New Carbide Clusters in the Cobalt Sub-group. Part 14.‡ Synthesis and Structural Characterization of the anion $[Co_{13}C_2(CO)_{24}]^{3-}$ as its Benzyltrimethylammonium Salt§

Vincenzo G. Albano* and Dario Braga
Istituto Chimico 'G. Ciamician' dell'Universita', Via F. Selmi 2, 40126 Bologna, Italy
Alessandro Fumagalli and Secondo Martinengo†
Centro C.N.R. di Studio per la Sintesi e la Struttura dei Composti dei Metalli di Transizione nei bassi Stati di Ossidazione c/o Dipartimento di Chimica Inorganica e Metallorganica, Via G. Venezian 21, 20133 Milano, Italy

The anion $[Co_{13}C_2(CO)_{24}]^{3-}$ has been obtained by oxidation of the corresponding paramagnetic tetra-anion with iodine in MeCN. Its molecular structure has been determined by single-crystal *X*-ray diffractometry, showing that the two anions are isostructural. The metal-atom polyhedron contains two trigonal-prismatic cavities in which two interstitial carbon atoms are accommodated. Average bond distances are Co–Co 2.575 Å and Co–C(carbide) 1.97 Å. The effect of the removal of the unpaired electron from the parent tetra-anion is discussed. A comparison of structural parameters is reported.

We have recently reported the synthesis and structure of the high-nuclearity carbido-carbonyl cluster $[Co_{13}C_2(CO)_{24}]^{4-}$ (**1**),[1] which was found to contain one unpaired electron. Paramagnetic carbonyl clusters are not common species and they occur only when occupation of a low-lying antibonding orbital localized on the cluster framework is important to the stabilization of the structure. As most paramagnetic clusters contain interstitial carbon atoms,[1–3] we had argued [1,2] that the extra electron helped in widening the prismatic cavities thus favouring the incorporation of the interstitial carbon atoms.

In the present case, $[Co_{13}C_2(CO)_{24}]^{4-}$, it was expected that the extra electron would not be very tightly bound and its removal could take place with retention of the overall molecular geometry. Controlled oxidation of the tetra-anion gave the expected trianion, whose i.r. spectrum indicated no changes in the ligand geometry. We decided to determine the structure of the trianion in order to ascertain whether the removal of an electron from a frontier orbital had measurable steric effects.

Results and Discussion

Synthesis and Chemical Characterization.—The $[Co_{13}C_2(CO)_{24}]^{3-}$ anion has been prepared by oxidation with iodine in MeCN of the corresponding tetra-anion according to equation (1). The reaction is clean and quantitative.

$$[Co_{13}C_2(CO)_{24}]^{4-} + 0.5\,I_2 \longrightarrow [Co_{13}C_2(CO)_{24}]^{3-} + I^- \quad (1)$$

Crystals of the benzyltrimethylammonium salt suitable for *X*-ray analysis were obtained by the slow-diffusion technique from acetone and propan-2-ol. The crystals are stable in air for a few hours, while the brown solutions are quickly oxidized; they are soluble in acetone and MeCN, sparingly soluble in tetrahydrofuran, and insoluble in propan-2-ol and hydrocarbons. The i.r. spectrum in MeCN solution (Figure 1) shows bands at 1 990vs, 1 975(sh), 1 812m, and 1 782mw cm^{-1} with the same shape as that of the corresponding tetra-anion, but shifted to higher frequencies by about 30 cm^{-1}, in accord with the lower negative charge.

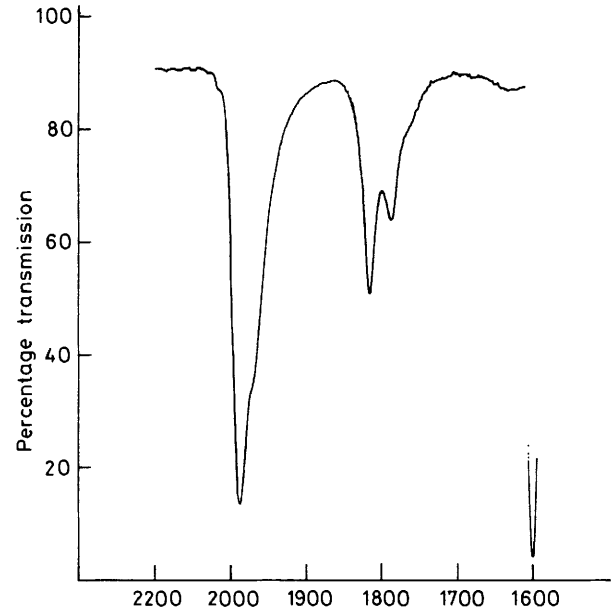

Figure 1. The i.r. spectrum of the anion $[Co_{13}C_2(CO)_{24}]^{3-}$ in MeCN solution

The $[Co_{13}C_2(CO)_{24}]^{3-}$ anion slowly reacts with CO to give eventually a mixture of $[Co(CO)_4]^-$, $Co_3(CO)_9CX$ species, and other cluster products at present under investigation. It can be converted back into the corresponding tetra-anion by reduction, for example with zinc powder in MeCN.

Structure.—The molecular structure of $[Co_{13}C_2(CO)_{24}]^{3-}$ (**2**), shown in Figure 2, was determined by single-crystal *X*-ray

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* For correspondence on structure.

† For correspondence on synthesis and chemical characterization.

‡ Part 13, S. Martinengo, D. Strumolo, P. Chini, V. G. Albano, and D. Braga, *J. Chem. Soc., Dalton Trans.*, 1985, 35

§ Benzyltrimethylammonium di-μ_6-carbido-dodeca-μ-carbonyl-dodecacarbonyl-*polyhedro*-tridecacobaltate(3−).

Supplementary data available (No. SUP 56188, 6 pp.): thermal parameters. See Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1985, Issue 1, pp. xvii–xix. Structure factors are available from the editorial office.

Table 1. Fractional atomic co-ordinates for complex (**2**)

Atom	*x*	*y*	*z*	Atom	*x*	*y*	*z*
Co(1)	−0.267 5(2)	−0.228 9(3)	0.258 9(3)	O(18)	−0.354 8(9)	−0.356 0(15)	0.362 1(16)
Co(2)	−0.166 5(2)	−0.131 4(3)	0.333 0(3)	C(19)	−0.205 3(13)	0.017 7(23)	0.216 2(23)
Co(3)	−0.189 5(2)	−0.312 4(3)	0.359 6(3)	O(19)	−0.179 1(10)	0.107 4(17)	0.232 6(17)
Co(4)	−0.126 1(2)	−0.398 8(3)	0.183 5(3)	C(20)	−0.311 2(13)	−0.085 6(22)	−0.038 5(24)
Co(5)	−0.194 9(2)	−0.320 6(3)	0.064 5(3)	O(20)	−0.330 2(10)	−0.070 4(16)	−0.127 3(17)
Co(6)	−0.097 3(2)	−0.219 0(3)	0.160 9(3)	C(21)	−0.393 7(17)	−0.043 6(27)	0.141 0(28)
Co(7)	−0.197 4(2)	−0.116 7(3)	0.137 3(3)	O(21)	−0.443 1(12)	−0.012 0(19)	0.125 9(19)
Co(8)	−0.306 0(2)	−0.045 2(3)	0.103 6(3)	C(22)	−0.150 7(12)	−0.199 9(21)	0.448 9(23)
Co(9)	−0.261 9(2)	−0.211 6(3)	−0.029 5(3)	O(22)	−0.127 7(10)	−0.168 8(17)	0.533 5(19)
Co(10)	−0.321 5(2)	−0.320 4(3)	0.082 5(3)	C(23)	−0.319 7(14)	−0.336 7(23)	−0.070 3(26)
Co(11)	−0.368 8(2)	−0.150 4(3)	0.210 2(3)	O(23)	−0.330 8(10)	−0.383 0(17)	−0.156 6(19)
Co(12)	−0.271 2(2)	−0.040 6(3)	0.297 3(3)	C(24)	−0.403 7(15)	−0.264 5(25)	0.116 9(26)
Co(13)	−0.248 2(2)	−0.413 2(3)	0.187 5(3)	O(24)	−0.454 8(11)	−0.292 2(18)	0.080 1(19)
C(0)	−0.284 9(13)	−0.183 0(21)	0.128 5(23)	C(25)	−0.307 0(14)	−0.136 0(23)	0.388 9(27)
C(1)	−0.179 8(12)	−0.273 7(20)	0.218 6(21)	O(25)	−0.329 1(9)	−0.121 4(15)	0.481 8(17)
C(2)	−0.175 2(12)	−0.013 7(22)	0.422 8(23)	N(1)	0.175 8(11)	0.847 8(17)	0.188 8(19)
O(2)	−0.172 9(9)	0.067 6(16)	0.493 1(17)	C(111)	0.220 4(13)	0.783 7(22)	0.239 6(23)
C(3)	−0.229 2(16)	−0.359 7(27)	0.468 5(31)	C(112)	0.289 5(7)	0.809 2(15)	0.227 3(17)
O(3)	−0.254 3(12)	−0.369 0(19)	0.548 2(22)	C(113)	0.317 2(7)	0.885 5(15)	0.309 5(17)
C(4)	−0.098 0(14)	−0.521 5(25)	0.112 4(25)	C(114)	0.381 5(7)	0.909 5(15)	0.294 8(17)
O(4)	−0.081 4(12)	−0.606 0(20)	0.066 3(21)	C(115)	0.418 2(7)	0.857 1(15)	0.197 8(17)
C(5)	−0.137 2(15)	−0.326 3(24)	−0.048 7(27)	C(116)	0.390 5(7)	0.780 8(15)	0.115 6(17)
O(5)	−0.102 8(10)	−0.344 7(16)	−0.125 1(18)	C(117)	0.326 2(7)	0.756 8(15)	0.130 3(17)
C(6)	−0.036 9(15)	−0.185 0(25)	0.072 3(27)	C(121)	0.186 9(14)	0.965 3(23)	0.236 9(25)
O(6)	0.006 9(11)	−0.161 8(18)	0.019 6(19)	C(131)	0.189 7(17)	0.814 7(27)	0.064 4(30)
C(7)	−0.144 8(15)	−0.084 3(24)	0.032 1(27)	C(141)	0.107 1(18)	0.830 3(29)	0.224 3(31)
O(7)	−0.116 6(11)	−0.056 8(18)	−0.035 8(20)	N(2)	0.986 2(11)	0.251 6(18)	0.329 1(20)
C(8)	−0.312 2(17)	0.094 0(31)	0.109 2(30)	C(211)	0.940 2(13)	0.212 7(22)	0.416 6(24)
O(8)	−0.320 1(13)	0.179 3(23)	0.122 1(22)	C(212)	0.901 6(9)	0.296 3(14)	0.497 1(16)
C(9)	−0.222 0(15)	−0.226 4(24)	−0.159 7(28)	C(213)	0.922 1(9)	0.354 0(14)	0.598 4(16)
O(9)	−0.192 4(11)	−0.236 2(18)	−0.246 3(21)	C(214)	0.881 5(9)	0.427 4(14)	0.673 2(16)
C(10)	−0.351 8(17)	−0.448 2(29)	0.065 4(29)	C(215)	0.820 2(9)	0.443 2(14)	0.646 7(16)
O(10)	−0.377 5(12)	−0.527 5(20)	0.054 1(20)	C(216)	0.799 7(9)	0.385 6(14)	0.545 4(16)
C(11)	−0.427 7(16)	−0.134 4(26)	0.313 1(30)	C(217)	0.840 4(9)	0.312 1(14)	0.470 6(16)
O(11)	−0.468 1(13)	−0.110 6(21)	0.388 7(23)	C(221)	0.944 6(15)	0.282 9(25)	0.251 3(27)
C(12)	−0.308 9(15)	0.077 3(26)	0.369 1(26)	C(231)	1.027 8(15)	0.343 0(25)	0.386 0(26)
O(12)	−0.335 4(13)	0.151 7(22)	0.416 1(23)	C(241)	1.026 5(15)	0.156 6(25)	0.258 2(26)
C(13)	−0.254 1(20)	−0.539 5(35)	0.215 3(35)	N(3)	0.458 9(14)	0.251 0(24)	0.313 3(25)
O(13)	−0.253 1(13)	−0.619 7(23)	0.233 3(23)	C(311)	0.387 5(19)	0.249 0(30)	0.338 1(32)
C(14)	−0.121 7(14)	−0.402 8(24)	0.332 8(25)	C(312)	0.459 1(14)	0.449 2(18)	0.373 1(26)
O(14)	−0.088 9(10)	−0.453 8(16)	0.377 4(17)	C(313)	0.488 8(14)	0.493 1(18)	0.292 5(26)
C(15)	−0.045 5(15)	−0.337 2(24)	0.171 4(26)	C(314)	0.466 2(14)	0.587 6(18)	0.280 9(26)
O(15)	0.008 1(11)	−0.355 2(18)	0.170 8(19)	C(315)	0.414 0(14)	0.638 2(18)	0.349 9(26)
C(16)	−0.082 7(15)	−0.119 0(24)	0.285 5(25)	C(316)	0.384 3(14)	0.594 3(18)	0.430 5(26)
O(16)	−0.038 0(11)	−0.062 3(18)	0.322 3(19)	C(317)	0.406 8(14)	0.499 8(18)	0.442 1(26)
C(17)	−0.212 8(13)	−0.462 0(23)	0.032 5(24)	C(321)	0.473 7(20)	0.234 4(33)	0.191 0(38)
O(17)	−0.211 5(9)	−0.543 0(17)	−0.041 1(17)	C(331)	0.491 1(17)	0.351 4(30)	0.391 0(31)
C(18)	−0.306 1(14)	−0.342 2(23)	0.302 7(25)	C(341)	0.489 4(22)	0.151 5(37)	0.345 2(38)

diffractometry, showing that (**1**) and (**2**) are strictly isostructural. Although features of the metal-atom polyhedron of complex (**1**) have already been discussed, it is worth summarizing the main points. The metal-atom polyhedron, shown in Figure 3, can be described as a three-layer sequence whose outer layers are squares and the inner one a system of condensed triangles. The stacking sequence is such as to generate two prismatic cavities accommodating the interstitial carbon atoms. A comparison of the average metal–metal bond parameters for complexes (**1**) and (**2**) shows that while the overall average Co–Co distance of 2.575 Å is coincident for both (**1**) and (**2**), the bond-length distribution is slightly more homogeneous for (**2**) [maximum and minimum values 2.789(4), 2.390(4) and 2.745(5), 2.387(5) Å for (**1**) and (**2**), respectively]. A slight shortening of the Co–C(carbide) average distances [1.98 and 1.97 Å for (**1**) and (**2**), respectively] confirms what was previously inferred about the role of the extra electron present in (**1**), *i.e.* it is used to widen the prismatic cavities in order to accommodate the carbide atoms. There are twelve terminal and twelve edge-bridging carbonyl ligands. Each cobalt atom possesses one terminal ligand, with the exception of Co(1) which has none, probably because of its very high connectivity (eight metal–metal, two metal–carbonyl, and two metal–carbide interactions). The bridging ligands span the four edges of the outer square layers and four out of five edges of the inner layer. The carbonyl ligands conform to idealized C_2 symmetry. The molecule exists in two enantiomeric forms and the crystal contains a racemic mixture.

The bond parameters for the carbonyl ligands are within normal ranges, with Co–C and C–O averages of 1.75 and 1.15 Å for terminal groups and 1.89 and 1.21 Å for bridging ones, respectively. These values, if compared with those in the tetra-anion (1.70, 1.18 and 1.96, 1.19 Å, respectively), show the expected trend for the terminal groups, *i.e.* longer Co–C and shorter C–O values for the species with lower anionic charge. Unexpectedly, this effect is reversed for the edge-bridging groups. The C–O stretching frequencies, on the other hand,

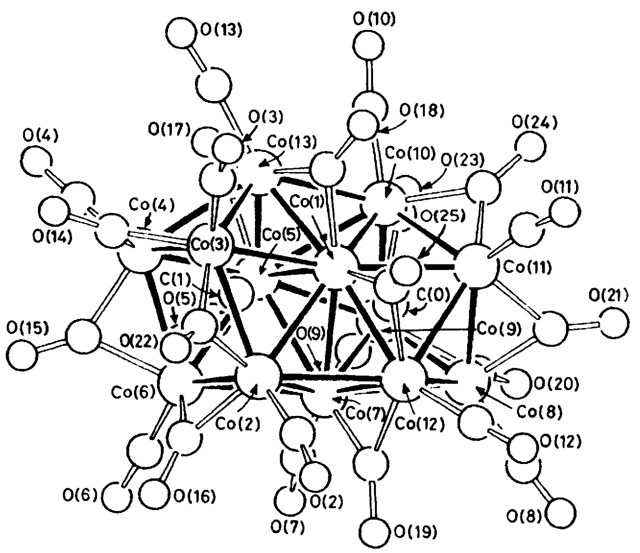

Figure 2. The anion $[Co_{13}C_2(CO)_{24}]^{3-}$ showing the atom labelling. The carbon atoms of the CO groups, not labelled, are numbered according to the corresponding oxygens

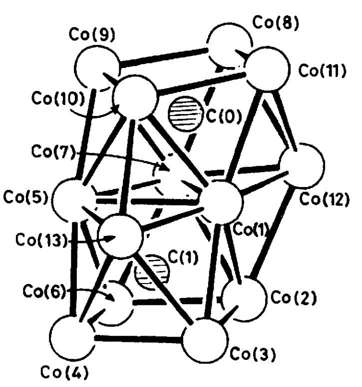

Figure 3. The metal-core geometry of the anion $[Co_{13}C_2(CO)_{24}]^{3-}$

Table 2. Bond distances (Å) and angles (°) with estimated standard deviations in parentheses

Co(1)–Co(2)	2.582(5)	Co(5)–Co(9)	2.605(6)	Co(1)–Co(3)	2.595(6)	Co(5)–Co(7)	2.602(5)
Co(1)–Co(5)	2.745(5)	Co(5)–Co(13)	2.412(6)	Co(1)–Co(7)	2.734(6)	Co(5)–Co(10)	2.659(6)
Co(1)–Co(10)	2.572(6)	Co(7)–Co(8)	2.607(6)	Co(1)–Co(11)	2.586(6)	Co(6)–Co(7)	2.610(6)
Co(1)–Co(12)	2.409(5)	Co(7)–Co(12)	2.418(5)	Co(1)–Co(13)	2.387(5)	Co(7)–Co(9)	2.615(6)
Co(2)–Co(3)	2.541(6)	Co(8)–Co(11)	2.474(6)	Co(2)–Co(6)	2.502(5)	Co(8)–Co(9)	2.507(5)
Co(2)–Co(7)	2.657(6)	Co(9)–Co(10)	2.520(6)	Co(3)–Co(4)	2.473(5)	Co(8)–Co(12)	2.589(6)
Co(3)–Co(13)	2.614(6)	Co(10)–Co(13)	2.640(7)	Co(4)–Co(5)	2.590(6)	Co(10)–Co(11)	2.539(5)
Co(4)–Co(6)	2.531(6)	Co(2)–Co(12)	2.662(6)	Co(4)–Co(13)	2.575(6)	Co(11)–Co(12)	2.643(3)
Co(5)–Co(6)	2.640(6)						
C(0)–Co(1)	1.96(3)	C(1)–Co(5)	1.92(3)	Co(13)–C(18)	1.86(3)	C(25)–O(25)	1.18(4)
C(0)–Co(7)	2.03(3)	C(1)–Co(6)	2.02(3)	Co(12)–C(19)	1.92(3)	Co(12)–C(12)	1.74(3)
C(0)–Co(8)	2.00(3)	C(1) ··· Co(7)	2.60(3)	Co(9)–C(20)	2.02(3)	Co(3)–C(14)	1.83(3)
C(0)–Co(9)	1.93(3)	C(1) ··· Co(13)	2.29(3)	C(9)–O(9)	1.18(4)	Co(4)–C(15)	1.88(3)
C(0)–Co(10)	1.92(3)	Co(2)–C(2)	1.66(2)	C(10)–O(10)	1.15(4)	Co(2)–C(16)	1.83(3)
C(0)–Co(11)	1.96(3)	Co(3)–C(3)	1.79(4)	C(11)–O(11)	1.19(4)	Co(5)–C(17)	1.84(3)
C(0) ··· Co(5)	2.57(3)	Co(4)–C(4)	1.71(3)	C(12)–O(12)	1.13(4)	Co(1)–C(18)	1.88(3)
C(2)–O(2)	1.19(3)	Co(5)–C(5)	1.77(3)	C(13)–O(13)	1.15(6)	Co(7)–C(19)	1.79(3)
C(3)–O(3)	1.12(5)	Co(6)–C(6)	1.75(3)	C(14)–O(14)	1.24(4)	Co(8)–C(20)	1.73(3)
C(4)–O(4)	1.15(3)	Co(7)–C(7)	1.79(3)	C(15)–O(15)	1.16(4)	Co(8)–C(21)	1.87(4)
C(5)–O(5)	1.12(3)	Co(8)–C(8)	1.83(4)	C(16)–O(16)	1.24(4)	Co(11)–C(21)	1.95(4)
C(6)–O(6)	1.17(4)	Co(9)–C(9)	1.73(3)	C(17)–O(17)	1.21(3)	Co(3)–C(22)	1.83(3)
C(7)–O(7)	1.13(4)	Co(10)–C(10)	1.76(4)	C(18)–O(18)	1.25(4)	Co(10)–C(23)	1.87(3)
C(8)–O(8)	1.11(5)	Co(11)–C(11)	1.68(3)	C(19)–O(19)	1.27(4)	Co(11)–C(24)	1.83(3)
C(0) ··· Co(12)	2.46(3)	Co(13)–C(13)	1.80(5)	C(20)–O(20)	1.29(4)	Co(12)–C(25)	2.02(4)
C(1)–Co(1)	1.93(3)	Co(4)–C(14)	1.90(3)	C(21)–O(21)	1.18(5)	Co(2)–C(22)	1.97(3)
C(1)–Co(2)	2.07(2)	Co(6)–C(15)	1.97(3)	C(22)–O(22)	1.18(4)	Co(9)–C(23)	2.02(3)
C(1)–Co(3)	1.97(3)	Co(6)–C(16)	1.80(3)	C(23)–O(23)	1.13(4)	Co(10)–C(24)	1.86(3)
C(1)–Co(4)	1.95(3)	Co(13)–C(17)	1.96(3)	C(24)–O(24)	1.22(4)	Co(1)–C(25)	1.89(3)
Co(2)–C(2)–O(2)	170(2)	Co(12)–C(12)–O(12)	177(3)	Co(5)–C(17)–O(17)	144(3)	Co(2)–C(22)–O(22)	132(2)
Co(3)–C(3)–O(3)	166(3)	Co(13)–C(13)–O(13)	175(4)	Co(13)–C(17)–O(17)	137(2)	Co(3)–C(22)–O(22)	145(3)
Co(4)–C(4)–O(4)	177(3)	Co(3)–C(14)–O(14)	143(2)	Co(1)–C(18)–O(18)	137(2)	Co(9)–C(23)–O(23)	128(3)
Co(5)–C(5)–O(5)	170(3)	Co(4)–C(14)–O(14)	138(2)	Co(13)–C(18)–O(18)	142(2)	Co(10)–C(23)–O(23)	151(3)
Co(6)–C(6)–O(6)	175(3)	Co(4)–C(15)–O(15)	141(3)	Co(7)–C(19)–O(19)	144(2)	Co(10)–C(24)–O(24)	133(2)
Co(7)–C(7)–O(7)	173(3)	Co(6)–C(15)–O(15)	137(3)	Co(12)–C(19)–O(19)	134(2)	Co(11)–C(24)–O(24)	139(2)
Co(3)–C(8)–O(8)	173(3)	Co(2)–C(16)–O(16)	135(2)	Co(8)–C(20)–O(20)	151(2)	Co(1)–C(25)–O(25)	149(3)
Co(9)–C(9)–O(9)	177(3)	Co(16)–C(16)–O(16)	138(2)	Co(9)–C(20)–O(20)	126(2)	Co(12)–C(25)–O(25)	134(2)
Co(10)–C(10)–O(10)	173(3)						
Co(11)–C(11)–O(11)	172(3)						

show the expected general increase for the species with lower charge.

Experimental

All the operations were carried out under nitrogen using purified solvents. The salt $[NMe_3(CH_2Ph)]_4[Co_{13}C_2(CO)_{24}]$ was prepared as already described.[1]

Synthesis of $[Co_{13}C_2(CO)_{24}]^{3-}$.—A solution of $[NMe_3(CH_2Ph)]_4[Co_{13}C_2(CO)_{24}]$ (0.22 g, 0.107 mmol) in MeCN (15 cm^3) was treated dropwise while stirring with a solution of iodine (0.015 g, 0.118 mol equiv.) in MeCN (4 cm^3) in slight excess over the stoicheiometric 1 : 1 ratio. At the end of the addition the i.r. bands of the starting tetra-anion [1 960s, 1 945(sh), 1 785ms, and 1 758mw cm^{-1}] were replaced by those of the trianion. The brown solution was evaporated to dryness by pumping in vacuum at room temperature, the residue was dissolved in acetone (6 cm^3), and the solution filtered. Crystals were obtained by cautiously layering propan-2-ol (24 cm^3) over this solution and leaving the solvents to diffuse for a few days. The crystals were filtered off, washed with propan-2-ol, and vacuum dried. Yields: 75—85% (Found: C, 35.35; H, 2.55; N, 2.05. Calc. for $C_{56}H_{48}Co_{13}N_3O_{24}$: C, 35.15; H, 2.55; N, 2.20%).

Crystallography.—Crystal data. $C_{56}H_{48}Co_{13}N_3O_{24}$, M = 1 913.13, triclinic, a = 21.082(3), b = 13.242(2), c = 12.556(2) Å, α = 105.88(2), β = 84.75(2), γ = 91.03(2)°, U = 3 357.2 Å^3, D_m = 1.90, Z = 2, D_c = 1.89 g cm^{-3}, space group $P\bar{1}$ (no. 2), $F(000)$ = 1 896, Mo-K_α radiation, λ = 0.710 69 Å, μ(Mo-K_α) = 31.9 cm^{-1}.

Intensity measurements. From a crystal fragment with dimensions 0.1 × 0.07 × 0.1 mm, 3 504 reflections intensities were collected in the range $3 < \theta < 20°$, by the ω—2θ scan method on a Philips PW1100 diffractometer with scan interval 0.9° and speed 3° min^{-1}. The background was measured at both sides of the reflections for a total time equal to the peak scanning time. Due to rapid decay of the crystal upon X-ray exposure, neither a wider θ range nor psi-scan data for empirical absorption correction could be collected. 3 325 Reflections $[F_o > 6\sigma(F_o)]$ were used for structure solution and refinement. The structure was solved by conventional Patterson methods and Fourier analysis. The computations were carried out with the SHELX[4] package of crystallographic programs. Metal atoms were allowed to vibrate anisotropically and the light atoms isotropically. The phenyl rings were treated as rigid groups (C–C 1.395 Å, C–C–C 120°). Hydrogens were omitted because of the very high number of atoms in the structure model. The final agreement indices were R = 0.088 and R' = 0.106. The weighting scheme employed was $w = k[\sigma^2(F) + |g|F^2]$ where k = 1.52 and g = 0.0011. A final Fourier difference map showed residual peaks not exceeding 1.3 e Å^{-3} in the vicinity of the cobalt atoms. Fractional atomic co-ordinates are reported in Table 1, bond distances and angles in Table 2.

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References

1 V. G. Albano, D. Braga, P. Chini, G. Ciani, and S. Martinengo, *J. Chem. Soc., Dalton Trans.*, 1982, 645.
2 V. G. Albano, P. Chini, G. Ciani, M. Sansoni, and S. Martinengo, *J. Chem. Soc., Dalton Trans.*, 1980, 163.
3 D. Strumolo, C. Seregni, S. Martinengo, V. G. Albano, and D. Braga, *J. Organomet. Chem.*, 1983, **252**, C93.
4 SHELX 76 System of Computer Programs, G. M. Sheldrick, Cambridge, 1976.

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