Table 3. *Thermogravimetric and differential thermal analysis results*

Step	% weight loss Calc.	Meas.	Temp. (K) Sample	ΔH (kJ mol^{-1})
1	28·1	28	307	66
2	56·1	56	433	89
3	84·1	84	515	166
				ΔH_{tot} = 339 kJ mol^{-1}

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Structure of a Chiral Diphosphine Ligand in a Trinuclear Ruthenium Cluster: $Ru_3(CO)_{10}\{(Ph_2PCH_2)_2C_5H_8O_2\}.xCH_2Cl_2$*

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Abstract. Decacarbonyl-μ-[4,5-bis(diphenylphosphinomethyl)-2,2-dimethyl-1,3-dioxolane]-*triangulo*-triruthenium dichloromethane solvate, $[Ru_3(CO)_{10}(C_{31}H_{32}O_2P_2)].xCH_2Cl_2$ ($x = 0{\cdot}29$), $M_r = 1081{\cdot}95 + 84{\cdot}93x$, orthorhombic, $P2_12_12_1$, $a = 14{\cdot}344\,(5)$, $b = 14{\cdot}763\,(3)$, $c = 21{\cdot}830\,(4)$ Å, $V = 4622{\cdot}7$ Å^3, $Z = 4$, $D_m = 1{\cdot}62\,(2)$, $D_x = 1{\cdot}590$ ($x = 0{\cdot}29$) g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 10{\cdot}4 \rightarrow 11{\cdot}5$ ($x = 0 \rightarrow 1$) cm^{-1}, $F(000) = 2144 + 168x$, room temperature, final $R = 0{\cdot}060$ for 3533 reflections $[F > 3\sigma(F)]$. The structure consists of discrete molecules in which the chelating diphosphine ligand ($C_{31}H_{32}O_2P_2$ = diop) bridges two Ru atoms to form an eight-membered ring [Ru—P = 2·328 (3), 2·337 (4) Å]. The longest Ru—Ru distance [2·888 (1) Å] is associated with the diphosphine bridge.

Introduction. The use of chiral organometallic species as potential catalysts in stereoselective synthesis is now well established (Kagan, 1982). As part of our study of metal-cluster catalysts, we have examined the Ru_3—carbonyl cluster with the chiral diphosphine ligand 4,5-bis(diphenylphosphinomethyl)-2,2-dimethyl-1,3-dioxolane (diop) and we now report the structure of $Ru_3(CO)_{10}\{(Ph_2PCH_2)_2C_5H_8O_2\}.xCH_2Cl_2$.

Experimental. The compound was prepared from the reaction of $Ru_3(CO)_{12}$ with the phosphine ligand using the radical-ion catalysis method (Bruce, Kehoe, Matisons, Nicholson, Rieger & Williams, 1982) and purified by flash column chromatography. Dark-red crystals were obtained from methylene chloride/octane by cooling and the density was measured by flotation ($CCl_4/C_2H_4Br_2$). Preliminary data were from photographic examination and accurate cell dimensions were obtained from 25 accurately centred reflections ($9{\cdot}0 < \theta < 12{\cdot}8°$) using an Enraf–Nonius CAD-4 diffractometer fitted with a graphite monochromator and Mo radiation. The intensities of 4746 reflections were recorded (ω–2θ scan, $1{\cdot}5 < \theta < 25°$; h 0–17, k 0–17, l 0–26) using a crystal 0·3 × 0·2 × 0·15 mm positioned in a thin-walled glass capillary. The standard reflections (three) showed no change with time during

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* μ-[4,5-Bis(diphenylphosphinomethyl)-2,2-dimethyl-1,3-dioxolane]-decacarbonyl-*triangulo*-triruthenium methylene dichloride solvate.

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the experiment and after data reduction 4509 unique reflections remained ($R_{\rm int} = 0·012$) of which 3537 with $F > 3\sigma(F)$ were used in the analysis. No absorption correction was applied. Systematic absences indicated the space group $P2_12_12_1$. The Ru atoms were located from the Patterson function and subsequent structure-factor and electron-density syntheses located the remaining non-H atoms of the cluster. The later maps gave evidence for some of the H atoms bonded to carbon and these were all introduced in geometrically calculated positions [d(C—H) = 1·08 Å]; methyl H atoms were given a common refined temperature factor as were the remaining H atoms (CH, CH_2). Phenyl C atoms were treated as a rigid group [d(C—C) = 1·395 Å]. A difference electron-density map revealed three peaks, the two largest of which (*ca* 3·5 e Å^{-3}) were separated by 2·9 Å, and these were interpreted as a fractional-occupancy molecule of CH_2Cl_2, one of the solvents used in the crystal growth, and this is consistent with the measured density. Four reflections (200, 211, 002, 113) thought to be subject to extinction were removed and full-matrix least-squares refinement minimizing $\sum w(\Delta F)^2$ converged to $R = 0·060$ {228 parameters, 3533 reflections, anisotropic (Ru, P) and isotropic (O, C, H) atoms, $w = 1/[\sigma^2(F) + 0·0007F^2]$, max. $\Delta/\sigma = 0·4$, $wR = 0·073$, $S = 1·88$; with all unique data $R = 0·087$, $wR = 0·078$}. The choice of the absolute configuration as *R*,*R* was confirmed by a calculation on the enantiomorph, when R and wR increased (0·0611, 0·0744) and the R-factor ratio (1·0134) was statistically highly significant (Hamilton, 1965); this was in agreement with the (−)-diop used in the initial synthesis. The residual electron density was in the range 1·6 to −1·1 e Å^{-3} with the largest peaks close to the solvent molecule. Scattering factors for neutral atoms and anomalous-dispersion corrections were taken from *SHELX*76 (Sheldrick, 1976) and *International Tables for X-ray Crystallography* (1974) (Ru only). All calculations were carried out using *SHELX*76 (Sheldrick, 1976) and *PLUTO* (Motherwell & Clegg, 1978) on ICL2970 and CDC7600 computers. The final atomic coordinates are given in Table 1 and selected molecular geometry is presented in Table 2.*

Table 1. *Atomic coordinates and isotropic temperature factors* (Å^2 × 10^3)

	x	y	z	U
Ru(1)	0·79632 (7)	0·24286 (8)	0·18460 (5)	35·2 (5)*
Ru(2)	0·61299 (7)	0·26807 (7)	0·13258 (5)	32·4 (5)*
Ru(3)	0·76604 (8)	0·21606 (8)	0·05752 (5)	42·1 (6)*
P(1)	0·8082 (2)	0·2441 (3)	0·2910 (2)	38·5 (18)*
P(2)	0·5053 (3)	0·3486 (2)	0·1917 (2)	36·8 (19)*
C(1)	0·8041 (11)	0·1117 (11)	0·1855 (7)	50 (4)
O(1)	0·8146 (9)	0·0367 (9)	0·1879 (6)	74 (4)
C(2)	0·9237 (11)	0·2556 (11)	0·1737 (6)	50 (4)
O(2)	1·0032 (11)	0·2651 (11)	0·1673 (6)	91 (4)
C(3)	0·7583 (11)	0·3675 (10)	0·1806 (7)	46 (4)
O(3)	0·7502 (8)	0·4460 (8)	0·1856 (5)	61 (3)
C(4)	0·6286 (10)	0·3733 (10)	0·0821 (7)	43 (3)
O(4)	0·6328 (8)	0·4351 (8)	0·0493 (5)	59 (3)
C(5)	0·5242 (10)	0·2199 (11)	0·0803 (7)	48 (4)
O(5)	0·4727 (9)	0·1869 (9)	0·0466 (6)	76 (4)
C(6)	0·6175 (11)	0·1635 (9)	0·1857 (6)	41 (3)
O(6)	0·6022 (8)	0·1011 (8)	0·2157 (5)	60 (3)
C(7)	0·7150 (12)	0·2268 (13)	−0·0225 (8)	66 (4)
O(7)	0·6844 (11)	0·2348 (11)	−0·0718 (7)	106 (5)
C(8)	0·8157 (11)	0·3348 (10)	0·0590 (7)	48 (4)
O(8)	0·8490 (9)	0·4066 (9)	0·0535 (6)	77 (4)
C(9)	0·8845 (13)	0·1689 (12)	0·0414 (8)	64 (5)
O(9)	0·9592 (9)	0·1412 (9)	0·0298 (6)	74 (4)
C(10)	0·7066 (12)	0·1018 (11)	0·0696 (8)	56 (4)
O(10)	0·6777 (10)	0·0311 (10)	0·0749 (7)	84 (4)
C(11)	0·7008 (10)	0·2333 (10)	0·3358 (6)	47 (3)
O(12)	0·6277 (8)	0·3269 (8)	0·4107 (5)	59 (3)
C(12)	0·6472 (10)	0·3228 (10)	0·3478 (7)	44 (4)
O(13)	0·4895 (8)	0·3497 (8)	0·3677 (5)	62 (3)
C(13)	0·5492 (11)	0·3273 (10)	0·3162 (7)	47 (4)
C(14)	0·5402 (11)	0·3987 (10)	0·2650 (6)	44 (4)
C(15)	0·5449 (12)	0·3762 (11)	0·4178 (8)	55 (4)
C(16)	0·4959 (16)	0·3483 (16)	0·4741 (10)	84 (6)
C(17)	0·5628 (17)	0·4756 (14)	0·4117 (11)	91 (7)
C(21)	0·4683 (7)	0·4490 (6)	0·1490 (4)	41 (3)
C(22)	0·5172 (7)	0·5304 (6)	0·1535 (4)	52 (4)
C(23)	0·4945 (7)	0·6027 (6)	0·1152 (4)	66 (5)
C(24)	0·4230 (7)	0·5936 (6)	0·0723 (4)	69 (5)
C(25)	0·3741 (7)	0·5122 (6)	0·0678 (4)	73 (5)
C(26)	0·3968 (7)	0·4399 (6)	0·1061 (4)	61 (4)
C(31)	0·3942 (6)	0·2928 (7)	0·2126 (5)	52 (4)
C(32)	0·3809 (6)	0·2004 (7)	0·2026 (5)	59 (4)
C(33)	0·2946 (6)	0·1608 (7)	0·2150 (5)	78 (6)
C(34)	0·2216 (6)	0·2137 (7)	0·2374 (5)	85 (6)
C(35)	0·2348 (6)	0·3061 (7)	0·2474 (5)	103 (8)
C(36)	0·3211 (6)	0·3457 (7)	0·2350 (5)	64 (5)
C(41)	0·8662 (9)	0·3401 (8)	0·3268 (6)	58 (4)
C(42)	0·8669 (9)	0·3490 (8)	0·3904 (6)	78 (6)
C(43)	0·9071 (9)	0·4252 (8)	0·4175 (6)	122 (9)
C(44)	0·9466 (9)	0·4925 (8)	0·3809 (6)	140 (11)
C(45)	0·9458 (9)	0·4836 (8)	0·3173 (6)	129 (10)
C(46)	0·9057 (9)	0·4074 (8)	0·2902 (6)	86 (6)
C(51)	0·8744 (8)	0·1470 (7)	0·3196 (5)	49 (4)
C(52)	0·9707 (8)	0·1553 (7)	0·3268 (5)	77 (6)
C(53)	1·0229 (8)	0·0816 (7)	0·3474 (5)	95 (7)
C(54)	0·9789 (8)	−0·0003 (7)	0·3606 (5)	104 (8)
C(55)	0·8827 (8)	−0·0086 (7)	0·3534 (5)	97 (7)
C(56)	0·8304 (8)	0·0650 (7)	0·3328 (5)	77 (6)
Cl(1)	0·6465 (8)	0·1720 (8)	0·5916 (5)	40†
Cl(2)	0·7624 (8)	0·3321 (8)	0·5738 (5)	40†
C	0·7508 (40)	0·2105 (38)	0·5908 (25)	60†

* Equivalent isotropic temperature factor from anisotropic atom: $U_{\rm eq} = \frac{1}{3}$ trace **U**.

† The temperature factor was fixed and these atoms were given a common refined population [0·287 (6)].

Discussion. The structure of the discrete cluster is shown in Fig. 1 and shows the *triangulo*-Ru_3 unit found in many species with this diphosphine bridging two Ru atoms. The bridged Ru—Ru distance is lengthened relative to that in $Ru_3(CO)_{12}$ by *ca* 0·03 Å (Churchill, Hollander & Hutchinson, 1977) although with other diphosphine ligands the corresponding Ru—Ru distance is shortened or not much changed, *e.g.* $Ru_3(CO)_{10}$(dppm) [dppm = bis(diphenylphosphino)methane] (Coleman, Jones, Dixneuf, Brisson, Bonnet & Lavigne, 1984). Reports of longer Ru—Ru distances in $Ru_3(CO)_{10}(L_2)$ structures are few, seemingly confined to one of the molecules in the asymmetric unit in $Ru_3(CO)_{10}(PPh_3)_2$ [2·893 (3) Å] (Bruce, Liddell, Hughes, Patrick, Skelton & White, 1988; Chin-Choy, Keder, Stucky & Ford, 1988). However, in this

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* Lists of structure factors, anisotropic thermal parameters, calculated H-atom positions and complete geometric details have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51570 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Selected distances* (Å) *and angles* (°)

Ru(1)–Ru(2)	2·888 (1)	P(1)–Ru(1)	2·328 (3)
Ru(2)–Ru(3)	2·845 (2)	P(2)–Ru(2)	2·337 (4)
Ru(3)–Ru(1)	2·836 (2)	P(1)···P(2)	5·094 (6)
Ru–C	min. 1·85 (2)	max. 1·94 (2)	mean 1·90 (3)
P–C	min. 1·82 (1)	max. 1·85 (1)	mean 1·83 (1)
C–O	min. 1·12 (2)	max. 1·17 (2)	mean 1·15 (2)
C(11)–C(12)	1·55 (2)	O(12)–C(12)	1·40 (2)
C(12)–C(13)	1·57 (2)	O(12)–C(15)	1·40 (2)
C(13)–C(14)	1·54 (2)	O(13)–C(13)	1·45 (2)
C(15)–C(16)	1·47 (2)	O(13)–C(15)	1·41 (2)
C(15)–C(17)	1·50 (3)		
Ru(1)···C(6)	2·82 (2)	Ru(2)···C(3)	2·76 (2)
Ru(1)–Ru(2)–Ru(3)	59·3 (1)	P(1)–Ru(1)–Ru(2)	117·2 (1)
Ru(2)–Ru(3)–Ru(1)	61·1 (1)	P(1)–Ru(1)–Ru(3)	171·1 (1)
Ru(3)–Ru(1)–Ru(2)	59·6 (1)	P(2)–Ru(2)–Ru(1)	116·7 (1)
		P(2)–Ru(2)–Ru(3)	164·8 (1)
Ru–P–C	min. 108·8 (4)	max. 120·5 (5)	
C–P–C	min. 100·7 (6)	max. 103·6 (5)	
Ru(1)–C(3)–O(3)	167 (1)	C(12)–C(13)–O(13)	101 (1)
Ru(2)–C(6)–O(6)	167 (1)	C(13)–O(13)–C(15)	109 (1)
		O(13)–C(15)–O(12)	104 (1)
P(1)–C(11)–C(12)	116 (1)	C(15)–O(12)–C(12)	108 (1)
P(2)–C(14)–C(13)	112 (1)	O(12)–C(12)–C(13)	104 (1)

structure the two phosphine groups are *trans* rather than *cis* to the lengthened bond, and a larger effect may be anticipated. For the ligand-bridged bonds, the Ru–Ru separation tends to increase with the ligand 'bite' distance, from 2·834(1)Å in $Ru_3(CO)_{10}$(dppm) (P···P = 3·12 Å) (Coleman *et al.*, 1984) through 2·856(1)Å in $Ru_3(CO)_{10}$(dppe) (P···P = 3·89 Å) (Bruce, Hambley, Nicolson & Snow, 1982) to 2·888(1)Å in the present diop cluster (P···P = 5·09 Å). The electronic properties of the Group V donor ligands will be very similar and any structural changes may be attributed to steric factors in the ligand bridge. Clearly, the metal–metal bond length parallels the bite variations, but to only a small degree. The ligand, diop, has been characterized by X-ray diffraction in several complexes and can coordinate to one metal centre, *e.g.* as in $RuHCl(diop)_2$ (Ball & Trotter, 1981) or bridge two metal atoms as in the present example and in $[Ir_4(CO)_{10}(diop)]$ (Tranqui, Durif, Nasr Eddine, Lieto, Rafalko & Gates, 1982). In the latter complex the bite distance (4·75 Å) is 0·34 Å less than in the present example, showing that the ligand is flexible when in a bridging mode. Again the direction of the metal–metal bond-length change parallels the bite variation; the diop-bridged Ir–Ir bond in the tetranuclear cluster has a length of 2·772 Å, 0·12 Å shorter than the pertinent Ru–Ru vector in this report. The rather strong Ir–Ir bonds and the different angular relationship to the substituent sites may well impose the reduced bite on the diop. In the ruthenium series it is unclear whether the small variations in the Ru–Ru bond length are a consequence merely of the bite change or due to a modification of the metal–metal bonding resulting from the twisting of the RuL_4 units caused by the substituent. One of the P atoms [P(1)] lies above (0·60 Å) and one [P(2)] below (0·34 Å) the Ru_3 plane and this twist is reflected in the CO groups bonded to each of the Ru atoms as is clearly shown in Fig. 2. Indeed, it seems that the approximate twofold symmetry of the phosphine is imposed on the metal residue. The P–Ru–Ru–P torsion angle is 26·3 (2)°, similar to the value of 25·1° in $Ru_3(CO)_{10}$(dppe) (Bruce, Hambley *et al.*, 1982) and rather larger than that in the dppm derivative [19·1 (1)°] (Coleman *et al.*, 1984). One consequence of this twist is that two of the carbonyl groups, C(3)O(3) and C(6)O(6), become semi-bridging and have Ru···C distances *ca* 2·8 Å whereas all other Ru···C's are > 3·0 Å. These two carbonyl groups are also the most non-linear [Ru–C–O = 167 (1)°]. These values may be compared with those reported for $Ru_3(CO)_{10}(PPh_3)_2$, one of the structures in which the distortion from an $Ru_3(CO)_{12}$ towards an $Fe_3(CO)_{12}$ structure is most pronounced (Ru···C = 2·74 Å, Ru–C–O = 158°) (Chin-Choy *et al.*, 1988). The five-membered heterocyclic ring has an envelope conformation with C(15) lying out of the plane formed by the other four atoms by 0·47 (2) Å.

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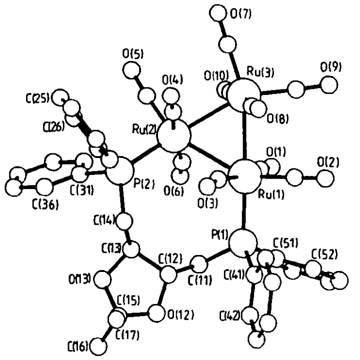

Fig. 1. Perspective view of the discrete molecule showing the atom-numbering scheme. Atoms are drawn with arbitrary size and H atoms omitted for clarity.

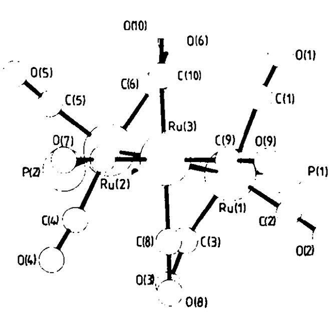

Fig. 2. View of the molecule showing the semi-bridging carbonyl groups. Only Ru and P atoms, and CO groups are drawn.

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Structure of Chloro{*N*-[2-(4-imidazolyl)ethyl]salicylideneaminato}copper(II) Monohydrate

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Abstract. $[CuCl(C_{12}H_{11}N_3O)].H_2O$, $M_r = 330{\cdot}25$, orthorhombic, $Pccn$, $a = 18{\cdot}952\,(9)$, $b = 19{\cdot}680\,(9)$, $c = 15{\cdot}028\,(8)$ Å, $V = 5605{\cdot}1\,(9)$ Å^3, $Z = 16$, $D_x = 1{\cdot}584$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 17{\cdot}5$ cm^{-1}, $F(000) = 2688$, $T = 293$ K, $R = 0{\cdot}059$ for 2569 observed data with $I \geq 2{\cdot}58\sigma(I)$. There are two independent molecules in the asymmetric unit. In each molecule the Cu atom is four-coordinate in an essentially square-planar environment with three of the basal coordination positions occupied by the donor atoms of the ligand whilst the fourth position is occupied by a chloride ion. The coordination is completed by a long, but significant (3·0 Å av.), bond between the Cu atom of one molecule and the chloride ion of a symmetry-related counterpart. This results in the existence of dimeric units in which the coordination of the metal atom is best described as belonging to the 4 + 1 type.

Introduction. Histidine and tyrosine residues are, generally, thought to be involved at many biological copper sites. In order to characterize the properties of a copper ion in such an environment, we started investigating systematically copper complexes of ligands involving a diamine in addition to a salicyl moiety. In a previous study, we reported on the behaviour of complexes of ligands involving a salicyl group together with an aminoalkylpyridine (Latour, Leonard, Limosin, Povey & Tandon, 1985; Latour, Tandon & Povey, 1989). In the present contribution, we describe the structural properties of the chlorocopper(II) complex of the ligand resulting from the condensation of histamine with salicylaldehyde.

Experimental. Histamine dihydrochloride (0·555 g; 3 mmol) was dissolved in 50 ml of aqueous 0·1 *M* sodium hydroxide solution. To this solution was added salicylaldehyde (0·366 g; 3 mmol) in 25 ml of absolute ethanol. The yellow reaction mixture was stirred for 30 min and, after addition of a solution of copper(II) nitrate trihydrate (0·793 g; 3 mmol) in 10 ml of water, it turned dark green. After stirring for 2 h, the resulting

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