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Key indicators

Single-crystal X-ray study
 T = 100 K
 Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
 R factor = 0.021
 wR factor = 0.042
 Data-to-parameter ratio = 34.6

For details of how these key indicators were
 automatically derived from the article, see
<http://journals.iucr.org/e>.

Bis(*tert*-butyl isocyanide)-1 κ^2 C-di- μ -carbonyl-2:3 κ^4 C-octacarbonyl-1 κ^2 C,2 κ^3 C,3 κ^3 C-triangulo-diironosmium

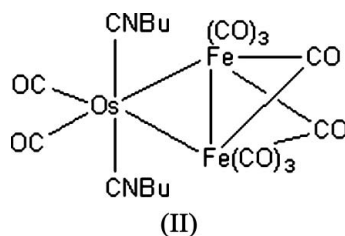
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The preparation of the mixed-metal cluster, $[\text{Fe}_2\text{Os}(\text{C}_5\text{H}_9\text{N})_2(\text{CO})_{10}]$, and its crystal structure at 100 K are reported. This complex, along with the cluster in the preceding paper, are the first structurally characterized substitution derivatives of $\text{Fe}_2\text{Os}(\text{CO})_{12}$. The isonitrile ligands adopt axial positions on the osmium centre and the cluster is isostructural with the Fe_2Ru analogue.

Comment

The background to this study has been set out in the preceding paper (Evans *et al.*, 2006). We report here and in that paper the synthesis and structures of $\text{Fe}_2\text{Os}(\text{CO})_{12-n}(\text{CNBu}^t)_n$ ($n = 1$ and 2).

$\text{Fe}_2\text{Os}(\text{CO})_{10}(\text{CNBu}^t)_2$, (II), was prepared by carbonyl substitution of the parent $\text{Fe}_2\text{Os}(\text{CO})_{12}$ cluster using standard methods (Farrugia & Mertes, 2002). The compound 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 (II) at 100 K is shown in Fig. 1. Both isonitrile ligands adopt axial positions on the Os atom, identical to that reported for the Fe_2Ru analogue but contrasting with $\text{Fe}_3(\text{CO})_{10}(\text{CNBu}^t)_2$ [where one isonitrile is axial and the other equatorial (Murray *et al.*, 1990)] and $M_3(\text{CO})_{10}(\text{CNR})_2$ [$M = \text{Ru}$ and Os ; $R = \text{Bu}^t$ and Me] (Dawson *et al.*, 1982; Bruce *et al.*, 1983; Farrugia *et al.*, 1998), where the two isonitrile ligands are axial but attached to different metal centres. The average Fe–Os distance [2.7590 (3) Å] and Fe–Fe distance [2.5738 (3) Å] are longer than those reported (Farrugia & Mertes, 2002) for the ruthenium analogue [$\text{Ru}-\text{Fe} = 2.7527$ (3) Å and $\text{Fe}-\text{Fe} = 2.5678$ (2) Å]. Two carbonyl ligands symmetrically bridge the Fe–Fe bond [$\delta(M-\text{C}) = 0.009$ and 0.003 \AA for C14 and C24, respectively].

Experimental

Complex (II) was prepared in the same manner as reported for the ruthenium analogue (Farrugia & Mertes, 2002) by reaction of the parent carbonyl with a 1:2 molar ratio of isonitrile. The product was purified by chromatography on Florisil using hexane/CH₂Cl₂ mixtures as eluant. Crystals were obtained from a concentrated hexane solution at 25 K. Analysis calculated for C₂₀H₁₈Fe₂N₂O₁₀Os: C 32.10, H 2.42, N 3.74%; found: C 32.15, H 2.20, N 3.74%. IR [$\nu(\text{CN})$, cm⁻¹] 2200 (*vw*), 2170 (*m*); IR [$\nu(\text{CO})$, cm⁻¹] 2053 (*w*), 2021 (*vs*), 2015 (*vs*), 1981 (*m*), 1975 (*m*), 1901 (*vw*), 1834 (*vw*), 1802 (*w*). ¹H NMR: δ 1.54 (*s*, CH₃). Mass spectrum, *m/z* = 750.2 [*M*⁺], 694.2 [*M*⁺ - 2CO], 666.2 [*M*⁺ - 3CO], 638.2 [*M*⁺ - 4CO], 610.2 [*M*⁺ - 5CO], 582.2 [*M*⁺ - 6CO], 554.3 [*M*⁺ - 7CO], 526.3 [*M*⁺ - 8CO], 498.3 [*M*⁺ - 9CO], 470.3 [*M*⁺ - 10CO].

Crystal data

[Fe₂Os(C₅H₉N)₂(CO)₁₀]
M_r = 748.26
 Monoclinic, *P*2₁/*a*
a = 11.6903 (2) Å
b = 12.4357 (2) Å
c = 17.6041 (3) Å
 β = 91.753 (1)°
V = 2558.03 (7) Å³
Z = 4

D_x = 1.943 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 11369 reflections
 θ = 2.2–35.0°
 μ = 6.13 mm⁻¹
T = 100 (2) K
 Prism, purple
 0.3 × 0.3 × 0.2 mm

Data collection

Nonius KappaCCD diffractometer
 φ or ω scans
 Absorption correction: multi-scan (Blessing, 1995)
*T*_{min} = 0.197, *T*_{max} = 0.294
 64585 measured reflections
 10967 independent reflections

9945 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.032
 θ_{max} = 35.0°
h = -18 → 18
k = -20 → 20
l = -27 → 28

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.021
wR (*F*²) = 0.042
S = 1.1
 10967 reflections
 317 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0113P)^2 + 1.9594P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.002$
 $\Delta\rho_{\text{max}} = 1.28 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.94 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.00024 (4)

All H atoms were placed in calculated positions and refined using a riding model [*C*–H = 0.98 Å and *U*_{iso}(H) = 1.5*U*_{eq}(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).

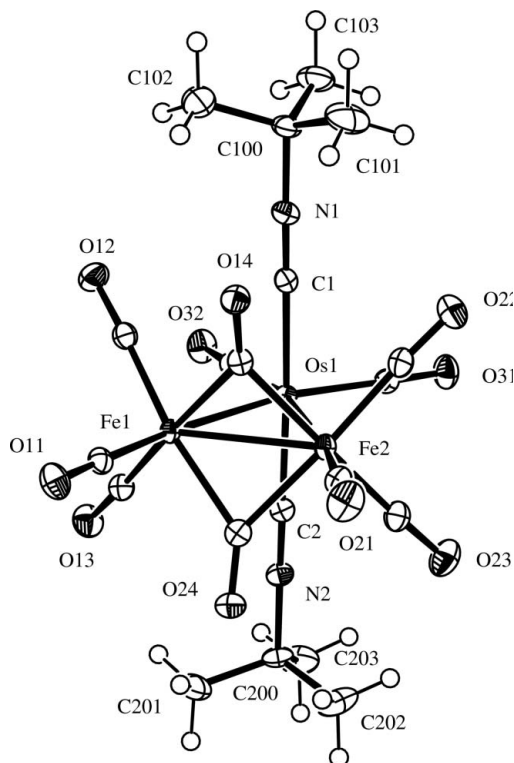


Figure 1

A view of Fe₂Os(CO)₁₀(CNBu)₂, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level, with H atoms represented by circles of arbitrary size.

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