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# Synthesis and characterisation of *cis* and *trans* isomers of the mixed chalcogen clusters $Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Se)$ and $Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Te)$

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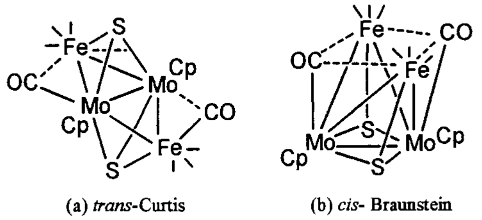
## Abstract

The reaction of $Fe_2(CO)_6(\mu\text{-}SSe)$ and $Cp_2Mo_2(CO)_4$ in benzene at room temperature for 2 h, yielded the new isomeric mixed-metal, mixed-dichalcogenide clusters *trans*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Se)$ (**1**) and *cis*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Se)$ (**4**). The new mixed-metal clusters **1** and **4** could also be obtained when $Fe_3(CO)_9(\mu_3\text{-}S)(\mu_3\text{-}Se)$ and $Cp_2Mo_2(CO)_6$ were heated at 65°C in benzene for 36 h. Similarly, the room-temperature reaction of $Fe_2(CO)_6(\mu\text{-}STe)$ with $Cp_2Mo_2(CO)_4$ in benzene for 2 h and the thermolytic reaction of $Fe_3(CO)_9(\mu_3\text{-}S)(\mu_3\text{-}Te)$ with $Cp_2Mo_2(CO)_6$ in benzene for 36 h afforded the new isomeric clusters *trans*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Te)$ (**9**) and *cis*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Te)$ (**11**). The new clusters have been characterised by IR and by $^{1}$H-, $^{77}$Se-, $^{125}$Te-NMR spectroscopy. Clusters **1** and **4** have also been structurally characterised by single-crystal X-ray diffraction methods. © 1999 Elsevier Science S.A. All rights reserved.

*Keywords:* Clusters; Iron; Molybdenum

## 1. Introduction

The last decade has witnessed a tremendous development in the chemistry of transition metal clusters stabilised by single-atom ligands derived from main group elements and a myriad of these types of compounds have been reported [1]. The main group elements act as bridging and stabilising ligands, thereby serving to maintain the cluster integrity and also to stabilise the overall cluster framework [2]. Development of facile synthetic methods for preparation of heterochalcogen atom stabilised mixed-transition metal cluster compounds of high nuclearity is a particularly intriguing area, as it gives an opportunity to study the influence of the different chalcogen atoms on the structure, reactivity and bonding pattern of these complexes.



(a) *trans*-Curtis (b) *cis*- Braunstein

Fig. 1.

The reactive $Mo\equiv Mo$ triple bond of $Cp_2Mo_2(CO)_4$ has been widely used for rational synthesis of metal cluster complexes [3]. Following the precedent of addition of organic species across the triple bond, both Braunstein and Curtis independently investigated the reaction of $Cp_2Mo_2(CO)_4$ with $Fe_2S_2(CO)_6$ [4,5]. Consequently, a pair of isomeric *trans*-'Curtis' and *cis*-'Braunstein' iron–molybdenum–sulphur clusters, $Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)_2$ was generated with different $Fe_2Mo_2S_2$ frameworks (Fig. 1).

In view of our longstanding interest in the chemistry of chalcogen atom stabilised transition metal clusters, we sought to synthesise the mixed chalcogen analogues of these compounds to study the effect of the heterochalcogens on the structure and bonding pattern of

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these classes of compounds. We report here on the synthesis and characterisation of the new isomeric mixed chalcogen clusters *cis*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Se)$, *trans*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Se)$, *cis*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Te)$ and *trans*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Te)$.

## 2. Experimental

### 2.1. General procedures

All reactions and other manipulations were carried out using standard Schlenk techniques under an inert atmosphere of argon. All solvents were deoxygenated immediately prior to use. Infrared spectra were recorded on a Nicolet Impact 400 FT-IR spectrophotometer, as dichloromethane solutions in 0.1 mm path length cells or as KBr pellets. Elemental analyses was performed using a Carlo–Erba automatic analyser. $^1H$-, $^{77}Se$- and $^{125}Te$-NMR spectra were recorded on a Varian VXR-300S spectrometer in $CDCl_3$. The operating frequency for $^{77}Se$-NMR was 57.23 MHz with a pulse width of 15 μs and a delay of 1.0 s and the operating frequency for $^{125}Te$ was 94.70 MHz with pulse width of 9.5 μs and a delay of 1 s. $^{77}Se$-NMR spectra were referenced to $Me_2Se$ ($\delta = 0$ ppm) and $^{125}Te$-NMR spectra were referenced to $Me_2Te$ ($\delta = 0$ ppm).

The starting materials $Cp_2Mo_2(CO)_6$ [6], $Cp_2Mo_2(CO)_4$ [7], $Fe_3(CO)_9(\mu_3\text{-}S)(\mu_3\text{-}Se)$, $Fe_2(CO)_6(\mu\text{-}SSe)$ [8], $Fe_3(CO)_9(\mu_3\text{-}S)(\mu_3\text{-}Te)$ and $Fe_2(CO)_6(\mu\text{-}STe)$ [9] were prepared as reported in the literature.

### 2.2. Reaction of $Fe_2(CO)_6(\mu\text{-}SSe)$ with $Cp_2Mo_2(CO)_4$

A benzene solution (50 ml) of $Fe_2(CO)_6(\mu\text{-}SSe)$ (0.5 g, 1.27 mmol) and $Cp_2Mo_2(CO)_4$ (0.5g, 1.15 mmol) was stirred constantly at room temperature (r.t.) for 2 h. The reaction was monitored by TLC. The solution was filtered through Celite to remove the insoluble material. After removal of the solvent from the filtrate, the residue was dissolved in dichloromethane and chromatographic work-up was performed. Using 1:1 hexane–dichloromethane solvent mixtures as eluent, three fractions were separated. The first two fractions were subjected to further chromatographic work-up. On eluting the first fraction with 7:3 dichloromethane–hexane solvent mixtures, the pure green compound *trans*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Se)$ (**1**; 70 mg, 8%) was isolated. The second fraction was eluted with 1:1 hexane–dichloromethane solvent mixture whereby three compounds were isolated: dark red $Cp_2Mo_2Fe(CO)_7(\mu_3\text{-}Se)$ (**2**; 45 mg, 6%); dark red $Cp_2Mo_2Fe(CO)_7(\mu_3\text{-}S)$ (**3**; 34.8 mg, 5%) and the reddish–brown *cis*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Se)$ (**4**; 132.7 mg, 15%). From the third fraction pure $Cp_2Mo_2Fe_2(CO)_7(\mu_3\text{-}S)(\mu_3\text{-}Se)$ (**5**; 230 mg, 27%) was obtained. **1**: Anal. Found: C, 27.5; H, 1.38. $C_{18}H_{10}Fe_2Mo_2O_8SSe$; Anal. Calc.: C, 28.1; H, 1.30%. **4**: Anal. Found: C, 27.4; H, 1.36. $C_{18}H_{10}Fe_2Mo_2O_8SSe$; Anal. Calc.: C, 28.1; H, 1.30%.

### 2.3. Reaction of $Fe_3(CO)_9(\mu_3\text{-}S)(\mu_3\text{-}Se)$ with $Cp_2Mo_2(CO)_6$

To a benzene solution (250 ml) of $Fe_3(CO)_9(\mu_3\text{-}S)(\mu_3Se)$ (2 g, 3.76 mmol), $Cp_2Mo_2(CO)_6$ (0.92 g, 1.88 mmol) was added and the solution heated to 65°C with constant stirring for 36 h. The reaction was closely monitored by TLC. The cooled solution was filtered through Celite to remove insoluble material. After removal of the solvent from the filtrate, the residue was dissolved in dichloromethane and subjected to chromatographic work-up. Using 1:1 hexane–dichloromethane mixture as eluent, three fractions were separated. The first two fractions were subjected to further chromatographic work-up using silica gel TLC plates. Chromatography of the first fraction, using 7:3 hexane–dichloromethane mixture as eluent, resulted in the following four compounds, in the order of elution: yellowish–brown $Cp_2Mo_2Fe_2(CO)_6(\mu_4\text{-}Se)(\mu_3\text{-}Se)(\mu_3\text{-}S)$ (**6**; 149 mg, 10%); deep yellowish–brown $Cp_2Mo_2Fe_2(CO)_6(\mu_4\text{-}Se)(\mu_3\text{-}S)_2$ (**7**; trace); brown $Cp_2Mo_2Fe_2(CO)_6(\mu_4\text{-}Se)(\mu_3\text{-}Se)_2$ (**8**; trace) and green *trans*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Se)$ (**1**; 333 mg, 23%). Chromatography of the second fraction with 1:1 hexane–dichloromethane mixture as eluent afforded the following three compounds, in order of elution: dark red $Cp_2Mo_2Fe(CO)_7(\mu_3\text{-}Se)$ (**2**; trace); dark red $Cp_2Mo_2Fe(CO)_7(\mu_3\text{-}S)$ (**3**; trace) and the reddish–brown compound *cis*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Se)$ (**4**; 506 mg, 35%). From the third fraction the pure $Cp_2Mo_2Fe_2(CO)_7(\mu_3\text{-}S)(\mu_3\text{-}Se)$ (**5**; 209 mg, 15%) was obtained.

### 2.4. Reaction of $Fe_2(CO)_6(\mu\text{-}STe)$ with $Cp_2Mo_2(CO)_4$

A benzene solution (50 mL) of $Fe_2(CO)_6(\mu\text{-}STe)$ (1.15 mmol, 0.5 g) containing $Cp_2Mo_2(CO)_4$ (1.15 mmol, 0.5 g) was stirred constantly at r.t. for 2 h. The reaction was closely monitored by TLC. The solution was filtered through Celite to remove the insoluble material. After removal of the solvent from the filtrate the residue was dissolved in dichloromethane and chromatographic work-up was performed. On elution with 1:1 hexane–dichloromethane solvent mixtures, three fractions were isolated. The first two fractions were subjected to further chromatographic work-up using silica gel TLC plates. Using 7:3 hexane–dichloromethane solvent mixture as eluent, green *trans*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Te)$ (**9**; 94 mg, 10%) was

Table 1
Crystal data and details of measurements for compounds **1** and **4** [a]

| Compound | **1** | **4** |
|---|---|---|
| Formula | $C_{18}H_{10}Fe_2Mo_2O_8SSe$ | $C_{18}H_{10}Fe_2Mo_2O_8SSe$ |
| Formula weight | 768.86 | 768.86 |
| Temperature (K) | 293(2) | 293(2) |
| Wavelength (Å) | 0.70930 | 0.70930 |
| Crystal system | Orthorhombic | Triclinic |
| Space group | *Pmnb* (No. 53) | $P\bar{1}$ (No. 2) |
| Crystal dimensions (mm$^3$) | $0.2 \times 0.2 \times 0.25$ | $0.2 \times 0.2 \times 0.15$ |
| $a$ (Å) | 10.505(1) | 8.383(4) |
| $b$ (Å) | 14.049(1) | 9.974(4) |
| $c$ (Å) | 14.938(2) | 15.410(8) |
| $\alpha$ (°) | – | 91.68(4) |
| $\beta$ (°) | – | 105.88(4) |
| $\gamma$ (°) | – | 114.88(3) |
| $U$ (Å$^3$) | 2204.6(4) | 1108.8(9) |
| $Z$ | 4 | 2 |
| $D$ (Calc) (Mg m$^{-3}$) | 2.316 | 2.303 |
| $F$(000) | 1472 | 736 |
| $\mu$(Mo–K$_\alpha$) (mm$^{-1}$) | 4.190 | 4.166 |
| $\theta$ range for data collection (°) | 2.37–23.91 | 2.28–22.91 |
| Index ranges | $0 \leq h \leq 12$;<br>$0 \leq k \leq 16$;<br>$0 \leq l \leq 17$ | $-9 \leq h \leq 8$;<br>$-10 \leq k \leq 10$;<br>$0 \leq l \leq 16$ |
| No. of reflections observed | 1822 | 3061 |
| No. of independent reflections ($R_{int}$ = 0.0000) | 1822 | 3061 |
| Data/restraints/parameters | 1821/0/158 | 3052/0/291 |
| Goodness-of-fit on $F^2$ | 1.076 | 1.046 |
| Final $R$ indices ($I > 2\sigma(I)$) | $R_1 = 0.0592$<br>$wR_2 = 0.1764$ | $R_1 = 0.0794$<br>$wR_2 = 0.1913$ |
| R indices (all data) | $R_1 = 0.0840$<br>$wR_2 = 0.1983$ | $R_1 = 0.1332$<br>$wR_2 = 0.2410$ |
| Largest diffracting, peak and hole (e Å$^{-3}$) | 1.154 and −1.208 | 1.545 and −1.159 |
| Maximum, minimum transmission corrections | 1.000 and 0.622 | 1.000 and 0.056 |
| Weighting scheme, $w_c$ | Calc. | Calc. |
| | $w = 1/[\sigma^2(F_o^2) + (0.0780P)^2 + 77.7415P]$ where $P = (F_o^2 + 2F_c^2)/3$ | $w = 1/[\sigma^2(F_o^2) + (0.1427P)^2 + 10.4791P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| Extinction coefficient | 0.0000(5) | 0.0015(9) |

[a] The refinement method used in the case of the two compounds was full-matrix least squares on $F^2$.

isolated from the first fraction. The second fraction was eluted with 1:1 hexane–dichloromethane solvent mixture. This afforded the following compounds in the order of elution: dark red $Cp_2Mo_2Fe(CO)_7(\mu_3\text{-Te})$ (**10**; trace), red $Cp_2Mo_2Fe(CO)_7(\mu_3\text{-S})$ (**3**; 49 mg, 7%) and brown *cis*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-S})(\mu_3\text{-Te})$ (**11**; 169 mg, 18%). From the third fraction, pure $Cp_2Mo_2Fe_2(CO)_7(\mu_3\text{-S})(\mu_3\text{-Te})$ (**12**; 318 mg, 35%) was obtained. **9**: Anal. Found: C, 25.6; H, 1.34. $C_{18}H_{10}Fe_2Mo_2O_8STe$; Anal. Calc.: C, 26.4; H, 1.22%. **11**: Anal. Found: C, 25.4; H, 1.35. $C_{18}H_{10}Fe_2Mo_2O_8STe$; Anal. Calc.: C, 26.4; H, 1.22%.

### 2.5. Reaction of $Fe_3(CO)_9(\mu_3\text{-S})(\mu_3Te)$ with $Cp_2Mo_2(CO)_6$

A benzene solution (250 ml) of $Fe_3(CO)_9(\mu_3\text{-S})(\mu_3Te)$ (2 g, 3.45 mmol) and $Cp_2Mo_2(CO)_6$ (0.805 g, 1.725 mmol) was heated to 65°C for 36 h with constant stirring. The reaction was monitored by TLC. The solution was filtered through Celite to remove the insoluble material. The solvent was removed in vacuo. The residue was dissolved in dichloromethane and chromatographic work-up was performed. On elution with 1:1 hexane–dichloromethane solvent mixture three fractions were isolated. The first two fractions were subjected to further chromatographic work-up using silica gel TLC plates. Chromatography of the first fraction with 7:3 hexane–dichloromethane solvent mixture as eluent afforded the following compounds in order of elution: yellowish–brown $Cp_2Mo_2Fe_2(CO)_6(\mu_4\text{-Te})(\mu_3\text{-Te})(\mu_3\text{-S})$ (**13**; 138 mg, 9%), yellowish-brown $Cp_2Mo_2Fe_2(CO)_6(\mu_4\text{-Te})(\mu_3\text{-S})_2$ (**14**; 68 mg, 5%), brown $Cp_2Mo_2Fe_2(CO)_6(\mu_4\text{-Te})(\mu_3\text{-Te})(\mu_3\text{-Te})$ (**15**; trace) and green *trans*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-S})(\mu_3\text{-Te})$ (**9**; 367 mg, 23%). Elution of the second fraction with 1:1 hexane–dichloromethane solvent mixtures yielded the following compounds in order of elution: dark red $Cp_2Mo_2Fe(CO)_7(\mu_3\text{-Te})$ (**10**; trace), red $Cp_2Mo_2Fe(CO)_7(\mu_3\text{-S})$ (**3**; 52 mg, 5%) and brown *cis*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-S})(\mu_3\text{-Te})$ (**11**; 465 mg, 33%). The third fraction was eluted with 3:7 hexane–dichloromethane solvent mixtures to isolate the compound $Cp_2Mo_2Fe_2(CO)_7(\mu_3\text{-S})(\mu_3\text{-Te})$ (**12**; 136 mg, 10%).

### 2.6. Crystal structure determination of **1** and **4**

Crystals of compounds **1** and **4** suitable for X-ray diffraction analysis were grown from hexane–dichloromethane solvent mixtures by slow evaporation of the solvents at 0°C.

Relevant crystallographic data and details of measurements are given in Table 1. Atomic coordinates and equivalent isotropic displacement parameters for **1** and **4** are given in Tables 2 and 3, respectively. Selected bond lengths and bond angles for **1** and **4** are given in Tables 4 and 5, respectively. Crystallographic measurements were made at 293(2) K on a CAD4 automatic four circle diffractometer. Data were corrected for Lorentz and polarisation and also for absorption [10].

In the final least-squares cycles, all atoms were allowed to vibrate anisotropically. Hydrogen atoms were included at calculated positions where relevant. The solution of the structure was done by SHELX86 [11] and the refinement was done by SHELXL93 [12]. The asymmetric units (shown in Figs. 2 and 3) along with the labelling scheme used were produced using ORTEX [13]. The asymmetric unit in compound **1** was seen to consist of one half of a complex above with the remaining half of the molecule being generated via a mirror plane intrinsic in the space group symmetry. Atoms Fe(1), Se(1), S(1), Fe(2), C(1), O(1), C(4) and O(4) were seen to be located on this mirror plane. In compound **4** the selenium and sulphur positions were disordered as it was evident from early difference Fourier electron density maps. Isotropic refinement of partial atoms (Se(1), Se(2), S(1) and S(2)) at both capping sites revealed that the disorder ratio was 50:50 for selenium and sulphur. Subsequent anisotropic refinement of the four partial atoms was conducted with fixed site-occupancy factors. Satisfactory convergence was achieved by treating each of the Se(1)/S(1) and Se(2)/S(2) atom pairs with common positional and thermal displacement parameters at their respective locations. The thermal displacement parameters for the cyclopentadienyl carbons would normally suggest disorder due to ring staggering. However, an attempt to model disorder of this type was abondoned because of the resulting 'non-positive definite' ellipsoids within the rings, in addition to destabilisation of the refinement.

Table 2
Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **1**

| Atom | $x$ | $y$ | $z$ | $U_{eq}$ [a] |
|---|---|---|---|---|
| Mo(1) | 1131(1) | 2196(1) | 687(1) | 29(1) |
| Se(1) | 2500 | 890(1) | 41(1) | 38(1) |
| S(1) | 2500 | 3062(3) | 1628(3) | 27(1) |
| Fe(1) | 2500 | 775(2) | 1563(2) | 38(1) |
| Fe(2) | 2500 | 3911(2) | 400(2) | 32(1) |
| O(1) | 2500 | 1615(13) | 3358(11) | 66(5) |
| O(2) | 4534(15) | −636(9) | 1794(9) | 79(4) |
| O(3) | 1004(13) | 2759(9) | −1324(7) | 63(4) |
| O(4) | 2500 | 4637(13) | −1459(11) | 62(5) |
| O(5) | 4549(11) | 5140(8) | 1039(9) | 61(3) |
| C(1) | 2500 | 1265(14) | 2658(13) | 37(5) |
| C(2) | 3750(19) | −78(10) | 1738(11) | 54(5) |
| C(3) | 1189(14) | 2642(11) | −591(10) | 41(4) |
| C(4) | 2500 | 4356(20) | −709(16) | 54(6) |
| C(5) | 3769(15) | 4666(10) | 774(10) | 41(4) |
| C(6) | −866(13) | 2902(15) | 689(13) | 59(5) |
| C(7) | −978(14) | 2062(14) | 187(13) | 57(5) |
| C(8) | −727(15) | 1282(10) | 801(11) | 46(4) |
| C(9) | −523(14) | 1724(10) | 1650(10) | 42(4) |
| C(10) | −594(14) | 2701(10) | 1555(12) | 44(4) |

[a] $U_{eq}$ is defined as one-third of the trace of the orthogonalized $U_{ij}$ tensor.

Table 3
Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **4**

| Atom | $x$ | $y$ | $z$ | $U_{eq}$ [a] |
|---|---|---|---|---|
| Se(1) | 4189(5) | 8629(4) | 8608(3) | 74(1) |
| S(1) | 4189(5) | 8629(4) | 8608(3) | 74(1) |
| Se(2) | 1950(5) | 6372(4) | 6394(3) | 75(1) |
| S(2) | 1950(5) | 6372(4) | 6394(3) | 75(1) |
| Mo(1) | 2697(2) | 8831(2) | 7088(1) | 45(1) |
| Mo(2) | 1777(2) | 6170(2) | 7914(1) | 45(1) |
| Fe(1) | 1628(4) | 8716(3) | 8691(2) | 56(1) |
| Fe(2) | −779(3) | 6279(3) | 6310(2) | 56(1) |
| O(1) | 2071(29) | 7916(25) | 10523(14) | 111(7) |
| O(2) | −2052(28) | 8476(31) | 8479(18) | 143(9) |
| O(3) | 3436(32) | 111987(24) | 9260(16) | 113(6) |
| O(4) | −2792(27) | 3005(24) | 5720(16) | 116(7) |
| O(5) | −4012(26) | 6540(30) | 6515(18) | 134(9) |
| O(6) | −1333(31) | 7087(24) | 4500(14) | 108(6) |
| O(7) | −520(22) | 9737(20) | 6804(13) | 93(5) |
| O(8) | −2050(20) | 5269(20) | 8174(13) | 88(5) |
| C(1) | 4581(80) | 9641(34) | 6179(42) | 149(21) |
| C(2) | 3270(44) | 10085(47) | 5895(21) | 111(12) |
| C(3) | 3530(43) | 11140(34) | 6600(30) | 103(11) |
| C(4) | 5008(40) | 11249(26) | 7316(18) | 84(8) |
| C(5) | 5636(32) | 10329(37) | 7018(26) | 85(8) |
| C(6) | 784(32) | 3825(28) | 8343(28) | 103(12) |
| C(7) | 2095(75) | 4826(50) | 9100(26) | 113(13) |
| C(8) | 3712(63) | 5357(30) | 8937(35) | 137(20) |
| C(9) | 3433(46) | 4795(41) | 8102(34) | 108(13) |
| C(10) | 1564(59) | 3791(32) | 7666(20) | 102(11) |
| C(11) | 1907(32) | 8230(26) | 9822(20) | 77(7) |
| C(12) | −688(35) | 8518(32) | 8505(19) | 88(8) |
| C(13) | 2721(34) | 10705(30) | 9031(16) | 73(6) |
| C(14) | −1990(30) | 4326(34) | 5982(17) | 79(7) |
| C(15) | −2702(31) | 6459(33) | 6492(20) | 92(8) |
| C(16) | −1121(28) | 6766(25) | 5224(18) | 73(6) |
| C(17) | 484(28) | 9153(23) | 6979(17) | 67(6) |
| C(18) | −667(30) | 5845(26) | 7986(18) | 74(6) |

[a] $U_{eq}$ is defined as one-third of the trace of the orthogonalized $U_{ij}$ tensor.

## 3. Results and discussion

### 3.1. Synthesis and spectroscopic characterisation

In contrast to the formation of 'Braunstein' and 'Curtis' isomers of $Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)_2$ in yields of 80 and 20%, respectively from the reaction of $Fe_2(CO)_6(\mu\text{-}S_2)$ with $Cp_2Mo_2(CO)_4$, we find that the mixed-chalcogenide, $Fe_2(CO)_6(\mu\text{-}SSe)$ reacts with $Cp_2Mo_2(CO)_4$ at r.t. to yield *trans*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Se)$ (**1**) and *cis*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Se)$ (**4**) in much lower yields, (8 and 15%, respectively) (Scheme 1). Also obtained from our reaction are the previously reported clusters: $Cp_2Mo_2Fe(CO)_7(\mu_3\text{-}Se)$ (**2**; 6%) [14], $Cp_2Mo_2Fe(CO)_7(\mu_3\text{-}S)$ (**3**; 5%) [15], and $Cp_2Mo_2Fe_2(CO)_7(\mu_3\text{-}S)(\mu_3\text{-}Se)$ (**5**; 27%) [16]. The new clusters **1** and **4** are formed in better yields (23 and

35%, respectively) when a benzene solution containing $Fe_3(CO)_9(\mu_3\text{-S})(\mu_3\text{-Se})$ and $Cp_2Mo_2(CO)_6$ is heated at 65°C for 36 h (Scheme 2). From this thermolysis reaction, the following previously reported clusters are also obtained: $Cp_2Mo_2Fe_2(CO)_6(\mu_4\text{-Se})(\mu_3\text{-Se})(\mu_3\text{-S})$ (**6**; 10%), $Cp_2Mo_2Fe_2(CO)_6(\mu_4\text{-Se})(\mu_3\text{-S})_2$ (**7**; trace) [16], $Cp_2Mo_2Fe_2(CO)_6(\mu_4\text{-Se})(\mu_3\text{-Se})_2$ (**8**; trace) [17], $Cp_2Mo_2Fe(CO)_7(\mu_3\text{-Se})$ (**2**; trace), $Cp_2Mo_2Fe(CO)_7(\mu_3\text{-S})$ (**3**; trace), and $Cp_2Mo_2Fe_2(CO)_7(\mu_3\text{-S})(\mu_3\text{-Se})$ (**5**; 15%).

Similarly, when a benzene solution of $Fe_2(CO)_6(\mu\text{-STe})$ and $Cp_2Mo_2(CO)_4$ was stirred at r.t. for 2 h, the following compounds were isolated: $Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-S})(\mu_3\text{-Te})$ (**9**; 10%), $Cp_2Mo_2Fe(CO)_7(\mu_3\text{-Te})$ (**10**; trace) [15], $Cp_2Mo_2Fe(CO)_7(\mu_3\text{-S})$ (**3**; 7%), $Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-S})(\mu_3\text{-Te})$ (**11**; 18%) and $Cp_2Mo_2Fe_2(CO)_7(\mu_3\text{-S})(\mu_3\text{-Te})$ (**12**; 35%) [15]. The new compounds **9** and **11** could be isolated in higher yields when a benzene solution containing $Fe_3(CO)_9(\mu_3\text{-S})(\mu_3\text{-Te})$ and $Cp_2Mo_2(CO)_6$ was heated at 65°C for 36 h. This reaction also produces the following compounds: $Cp_2Mo_2Fe_2(CO)_6(\mu_4\text{-Te})(\mu_3\text{-Te})(\mu_3\text{-S})$ (**13**; 9%), $Cp_2Mo_2Fe_2(CO)_6(\mu_4\text{-Te})(\mu_3\text{-S})_2$ (**14**; 5%), $Cp_2Mo_2Fe_2(CO)_6(\mu_4\text{-Te})(\mu_3\text{-Te})_2$ (**15**; trace) [15], $Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-S})(\mu_3\text{-Te})$ (**9**; 23%), $Cp_2Mo_2Fe(CO)_7(\mu_3\text{-Te})$ (**10**; trace), $Cp_2Mo_2Fe(CO)_7(\mu_3\text{-S})$ (**3**; 5%), $Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-S})(\mu_3\text{-Te})$ (**11**; 33%), $Cp_2Mo_2Fe_2(CO)_7(\mu_3\text{-S})(\mu_3\text{-Te})$ (**12**; 10%).

Table 4
Selected bond lengths (Å) and bond angles (°) for **1** [a]

| | | | |
|---|---|---|---|
| *Bond lengths* (Å) | | | |
| Mo(1)–C(3) | 2.01(2) | Se(1)–Mo(1) # 1 | 2.523(2) |
| Mo(1)–C(6) | 2.32(2) | S(1)–Fe(2) | 2.188(5) |
| Mo(1)–C(10) | 2.34(2) | S(1)–Mo(1) # 1 | 2.351(4) |
| Mo(1)–C(8) | 2.343(14) | Fe(1)–C(1) | 1.77(2) |
| Mo(1)–C(7) | 2.35(2) | Fe(1)–C(2) | 1.80(2) |
| Mo(1)–S(1) | 2.351(4) | Fe(1)–C(2) # 1 | 1.80(2) |
| Mo(1)–C(9) | 2.351(14) | Fe(1)–Mo(1) # 1 | 2.786(3) |
| Mo(1)–Se(1) | 2.523(2) | Fe(2)–C(4) | 1.77(3) |
| Mo(1)–Fe(1) | 2.786(3) | Fe(2)–C(5) | 1.79(2) |
| Mo(1)–Fe(2) | 2.839(3) | Fe(2)–C(5) # 1 | 1.79(2) |
| Mo(1)–Mo(1) # 1 | 2.876(2) | Fe(2)–C(3) | 2.70(2) |
| Se(1)–Fe(1) | 2.279(4) | Fe(2)–Mo(1) # 1 | 2.839(3) |
| *Bond angles* (°) | | | |
| Se(1)–Mo(1)–Fe(2) | 105.73(6) | Mo(1)–S(1)–Mo(1) # 1 | 75.43(14) |
| Fe(1)–Mo(1)–Fe(2) | 114.67(6) | Se(1)–Fe(1)–Mo(1) # 1 | 58.73(8) |
| S(1)–Mo(1)–Mo(1) # 1 | 52.28(7) | Se(1)–Fe(1)–Mo(1) | 58.73(8) |
| Se(1)–Mo(1)–Mo(1) # 1 | 55.25(4) | Mo(1) # 1–Fe(1)–Mo(1) | 62.14(8) |
| Fe(1)–Mo(1)–Mo(1) # 1 | 58.93(4) | S(1)–Mo(1)–Se(1) | 104.86(9) |
| Fe(2)–Mo(1)–Mo(1) # 1 | 59.57(4) | S(1)–Mo(1)–Fe(1) | 76.93(12) |
| Fe(1)–Se(1)–Mo(1) # 1 | 70.72(8) | Se(1)–Mo(1)–Fe(1) | 50.55(8) |
| Fe(1)–Se(1)–Mo(1) | 70.72(8) | S(1)–Mo(1)–Fe(2) | 48.77(12) |
| Mo(1) # 1–Se(1)–Mo(1) | 69.50(7) | S(1)–Fe(2)–Mo(1) # 1 | 53.89(11) |
| Fe(2)–S(1)–Mo(1) | 77.34(14) | Mo(1)–Fe(2)–Mo(1) # 1 | 60.86(7) |
| Fe(2)–S(1)–Mo(1) # 1 | 77.34(14) | | |

[a] Symmetry transformations used to generate equivalent atoms: # 1 $-x+1/2$, $y$, $z$.

Table 5
Selected bond lengths (Å) and bond angles (°) for **4**

| | | | |
|---|---|---|---|
| *Bond lengths* (Å) | | | |
| Se(1)–Fe(1) | 2.222(4) | Mo(2)–C(18) | 1.97(2) |
| Se(1)–Mo(1) | 2.394(4) | Mo(2)–C(9) | 2.30(2) |
| Se(1)–Mo(2) | 2.395(4) | Mo(2)–C(6) | 2.31(2) |
| Se(2)–Fe(2) | 2.218(4) | Mo(2)–C(10) | 2.32(2) |
| Se(2)–Mo(2) | 2.395(4) | Mo(2)–C(7) | 2.32(2) |
| Se(2)–Mo(1) | 2.395(4) | Mo(2)–C(8) | 2.36(3) |
| Mo(1)–C(17) | 1.98(2) | Mo(2)–Fe(2) | 2.833(4) |
| Mo(1)–C(2) | 2.30(3) | Mo(2)–Fe(1) | 2.840(3) |
| Mo(1)–C(1) | 2.30(2) | Fe(1)–C(13) | 1.79(3) |
| Mo(1)–C(4) | 2.31(2) | Fe(1)–C(11) | 1.80(3) |
| Mo(1)–C(5) | 2.32(2) | Fe(1)–C(12) | 1.81(2) |
| Mo(1)–C(3) | 2.32(2) | Fe(2)–C(16) | 1.74(3) |
| Mo(1)–Fe(1) | 2.839(3) | Fe(2)–C(14) | 1.75(3) |
| Mo(1)–Fe(2) | 2.842(4) | Fe(2)–C(15) | 1.79(2) |
| Mo(1)–Mo(2) | 2.876(3) | | |
| *Bond angles* (°) | | | |
| Fe(1)–Se(1)–Mo(1) | 75.81(13) | S(2)–Fe(2)–Mo(1) | 54.85(12) |
| Fe(1)–Se(1)–Mo(2) | 75.82(14) | Mo(2)–Fe(2)–Mo(1) | 60.91(8) |
| Mo(1)–Se(1)–Mo(2) | 73.83(13) | S(2)–Mo(2)–Se(2) | 0.0(3) |
| Fe(2)–Se(2)–Mo(2) | 75.68(13) | S(2)–Mo(2)–Se(1) | 96.24(14) |
| Fe(2)–Se(2)–Mo(1) | 75.94(14) | Se(2)–Mo(2)–Se(1) | 96.24(14) |
| Mo(2)–Se(2)–Mo(1) | 73.81(13) | S(2)–Mo(2)–S(1) | 96.24(14) |
| Se(1)–Mo(1)–Se(2) | 96.27(14) | Se(2)–Mo(2)–S(1) | 96.24(14) |
| S(1)–Mo(1)–Se(2) | 96.27(14) | Se(1)–Mo(2)–S(1) | 0.0(4) |
| S(2)–Mo(2)–Fe(1) | 110.79(12) | S(2)–Mo(2)–Fe(2) | 49.33(10) |
| Se(2)–Mo(2)–Fe(1) | 110.79(12) | Se(2)–Mo(2)–Fe(2) | 49.33(10) |
| Se(1)–Mo(2)–Fe(1) | 49.33(10) | Se(1)–Mo(2)–Fe(2) | 110.91(12) |
| S(1)–Mo(2)–Fe(1) | 49.33(10) | S(1)–Mo(2)–Fe(2) | 110.91(12) |
| Fe(2)–Mo(2)–Fe(1) | 85.17(10) | Se(1)–Mo(1)–Fe(1) | 49.36(10) |
| Se(1)–Fe(1)–Mo(1) | 54.83(11) | S(1)–Mo(1)–Fe(1) | 49.36(10) |
| S(1)–Fe(1)–Mo(1) | 54.83(11) | S(2)–Mo(1)–Fe(1) | 110.81(12) |
| Se(1)–Fe(1)–Mo(2) | 54.85(12) | Se(2)–Mo(1)–Fe(1) | 110.81(12) |
| S(1)–Fe(1)–Mo(2) | 54.85(12) | Se(1)–Mo(1)–Fe(2) | 110.67(12) |
| Mo(1)–Fe(1)–Mo(2) | 60.86(7) | S(1)–Mo(1)–Fe(2) | 110.67(12) |
| Se(2)–Fe(2)–Mo(2) | 54.99(12) | S(2)–Mo(1)–Fe(2) | 49.20(10) |
| S(2)–Fe(2)–Mo(2) | 54.99(12) | Se(2)–Mo(1)–Fe(2) | 49.20(10) |
| Se(2)–Fe(2)–Mo(1) | 54.85(12) | Fe(1)–Mo(1)–Fe(2) | 85.03(10) |

The new dichalcogenide clusters **1**, **4**, **9** and **11** were characterised by IR and $^1$H-, $^{77}$Se- and $^{125}$Te-NMR spectroscopy (Table 6). All other compounds were identified on the basis of comparison of their IR spectra in the carbonyl stretching region with those reported earlier. The IR spectra of **1** and **9** indicate the presence of terminal carbonyl groups, in the range of 1940 and 2030 $cm^{-1}$ as well as semi-doubly bridging carbonyl groups in the range of 1850 and 1900 $cm^{-1}$. The presence of semi-triply bridging and terminal carbonyl groups in the infrared spectra of **4** and **11** is evidenced by $\nu$ CO in the range of 1750 to 2030 $cm^{-1}$. The carbonyl stretching frequencies of the corresponding bands decrease from the chalcogen combination SSe to STe. The $^1$H-NMR spectra of the new dichalcogenide clusters **1**, **4**, **9** and **11** each show a single peak for the two equivalent Cp ligands. $^{77}$Se-NMR spectrum of both **1** and **4** show single peaks, corresponding to the $\mu_3$-Se

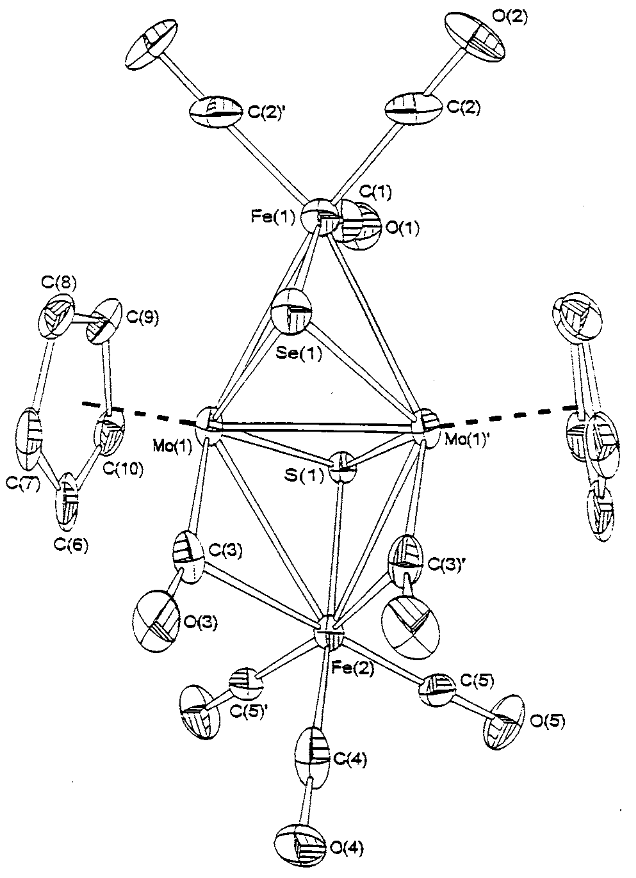


Fig. 2. Molecular structure of *trans*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Se)$ (**1**).

atom. The $^{77}$Se-NMR signal for cluster **1** ($\delta$ 639.4 ppm) is downfield as compared to that in cluster **4** ($\delta$ 418.63 ppm). The $^{125}$Te-NMR spectra shows a single peak indicating the presence of triply bridging Te atoms in clusters **9** and **11,** the value for cluster **9** ($\delta$ 637.45 ppm) being downfield as compared to that in cluster **11** ($\delta$ 322 ppm).

### 3.2. Molecular structures of **1** and **4**

The molecular structure of **1,** shown in Fig. 2, consists of an open $Mo_2Fe_2$ butterfly core with a $\mu_3$-S and a $\mu_3$-Se capping opposite sides of the two $Mo_2Fe$ faces. Each Mo atom has a ($\eta^5$-$C_5H_5$) group and one carbonyl group attached to it; the latter semibridges to one of the Fe atoms. Each Fe atom has three terminal carbonyl groups. Assuming that the $\mu_3$-S and $\mu_3$-Se are four-electron donors each, the cluster is electron precise, according to the 18-electron rule.

The molecular structure of **4,** shown in Fig. 3, also consists of an open $Mo_2Fe_2$ butterfly tetrahedron with a $\mu_3$-S and a $\mu_3$-Se capping the two closed $Mo_2Fe$ faces on the same side. The two open $Fe_2Mo$ faces bear a semi-triply bridging carbonyl group each. In addition, each Fe atom bears three terminal carbonyl groups and each Mo atom a ($\eta^5$-$C_5H_5$) group. The Mo–Mo bond distance in **1** (2.876(2) Å) is identical to that in **4** (2.876(3)Å), but is slightly longer than the Mo–Mo

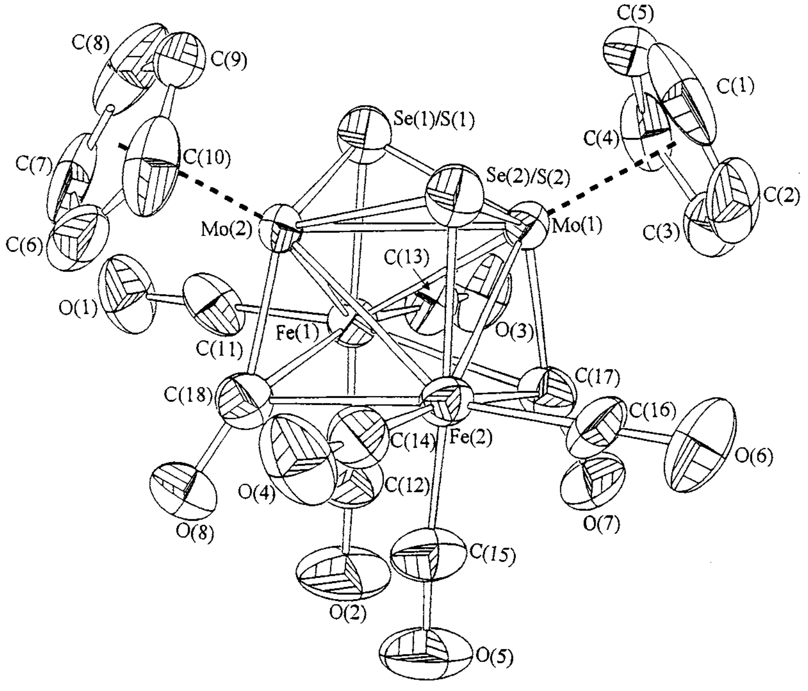
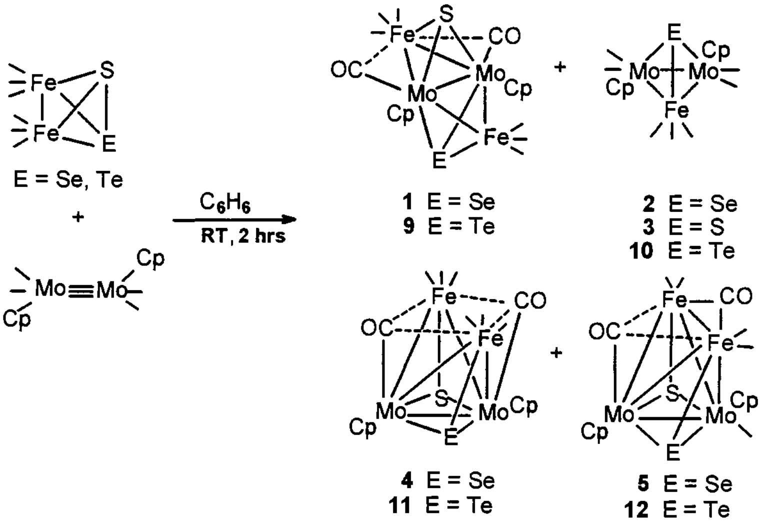
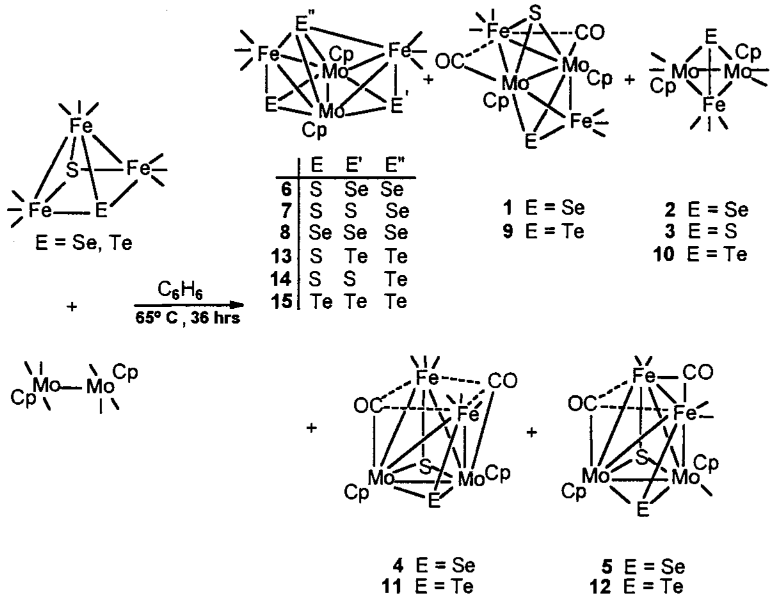


Fig. 3. Molecular structure of *cis*-$Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)(\mu_3\text{-}Se)$ (**4**).



Scheme 1.



Scheme 2.

bond lengths in the trichalcogen-bridged clusters of the type $Cp_2Mo_2Fe_2(CO)_6(\mu_4\text{-}E)(\mu_3\text{-}E)(\mu_3\text{-}E')$: **6** (2.7194(12)Å), **13** (2.7510(13) Å), **14** (2.752(2) Å) and **15** (2.8172 Å), and the dichalcogen-bridged cluster, $Cp_2Mo_2Fe_2(CO)_7(\mu_3\text{-}S)(\mu_3\text{-}Te)$, **12** (2.783(1) Å), but is shorter than the Mo–Mo bond lengths in the mono-chalcogen-bridged clusters, $Cp_2Mo_2Fe(CO)_7(\mu_3\text{-}E)$, **2** (3.096 Å) and **10** (3.129(1) Å), as well as in $Cp_2Mo_2(CO)_6$ (3.235 Å).

## 4. Conclusions

The *cis*-Braunstein and the *trans*-Curtis isomers of $Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}S)_2$ have previously been prepared from the reaction of $Cp_2Mo_2(CO)_4$ and $Fe_2(CO)_6(\mu\text{-}S_2)$. We have previously reported a r.t. reaction of $Fe_2(CO)_6(\mu\text{-}Se_2)$ with $Cp_2Mo_2(CO)_4$ and the thermolysis reaction of $Fe_3(CO)_9(\mu_3\text{-}Se)_2$ and $Cp_2Mo_2(CO)_6$, which yielded the clusters $Cp_2Mo_2Fe_2(CO)_6(\mu_4\text{-}Se)(\mu_3\text{-}Se)_2$, $Cp_2Mo_2Fe_2(CO)_7(\mu_3\text{-}Se)_2$ and $Cp_2Mo_2Fe(CO)_7(\mu_3\text{-}Se)$. The Se-bridged Braunstein and Curtis isomers of $Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}Se)_2$ were not obtained. Similarly, the Te-analogue, $Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}Te)_2$, is not known. In this paper, we have described the synthesis of the mixed-chalcogenide analogues of the Braunstein and Curtis clusters, **1**, **4**, **9** and **11**. Thus, whereas $Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}Se)_2$ or $Cp_2Mo_2Fe_2(CO)_8(\mu_3\text{-}Te)_2$ has not been observed, the presence of a $\mu_3$-Se and $\mu_3$-S or $\mu_3$-Te and $\mu_3$-S as bridging ligands in these types of clusters imparts sufficient stability for isolation of **1**, **4**, **9** and **11**.

Table 6
Spectroscopic data for compounds **1**, **4**, **9**, **11**

| Compounds | IR (ν, $cm^{-1}$) | $^1H$-NMR ($\delta$, $CDCl_3$) | $^{77}Se$-NMR ($\delta$, $CDCl_3$) | $^{125}Te$-NMR ($\delta$, $CDCl_3$) |
|---|---|---|---|---|
| $Cp_2Mo_2Fe_2SSe(CO)_8$ (**1**) | 2040(s) [a], 2001(s), 1980(s), 1965(s), 1933(m), 1896(m) | | | |
| | 2043(m) [b], 2003(s), 1981(m), 1934(m) | 5.25 ($C_5H_5$) | 639.4 | – |
| $Cp_2Mo_2Fe_2SSe(CO)_8$ (**4**) | 2042(s) [a], 2010(vs), 1978(s), 1948(s), 1832(m), 1780(m) | | | |
| | 2050(s) [b], 2024(vs), 1969(s), 1802(m) | 5.27 ($C_5H_5$) | 418.6 | – |
| $Cp_2Mo_2Fe_2STe(CO)_8$ (**9**) | 2037(s) [a], 1995(s), 1978(s), 1964(s), 1931(m), 1895(m) | | | |
| | 2040(m) [b], 1998(s), 1979(m), 1932(m) | 5.18 ($C_5H_5$) | – | 637.5 |
| $Cp_2Mo_2Fe_2STe(CO)_8$ (**11**) | 2034(s) [a], 2007(s), 1962(s), 1834(m), 1782(s) | | | |
| | 2045(s) [b], 2019(vs), 1964(m), 1820(m), 1794(m) | 5.18 ($C_5H_5$) | – | 322.0 |

[a] KBr pellet.
[b] $CH_2Cl_2$.

## 5. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC, No. 116940 for compound **1** and 116941 for compound **4**. Copies of the information may be obtained free of charge from The Director, CCDC, 12, Union Road, Cambridge CB2 1EZ (Fax +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

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