

distortion. Steric congestion around the metal is also evidenced by the small Cl(1)—Nb—Cl(2) and Cl(1)—Nb—P(1) angles (79 and 73.8°, respectively). The Nb—PMe₃ distances are of two types depending on the ligand in the *trans* position; they are shorter than those observed in Nb₄Cl₁₀(PMe₃)₆ (Cotton & Shang, 1988), one of the few niobium–phosphine adducts structurally characterized. The lability of the phosphine ligand and/or metathesis reactions opens synthetic routes to other monocyclopentadienyl derivatives.

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Tricarbonyl[(1,2,3,4- η)-2,3-dimethylbutadiene]cobalt Acetonitriletrichloroferrate(1-)

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Abstract. [Co(C₆H₁₀)(CO)₃][Fe(Cl)₃(C₂H₃N)], *M*_r = 428.35, monoclinic, *P*2₁/*a*, *a* = 11.259 (10), *b* = 12.043 (4), *c* = 12.727 (7) Å, β = 97.53 (6)°, *V* = 1710.8 Å³, *Z* = 4, *D*_x = 1.663 Mg m⁻³, Mo *K* α , λ = 0.71069 Å, μ = 2.291 mm⁻¹, *F*(000) = 856, *T* = 291 (1) K, *R* = 0.032 for 1383 independent observed reflections. The title compound, an archetypal Co^I species, was studied in the hope of gaining further information about [7,7,7-(CO)₃-7-CoB₁₀H₁₂]⁻, which had previously been described formally as a Co^{III} complex. Comparison of Co—CO distances and C—O IR stretching frequencies lends support to this conclusion.

Introduction. Recently (Macgregor, Yellowlees & Welch, 1990) we reported that analysis of the structure of the anion [7,7,7-(CO)₃-7-CoB₁₀H₁₂]⁻ (1) implied that the {B₁₀H₁₂} moiety in this species could best be described as the *arachno* fragment {B₁₀H₁₂}⁴⁻, and therefore that the formal oxidation state of the Co atom was +3.

In an attempt to gain further information on this system we describe here the results of a structural study of the cation [(CO)₃Co(dmbd)]⁺ (2) [dmbd = 2,3-dimethylbutadiene, η -CH₂C(Me)C(Me)CH₂]. This species has been selected for comparison with the cobaltaborane anion since (i) it contains a

directly analogous metal coordination sphere (a tricarbonyl unit and an acyclic η^4 -bonded ligand) and (ii) the oxidation state of cobalt in the title compound is clearly known (+1). Spectroscopic evidence for the cation [(CO)₃Co(dmbd)]⁺ was first furnished in 1974 (Chauldry & Paulson, 1974), but no bulk preparation and characterization was carried out. Here we report the isolation of (2) as its [FeCl₃(NCMe)]⁻ salt.

Experimental. Compound prepared as the [FeCl₃(NCMe)]⁻ salt by oxidation of a CH₂Cl₂ solution of [Co(CO)₂(dmbd)]₂ with FeCl₃, removal of solvent *in vacuo*, and extraction of the resultant solid into acetonitrile; identity checked by IR spectroscopy (KBr disc, ν_{C-O} = 2130, 2100 and 2080 cm⁻¹), and established by diffraction study; marine blue crystal, 0.2 × 0.2 × 0.3 mm, mounted on an Enraf–Nonius CAD-4 diffractometer (Mo *K* α radiation, graphite monochromator); cell parameters and orientation matrix from least-squares refinement of the setting angles (9 < θ < 12°) of 25 centred reflections; data collection by ω -2 θ scans in 96 steps with ω scan width (0.8 + 0.34tan θ)°; data (*h*: 0 to 10, *k*: 0 to 11, *l*: -12 to 12) measured for 1 ≤ θ ≤ 20° over 18 X-ray hours; corrections for Lorentz and polarization effects applied (Gould & Smith, 1986); 1699 indepen-

dent reflections measured, of which 1383 [$F \geq 1.0\sigma(F)$] retained; structure solution *via* automatic direct methods (Co, Fe) (Sheldrick, 1986), and iterative full-matrix least-squares refinement (on $F/\Delta F$ syntheses (Cl, O, N, C, H) (Sheldrick, 1976); empirical absorption correction (Walker & Stuart, 1983) applied after isotropic convergence (maximum and minimum correction factors 1.053 and 0.933, respectively); CH₃ moieties treated as rigid groups; vinyl H atoms located and positionally refined; all H atoms given a fixed isotropic thermal parameter ($U_H = 0.04 \text{ \AA}^2$); weighting scheme $w^{-1} = \sigma^2(F) + 0.000506F^2$; model refined in two blocks of 68 and 137 variables, data:variable ratio > 10:1; max. shift/e.s.d. in final cycle < 0.06; $R = 0.032$, $wR = 0.033$, $S = 1.294$; max. and min. residues in final ΔF synthesis 0.28 and -0.35 e \AA^{-3} , respectively; scattering factors for C, H, N, O and Cl inlaid in *SHELX76* (Sheldrick, 1976), those for Co and Fe from *International Tables for X-ray Crystallography* (1974, Vol. IV); Fig. 1 drawn using *EASYORTEP* (Mallinson & Muir, 1985); molecular geometry calculations *via* *CALC* (Gould & Taylor, 1986).

Discussion. Table 1* lists the coordinates of refined atoms and equivalent isotropic thermal parameters, and Table 2 details internuclear distances and interbond angles between these. The cation (2) has effective, non-crystallographically imposed, C_s symmetry about the plane containing CoC(2)O(2), and the midpoint of the C(5)—C(6) bond, and the {Co(CO)₃} unit is oriented such that one carbonyl ligand, C(2)O(2), lies over the open edge of the dmbd ligand (see Fig. 1). This (*endo*) conformation is analogous to that observed in the cobaltaborane anion (1), confirming the similarities of the metal coordination spheres in both species.

Table 3 summarizes Co—CO and C—O distances for (1) and (2) (parameters related by the effective mirror planes present in both species are averaged). The bond angles are highly similar, the only perceptible difference being a slight widening of C(1)—Co—C(3) in (1). Co—CO bonds are consistently shorter in the cobaltaborane anion than in the cobalt-dmbd cation, and are thus not inconsistent with a greater formal metal oxidation state in the former species. However, at least two other differences (discussed below) between (1) and (2) could contribute to this result, making it somewhat difficult to factorize out the causes. The C—O distances in (2) are marginally shorter than those in (1), but the

* Lists of structure factors, H-atom positions and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53518 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Coordinates of refined atoms and equivalent isotropic thermal parameters* (\AA^2)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
Fe	0.22731 (6)	0.52612 (5)	0.69958 (5)	0.0231 (4)
Cl(1)	0.33475 (12)	0.46608 (11)	0.57326 (9)	0.0409 (8)
Cl(2)	0.34165 (11)	0.61722 (10)	0.83185 (9)	0.0328 (8)
Cl(3)	0.10651 (12)	0.40022 (11)	0.76295 (10)	0.0414 (9)
N(1)	0.1174 (4)	0.6387 (4)	0.6165 (3)	0.035 (3)
C(10)	0.0509 (5)	0.6898 (4)	0.5610 (4)	0.034 (3)
Cl(11)	-0.0364 (4)	0.7552 (4)	0.4906 (4)	0.039 (3)
Co	0.69182 (6)	0.43230 (6)	0.81949 (5)	0.0251 (4)
C(1)	0.7827 (5)	0.3509 (4)	0.9218 (4)	0.034 (4)
O(1)	0.8410 (4)	0.3033 (3)	0.9855 (3)	0.045 (3)
C(2)	0.5438 (6)	0.3696 (4)	0.8211 (4)	0.034 (4)
O(2)	0.4504 (4)	0.3338 (3)	0.8254 (3)	0.049 (3)
C(3)	0.7531 (4)	0.3602 (4)	0.7113 (4)	0.028 (3)
O(3)	0.7927 (3)	0.3197 (3)	0.6445 (3)	0.042 (3)
C(4)	0.6628 (5)	0.5538 (4)	0.9292 (4)	0.034 (4)
HC(41)	0.585 (4)	0.581 (4)	0.906 (4)	*
HC(42)	0.672 (4)	0.533 (4)	1.006 (4)	*
C(5)	0.7608 (4)	0.5837 (4)	0.8768 (4)	0.022 (3)
C(6)	0.7399 (4)	0.5865 (4)	0.7645 (4)	0.023 (3)
C(7)	0.6222 (5)	0.5584 (4)	0.7177 (4)	0.028 (3)
HC(71)	0.553 (4)	0.580 (4)	0.751 (4)	*
HC(72)	0.619 (4)	0.549 (4)	0.651 (4)	*
C(8)	0.8848 (4)	0.6017 (4)	0.9367 (4)	0.033 (3)
C(9)	0.8387 (4)	0.6087 (4)	0.6983 (4)	0.031 (3)

* U_{iso} fixed at 0.04 \AA^2 .

Table 2. *Internuclear distances* (\AA) *and interbond angles* ($^\circ$)

Fe—Cl(1)	2.2540 (14)	C(1)—O(1)	1.130 (7)
Fe—Cl(2)	2.2642 (14)	C(2)—O(2)	1.144 (7)
Fe—Cl(3)	2.2554 (15)	C(3)—O(3)	1.122 (6)
Fe—N(1)	2.035 (4)	C(4)—C(5)	1.408 (7)
N(1)—C(10)	1.140 (7)	C(4)—HC(41)	0.94 (5)
C(10)—C(11)	1.469 (7)	C(4)—HC(42)	0.99 (5)
Co—C(1)	1.831 (5)	C(5)—C(6)	1.418 (7)
Co—C(2)	1.832 (6)	C(5)—C(8)	1.516 (7)
Co—C(3)	1.837 (5)	C(6)—C(7)	1.420 (7)
Co—C(4)	2.077 (6)	C(6)—C(9)	1.504 (7)
Co—C(5)	2.076 (5)	C(7)—HC(71)	0.97 (5)
Co—C(6)	2.082 (5)	C(7)—HC(72)	0.85 (5)
Co—C(7)	2.082 (5)		
Cl(1)—Fe—Cl(2)	112.32 (5)	C(6)—Co—C(7)	39.90 (20)
Cl(1)—Fe—Cl(3)	116.40 (5)	Co—C(1)—O(1)	177.9 (5)
Cl(1)—Fe—N(1)	100.94 (13)	Co—C(2)—O(2)	177.0 (5)
Cl(2)—Fe—Cl(3)	111.76 (5)	Co—C(3)—O(3)	177.4 (5)
Cl(2)—Fe—N(1)	108.13 (13)	Co—C(4)—C(5)	70.2 (3)
Cl(3)—Fe—N(1)	106.22 (13)	Co—C(4)—HC(41)	105 (3)
Fe—N(1)—C(10)	170.6 (4)	Co—C(4)—HC(42)	119 (3)
N(1)—C(10)—C(11)	179.0 (6)	C(5)—C(4)—HC(41)	121 (3)
C(1)—Co—C(2)	101.27 (25)	C(5)—C(4)—HC(42)	123 (3)
C(1)—Co—C(3)	92.85 (24)	HC(41)—C(4)—HC(42)	111 (4)
C(1)—Co—C(4)	91.26 (23)	Co—C(5)—C(4)	70.2 (3)
C(1)—Co—C(5)	94.16 (22)	Co—C(5)—C(6)	70.3 (3)
C(1)—Co—C(6)	124.83 (22)	Co—C(5)—C(8)	125.4 (3)
C(1)—Co—C(7)	164.30 (23)	C(4)—C(5)—C(6)	116.6 (4)
C(2)—Co—C(3)	104.20 (24)	C(4)—C(5)—C(8)	121.7 (4)
C(2)—Co—C(4)	93.41 (24)	C(6)—C(5)—C(8)	121.6 (4)
C(2)—Co—C(5)	131.06 (22)	Co—C(6)—C(5)	69.9 (3)
C(2)—Co—C(6)	130.45 (22)	Co—C(6)—C(7)	70.0 (3)
C(2)—Co—C(7)	92.24 (23)	Co—C(6)—C(9)	126.2 (3)
C(3)—Co—C(4)	160.74 (23)	C(5)—C(6)—C(7)	116.0 (4)
C(3)—Co—C(5)	121.21 (21)	C(5)—C(6)—C(9)	122.1 (4)
C(3)—Co—C(6)	91.69 (21)	C(7)—C(6)—C(9)	121.7 (4)
C(3)—Co—C(7)	91.51 (22)	Co—C(7)—C(6)	70.1 (3)
C(4)—Co—C(5)	39.64 (20)	Co—C(7)—HC(71)	100 (3)
C(4)—Co—C(6)	70.63 (20)	Co—C(7)—HC(72)	119 (3)
C(4)—Co—C(7)	79.90 (22)	C(6)—C(7)—HC(71)	120 (3)
C(5)—Co—C(6)	39.88 (18)	C(6)—C(7)—HC(72)	111 (3)
C(5)—Co—C(7)	70.75 (20)	HC(71)—C(7)—HC(72)	123 (4)

unfortunately high e.s.d.'s associated with the structural study of (1) prevent detailed comparison.

In addition to a higher oxidation state resulting in shorter Co—CO distances (by dint of the smaller radius of Co³⁺ versus Co⁺), both the overall charge on the metal complexes and the nature of the η -bonded ligand *trans* to the carbonyl functions could influence the Co—CO bond lengths. Thus, if the formal metal oxidation states in both (1) and (2) were the same, both the higher negative charge on (1) and the presence of an η -bonded ligand face composed of B atoms rather than C atoms might reasonably be expected to increase the electron density at the metal atom, resulting in greater Co—CO π back bonding and shorter Co—CO distances.

To an extent, the influence of the difference in overall charge between (1) and (2) on the extent of Co—CO back bonding can be assessed by consideration of CO stretching frequencies. The {M(CO)₃} fragment has local C_{3v} symmetry, giving rise to C—O stretches of *a*₁ and *e* symmetry. Table 4 lists the '*a*₁' and '*e*' IR stretching frequencies for the family of *d*⁶ complexes [CpM(CO)₃]ⁿ (Cp = η -C₅H₅; M = Fe, *n* = +1; M = Mn, *n* = 0; M = Cr, *n* = -1) and for (2) and (1). In moving from the (cationic) Fe complex to the (anionic) Cr complex the frequency of the *a*₁ stretch decreases by 170 cm⁻¹ and that of the *e*

Table 3. Comparison of key molecular parameters (Å, °) for (1) and (2)

	[7,7,7-(CO) ₃ -7-CoB ₁₀ H ₁₂] ⁻ (1)	[(CO) ₃ Co(dmbd)] ⁺ (2)
Co—C(1,3)	1.802 (14)	1.834 (5)
Co—C(2)	1.763 (11)	1.832 (6)
C(1,3)—O(1,3)	1.134 (18)	1.126 (7)
C(2)—O(2)	1.165 (15)	1.144 (7)
C(1,3)—Co—C(2)	102.97 (58)	102.74 (25)
C(1)—Co—C(3)	95.58 (63)	92.85 (24)

Table 4. Carbonyl IR stretching frequencies (cm⁻¹) in complexes containing {M(CO)₃} fragments

Complex	' <i>a</i> ₁ ' stretch	' <i>e</i> ' stretch	Reference
[CpFe(CO) ₃] ⁺	2070	2127	Kockhar & Pettit (1966)
[CpMn(CO) ₃]	2025	1945	Haas & Sheline (1967)
[CpCr(CO) ₃] ⁻	1900	1776*	Ellis & Flum (1975)
[(CO) ₃ Co(dmbd)] ⁺ (2)	2130	2090*	This work
[(CO) ₃ CoB ₁₀ H ₁₂] ⁻ (1)	2065	2010*	Macgregor, Yellowlees & Welch (1990)

* Components of '*e*' stretch resolved; average value given.

stretch by 250 cm⁻¹ (as more electron density is available for M—CO back bonding).

In moving from the (cationic) (2) to the (anionic) (1), the changes are considerably smaller (65 and 80 cm⁻¹, respectively). This implies that considerably less electron density is available to the metal centre in (1) than is expected from overall charge considerations alone. Given that the non-carbonyl ligand in (1) is anticipated to be a somewhat stronger donor than that in (2) (Cowie, Hamilton, Laurie & Welch, 1990), this analysis of IR stretching frequencies appears to lend support to the idea that the metal atom in (1) is formally in a higher oxidation state than that in (2). Indeed, this result is fully consistent with analysis of the dimensions of the borane ligand in (1) (Macgregor, Yellowlees & Welch, 1990), and with the results of XANES studies on (1) in comparison to those on standard Co^I and Co^{III} compounds (Wynd, Macgregor, Gould, Taylor, Yellowlees & Welch, 1990).

Bond distances and angles within the metallabutadiene moiety of (2) are entirely consistent with those found in a large number of isoelectronic iron analogues (Herbstein & Reisner, 1977). As has previously been described by Immirzi (1974) for iron-butadiene systems, we find that the *syn* and *anti* H atoms of the dmbd ligand are displaced out of the C(4)C(5)C(6)C(7) plane, the former by (average) 0.172 Å away from Co, and the latter by (average) 0.516 Å towards Co.

As far as we can ascertain, the anion in the current study, [FeCl₃(NCMe)]⁻, has not previously been structurally characterized. The environment around the Fe atom is slightly distorted from tetrahedral; the Cl—Fe—Cl angles average 113.5 (1)°, whilst N—Fe—Cl averages 105.1 (2)°. The angle at the

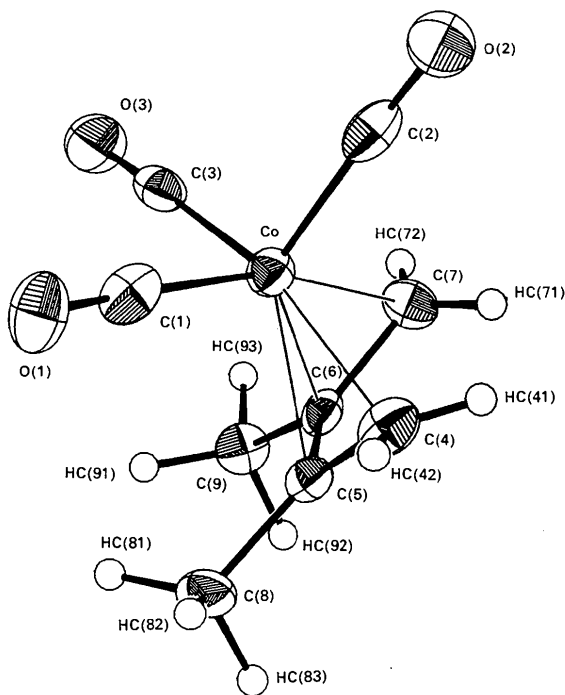


Fig. 1. Perspective view of, and atomic numbering scheme adopted for, the cation [Co(C₆H₁₀)(CO)₃]⁺ (50% thermal ellipsoids, except for H atoms which have an artificial radius of 0.1 Å for clarity).

acetonitrile N atom is significantly bent away from 180°, whilst the sequence N(1)–C(10)–C(11) is linear.

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Structure of Bis(1,5-cyclooctadiene)rhodium(I) Hexacarbonyl-tri- μ -chloro-dirhenate(I)

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Abstract. $[\text{Rh}(\text{C}_8\text{H}_{12})_2]^+[\text{Re}_2(\text{CO})_6\text{Cl}_3]^-$, $M_r = 966.07$, monoclinic, $P2_1/n$ (non-standard setting of $P2_1/c$, No. 13), $a = 11.221$ (3), $b = 7.018$ (2), $c = 16.562$ (6) Å, $\beta = 91.30$ (3)°, $V = 1303.9$ (7) Å³, $Z = 2$, $D_m = 2.30$ (10), $D_x = 2.46$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 107.45$ cm⁻¹, $F(000) = 900$, $T = 297$ K. Final $R_F = 0.053$ for 2448 observed reflections [$F_o^2 > 3\sigma(F_o^2)$]. The crystal structure of the title compound consists of $(\text{C}_8\text{H}_{12})_2\text{Rh}^+$ cations and $(\text{CO})_3\text{-ReCl}_3\text{Re}(\text{CO})_3^-$ anions. The 1,5-cyclooctadiene ligands are chelated to the Rh atom. The coordination about rhodium is square planar and the olefin midpoint distances are 2.128 (13) and 2.131 (14) Å. The Re atoms display pseudo-octahedral coordination bridged by the three Cl atoms. The carbonyl groups occupy the non-bridging positions about the Re atom, $\text{Re}-\text{Cl} = 2.506$ (3) and 2.524 (3) Å, $\text{Re}-\text{Re} = 3.380$ (1) Å, $\text{Re}-\text{C}_{av} = 1.89$ (1) Å.

Introduction. In conjunction with a study of potential catalytic properties of bimetallic compounds the title compound was synthesized and found to function in a limited manner as a hydrogenation, isomerization or polymerization catalyst for certain cyclic poly-

olefins. To gain a better understanding of their catalytic properties we have been studying the structures of these bimetallic ionic compounds and report here the synthesis and structure of the title compound.

Experimental. $[\text{Rh}(\text{C}_8\text{H}_{12})_2]^+[\text{Re}_2(\text{CO})_6\text{Cl}_3]^-$ was prepared by the addition of 0.100 g (0.203 mmol) of $[\text{Rh}(\text{C}_8\text{H}_{12})\text{Cl}]_2$ to 0.30 ml (0.250 mmol) of 1,5-cyclooctadiene and 0.300 g (0.420 mmol) of $[\text{Re}(\text{CO})_4\text{Cl}]_2$ in 20 ml of dichloromethane. D_m measured pycnometrically in water. The red solution was stirred for 24 h and upon evaporation dark-red diamond-shaped prismatic crystals separated from the mixture. Crystal size 0.23 × 0.24 × 0.04 mm, ω - 2θ scan with a maximum 2θ value of 57.5° ($h = 0$ to ± 15 , $k = 0$ to ± 9 , $l = -22$ to 21) on a Picker four-circle diffractometer using graphite-monochromated Mo $K\alpha$ radiation. Three standard reflections (400, 040, 006) monitored every sixty reflections, showed small random variations (2.5, 5.0 and 1.7% respectively). Twelve non-collinear reflections were measured and their setting angles were fitted by a least-squares program to give the orientation matrix and cell dimension constants. Data were corrected for Lorentz and polarization effects. The compound has a linear absorption coefficient $\mu =$

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