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tert-Butyl isocyanide-1κC-di-μ-carbonyl-2:3$\kappa^4 C$-nonacarbonyl-1$\kappa^3 C$,2$\kappa^3 C$,3$\kappa^3 C$-*triangulo*-diironosmium

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.006 Å
R factor = 0.022
wR factor = 0.054
Data-to-parameter ratio = 14.7

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

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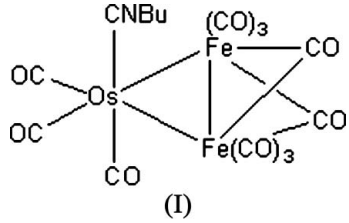
The preparation of the mixed-metal cluster, $[Fe_2Os(C_5H_9N)(CO)_{11}]$, and its crystal structure at 100 K are reported. This complex, along with the cluster reported in the following paper, are the first structurally characterized substitution derivatives of $Fe_2Os(CO)_{12}$. The isonitrile ligand adopts an axial position on the osmium centre and the cluster is isostructural with the Fe_2Ru analogue.

Comment

The structures of the mixed metal clusters $Fe_2M(CO)_{12}$ (M = Ru and Os) have been examined in detail (Churchill & Fettinger, 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- (Venalainen & Pakkanen, 1984) and isonitrile-substituted (Farrugia & Mertes, 2002) derivatives of $Fe_2Ru(CO)_{12}$ have been structurally characterized. Phosphine and phosphite derivatives of $Fe_2Os(CO)_{12}$ 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 $Fe_2Os(CO)_{12-n}(CNBu^t)_n$ (n = 1 and 2).

$Fe_2Os(CO)_{11}(CNBu^t)$, (I), was prepared by carbonyl substitution of the parent $Fe_2Os(CO)_{12}$ 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.



(I)

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 $Fe_2Ru(CO)_{11}(CNBu^t)$ (Farrugia & Mertes, 2002) and isomer B of $Fe_3(CO)_{11}PPh_3$ (Dahm & Jacobson, 1968) but contrasting with the equatorial positions adopted by the phosphine and phosphite ligands in $Fe_2Ru(CO)_{11}(PR_3)$ (R = Ph and OMe; Venalainen & Pakkanen,

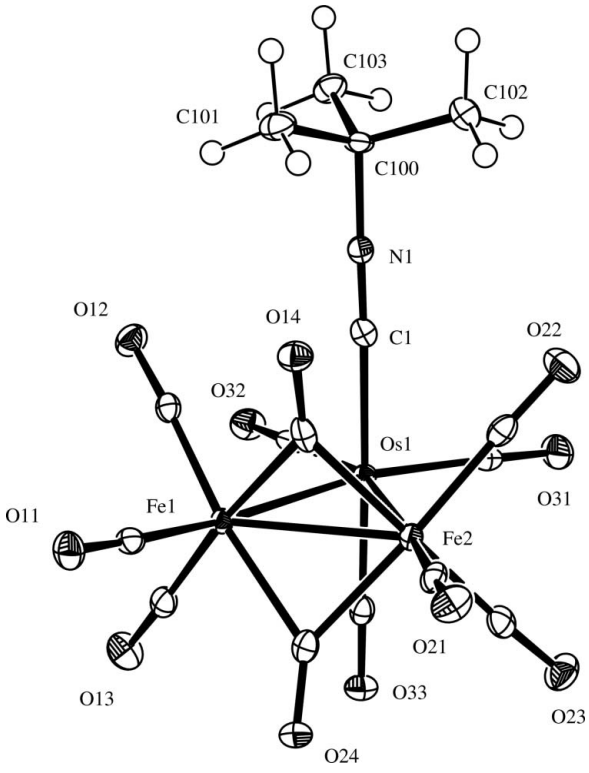


Figure 1
A view of $Fe_2Os(CO)_{11}(CNBu^t)$ showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level, with H atoms represented by circles of arbitrary size.

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 $Os_3(CO)_{11}(CNMe)$ [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 $C_{16}H_9Fe_2NO_{11}Os$: C 27.73, H 1.31, N 2.02%; found: C 27.72, H 1.30, N 1.97%. IR [ν(CN), cm^{-1}] 2200 (*vw*); IR [ν(CO), cm^{-1}] 2042 (*vs*), 2033 (*vs*), 2019 (*m*), 1994 (*w*), 1984 (*w*), 1895 (*vw*), 1856 (*vw*), 1813 (*w*). ^{1}H NMR: δ 1.54 (*s*, CH_3). Mass spectrum, *m*/*z* = 695.1 [M^+], 667.2 [M^+ − CO], 639.2 [M^+ − 2CO], 611.1 [M^+ − 3CO], 583.1 [M^+ − 4CO], 555.1 [M^+ − 5 CO], 527.1 [M^+ − 6CO], 499.1 [M^+ − 7CO], 471.2 [M^+ − 8CO], 443.1 [M^+ − 9 CO].

Crystal data

$[Fe_2Os(C_5H_9N)(CO)_{11}]$	D_x = 2.296 Mg m^{-3}
M_r = 693.14	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 8960 reflections
a = 11.6861 (2) Å	θ = 2.2–35.0°
b = 11.6142 (2) Å	μ = 7.81 mm^{-1}
c = 15.5189 (2) Å	T = 100 (2) K
β = 107.829 (1)°	Prism, black
V = 2005.14 (6) Å^3	0.3 × 0.2 × 0.1 mm
Z = 4	

Data collection

Nonius KappaCCD diffractometer	4019 reflections with $I > 2\sigma(I)$
φ or ω scans	R_{int} = 0.044
Absorption correction: multi-scan (Blessing, 1995)	θ_{max} = 27.6°
T_{min} = 0.215, T_{max} = 0.452	$h = -15 \rightarrow 15$
40723 measured reflections	$k = -15 \rightarrow 14$
4158 independent reflections	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0019P)^2 + 10.979P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.022	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.054	$\Delta\rho_{max}$ = 1.55 e Å^{-3}
S = 1.12	$\Delta\rho_{min}$ = −1.23 e Å^{-3}
4158 reflections	Extinction correction: *SHELXL97*
282 parameters	Extinction coefficient: 0.00067 (8)
H-atom parameters constrained	

All H atoms were placed in calculated positions and refined using a riding model [C—H = 0.98 Å and U_{iso}(H) = 1.5U_{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).

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supporting information

Acta Cryst. (2006). E**62**, m475–m477 [https://doi.org/10.1107/S1600536806004235]

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Crystal data

$[Fe_2Os(C_5H_9N)(CO)_{11}]$
$M_r = 693.14$
Monoclinic, $P2_1/n$
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$a = 11.6861$ (2) Å
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$D_x = 2.296$ Mg m^{-3}
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Cell parameters from 8960 reflections
$\theta = 2.2$–$35.0°$
$\mu = 7.81$ mm^{-1}
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Prism, black
0.3 × 0.2 × 0.1 mm

Data collection

KappaCCD diffractometer
Radiation source: Enraf Nonius FR590
Graphite monochromator
CCD rotation images, thick slices scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{min} = 0.215$, $T_{max} = 0.452$
40723 measured reflections
4158 independent reflections
4019 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 27.6°$, $\theta_{min} = 1.9°$
$h = -15 \rightarrow 15$
$k = -15 \rightarrow 14$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.054$
$S = 1.12$
4158 reflections
282 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
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, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00067 (8)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	1.0863 (4)	0.1792 (4)	0.1082 (3)	0.0125 (8)
C11	0.8759 (4)	0.5245 (4)	0.1518 (3)	0.0140 (8)
C12	0.9341 (4)	0.3731 (3)	0.0368 (3)	0.0132 (8)
C13	0.7239 (4)	0.3506 (4)	0.0697 (3)	0.0162 (9)
C14	1.0483 (4)	0.3761 (4)	0.2118 (3)	0.0145 (8)
C21	1.0292 (4)	0.3552 (3)	0.3803 (3)	0.0134 (8)
C22	1.1308 (4)	0.1910 (4)	0.3052 (3)	0.0152 (8)
C23	0.9238 (4)	0.1467 (4)	0.3302 (3)	0.0166 (9)
C24	0.8239 (4)	0.3338 (3)	0.2418 (3)	0.0142 (8)
C31	0.9743 (4)	−0.0146 (4)	0.1611 (3)	0.0159 (8)
C32	0.8590 (4)	0.1093 (4)	−0.0026 (3)	0.0143 (8)
C33	0.7690 (4)	0.1149 (4)	0.1436 (3)	0.0140 (8)
C100	1.2935 (4)	0.2314 (4)	0.0850 (3)	0.0138 (8)
C101	1.3028 (4)	0.3629 (4)	0.0842 (3)	0.0199 (9)
H10A	1.3025	0.3935	0.143	0.03*
H10B	1.2342	0.3943	0.0365	0.03*
H10C	1.3777	0.3851	0.0726	0.03*
C102	1.3899 (4)	0.1792 (4)	0.1650 (3)	0.0205 (9)
H10D	1.3801	0.0954	0.1644	0.031*
H10E	1.382	0.2104	0.2216	0.031*
H10F	1.4695	0.1981	0.1604	0.031*
C103	1.2950 (4)	0.1802 (4)	−0.0054 (3)	0.0189 (9)
H10G	1.2901	0.0961	−0.0028	0.028*
H10H	1.3696	0.2022	−0.0173	0.028*
H10I	1.2261	0.2095	−0.0539	0.028*
N1	1.1767 (3)	0.2023 (3)	0.0969 (2)	0.0131 (7)
O11	0.8763 (3)	0.6214 (3)	0.1617 (2)	0.0191 (7)
O12	0.9639 (3)	0.3781 (3)	−0.0264 (2)	0.0198 (7)
O13	0.6252 (3)	0.3433 (3)	0.0286 (2)	0.0232 (7)
O14	1.1403 (3)	0.4232 (3)	0.2157 (2)	0.0174 (6)
O21	1.0583 (3)	0.4115 (3)	0.4435 (2)	0.0205 (7)
O22	1.2228 (3)	0.1478 (3)	0.3243 (2)	0.0253 (8)
O23	0.8907 (3)	0.0728 (3)	0.3652 (2)	0.0235 (7)
O24	0.7414 (3)	0.3486 (3)	0.2664 (2)	0.0184 (6)
O31	1.0013 (3)	−0.1066 (3)	0.1831 (3)	0.0268 (8)

O32	0.8180 (3)	0.0910 (3)	−0.0788 (2)	0.0224 (7)
O33	0.6785 (3)	0.0980 (3)	0.1540 (2)	0.0185 (6)
Fe1	0.88075 (5)	0.37081 (5)	0.13486 (4)	0.01044 (12)
Fe2	0.98706 (5)	0.26332 (5)	0.28223 (4)	0.01091 (12)
Os1	0.923356 (15)	0.139606 (12)	0.123351 (11)	0.00966 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.014 (2)	0.0116 (18)	0.0107 (18)	0.0013 (15)	0.0022 (15)	−0.0031 (15)
C11	0.0128 (19)	0.018 (2)	0.0123 (19)	0.0020 (16)	0.0049 (15)	0.0012 (15)
C12	0.014 (2)	0.0104 (18)	0.014 (2)	−0.0002 (15)	0.0028 (16)	0.0004 (15)
C13	0.022 (2)	0.0119 (19)	0.016 (2)	0.0028 (17)	0.0077 (18)	0.0004 (16)
C14	0.020 (2)	0.0130 (19)	0.0090 (18)	0.0076 (16)	0.0017 (16)	−0.0031 (15)
C21	0.015 (2)	0.0121 (18)	0.014 (2)	−0.0020 (15)	0.0045 (16)	0.0034 (16)
C22	0.023 (2)	0.0128 (19)	0.0116 (19)	−0.0019 (17)	0.0080 (16)	−0.0014 (15)
C23	0.014 (2)	0.020 (2)	0.013 (2)	0.0016 (17)	0.0002 (16)	−0.0003 (17)
C24	0.019 (2)	0.0091 (18)	0.0133 (19)	−0.0011 (16)	0.0040 (17)	−0.0006 (15)
C31	0.014 (2)	0.019 (2)	0.017 (2)	−0.0004 (16)	0.0077 (16)	−0.0007 (16)
C32	0.0108 (19)	0.0159 (19)	0.017 (2)	0.0010 (15)	0.0054 (16)	−0.0024 (16)
C33	0.018 (2)	0.0127 (19)	0.0110 (18)	0.0010 (16)	0.0040 (16)	0.0002 (16)
C100	0.0106 (19)	0.0155 (19)	0.018 (2)	0.0006 (15)	0.0081 (16)	−0.0002 (16)
C101	0.020 (2)	0.016 (2)	0.027 (2)	−0.0029 (17)	0.0129 (19)	−0.0004 (18)
C102	0.015 (2)	0.025 (2)	0.020 (2)	0.0023 (18)	0.0027 (17)	0.0034 (18)
C103	0.024 (2)	0.016 (2)	0.020 (2)	0.0002 (17)	0.0131 (19)	−0.0014 (17)
N1	0.0138 (17)	0.0129 (16)	0.0122 (16)	0.0012 (13)	0.0036 (13)	0.0001 (13)
O11	0.0234 (17)	0.0122 (15)	0.0212 (16)	0.0019 (12)	0.0061 (13)	−0.0004 (12)
O12	0.0257 (18)	0.0196 (16)	0.0164 (16)	0.0008 (13)	0.0101 (13)	0.0012 (13)
O13	0.0193 (18)	0.0243 (18)	0.0216 (17)	−0.0002 (14)	0.0001 (14)	−0.0041 (14)
O14	0.0154 (15)	0.0176 (15)	0.0200 (15)	−0.0027 (12)	0.0064 (12)	−0.0011 (12)
O21	0.0239 (17)	0.0217 (16)	0.0166 (16)	−0.0045 (14)	0.0072 (13)	−0.0039 (13)
O22	0.0185 (18)	0.0304 (19)	0.0271 (18)	0.0084 (14)	0.0068 (14)	0.0025 (15)
O23	0.0237 (18)	0.0233 (17)	0.0233 (17)	−0.0058 (14)	0.0069 (14)	0.0051 (14)
O24	0.0170 (16)	0.0226 (16)	0.0186 (16)	0.0032 (13)	0.0100 (13)	0.0019 (13)
O31	0.0277 (19)	0.0151 (16)	0.041 (2)	0.0052 (14)	0.0152 (16)	0.0084 (15)
O32	0.0232 (17)	0.0274 (18)	0.0174 (16)	0.0000 (14)	0.0072 (13)	−0.0046 (14)
O33	0.0151 (15)	0.0218 (16)	0.0202 (16)	−0.0015 (13)	0.0079 (12)	−0.0010 (13)
Fe1	0.0122 (3)	0.0099 (3)	0.0092 (3)	0.0013 (2)	0.0033 (2)	0.0003 (2)
Fe2	0.0128 (3)	0.0102 (3)	0.0096 (3)	0.0007 (2)	0.0032 (2)	0.0006 (2)
Os1	0.00983 (9)	0.00890 (8)	0.01084 (8)	−0.00011 (6)	0.00403 (6)	−0.00070 (6)

Geometric parameters (Å, º)

C1—N1	1.154 (6)	C31—Os1	1.922 (4)
C1—Os1	2.042 (4)	C32—O32	1.150 (5)
C11—O11	1.136 (5)	C32—Os1	1.901 (4)
C11—Fe1	1.807 (4)	C33—O33	1.135 (5)
C12—O12	1.139 (5)	C33—Os1	1.944 (4)

C12—Fe1	1.813 (4)	C100—N1	1.472 (5)
C13—O13	1.138 (6)	C100—C102	1.524 (6)
C13—Fe1	1.817 (5)	C100—C103	1.528 (6)
C14—O14	1.191 (6)	C100—C101	1.531 (6)
C14—Fe1	1.958 (5)	C101—H10A	0.98
C14—Fe2	1.976 (5)	C101—H10B	0.98
C21—O21	1.141 (5)	C101—H10C	0.98
C21—Fe2	1.799 (4)	C102—H10D	0.98
C22—O22	1.140 (6)	C102—H10E	0.98
C22—Fe2	1.814 (5)	C102—H10F	0.98
C23—O23	1.144 (6)	C103—H10G	0.98
C23—Fe2	1.810 (5)	C103—H10H	0.98
C24—O24	1.154 (6)	C103—H10I	0.98
C24—Fe2	1.991 (5)	Fe1—Fe2	2.5675 (8)
C24—Fe1	2.014 (4)	Fe1—Os1	2.7467 (6)
C31—O31	1.136 (5)	Fe2—Os1	2.7522 (6)

N1—C1—Os1	177.9 (4)	C13—Fe1—C24	84.27 (19)
O11—C11—Fe1	177.7 (4)	C14—Fe1—C24	91.44 (18)
O12—C12—Fe1	177.0 (4)	C11—Fe1—Fe2	112.27 (13)
O13—C13—Fe1	176.9 (4)	C12—Fe1—Fe2	123.90 (14)
O14—C14—Fe1	139.8 (4)	C13—Fe1—Fe2	124.31 (14)
O14—C14—Fe2	138.7 (3)	C14—Fe1—Fe2	49.55 (13)
Fe1—C14—Fe2	81.50 (18)	C24—Fe1—Fe2	49.75 (13)
O21—C21—Fe2	178.2 (4)	C11—Fe1—Os1	171.57 (14)
O22—C22—Fe2	176.3 (4)	C12—Fe1—Os1	81.01 (13)
O23—C23—Fe2	175.5 (4)	C13—Fe1—Os1	90.56 (13)
O24—C24—Fe2	140.7 (4)	C14—Fe1—Os1	84.42 (12)
O24—C24—Fe1	139.5 (4)	C24—Fe1—Os1	87.53 (12)
Fe2—C24—Fe1	79.74 (17)	Fe2—Fe1—Os1	62.273 (18)
O31—C31—Os1	178.2 (4)	C21—Fe2—C23	97.84 (19)
O32—C32—Os1	178.8 (4)	C21—Fe2—C22	96.4 (2)
O33—C33—Os1	178.2 (4)	C23—Fe2—C22	92.48 (19)
N1—C100—C102	106.8 (3)	C21—Fe2—C14	91.34 (18)
N1—C100—C103	107.3 (3)	C23—Fe2—C14	170.81 (19)
C102—C100—C103	112.3 (4)	C22—Fe2—C14	86.81 (18)
N1—C100—C101	107.6 (3)	C21—Fe2—C24	90.61 (19)
C102—C100—C101	111.5 (4)	C23—Fe2—C24	88.0 (2)
C103—C100—C101	111.1 (4)	C22—Fe2—C24	172.84 (18)
C100—C101—H10A	109.5	C14—Fe2—C24	91.58 (18)
C100—C101—H10B	109.5	C21—Fe2—Fe1	113.43 (13)
H10A—C101—H10B	109.5	C23—Fe2—Fe1	125.69 (14)
C100—C101—H10C	109.5	C22—Fe2—Fe1	124.50 (13)
H10A—C101—H10C	109.5	C14—Fe2—Fe1	48.95 (13)
H10B—C101—H10C	109.5	C24—Fe2—Fe1	50.51 (13)
C100—C102—H10D	109.5	C21—Fe2—Os1	174.99 (13)
C100—C102—H10E	109.5	C23—Fe2—Os1	86.86 (14)
H10D—C102—H10E	109.5	C22—Fe2—Os1	85.08 (13)

C100—C102—H10F	109.5	C14—Fe2—Os1	83.95 (12)
H10D—C102—H10F	109.5	C24—Fe2—Os1	87.82 (12)
H10E—C102—H10F	109.5	Fe1—Fe2—Os1	62.058 (18)
C100—C103—H10G	109.5	C32—Os1—C31	97.42 (18)
C100—C103—H10H	109.5	C32—Os1—C33	92.55 (18)
H10G—C103—H10H	109.5	C31—Os1—C33	91.67 (18)
C100—C103—H10I	109.5	C32—Os1—C1	90.72 (17)
H10G—C103—H10I	109.5	C31—Os1—C1	91.88 (17)
H10H—C103—H10I	109.5	C33—Os1—C1	174.83 (17)
C1—N1—C100	178.5 (4)	C32—Os1—Fe1	103.04 (13)
C11—Fe1—C12	98.15 (18)	C31—Os1—Fe1	159.51 (13)
C11—Fe1—C13	97.87 (19)	C33—Os1—Fe1	86.44 (12)
C12—Fe1—C13	94.71 (19)	C1—Os1—Fe1	88.92 (11)
C11—Fe1—C14	87.18 (18)	C32—Os1—Fe2	158.70 (13)
C12—Fe1—C14	88.53 (19)	C31—Os1—Fe2	103.87 (13)
C13—Fe1—C14	173.54 (19)	C33—Os1—Fe2	86.67 (13)
C11—Fe1—C24	93.35 (17)	C1—Os1—Fe2	88.83 (11)
C12—Fe1—C24	168.48 (18)	Fe1—Os1—Fe2	55.669 (17)

O14—C14—Fe1—C11	−56.5 (5)	Os1—Fe1—Fe2—C14	107.68 (15)
Fe2—C14—Fe1—C11	122.73 (17)	C11—Fe1—Fe2—C24	74.7 (2)
O14—C14—Fe1—C12	41.7 (5)	C12—Fe1—Fe2—C24	−167.9 (2)
Fe2—C14—Fe1—C12	−139.04 (17)	C13—Fe1—Fe2—C24	−42.6 (2)
O14—C14—Fe1—C24	−149.8 (5)	C14—Fe1—Fe2—C24	139.9 (2)
Fe2—C14—Fe1—C24	29.44 (16)	Os1—Fe1—Fe2—C24	−112.40 (16)
O14—C14—Fe1—Fe2	−179.2 (6)	C11—Fe1—Fe2—Os1	−172.90 (14)
O14—C14—Fe1—Os1	122.8 (5)	C12—Fe1—Fe2—Os1	−55.53 (16)
Fe2—C14—Fe1—Os1	−57.93 (9)	C13—Fe1—Fe2—Os1	69.81 (16)
O24—C24—Fe1—C11	61.8 (5)	C14—Fe1—Fe2—Os1	−107.68 (15)
Fe2—C24—Fe1—C11	−116.61 (17)	C24—Fe1—Fe2—Os1	112.40 (15)
O24—C24—Fe1—C12	−121.2 (9)	C12—Fe1—Os1—C32	−43.66 (19)
Fe2—C24—Fe1—C12	60.4 (10)	C13—Fe1—Os1—C32	51.02 (19)
O24—C24—Fe1—C13	−35.8 (5)	C14—Fe1—Os1—C32	−133.05 (18)
Fe2—C24—Fe1—C13	145.81 (17)	C24—Fe1—Os1—C32	135.26 (18)
O24—C24—Fe1—C14	149.0 (5)	Fe2—Fe1—Os1—C32	−179.81 (13)
Fe2—C24—Fe1—C14	−29.35 (16)	C12—Fe1—Os1—C31	139.3 (4)
O24—C24—Fe1—Fe2	178.4 (6)	C13—Fe1—Os1—C31	−126.0 (4)
O24—C24—Fe1—Os1	−126.6 (5)	C14—Fe1—Os1—C31	49.9 (4)
Fe2—C24—Fe1—Os1	55.00 (10)	C24—Fe1—Os1—C31	−41.8 (4)
O14—C14—Fe2—C21	58.8 (5)	Fe2—Fe1—Os1—C31	3.2 (4)
Fe1—C14—Fe2—C21	−120.46 (17)	C12—Fe1—Os1—C33	−135.45 (19)
O14—C14—Fe2—C22	−37.5 (5)	C13—Fe1—Os1—C33	−40.77 (19)
Fe1—C14—Fe2—C22	143.21 (17)	C14—Fe1—Os1—C33	135.16 (18)
O14—C14—Fe2—C24	149.4 (5)	C24—Fe1—Os1—C33	43.46 (18)
Fe1—C14—Fe2—C24	−29.81 (16)	Fe2—Fe1—Os1—C33	88.39 (13)
O14—C14—Fe2—Fe1	179.2 (6)	C12—Fe1—Os1—C1	46.83 (18)
O14—C14—Fe2—Os1	−122.9 (5)	C13—Fe1—Os1—C1	141.51 (18)
Fe1—C14—Fe2—Os1	57.83 (10)	C14—Fe1—Os1—C1	−42.56 (17)

O24—C24—Fe2—C21	−57.9 (6)	C24—Fe1—Os1—C1	−134.25 (17)
Fe1—C24—Fe2—C21	120.42 (16)	Fe2—Fe1—Os1—C1	−89.32 (11)
O24—C24—Fe2—C23	39.9 (6)	C12—Fe1—Os1—Fe2	136.15 (14)
Fe1—C24—Fe2—C23	−141.76 (17)	C13—Fe1—Os1—Fe2	−129.17 (14)
O24—C24—Fe2—C14	−149.3 (5)	C14—Fe1—Os1—Fe2	46.76 (13)
Fe1—C24—Fe2—C14	29.06 (16)	C24—Fe1—Os1—Fe2	−44.93 (13)
O24—C24—Fe2—Fe1	−178.4 (6)	C23—Fe2—Os1—C32	134.2 (4)
O24—C24—Fe2—Os1	126.8 (5)	C22—Fe2—Os1—C32	−133.1 (4)
Fe1—C24—Fe2—Os1	−54.82 (10)	C14—Fe2—Os1—C32	−45.8 (4)
C11—Fe1—Fe2—C21	4.7 (2)	C24—Fe2—Os1—C32	46.1 (4)
C12—Fe1—Fe2—C21	122.1 (2)	Fe1—Fe2—Os1—C32	0.5 (3)
C13—Fe1—Fe2—C21	−112.6 (2)	C23—Fe2—Os1—C31	−45.19 (19)
C14—Fe1—Fe2—C21	69.9 (2)	C22—Fe2—Os1—C31	47.57 (19)
C24—Fe1—Fe2—C21	−70.0 (2)	C14—Fe2—Os1—C31	134.88 (18)
Os1—Fe1—Fe2—C21	177.59 (15)	C24—Fe2—Os1—C31	−133.30 (18)
C11—Fe1—Fe2—C23	124.3 (2)	Fe1—Fe2—Os1—C31	−178.86 (13)
C12—Fe1—Fe2—C23	−118.3 (2)	C23—Fe2—Os1—C33	45.70 (19)
C13—Fe1—Fe2—C23	7.0 (2)	C22—Fe2—Os1—C33	138.47 (18)
C14—Fe1—Fe2—C23	−170.5 (2)	C14—Fe2—Os1—C33	−134.23 (18)
C24—Fe1—Fe2—C23	49.6 (2)	C24—Fe2—Os1—C33	−42.40 (17)
Os1—Fe1—Fe2—C23	−62.79 (18)	Fe1—Fe2—Os1—C33	−87.96 (12)
C11—Fe1—Fe2—C22	−111.7 (2)	C23—Fe2—Os1—C1	−136.84 (19)
C12—Fe1—Fe2—C22	5.6 (2)	C22—Fe2—Os1—C1	−44.08 (17)
C13—Fe1—Fe2—C22	131.0 (2)	C14—Fe2—Os1—C1	43.23 (17)
C14—Fe1—Fe2—C22	−46.5 (2)	C24—Fe2—Os1—C1	135.05 (17)
C24—Fe1—Fe2—C22	173.6 (2)	Fe1—Fe2—Os1—C1	89.49 (12)
Os1—Fe1—Fe2—C22	61.16 (17)	C23—Fe2—Os1—Fe1	133.67 (15)
C11—Fe1—Fe2—C14	−65.2 (2)	C22—Fe2—Os1—Fe1	−133.57 (13)
C12—Fe1—Fe2—C14	52.2 (2)	C14—Fe2—Os1—Fe1	−46.26 (13)
C13—Fe1—Fe2—C14	177.5 (2)	C24—Fe2—Os1—Fe1	45.56 (12)
C24—Fe1—Fe2—C14	−139.9 (2)		