

Cameron Evans,* Louis J.
Farrugia and Marcus TegelWest CHEM, Department of Chemistry,
University of Glasgow, Joseph Black Building,
University Avenue, Glasgow G12 8QQ,
ScotlandCorrespondence e-mail:
cevens@chem.gla.ac.uk

Key indicators

Single-crystal X-ray study
 $T = 100$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
 R factor = 0.022
 wR factor = 0.054
Data-to-parameter ratio = 14.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.***tert*-Butyl isocyanide-1 κ C-di- μ -carbonyl-
2:3 κ^4 C-nonacarbonyl-1 κ^3 C,2 κ^3 C,3 κ^3 C-
triangulo-diironosmium**

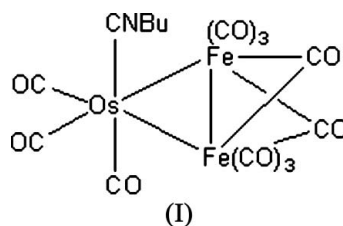
The preparation of the mixed-metal cluster, $[\text{Fe}_2\text{Os}(\text{C}_5\text{H}_9\text{N})(\text{CO})_{11}]$, and its crystal structure at 100 K are reported. This complex, along with the cluster reported in the following paper, are the first structurally characterized substitution derivatives of $\text{Fe}_2\text{Os}(\text{CO})_{12}$. The isonitrile ligand adopts an axial position on the osmium centre and the cluster is isostructural with the Fe_2Ru analogue.

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Comment

The structures of the mixed metal clusters $\text{Fe}_2M(\text{CO})_{12}$ ($M = \text{Ru}$ and Os) have been examined in detail (Churchill & Fetting, 1990; Braga *et al.*, 1995, 1996; Farrugia *et al.*, 1996), with particular focus on dynamic disorder within the metal triangle. In addition, various phosphine-, phosphite- (Venäläinen & Pakkanen, 1984) and isonitrile-substituted (Farrugia & Mertes, 2002) derivatives of $\text{Fe}_2\text{Ru}(\text{CO})_{12}$ have been structurally characterized. Phosphine and phosphite derivatives of $\text{Fe}_2\text{Os}(\text{CO})_{12}$ have been reported (Shojaie & Atwood, 1988) though, to date, no derivatives have been structurally characterized. We report here (and in Evans *et al.*, 2006) the synthesis and structures of $\text{Fe}_2\text{Os}(\text{CO})_{12-n}(\text{CNBu}^t)_n$ ($n = 1$ and 2).

$\text{Fe}_2\text{Os}(\text{CO})_{11}(\text{CNBu}^t)$, (I), was prepared by carbonyl substitution of the parent $\text{Fe}_2\text{Os}(\text{CO})_{12}$ cluster using standard methods (Farrugia & Mertes, 2002). The complex was characterized spectroscopically, by FAB mass spectrometry, and by single-crystal X-ray structure determination. The structure was determined at room temperature and 100 K with no discernible metal atom disorder at either temperature. As the structures at different temperatures are essentially identical, only the more precise low-temperature structure will be discussed here.



The structure of (I) is shown in Fig. 1. The isonitrile ligand adopts an axial position on the Os atom, equivalent to the orientation observed for $\text{Fe}_2\text{Ru}(\text{CO})_{11}(\text{CNBu}^t)$ (Farrugia & Mertes, 2002) and isomer *B* of $\text{Fe}_3(\text{CO})_{11}\text{PPh}_3$ (Dahm & Jacobson, 1968) but contrasting with the equatorial positions adopted by the phosphine and phosphite ligands in $\text{Fe}_2\text{Ru}(\text{CO})_{11}(\text{PR}_3)$ ($R = \text{Ph}$ and OME ; Venäläinen & Pakkanen,

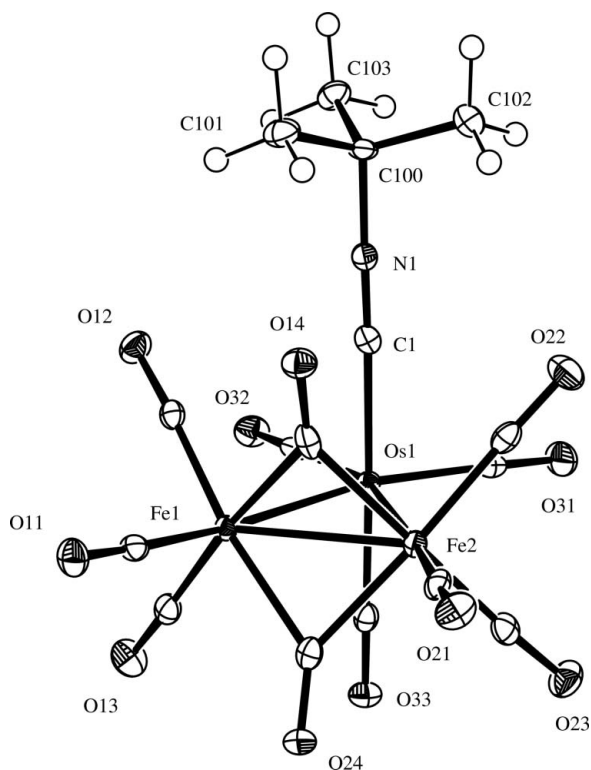


Figure 1
A view of $\text{Fe}_2\text{Os}(\text{CO})_{11}(\text{CNBu}')$ showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level, with H atoms represented by circles of arbitrary size.

1984). The average Os—Fe distance [2.7495 (8) Å] is marginally longer than that observed (Farrugia & Mertes, 2002) for Ru—Fe in the ruthenium analogue [2.7441 (7) Å], while the Fe—Fe distance [2.5675 (8) Å] is slightly shorter [2.5724 (6) Å]. The C—Os distance [2.042 (4) Å] and N—C—Os angle [177.9 (4)°] are comparable to those reported for the ruthenium analogue [2.045 (3) Å and 177.9 (2)°] and $\text{Os}_3(\text{CO})_{11}(\text{CNMe})$ [2.074 (23) Å and 173.6 (17)°; Dawson *et al.*, 1982]. Two carbonyl ligands symmetrically bridge the Fe—Fe bond [$\delta(M-C) = 0.018$ and 0.023 Å for C14 and C24, respectively].

The crystal of (I) is affected by twinning in a manner similar to that reported for the ruthenium analogue (Farrugia & Mertes, 2002). The twin axis is [101] and the non-merohedral twinning results in a significant number of seriously overlapped reflections, which were removed from the data file used for refinement. At 100 K, the proportion of the second component refines to 0.207 (1) compared with 0.172 (2) at room temperature.

Experimental

Complex (I) was prepared in the same manner as reported for the ruthenium analogue (Farrugia & Mertes, 2002), by reaction of the parent carbonyl with a 1:1 molar ratio of isonitrile. The product was purified by chromatography on Florisil using hexane/ CH_2Cl_2 mixtures as eluant. Crystals were obtained from a concentrated hexane solution at 253 K. Analysis calculated for $\text{C}_{16}\text{H}_9\text{Fe}_2\text{NO}_{11}\text{Os}$: C

27.73, H 1.31, N 2.02%; found: C 27.72, H 1.30, N 1.97%. IR [$\nu(\text{CN})$, cm^{-1}] 2200 (vw); IR [$\nu(\text{CO})$, cm^{-1}] 2042 (vs), 2033 (vs), 2019 (m), 1994 (w), 1984 (w), 1895 (vw), 1856 (vw), 1813 (w). $^1\text{H NMR}$: δ 1.54 (s, CH_3). Mass spectrum, $m/z = 695.1$ [M^+], 667.2 [$M^+ - \text{CO}$], 639.2 [$M^+ - 2\text{CO}$], 611.1 [$M^+ - 3\text{CO}$], 583.1 [$M^+ - 4\text{CO}$], 555.1 [$M^+ - 5\text{CO}$], 527.1 [$M^+ - 6\text{CO}$], 499.1 [$M^+ - 7\text{CO}$], 471.2 [$M^+ - 8\text{CO}$], 443.1 [$M^+ - 9\text{CO}$].

Crystal data

$[\text{Fe}_2\text{Os}(\text{C}_5\text{H}_9\text{N})(\text{CO})_{11}]$
 $M_r = 693.14$
Monoclinic, $P2_1/n$
 $a = 11.6861$ (2) Å
 $b = 11.6142$ (2) Å
 $c = 15.5189$ (2) Å
 $\beta = 107.829$ (1)°
 $V = 2005.14$ (6) Å³
 $Z = 4$

$D_x = 2.296$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 8960 reflections
 $\theta = 2.2\text{--}35.0^\circ$
 $\mu = 7.81$ mm⁻¹
 $T = 100$ (2) K
Prism, black
0.3 × 0.2 × 0.1 mm

Data collection

Nonius KappaCCD diffractometer
 φ or ω scans
Absorption correction: multi-scan (Blessing, 1995)
 $T_{\min} = 0.215$, $T_{\max} = 0.452$
40723 measured reflections
4158 independent reflections

4019 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.044$
 $\theta_{\max} = 27.6^\circ$
 $h = -15 \rightarrow 15$
 $k = -15 \rightarrow 14$
 $l = -20 \rightarrow 20$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.022$
 $wR(F^2) = 0.054$
 $S = 1.12$
4158 reflections
282 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0019P)^2 + 10.979P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.55$ e Å⁻³
 $\Delta\rho_{\min} = -1.23$ e Å⁻³
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00067 (8)

All H atoms were placed in calculated positions and refined using a riding model [C—H = 0.98 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$]. The highest features in the difference map are associated with the Os atom.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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