Improved Synthesis, Solution and Solid-state Structure, and Reactivity of $[Ru_2(\mu\text{-}SePh)_2(CO)_6]$†

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Both molecules consist of two metal–metal bonded $Ru(CO)_3$ moieties bridged by two SePh ligands. Each Ru atom is in a very distorted octahedral arrangement due to the strain imposed by the bridging ligands [Se–Ru–Se angles 79.1(1) and 79.0(1) (molecule 1), 78.8(1) and 79.1(1)° (molecule 2)] which force the axial CO ligands to deviate significantly from linearity with the Ru–Ru bonds [average OC_{ax}–Ru–Ru 153.8(1)°], while the equatorial CO ligands are approximately *trans* to the selenium atoms. The Ru–Ru distances, 2.700(1) (in molecule 1) and 2.703(1) Å (in molecule 2), are consistent with metal–metal bonds. The Ru_2Se_2 frameworks adopt butterfly conformations [average Ru–Se 2.521(1) Å] with the wings forming dihedral angles of 97.8(1)°. In both molecules the co-ordination around the Se atoms is a very distorted tetrahedron [Ru–Se–Ru angles 64.8(1) and 64.7(1) (molecule 1), 64.8(1) and 64.7(1)° (molecule 2)] with one co-ordination site occupied by the lone pair and with the phenyl groups adopting an *anti* configuration. Fig. 2 shows the main differences between molecules 1 and 2. In the former the ligands are much more eclipsed than in the latter (see torsion angles in Table 1) and the phenyl rings are not twisted equally about the Se–C bonds. Overall, the molecular structure of complex **1** is comparable to that found for the iron(I) dimer $[Fe_2(\mu\text{-SeEt})_2(CO)_6]$.[10]

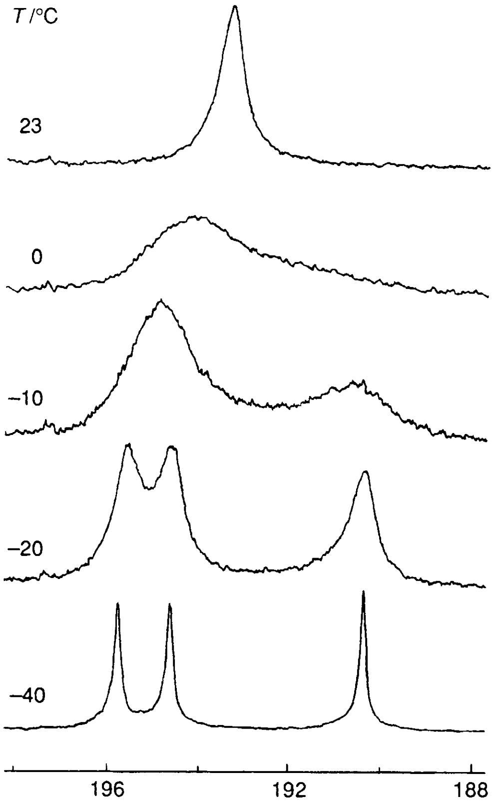


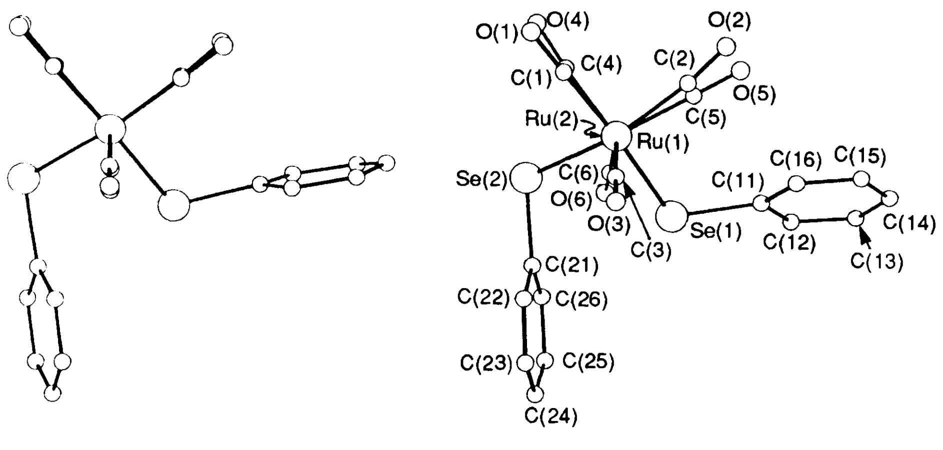
Fig. 1 Variable-temperature $^{13}C\text{-}\{^1H\}$ NMR spectra (CD_2Cl_2, 75.4 MHz) of a ^{13}CO-enriched sample of complex **1**

Although the reactivity of iron(I) dimers of the type $[Fe_2(\mu\text{-SR})_2(CO)_6]$ has been studied extensively,[11] that of the analogous ruthenium(I) sulphide, selenide, or telluride complexes remains almost unexplored.[12] We therefore decided to investigate some reactions of complex **1**. This complex reacted with one equivalent of triphenylphosphine giving a mixture of at least three compounds, as demonstrated by ^{31}P NMR spectroscopy, which could not be separated and were not investigated further. However, the reaction of complex **1** with two equivalents of triphenylphosphine gave a product which analysed for the disubstituted compound $[Ru_2(\mu\text{-SePh})_2(CO)_4(PPh_3)_2]$ **3**, but curiously its $^{31}P\text{-}\{^1H\}$ NMR spectrum showed two singlets with a 2:1 integral ratio. In most binuclear ruthenium(I) hexacarbonyl complexes the substitution of CO ligands takes place selectively at the positions *trans* to the Ru–Ru bond.[13] Accordingly, we propose that compound **3** is in fact a 2:1 mixture of *anti* and *syn* isomers. Although these isomers could not be separated, our proposal is supported by the $^{13}C\text{-}\{^1H\}$ NMR spectrum of the mixture, which showed the carbonyl resonances as three equal intensity singlets, two corresponding to the major component, the *anti* isomer, and one to the *syn* isomer.

The reactivity of complex **1** with other reagents has also been investigated. However, we have been unable to identify any well defined products from the reactions with acetonitrile, pyridine, bis(diphenylphosphino)methane or tetrafluoroboric acid and surprisingly, complex **1** does not react at room temperature with $Ag[BF_4]$–PPh_3, $[Cu(MeCN)_4][BF_4]$–PPh_3 or $[AuCl(PPh_3)]$–$Tl[PF_6]$ despite having lone pairs on the selenium atoms available for further co-ordination and presumably an electron rich Ru–Ru bond.

Experimental

Solvents were dried and distilled prior to use. All reactions were carried out under nitrogen, using standard Schlenk techniques. The compound $[Ru_3(CO)_{12}]$ was prepared by a literature method[14] and $[Ru_3(CO)_{12}]$, *ca.* 30% enriched in ^{13}CO, by stirring a solution of $[Ru_3(CO)_{12}]$ (1.2 g) in methylcyclohexane (100 cm^3) under *ca.* 1.5 atm (1.5 × 10^5 Pa) of ^{13}CO at 90 °C for



Molecule 1 Molecule 2

Fig. 2 Views, along the Ru–Ru vectors, of the two crystallographically independent molecules of *anti*-$[Ru_2(\mu\text{-SePh})_2(CO)_6]$

Table 1 Selected bond lengths (Å), angles (°) and torsion angles (°) in complex **1**

Molecule 1				Molecule 2			
Ru(11)–Ru(21)	2.700(1)	Ru(21)–C(51)	1.914(13)	Ru(12)–Ru(22)	2.703(1)	Ru(22)–C(52)	1.898(13)
Ru(11)–C(11)	1.871(14)	Ru(21)–C(61)	1.935(13)	Ru(12)–C(12)	1.860(15)	Ru(22)–C(62)	1.890(13)
Ru(11)–C(21)	1.887(12)	Ru(21)–Se(11)	2.519(2)	Ru(12)–C(22)	1.918(13)	Ru(22)–Se(12)	2.523(1)
Ru(11)–C(31)	1.933(13)	Ru(21)–Se(21)	2.529(2)	Ru(12)–C(32)	1.937(13)	Ru(22)–Se(22)	2.506(2)
Ru(11)–Se(11)	2.521(1)	Se(11)–C(111)	1.905(11)	Ru(12)–Se(12)	2.522(2)	Se(12)–C(112)	1.926(12)
Ru(11)–Se(21)	2.519(2)	Se(21)–C(211)	1.941(12)	Ru(12)–Se(22)	2.522(2)	Se(22)–C(212)	1.921(12)
Ru(21)–C(41)	1.847(15)			Ru(22)–C(42)	1.856(14)		
Ru(21)–Ru(11)–C(11)	104.0(4)	Ru(11)–Ru(21)–C(61)	153.4(4)	Ru(22)–Ru(12)–C(12)	107.1(4)	Ru(12)–Ru(22)–C(62)	154.2(4)
Ru(21)–Ru(11)–C(21)	97.9(4)	C(41)–Ru(21)–C(61)	97.8(6)	Ru(22)–Ru(12)–C(22)	95.5(4)	C(42)–Ru(22)–C(62)	95.5(6)
C(11)–Ru(11)–C(21)	92.6(5)	C(51)–Ru(21)–C(61)	98.7(5)	C(12)–Ru(12)–C(22)	90.1(6)	C(52)–Ru(22)–C(62)	97.3(6)
Ru(21)–Ru(11)–C(31)	155.4(3)	Ru(11)–Ru(21)–Se(11)	57.7(1)	Ru(22)–Ru(12)–C(32)	152.4(4)	Ru(12)–Ru(22)–Se(12)	57.6(1)
C(11)–Ru(11)–C(31)	94.4(5)	C(41)–Ru(21)–Se(11)	160.2(4)	C(12)–Ru(12)–C(32)	94.4(6)	C(42)–Ru(22)–Se(12)	160.7(4)
C(21)–Ru(11)–C(31)	97.5(5)	C(51)–Ru(21)–Se(11)	93.7(4)	C(22)–Ru(12)–C(32)	101.8(6)	C(52)–Ru(22)–Se(12)	89.7(4)
Ru(21)–Ru(11)–Se(11)	57.6(1)	C(61)–Ru(21)–Se(11)	100.6(4)	Ru(22)–Ru(12)–Se(12)	57.6(1)	C(62)–Ru(22)–Se(12)	102.2(4)
C(11)–Ru(11)–Se(11)	161.3(4)	Ru(11)–Ru(21)–Se(21)	57.5(1)	C(12)–Ru(12)–Se(12)	164.1(4)	Ru(12)–Ru(22)–Se(22)	57.8(1)
C(21)–Ru(11)–Se(11)	92.7(3)	C(41)–Ru(21)–Se(21)	88.8(4)	C(22)–Ru(12)–Se(12)	95.8(5)	C(42)–Ru(22)–Se(22)	88.9(5)
C(31)–Ru(11)–Se(11)	102.6(4)	C(51)–Ru(21)–Se(21)	154.4(4)	C(32)–Ru(12)–Se(12)	98.9(4)	C(52)–Ru(22)–Se(22)	156.1(4)
Ru(21)–Ru(11)–Se(21)	57.9(1)	C(61)–Ru(21)–Se(21)	106.7(4)	Ru(22)–Ru(12)–Se(22)	57.2(1)	C(62)–Ru(22)–Se(22)	105.6(4)
C(11)–Ru(11)–Se(21)	88.6(4)	Se(11)–Ru(21)–Se(21)	79.0(1)	C(12)–Ru(12)–Se(22)	88.8(4)	Se(12)–Ru(22)–Se(22)	79.1(1)
C(21)–Ru(11)–Se(21)	155.1(4)	Ru(11)–Se(11)–Ru(21)	64.8(1)	C(22)–Ru(12)–Se(22)	150.7(4)	Ru(12)–Se(12)–Ru(22)	64.8(1)
C(31)–Ru(11)–Se(21)	107.3(4)	Ru(11)–Se(11)–C(111)	110.0(3)	C(32)–Ru(12)–Se(22)	107.5(4)	Ru(12)–Se(12)–C(112)	112.7(4)
Se(11)–Ru(11)–Se(21)	79.1(1)	Ru(21)–Se(11)–C(111)	110.4(3)	Se(12)–Ru(12)–Se(22)	78.8(1)	Ru(22)–Se(12)–C(112)	109.1(3)
Ru(11)–Ru(21)–C(41)	102.6(4)	Ru(11)–Se(21)–Ru(21)	64.7(1)	Ru(12)–Ru(22)–C(42)	103.2(4)	Ru(12)–Se(22)–Ru(22)	65.0(1)
Ru(11)–Ru(21)–C(51)	97.9(4)	Ru(11)–Se(21)–C(211)	109.1(4)	Ru(12)–Ru(22)–C(52)	98.4(4)	Ru(12)–Se(22)–C(212)	109.2(4)
C(41)–Ru(21)–C(51)	90.6(6)	Ru(21)–Se(21)–C(211)	108.1(3)	C(42)–Ru(22)–C(52)	95.6(6)	Ru(22)–Se(22)–C(212)	108.1(4)
		Se(21)–Ru(11)–Ru(21)–Se(11)	−97.77(9)	Se(12)–Ru(12)–Ru(22)–Se(22)	97.82(9)		
		C(11)–Ru(11)–Ru(21)–C(41)	1.1(6)	C(12)–Ru(12)–Ru(22)–C(42)	3.1(6)		
		C(21)–Ru(11)–Ru(21)–C(51)	−1.2(5)	C(22)–Ru(12)–Ru(22)–C(52)	9.2(6)		
		C(31)–Ru(11)–Ru(21)–C(61)	−1.0(1)	C(32)–Ru(12)–Ru(22)–C(62)	7.0(1)		

48 h, the ^{13}CO (99.7% ^{13}C, 14.3% ^{18}O) being obtained from Isotec Inc. All other reagents were purchased from Aldrich and used as received. The ^{13}C NMR spectra were recorded using ^{13}CO-enriched $[Ru_3(CO)_{12}]$. Instrumentation was as follows: Perkin-Elmer FT 1720-X (IR), Bruker AC-300 (NMR) and Perkin-Elmer 240-B (microanalysis).

Reaction of $[Ru_3(CO)_{12}]$ *with Diphenyl Diselenide.*—The compounds $[Ru_3(CO)_{12}]$ (600 mg, 0.936 mmol) and Ph_2Se_2 (444 mg, 1.42 mmol) were stirred in thf (25 cm^3) at reflux for 1.5 h. The solvent was removed under reduced pressure from the red-orange solution and the residue extracted with four 5 cm^3 portions of hexane–diethyl ether (2:1 v/v). The insoluble yellow-brown material was washed with diethyl ether and identified as the known[1] $[\{Ru(\mu\text{-}SePh)_2(CO)_2\}_n]$ (260 mg, 20% based on Ru) (Found: C, 35.6; H, 2.1. $C_{14}H_{10}O_2RuSe_2$ requires C, 35.85; H, 2.15%). IR (CH_2Cl_2): ν(CO) at 2100m, 2043s and 1974m cm^{-1}. The combined extracts were evaporated to dryness and the residue washed with two 1 cm^3 portions of methanol to give *anti*-$[Ru_2(\mu\text{-}SePh)_2(CO)_6]$ as a yellow-orange powder (483 mg, 50% based on Ru) (Found: 31.6; H, 1.4. $C_{18}H_{10}O_6Ru_2Se_2$ requires C, 31.7; H, 1.5%). IR (hexane): ν(CO) at 2081s, 2053s, 2009s, 2005s, 1994m and 1963w cm^{-1}. ^{13}C-$\{^1H\}$ NMR (CD_2Cl_2, −40 °C, internal $SiMe_4$, 75.47 MHz): δ(CO) 195.7(s), 194.6(s) and 190.3(s) ppm.

Reaction of anti-$[Ru_2(\mu\text{-}SePh)_2(CO)_2]$ *with Triphenylphosphine.*—Triphenylphosphine (24 mg, 0.09 mmol) and *anti*-$[Ru_2(\mu\text{-}SePh)_2(CO)_6]$ (30 mg, 0.044 mmol) were stirred in refluxing thf (10 cm^3) for 45 min. The solvent was removed under reduced pressure and the residue washed with two 5 cm^3 portions of hexane to give a dark orange solid, subsequently identified as a 2:1 mixture of *anti*- and *syn*-$[Ru_2(\mu\text{-}SePh)_2(CO)_4(PPh_3)_2]$ (30 mg, 60%) (Found: C, 54.0; H, 3.4. $C_{52}H_{40}P_2O_4Ru_2Se_2$ requires C, 54.25; H, 3.5%). IR (CH_2Cl_2): ν(CO) at 2032m, 2012s, 1975s and 1984s cm^{-1}. ^{31}P-$\{^1H\}$ NMR (CD_2Cl_2, 23 °C, external 85% H_3PO_4, 121.5 MHz); δ 35.2(s) and 30.6(s) ppm, integral ratio 2:1. ^{13}C-$\{^1H\}$ NMR (CD_2Cl_2, 23 °C, internal $SiMe_4$, 75.47 MHz): δ(CO) 203.7(s), 202.9(s) and 201.5(s) ppm, integral ratio 1:1:1.

Crystal and Refinement Data for Compound **1**.—$C_{18}H_{10}O_6Ru_2Se_2$, M = 682.3, monoclinic, $P2_1/n$ (from systematic absences), a = 17.305(7), b = 16.798(5), c = 15.254(3) Å, β = 101.28(2)°, U = 4348(2) $Å^3$, D_c = 2.08 g cm^{-3}, Z = 8, $F(000)$ = 2576, λ(Mo-Kα) = 0.710 73 Å, μ(Mo-Kα) = 47.08 cm^{-1}, room temperature. Orange prismatic crystal (0.23 × 0.17 × 0.13 mm), obtained from a saturated hexane solution at −20 °C. Intensities were collected on an Enraf-Nonius CAD4 diffractometer, using the ω–2θ scan technique. 7626 Reflections were measured in the range $0 \leqslant \theta \leqslant 25°$ ($-20 \leqslant h \leqslant 20$, $0 \leqslant k \leqslant 19$, $0 \leqslant l \leqslant 18$); 2889 with $I \geqslant 3\sigma(I)$ were used in the refinement. Profile analysis[15] was made in all reflections. An empirical (ψ-scan based) absorption correction was applied (min. 0.961, max. 1.000). Several attempts to solve the structure by Patterson interpretation with SHELX 86[16] in the proper space group $P2_1/n$ were fruitless. Symmetry was lowered and the structure was solved in the space group $P2_1$ (heavy atoms from Patterson synthesis and the remaining non-H atoms with DIRDIF[17]). The symmetry centre was located and the refinement was continued in the centrosymmetric space group $P2_1/n$. Least-squares refinement was made with SHELX 76[18] in two blocks (one for each independent molecule in the asymmetric unit). After isotropic refinement of the non-H atoms (R = 0.081), an additional absorption correction was applied with DIFABS[19] (min. 0.749, max. 1.163). The R factor decreased to 0.061. All non-H atoms were then refined anisotropically; H atoms were positioned geometrically, riding on their adjacent atoms, with overall isotropic thermal parameters of 0.08 $Å^2$, and were not refined. Applied weighting $w = [\sigma^2(F) + 0.0003\,F^2]^{-1}$. Number of parameters 507. Final R 0.035 (R' 0.034). Maximum residual 0.59 e $Å^{-3}$ in the proximity

Table 2 Fractional atomic coordinates for complex **1**

Molecule 1

Atom	*x*	*y*	*z*
Ru(11)	0.094 82(5)	−0.306 00(5)	0.071 21(6)
Ru(21)	−0.026 13(6)	−0.202 03(6)	0.054 89(7)
C(11)	0.113 9(7)	−0.334 6(7)	0.192 1(9)
O(11)	0.125 7(6)	−0.351 5(5)	0.266 5(6)
C(21)	0.177 8(7)	−0.231 2(7)	0.089 2(8)
O(21)	0.224 9(5)	−0.183 0(5)	0.101 1(6)
C(31)	0.153 0(7)	−0.394 6(7)	0.035 7(9)
O(31)	0.188 9(5)	−0.445 6(5)	0.016 1(7)
C(41)	−0.044 0(7)	−0.196 1(8)	0.170 2(9)
O(41)	−0.052 4(6)	−0.192 8(7)	0.243 3(6)
C(51)	0.034 7(8)	−0.106 1(8)	0.072 5(9)
O(51)	0.072 8(6)	−0.051 7(5)	0.087 5(8)
C(61)	−0.125 2(8)	−0.157 2(8)	−0.005 8(9)
O(61)	−0.183 7(6)	−0.130 0(7)	−0.038 1(7)
Se(11)	0.031 42(7)	−0.245 67(6)	−0.076 75(8)
Se(21)	−0.045 67(7)	−0.351 08(7)	0.059 27(9)
C(111)	0.097 5(6)	−0.164 5(6)	−0.109 1(7)
C(121)	0.071 8(8)	−0.087 8(7)	−0.124 8(9)
C(131)	0.118(1)	−0.031 7(9)	−0.157(1)
C(141)	0.190(1)	−0.052(1)	−0.168(1)
C(151)	0.217 7(8)	−0.131(1)	−0.159(1)
C(161)	0.168 5(7)	−0.186 4(7)	−0.128 9(8)
C(211)	−0.086 1(8)	−0.387 4(6)	−0.061 8(8)
C(221)	−0.042 7(8)	−0.434 0(7)	−0.107 7(9)
C(231)	−0.074 6(9)	−0.460 6(7)	−0.192(1)
C(241)	−0.149 4(9)	−0.440 7(8)	−0.232 0(9)
C(251)	−0.193 0(8)	−0.394 2(8)	−0.186 8(10)
C(261)	−0.162 0(8)	−0.367 3(7)	−0.101(1)

Molecule 2

Atom	*x*	*y*	*z*
Ru(12)	−0.079 81(6)	0.194 91(5)	−0.510 67(7)
Ru(22)	0.023 85(5)	0.315 99(5)	−0.505 35(7)
C(12)	−0.106 7(7)	0.189 3(7)	−0.398 6(9)
O(12)	−0.125 4(6)	0.182 7(6)	−0.329 2(7)
C(22)	−0.001 5(8)	0.114 1(7)	−0.476 0(9)
O(22)	0.046 3(6)	0.070 3(6)	−0.447 0(7)
C(32)	−0.169 4(8)	0.132 7(8)	−0.569 3(9)
O(32)	−0.221 8(7)	0.096 4(7)	−0.605 3(8)
C(42)	0.038 5(8)	0.350 4(8)	−0.387 7(9)
O(42)	0.051 4(6)	0.370 4(6)	−0.312 8(7)
C(52)	0.118 0(8)	0.255 1(7)	−0.490 0(9)
O(52)	0.173 2(5)	0.216 5(5)	−0.480 2(6)
C(62)	0.063 7(7)	0.409 3(8)	−0.549 6(9)
O(62)	0.089 3(6)	0.467 0(5)	−0.572 7(7)
Se(12)	−0.029 48(7)	0.239 96(6)	−0.646 93(8)
Se(22)	−0.121 13(7)	0.339 14(7)	−0.519 72(9)
C(112)	0.052 0(7)	0.171 7(7)	−0.674 5(8)
C(122)	0.049 2(9)	0.090 3(8)	−0.666 8(9)
C(132)	0.108(1)	0.045 0(9)	−0.693(1)
C(142)	0.163(1)	0.081(1)	−0.733(1)
C(152)	0.167 0(8)	0.162 5(9)	−0.740 8(9)
C(162)	0.109 1(7)	0.206 8(7)	−0.712 9(8)
C(212)	−0.163 9(7)	0.365 9(7)	−0.642 1(8)
C(222)	−0.233 2(8)	0.332 5(7)	−0.684 7(9)
C(232)	−0.267 3(8)	0.352 8(9)	−0.772 3(10)
C(242)	−0.232 3(9)	0.407 3(9)	−0.817 1(9)
C(252)	−0.162 4(9)	0.440 1(8)	−0.775(1)
C(262)	−0.129 3(7)	0.421 4(7)	−0.686 9(10)

of Se(11). Maximum shift–error ratio in the last cycle 0.043. The final atomic coordinates are listed in Table 2. Drawings were made with PLUTO.[20] Least-squares planes and torsion angles calculations were made with PARST.[21]

Additional material available from the Cambridge Crystallographic Data Centre comprises H-atom coordinates, thermal parameters and remaining bond distances and angles.

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