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XXXXIV *. X-RAY STRUCTURES OF [Ru$_3$(CO)$_{11}$(CNBu^t)] (AT 130 K) AND [Os$_3$(CO)$_{11}$(CNBu^t)] (AT 295 K)

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Summary

Single crystal X-ray structure determinations are reported for [Os$_3$(CO)$_{11}$(CNBu^t)] at 295 K and for [Ru$_3$(CO)$_{11}$(CNBu^t)] at ~ 130 K; both structures are isomorphous with the previously determined room-temperature structure for the latter, which exhibited ~ 14% disorder in the Ru$_3$ core. Residuals for the present studies were 0.042 and 0.033 for 4491 and 6072 observed reflections respectively. The osmium complex is fully ordered; disorder in the ruthenium complex is reduced to ~ 6% at low temperature, indicating the disorder to be dynamic rather than a consequence of packing error.

Introduction

Three structural types are known for the M$_3$(CO)$_{12}$ Group VIII metal carbonyls and their derivatives with 2e-donor ligands. These are typified by [Fe$_3$(CO)$_{12}$] (two CO groups bridging one M–M bond) [2], [Ru$_3$(CO)$_{12}$] (all terminal CO groups) [3] and [FeRu$_2$(CO)$_{10}$(PPh$_3$)$_2$] (two CO groups each semibridging a Fe–Ru bond) [4]. The molecular structure of [Fe$_3$(CO)$_{11}$(CNBu^t)] is of the [Fe$_3$(CO)$_{12}$]-type with the isocyanide ligand axial on the unique Fe atom [5]. The analogous ruthenium complex adopts the [Ru$_3$(CO)$_{12}$] structure, again with an axial CNBu^t substituent, but the Ru$_3$ core exhibits ~ 14% disorder [6]. As part of an on-going study of the

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* For Part XXXXIII, see ref. 1.

influence of various ligands on the structures of derivatives of this type [5–9], we have determined the structure of $[Os_3(CO)_{11}(CNBu^t)]$ by single crystal X-ray methods, and also extended the study of the ruthenium complex to encompass a determination at low temperature in an attempt to ascertain whether the disorder observed at room temperature is structural (in the sense of being frozen into the crystal lattice during the crystallization process) or whether it remains dynamic even in the solid state. The results are reported hereunder.

Results and discussion

The complex $[Os_3(CO)_{11}(CNBu^t)]$ (**1**) was first described by Mays and Gavens in 1980 [10], but an X-ray structural characterisation has not been reported subsequently. The solution behaviour of this complex indicated that at $-60°C$, both the axial and equatorial isomers are present. The former is present in major amount; a limiting ^{13}C NMR spectrum was not obtained for the equatorial isomer. At $50°C$, only a single ^{13}C carbonyl resonance was present, showing that rapid interconversion (on the NMR time scale) of the axial and equatorial isomers occurs at this temperature. The ruthenium analogue (**2**) is also fluxional in solution at $-100°C$, all ^{13}CO resonances appearing as a sharp singlet at this temperature [11].

The results of the present structure determinations are consistent with the stoichiometry expected; the two complexes are isomorphous with each other and with the room temperature determination for the ruthenium complex. Subtle variations are found in respect of the fact that the osmium complex is a totally ordered structure, whereas the Ru_3 core of the ruthenium complex is disordered and to a different degree to that of the room temperature determination, the degree of disorder reverting to 14% on return of the crystal to room temperature.

A molecule of the osmium complex is shown in Fig. 1a, and the unit cell contents are depicted in Fig. 1b; it is evident from consideration of this Figure and the atom coordinates that the crystal array may be regarded as a superlattice on a $P2_1$ structure. The unit cell contains discrete molecules of the complex, which contains an Os_3 triangle, in which the isocyanide ligand occupies a terminal site; the eleven CO groups are all terminal. The average Os–Os separation is 2.875 Å, about 0.02 Å longer than found for the ruthenium analogue; in $[M_3(CO)_{12}]$, values of 2.877 (M = Os [12]) and 2.854(1) Å (M = Ru [3]) were found. In both osmium and ruthenium complexes the longer M–M bond is within the $M_2(CO)_8$ fragment. A comparison can also be made with $[Os_3(CO)_{11}(NCMe)]$, in which the average Os–Os distance is 2.870 Å; again the longest Os–Os separation [2.894(2) Å] is associated with the $Os_2(CO)_8$ unit [13]. The Os–CO distances range between 1.91–1.95(1) Å and the Os–$CNBu^t$ distance is 2.03(1) Å; there are no significant differences between these values and those found for the ruthenium analogue. The twisting of the $M(CO)_4$ groups about the M(2)–M(3) bond is comparable, and is seen as a perturbation of the $(CO)_{12}$ anticuboctahedron resulting from replacement of one CO by the isocyanide ligand.

As mentioned above, the Ru_3 core in $[Ru_3(CO)_{11}(CNBu^t)]$ at 295 K is disordered to the extent of ca. 14% within the ligand array [6]. We noted also that in this complex, as well as other derivatives of $[Ru_3(CO)_{12}]$ containing 2e-donors, such as tertiary phosphines or phosphites, a peripheral atom polyhedron (p.a.p.), constructed from appropriate ligand atoms [O of CO, N of CNR, P of PR_3 or $P(OR)_3$] remains

essentially unaltered about the disordered metal core. It was of interest to determine whether the disorder is a kinetic phenomenon, in which case the populations of the two forms should vary with temperature. Accordingly we have redetermined the structure of **2** at 130 K. The results of this study show that the degree of disorder of the Ru_3 core is significantly different at the two temperatures of the determinations, indicating that the disorder is a dynamic phenomenon; the time scale of this dynamic process cannot be determined at present. In the present study, we have shown (Experimental) that the same crystal exhibits an increase in the disorder of the Ru_3 core on heating from 130 to 295 K, thus not only confirming our previous result, but showing that a real change occurs.

Insofar as these determinations allow a determination of the equilibrium constants of the dynamic process, and the derived thermodynamic properties, our studies go a little way to quantifying the process. However, the lack of any information concerning the rate of this process (which may be available from solid state variable temperature NMR studies in the future) means that no statement can be made about possible barriers to the rotation of the Ru_3 core within the ligand shell. We have previously commented on the reluctance of $CNBu^t$ to adopt a position bridging two metal atoms: the lack of disorder in $Fe_3(CO)_{11}(CNBu^t)$ in the solid state was attributed to the reluctance of $CNBu^t$ to take the place of one of the two μ-CO ligands [5]. Although examination of the structure of the minor component of **2** reveals that in this, the higher energy isomer, the $CNBu^t$ ligand adopts a position bridging the two metal atoms, this may be an artefact of our inability to meaningfully deconvolute the disordered components of the coordinating atoms.

Experimental

The osmium complex was obtained from a reaction between $CNBu^t$ and $[Os_3(CO)_{11}(NCMe)]$ and was identified by comparison of its infrared $\nu(CO)$ spectrum with that reported by Mays and Gavens [10]; the ruthenium derivative was prepared by the sodium diphenylketyl-initiated reaction between $CNBu^t$ and $[Ru_3(CO)_{12}]$ [14], and was crystallised from light petroleum as in the previous study.

Crystallography

Crystal data. **1.** $C_{16}H_9NO_{11}Os_3$, $M = 961.9$, Monoclinic, space group $P2_1/c$ (C_{2h}^5, No.14), a 11.862(5), b 11.900(5), c 16.692(7) Å, β 112.58(2)°, U 2176(2) Å^3. D_c ($Z = 4$) 2.94 g cm^{-3}, $F(000) = 1712$. μ_{Mo} 169 cm^{-1}. Specimen: $0.16 \times 0.40 \times 0.22$ mm. $2\theta_{max}$ 65°. $N = 7235$, $N_0 = 4491$. $R = 0.042$, $R' = 0.047$. T 295 K.

2. $C_{16}H_9NO_{11}Ru_3$, $M = 694.5$, Monoclinic, space group $P2_1/c$, a 11.811(7), b 11.780(7), c 16.401(13) Å, β 112.75(5)°, U 2104(2) Å^3. D_c ($Z = 4$) 2.19 g cm^{-3}. $F(000) = 1328$. μ_{Mo} 20.1 cm^{-1}. Specimen: $0.22 \times 0.25 \times 0.31$mm. $2\theta_{max}$ 70°C. $N = 7347$, $N_0 = 6072$. $R = 0.033$, $R' = 0.037$. $T \sim 130$ K.

Structure determination. Unique data sets were measured within the specified $2\theta_{max}$ limits using a Syntex P2$_1$ four circle diffractometer fitted with monochromatic Mo-K_α radiation source (λ 0.71069 Å) and operating in conventional $2\theta/\theta$ scan mode. N independent reflections were measured, N_0 with $I > 3\sigma(I)$ being considered 'observed' and used in the full matrix least-squares refinement after analytical absorption correction. Refinement was initiated using the parameters of the room temperature ruthenium structure (high population component). Anisotropic thermal
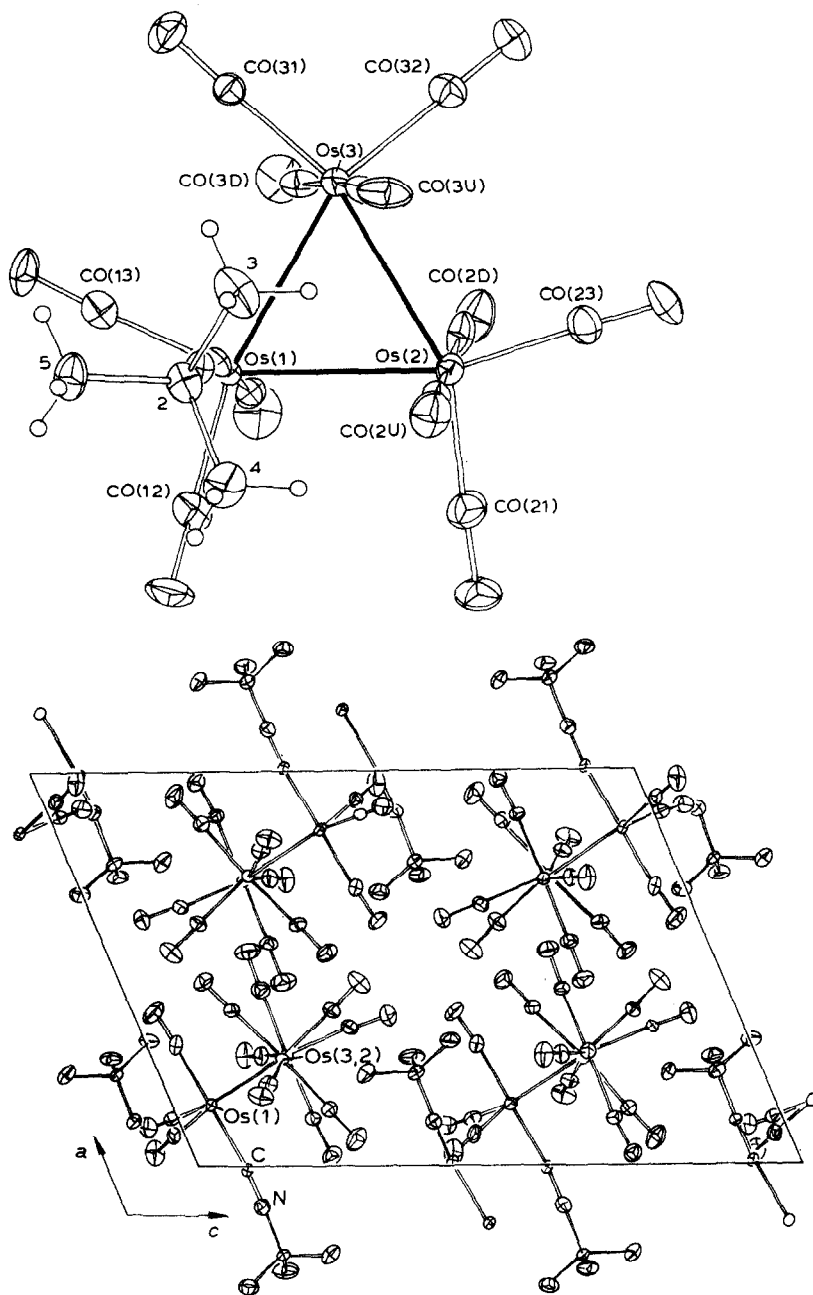
Fig. 1. (a) The osmium complex, projected normal to the Os_3 plane; 20% thermal ellipsoids are shown for the non-hydrogen atoms. Disordered Ru components of the Ru_3 system lie on an equilateral triangle rotated 60° from the parent with Ru(12) lying between Ru(1) and (2) (etc.) [6]. (b) Unit cell contents projected down b.

parameters were refined for the non-hydrogen atoms of both structures; for **1**, $(x, y, z, U_{iso})_H$ were included constrained at estimated values. In **2**, these parameters could be meaningfully refined. Populations of the fragments of the Ru_3 triangle in the latter were refined independently initially. As it became evident that the population of each set of three atoms were, as expected, almost identical, the independent parameters were replaced by a single population, x, for the major triangle fragment and $1 - x$ for the minor fragment. At convergence x, was 0.941(1). Attempts to improve the model by an extension of the description to incorporate fragmented inner ligand atom components similarly were unsuccessful and these were refined ultimately as single units of unit population. After it had become apparent that the population of the Ru_3 molecular core components were indeed greatly different at low temperature from the previously determined room tempera-

TABLE 1

NON-HYDROGEN ATOM COORDINATES FOR 1 (M = Os) AND 2 (M = Ru)

Atom	**1**			**2** [a]		
	x	y	z	x	y	z
M(1)	0.15161(3)	0.24558(3)	0.06098(2)	0.15219(2)	0.24567(2)	0.06039(2)
M(2)	0.27828(4)	0.13616(3)	0.22469(2)	0.28175(2)	0.13656(2)	0.22556(2)
M(3)	0.26896(4)	0.37820(3)	0.21400(2)	0.27590(2)	0.37890(2)	0.21367(2)
C(1)	−0.0086(9)	0.2593(9)	0.0772(6)	−0.0088(3)	0.2625(3)	0.0781(2)
N(1)	−0.1065(8)	0.2630(7)	0.0768(6)	−0.1059(3)	0.2642(2)	0.0781(2)
C(2)	−0.2313(10)	0.2570(10)	0.0748(7)	−0.2291(3)	0.2590(3)	0.0786(2)
C(3)	−0.2296(14)	0.3177(15)	0.1568(9)	−0.2242(4)	0.3165(4)	0.1637(3)
C(4)	−0.2680(12)	0.1343(12)	0.0705(10)	−0.2648(9)	0.1350(3)	0.0767(3)
C(5)	−0.3111(12)	0.3192(13)	−0.0061(9)	−0.3159(3)	0.3214(3)	−0.0033(2)
C(1D)	0.3015(12)	0.2154(9)	0.0464(7)	0.3019(4)	0.2111(3)	0.0457(2)
O(1D)	0.3858(9)	0.1915(10)	0.0321(6)	0.3849(2)	0.1858(2)	0.0292(2)
C(12)	0.0754(12)	0.1287(11)	−0.0195(7)	0.0713(4)	0.1272(3)	−0.0204(2)
O(12)	0.0291(11)	0.0578(8)	−0.0675(6)	0.0215(3)	0.0608(3)	−0.0711(2)
C(13)	0.1118(11)	0.3712(10)	−0.0151(7)	0.1161(3)	0.3745(3)	−0.0160(2)
O(13)	0.0941(9)	0.4478(8)	−0.0597(6)	0.0979(3)	0.4491(2)	−0.0633(2)
C(2U)	0.1139(10)	0.1169(9)	0.2240(7)	0.1171(3)	0.1162(3)	0.2225(2)
O(2U)	0.0230(8)	0.0976(8)	0.2270(5)	0.0276(2)	0.0950(2)	0.2282(2)
C(2D)	0.4398(11)	0.1669(10)	0.2267(7)	0.4443(4)	0.1706(3)	0.2281(2)
O(2D)	0.5390(9)	0.1761(10)	0.2317(6)	0.5443(2)	0.1795(2)	0.2356(2)
C(21)	0.2743(13)	−0.0119(10)	0.1787(8)	0.2789(3)	−0.0130(3)	0.1783(2)
O(21)	0.2752(11)	−0.0998(8)	0.1524(6)	0.2824(3)	−0.1016(2)	·0.1522(2)
C(23)	0.3454(10)	0.1100(10)	0.3478(6)	0.3463(3)	0.1122(3)	0.3515(2)
O(23)	0.3790(9)	0.0925(9)	0.4201(5)	0.3767(2)	0.0920(2)	0.4247(2)
C(3U)	0.1358(11)	0.3542(10)	0.2545(7)	0.1458(4)	0.3488(3)	0.2555(2)
O(3U)	0.0622(10)	0.3432(9)	0.2801(6)	0.0734(3)	0.3409(2)	0.2848(2)
C(3D)	0.3920(11)	0.3961(10)	0.1642(7)	0.3958(3)	0.3947(3)	0.1604(2)
O(3D)	0.4653(9)	0.4108(9)	0.1381(6)	0.4684(2)	0.4137(2)	0.1331(2)
C(31)	0.2072(11)	0.5259(10)	0.1737(7)	0.2104(3)	0.5265(3)	0.1707(2)
O(31)	0.1742(9)	0.6132(7)	0.1527(6)	0.1720(3)	0.6147(2)	0.1496(2)
C(32)	0.3882(12)	0.4110(10)	0.3298(7)	0.3959(3)	0.4137(3)	0.3302(2)
O(32)	0.4589(10)	0.4314(9)	0.3946(5)	0.4701(3)	0.4374(2)	0.3964(2)

[a] For **2**: Ru(12): 0.2251(5), 0.1298(4), 0.1225(3)
Ru(13): 0.1906(5), 0.3666(4), 0.1172(3)
Ru(23): 0.3336(5), 0.2579(4), 0.2797(3)

TABLE 2

MOLECULAR CORE GEOMETRY FOR 1 AND 2 (N refers to the cyclic permutation 1, 2, 3 (note that for $N = 1$, '$N-1$' is 3; for $N = 3$, '$N+1$' is 1. For C(1U) read C(1)). The values in each entry are for 1 (M = Os), 2 (M = Ru) respectively, followed by the value for the room temperature determination of 2)

	$N = 1$	$N = 2$	$N = 3$
Distances (Å)			
M(N)–M($N+1$)	2.875(1), 2.851(2), 2.8575(8);	2.885(1), 2.860(2), 2.8668(11);	2.867(1), 2.842(2), 2.8477(8)
M(N)–C(NU)	2.03(1), 2.042(4), 2.041(5);	1.96(1), 1.941(4), 1.940(4);	1.96(1), 1.942(5), 1.949(6)
M(N)–C(ND)	1.92(2), 1.919(4), 1.940(6);	1.94(1), 1.946(4), 1.961(5);	1.95(1), 1.938(5), 1.938(6)
M(N)–C($NN+1$)	1.90(1), 1.908(3), 1.892(4);	1.92(1), 1.927(4), 1.943(4);	1.92(1), 1.923(3), 1.923(4)
M(N)–C($NN-1$)	1.90(1), 1.909(3), 1.917(4);	1.91(1), 1.919(4), 1.942(5);	1.95(1), 1.932(3), 1.950(4)
C(NU)–O(NU)		1.12(2), 1.124(5), 1.101(6);	1.12(2), 1.136(6), 1.131(7)
C(ND)–O(ND)	1.15(2), 1.152(5), 1.149(7);	1.15(2), 1.144(5), 1.123(6);	1.13(2), 1.134(5), 1.115(7)
C($NN-1$)–O($NN-1$)	1.14(1), 1.136(4), 1.118(6);	1.14(2), 1.136(5), 1.126(6);	1.11(1), 1.136(4), 1.111(5)
C($NN+1$)–O($NN+1$)	1.15(1), 1.127(4), 1.108(5);	1.14(1), 1.137(4), 1.127(5);	1.12(1), 1.133(4), 1.127(5)
Ru($NN+1$)–Ru($N-1N$)	2.816(7), 2.831(3);	2.830(7), 2.853(3);	2.849(6), 2.856(3)
Ru(N)–Ru($NN+1$)	1.722(5), 1.746(2);	1.669(5), 1.665(2);	1.519(5), 1.537(2)
Ru(N)–Ru($N-1N$)	1.666(5), 1.646(2);	1.561(5), 1.563(2);	1.760(5), 1.767(2)
Angles (°)			
M($N-1$)–M(N)–M($N+1$)	60.32(4), 60.33(5), 60.33(2);	59.70(2), 59.69(2), 59.67(1);	59.98(3), 59.99(3), 60.00(2)
M($N-1$)–M(N)–C(NU)	89.8(3), 90.5(1), 89.3(1);	83.8(3), 82.3(1), 80.3(1);	81.4(4), 78.2(1), 77.2(1)
M($N-1$)–M(N)–C(ND)	93.4(3), 92.6(1), 92.7(1);	94.8(3), 95.2(1), 96.5(1);	96.8(3), 97.1(1), 97.0(1)
M($N-1$)–M(N)–C($NN+1$)	165.3(4), 165.2(1), 164.3(1);	160.0(4), 158.5(1), 157.4(1);	159.1(3), 157.4(1), 156.0(1)
M($N-1$)–M(N)–C($NN-1$)	94.2(3), 92.8(1), 92.7(1);	96.4(3), 96.4(1), 96.2(1);	98.2(4), 98.9(1), 98.9(1)
M($N+1$)–M(N)–C(NU)	94.8(3), 95.5(1), 96.5(1);	95.9(3), 97.1(1), 97.1(1);	92.7(3), 90.3(1), 91.1(1)

M(N+1)–M(N)–C(ND)	82.2(3), 80.9(1), 79.0(1);	80.0(4), 78.1(1), 78.1(1);	82.4(3), 82.5(1), 82.1(10
M(N+1)–M(N)–C(NN+1)	105.8(3), 105.7(1), 105.0(1);	102.6(3), 102.2(1), 100.9(1);	101.6(3), 100.3(1), 98.7(1)
M(N+1)–M(N)–C(NN−1)	153.7(3), 152.1(1), 151.6(1);	154.0(4), 153.2(1), 152.8(2);	156.0(4), 157.1(1), 157.3(1)
C(NU)–M(N)–C(ND)	173.8(4), 173.3(1), 173.3(2);	175.8(5), 175.2(1), 175.1(2);	175.0(4), 172.7(1), 172.6(2)
C(NU)–M(N)–C(NN−1)	91.2(5), 91.9(2), 91.5(2);	91.1(6), 90.9(2), 90.3(2);	93.5(5), 93.9(2), 92.0(2)
C(NU)–M(N)–C(NN+1)	86.4(5), 85.8(2), 86.8(2);	89.4(5), 89.2(1), 91.8(2);	90.3(6), 91.9(2), 93.4(2)
C(ND)–M(N)–C(NN−1)	93.9(5), 94.0(2), 94.8(2);	93.0(6), 93.5(2), 93.8(2);	91.4(5), 92.3(2), 93.3(2)
C(ND)–M(N)–C(NN+1)	89.2(6), 89.8(2), 89.5(2);	90.7(5), 91.7(2), 89.8(2);	89.9(5), 90.5(2), 90.2(2)
C(NN−1)–M(N)–C(NN+1)	100.1(5), 101.6(1), 102.0(8);	102.5(5), 103.5(1), 105.1(2);	101.4(5), 102.0(1), 103.6(2)
M(N)–C(NU)–O(NU)		174(1), 171.8(3), 169.1(3);	177(1), 173.0(3), 171.0(4)
M(N)–C(ND)–O(ND)	174(1), 173.5(3), 172.6(3);	174(1), 172.0(3), 170.3(3);	177(1), 173.5(3), 172.2(4)
M(N)–C(NN−1)–O(NN−1)	176(1), 176.9(4), 173.1(4);	178(1), 177.1(3), 173.8(3);	177(1), 176.2(4), 173.2(4)
M(N)–C(NN+1)–O(NN+1)	179(1), 176.8(3), 174.4(4);	176(1), 174.5(3), 172.8(4);	178(1), 176.4(4), 173.1(4)
Ru(NN−1)–Ru(NN+1)– Ru(N+1 N+2)	60.6(2), 60.33(7);	59.4(2), 59.45(7);	60.0(2), 60.22(7)

Ligand: Distances (Å)

C(1)–N(1)	1.16(2), 1.147(5), 1.139(6)		
N(1)–C(2)	1.47(2), 1.459(5), 1.465(6)		
C(2)–C(3)	1.54(2), 1.533(6), 1.500(8)		
C(2)–C(4)	1.52(2), 1.518(5), 1.505(7)		
C(2)–C(5)	1.51(2), 1.527(5), 1.515(6)		

Angles (°)

M–C(1)–N(1)	172.3(8), 171.1(3), 171.4(3)
C(1)–N(1)–C(2)	175(1), 176.7(3), 174.1(4)
N(1)–C(2)–C(3)	107(1), 107.5(3), 107.8(3)
N(1)–C(2)–C(4)	108(1), 108.1(3), 108.3(4)
N(1)–C(2)–C(5)	106(1), 107.9(3), 107.6(4)
C(3)–C(2)–C(4)	114(1), 110.9(3), 110.8(5)
C(3)–C(2)–C(5)	111(1), 111.4(3), 112.1(4)
C(4)–C(2)–C(5)	111(1), 111.0(3), 110.0(4)

ture values (~ 1 month), the same crystal was remounted in similar orientation on the same diffractometer and a new room temperature data set remeasured and processed as described previously. Refinement converged with atom parameters (including populations) not non-trivially different from those of the earlier room temperature experiment; accordingly they are not recorded. The osmium structure is fully ordered and refined normally. Residuals quoted at convergence are conventional R, R'. Neutral complex scattering factors were used [15]; computation used the XTAL 83 program system [16], implemented on a Perkin–Elmer 3240 computer by S.R. Hall. Non-hydrogen atom numbering follows that previously defined for the room temperature ruthenium study [6], and is shown in Fig. 1a.

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