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# Redetermination of hexacarbonyl- $(\eta^5\text{-cyclopentadienyl})\text{bis}(\mu_3\text{-selenido})\text{-diiron(II)cobalt(II)}$

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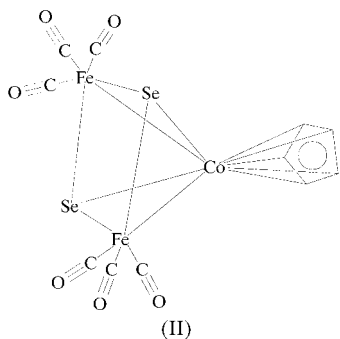
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The structure of the title compound, hexacarbonyl- $1\kappa^3\text{C}_2,2\kappa^3\text{C}-[3(\eta^5\text{-cyclopentadienyl})\text{bis}(\mu_3\text{-selenido})\text{diiron(II)cobalt(II)}, [\text{CoFe}_2(\mu_3\text{-Se})_2(\text{C}_5\text{H}_5)(\text{CO})_6]$ , was redetermined at room temperature and the correct  $C2/c$  space group was assumed instead of the previously reported  $P\bar{1}$  space group [Mathur *et al.* (1995). *Organometallics*, **14**, 2115–2118]. Analysis of the literature data showed that the previously reported triclinic parameters correspond to a primitive subcell of the actual monoclinic C-centred cell with cell dimensions close to those found by us. The title compound appeared to be isostructural with the sulfur–selenium analogue.

## Comment

The crystal structures of two compounds with similar composition, *i.e.*  $(\mu_3\text{-S})(\mu_3\text{-Se})\text{Fe}_2\text{CoCp}(\text{CO})_6$ , (I), and  $(\mu_3\text{-Se})_2\text{Fe}_2\text{CoCp}(\text{CO})_6$  (II), have been published (Mathur *et al.*, 1995). Usually similar compounds are isostructural but in this case, the reported space groups were different, with  $C2/c$  and  $Z = 8$  for the former, and  $P\bar{1}$  and  $Z = 4$  for the latter. We had a good crystal of compound (II) and redetermined the structure.



We found that the correct space group is  $C2/c$  and that (II) is actually isostructural with the sulfur–selenium analogue (I). Further analysis of the literature data revealed that the unit

cell found in 1995 for (II) corresponds to a primitive triclinic subcell of the actual monoclinic C-centred cell with parameters close to those found by us [ $a = 35.06$  (1),  $b = 6.638$  (1),  $c = 14.675$  (4) Å,  $\alpha = 90.12$  (2),  $\beta = 111.75$  (3),  $\gamma = 89.92$  (2)°, transformation matrix  $[\bar{1}0\bar{2}; 100; 0\bar{1}0]$ ]. The atomic coordinates of the triclinic determination transformed by the corresponding  $M^{-1T}$  matrix, applying the origin shift of  $(\frac{3}{4}, \frac{1}{4}, \frac{1}{2})$ , gives practically the same atomic coordinates as were found in our investigation. Two independent molecules in the triclinic cell are actually related by twofold axes.

The s.u.'s in our investigation are better than in the previous determination probably due to the avoiding the correlation effects.

The situation when the monoclinic structure is solved in triclinic subcell is not unusual. A good review was published on the subject by Marsh (1995).

## Experimental

The title compound was obtained from the reaction of  $(\text{Et}_4\text{N})_2[\text{Fe}_3(\mu_3\text{-Se})(\text{CO})_9]$  with  $\text{CpCo}(\text{CO})\text{I}_2$  in tetrahydrofuran. Single crystals were obtained from a saturated hexane solution of the product at 263 K.

### Crystal data

$[\text{CoFe}_2\text{Se}_2(\text{C}_5\text{H}_5)(\text{CO})_6]$   
 $M_r = 561.70$   
 Monoclinic,  $C2/c$   
 $a = 34.992$  (4) Å  
 $b = 6.6249$  (6) Å  
 $c = 14.651$  (2) Å  
 $\beta = 111.751$  (9)°  
 $V = 3154.6$  (6) Å<sup>3</sup>  
 $Z = 8$

$D_x = 2.365$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 24 reflections  
 $\theta = 9\text{--}14^\circ$   
 $\mu = 7.490$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Plate, black  
 $0.28 \times 0.24 \times 0.10$  mm

### Data collection

Enraf–Nonius CAD-4 diffractometer  
 $\omega$  scans  
 Absorption correction:  $\psi$  scan (modified *CADDPAT*; unpublished)  
 $T_{\min} = 0.141$ ,  $T_{\max} = 0.473$   
 2811 measured reflections  
 2765 independent reflections

1297 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.0543$   
 $\theta_{\text{max}} = 24.97^\circ$   
 $h = 0 \rightarrow 41$   
 $k = 0 \rightarrow 7$   
 $l = -17 \rightarrow 16$   
 3 standard reflections  
 frequency: 60 min  
 intensity decay: none

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.028$   
 $wR(F^2) = 0.062$   
 $S = 0.693$   
 2765 reflections  
 200 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0086P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.45$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.39$  e Å<sup>-3</sup>  
 Extinction correction: *SHELXL97*  
 Extinction coefficient: 0.00086 (2)

**Table 1**

Selected geometric parameters (Å, °).

Se1–Fe1	2.3569 (14)	Fe2–Co1	2.5467 (16)
Se1–Fe2	2.3583 (15)	Fe2–C21	1.800 (10)
Se1–Co1	2.2853 (14)	Fe2–C22	1.751 (9)
Se2–Fe1	2.3615 (15)	Fe2–C23	1.773 (9)
Se2–Fe2	2.3605 (14)	Co1–C1CP	2.035 (9)
Se2–Co1	2.2870 (13)	Co1–C2CP	2.064 (8)
Fe1–Co1	2.5610 (16)	Co1–C3CP	2.057 (8)
Fe1–C11	1.810 (10)	Co1–C4CP	2.035 (8)
Fe1–C12	1.792 (9)	Co1–C5CP	2.014 (9)
Fe1–C13	1.750 (10)		

Fe1—Se1—Fe2	97.30 (5)	C11—Fe1—Co1	106.2 (3)
Co1—Se1—Fe1	66.94 (5)	C12—Fe1—Se1	156.6 (3)
Co1—Se1—Fe2	66.50 (5)	C12—Fe1—Se2	89.3 (3)
Fe2—Se2—Fe1	97.11 (5)	C12—Fe1—Co1	102.3 (3)
Co1—Se2—Fe1	66.84 (5)	C13—Fe1—Se1	104.1 (3)
Co1—Se2—Fe2	66.43 (5)	C13—Fe1—Se2	100.1 (3)
Se1—Fe1—Se2	82.25 (5)	C13—Fe1—Co1	146.8 (3)
Se1—Fe1—Co1	55.19 (4)	Se1—Fe2—Se2	82.24 (5)
Se2—Fe1—Co1	55.19 (4)	Se1—Fe2—Co1	55.38 (4)
C11—Fe1—Se1	88.6 (3)	Se2—Fe2—Co1	55.40 (4)
C11—Fe1—Se2	161.2 (3)		

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Data collection: *CD4CA0* in *CAD-4 User's Manual* (Enraf-Nonius, 1985); cell refinement: *CD4CA0*; data reduction: *XCAD* (local program); program(s) used to solve structure: *SHELXS97*

(Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997).

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