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Bis(tricarbonyl-η^5-methylcyclopentadienylchromium)(*Cr*—*Cr*)

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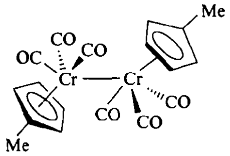
Abstract

The structure of $[Cr(MeC_5H_4)(CO)_3]_2$ is centrosymmetric and is similar to those of the pentamethylcyclopentadienyl and cyclopentadienyl analogues.

Comment

The title compound, (I), was synthesized from $Cr(CO)_6$ and methylcyclopentadiene using published procedures (Birdwhistell, Hackett & Manning, 1978) and recrystallized from toluene–hexane. The interest in this structure lies in a comparison of its *M*—*M* bond with those in its cyclopentadienyl (Adams, Collins & Cotton, 1974) and pentamethylcyclopentadienyl (Goh, Hambley, Darensbourg & Reibenspies, 1990) analogues, both of which readily dissociate to give highly reactive 17-electron radical species (Baird, 1988; McLain, 1988; Goh, Khoo & Lim, 1990; Goh & Lim, 1991; Watkins, Jaeger, Kidd, Fortier, Baird, Kiss, Roper & Hoff, 1992).



(I)

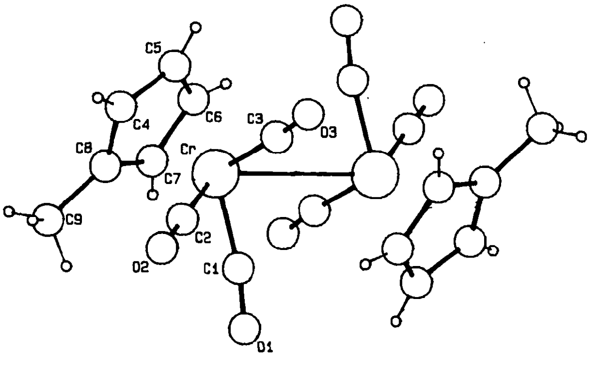


Fig. 1. A perspective view of $[Cr(MeC_5H_4)(CO)_3]_2$ showing the numbering scheme.

The molecular structure of the title compound (Fig. 1) is similar to those of the C_5Me_5 and C_5H_5 analogues (see Table 3). The Cr—Cr bond is shortest in the title compound and there is a decrease in the Cr—C(carbonyl) and an increase in the C≡O bond distances as the number of ring methyl substituents increases. The Cr—Cr—C angles of the title compound are almost identical to those of the C_5H_5 analogue, whereas one of the angles in the C_5Me_5 compound has a larger value of 117.3 (1)°.

Experimental

Crystal data

$[Cr_2(C_6H_7)_2(CO)_6]$
$M_r = 430.30$
Triclinic
$P\bar{1}$
$a = 7.5371$ (7) Å
$b = 7.7348$ (7) Å
$c = 8.5907$ (8) Å
$\alpha = 79.031$ (7)°
$\beta = 81.474$ (8)°
$\gamma = 64.451$ (7)°
$V = 442.4$ (1) Å^3
$Z = 1$
$D_x = 1.615$ Mg m^{-3}

Mo *Kα* radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 14$–16°
$\mu = 1.23$ mm^{-1}
$T = 300$ K
Chunky parallelepiped
0.32 × 0.30 × 0.30 mm
Dark green

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: empirical (ψ-scans; North, Phillips & Mathews, 1968)
$T_{\min} = 0.755$, $T_{\max} = 0.998$
2164 measured reflections
2011 independent reflections

1735 observed reflections [$I > 3\sigma(I)$]
$\theta_{\max} = 27.5$°
$h = 0 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -11 \rightarrow 11$
3 standard reflections monitored every 400 reflections
intensity variation: −0.0134% h^{-1}

Refinement

Refinement on *F*
$R = 0.030$
$wR = 0.035$
$S = 0.587$
1735 reflections
146 parameters
All H-atom parameters refined
$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{\max} < 0.01$
$\Delta\rho_{\max} = 0.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.30$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	*y*	*z*	B_{eq}
Cr	0.07440 (5)	0.37926 (5)	0.17186 (4)	3.015 (6)
O1	0.4463 (2)	0.3148 (3)	−0.0389 (2)	5.26 (5)
O2	0.3268 (3)	0.4344 (3)	0.3775 (2)	5.98 (5)
O3	−0.1549 (3)	0.7923 (3)	0.2281 (2)	5.03 (5)
C1	0.2971 (3)	0.3473 (3)	0.0324 (3)	3.70 (5)

C2	0.2299 (3)	0.4120 (3)	0.2985 (3)	4.01 (5)
C3	−0.0706 (3)	0.6384 (3)	0.1959 (3)	3.70 (5)
C4	−0.0107 (4)	0.2173 (4)	0.3827 (3)	4.29 (6)
C5	−0.1688 (3)	0.3039 (4)	0.2852 (4)	4.58 (6)
C6	−0.1042 (3)	0.2252 (3)	0.1417 (3)	4.52 (5)
C7	0.0929 (4)	0.0940 (3)	0.1498 (3)	4.20 (5)
C8	0.1540 (3)	0.0846 (3)	0.3000 (3)	3.97 (5)
C9	0.3466 (4)	−0.0494 (5)	0.3638 (4)	5.72 (8)

Table 2. *Bond lengths* (Å) *and angles* (°)

Cr—Cr	3.247 (1)	O3—C3	1.148 (3)
Cr—C1	1.863 (2)	Cr—C2	1.833 (2)
Cr—C3	1.859 (3)	Cr—C4	2.179 (3)
Cr—C5	2.197 (3)	Cr—C6	2.214 (3)
Cr—C7	2.193 (3)	Cr—C8	2.198 (3)
O1—C1	1.146 (3)	O2—C2	1.151 (3)
C4—C8	1.420 (4)	C5—C6	1.407 (4)
C7—C8	1.412 (4)	C8—C9	1.484 (4)
C6—C7	1.395 (4)	C4—C5	1.401 (4)
Cr—Cr—C1	72.27 (8)	C7—C8—C9	126.7 (3)
Cr—Cr—C3	71.65 (7)	Cr—Cr—C2	128.21 (9)
Cr—Cr—C4	141.71 (8)	Cr—Cr—C5	104.53 (8)
Cr—Cr—C6	84.00 (8)	Cr—Cr—C7	101.31 (7)
Cr—Cr—C8	138.80 (7)	Cr—Cr—C9	145.17 (8)
C2—Cr—C3	79.3 (1)	C1—Cr—C2	79.7 (1)
C1—Cr—C3	110.8 (1)	Cr—C1—O1	172.2 (2)
Cr—C2—O2	179.4 (3)	Cr—C3—O3	172.4 (2)
C5—C4—C8	108.8 (3)	C4—C5—C6	107.6 (3)
C5—C6—C7	108.3 (3)	C6—C7—C8	108.9 (2)
C4—C8—C7	106.4 (2)	C4—C8—C9	126.7 (3)

Table 3. *A comparison of geometric parameters* (Å, °) *for* $[Cr(MeC_5H_4)(CO_3)]_2$ *and its* C_5H_5 *and* C_5Me_5 *analogues*

	$(Cp)_2Cr_2(CO)_6$	$(MeCp)_2Cr_2(CO)_6$	$(Me_5Cp)_2Cr_2(CO)_6$
Cr—Cr	3.281 (1)	3.247 (1)	3.310 (1)
Cr—C	1.846–1.870 (3)	1.833–1.863 (2)	1.818–1.859 (3)
Cr—C(Cp)	2.174–2.227 (3)	2.179–2.214 (3)	2.179–2.264 (3)
C—O	1.141–1.146 (3)	1.146–1.151 (3)	1.152–1.157 (4)
Cr—C—O	172.0–178.7 (2)	172.2–179.4 (2)	167.8–178.6 (3)
Cr—Cr—C	79.2 (1)	79.3 (1)	77.9 (1)
	80.8 (1)	79.7 (1)	78.9 (1)
	110.4 (1)	110.8 (1)	117.3 (1)

The structure was solved by Patterson synthesis and refined by full-matrix least-squares methods using *MolEN* (Fair, 1990) on a DEC MicroVAX II minicomputer. The H atoms were located from difference Fourier maps and were refined isotropically.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1098). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structural Analogues of Ferroverdin. Tris(methyl isonitrosoacetoacetato)cobalt(III)

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Abstract

The title compound (*OC*-6-22)-tris[methyl 2-(nitroso-*N*)acetoacetato-*O*]cobalt(III), $[Co(C_5H_6NO_4)_3]$, has been synthesized and its structure solved by X-ray diffraction. Each of the three bidentate ligands binds through the N atom of the nitroso group and the ketonic O atom to the Co atom, which displays trigonally distorted *fac*-octahedral coordination. The three ligands display different conformations around the C—C(O)OMe bond.

Comment

Isonitrosoketones and related ligands such as nitrosophenols are used as analytical reagents (Welcher, 1947). Complexes of transition metals with these ligands (Wilkinson, Gillard & McCleverty, 1987; Basu & Chakravorty, 1992) are potential models for metal binding sites in ferroverdin (Ballio, Barcellona, Chain, Tonolo & Vero-Barcellona, 1964; Candeloro *et al.*, 1969) and have industrial applications as dyes and light-absorbing agents for accelerating the solar evaporation

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