

Fig. 1. Configuration of the cluster core $\{WCu_3S_3Cl\}(PPh_3)_3(S)$.

which were in the vicinity of the W atom. All calculations were performed on a VAX 785 computer using the *TEXSAN* (Molecular Structure Corporation, 1985) program package, the scattering factors were taken from Cromer & Waber (1974). The view of the molecule was produced by the *ORTEPII* program (Johnson, 1976) (Fig. 1). The atom coordinates and thermal parameters are listed in Table 1;* the important bond lengths and bond angles are

* Lists of structure factors, anisotropic thermal parameters complete bond lengths and angles, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53550 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

given in Table 2. The main differences in bond distances between the title compound and a related compound which was prepared by Müller, Bögge & Schimanski (1983) are appended to Table 2.

Related literature. μ_3 -Chloro-tri- μ_3 -sulfido-tris[(triphenylphosphine)copper](sulfidotungsten)(3Cu—W) was prepared by allowing WS_4^{2-} to react with PPh_3 and $CuCl_2 \cdot 2H_2O$ (Müller, Bögge & Schimanski, 1983). This crystal belongs to the orthorhombic system with space group $P2_12_12_1$.

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Structure of Pentacarbonyl(morpholine- κN)chromium(0)

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Abstract. $[Cr(CO)_5(C_4H_9NO)]$, $M_r = 279.2$, monoclinic, $P2_1/c$, $a = 9.391(7)$, $b = 10.946(7)$, $c = 12.259(9)$ Å, $\beta = 108.14(5)^\circ$, $V = 1197.6(2)$ Å³, $Z = 4$, $D_x = 1.55$ g cm⁻³, $\lambda(Cu K\alpha) = 1.5418$ Å, $\mu = 81.71$ cm⁻¹, $F(000) = 568$, room temperature, $R = 0.039$ for 1332 reflections with $I \geq 3\sigma(I)$. The metal

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atom is coordinated to the morpholine ligand via the N atom and to five CO ligands in a slightly distorted octahedral arrangement with bond lengths Cr—C(*cis*, mean) = 1.899 (5), Cr—C(*trans*) = 1.819 (6), Cr—N = 2.197 (4) Å. There are no unusual features in the morpholine ligand.

Experimental. The compound under study was prepared by methods described previously (Gmelin, 1971). A yellow crystal (0.4 × 0.2 × 0.2 mm) of the title compound was used for measurements on a Siemens AED diffractometer, with graphite-monochromatized Cu K α radiation, ω : θ scan mode; unit-cell dimensions were determined by a least-squares fit of 26 centred reflections with $23 \leq 2\theta \leq 33^\circ$; two standard reflections monitored every hour showed no significant intensity decay. Of the 1645 unique reflections with $3 \leq 2\theta \leq 60^\circ$, 1332 were considered observed with $I \geq 3\sigma(I)$, index range $h = 0 \rightarrow 10$, $k = 0 \rightarrow 12$, $l = -13 \rightarrow 13$. Intensities were corrected for Lp effects as usual and an absorption correction was made after isotropic refinement; correction factors ranged from 0.812 to 1.259.

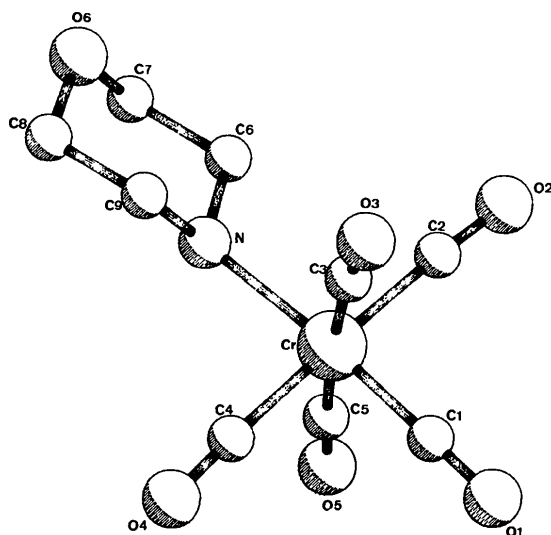


Fig. 1. Plot of the complex showing the atom-numbering scheme.

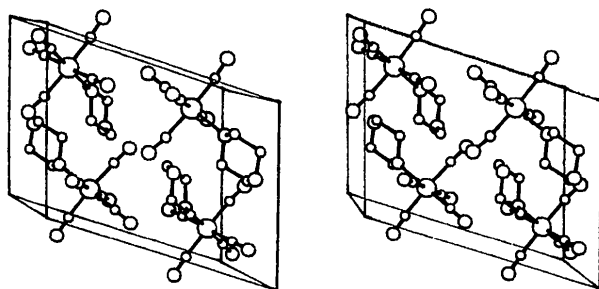


Fig. 2. Packing diagram.

Table 1. Positional parameters and equivalent isotropic values of the anisotropic temperature factors ($\times 10^4$) for the non-H atoms

	x	y	z	U_{eq} (Å ²)
Cr	0.1853 (1)	0.1548 (1)	0.3469 (1)	355 (2)
N	0.2672 (4)	0.0544 (3)	0.2232 (3)	373 (14)
C1	0.1174 (5)	0.2476 (5)	0.4434 (4)	467 (19)
C2	0.1619 (6)	0.0113 (5)	0.4279 (4)	503 (20)
C3	0.3804 (6)	0.1708 (5)	0.4540 (4)	487 (20)
C4	0.2049 (6)	0.2995 (4)	0.2694 (4)	441 (18)
C5	-0.0158 (6)	0.1417 (5)	0.2493 (4)	480 (20)
C6	0.2427 (6)	-0.0788 (4)	0.2149 (4)	501 (20)
C7	0.2916 (6)	-0.1338 (5)	0.1178 (5)	658 (25)
C8	0.4655 (7)	0.0208 (5)	0.1321 (5)	654 (25)
C9	0.4255 (6)	0.0792 (5)	0.2307 (5)	520 (21)
O1	0.0748 (4)	0.3085 (3)	0.5043 (3)	650 (16)
O2	0.1474 (5)	-0.0700 (4)	0.4808 (4)	848 (21)
O3	0.4920 (5)	0.1814 (4)	0.5249 (3)	785 (18)
O4	0.2104 (5)	0.3896 (4)	0.2221 (4)	785 (19)
O5	-0.1404 (5)	0.1413 (4)	0.1954 (4)	771 (18)
O6	0.4434 (4)	-0.1071 (4)	0.1325 (4)	708 (18)

Table 2. Interatomic distances (Å), angles ($^\circ$) and torsion angles ($^\circ$) with *e.s.d.*'s in parentheses

Cr—N	2.197 (4)	C2—O2	1.134 (7)
Cr—C1	1.819 (6)	C3—O3	1.140 (6)
Cr—C2	1.907 (5)	C4—O4	1.153 (7)
Cr—C3	1.901 (5)	C5—O5	1.155 (6)
Cr—C4	1.886 (5)	C6—C7	1.526 (9)
Cr—C5	1.901 (5)	C7—O6	1.411 (7)
N—C6	1.474 (6)	C8—C9	1.515 (9)
N—C9	1.485 (7)	C8—O6	1.415 (7)
C1—O1	1.161 (7)		
C1—Cr—C3	87.6 (2)	C4—Cr—C5	88.8 (2)
C1—Cr—C2	90.1 (2)	C3—Cr—C5	175.6 (2)
N—Cr—C5	91.2 (2)	C3—Cr—C4	92.7 (2)
N—Cr—C4	87.9 (2)	C2—Cr—C5	90.7 (2)
N—Cr—C3	93.1 (2)	C2—Cr—C4	178.1 (2)
N—Cr—C2	94.0 (2)	C2—Cr—C3	87.7 (2)
N—Cr—C1	175.9 (2)	C1—Cr—C5	88.3 (2)
C1—Cr—C4	88.1 (2)		
C2—Cr—N—C9	-113.8 (3)	Cr—N—C9—C8	-171.8 (3)
C2—Cr—N—C6	15.9 (4)	Cr—N—C6—C7	174.5 (3)
C3—Cr—N—C9	-25.9 (3)	C6—N—C9—C8	54.2 (5)
C3—Cr—N—C6	103.8 (3)	C9—N—C6—C7	-52.8 (5)
C4—Cr—N—C9	66.6 (3)	N—C6—C7—O6	57.0 (6)
C4—Cr—N—C6	-163.7 (3)	C6—C7—O6—C8	-60.5 (6)
C5—Cr—N—C9	155.4 (3)	C9—C8—O6—C7	61.4 (6)
C5—Cr—N—C6	-74.9 (4)	O6—C8—C9—N	-59.0 (6)

The structure was solved by direct methods (Sheldrick, 1986) and refined on F by full-matrix least squares (Stewart, Kundell & Baldwin, 1980), H-atom positions were calculated (Fayos & Martínez-Ripoll, 1980) and refined isotropically; refinement converged to $R = 0.039$ for 154 refined parameters; unit weights; overdetermination ratio 8.7; max. shift/*e.s.d.* = 0.982; max. electron density in final difference map was 0.52 e Å⁻³; atomic scattering factors were from *International Tables for X-ray Crystallography* (1974, Vol. IV). The molecule and numbering scheme are shown in Fig. 1, and the molecular packing in Fig. 2. Positional parameters and equivalent values of the anisotropic temperature

factors for the non-H atoms are given in Table 1,* and interatomic distances, angles and torsion angles in Table 2.

Related literature. For the preparation of the title compound see Gmelin (1971). Spectral studies and the kinetics of thermal decomposition were described by Dennenberg & Paronbourg (1972). Electrochemical studies and solvent effects were described by Chadwick, Díaz, González, Santa Ana & Yútronic (1986).

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53607 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of (η^5 -Cyclopentadienyl)(η^6 -*p*-isopropylnitrobenzene)iron(II) Trifluoroacetate at 198 K

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Abstract. $[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_9\text{H}_{11}\text{NO}_2)]\cdot\text{C}_2\text{F}_3\text{O}_2$, (I), $M_r = 399.1$, monoclinic, $P2_1/c$, $a = 8.995$ (2), $b = 10.641$ (2), $c = 17.538$ (5) Å, $\beta = 95.22$ (2)°, $V = 1671.7$ (8) Å³, $Z = 4$, $D_x = 1.586$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 9.50$ cm⁻¹, $F(000) = 816$, $T = 163$ K, $R = 0.0648$ and $wR = 0.0713$ for 2502 reflections [$I \geq 3\sigma(I)$]. The phenyl ring exhibits a significant asymmetry in the C—C and Fe—C bond lengths. C(3)—C(4) and C(4)—C(5) [1.427 (6) and 1.424 (6) Å] are longer than the rest of the phenyl bonds; the longest bond with the Fe atom is that of C(4) [2.131 (4) Å]. The lengthening of these bonds may be attributed to the bulky isopropyl group and the presence of H(7) on the same side of the phenyl ring as the FeCp moiety. The phenyl ring and the cyclopentadienyl (Cp) are nearly parallel to each other [dihedral angle 1.3 (2)°], and the plane of the nitro group is twisted by an angle of 5.7 (2)° with respect to the phenyl ring. The Fe atom is closer to the phenyl ring [1.5451 (8) Å] than the Cp ring [1.6711 (9) Å]; this phenomenon was also seen in all similar compounds studied in this laboratory. Each acetyl O atom is involved in a close contact with an

aromatic H atom. For C(3)—H(3)⋯O(3) ($1-x, -0.5+y, 0.5-z$): C—H = 0.86 (5), H⋯O = 2.59 (6), C⋯O = 3.207 (6) Å, C—H⋯O = 130 (3)°. For C(5)—H(5)⋯O(4) ($1-x, 0.5+y, 0.5-z$): C—H = 0.80 (5), H⋯O = 2.46 (6), C⋯O = 3.063 (6) Å, C—H⋯O = 132 (3)°.

Experimental. Compound (I) was synthesized by oxidation of the (η^5 -cyclopentadienyl)(η^6 -*p*-aminoisopropylbenzene)iron(II) hexafluorophosphate complex with H₂O₂ in trifluoroacetic acid according to a method similar to that reported by Sheats & Rausch (1970). Crystals were grown by slow evaporation from a mixture of acetone–ethyl ether–methylene chloride. A suitable crystal of dimensions 0.14 × 0.27 × 0.40 mm was chosen for X-ray investigation. Data were collected on a Siemens *R3m/V* diffractometer equipped with a graphite monochromator utilizing Mo $K\alpha$ radiation. 50 reflections with $20.82 \leq 2\theta \leq 26.08^\circ$ were used to refine the cell parameters. 7564 reflections were collected using the ω -scan method ($h, 0 \rightarrow 11$; $k, -13 \rightarrow 13$; $l, -22 \rightarrow 22$), 3835 unique reflections, $R_{\text{int}} = 0.0259$; 2θ range $3 \rightarrow 55^\circ$, $1.2^\circ \omega$