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# Synthesis and characterisation of CpCoFe$_2$(CO)$_6$( $\mu_3$-S)( $\mu_3$-Te). Crystal structure of CpCoFe$_2$(CO)$_6$( $\mu_3$-Se)( $\mu_3$-Te)

Pradeep Mathur [a,*], P. Sekar [a], C.V.V. Satyanarayana [b], Mary F. Mahon [c]

[a] Department of Chemistry, Indian Institute of Technology, Powai, Bombay 400 076, India
[b] Regional Sophisticated Instrumentation Centre, Indian Institute of Technology, Powai, Bombay 400 076, India
[c] School of Chemistry, University of Bath, Claverton Down, Bath BA2 7AY, UK

## Abstract

The new mixed-metal, mixed-chalcogenide cluster CpCoFe$_2$(CO)$_6$( $\mu_3$-S)( $\mu_3$-Te) (1) has been prepared from the room temperature reaction of Fe$_2$(CO)$_6$( $\mu$-STe) with CpCo(CO)$_2$. Compound 1 has been characterised by IR and $^1$H, $^{13}$C and $^{125}$Te NMR spectroscopy and its composition has been confirmed by elemental analysis. The previously reported compound CpCoFe$_2$(CO)$_6$( $\mu_3$-Se)( $\mu_3$-Te) (3) has been further characterised by $^{77}$Se and $^{125}$Te NMR spectroscopy and its solid state structure elucidated by X-ray crystallography. Compound 3 crystallised in the monoclinic space group $C2/c$ with $a = 35.125(2)$, $b = 6.643(1)$, $c = 14.910(1)$ Å, $\beta = 111.79(1)°$, $V = 3230.6$ Å$^3$, $Z = 8$, $D_c = 2.51$ g cm$^{-3}$ and $R = 0.0297$ ($R_w = 0.0283$). Its structure consists of an Fe$_2$CoSeTe distorted square pyramidal core in which the Co atom occupies the apical site of the square pyramid, and the Fe and chalcogen atoms occupy alternate corners of the distorted square with three carbonyl groups terminally bonded to each Fe atom and an $\eta^5$-Cp ligand bonded to the Co atom.

*Keywords:* Iron; Cobalt; Selenium; Tellurium; Carbonyl; Cluster; X-ray diffraction

## 1. Introduction

Transition metal clusters which contain S, Se or Te ligands are of considerable current interest because they exhibit unusual structural and reactivity patterns [1–4]. Chalcogen atoms have been recognised as bridging and stabilising ligands in numerous metal carbonyl compounds [5]. The dinuclear compounds Fe$_2$(CO)$_6$( $\mu$-EE'), where E = S, Se; E' = Te, have been used as convenient starting materials for the synthesis of several mixed-metal, mixed-chalcogenide clusters [6–8]. A characteristic feature of these compounds is the presence of a reactive E–E' bond across which addition of organic and inorganic moieties occurs readily [7–11]. The oxidative addition across the E–E linkage of Fe$_2$(CO)$_6$( $\mu$-E$_2$), where E = S, Se or Te, with various low-valent transition-metal species has been demonstrated in detail [12–21]. We have previously reported the synthesis of the mixed-chalcogenide compounds Fe$_2$(CO)$_6$( $\mu$-SeTe) and Fe$_2$(CO)$_6$( $\mu$-STe) and their reactions with coordi-

natively unsaturated metal carbonyl species to yield new mixed-metal, mixed-chalcogenide clusters [6–8,18]. Here we report the reaction of Fe$_2$(CO)$_6$( $\mu$-STe) with CpCo(CO)$_2$ and the characterisation of the product isolated. We also report the $^{77}$Se and $^{125}$Te NMR data and single crystal X-ray diffraction analysis of CpCoFe$_2$(CO)$_6$( $\mu_3$-Se)( $\mu_3$-Te), whose preparation has been described earlier [6].

## 2. Results and discussion
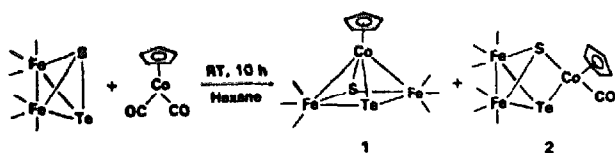
The mixed-chalcogen compounds Fe$_2$(CO)$_6$( $\mu$-EE') (E = S, Se; E' = Te) were obtained in pure form using the general method reported earlier [8,18].

### 2.1. Reaction of Fe$_2$(CO)$_6$( $\mu$-STe) with CpCo(CO)$_2$

When a hexane solution containing Fe$_2$(CO)$_6$( $\mu$-STe) and CpCo(CO)$_2$ was stirred at room temperature for 10 h, two products were formed and identified as green coloured CpCoFe$_2$(CO)$_6$( $\mu_3$-S)( $\mu_3$-Te) (1) and dark

* Corresponding author.

Scheme 1. Formation of 1 and 2.

Table 1
Multinuclear NMR data ($\delta$ ppm) for 1, 3 and 4

| Com-pound | $^1$H NMR ($\delta$ w.r.t. TMS) | $^{13}$C NMR ($\delta$ w.r.t. TMS) | $^{77}$Se NMR ($\delta$ w.r.t. Me$_2$Se) | $^{125}$Te NMR ($\delta$ w.r.t. Me$_2$Te) |
|---|---|---|---|---|
| 1 | 5.83(s) | 82.17(s) 208.48(s) | — | 933.85(s) |
| 3 | 5.83(s) | 81.16(s) 209.13(s) | 857.18(s) | 1018.49(s) 947.48(s) |
| 4 | 5.84(s) | 89.01(s) 208.93(s) | −473.57(s) | −958.17(s) |

red coloured CpCoFe$_2$(CO)$_7$( $\mu_3$-S)( $\mu_3$-Te) (2) (Scheme 1). The infrared spectrum of 1 indicates the presence of only terminally bonded carbonyl groups and the pattern is similar to that of previously reported compounds CpCoFe$_2$(CO)$_6$( $\mu_3$-E)( $\mu_3$-E') (E, E' = S, Se; E = Se, Te and E' = Te) [6,13,15,22]. $^1$H and $^{13}$C NMR spectra confirm the presence of Cp ligand in 1. For the series of compounds CpCoFe$_2$(CO)$_6$( $\mu_3$-Te)( $\mu_3$-E') (E' = S, Se, Te), a downfield shift of the $^{125}$Te NMR signal along the series E = S, Se, Te is observed. A similar trend is seen in the $^{125}$Te NMR spectra of the series of compounds Fe$_3$(CO)$_9$( $\mu_3$-S)( $\mu_3$-Te) ($\delta$ 828 ppm), Fe$_3$(CO)$_9$( $\mu_3$-Se)( $\mu_3$-Te) ($\delta$ 982 ppm) and Fe$_3$(CO)$_9$-( $\mu_3$-Te)$_2$ ($\delta$ 1123 ppm), and also the mixed-metal series Fe$_2$W(CO)$_{10}$( $\mu_3$-S)( $\mu_3$-Te) ($\delta$ 244 ppm), Fe$_2$W(CO)$_{10}$( $\mu_3$-Se)( $\mu_3$-Te) ($\delta$ 356 ppm) and Fe$_2$W(CO)$_{10}$( $\mu_3$-Te)$_2$ ($\delta$ 467 ppm). Comparison with the $^{125}$Te NMR spectrum of Fe$_3$(CO)$_9$( $\mu_3$-S)( $\mu_3$-Te) shows that a formal replacement of an Fe(CO)$_3$ group by a CpCo group shifts the signal downfield by 106 ppm.

Characterisation of 2 is based on comparison of its infrared spectrum in the carbonyl region (in hexane solvent, $\nu$(CO): 2058(m), 2042(m), 2034(vs), 1992(s), 1980(vs), 1951(w), 1942(w) cm$^{-1}$) with the previously reported compounds CpCoFe$_2$(CO)$_7$( $\mu_3$-E)( $\mu_3$-E'), where E = Se, Te; E' = Te [6,13]. In solution, 2 was found to convert readily to the nido-hexacarbonyl compound 1. The related compound CpCoFe$_2$(CO)$_7$( $\mu_3$-Se)( $\mu_3$-Te) is also unstable in solution, and on standing converts to CpCoFe$_2$(CO)$_6$( $\mu_3$-Se)( $\mu_3$-Te).

2.2. $^{77}$Se and $^{125}$Te NMR study of CpCoFe$_2$(CO)$_6$( $\mu_3$-Se)( $\mu_3$-Te) (3) and CpCoFe$_2$(CO)$_7$( $\mu_3$-Se) ( $\mu_3$-Te) (4)

The $^{77}$Se NMR spectrum of 3 showed a singlet at $\delta$ 857.18 ppm. The $^{125}$Te NMR spectrum of 3 showed a signal at $\delta$ 1018.49 and a much weaker signal at 947.48 ppm downfield of Me$_2$Te. It indicates the presence of two types of Te atom due to two isomeric forms. For the related compounds CpCoFe$_2$(CO)$_6$( $\mu_3$-Te)$_2$ and CpRhFe$_2$(CO)$_6$( $\mu_3$-Te)$_2$, Rauchfuss and coworker [13] have reported two isomeric forms for each on the basis of $^{125}$Te NMR spectra. Comparison of the spectra of 3 with the $^{77}$Se and $^{125}$Te NMR spectra of Fe$_3$(CO)$_9$( $\mu_3$-Se)( $\mu_3$-Te) shows that the replacement of an Fe(CO)$_3$ group by a CpCo group shifts the $^{77}$Se signal upfield by 10 ppm and the $^{125}$Te signal downfield by 36 ppm [23].

The $^{77}$Se NMR spectrum of 4 showed a signal at $\delta$ −473.57 ppm. The $^{125}$Te NMR spectrum of 4 also shows only one signal at $\delta$ −958.17 ppm. Compound 4 slowly decomposes in solution: after 24 h only the Se$_2$ and Te$_2$ compounds were found by $^{77}$Se and $^{125}$Te NMR spectroscopy. Table 1 lists the multinuclear NMR data for compounds 1, 3 and 4.

2.3. Molecular structure of 3

An ORTEP diagram of the molecular structure of 3 is shown in Fig. 1. The metal core geometry of 3 can be described as an Fe$_2$CoSeTe distorted square pyramid in which the Co atom occupies the apical site. Overall, the core structure is similar to that of the other members of the class of compounds CpCoFe$_2$(CO)$_6$( $\mu_3$-E)( $\mu_3$-E') (E, E' = S, S; Se, Se; S, Se) [22,24]. The average Co–Fe bond distance in 3 (2.594 Å) is somewhat longer than the average Co–Fe bond distances in ($\eta^5$-C$_5$H$_5$)CoFe$_2$(CO)$_6$( $\mu_3$-S)$_2$ (2.539 Å) [24], CpCo-Fe$_2$(CO)$_6$( $\mu_3$-S)( $\mu_3$-Se) (2.529 Å), CpCoFe$_2$(CO)$_6$-( $\mu_3$-Se)$_2$ (2.558 Å) [22], FeCo$_2$(CO)$_9$( $\mu_3$-Se) (2.581 Å)
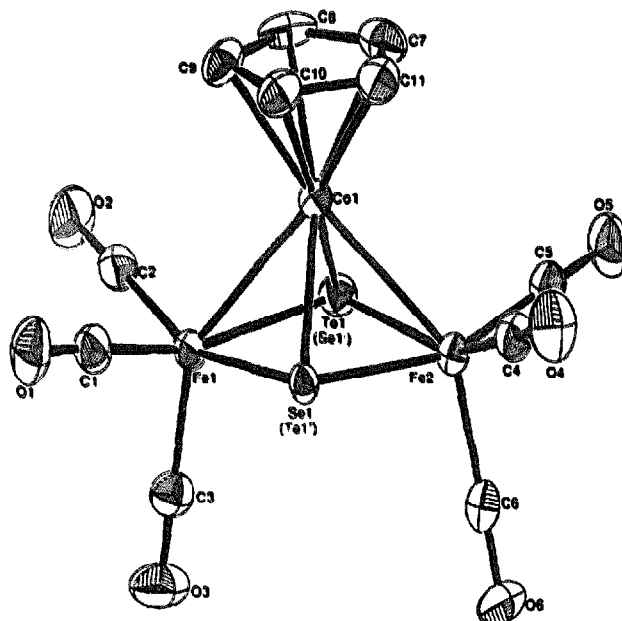


Fig. 1. Molecular structure of 3.

[25] and $Fe_2Co_2(CO)_{11}(\mu_4\text{-S})_2$ (2.58 Å) [12], but is shorter than the average Co–Fe bond distances reported for $Fe_2Co_2(CO)_{11}(\mu_4\text{-PPh})_2$ (2.62 Å) [12] and $H(Cp)MoCoFe(CO)_8(\mu_3\text{-Ge}^tBu)$ (2.684 Å) [26]. However, the average Fe–Se bond distance in **3** (2.389 Å) is longer than the average Fe–Se bond distance in $Fe_3(CO)_9(\mu_3\text{-Se})_2$ (2.35 Å) [27] and in $Fe_3(CO)_9(\mu_3\text{-S})(\mu_3\text{-Se})$ (2.351 Å) [28]. It is shorter than the average Fe–Se bond distance of 2.437 Å reported for $Fe_3(CO)_9(\mu_3\text{-Se})(\mu_3\text{-Te})$ [28]. The average Fe–Te bond distance in **3** (2.490 Å) is similar to the average Fe–Te bond distance in $Fe_3(CO)_9(\mu_3\text{-Se})(\mu_3\text{-Te})$ (2.480 Å), but is shorter than the average Fe–Te bond distance reported for $Fe_3(CO)_9(\mu_3\text{-S})(\mu_3\text{-Te})$ (2.533 Å) [28]. The Fe–Co–Fe angle in **3** is 89.1°, similar to the corresponding angle in $CpCoFe_2(CO)_6(\mu_3\text{-Se})_2$ (87.8°), but slightly greater than the angle in $CpCoFe_2(CO)_6\text{-}(\mu_3\text{-S})(\mu_3\text{-Se})$ (86.4°) [22].

Assuming that the Se and Te atoms act as four-electron donors, compound **3** is a 50-electron cluster, and

Table 2
Crystallographic data for **3**

| *Crystal data* | |
|---|---|
| Molecular formula | $C_{11}H_5O_6Fe_2SeTeCo$ |
| Molecular weight | 610.3 |
| Crystal colour and description | dark red plates |
| Crystal dimensions (mm³) | 0.2×0.2×0.24 |
| Crystal system | monoclinic |
| Space group | $C2/c$ |
| Cell parameters | |
| $a$ (Å) | 35.125(2) |
| $b$ (Å) | 6.643(1) |
| $c$ (Å) | 14.910(1) |
| $\beta$ (°) | 111.79(1) |
| $V$ (Å³) | 3230.2 |
| $Z$ | 8 |
| $D_{calc}$ (g cm⁻³) | 2.51 |
| $F(000)$ | 2272 |
| *Data collection* | |
| Diffractometer | CAD-4 automatic four-circle diffractometer |
| Radiation ($\lambda$ (Å)) | Mo K $\alpha$ (0.7093) |
| Temperature (K) | 298 |
| $\theta_{max}$ (°) | 24 |
| Reflections collected | 2825 |
| Observed reflections | 1850 |
| Max./min. absorption correction | 1.271, 0.832 |
| *Refinement* | |
| Refinement method | Full-matrix least-squares |
| Weighting scheme $w$ | $2.5191/[\sigma^2(F)+0.000275(F)^2]$ |
| Final $R$ | 0.0297 |
| Weighted $R$ | 0.0283 |
| $(\Delta/\sigma)$ max. | 0.004 |
| $\Delta\rho$ max. (e Å⁻³) | 0.39 |
| $\Delta\rho$ min. (e Å⁻³) | −0.39 |

Table 3
Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($\text{Å}^2 \times 10^3$) for **3**

| Atom | $x$ | $y$ | $z$ | $U$ |
|---|---|---|---|---|
| Te(1) | 3865(1) | 237(4) | 7066(2) | 54(1) |
| Te(1') | 3654(3) | 4714(16) | 7623(8) | 96(4) |
| Co(1) | 3739.4(2) | 1682(1) | 8423(1) | 37.6(5) |
| Fe(1) | 4298.2(2) | 3179(1) | 7863(1) | 42.5(6) |
| Fe(2) | 3209.4(3) | 2107(1) | 6682(1) | 41.0(5) |
| Se(1) | 3651(1) | 4713(3) | 7605(2) | 39(1) |
| Se(1') | 3871(3) | 184(14) | 7061(6) | 13(3) |
| O(1) | 4699(2) | 6399(9) | 9244(4) | 90(3) |
| O(2) | 4995(2) | 434(9) | 8707(5) | 95(3) |
| O(3) | 4491(2) | 4655(10) | 6244(5) | 101(3) |
| O(4) | 2541(2) | 3976(10) | 7117(4) | 92(3) |
| O(5) | 2787(2) | −1775(9) | 6105(5) | 90(3) |
| O(6) | 3021(2) | 3898(9) | 4770(4) | 76(2) |
| C(1) | 4543(2) | 5123(12) | 8712(6) | 58(3) |
| C(2) | 4722(2) | 1497(12) | 8386(5) | 59(3) |
| C(3) | 4413(2) | 4081(12) | 6867(5) | 60(3) |
| C(4) | 2800(2) | 3234(11) | 6944(5) | 59(3) |
| C(5) | 2950(2) | −248(12) | 6351(5) | 56(3) |
| C(6) | 3096(2) | 3174(11) | 5511(5) | 53(3) |
| C(7) | 3514(3) | −486(13) | 9062(6) | 69(4) |
| C(8) | 3938(3) | −515(13) | 9463(5) | 70(4) |
| C(9) | 4076(2) | 1381(13) | 9893(5) | 63(3) |
| C(10) | 3727(2) | 2552(13) | 9742(5) | 61(3) |
| C(11) | 3379(2) | 1409(13) | 9236(5) | 64(4) |

the formal application of the 18-electron rule would predict two metal–metal bonds as observed. According to the PSEP theory, the presence of seven skeletal electron pairs for **3** correctly predicts the *nido* octahedral structure.

## 3. Experimental details

### 3.1. General procedures

Reactions and manipulations were carried out under an inert atmosphere of argon by means of standard Schlenk techniques. The solvents were purified and distilled under a nitrogen or argon atmosphere prior to use. Infrared spectra were recorded on a Nicolet Impact 400 FT infrared spectrometer in NaCl cells of 0.1 mm path length as hexane solutions. $^1$H, $^{13}$C, $^{77}$Se and $^{125}$Te NMR spectra were obtained on a Varian VXR-300S NMR spectrometer in $CDCl_3$ solution using appropriate references at 25°C. The $^{77}$Se NMR spectra were referenced to $Me_2Se$ as an external standard ($\delta$ 0 ppm) and the spectra were obtained at an operating frequency of 57.23 MHz; 90° pulses were used with 1.0 s delay and 1.0 s acquisition time. $^{125}$Te NMR spectra were referenced to $Me_2Te$ as an external standard ($\delta$ 0 ppm) and the spectra were obtained at an operating frequency of 94.75 MHz; 90° pulses were used with 1.0 s delay and 1.0 s acquisition time. $Fe_2(CO)_6(\mu\text{-STe})$ [8] and $CpCoFe_2(CO)_6(\mu_3\text{-Se})(\mu_3\text{-Te})$ (**3**) [6] were prepared as

previously reported. $CpCo(CO)_2$ was prepared as described in the literature [29].

### 3.2. Reaction of $Fe_2(CO)_6(\mu\text{-}STe)$ with $CpCo(CO)_2$

To a hexane solution (60 ml) of $Fe_2(CO)_6(\mu\text{-}STe)$ (0.2 g, 0.46 mmol) was added $CpCo(CO)_2$ (0.4 ml) and the reaction mixture was stirred at room temperature for 10 h. The solvent was removed in vacuo, and the residue chromatographed on a silica gel column using hexane as eluant to separate a trace of unreacted $Fe_2(CO)_6(\mu\text{-}STe)$ and the unstable dark red CpCo-$Fe_2(CO)_7(\mu_3\text{-}S)(\mu_3\text{-}Te)$ (2) from the major second band of green $CpCoFe_2(CO)_6(\mu_3\text{-}S)(\mu_3\text{-}Te)$ (1); yield 0.085 g (33%). IR ($\nu$(CO), cm$^{-1}$): 2057(m), 2033(vs), 1991(s), 1980(vs). $^1$H NMR: $\delta$ 5.83 (s, $C_5H_5$). $^{13}$C NMR: $\delta$ 82.2 (s, $C_5H_5$), 208.48 (s, CO). $^{125}$Te NMR: $\delta$ 933.9 (s). M.p. 156°C(dec.). Anal. Found: C, 21.83; H, 1.17. $C_{11}H_5O_6CoFe_2STe$ Calc.: C, 21.65; H, 0.83%.

### 3.3. X-ray diffraction study of 3

Black crystals of 3 were grown from a hexane/dichloromethane solution by slow evaporation of solvent at −5°C. A crystal of approximate dimensions $0.2 \times 0.2 \times 0.24$ mm$^3$ was used for data collection. Crystal data is summarised in Table 2. The data were measured at room temperature on a CAD-4 automatic four-circle diffractometer in the range $2° \leq \theta \leq 24°$; 2825 reflections were collected of which 1850 were unique with $I \geq 2\sigma(I)$. Data were corrected for Lorentz and polarization and also for absorption [30]. (Max. and min. absorption corrections 1.271 and 0.832 respectively.)

The structure was solved by direct methods and refined using the SHELX [31,32] suite of programs. Early convergence revealed that the Te1 and Se1 atoms were disordered in the ratio 70.6:29.4 with their primed analogues. Disorder was modelled as follows. Initially the site occupancy was refined with isotropic thermal parameters for both the Te and Se atoms and their disordered counterparts. The refined occupancy parameters were then input (and fixed) in the SHELX file, and anisotropic refinement of the thermal parameters for Te1, Se1, Te1' and Se1' was subsequently performed. This disorder is not depicted in the ORTEP plot where only the position of the unprimed atoms is illustrated. The very proximate disordered moieties are indicated by bracketed labels. In the final least-squares cycles all atoms were allowed to vibrate anisotropically except for the partial occupiers Te1' and Se1'. Hydrogen atoms were included at calculated positions. Final residuals after 12 cycles of least-squares were $R = 0.0297$, $R_w = 0.0283$, for a weighting scheme of $w = 2.5191/[\sigma^2(F) + 0.000275(F)^2]$. Convergence of the structure is evidenced by the quoted largest shift/e.s.d. value of 0.004. The max. and min. residual densities were 0.39 and −0.39 e Å$^{-3}$ respectively. Final fractional atomic coordinates and isotropic thermal parameters, bond distances and angles are given in Tables 3 and 4.

Final fractional atomic coordinates and equivalent isotropic temperature factors, anisotropic thermal parameters, bond lengths and angles, intermolecular distances, intramolecular distances, and the structure factor tables are available from the authors.

#### Table 4
Selected bond lengths (Å) and angles (°) for 3

| | | | |
|---|---|---|---|
| Fe(1)–Co(1) | 2.599(4) | Fe(2)–Te(1) | 2.489(5) |
| Fe(2)–Co(1) | 2.588(4) | C(1)–Fe(1) | 1.789(10) |
| Se(1)–Co(1) | 2.314(4) | C(2)–Fe(1) | 1.792(10) |
| Te(1)–Co(1) | 2.424(5) | C(3)–Fe(1) | 1.782(10) |
| Se(1)–Fe(1) | 2.388(4) | C(4)–Fe(2) | 1.787(9) |
| Se(1)–Fe(2) | 2.390(4) | C(5)–Fe(2) | 1.786(10) |
| Fe(1)–Te(1) | 2.491(5) | C(6)–Fe(2) | 1.787(10) |
| | | | |
| Fe(2)–Co(1)–Fe(1) | 89.1(2) | C(1)–Fe(1)–Se(1) | 89.2(3) |
| Fe(2)–Se(1)–Fe(1) | 99.2(2) | C(1)–Fe(1)–Te(1) | 162.2(2) |
| Fe(2)–Te(1)–Fe(1) | 93.8(2) | C(2)–Fe(1)–Se(1) | 157.5(2) |
| Fe(1)–Se(1)–Te(1) | 49.8(1) | C(2)–Fe(1)–Te(1) | 88.9(3) |
| Fe(2)–Se(1)–Te(1) | 49.7(1) | C(3)–Fe(1)–Se(1) | 102.7(3) |
| Se(1)–Co(1)–Fe(1) | 57.8(2) | C(3)–Fe(1)–Te(1) | 98.8(4) |
| Se(1)–Co(1)–Fe(2) | 58.0(2) | C(4)–Fe(2)–Se(1) | 88.5(3) |
| Se(1)–Co(1)–Te(1) | 86.3(2) | C(4)–Fe(2)–Te(1) | 155.4(2) |
| Fe(1)–Co(1)–Te(1) | 59.4(2) | C(5)–Fe(2)–Se(1) | 161.7(2) |
| Fe(2)–Co(1)–Te(1) | 59.5(2) | C(5)–Fe(2)–Te(1) | 87.7(3) |
| Se(1)–Fe(1)–Te(1) | 83.2(2) | C(6)–Fe(2)–Se(1) | 97.9(3) |
| Se(1)–Fe(2)–Te(1) | 83.2(2) | C(6)–Fe(2)–Te(1) | 105.9(3) |

Numbers in parentheses are estimated standard deviations in the least significant digits.

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