

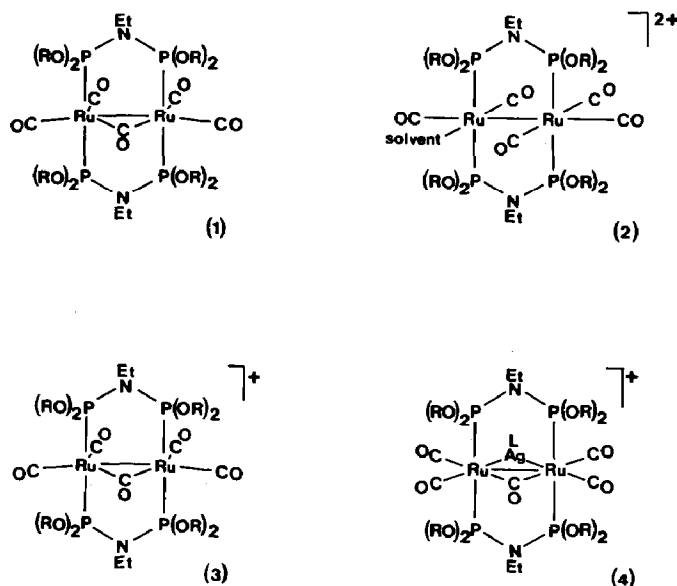
Preliminary communication**Synthesis of the solvento species $[\text{Ru}_2(\text{CO})_5(\text{solvent})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ and its potential as a source for a wide range of dinuclear derivatives of ruthenium****John S. Field, Raymond, J. Haines*, Ute Honrath, Jörg Sundermeyer and Stephen F. Woollam***Unit of Metal Cluster Chemistry, Department of Chemistry, University of Natal, P.O. Box 375, Pietermaritzburg 3200 (Republic of South Africa)*

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Abstract

Treatment of $[\text{Ru}_2(\mu\text{-CO})(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]$ ($\text{R} = \text{Me}$ or Pr^i), electron-rich derivatives of $[\text{Ru}_2(\text{CO})_9]$, with a twice molar amount of a silver(I) salt in aprotic, weakly co-ordinating solvents such as acetone, acetonitrile or benzonitrile leads to the formation of the solvento species $[\text{Ru}_2(\text{CO})_5(\text{solvent})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$. The structure of the benzonitrile derivative, $[\text{Ru}_2(\text{CO})_5(\text{PhCN})\{\mu\text{-(Pr}^i\text{O)}_2\text{PN(Et)P(OPr}^i)_2\}_2](\text{SbF}_6)_2$, has been established by X-ray crystallography. The acetone molecule in $[\text{Ru}_2(\text{CO})_5(\text{acetone})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ is readily replaced by various nucleophiles to afford products of the type $[\text{Ru}_2(\text{CO})_5\text{L}\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$, where L is a neutral ligand such as CO, $\text{Me}_2\text{C}_6\text{H}_3\text{NC}$, PhCN, $\text{C}_5\text{H}_5\text{N}$, H_2O , Me_2S or SC_4H_8 , $[\text{Ru}_2\text{Y}(\text{CO})_5\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^+$, where Y^- is an anionic ligand such as Cl^- , Br^- , I^- , CN^- , SCN^- , MeCO_2^- , CF_3CO_2^- or $[\text{Ru}_2(\mu\text{-Y})(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^+$ where Y^- is an anionic ligand such as Cl^- , Br^- , I^- , SPh^- , $\text{S}_2\text{CNEt}_2^-$, MeCO_2^- or CF_3CO_2^- .

The one-electron oxidation of $[\text{Ru}_2(\mu\text{-CO})(\text{CO})_4\{\mu\text{-(MeO)}_2\text{PN(Et)P(OMe)}_2\}_2]$ (**1**; $\text{R} = \text{Me}$) in acetone or benzonitrile has been established previously from cyclic voltammetry studies to be irreversible, with the one-electron oxidized product participating in a chemical process involving solvent attack and the product of this attack being immediately oxidized to afford $[\text{Ru}_2(\text{CO})_5(\text{solvent})\{\mu\text{-(MeO)}_2\text{PN(Et)P(OMe)}_2\}_2]^{2+}$ (solvent = acetone or benzonitrile) (**2**; $\text{R} = \text{Me}$) [1]. On the other hand, the corresponding oxidation of $[\text{Ru}_2(\mu\text{-CO})(\text{CO})_4\{\mu\text{-(Pr}^i\text{O)}_2\text{PN(Et)P(OPr}^i)_2\}_2]$ in these solvents was found to be reversible, although further oxidation of the oxidized product $[\text{Ru}_2(\mu\text{-CO})(\text{CO})_4\{\mu\text{-(Pr}^i\text{O)}_2\text{PN(Et)P(OPr}^i)_2\}_2]^+$ (**3**; $\text{R} =$



Pr^i) also proved to be irreversible, again leading to the formation of the solvento dication, viz. $[\text{Ru}_2(\text{CO})_5(\text{solvent})\{\mu\text{-(Pr}^i\text{O)}_2\text{PN(Et)P(OPr}^i)_2\}_2]^{2+}$ (**2**; $\text{R} = \text{Pr}^i$) [1]. It has also been established that $[\text{Ru}_2(\mu\text{-CO})(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]$ ($\text{R} = \text{Me}$ or Pr^i) is readily oxidized by chemical oxidants and, in particular, that oxidation by silver(I) salts occurs via the inner sphere intermediate $[\text{Ru}_2(\mu\text{-AgL})(\mu\text{-CO})(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^+$ ($\text{L} = \text{acetone, THF, etc.}$) (**4**). With loss of elemental silver this adduct decomposes to $[\text{Ru}_2(\mu\text{-CO})(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^+$ (**3**) which in turn spontaneously disproportionates in solution to $[\text{Ru}_2(\text{CO})_5(\text{solvent})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ and $[\text{Ru}_2(\mu\text{-CO})(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]$ [2]. The synthesis of the solvento species $[\text{Ru}_2(\text{CO})_5(\text{solvent})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2](\text{SbF}_6)_2$ and their reactivity towards a wide range of nucleophiles is now reported.

Reaction of $[\text{Ru}_2(\mu\text{-CO})(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]$ ($\text{R} = \text{Me}$ or Pr^i) (**1**) with a twice molar amount of AgSbF_6 in the aprotic weakly co-ordinating solvent benzonitrile, was found to lead to the quantitative formation of a product characterized as $[\text{Ru}_2(\text{CO})_5(\text{PhCN})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2](\text{SbF}_6)_2$. The structure of the tetraisopropoxydiphosphazane derivative has been established by X-ray crystallography and is illustrated in Fig. 1*. The two ruthenium atoms, each of which is approximately octahedral, are not only linked through two bridging tetraisopropoxydiphosphazane ligands but also by a formal ruthenium–ruthenium bond $\{\text{Ru}(1)\text{--Ru}(2) 2.890(3) \text{ \AA}\}$. The cation adopts an essentially staggered conforma-

* *Crystal data:* $\text{C}_{40}\text{H}_{71}\text{N}_5\text{F}_{12}\text{O}_{13}\text{P}_4\text{Ru}_2\text{Sb}_2$, $M = 1599.5$, orthorhombic, space group $P2_12_12$ (No. 18), a 22.710(5), b 23.695(5), c 12.256(3) Å, U 6594.9 Å³, D_c 1.61 g cm⁻³, $Z = 4$, Mo- K_α radiation, λ 0.71069 Å, μ 13.1 cm⁻¹. Nonius CAD4 diffractometer, 3877 unique reflections with $I > 3\sigma(I)$ in the range $3^\circ \leq \theta \leq 23^\circ$. Structure solved from a Patterson synthesis and successive Fourier maps and refined to $R = 0.101$ (319 parameters). Tables of atomic co-ordinates, interatomic distances and angles and thermal parameters have been deposited at the Cambridge Crystallographic Data Centre.

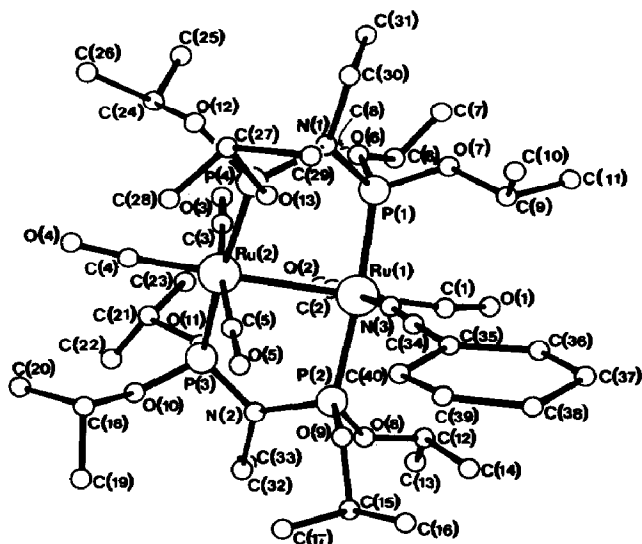
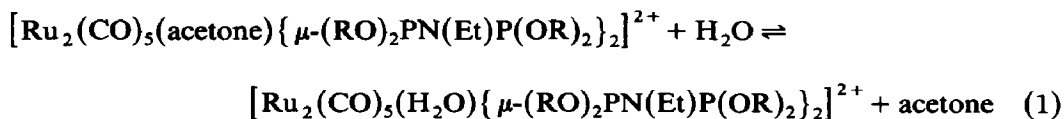


Fig. 1. The stereochemistry of $[\text{Ru}_2(\text{CO})_5(\text{PhCN})\{\mu\text{-(Pr}^i\text{O)}_2\text{PN(Et)P(OPr}^i)_2\}_2]^{2+}$ showing the atom labelling scheme. Relevant interatomic distances: Ru(1)–Ru(2), 2.889(3); Ru(1)–P(1), 2.341(8); Ru(1)–P(2), 2.353(8); Ru(2)–P(3), 2.339(8); Ru(2)–P(4), 2.337(8); Ru(1)–N(3), 2.079(24); N(3)–C(34), 1.125(35); C(34)–C(35), 1.433(41) Å.

tion, as reflected by P(1)–Ru(1)–Ru(2)–P(4) and P(2)–Ru(1)–Ru(2)–P(3) torsion angles of 28.4 and 31.0°, respectively, with the benzonitrile ligand co-ordinating equatorially. The $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of this compound measured in acetone- d^6 at -77°C exhibits an AA'BB' pattern of peaks centred at 133.8 ppm (separation of the outer peaks 20.4 Hz), consistent with the structure established, but the room temperature spectrum measured in both acetone- d^6 and benzonitrile exhibits a sharp singlet (at 128.5 and 130.3 ppm, respectively). This is interpreted in terms of this species participating in some fluxional process involving the migration of the co-ordinated benzonitrile ligand from the equatorial site of one ruthenium atom to the adjacent equatorial site of the other ruthenium atom.

The corresponding acetonitrile, acetone and tetrahydrofuran solvento species were synthesized similarly by treatment of $[\text{Ru}_2(\mu\text{-CO})(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]$ with AgSbF_6 in the appropriate solvent. Significantly, the room temperature $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of $[\text{Ru}_2(\text{CO})_5(\text{acetone})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ measured in acetone- d^6 , was found to exhibit both an AA'BB' pattern of peaks (centred at 127.5 ppm for $\text{R} = \text{Pr}^i$) as well as a sharp singlet (at 129.6 ppm for $\text{R} = \text{Pr}^i$), while the corresponding low temperature spectrum (-77°C) was shown to contain two sets of peaks with AA'BB' patterns. However, a more detailed spectroscopic study of this species revealed that the co-ordinated acetone is readily and reversibly displaced by water, that the equilibrium



is such that the aquo species is present in significant quantities, even in convention-

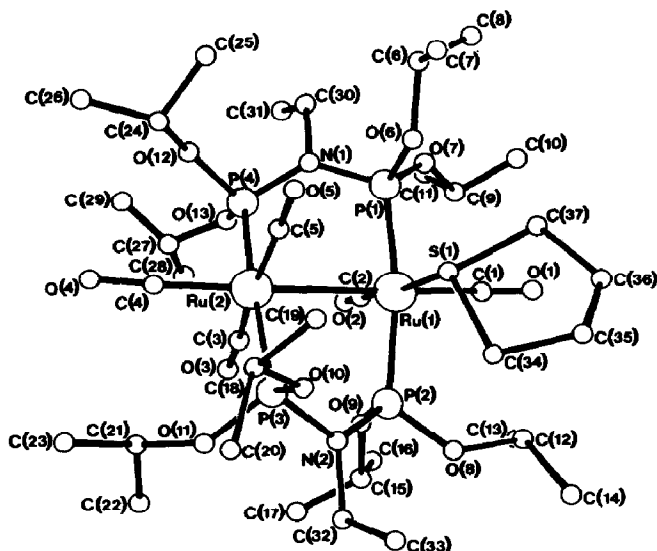
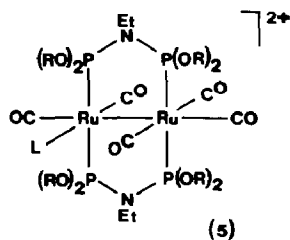


Fig. 2. The stereochemistry of $[\text{Ru}_2(\text{CO})_5(\text{SC}_4\text{H}_8)\{\mu\text{-(Pr}^i\text{O)}_2\text{PN(Et)P(OPr}^i)_2\}_2]^{2+}$ showing the atom labelling scheme. Relevant interatomic distances: Ru(1)–Ru(2), 2.926(2); Ru(1)–P(1), 2.346(7); Ru(1)–P(2), 2.358(8); Ru(2)–P(3), 2.366(7); Ru(2)–P(4), 2.334(7); Ru(1)–S(1), 2.487(6) Å.

ally dried acetone *, and that the singlet in the room temperature spectrum results from the presence of the aquo species.

The acetone molecule in $[\text{Ru}_2(\text{CO})_5(\text{acetone})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ and likewise the water molecule in $[\text{Ru}_2(\text{CO})_5(\text{H}_2\text{O})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ was established to be readily and reversibly displaced by a wide range of neutral nucleophiles leading to the formation of products of the type $[\text{Ru}_2(\text{CO})_5\text{L}\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ (5) where L is a neutral ligand such as CO, $\text{Me}_2\text{C}_6\text{H}_3\text{NC}$, PhCN, MeCN, $\text{C}_5\text{H}_5\text{N}$, Me_2S and tetrahydrothiophene. $[\text{Ru}_2(\text{CO})_5(\text{SC}_4\text{H}_8)\{\mu\text{-(Pr}^i\text{O)}_2\text{PN(Et)P(OPr}^i)_2\}_2](\text{SbF}_6)_2$ has been structurally characterised and the stereochemistry of the cation of this species is illustrated in Fig. 2 **. The structure is



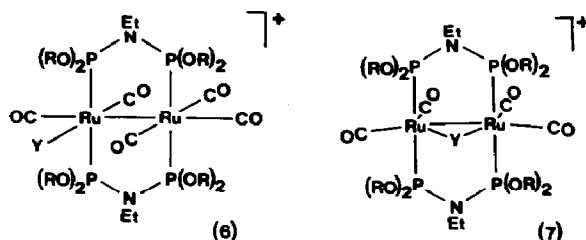
* The acetone used in this study was freshly distilled from anhydrous calcium sulphate (twice) and stored over molecular sieves (3 Å).

** *Crystal data:* $\text{C}_{37}\text{H}_{74}\text{N}_2\text{F}_{12}\text{O}_{13}\text{P}_4\text{SRu}_2\text{Sb}_2\cdot\text{CH}_2\text{Cl}_2$. $M = 1669.7$, orthorhombic, space group $Pna2_1$ (No. 33), a 27.167(2), b 14.629(2), c 16.704(2) Å, U 6638.9 Å³, D_c 1.67 g cm⁻³, $Z = 4$, Mo- K_α radiation, λ 0.71069 Å, μ 16.33 cm⁻¹. Nonius CAD4 diffractometer, 2788 unique reflections with $I > 3\sigma(I)$ in the range $3^\circ \leq \theta \leq 23^\circ$. Structure solved using direct methods and successive Fourier maps, and refined to $R = 0.058$, $R_w = 0.066$ (349 parameters). Anomalous scattering factors were used but changing the signs of the atom co-ordinates merely resulted in the same R values and, therefore, it was not possible to determine the chirality of the crystal. Tables of atomic co-ordinates, interatomic distances and angles and thermal parameters have been deposited at the Cambridge Crystallographic Data Centre.

very similar to that of $[\text{Ru}_2(\text{CO})_5(\text{PhCN})\{\mu\text{-(Pr}^i\text{O)}_2\text{PN(Et)P(OR}^i)_2\}_2]^{2+}$ with the tetrahydrothiofuran co-ordinating equatorially and with the cation adopting an essentially staggered conformation [P(1)–Ru(1)–Ru(2)–P(4) torsion angle 26.6° ; P(2)–Ru(1)–Ru(2)–P(3) torsion angle 30.5°].

The solvento species $[\text{Ru}_2(\text{CO})_5(\text{solvent})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ where the co-ordinated solvent is a protic solvent of the type $\text{R}'\text{OH}$ ($\text{R}' = \text{H, Me, Et, etc.}$) has been shown to be readily deprotonated to $[\text{Ru}_2\{\mu\text{-OC(OR}'')\}(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ and in the case of $[\text{Ru}_2(\text{CO})_5(\text{H}_2\text{O})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ to $[\text{Ru}_2\{\mu\text{-OC(O)}\}(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]$ [3], such that a competing reaction in the above nucleophilic substitution reactions would be the deprotonation of any $[\text{Ru}_2(\text{CO})_5(\text{H}_2\text{O})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ present in the acetone solution by the ligand L functioning as a base. The extent of this deprotonation was found to be dependent not only on the amount of water present but also on the relative nucleophilic and basic strengths of the ligand. Thus, while the reaction of $[\text{Ru}_2(\text{CO})_5(\text{acetone})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]\text{SbF}_6$ with tetrahydrothiofuran in acetone afforded solely $[\text{Ru}_2(\text{CO})_5(\text{SC}_4\text{H}_8)\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]\text{SbF}_6$, the corresponding reaction involving pyridine produced $[\text{Ru}_2(\text{CO})_5(\text{pyridine})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]\text{SbF}_6$ and $[\text{Ru}_2\{\mu\text{-OC(OH)}\}(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]\text{SbF}_6$ in approximately equal amounts and that involving NEt_3 gave solely $[\text{Ru}\{\mu\text{-OC(O)}\}(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]$.

Anionic ligands Y^- were also established to readily displace the acetone from $[\text{Ru}_2(\text{CO})_5(\text{acetone})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ affording either cationic products of the type $[\text{Ru}_2\text{Y}(\text{CO})_5\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ (6) or, for those ligands which are capable of effecting further substitution, of the type $[\text{Ru}_2(\mu\text{-Y})(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ (7). However, those anionic ligands with basic properties were again found to effect the deprotonation of the $[\text{Ru}_2(\text{CO})_5(\text{H}_2\text{O})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ present in the acetone solution. In particular while cyanide, halide, thiocyanate, mercaptide, trifluoroacetate and xanthate ions gave the pentacarbonyl $[\text{Ru}_2\text{Y}(\text{CO})_5\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ and/or tetracarbonyl $[\text{Ru}_2(\mu\text{-Y})(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ substituted species, acetate ions afforded $[\text{Ru}_2\{\mu\text{-OC(OH)}\}(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ as well as $[\text{Ru}_2\{\mu\text{-OC(Me)O}\}(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ and alkoxide and hydroxide ions produced solely $[\text{Ru}_2\{\mu\text{-OC(OH)}\}(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ or $[\text{Ru}_2\{\mu\text{-OC(O)}\}(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]$.



The nature of equilibrium (1) is such that while acetate ions deprotonate the aquo species present in acetone solution as described above, acetic acid was observed to effect only the displacement of the acetone from the acetone solvento species in this solution to afford $[\text{Ru}_2(\text{CO})_5(\text{MeCOOH})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$ which is readily deprotonated to $[\text{Ru}_2(\text{MeCO}_2)(\text{CO})_5\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]^{2+}$. Similarly, although $[\text{Ru}_2(\text{SPh})(\text{CO})_5\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]\text{SbF}_6$ can be synthesised

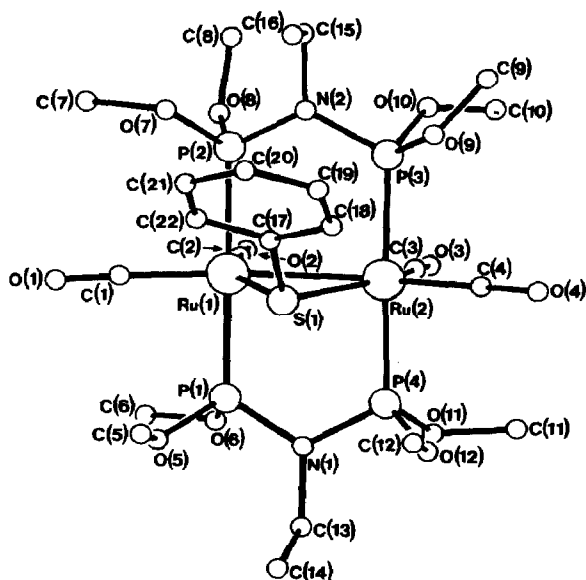
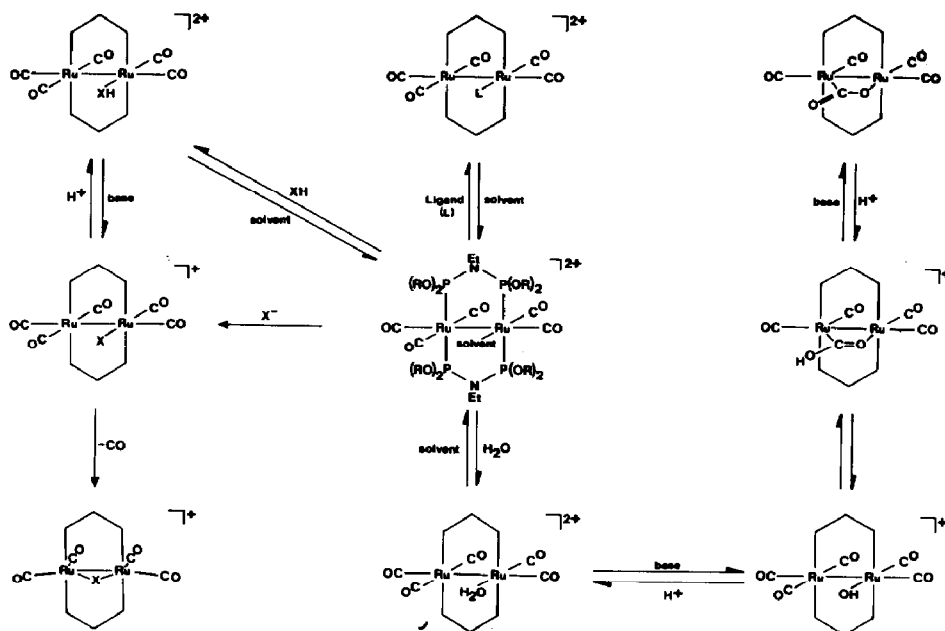


Fig. 3. The stereochemistry of $[\text{Ru}_2(\mu\text{-SPh})(\text{CO})_4\{\mu\text{-(MeO)}_2\text{PN(Et)P(OMe)}_2\}_2]^+$ showing the atom labelling scheme. Relevant interatomic distances and angles: Ru(1)–Ru(2), 2.796(3); Ru(1)–P(1), 2.316(2); Ru(1)–P(2), 2.335(2); Ru(2)–P(3), 2.326(2); Ru(2)–P(4), 2.323(2); Ru(1)–S(1), 2.380(2); Ru(2)–S(1), 2.387(1) Å; Ru(1)–S(1)–Ru(2), 71.8(0)°.

by treatment of $[\text{Ru}_2(\text{CO})_5(\text{acetone})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2](\text{SbF}_6)_2$ with phenyl mercaptide ions in acetone solution, it is obtained in best yield by reaction of the acetone solvento species with phenyl mercaptan. Indeed, the synthesis of derivatives of the type $[\text{Ru}_2\text{Y}(\text{CO})_5\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]\text{SbF}_6$ or of the type $[\text{Ru}_2\{\mu\text{-OC(Y)}\}(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]\text{SbF}_6$ where Y^- is the conjugate base of a weak acid is best achieved by addition of the acid rather than the conjugate base to $[\text{Ru}_2(\text{CO})_5(\text{acetone})\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2](\text{SbF}_6)_2$.

Interestingly, although $[\text{Ru}_2(\text{SPh})(\text{CO})_5\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]\text{SbF}_6$ has been identified spectroscopically, it readily decarbonylates to $[\text{Ru}_2(\mu\text{-SPh})(\text{CO})_4\{\mu\text{-(RO)}_2\text{PN(Et)P(OR)}_2\}_2]\text{SbF}_6$, which for the tetramethoxydiphosphazane hexafluorophosphate salt has been characterised crystallographically as well as by conventional methods. The stereochemistry of the cation is illustrated in Fig. 3*. In contrast to $[\text{Ru}_2(\text{CO})_5(\text{PhCN})\{\mu\text{-(Pr}^i\text{O)}_2\text{PN(Et)P(OPr}^i\text{)}_2\}_2]^{2+}$ and $[\text{Ru}_2(\text{CO})_5(\text{SC}_4\text{H}_8)\{\mu\text{-(Pr}^i\text{O)}_2\text{PN(Et)P(OPr}^i\text{)}_2\}_2]^{2+}$, this cation adopts an eclipsed conformation with the two ruthenium atoms bridged by the phenylmercapto as well as by the two diphosphazane ligands and separated by a distance of 2.796(1) Å.

* *Crystal data:* $\text{C}_{22}\text{H}_{39}\text{N}_2\text{F}_6\text{O}_{12}\text{P}_5\text{SRu}_2$, $M = 1028.7$, triclinic, space group $P\bar{1}$ (No. 2), a 9.424(8), b 12.899(1), c 16.628(2) Å, α 93.386(9), β 102.118(8), γ 99.135(9)°, U 1942.3 Å³, D_c 1.76 g cm⁻³, $Z = 2$, Mo- K_α radiation, λ 0.71069 Å, μ 13.95 cm⁻¹. Nonius CAD4 diffractometer, 4962 unique reflections with $I > 3\sigma(I)$ in the range $3^\circ \leq \theta \leq 23^\circ$. Structure solved from a Patterson synthesis and successive Fourier maps and refined to $R = 0.053$ (341 parameters). Tables of atomic co-ordinates, interatomic distances and angles and thermal parameters have been deposited at the Cambridge Crystallographic Data Centre.



Scheme 1.

The behaviour of the solvent species $[\text{Ru}_2(\text{CO})_5(\text{solvent})\{\mu-(\text{RO})_2\text{PN}(\text{Et})\text{P}(\text{OR})_2\}]^{2+}$ towards various nucleophiles, as described above, is summarized in Scheme 1.

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