3-H-3,1,2-$RhC_2B_9H_{11}$][1] with excess nitric acid in dichloromethane or NO_2/N_2O_4 in benzene, at room temperature, affords an air-stable red complex(I) isolated in good yield (ca. 70%) by column chromatography (silicagel-CH_2Cl_2) and crystallization.

The i.r. spectrum of (I), as a nujol mull, showed absorptions characteristic of terminal B-H bonds and coordinated triphenylphosphine. The $^{31}P\{^1H\}$ nmr spectrum[5] of (I) in $CDCl_3$ showed a doublet centered at +36.1 ppm, J_{Rh-P} = 168.5 Hz. The $^{11}B\{^1H\}$ nmr spectrum,[6] also in $CDCl_3$, showed resonances at -25.7, -9.2, -3.1 and +11.8 of relative intensities 1:2:4:2. Also no signal attributable to a Rh-H group could be detected in the ^1H nmr spectrum of (I).

Microanalytical data for crystalline (I) proved inconclusive apparently because of solvent loss; however the presence of Rh, N, P and B in the ratio 1:1:1:9 was clearly indicated. In a further attempt to elucidate the nature of (I) we noted that (I) reacts with PPh_3 and either H_2 or hydrochloric acid in THF solution to quantitatively generate [3,3-$(PPh_3)_2$-3-X-3,1,2-$RhC_2B_9H_{11}$] (X = H[1] or Cl[7,8] respectively). Further, it has been shown that transition metal nitrato complexes can be obtained by the action of nitric acid on platinum-metal phosphine complexes.[9] Accordingly we tentatively formulated (I) as [3-(PPh_3)-3,3-(NO_3)-3,1,2-$RhC_2B_9H_{11}$] in which a <u>closo</u>-rhodacarborane cage and a bidentate nitrato ligand acting as a three-electron donor towards rhodium are present. This formulation was subsequently confirmed by an X-ray crystallographic study.

<u>Crystal Data</u>

$[P(C_6H_5)_3]NO_3RhC_2B_9H_{11}\cdot 3CH_2Cl_2$ M = 814.4, triclinic, space group P1, a = 12.592(5), b = 16.382(8), c = 17.059(6) Å, α = 75.74(4)°, β = 105.34(3)°,

$\gamma = 120.05(3)^{o}$, $z = 2$, $\mu(MoK_{\alpha}) = 7.29\ cm^{-1}$. Data were collected on a Picker FACS-I four circle diffractometer at room temperature with the crystal sealed in a thin-walled capillary to prevent solvent loss. The structure was solved by standard Patterson and Fourier techniques. Severe disorder in the solvent molecules has prevented a satisfactory refinement of the structure and at present the agreement factor stands at 0.123 (3497 reflections).

The molecule is illustrated in the figure along with some pertinent bond distances and angles. As postulated the complex consists of a rhodium atom bonded to a $C_2B_9H_{11}^{-2}$ anion, triphenylphosphine and nitrato ligands with the closo-$RhC_2B_9H_{11}$ fragment in its usual distorted icosahedral geometry. The nitrato group is bound in an apparently symmetrically bidentate fashion with an average Rh-O bond length of 2.20(1) $\mathring{A}$. This value is 0.1 $\mathring{A}$ larger than various Rh(III)-O bonds in complexes containing carbonato or acetylacetonato ligands[10] but does compare favorably with Rh(III)-O distances in complexes with weakly bound water molecules (2.24(1) and 2.28(1) $\mathring{A}$).[11] The only other metallocarborane with a metal-oxygen bond is [3,3-$(PPh_3)_2$-3-(HSO_4)-3,1,2-$RhC_2B_9H_{11}$] with a Rh-O distance of 2.245(8) $\mathring{A}$.[12]

[3-(PPh_3)-3,3-(NO_3)-3,1,2-$RhC_2B_9H_{11}$](I) has proved to be an extremely useful precursor in the synthesis of previously inaccessible rhodacarborane derivatives. For example (I) reacts with CO gas and hydrochloric acid in THF at room temperature to afford, in 85% yield, air-stable [3-(PPh_3)-3-(CO)-3-Cl-3,1,2-$RhC_2B_9H_{11}$](II) characterized by elemental analysis, i.r. and n.m.r. spectroscopy.[13] Previous attempts in this laboratory to prepare (II) by direct reaction of CO with the previously documented [3,3-$(PPh_3)_2$-3-Cl-3,1,2-$RhC_2B_9H_{11}$][7,8] have failed.

Complex (I) also reacts with PPh_3 in diethylether at room temperature to give, in 81% yield, orange [3,3-$(PPh_3)_2$-3-(NO_3)-3,1,2-$RhC_2B_9H_{11}$](III) charac-

terized by elemental analysis[14] and i.r. and n.m.r. spectroscopy. In particular the $^{31}P\{^{1}H\}$ n.m.r. spectrum of (III) at room temperature in $CDCl_3$ shows, in addition to a doublet centered at +28.0 ppm (J_{Rh-P} = 133.5 Hz) attributed to (III), resonances characteristic of uncoordinated triphenylphosphine and complex (I). Thus (III) apparently exhibits behavior in solution similar to that reported for $[3,3\text{-}(PPh_3)_2\text{-}3\text{-}(HSO_4)\text{-}3,1,2\text{-}RhC_2B_9H_{11}]$.[12]

Finally we note that (II) reacts with PPh_3 in THF at room temperature to afford $[3,3\text{-}(PPh_3)_2\text{-}3\text{-}Cl\text{-}3,1,2\text{-}RhC_2B_9H_{11}]$[7,8] in 81% yield and that (III) reacts with either H_2 or hydrochloric acid, also in THF at room temperature, to give $[3,3\text{-}(PPh_3)_2\text{-}3\text{-}X\text{-}3,1,2\text{-}Rh(C_2B_9H_{11}]$ (X = H[1] in 79% yield, or X = Cl[7,8] in 90% yield respectively).

Acknowledgments

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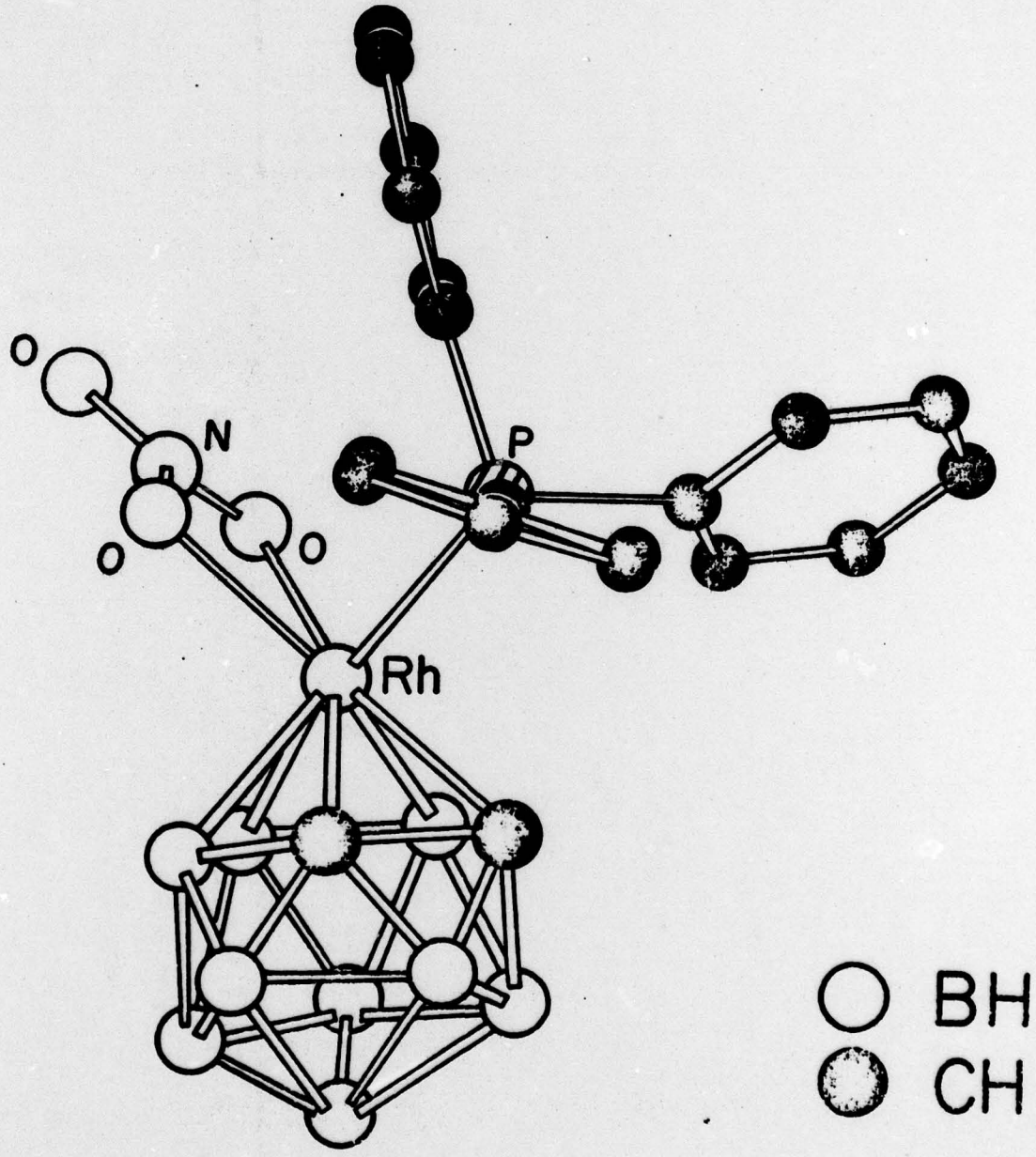
12. W. C. Kalb, R. G. Teller and M. F. Hawthorne; J. Am. Chem. Soc., in press.
13. Analysis: Found; C = 45.8, H = 4.6, Cl = 6.7, B = 17.6, P = 5.6, Rh = 18.2%. $C_{21}H_{26}B_9ClOPRh$ requires C = 45.0, H = 4.7, Cl = 6.3, B = 17.3, P = 5.5, Rh = 18.3%. i.r. spectrum, as a nujol mull, showed ν(CO) at ca. 2062 cm^{-1}. $^{31}P\{^1H\}$ nmr spectrum in $CDCl_3$ showed a doublet centered at +39.6 ppm, J_{Rh-P} = 107.4 Hz.
14. Analysis: Found; C = 55.6, H = 5.3, N = 1.6, P = 7.4, B = 11.6, Rh = 12.3%. $C_{38}H_{41}B_9NO_3P_2Rh$ requires C = 55.5, H = 5.0, N = 1.7, P = 7.5, B = 11.8, Rh = 12.5%.

Figure 1 A molecular plot of (I). Some distances (averaged from two unique molecules) follow: Rh-O 2.20(1) Å, Rh-P 2.38(1) Å, N-O 1.22(1) Å, Rh-C 2.14(2) Å, Rh-B 2.14(3) Å, C-C 1.88(7) Å, C-B 1.80(2) Å, B-B 1.82(2) Å.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1. Positional (in Fractional Coordinates) and Thermal[a] Parameters for (the Non-group Atoms of) 3-PPh_3-3,3-NO_3-3,1,2-$RhC_2B_9H_{11}$

Atom	10^4x	10^4y	10^4z	$B(A^{o2})$
Rh[b]	0	0	0	c
C(1)	-521(34)	744(26)	589(22)	2.7(8)
C(2)	-587(34)	-417(24)	1136(21)	2.5(8)
B(4)	-1639(35)	-1227(24)	422(21)	1.8(7)
B(5)	-2305(43)	-1269(31)	1203(26)	2.6(10)
B(6)	1576(38)	22(27)	1283(23)	1.9(8)
B(7)	-2017(34)	-692(24)	-425(20)	0.9(7)
B(8)	-3217(40)	-1393(28)	169(24)	2.4(9)
B(9)	-3244(47)	-677(34)	717(29)	3.6(11)
B(10)	-1974(65)	704(49)	170(41)	5.5(17)
B(11)	-1296(37)	477(26)	-456(22)	1.3(8)
B(12)	-2886(46)	-356(32)	-340(27)	3.0(10)
N	940(34)	-807(26)	-608(23)	4.5(8)
O(1)	369(25)	-220(18)	-1097(15)	3.4(6)
O(2)	922(21)	-875(16)	119(14)	2.3(5)
O(3)	1253(25)	-1151(19)	-994(17)	4.2(6)
P	1864(17)	1441(13)	-26(11)	c
Rh'	3908(8)	5631(5)	5620(4)	c
C(1)'	2688(37)	5909(27)	6052(22)	3.0(9)
C(2)'	3072(38)	6557(28)	4999(24)	3.7(9)
B(4)'	2803(40)	5608(29)	4475(24)	2.4(9)
B(5)'	1672(42)	6073(31)	4281(26)	2.8(9)
B(6)'	1675(39)	6300(28)	5239(24)	2.2(9)
B(7)'	2197(40)	4545(29)	5111(25)	2.2(9)
B(8)'	1150(44)	4761(32)	4336(27)	3.4(10)
B(9)'	274(48)	5239(42)	4811(33)	5.4(15)
B(10)'	996(45)	5112(33)	5940(28)	3.1(10)
B(11)'	1990(64)	4742(47)	6071(41)	4.0(17)
B(12)'	656(62)	4155(46)	5335(38)	7.0(15)
N'	5047(31)	4646(23)	6334(22)	3.3(8)
O(1)'	4823(23)	4766(18)	5657(16)	3.0(6)
O(2)'	4822(23)	5096(17)	6769(14)	3.0(5)
O(3)'	5617(26)	4230(19)	6784(16)	4.5(6)
P'	5897(18)	7044(15)	5690(11)	c

2. Anisotropic Temperature Factors[d]

Atom	$10^4\beta_{11}$	$10^4\beta_{22}$	$10^5\beta_{33}$	$10^5\beta_{12}$	$10^5\beta_{13}$	$10^5\beta_{23}$
Rh	126(7)	70(4)	48(2)	44(4)	16(3)	-8(2)
Rh'	114(6)	67(4)	63(3)	40(4)	9(3)	-4(2)
P	113(19)	68(10)	46(8)	44(11)	4(8)	-2(6)
P'	140(23)	106(14)	49(8)	33(14)	41(10)	7(8)

3. Rigid-Group Parameters[e]

Group	x	y	z	Phi	Theta	Rho	B(Å^2)
Phenyl 1	.309(2)	.132(2)	-.037(2)	-2.98(2)	-2.19(1)	-2.82(3)	3.7(4)
Phenyl 2	.274(2)	.194(2)	.090(1)	2.86(2)	-2.57(1)	1.66(2)	2.3(3)
Phenyl 3	.150(2)	.231(2)	-.079(1)	0.43(2)	-2.36(1)	-1.76(2)	3.1(3)
Phenyl 4	.719(2)	.687(1)	.601(2)	-2.98(2)	2.19(1)	2.73(2)	3.4(4)
Phenyl 5	.623(2)	.791(2)	.479(1)	2.85(2)	2.53(1)	-1.58(2)	3.8(4)
Phenyl 6	.580(2)	.764(2)	.649(1)	0.46(2)	2.40(1)	1.80(2)	3.0(4)

a) Standard deviations in the least significant figures are given in parentheses. All phenyl moieties were refined as rigid groups with C-C 1.39 and C-H 1.00 Å.

b) The position of the atom was fixed in the least squares process.

c) Anisotropic thermal parameter.

d) The form of the anisotropic thermal ellipsoidal is: $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}\ell + 2\beta_{12}hk + 2\beta_{13}h\ell + 2\beta_{23}k\ell)]$.

e) x, y, and z are the coordinates of the origin of the rigid group and phi, theta, and rho the rotation angles.

Selected Distances (in Å) and Angles (in degrees) in 3-(PPh_3)-3,3-(NO_3)-3,1,2-$RhC_2B_9H_{11}$·$3CH_2Cl_2$

1. Distances

	Molecule 1	Molecule 2
Rh-C(1)	2.16	2.14
Rh-C(2)	2.09	2.18
Rh-B(4)	2.18	2.07
Rh-B(7)	2.19	2.12
Rh-B(11)	2.05	2.23
Rh-N(1)	2.67	2.58
Rh-O(1)	2.19	2.21
Rh-O(3)	2.20	2.22
Rh-P	2.35	2.41
P-C(11)	1.90	1.73
P-C(21)	1.79	1.79
P-C(31)	1.83	1.91
N-O(1)	1.26	1.19
N-O(2)	1.20	1.25
N-O(3)	1.22	1.21
C(1)-C(2)	1.87	1.90
C(1)-B(4)	1.78	1.82
C(1)-B(5)	1.92	1.78
C(1)-B(6)	1.81	1.73
C(2)-B(6)	1.77	1.85
C(2)-B(10)	1.76	1.84
C(2)-B(11)	1.83	1.65
B(4)-B(5)	1.72	1.85
B(4)-B(7)	1.58	1.72
B(4)-B(8)	1.81	1.82
B(5)-B(6)	1.86	1.76
B(5)-B(8)	1.83	1.89
B(5)-B(9)	1.81	1.90

	Molecule 1 (cont'd)	Molecule 2 (cont'd)
B(6)-B(9)	1.93	1.88
B(6)-B(10)	2.03	1.91
B(7)-B(8)	1.77	1.71
B(7)-B(11)	1.65	1.85
B(7)-B(12)	1.50	1.83
B(8)-B(9)	1.69	2.03
B(8)-B(12)	1.60	1.84
B(9)-B(10)	2.16	1.91
B(9)-B(12)	1.86	2.01
B(10)-B(11)	1.74	1.60
B(10)-B(12)	1.82	1.90
B(11)-B(12)	1.80	1.78

2. Angles

P-Rh-O(1)	87	90
P-Rh-O(3)	94	84
P-Rh-C(1)	111	107
P-Rh-C(2)	83	87
P-Rh-B(4)	146	112
P-Rh-B(7)	161	160
P-Rh-B(11)	101	147
O(1)-Rh-C(1)	150	152
O(1)-Rh-C(2)	155	153
O(1)-Rh-B(4)	111	107
O(1)-Rh-B(7)	92	89
O(1)-Rh-B(11)	104	113
O(3)-Rh-C(1)	148	102
O(3)-Rh-C(2)	101	148
O(3)-Rh-B(4)	92	158
O(3)-Rh-B(7)	115	112
O(3)-Rh-B(11)	157	94
O(1)-Rh-O(3)	60	56
O(1)-N-O(2)	115	125
O(1)-N-O(3)	110	118
O(2)-N-O(3)	134	115

	Molecule 1 (cont'd)	Molecule 2 (cont'd)
Rh-P-C(11)	112	115
Rh-P-C(21)	120	118
Rh-P-C(31)	107	102
C(11)-P-C(27)	100	107
C(11)-P-C(31)	108	109
C(21)-P-C(31)	110	105

a) Estimated standard deviations in bond lengths: Rh-P 0.01, Rh-O 0.02, Rh-N 0.03, Rh-C 0.03, Rh-B 0.03, N-O 0.05, C-C 0.07, C-B 0.08, B-B 0.08 Å; and in bond angles: P-Rh-O, P-Rh-C, P-Rh-B 1°, O-Rh-C, O-Rh-B 2°; O-N-O 3°.

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The action of nitric acid or NO_2/N_2O_4 mixture on [3,3-(PPh_3)2-3-H-3,1,2-$RhC_2B_9H_{11}$] affords the nitratorhodacarborane [3-(PPh_3)-3,3-(NO_3)-3,1,2-$RhC_2B_9H_{11}$](I) whose structure has been determined by X-ray crystallography, triclinic, space group P1, a = 12.592(5), b = 16.382(8), c = 17.059(6) Å, α = 75.74(4)°, β = 105.34(3)°, γ = 120.05(3)°, z = 2. The latter species has been shown to be a useful precursor in the synthesis of other rhodacarborane derivatives.

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