

and two with Pt^{II} (Spagna & Zambonelli, 1973; Dubey, 1976), but there is a considerable discrepancy concerning the important C≡C bond lengths in these latter two, chemically very similar, compounds.

Fig. 2 shows the unit cell of compound (2). There are no close contacts between the individual Ni atoms and/or alkynes which could be interpreted in terms of initial steps of the catalytic reaction discussed above.

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References

- DICKSON, R. S. & IBERS, J. A. (1972). *J. Organomet. Chem.* **36**, 191–207.
 DIECK, H. TOM, LAUER, A. & DIERCKS, R. (1983). *J. Mol. Catal.* Submitted.
 DIECK, H. TOM, SVOBODA, M. & GREISER, T. (1981). *Z. Naturforsch. Teil B*, **36**, 823–832.
 DIECK, H. TOM, SVOBODA, M. & KOPF, J. (1978). *Z. Naturforsch. Teil B*, **33**, 1381–1385.
 DIERCKS, R. & TOM DIECK, H. (1983). *Chem. Ber.* Submitted.
 DUBEY, R. J. (1975). *Acta Cryst.* **B31**, 1860–1864.
 DUBEY, R. J. (1976). *Acta Cryst.* **B32**, 199–202.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
 JOLLY, P. W. (1982). *Comprehensive Organometallic Chemistry*, Vol. 8, ch. 56.3, edited by G. WILKINSON, F. G. A. STONE & E. W. ABEL, pp. 649–670. London: Pergamon Press.
 JOLLY, P. W. & WILKE, G. (1975). *The Organic Chemistry of Nickel*, Vol. 2, ch. 2, pp. 94–132. New York: Academic Press.
 REPPE, W., SCHLICHTING, O., KLAGER, K. & TOEPEL, T. (1948). *Justus Liebigs Ann. Chem.* **560**, 1–92.
 SHELDRIK, G. M. (1977). *SHELX77*. Program for crystal structure determination. Univ. of Cambridge, England.
 SPAGNA, R. & ZAMBONELLI, L. (1973). *Acta Cryst.* **B29**, 2302–2304.
 WILKE, G. (1978). *Pure Appl. Chem.* **50**, 677–688.

Acta Cryst. (1984). **C40**, 365–368

Structure of Octadecacarbonyl-1,2,3:4,5,6-bis- μ_3 [(η -toluene)cuprio]-octahedro-hexaruthenium, (C₆H₅CH₃)₂Cu₂Ru₆(CO)₁₈

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Abstract. $M_r = 1421.9$, triclinic, $P1$, $a = 10.236$ (2), $b = 10.395$ (3), $c = 19.267$ (2) Å, $\alpha = 82.64$ (1), $\beta = 75.27$ (1), $\gamma = 82.44$ (2)°, $V = 1955.9$ Å³, $Z = 2$, $D_x = 2.415$ Mg m⁻³, D_m not measured, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 67.52$ mm⁻¹, $F(000) = 2696$, $T = 293$ K. Final $R = 0.0377$ for 4357 observed independent reflections. Two essentially similar but independent metal–carbonyl clusters contain Ru₆ octahedra capped by two Cu atoms on opposite trigonal faces. Three terminal carbonyl ligands are bound to each Ru atom, and a toluene molecule is η -bonded to each Cu atom. Cu–Ru distances average 2.66 (6) Å. Ru–Ru distances range from 2.87 to 2.94 Å.

Introduction. The title compound has an identical Cu₂Ru₆ core stoichiometry to that found in (CH₃CN)₂Cu₂Ru₆C(CO)₁₆ (Bradley, Pruett, Hill, Ansell, Leonowicz & Modrick, 1982; Ansell, Leonowicz, Modrick & Bradley, 1981), and might be expected to

have a similar structure. We report here that in fact the cluster has a radically different geometry to its carbide-containing analog.

Experimental. Material synthesized by reacting [PPh₃]₂N][Ru₆(CO)₁₈] with [Cu(CH₃CN)₄][BF₄] in dry acetone; after extraction from evaporated reaction solution with methylene chloride, crude product recrystallized from warm toluene to give crystals suitable for X-ray examination. Crystal with dimensions 0.20 × 0.19 × 0.23 mm mounted in a thin-walled Lindemann capillary tube, Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation monochromated by a graphite crystal in incident beam. Indexing on 25 centered reflections with intensities ranging from weak to very intense gave lattice parameters. Total of 7092 reflections measured in $\theta/2\theta$ scan mode, $0 < \theta < 25^\circ$, range of hkl : 0–12, –12–12, –20–22; scan begun 0.75° below calculated 2θ position for $K\alpha_1$, and

terminated 0.75° above calculated $K\alpha_2$ angle; total background counting time half scan time and evenly divided on either side of the peak. 4357 independent reflections regarded as observed with $I > 3.0\sigma(F)^2$ where $I = S(C - RB)$ and $\sigma(F)^2 = [S^2(C + R^2B) + (kI)^2]^{1/2}$, where C = total counts recorded during scan, $R = 2.0$ = ratio of scanning time to total background B , S = scan rate (varied between 4° and 20° min⁻¹) and $k = 0.05$, a factor introduced to reflect instrumental stability. From these data structure factors and their e.s.d.'s computed using $|F_o| = (I/Lp)^{1/2}$ and $\sigma(F_o) = \sigma(I)/2|F_o|Lp$, where Lp is the Lorentz-polarization correction. Three reflections checked

periodically during data collection showed no substantial decrease in intensity. Corrections for absorption not made since ψ -scan data showed effect to be small. Positions of Cu and Ru atoms found using direct methods (*SDP*; Enraf-Nonius, 1980) on the 11/60 computer; remaining non-H atoms located by subsequent difference Fourier calculations, alternated with cycles of least-squares refinement. During latter stages of refinement it was found that the C atoms in the two toluene molecules associated with one Cu₂Ru₆ cluster gave large parameter shift/e.s.d. ratios which prevented the refinement from converging. Using a combination of the idealized atomic positions and those

Table 1. Positional and isotropic thermal parameters (Å²)

Cluster A					Cluster B					
	x	y	z	B_{eq}/B_{iso}		x	y	z	B_{eq}/B_{iso}	Ave. B
Ru(1)	0.8055 (1)	-0.0618 (1)	-0.01956 (7)	1.9895*	Ru(7)	0.5745 (1)	0.9404 (1)	0.39433 (7)	2.2482*	2.119
Ru(2)†	0.9434	0.1387	0.01560	2.03176*	Ru(8)	0.2959 (1)	0.9593 (1)	0.48581 (7)	2.46907*	2.250
Ru(3)	1.0784 (1)	-0.0149 (1)	-0.10484 (7)	2.26999*	Ru(9)	0.4848 (2)	1.1438 (1)	0.49355 (8)	2.91337*	2.592
Ru(4)	1.0328 (1)	-0.2523 (1)	-0.00964 (7)	2.17976*	Ru(10)	0.6788 (1)	0.9303 (1)	0.52011 (7)	2.50781*	2.344
Ru(5)	0.8969 (1)	-0.0932 (1)	0.11030 (7)	1.82075*	Ru(11)	0.4927 (1)	0.7457 (1)	0.51218 (7)	2.13909*	1.980
Ru(6)	1.1719 (1)	-0.0472 (1)	0.02550 (7)	2.00302*	Ru(12)	0.4024 (1)	0.9506 (1)	0.61073 (7)	2.57039*	2.287
Cu(1)	0.8591 (2)	0.1524 (2)	-0.1057 (1)	2.74553*	Cu(3)	0.3824 (3)	1.1350 (2)	0.3785 (1)	3.25560*	3.000
Cu(2)	1.1105 (2)	-0.2669 (2)	0.1124 (1)	3.18608*	Cu(4)	0.5861 (2)	0.7538 (2)	0.6270 (1)	3.17094*	3.179
O(1A)	0.713 (2)	-0.130 (2)	-0.1449 (8)	4.8 (3)	O(7A)	0.471 (1)	0.874 (1)	0.2711 (6)	2.7 (2)	3.8
O(1B)	0.658 (1)	-0.282 (1)	0.0663 (7)	3.3 (2)	O(7B)	0.719 (1)	1.146 (1)	0.2819 (7)	3.3 (2)	3.3
O(1C)	0.539 (1)	0.112 (1)	0.0101 (7)	3.6 (3)	O(7C)	0.827 (1)	0.755 (1)	0.3540 (8)	4.2 (3)	3.9
O(2A)	0.682 (1)	0.320 (1)	0.0391 (8)	4.8 (3)	O(8A)	0.051 (2)	1.156 (2)	0.5163 (9)	5.7 (4)	5.3
O(2B)	0.989 (1)	0.219 (1)	0.1535 (7)	3.8 (3)	O(8B)	0.101 (1)	0.809 (1)	0.5960 (7)	3.9 (3)	3.9
O(2C)	1.054 (1)	0.380 (1)	-0.0754 (7)	3.1 (2)	O(8C)	0.174 (2)	0.869 (2)	0.3733 (9)	5.1 (3)	4.1
O(3A)	1.001 (1)	-0.047 (1)	-0.2404 (7)	3.6 (3)	O(9A)	0.621 (2)	1.348 (1)	0.3866 (8)	4.9 (3)	4.3
O(3B)	1.344 (1)	-0.187 (1)	-0.1543 (6)	2.1 (2)	O(9B)	0.608 (1)	1.243 (1)	0.5995 (8)	4.8 (3)	3.5
O(3C)	1.188 (1)	0.224 (1)	-0.1966 (7)	3.2 (2)	O(9C)	0.260 (1)	1.364 (1)	0.5102 (8)	4.6 (3)	3.9
O(4A)	0.907 (1)	-0.489 (1)	0.0651 (8)	4.1 (3)	O(10A)	0.795 (1)	0.993 (1)	0.6380 (7)	4.2 (3)	4.2
O(4B)	1.299 (1)	-0.419 (1)	-0.0567 (7)	3.7 (3)	O(10B)	0.908 (1)	0.709 (1)	0.5067 (7)	3.6 (3)	3.7
O(4C)	0.959 (1)	-0.333 (1)	-0.1367 (7)	3.7 (3)	O(10C)	0.857 (2)	1.126 (2)	0.4187 (8)	5.1 (3)	4.4
O(5A)	0.619 (2)	0.042 (2)	0.1596 (8)	4.9 (3)	O(11A)	0.715 (2)	0.526 (2)	0.4849 (10)	6.7 (4)	5.8
O(5B)	0.989 (1)	-0.037 (1)	0.2402 (7)	4.0 (3)	O(11B)	0.324 (2)	0.553 (2)	0.6141 (9)	6.1 (4)	5.1
O(5C)	0.755 (1)	-0.318 (1)	0.1981 (8)	4.7 (3)	O(11C)	0.417 (1)	0.640 (1)	0.3918 (7)	4.1 (3)	4.4
O(6A)	1.269 (1)	-0.007 (1)	0.1570 (7)	3.7 (3)	O(12A)	0.243 (2)	0.763 (2)	0.7144 (8)	5.2 (3)	4.5
O(6B)	1.300 (1)	0.199 (1)	-0.0466 (7)	3.5 (3)	O(12B)	0.176 (1)	1.163 (1)	0.6511 (7)	3.8 (3)	3.7
O(6C)	1.451 (1)	-0.193 (1)	-0.0201 (7)	4.0 (3)	O(12C)	0.507 (2)	1.044 (2)	0.7266 (11)	7.1 (5)	5.6
C(1A)	0.750 (2)	-0.092 (2)	-0.1022 (9)	2.3 (3)	C(7A)	0.513 (2)	0.911 (2)	0.3171 (12)	4.6 (5)	3.5
C(1B)	0.722 (2)	-0.194 (2)	0.0317 (13)	5.0 (5)	C(7B)	0.650 (3)	1.055 (3)	0.3334 (16)	6.9 (7)	6.0
C(1C)	0.633 (2)	0.030 (2)	0.0155 (11)	3.7 (4)	C(7C)	0.732 (2)	0.820 (2)	0.3751 (9)	2.7 (3)	3.2
C(2A)	0.775 (1)	0.244 (1)	0.0284 (8)	1.8 (3)	C(8A)	0.158 (2)	1.101 (2)	0.4982 (10)	3.1 (3)	2.5
C(2B)	0.975 (2)	0.175 (2)	0.1054 (12)	4.2 (4)	C(8B)	0.179 (1)	0.857 (1)	0.5573 (8)	1.9 (3)	3.1
C(2C)	1.022 (2)	0.290 (1)	-0.0392 (8)	2.0 (3)	C(8C)	0.231 (3)	0.898 (3)	0.4185 (15)	6.0 (6)	4.0
C(3A)	1.029 (2)	-0.059 (2)	-0.1877 (9)	2.9 (3)	C(9A)	0.591 (2)	1.261 (2)	0.4251 (9)	2.2 (3)	2.6
C(3B)	1.248 (2)	-0.126 (2)	-0.126 (10)	3.3 (4)	C(9B)	0.563 (2)	1.196 (2)	0.5612 (9)	2.6 (3)	3.0
C(3C)	1.147 (1)	-0.142 (1)	-0.1584 (7)	1.4 (2)	C(9C)	0.345 (2)	1.282 (2)	0.5006 (10)	3.3 (4)	2.4
C(4A)	0.961 (2)	-0.394 (2)	0.0493 (11)	4.0 (4)	C(10A)	0.747 (1)	0.967 (1)	0.5994 (8)	1.7 (3)	2.9
C(4B)	1.199 (2)	-0.350 (2)	-0.0439 (11)	3.8 (4)	C(10B)	0.824 (2)	0.797 (2)	0.5105 (12)	4.8 (5)	4.3
C(4C)	0.986 (2)	-0.296 (2)	-0.0899 (8)	2.1 (3)	C(10C)	0.774 (3)	1.052 (3)	0.4580 (14)	5.8 (6)	4.0
C(5A)	0.735 (2)	-0.002 (2)	0.1408 (9)	2.8 (3)	C(11A)	0.644 (2)	0.621 (2)	0.4954 (12)	4.0 (4)	3.4
C(5B)	0.964 (1)	-0.078 (1)	0.1910 (8)	1.9 (3)	C(11B)	0.420 (2)	0.618 (2)	0.5834 (10)	3.5 (4)	2.7
C(5C)	0.820 (2)	-0.229 (2)	0.1616 (13)	4.6 (5)	C(11C)	0.450 (2)	0.693 (2)	0.4326 (11)	3.8 (4)	4.2
C(6A)	1.215 (2)	-0.019 (2)	0.1114 (12)	4.2 (4)	C(12A)	0.309 (1)	0.819 (1)	0.6785 (6)	0.7 (2)	2.5
C(6B)	1.247 (1)	0.106 (1)	-0.0273 (7)	1.3 (2)	C(12B)	0.264 (2)	1.088 (2)	0.6293 (10)	3.0 (3)	2.2
C(6C)	1.334 (1)	-0.166 (1)	0.0081 (8)	1.6 (2)	C(12C)	0.477 (1)	1.000 (1)	0.6821 (8)	1.9 (3)	1.8
C(1)	0.826 (2)	0.253 (2)	-0.2091 (10)	3.1 (3)	C(15)	0.389	1.279	0.2832	4.5 (5)	3.8
C(2)	0.814 (2)	0.358 (2)	-0.1667 (9)	2.2 (3)	C(16)	0.335	1.365	0.3363	12.3	7.3
C(3)	0.684 (2)	0.405 (2)	-0.1301 (11)	3.8 (4)	C(17)	0.197	1.370	0.3718	12.0	7.9
C(4)	0.578 (3)	0.349 (3)	-0.1306 (14)	5.3 (5)	C(18)	0.113	1.290	0.3542	12.0	8.7
C(5)	0.593 (2)	0.241 (2)	-0.1690 (10)	3.4 (4)	C(19)	0.167	1.204	0.3011	3.5 (4)	3.5
C(6)	0.714 (2)	0.194 (2)	-0.2106 (11)	3.8 (4)	C(20)	0.305	1.199	0.2656	2.5 (3)	3.2
C(7)	0.672 (2)	0.528 (2)	-0.0862 (12)	4.1 (4)	C(21)	0.141	1.459	0.4268	12.0	8.1
C(8)	1.126 (2)	-0.391 (2)	0.2094 (11)	3.6 (4)	C(22)	0.582	0.600	0.7148	5.0 (5)	4.3
C(9)	1.149 (2)	-0.505 (2)	0.1719 (13)	4.8 (5)	C(23)	0.668	0.498	0.6809	3.6 (4)	4.2
C(10)	1.292 (2)	-0.528 (2)	0.1305 (9)	3.0 (3)	C(24)	0.805	0.513	0.6500	3.3 (4)	3.2
C(11)	1.396 (2)	-0.454 (2)	0.1358 (9)	2.8 (3)	C(25)	0.856	0.630	0.6531	12.0	7.4
C(12)	1.359 (3)	-0.346 (3)	0.1814 (16)	6.9 (7)	C(26)	0.771	0.732	0.6870	12.0	9.5
C(13)	1.226 (2)	-0.316 (2)	0.2152 (11)	4.0 (4)	C(27)	0.633	0.717	0.7179	8.1 (8)	6.1
C(14)	1.343 (3)	-0.652 (3)	0.0922 (15)	6.1 (6)	C(28)	0.894	0.408	0.6148	7.3 (7)	6.7

$$* B_{eq} = \frac{1}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

† Atomic parameters fixed during refinement.

obtained from the refinement as a criterion, positional coordinates from these 14 atoms were then computed and fixed. During last cycles of refinement 495 parameters were varied, which included positional and anisotropic thermal parameters and anomalous-dispersion corrections for four Cu and 12 Ru atoms, positional and isotropic thermal parameters for the 36 CO and two ordered $C_6H_5CH_3$ ligands, and some isotropic thermal parameters for the remaining two disordered $C_6H_5CH_3$ ligands. Refinement converged to $R = 0.0377$, $R_w = 0.0531$, $S = 1.617$. The small peaks in final difference Fourier map ($<0.9 \text{ e } \text{Å}^3$) appeared to be associated either with the H atoms in the toluene ligands or with the heavy metals in the cluster. It was not considered worthwhile to continue refinement including the H atoms. Quantity minimized $w(|F_o| - |F_c|)^2$ where $w = 1/\sigma(F_o)^2$ with $\sigma(F_o)$ defined above. $(\Delta/\sigma)_{\text{max}} = <0.01$. Largest peaks in final difference Fourier map $\leq 0.9 \text{ e } \text{Å}^{-3}$. The form of the extinction correction applied was $F_c(I + gI_c)^{-1}$ where the parameter g was refined in the least squares and converged to a value of 0.12073×10^{-6} . Atomic scattering factors from Cromer & Waber (1974); anomalous-scattering-factor corrections (Cromer, 1974) included in least-squares calculations. *SDP* (Enraf-Nonius, 1980) used for all calculations.

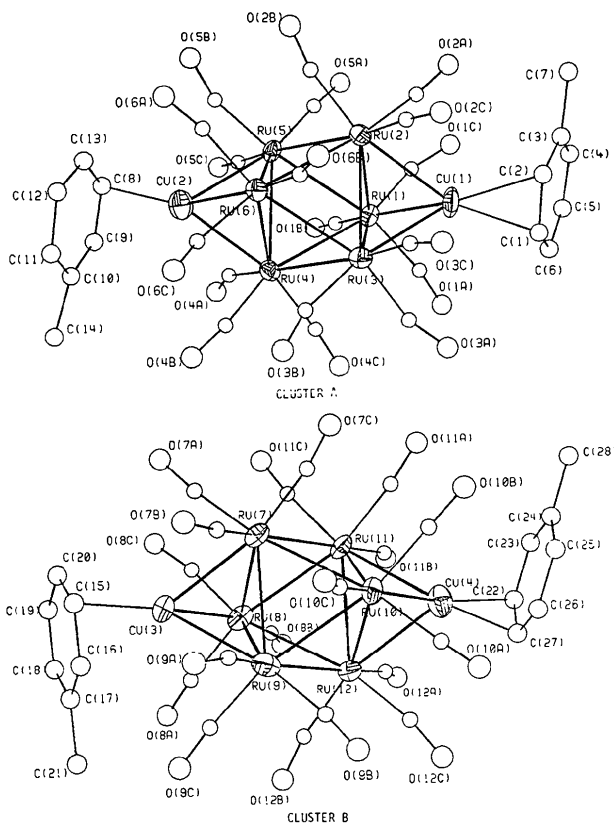


Fig. 1. The atomic configuration of the two $(C_6H_5CH_3)_2Cu_2Ru_6(CO)_{18}$ clusters.

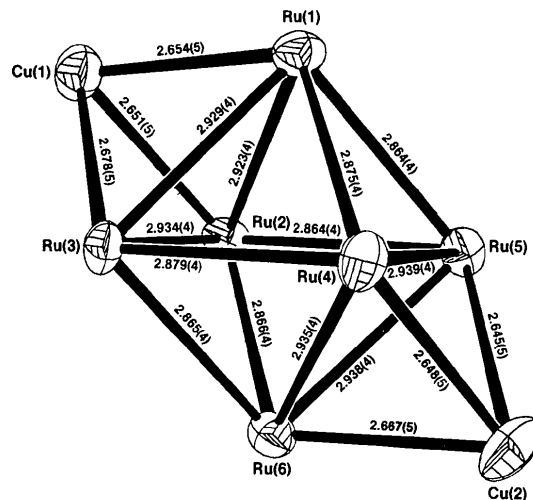


Fig. 2. Average metal-metal bonds (Å) within the Ru_6Cu_2 framework in $(C_6H_5CH_3)_2Cu_2Ru_6(CO)_{18}$.

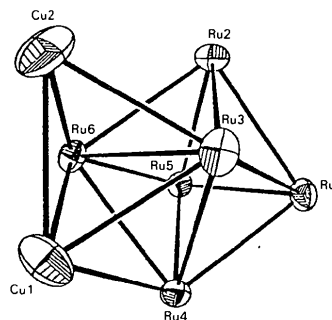


Fig. 3. The Cu_2Ru_6 core in $(CH_3CN)_2Cu_2Ru_6(CO)_{16}$.

Discussion. The final positional and isotropic thermal parameters are listed in Table 1.*

$(C_6H_5CH_3)_2Cu_2Ru_6(CO)_{18}$ possesses two crystallographically independent but essentially similar molecules (Fig. 1) which consists of a bimetallic Cu_2Ru_6 core (Fig. 2) which consists of a distorted octahedron of six atoms, each bearing three terminal carbonyls. Two opposite (trigonal) faces of the Ru_6 octahedra are capped by Cu atoms, each bonded to a toluene molecule. The distal arrangement of the Cu atoms is in contrast to their adjacent positions in $(CH_3CN)_2Cu_2Ru_6(CO)_{16}$ (Bradley *et al.*, 1982), shown in Fig. 3, but analogous to their location on opposite trigonal faces of the trigonal prismatic Rh_6 core in the $Rh-Cu$ cluster $(CH_3CN)_2Cu_2Rh_6(CO)_{15}$

* Lists of major bond lengths and angles, structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38988 (36 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(Albano, Braga, Martinengo, Chini, Sansoni & Strumolo, 1980). The mean Cu—Ru distance for the four Cu atoms in the two independent clusters is 2.66 (6) Å compared with 2.65 (9) Å in $(CH_3CN)_2Cu_2Ru_6C(CO)_{16}$. The Ru—Ru bonds in the octahedral core fall into two groups. Those on the opposite copper-capped trigonal faces [Ru(1)—Ru(2), Ru(1)—Ru(3), Ru(2)—Ru(3) and Ru(4)—Ru(5), Ru(4)—Ru(6), Ru(5)—Ru(6)] have significantly longer bond lengths than the Ru—Ru distances between these faces [2.93 (2) Å as compared to 2.87 (2) Å]. This effect was difficult to identify in $(CH_3CN)_2Cu_2Ru_6C(CO)_{16}$ because of the asymmetry of the cluster, where the range of Ru—Ru distances was wide (2.798–3.072 Å), with the long Ru—Ru bond (3.072 Å) associated with both Cu atoms. All 18 carbonyls on each cluster are essentially terminal with Ru—C—O angles ranging from 150–170°. This contrasts with the geometry of the starting material $[Ru_6(CO)_{18}]^{2-}$ (Jackson, Johnson, Lewis, McPartlin & Nelson, 1979) which contains two μ_2 -bridging carbonyls and two μ_3 -bridging carbonyls. The toluene molecules on each of the four Cu atoms are disordered to some degree. This results in only Cu(1) and Cu(4) with the expected μ_2 -bonding mode usable in the refinement.

The difference between the structures of the two bimetallic clusters $(CH_3CN)_2Cu_2Ru_6C(CO)_{16}$ and $(C_6H_5CH_3)_2Cu_2Ru_6(CO)_{18}$ is striking. In both cases, the Ru atoms form an octahedral core. In the former

carbide-containing cluster the Cu atoms occupy adjacent positions on the Ru_6 core, and are directly bonded to one another (Bradley *et al.*, 1982), whereas in the non-carbide analog, the two Cu atoms are found on opposite faces of the Ru_6 octahedron. This may reflect the spatial constraints imposed by the presence of 18 carbonyls in the latter case, in comparison to the relatively flexible carbonyl configuration allowed in the former, where the local perturbation caused by the presence of two adjacent heterometal atoms is more easily accommodated.

References

- ALBANO, V. G., BRAGA, D., MARTINENGO, S., CHINI, P., SANSONI, M. & STRUMOLO, D. (1980). *J. Chem. Soc. Dalton Trans.* pp. 52–54.
- ANSELL, G. B., LEONOWICZ, M. E., MODRICK, M. A. & BRADLEY, J. S. (1981). *Acta Cryst.* A37, C151.
- BRADLEY, J. S., PRUETT, R. L., HILL, E., ANSELL, G. B., LEONOWICZ, M. E. & MODRICK, M. A. (1982). *Organometallics*, 1, 748–752.
- CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Tables 2.2B, 2.3.1. Birmingham: Kynoch Press.
- CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press.
- Enraf-Nonius (1980). *Structure Determination Package*. Enraf-Nonius, Delft.
- JACKSON, P. F., JOHNSON, B. F. G., LEWIS, J., MCPARTLIN, M. & NELSON, W. J. H. (1979). *J. Chem. Soc. Chem. Commun.* pp. 735–736.

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Bis(2,6-dimethyl-4H-pyran-4-one)dinitratozinc, $[Zn(NO_3)_2(C_7H_8O_2)_2]$

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Abstract. $M_r = 437.66$, monoclinic, $A2/a$, $a = 16.786$ (8), $b = 12.922$ (8), $c = 9.089$ (4) Å, $\beta = 106.80$ (4)°, $V = 1887.34$ Å³, $Z = 4$, $D_x = 1.540$, $D_m = 1.536$ Mg m⁻³, $\lambda(Cu K\alpha) = 1.5418$ Å, $\mu = 2.38$ mm⁻¹, $F(000) = 896$, $T = 293$ K. Final $R = 0.043$ for 1500 observed visually estimated reflexions. The Zn atom lies on a twofold axis and coordinates tetrahedrally to two O atoms of the dimethyl-4-pyrone ligands, and to two O atoms of nitrate groups.

Introduction. The synthesis of the title compound was described originally by Hill (1971). Its properties have been studied by spectroscopic and other physical

methods (Briggs & Hill, 1969, 1970) and this work was undertaken to provide conclusive structural information particularly concerning the Zn coordination. Similar determinations have been concluded for the corresponding Cu and Cd complexes.

Experimental. Sample prepared by Dr A. E. Hill of this Polytechnic; slightly unstable crystals $ca 0.5 \times 0.5 \times 0.1$ mm, m.p. 421 K, which decompose in air within a few weeks. D_m by flotation in organic liquids. Lattice parameters from high-angle reflexions on Weissenberg photographs; intensity data from $0kl$, $h0l \rightarrow h1l$, and $hk0$ levels on a Stoe integrating Weissenberg camera