New Carbide Clusters in the Cobalt Sub-group. Part 7.[1–6] Preparation and Structural Characterization of Carbido-hexa-μ-carbonyl-hepta-carbonyl-*polyhedro*-hexarhodate(2−) as its Bis(tetraphenylphosphonium) Salt

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The anion $[Rh_6C(CO)_{13}]^{2-}$ has been prepared by refluxing a solution of $K_2[Rh_6C(CO)_{15}]\cdot 6CH_3O(CH_2)_4OCH_3$ in Pr^{i}OH at 100 °C for 2 h under nitrogen and pumping off the evolved CO. The new anion has been characterized by a crystallographic study on the salt $[PPh_4]_2[Rh_6C(CO)_{13}]$, which crystallizes in the monoclinic space group $P2_1/c$ with cell constants a = 24.232(8), b = 12.587(5), c = 19.754(7) Å, and β = 92.0(5)°. The structure has been solved by direct methods and refined by least-squares calculations to R = 0.055 for 4 350 significant diffraction intensities. The anion contains a distorted octahedron of rhodium atoms with the carbide ion in the centre [Rh–Rh 2.733—3.188(2), Rh–C(carbide) 1.99—2.15(1) Å]. Six of the carbonyl groups are edge bridging, spanning consecutive edges of the three octahedron equators, and seven are terminal. The idealized molecular symmetry is C_s-m. A strong dependence of the metal–metal distances on the ligand geometry has been noticed, and the driving force of the octahedron deformation is very likely the steric requirement of the interstitial carbon.

THE prismatic dianion [1] $[Rh_6C(CO)_{15}]^{2-}$ has proved to be a prolific source of more complicated cluster complexes. When treated with an oxidizing agent such as the iron(III) cation the species $[Rh_8C(CO)_{19}]$,[2] $[Rh_{15}C_2(CO)_{28}]^-$,[3] and $[Rh_{12}C_2(CO)_{25}]^-$ [4] have been isolated under different experimental conditions.

We are now investigating the effect of heat on this dianion and, by moderate thermal treatment in an inert atmosphere, we have noted reversible formation of the new anion $[Rh_6C(CO)_{13}]^{2-}$ according to reaction (1).

$$K_2[Rh_6C(CO)_{15}] \underset{25\ °C}{\overset{Pr^iOH,\ N_2,\ reflux,\ 2\ h}{\rightleftharpoons}} K_2[Rh_6C(CO)_{13}] + 2CO \quad (1)$$

The new species has 86 valence electrons and was expected to contain an octahedron of metal atoms. The unprecedented number of outer ligands, on the other hand, left some doubt about its structure. In fact, while the carbonyl clusters with 14 ligands, such as $[Co_6(CO)_{14}]^{4-}$ (ref. 7) and $[Co_4Ni_2(CO)_{14}]^{2-}$,[8] contain almost regular octahedra of metal atoms, the known examples with 12 ligands, $[Ni_6(CO)_{12}]^{2-}$ (ref. 9) and $[Pt_6(CO)_{12}]^{2-}$,[10] have shown unexpected structural changes.

The structure of the salt $[PPh_4]_2[Rh_6C(CO)_{13}]$, determined by single-crystal X-ray methods, has confirmed that the number of outer ligands has remarkable effects on the metal–metal interactions. Another interesting outcome of this work is the appreciation of how the carbonyl ligands, thanks to their high bonding flexibility, succeed in equalizing the number of electrons on all the metal atoms, even in the rather unique case of 13 ligands bonded to a potentially highly symmetric metal-atom polyhedron.

EXPERIMENTAL

Preparation of $[PPh_4]_2[Rh_6C(CO)_{13}]$.—All the operations were carried out with the rigorous exclusion of air. The compound $K_2[Rh_6C(CO)_{15}]\cdot CH_3O(CH_2)_4OCH_3$ (0.5 g) [11] {obtained by dissolution of $K_2[Rh_6C(CO)_{15}]$ [1,9] in acetone, addition of diethylene glycol dimethyl ether, concentration *in vacuo* until the acetone is eliminated, filtration, washing with n-hexane, and vacuum drying} was dissolved in Pr^{i}OH (35 cm^3) and refluxed under nitrogen on an oil bath at 100 °C for 2 h, pumping off the evolved CO every 30 min. The originally yellow solution turned red. After brief degassing *in vacuo* the solution was left aside overnight under nitrogen to allow the separation of some less soluble by-products presently under investigation. These by-products were filtered off leaving in solution the pure potassium salt from which the $[PPh_4]^+$ salt could be obtained by slow addition of a solution of $[PPh_4]Br$ in Pr^{i}OH, filtration, washing with Pr^{i}OH, and vacuum drying (Found: C, 44.2; H, 2.45. Calc. for $C_{62}H_{40}O_{13}P_2Rh_6$: C, 44.55; H, 2.40). Crystals for the X-ray analysis were obtained by recrystallization from acetone–Pr^{i}OH with the slow diffusion technique. The i.r. spectrum in thf solution showed bands at 1 968vs cm^{-1} in the terminal carbonyl stretching region, and at 1 825m, 1 800w(sh) cm^{-1} in the bridging carbonyl region.

The anion reacts with CO to give back the prismatic anion $[Rh_6C(CO)_{15}]^{2-}$ quantitatively and, by thermal rearrangement, gives different brown products, depending on temperature and time. More details on these reactions, on reactions with electrophiles, and on ^{13}C and ^{103}Rh n.m.r. spectra will be reported later.[12]

Crystal Data.—$C_{62}H_{40}O_{13}P_2Rh_6$, M = 1 672.3, Monoclinic, a = 24.232(8), b = 12.587(5), c = 19.754(7) Å, β = 92.0(5)°, U = 6 021.5 Å^3, D_m = 1.86, Z = 4, D_c = 1.84 g cm^{-3}, $F(000)$ = 3 264, space group $P2_1/c$ (no. 14), Mo-K_α radiation, λ = 0.710 7 Å, μ(Mo-K_α) = 15.6 cm^{-1}.

Intensity Measurements.—A crystal fragment with average dimensions 0.14 × 0.27 × 0.30 mm was mounted on a Siemens diffractometer. Diffraction intensities in two octants of the reciprocal lattice were measured in the range $3 < \theta < 24°$ by the ω-scan method, with scan range 2° and speed 2° min^{-1}. The background was measured at both sides of the reflections for a total time equal to the peak scanning time. 11 233 Diffraction intensities were collected, 4 350 of which $[F_o > 5\ \sigma(F_o)]$ were used for the computations. The integrated intensities were reduced to F_o

TABLE 1

Final positional parameters ($\times 10^4$) with estimated standard deviations in parentheses

Atom	X	Y	Z
Rh(1)	2 481(1)	4 862(1)	2 470(1)
Rh(2)	3 387(1)	4 708(1)	3 350(1)
Rh(3)	2 790(1)	5 344(1)	4 440(1)
Rh(4)	1 681(1)	5 410(1)	3 592(1)
Rh(5)	2 576(1)	6 702(1)	3 353(1)
Rh(6)	2 312(1)	3 613(1)	3 686(1)
C(0)	2 536(5)	5 131(11)	3 479(7)
C(1)	2 460(10)	6 036(21)	1 857(13)
C(2)	2 152(9)	3 768(20)	1 954(11)
C(3)	4 104(7)	4 352(15)	3 244(9)
C(4)	2 808(7)	5 679(14)	5 361(9)
C(5)	963(8)	5 580(16)	3 790(9)
C(6)	2 746(7)	7 970(15)	2 912(9)
C(7)	2 336(9)	2 223(20)	3 414(11)
C(8)	3 277(7)	4 477(15)	2 344(9)
C(9)	3 587(6)	4 942(13)	4 368(8)
C(10)	2 896(6)	7 045(14)	4 228(8)
C(11)	2 400(8)	3 534(15)	4 655(10)
C(12)	1 754(7)	6 960(15)	3 467(9)
C(13)	1 495(8)	3 849(16)	3 630(9)
O(1)	2 450(8)	6 632(15)	1 461(9)
O(2)	1 917(8)	3 151(15)	1 628(9)
O(3)	4 564(7)	4 085(13)	3 208(8)
O(4)	2 817(5)	5 942(11)	5 911(7)
O(5)	508(7)	5 584(13)	3 932(8)
O(6)	2 877(6)	8 750(14)	2 648(8)
O(7)	2 276(8)	1 382(18)	3 187(10)
O(8)	3 547(6)	4 250(11)	1 877(7)
O(9)	3 973(6)	4 917(11)	4 734(7)
O(10)	3 084(5)	7 730(11)	4 575(6)
O(11)	2 319(5)	3 162(10)	5 176(7)
O(12)	1 490(6)	7 781(12)	3 487(7)
O(13)	1 106(7)	3 238(13)	3 621(8)
P(1)	5 547(2)	5 511(3)	1 400(2)
P(2)	9 269(2)	1 846(4)	4 288(2)
C(14)	5 967(4)	5 874(8)	714(4)
C(15)	6 099(4)	6 937(8)	602(4)
C(16)	6 440(4)	7 210(8)	74(4)
C(17)	6 648(4)	6 420(8)	−341(4)
C(18)	6 516(4)	5 358(8)	−229(4)
C(19)	6 176(4)	5 084(8)	299(4)
C(20)	5 370(4)	4 122(6)	1 337(5)
C(21)	5 784(4)	3 356(6)	1 411(5)
C(22)	5 655(4)	2 282(6)	1 330(5)
C(23)	5 112(4)	1 974(6)	1 175(5)
C(24)	4 698(4)	2 740(6)	1 101(5)
C(25)	4 827(4)	3 814(6)	1 182(5)
C(26)	5 918(4)	5 694(9)	2 185(4)
C(27)	5 774(4)	5 082(9)	2 740(4)
C(28)	6 048(4)	5 227(9)	3 366(4)
C(29)	6 467(4)	5 983(9)	3 436(4)
C(30)	6 611(4)	6 595(9)	2 881(4)
C(31)	6 336(4)	6 451(9)	2 255(4)
C(32)	4 936(3)	6 291(8)	1 354(5)
C(33)	4 622(3)	6 385(8)	1 929(5)
C(34)	4 116(3)	6 913(8)	1 891(5)
C(35)	3 924(3)	7 348(8)	1 277(5)
C(36)	4 238(3)	7 255(8)	702(5)
C(37)	4 744(3)	6 726(8)	740(5)
C(38)	8 802(4)	951(9)	4 657(6)
C(39)	8 949(4)	−118(9)	4 714(6)
C(40)	8 588(4)	−840(9)	5 001(6)
C(41)	8 081(4)	−493(9)	5 232(6)
C(42)	7 935(4)	576(9)	5 175(6)
C(43)	8 295(4)	1 298(9)	4 887(6)
C(44)	8 927(5)	2 963(8)	3 943(6)
C(45)	8 822(5)	3 036(8)	3 246(6)
C(46)	8 544(5)	3 918(8)	2 976(6)
C(47)	8 373(5)	4 728(8)	3 402(6)
C(48)	8 478(5)	4 655(8)	4 099(6)
C(49)	8 756(5)	3 773(8)	4 370(6)
C(50)	9 745(5)	2 347(10)	4 914(6)
C(51)	10 106(5)	3 160(10)	4 746(6)
C(52)	10 515(5)	3 493(10)	5 214(6)
C(53)	10 561(5)	3 013(10)	5 850(6)
C(54)	10 200(5)	2 200(10)	6 018(6)
C(55)	9 791(5)	1 867(10)	5 550(6)
C(56)	9 622(5)	1 138(10)	3 647(6)
C(57)	10 194(5)	1 202(10)	3 604(6)
C(58)	10 461(5)	630(10)	3 106(6)
C(59)	10 156(5)	−5(10)	2 652(6)
C(60)	9 583(5)	−69(10)	2 696(6)
C(61)	9 316(5)	502(10)	3 193(6)

TABLE 2

Relevant bond distances (Å) and angles (°) with estimated standard deviations in parentheses

Rh(1)–Rh(2)	2.760(2)	C(2)–O(2)	1.15(2)
Rh(1)–Rh(4)	3.072(2)	C(3)–O(3)	1.17(2)
Rh(1)–Rh(5)	2.904(2)	C(4)–O(4)	1.14(2)
Rh(1)–Rh(6)	2.911(2)	C(5)–O(5)	1.15(2)
Rh(2)–Rh(3)	2.755(2)	C(6)–O(6)	1.16(2)
Rh(2)–Rh(5)	3.188(2)	C(7)–O(7)	1.16(3)
Rh(2)–Rh(6)	3.043(2)	C(8)–O(8)	1.18(2)
Rh(3)–Rh(4)	3.118(2)	C(9)–O(9)	1.16(2)
Rh(3)–Rh(5)	2.779(2)	C(10)–O(10)	1.18(2)
Rh(3)–Rh(6)	2.862(2)	C(11)–O(11)	1.15(2)
Rh(4)–Rh(5)	2.763(2)	C(12)–O(12)	1.22(2)
Rh(4)–Rh(6)	2.733(2)	C(13)–O(13)	1.22(2)
C(O)–Rh(1)	2.02(1)	P(1)–C(14)	1.78(1)
C(O)–Rh(2)	2.15(1)	P(1)–C(20)	1.80(1)
C(O)–Rh(3)	1.99(1)	P(1)–C(26)	1.78(1)
C(O)–Rh(4)	2.12(1)	P(1)–C(32)	1.78(1)
C(O)–Rh(5)	1.99(1)	P(2)–C(38)	1.77(1)
C(O)–Rh(6)	2.03(1)	P(2)–C(44)	1.76(1)
C(1)–Rh(1)	1.91(3)	P(2)–C(50)	1.78(1)
C(2)–Rh(1)	1.87(2)	P(2)–C(56)	1.79(1)
C(3)–Rh(2)	1.81(2)	Rh(1)–C(1)–O(1)	173(2)
C(4)–Rh(3)	1.87(2)	Rh(1)–C(2)–O(2)	175(2)
C(5)–Rh(4)	1.81(2)	Rh(2)–C(3)–O(3)	176(2)
C(6)–Rh(5)	1.87(2)	Rh(3)–C(4)–O(4)	176(2)
C(7)–Rh(6)	1.83(2)	Rh(4)–C(5)–O(5)	173(2)
C(8)–Rh(1)	2.01(2)	Rh(5)–C(6)–O(6)	177(2)
C(8)–Rh(2)	2.02(2)	Rh(6)–C(7)–O(7)	169(2)
C(9)–Rh(2)	2.07(1)	Rh(1)–C(8)–O(8)	135(1)
C(9)–Rh(3)	2.01(1)	Rh(2)–C(8)–O(8)	138(1)
C(10)–Rh(3)	2.20(2)	Rh(2)–C(9)–O(9)	139(1)
C(10)–Rh(5)	1.92(2)	Rh(3)–C(9)–O(9)	136(1)
C(11)–Rh(3)	2.51(2)	Rh(3)–C(10)–O(10)	130(1)
C(11)–Rh(6)	1.92(2)	Rh(5)–C(10)–O(10)	145(1)
C(12)–Rh(4)	1.98(2)	Rh(3)–C(11)–O(11)	127(1)
C(12)–Rh(5)	2.04(2)	Rh(6)–C(11)–O(11)	153(2)
C(13)–Rh(4)	2.02(2)	Rh(4)–C(12)–O(12)	142(1)
C(13)–Rh(6)	2.00(2)	Rh(5)–C(12)–O(12)	131(1)
C(1)–O(1)	1.08(3)	Rh(4)–C(13)–O(13)	142(2)
		Rh(6)–C(13)–O(13)	132(2)

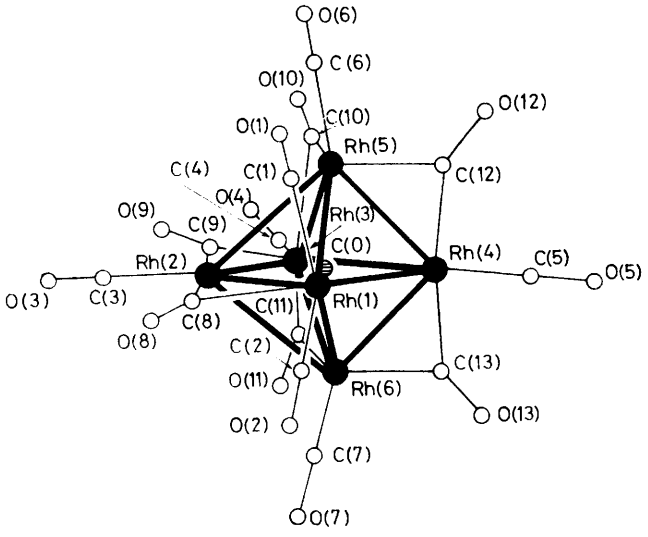
values by correction for Lorentz and polarization effects; the experimental correction for absorption was applied.

The structure was solved using the SHELX package of crystallographic programs.[13] The heavy atoms were located by direct methods and a difference-Fourier map revealed all the non-hydrogen atoms. The structure model, refined by full-matrix least squares, comprised anisotropic thermal treatment of the metal and phosphorus atoms and rigid geometry for the phenyl carbons (C–C 1.395 Å, C–C–C 120°). The hydrogen atoms were omitted in order to save computing time. The agreement indices were R 0.055 and R' 0.064. A final difference-Fourier map showed residual peaks lower than 1 e Å^{-3} in the vicinity of the oxygen atoms. The atomic co-ordinates are reported in Table 1, bond distances and relevant angles are listed in Table 2. Thermal parameters and structure factors are reported in Supplementary Publication No. SUP 22938 (29 pp.).*

* For details see Notices to Authors No. 7, *J. Chem. Soc., Dalton Trans.*, 1979, Index issue.

RESULTS AND DISCUSSION

The structure consists of discrete $[Rh_6C(CO)_{13}]^{2-}$ anions and $[PPh_4]^+$ cations. The anion contains the expected octahedron of rhodium atoms although somewhat distorted. The cluster cavity is occupied by the carbide ion and there are seven terminal and six edge-bridging carbonyl ligands. The bridging ligands span two consecutive edges of all three octahedron equators, as shown in the Figure. In this way four metal atoms are two-connected to the bridging ligands, one is three-connected [Rh(3)], and one mono-connected [Rh(1)]. Such unbalanced distribution of the bridging ligands is compensated by co-ordination of two terminal ligands to Rh(1) and making two out of the three bridging interactions involving Rh(3) unsymmetrical, with longer



The anion $[Rh_6C(CO)_{13}]^{2-}$ showing the atom labelling

distances from that atom. That seems to be the best way of coping with the problem of an even-electron distribution among the anion atoms. The anion is actually asymmetric but the C_s-m symmetry is a useful idealization. Very probably this solid-state structure is an instant stop image taken out of a fluxional behaviour in solution.

The metal–metal distances in this anion show strong dependence on the ligand geometry. Even the smallest deviation of the octahedron edges from the idealized symmetry can be rationalized in terms of non-equivalence of the metal–ligand interactions. The distances, average value 2.91 Å, fall in two broad sets, bridged and unbridged, with mean lengths 2.78 and 3.04 Å respectively. In either group one can tell apart two subsets. Among the six bridged edges those bearing symmetric ligands (four) are shorter [2.75(1) Å] than those bearing unsymmetric bridges [2.83(1) Å]. Moreover, as the degree of asymmetry happens to be different, the edge corresponding to the less asymmetric ligand [Rh(3)–Rh(5)] is shorter than the other [Rh(3)–Rh(6)], 2.789 against 2.862(2) Å. Among the unbridged edges two shorter and four longer values are found [2.91 and 3.11(1) Å] but, on inspecting the molecular stereochemistry, one finds that the shorter edges are in an eclipsed configuration with the two terminal ligands bonded to Rh(1) [C(1)–O(1) and C(2)–O(2)]. The Rh · · · C contacts of these ligands with Rh(5) and Rh(6) [3.1 and 3.4(1) Å respectively] are shorter than the sum of the van der Waals radii, and the shortening can be attributed to weak bonding interactions. The attractive nature of these interactions is demonstrated by the C(1) · · · C(2) contact and C(1)–Rh(1)–C(2) angle [2.95(1) Å and 103(1)° respectively], which are larger than those found in $[Rh_6(CO)_{15}I]^-$.[14] In the latter anion the average values for five pairs of terminal ligands are 2.71 Å and 94°, respectively. Once again, there being two rhodium–carbonyl contacts of different length, a tiny but significant effect on the Rh–Rh distances can be noticed; Rh(1)–Rh(5), corresponding to the shorter contact, is 2.904(2) Å long, and Rh(1)–Rh(6), corresponding to the longer contact, is 2.911(2) Å long. The non-equivalence of these contacts is related to the non-equivalence of the unsymmetrical bridging groups, both being a consequence of unsymmetrical packing forces.

The remaining unbridged edges (four) exhibit values among the longest found so far [average 3.10(10) Å]. On comparing the last figure with the mean Rh–Rh bond lengths in the non-carbido-octahedral clusters $[Rh_6(CO)_{16}]$ 2.78 [15] and $[Rh_6(CO)_{15}I]^-$ 2.75 Å,[14] one can infer that the interstitial carbon has something to do with the weakening of the rhodium–rhodium interactions because the octahedral cavity is a tight one for the neutral carbon atom. The apparent radius of the carbide ion is 0.60 Å, equal to the value found in $[Rh_{15}C_2(CO)_{25}]^-$ (ref. 3) and significantly smaller than that found in the prismatic cavity of $[Rh_6C(CO)_{15}]^{2-}$, 0.74 Å.[1] The trend is confirmed by what was found in $[Os_{10}C(CO)_{24}]^{2-}$ in which the carbon atom is an octahedral cavity.[16] The octahedral edges are 2.88 Å long and the others 2.79 Å. This is a particularly significant test because there are no disturbing effects of bridging ligands, all the CO groups being terminal.* It is interesting to note that the enlargement of the cavity is obtained at the expense of the unbridged edges, while the bridged ones show no appreciable effect, the net lengthening being 0.37 Å. That means that the bridging-carbonyl–metal interactions add a notable contribution to the metal–metal bonds. Similar effects had already been noticed in the anions $[Ni_6(CO)_{12}]^{2-}$ (ref. 9) and $[Pt_6(CO)_{12}]^{2-}$ (ref. 10) in which six bridged edges span two triangular faces with normal edges (2.38 and 2.77 Å respectively), while the remaining edges are elongated (2.77 and 3.04 Å respectively).

After looking at the ligand stereogeometry of this anion one wonders why a more regular allocation, among the various ones possible, has not been adopted. The

* In this case one should reconsider the $[Co_6C(CO)_{14}]^-$ anion,[6] in which the octahedron of cobalt atoms was swelled up and distorted by an extra electron occupying an antibonding cluster orbital. Very probably that orbital was stabilized by a synergic mechanism because it allowed a widening of the cavity and, consequently, a better settlement of the carbide ion.

answer is that all of them would imply an unfavourable charge localization, in order to assign the same number of electrons to all the metal atoms. Therefore the aim of an even distribution of electrons is achieved at the expense of the steric regularity, taking advantage of the CO flexibility, a ligand that can choose its mode of bonding in a continuous range of geometries.

The authors acknowledge the support of the National Research Council of Italy (CNR).

[0/1187 *Received*, 28*th July*, 1980]

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