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Molecular structures of $Fe_4(CO)_{10}(\mu\text{-}CO)(\mu_4\text{-}Se)_2$ and $Fe_3Ru(CO)_{10}(\mu\text{-}CO)(\mu_4\text{-}Se)_2$

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Abstract

The results of the X-ray analyses of $Fe_4(CO)_{10}(\mu\text{-}CO)(\mu_4\text{-}Se)_2$ and $Fe_3Ru(CO)_{10}(\mu\text{-}CO)(\mu_4\text{-}Se)_2$ are reported. The two compounds are isomorphous and belong to the orthorhombic space group *Pccn* with $a = 6.655(1)$, $b = 15.587(2)$, $c = 17.387(2)$ Å for $Fe_4(CO)_{10}(\mu\text{-}CO)(\mu_4\text{-}Se)_2$ and $a = 6.630(1)$, $b = 15.785(2)$, $c = 17.629(3)$ Å for $Fe_3Ru(CO)_{10}(\mu\text{-}CO)(\mu_4\text{-}Se)_2$. The structures were solved by direct methods and refined, correcting for extinction, to $R = 0.0306$ and 0.0312 for 1171 and 1277 observed reflections for $Fe_4(CO)_{10}(\mu\text{-}CO)(\mu_4\text{-}Se)_2$ and $Fe_3Ru(CO)_{10}(\mu\text{-}CO)(\mu_4\text{-}Se)_2$ respectively. They belong to the $[M_4(CO)_{11}XY]$ clusters which are formally unsaturated according to the 18-electron rules but conform to the Wade's rules of electron counting. Both complexes contain terminal, bridging and semibridging carbonyl groups.

Key words: Iron; Ruthenium; Selenium; Carbonyl; Cluster

1. Introduction

The closo square-bipyramidal $M_4E_2(CO)_{11}$ clusters, where the ligand E is GeR, PR, AsR, S, Se or Te have been of considerable interest in recent times [1]. These clusters have attracted interest from both the synthetic as well as the theoretical chemists [2]. The preparation of these compounds can be accomplished in any of a number of different ways. Two electron counts for these clusters are possible and the number of skeletal electron pairs can be eight or seven depending on whether a metal–metal π antibonding molecular orbital (MO) is occupied or not.

We have recently reported facile synthesis of a series of $M_4E_2(CO)_{11}$ clusters [3], including the single crystal X-ray analyses of $Ru_4Te_2(CO)_{11}$ [4] and $Fe_2Ru_2Te_2(CO)_{11}$ [5]. The structures of $Fe_4S_2(CO)_{11}$ [6], $Ru_4S_2(CO)_9(PMe_2Ph)_2$ [7], $Ru_4Se_2(CO)_{11}$ [8] and $Ru_4Se_2(CO)_{10}(SbPh_3)$ [9] have also been reported. In continuation of our interest in the $M_4E_2(CO)_{11}$ clusters, where E = Se, we report here the results of single-crystal X-ray structure analyses of $Fe_4Se_2(CO)_{11}$ and $Fe_3RuSe_2(CO)_{11}$.

2. Results and discussion

The molecular structures of the two isomorphous compounds $Fe_4Se_2(CO)_{11}$ and $Fe_3RuSe_2(CO)_{11}$ are shown in Figs. 1 and 2 respectively. Tables 1 and 2 list the bond parameters for the two compounds. The structures consist of a planar arrangement of the four metal atoms which are quadruply bridged on either side of the M_4 plane by the two Se atoms. In both clusters, there are three types of carbonyl group attached to the metal atoms. Each metal is associated with two terminal carbonyl groups. One edge of the M_4 unit is bridged by a carbonyl group and the two sides adjacent to the bridged edge contain one semibridging carbonyl group each. Typically, in $Ru_4Te_2(CO)_{11}$, $Ru_4Se_2(CO)_{11}$ and $Fe_4S_2(CO)_{11}$, the shortest edge of the M_4 unit is the edge which is bridged by a carbonyl group while the side opposite to it forms the longest edge. The two sides which contain the semibridging carbonyl groups are typically equal and intermediate in length in these clusters. The shortest edge of the Fe_4 unit in $Fe_4Se_2(CO)_{11}$ is the Fe2–Fe2′ (2.534(1) Å) and this edge is bridged by a carbonyl group. The edge opposite to this, Fe1–Fe1′ (2.692(1) Å) is the longest side and is not associated with any bridging carbonyl groups. The two remaining sides, Fe1–Fe2 and Fe1′–

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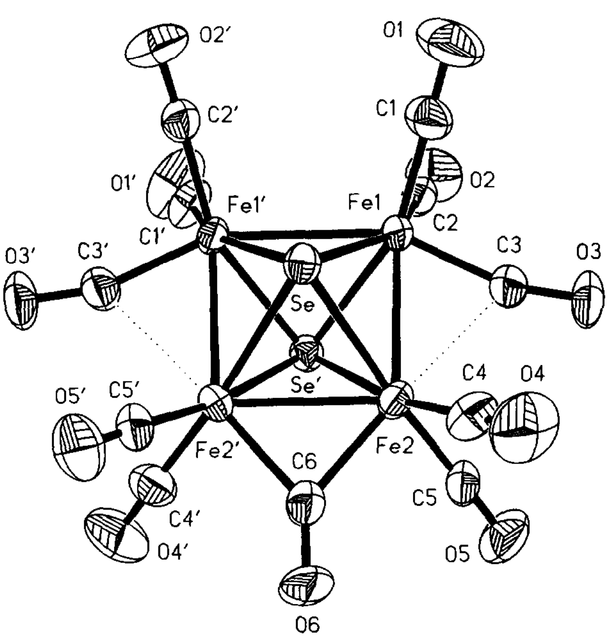

Fig. 1. Molecular structure of $Fe_4Se_2(CO)_{11}$.

TABLE 1. Bond lengths (Å) and bond angles (°) for $Fe_4Se_2(CO)_{11}$

Se–Fe(1)	2.406(1)	Se–Fe(2)	2.449(1)
Se–Fe(1A)	2.398(1)	Se–Fe(2A)	2.458(1)
Fe(1)–Fe(2)	2.596(1)	Fe(1)–C(1)	1.791(7)
Fe(1)–C(2)	1.796(6)	Fe(1)–C(3)	1.807(6)
Fe(1)–SeA	2.398(1)	Fe(1)–Fe(1A)	2.692(1)
Fe(2)–C(3)	2.418(6)	Fe(2)–C(4)	1.790(6)
Fe(2)–C(5)	1.781(6)	Fe(2)–C(6)	1.950(6)
Fe(2)–SeA	2.458(1)	Fe(2)–Fe(2A)	2.534(1)
C(1)–O(1)	1.121(8)	C(2)–O(2)	1.124(7)
C(3)–O(3)	1.147(8)	C(4)–O(4)	1.128(8)
C(5)–O(5)	1.141(7)	C(6)–O(6)	1.145(10)
C(6)–Fe(2A)	1.950(6)		
Fe(1)–Se–Fe(2)	64.6(1)	Fe(1)–Se–Fe(1A)	68.2(1)
Fe(2)–Se–Fe(1A)	98.9(1)	Fe(1)–Se–Fe(2A)	98.4(1)
Fe(2)–Se–Fe(2A)	62.2(1)	Fe(1A)–Se–Fe(2A)	64.6(1)
Se–Fe(1)–Fe(2)	58.5(1)	Se–Fe(1)–C(1)	90.2(2)
Fe(2)–Fe(1)–C(1)	131.4(2)	Se–Fe(1)–C(2)	158.0(2)
Fe(2)–Fe(1)–C(2)	133.5(2)	C(1)–Fe(1)–C(2)	89.7(3)
Se–Fe(1)–C(3)	106.3(2)	Fe(2)–Fe(1)–C(3)	63.7(2)
C(1)–Fe(1)–C(3)	96.6(3)	C(2)–Fe(1)–C(3)	95.6(3)
Se–Fe(1)–SeA	82.2(1)	Fe(2)–Fe(1)–SeA	58.8(1)
C(1)–Fe(1)–SeA	159.8(2)	C(2)–Fe(1)–SeA	90.4(2)
C(3)–Fe(1)–SeA	103.5(2)	Se–Fe(1)–Fe(1A)	55.8(1)
Fe(2)–Fe(1)–Fe(1A)	88.3(1)	C(1)–Fe(1)–Fe(1A)	104.3(2)
C(2)–Fe(1)–Fe(1A)	103.0(2)	C(3)–Fe(1)–Fe(1A)	151.9(2)
SeA–Fe(1)–Fe(1A)	56.1(1)	Se–Fe(2)–Fe(1)	56.9(1)
Se–Fe(2)–C(3)	88.3(1)	Fe(1)–Fe(2)–C(3)	42.0(1)
Se–Fe(2)–C(4)	92.9(2)	Fe(1)–Fe(2)–C(4)	113.9(2)
C(3)–Fe(2)–C(4)	87.1(2)	Se–Fe(2)–C(5)	172.5(2)
Fe(1)–Fe(2)–C(5)	115.7(2)	C(3)–Fe(2)–C(5)	85.4(2)
C(4)–Fe(2)–C(5)	90.8(3)	Se–Fe(2)–C(6)	95.5(1)
Fe(1)–Fe(2)–C(6)	141.2(2)	C(3)–Fe(2)–C(6)	176.1(2)
C(4)–Fe(2)–C(6)	92.2(2)	C(5)–Fe(2)–C(6)	90.8(2)
Se–Fe(2)–SeA	80.2(1)	Fe(1)–Fe(2)–SeA	56.6(1)
C(3)–Fe(2)–SeA	85.9(1)	C(4)–Fe(2)–SeA	170.3(2)
C(5)–Fe(2)–SeA	95.3(2)	C(6)–Fe(2)–SeA	95.2(1)
Se–Fe(2)–Fe(2A)	59.1(1)	Fe(1)–Fe(2)–Fe(2A)	91.7(1)
C(3)–Fe(2)–Fe(2A)	133.7(1)	C(4)–Fe(2)–Fe(2A)	123.2(2)
C(5)–Fe(2)–Fe(2A)	123.5(2)	C(6)–Fe(2)–Fe(2A)	49.5(2)
SeA–Fe(2)–Fe(2A)	58.7(1)	Fe(1)–C(1)–O(1)	178.7(6)
Fe(1)–C(2)–O(2)	177.9(5)	Fe(1)–C(3)–Fe(2)	74.2(2)
Fe(1)–C(3)–O(3)	161.6(5)	Fe(2)–C(3)–O(3)	124.1(4)
Fe(2)–C(4)–O(4)	178.8(6)	Fe(2)–C(5)–O(5)	177.9(5)
Fe(2)–C(6)–O(6)	139.5(2)	Fe(2)–C(6)–Fe(2A)	81.0(3)
O(6)–C(6)–Fe(2A)	139.5(2)		

Fe2' are equal in length (2.596(1) Å) and are associated with one semibridging carbonyl group each (Fe1–C3–O3 = 161.6(5)°). The remaining eight carbonyl groups are terminally bonded, two on each Fe atom. Prior to this report, there has been only one previous report of a mixed metal example of these type of clusters, $Fe_2Ru_2Te_2(CO)_{11}$, to have been structurally characterized by X-ray methods. The disposition of the carbonyl groups on the Fe_2Ru_2 framework is similar to that observed in the other clusters belonging to the $M_4E_2(CO)_{11}$ type which have been structurally characterized. In both $Fe_2Ru_2Te_2(CO)_{11}$ and $Fe_3RuSe_2(CO)_{11}$, the Ru atoms are associated with more bridging carbonyl groups than are the Fe atoms. Whereas in $Fe_2Ru_2Te_2(CO)_{11}$ the bridged Ru–Ru side forms the shortest metal–metal bond, in $Fe_3RuSe_2\text{-}(CO)_{11}$ the Fe–Ru bond, which is bridged by a carbonyl group, is equal in length to the unbridged Fe–Fe bond opposite to it (2.678(1) Å). The two bonds which are adjacent to the bridged bond, Fe'–Ru' (2.721(1) Å) and the Fe–Fe (2.717(1) Å) contain the semibridging carbonyl groups. The distances between the Fe atoms and the quadruply

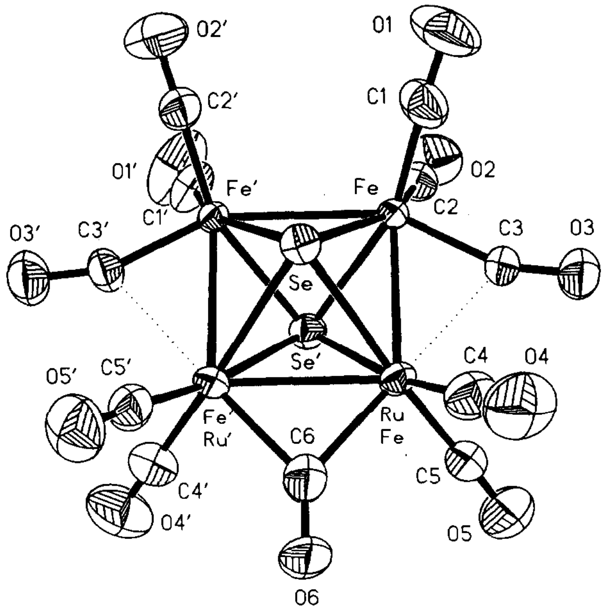

Fig. 2. Molecular structure of $Fe_3RuSe_2(CO)_{11}$.

TABLE 2. Bond lengths (Å) and bond angles (°) for $Fe_3Se_2Ru(CO)_{11}$

Se–Fe	2.443(1)	Se–Ru′	2.547(1)
Se–Fe′	2.547(1)	Se–FeA	2.433(1)
Se–Ru′A	2.562(1)	Fe–Ru′	2.678(1)
Fe–Fe′	2.678(1)	Fe–C(1)	1.791(8)
Fe–C(2)	1.785(6)	Fe–C(3)	1.823(6)
Fe–SeA	2.433(1)	Fe–FeA	2.717(1)
Fe′–C(3)	2.411(6)	Fe′–C(4)	1.839(8)
Fe′–C(5)	1.845(7)	Fe′–C(6)	2.025(8)
Fe′–SeA	2.562(1)	Fe′–Ru′A	2.721(1)
C(1)–O(1)	1.123(10)	C(2)–O(2)	1.125(8)
C(3)–Ru′	2.411(6)	C(3)–O(3)	1.147(9)
C(4)–Ru′	1.839(8)	C(4)–O(4)	1.113(10)
C(5)–Ru′	1.845(7)	C(5)–O(5)	1.135(9)
C(6)–Ru′	2.025(8)	C(6)–O(6)	1.168(13)
C(6)–Ru′A	2.025(8)		
Fe–Se–Ru′	64.9(1)	Fe–Se–Fe′	64.9(1)
Fe–Se–FeA	67.7(1)	Ru′–Se–FeA	100.0(1)
Fe′–Se–FeA	100.0(1)	Fe–Se–Ru′A	99.3(1)
Ru′–Se–Ru′A	64.3(1)	Fe′–Se–Ru′A	64.3(1)
FeA–Se–Ru′A	64.8(1)	Se–Fe–Ru′	59.4(1)
Se–Fe–Fe′	59.4(1)	Se–Fe–C(1)	90.0(2)
Ru′–Fe–C(1)	130.4(3)	Fe′–Fe–C(1)	130.4(3)
Se–Fe–C(2)	159.4(2)	Ru′–Fe–C(2)	131.9(2)
Fe′–Fe–C(2)	131.9(2)	C(1)–Fe–C(2)	90.7(3)
Se–Fe–C(3)	105.5(2)	Ru′–Fe–C(3)	61.4(2)
Fe′–Fe–C(3)	61.4(2)	C(1)–Fe–C(3)	96.4(3)
C(2)–Fe–C(3)	94.9(3)	Se–Fe–SeA	82.5(1)
Ru′–Fe–SeA	60.0(1)	Fe′–Fe–SeA	60.0(1)
C(1)–Fe–SeA	160.4(2)	C(2)–Fe–SeA	90.1(2)
C(3)–Fe–SeA	103.1(2)	Se–Fe–FeA	56.0(1)
Ru′–Fe–FeA	90.0(1)	Fe′–Fe–FeA	90.0(1)
C(1)–Fe–FeA	104.6(2)	C(2)–Fe–FeA	104.1(2)
C(3)–Fe–FeA	151.3(2)	SeA–Fe–FeA	56.3(1)
Se–Ru′–Fe	55.7(1)	Se–Ru′–C(3)	87.1(1)
Fe–Ru′–C(3)	41.6(1)	Se–Ru′–C(4)	93.8(2)
Fe–Ru′–C(4)	115.9(2)	C(3)–Ru′–C(4)	89.9(3)
Se–Ru′–C(5)	172.5(2)	Fe–Ru′–C(5)	117.1(2)
C(3)–Ru′–C(5)	87.6(3)	C(4)–Ru′–C(5)	91.5(3)
Se–Ru′–C(6)	93.8(2)	Fe–Ru′–C(6)	137.8(2)
C(3)–Ru′–C(6)	177.7(2)	C(4)–Ru′–C(6)	92.2(3)
C(5)–Ru′–C(6)	91.4(3)	Se–Ru′–SeA	78.0(1)
Fe–Ru′–SeA	55.3(1)	C(3)–Ru′–SeA	84.8(2)
C(4)–Ru′–SeA	170.3(2)	C(5)–Ru′–SeA	96.3(2)
C(6)–Ru′–SeA	93.3(2)	Se–Ru′–Ru′A	58.1(1)
Fe–Ru′–Ru′A	90.0(1)	C(3)–Ru′–Ru′A	131.5(1)
C(4)–Ru′–Ru′A	122.1(2)	C(5)–Ru′–Ru′A	122.9(2)
C(6)–Ru′–Ru′A	47.8(2)	SeA–Ru′–Ru′A	57.6(1)
Se–Fe′–Fe	55.7(1)	Se–Fe′–C(3)	87.1(1)
Fe–Fe′–C(3)	41.6(1)	Se–Fe′–C(4)	93.8(2)
Fe–Fe′–C(4)	115.9(2)	C(3)–Fe′–C(4)	89.9(3)
Se–Fe′–C(5)	172.5(2)	Fe–Fe′–C(5)	117.1(2)
C(3)–Fe′–C(5)	87.6(3)	C(4)–Fe′–C(5)	91.5(3)
Se–Fe′–C(6)	93.8(2)	Fe–Fe′–C(6)	137.8(2)
C(3)–Fe′–C(6)	177.7(2)	C(4)–Fe′–C(6)	92.2(3)
C(5)–Fe′–C(6)	91.4(3)	Se–Fe′–SeA	78.0(1)
Fe–Fe′–SeA	55.3(1)	C(3)–Fe′–SeA	84.8(2)
C(4)–Fe′–SeA	170.3(2)	C(5)–Fe′–SeA	96.3(2)
C(6)–Fe′–SeA	93.3(2)	Se–Fe′–Ru′A	58.1(1)
Fe–Fe′–Ru′A	90.0(1)	C(3)–Fe′–Ru′A	131.5(1)
C(4)–Fe′–Ru′A	122.1(2)	C(5)–Fe′–Ru′A	122.9(2)
C(6)–Fe′–Ru′A	47.8(2)	SeA–Fe′–Ru′A	57.6(1)
Fe–C(1)–O(1)	177.2(7)	Fe–C(2)–O(2)	176.7(6)

TABLE 2 (continued).

Fe–C(3)–Ru′	77.1(2)	Fe–C(3)–Fe′	77.1(2)
Fe–C(3)–O(3)	158.0(5)	Ru′–C(3)–O(3)	124.9(5)
Fe′–C(3)–O(3)	124.9(5)	Ru′–C(4)–O(4)	179.3(7)
Fe′–C(4)–O(4)	179.3(7)	Ru′–C(5)–O(5)	177.0(6)
Fe′–C(5)–O(5)	177.0(6)	Ru′–C(6)–O(6)	137.8(2)
Fe′–C(6)–O(6)	137.8(2)	Ru′–C(6)–Ru′A	84.4(4)
Fe′–C(6)–Ru′A	84.4(4)	O(6)–C(6)–Ru′A	137.8(2)

bridging Se atoms in $Fe_4Se_2(CO)_{11}$ (2.398(1)–2.458(1) Å) and in $Fe_3RuSe_2(CO)_{11}$ (2.433(1)–2.562(1) Å) are slightly longer than the distances between the Fe atoms and the triply bridging Se atoms in $Fe_3Se_2(CO)_{11}$ (2.34–2.37 Å) [10].

3. Experimental details

The two clusters were prepared as reported earlier [11] and crystallized at −10°C from petroleum ether solutions. Crystal data are collected in Table 3. Data collection, solution and refinement details are given in Table 4. The atomic coordinates and equivalent isotropic displacements for $Fe_4(CO)_{10}(\mu\text{-}CO)(\mu_4\text{-}Se)_2$ and $Fe_3Ru(CO)_{10}(\mu\text{-}CO)(\mu_4\text{-}Se)_2$ are listed in Tables 5 and 6, respectively. Crystals were checked for quality by polaroid photographs and measured with Mo radiation (50 kV, 45 mA). The XSCANS data collection package was used in fully automatic mode to determine the unit cell by hemispherical search and verified by a fractional search, obtaining better esd's by increasing the number of reflections in the list with a thin-shell search, transforming the cell to a conventional cell with the LePage–Bravais algorithm and verifying the symmetry with an automatic Laue measurement (including absorption correction). Finally the unit cell was refined by non-linear least-squares refinement including refinement of diffractometer zeros and crystal offset. For

TABLE 3. Crystal data

Empirical formula	$Fe_4Se_2(CO)_{11}$	$Fe_3Se_2Ru(CO)_{11}$
Colour; habit	black; plate	black; plate
Crystal size	0.03 × 0.08 × 0.14 mm	0.01 × 0.05 × 0.12 mm
Crystal system	Orthorhombic	Orthorhombic
Space group	*Pccn*	*Pccn*
Unit cell dimensions	$a = 6.655(1)$ Å	$a = 6.630(1)$ Å
	$b = 15.587(2)$ Å	$b = 15.785(2)$ Å
	$c = 17.387(2)$ Å	$c = 17.629(3)$ Å
Volume	1803.6(4) Å^3	1845.1(6) Å^3
Z	4	4
Formula weight	847.3	734.6
Density (calc.)	3.121 Mg m^{-3}	2.645 Mg m^{-3}
Absorption coefficient	11.285 mm^{-1}	7.114 mm^{-1}
F(000)	1576	1376

TABLE 4. X-ray data collection, solution and refinement

	$Fe_4Se_2(CO)_{11}$	$Fe_3Se_2Ru(CO)_{11}$
Data collection		
Diffractometer used	Siemens P4	Siemens P4
Radiation	Mo-Kα (λ = 0.71073 Å)	Mo-Kα (λ = 0.71073 Å)
Temperature	299 K	299 K
Monochromator	Highly oriented graphite crystal	Highly oriented graphite crystal
2θ range	2.0 to 50.0°	2.0 to 50.0°
Scan type	ω–2θ	ω–2θ
Scan speed	Variable; 8.00–60.00° min^{-1} in ω	Variable; 10.00–60.00° min^{-1} in ω
Scan range (ω)	0.90°	1.20°
Background measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 25% of total scan time	Stationary crystal and stationary counter at beginning and end of scan, each for 25% of total scan time
Standard reflections	3 measured every 250 reflections	3 measured every 250 reflections
Index ranges	$-1 \le h \le 7,\ -1 \le k \le 18,\ -1 \le l \le 20$	$-1 \le h \le 7,\ -1 \le k \le 18,\ -1 \le l \le 20$
Reflections collected	2190	2213
Independent reflections	1584 (R_{int} = 2.04%)	1632 (R_{int} = 1.74%)
Observed reflections	1171 ($F > 4.0\sigma(F)$)	1277 ($F > 4.0\sigma(F)$)
Absorption correction	Semiempirical	Semiempirical
Min./max. transmission	0.4309/0.9365	0.3884/0.8993
Solution and refinement		
System used	Siemens SHELXTL PLUS (PC Version)	Siemens SHELXTL PLUS (PC Version)
Solution	Direct methods	Direct methods
Refinement method	Full-matrix least-squares	Full-matrix least-squares
Quantity minimized	$\Sigma w(F_o - F_c)^2$	$\Sigma w(F_o - F_c)^2$
Absolute structure	N/A	N/A
Extinction correction	χ = 0.00112(6), where $F^* = F[1 + 0.002\chi F^2/\sin(2\theta)]^{-1/4}$	χ = 0.00009(2), where $F^* = F[1 + 0.002\chi F^2/\sin(2\theta)]^{-1/4}$
Weighting scheme	$w^{-1} = \sigma^2(F) + 0.0005F^2$	$w^{-1} = \sigma^2(F) + 0.0010F^2$
Number of parameters, refined	129	130
Final R indices (obs. data)	R = 3.06%, wR = 3.52%	R = 3.12%, wR = 4.21%
R indices (all data)	R = 4.66%, wR = 3.97%	R = 4.15%, wR = 5.97%
Goodness-of-fit	1.00	1.00
Largest and mean Δ/σ	0.001, 0.000	0.001, 0.000
Data-to-parameter ratio	9.1 : 1	9.8 : 1
Largest difference peak	0.53 e $Å^{-3}$	0.72 e $Å^{-3}$
Largest difference hole	−0.44 e $Å^{-3}$	−0.67 e $Å^{-3}$

TABLE 5. Atomic coordinates and equivalent isotropic displacement coefficients ($Å^2 \times 10^3$) for $Fe_4Se_2(CO)_{11}$

Atom	x	y	z	U_{eq}
Se	271(1)	7152(1)	2627(1)	29(1)
Fe(1)	1814(1)	8312(1)	1932(1)	27(1)
Fe(2)	1835(1)	8262(1)	3424(1)	31(1)
C(1)	−212(10)	8302(4)	1253(3)	48(2)
O(1)	−1459(8)	8306(3)	820(3)	86(2)
C(2)	3382(9)	8830(4)	1231(3)	39(2)
O(2)	4322(7)	9174(3)	791(3)	67(2)
C(3)	1084(9)	9287(4)	2420(3)	40(2)
O(3)	599(8)	9982(3)	2531(2)	65(2)
C(4)	−505(10)	8561(4)	3854(3)	47(2)
O(4)	−1988(7)	8735(4)	4127(3)	78(2)
C(5)	3074(9)	9132(4)	3889(3)	42(2)
O(5)	3842(7)	9682(3)	4207(3)	65(2)
C(6)	2500	7500	4277(5)	45(3)
O(6)	2500	7500	4935(3)	80(3)

TABLE 6. Atomic coordinates and equivalent isotropic displacement coefficient ($Å^2 \times 10^3$) for $Fe_3Se_2Ru(CO)_{11}$

Atom	x	y	z	U_{eq}
Se	235(1)	7137(1)	2629(1)	33(1)
Fe	1780(1)	8306(1)	1931(1)	25(1)
Ru′ + Fe′	1752(1)	8302(1)	3449(1)	30(1)
C(1)	−265(12)	8289(4)	1267(4)	55(3)
O(1)	−1514(10)	8305(4)	837(4)	101(3)
C(2)	3337(10)	8845(4)	1260(4)	44(2)
O(2)	4240(9)	9189(3)	816(3)	75(2)
C(3)	1002(10)	9265(4)	2426(3)	41(2)
O(3)	469(11)	9951(4)	2501(3)	73(2)
C(4)	−669(12)	8570(4)	3898(4)	53(2)
O(4)	−2143(9)	8731(4)	4163(4)	88(3)
C(5)	3022(10)	9193(4)	3930(4)	48(2)
O(5)	3749(9)	9735(3)	4253(3)	76(2)
C(6)	2500	7500	4300(6)	53(3)
O(6)	2500	7500	4962(4)	95(4)

complete crystal and experimental parameters, see Tables 3 and 4. Raw data were corrected for Lorenz-polarization and absorption by the empirical method using ψ-scans bringing R_{int} down from 18% to 3%. The structures were solved by direct methods and refined, correcting for extinction. The two compounds are isomorphous. $Fe_3RuSe_2(CO)_{11}$ is found to be statistically disordered around its two-fold axis resulting in a mixed occupancy of one site by Fe and Ru (refined to 48.1(6) and 51.9(6)%). In order to check for possible order, refinement was done in *Pna*2(1), which, however, resulted in inconsistent bond lengths and high esd's and moreover did not show any preference of one of the two sites for Ru or Fe whatsoever (equal number of difference electrons, equal temperature factors and no change in *R*).

The lists of observed and calculated structure factors and lists of anisotropic thermal parameters are available from the authors.

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