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Synthesis of double and multiple butterfly Fe/E (E = S, Se, Te) cluster complexes via reactions of complex anions $[(\mu\text{-RE})(\mu\text{-CO})Fe_2(CO)_6]^-$ (E = S, Se, Te). Crystal structures of $(\mu\text{-}p\text{-MeC}_6H_4Se)(\mu\text{-MeS})[Fe_2(CO)_6]_2(\mu_4\text{-S})$ and $\{(\mu\text{-}t\text{-BuS})[Fe_2(CO)_6]_2(\mu_4\text{-S})\}_2[(\mu\text{-}p\text{-SCH}_2)_2C_6H_4]$

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Abstract

The $[Et_3NH]^+$ salts of μ-CO anions $[(\mu\text{-RE})(\mu\text{-CO})Fe_2(CO)_6]^-$ (E = S, Se) prepared from $Fe_3(CO)_{12}$, REH (R = S,Se) and Et_3N reacted with $\mu\text{-S}_2Fe_2(CO)_6$ and subsequent treatment of the intermediate $[Et_3NH]^+$ salts of S-centered anions $(\mu\text{-RE})(\mu\text{-S}^-)[Fe_2(CO)_6]_2(\mu_4\text{-S})$ with MeI, $PhCH_2Br$, $p\text{-(BrCH}_2)_2C_6H_4$ or 1,3,5-$(BrCH_2)_3C_6H_3$ to give a series of μ_4-S-containing double and multiple butterfly clusters $(\mu\text{-RE})(\mu\text{-MeS})[Fe_2(CO)_6]_2(\mu_4\text{-S})$ (**7**–**9**, RE = *t*-BuS, PhSe, *p*-MeC_6H_4Se), $(\mu\text{-}p\text{-MeC}_6H_4Se)(\mu\text{-PhCH}_2S)[Fe_2(CO)_6]_2(\mu_4\text{-S})$ (**11**), $\{(\mu\text{-}t\text{-BuS})[Fe_2(CO)_6]_2(\mu_4\text{-S})\}_2[(\mu\text{-}p\text{-(SCH}_2)_2C_6H_4]$ (**12**) and $\{(\mu\text{-}t\text{-BuS})[Fe_2(CO)_6]_2(\mu_4\text{-S})\}_3[\mu\text{-1,3,5-(SCH}_2)_3C_6H_3]$ (**13**), whereas the $[MgBr]^+$ salts of μ-CO anions $[(\mu\text{-RTe})(\mu\text{-CO})Fe_2(CO)_6]^-$ prepared from Grignard reagents RMgBr and elemental Te followed by treatment of the intermediates RTeMgBr with $Fe_3(CO)_{12}$, reacted with S_2Cl_2 to afford a series of RTe-containing and μ-S–S-μ bridged double butterfly clusters $[(\mu\text{-RTe})\,Fe_2(CO)_6]_2(\mu\text{-S–S-}\mu)$ (**14**–**17**, R = Et, *i*-Bu, Ph, *p*-MeC_6H_4). Similarly, the RTe- and μ_4-S-containing double butterfly cluster $(\mu\text{-}p\text{-MeC}_6H_4Te)(\mu\text{-MeS})[Fe_2(CO)_6]_2(\mu_4\text{-S})$ (**10**) could be prepared by reaction of the $[MgBr]^+$ salt of μ-CO anion $(\mu\text{-}p\text{-MeC}_6H_4Te)(\mu\text{-CO})Fe_2(CO)_6]^-$ with $\mu\text{-S}_2Fe_2(CO)_6$ followed by treatment with MeI. All these new clusters **7**–**17** have been characterized by combustion analysis, IR, ^{1}H-NMR, ^{77}Se-NMR and ^{125}Te-NMR spectroscopies, as well as by X-ray diffraction analyses for **9** (RE = *p*-MeC_6H_4Se) and **12** (RE = *t*-BuS). © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Sulfur; Selenium; Tellurium; Iron; CO-bridged anions; X-ray structures

1. Introduction

Since the first preparations of complex anions $[(\mu\text{-RE})(\mu\text{-CO})Fe_2(CO)_6]^-$ (**1**, E = S [1]; **2**, E = Se [2]; **3**, E = Te [3]), their reactions have been intensively studied and successfully utilized in the synthesis of single, double and multiple butterfly Fe/E cluster complexes containing organic and inorganic structural moieties [1–23]. Reactions of such anions with most electrophiles can be rationalized in terms of their behavior as iron-centered nucleophiles and may be classified as two major types. In one type, reactions with the electrophiles containing a leaving group gave neutral products in which the organic or inorganic group replaced the μ-CO ligand. In the other type, reactions with the electrophiles without a leaving group initially produced another anions and finally afforded neutral products by incorporating an additional electrophile. On the basis of our previous studies related to the anions $[(\mu\text{-RE})(\mu\text{-CO})Fe_2(CO)_6]^-$ (1-3) [3,10–23] we continued to study the above-mentioned two types of reactions with the electrophiles containing a perthio group, i.e. sulfur(I) chloride S_2Cl_2 and dithiobis(tricarbonyliron) $\mu\text{-S}_2Fe_2(CO)_6$. Interestingly, these reactions investigated have led us to obtain a series of new double and

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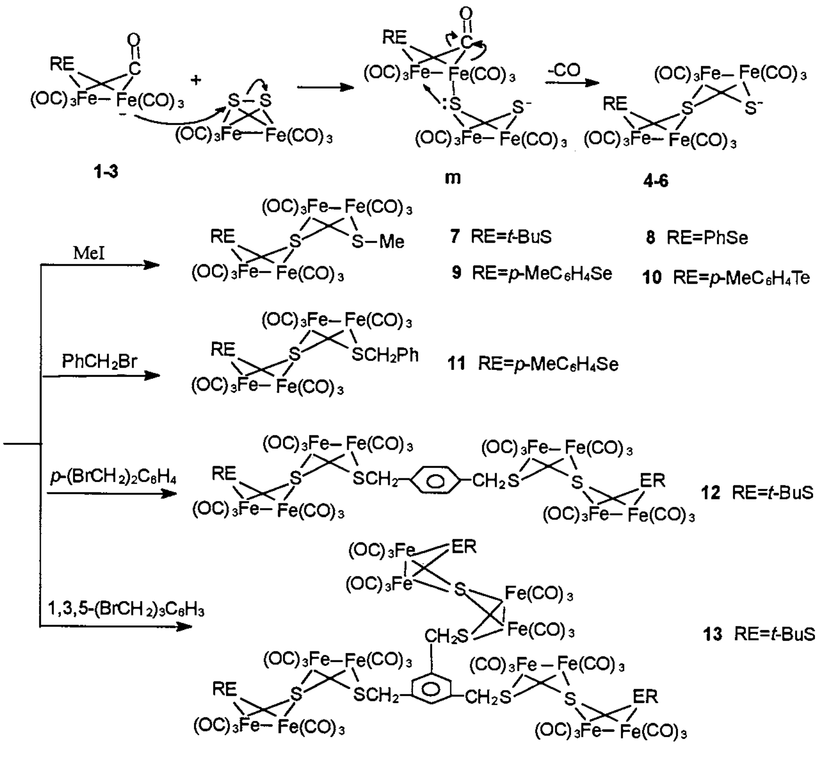
multiple butterfly Fe/E (E = S, Se, Te) cluster complexes. Herein we report their syntheses and spectroscopic characterization, as well as the crystal structures of two representative cluster complexes.

2. Results and discussion

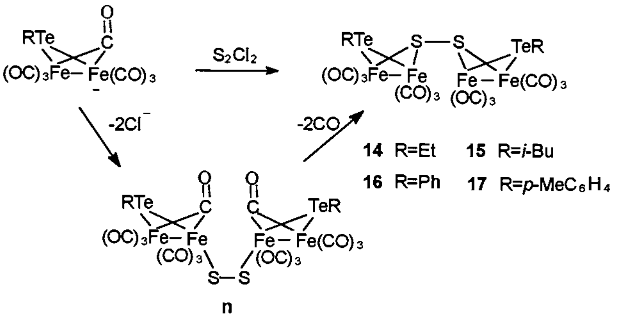
2.1. Reactions of $[(\mu\text{-}RE)(\mu\text{-}CO)Fe_2(CO)_6]^-$ ***(1–3)*** *with* $\mu\text{-}S_2Fe_2(CO)_6$*/alkyl halides leading to* $(\mu\text{-}RE)(\mu\text{-}MeS)\text{-}[Fe_2(CO)_6]_2(\mu_4\text{-}S)$ ***(7–10****; RE = t-BuS, PhSe, p-*MeC_6H_4Se*, p-*MeC_6H_4Te*),* $(\mu\text{-}p\text{-}MeC_6H_4Se)(\mu\text{-}PhCH_2S)[Fe_2(CO)_6]_2(\mu_4\text{-}S)$ ***(11)****,* $\{(\mu\text{-}t\text{-}BuS)[Fe_2(CO)_6]_2(\mu_4\text{-}S)\}_2[(\mu\text{-}p\text{-}(SCH_2)_2C_6H_4]$ ***(12)*** *and* $\{(\mu\text{-}t\text{-}BuS)[Fe_2(CO)_6]_2(\mu_4\text{-}S)\}_3[\mu\text{-}1,3,5\text{-}(SCH_2)_3C_6H_3]$ ***(13)***

Interestingly, we found that the Fe-centered anions $(\mu\text{-RE})(\mu\text{-CO})Fe_2(CO)_6]^-$ (**1**–**3**) could react with $\mu\text{-}S_2Fe_2(CO)_6$, via nucleophilic attack of the negatively charged Fe atom of **1**–**3** at the S atom of $\mu\text{-}S_2Fe_2(CO)_6$ followed by loss of the μ-CO ligand through coordination of the μ_3-S atom to another Fe atom in intermediates m, to give corresponding sulfur-centered anions (**4**–**6**, E = S, Se, Te) (Scheme 1). More interestingly, these anions **4**–**6** were found to be able to further react with alkyl mono-, di- and trihalides, giving a series of double and multiple butterfly Fe/E (E = S, Se, Te) cluster complexes **7**–**13** (Scheme 1). It is worth noting that although the intermediate anions **4**–**6** could not be isolated and directly characterized due to their very unstable nature, the suggested formation of **4**–**6** (Scheme 1) is reasonable in terms of the well-known reductive cleavage of the S–S bond in $\mu\text{-}S_2Fe_2(CO)_6$ under the action of nucleophiles [22]. In addition, their existence has been indirectly proved by the full characterization of their derivatives **7**–**13** (Scheme 1) obtained via in situ alkylations of **4**–**6** by alkyl halides [22].

For the preparations of these double and multiple butterfly clusters, the following points should be mentioned. (i) Anions **1** (RE = *t*-BuS) and **2** (RE = PhSe, *p*-MeC_6H_4Se) were prepared as their $[Et_3NH]^+$ salts from $Fe_3(CO)_{12}$, REH (RE = *t*-BuS, PhSe, *p*-MeC_6H_4Se) and Et_3N [1–16], whereas anion **3** (RE = *p*-MeC_6H_4Te) as its $[MgBr]^+$ salt was prepared by an insertion reaction of elemental Te with Grignard reagent *p*-MeC_6H_4MgBr followed by treatment of the intermediate *p*-$MeC_6H_4TeMgBr$ with $Fe_3(CO)_{12}$ [20]. (ii) Alkyl halides could be added when S-centered anions **4**–**6** were completely formed or they were added with $\mu\text{-}S_2Fe_2(CO)_6$ together since they could not react with either Fe-centered anions **1**–**3** or $\mu\text{-}S_2Fe_2(CO)_6$ between − 78°C and room temperature. (iii) The yields of products with E = S (**7**, **12**, **13**) are greater than those of products with E = Se (**8**, **9**, **11**) and in turn greater than the yield of product with E = Te (**10**). Such an order is consistent with the order of the decreased



Scheme 1.



Scheme 2.

nucleophilicity of the anions **1** > **2** > **3** and is also in good agreement with the increased decomposition trend of the anions **1** < **2** < **3** [3,12,13,16]. (iv) The corresponding single butterfly clusters (μ-RE)$_2$Fe$_2$(CO)$_6$ (RE = t-BuS, PhSe, p-MeC$_6$H$_4$Se, p-MeC$_6$H$_4$Te) were always produced along with products **7**–**13**. These single clusters are believed to be formed by decomposition of anions **1**–**3** to give fragments (μ-RE)Fe(CO)$_3$ followed by their dimerization [3,12,13,16].

It is noteworthy that while products **7** and **9** were reported preliminarily in our communication [22] in connection with the study of the formation and reactions of novel anions **4** (E = S) and **5** (E = Se), products **8** and **10**–**13** prepared from anions **4**–**6** have never been reported so far. All the clusters **7**–**13** were characterized by elemental analysis, IR, ^{1}H-NMR and ^{77}Se-NMR spectroscopies. For example, the ^{1}H-NMR spectra of double clusters **7**–**10** showed one singlet at 1.44 and 2.14–2.17 ppm for their t-Bu and Me groups bonded to bridged S atoms, whereas the ^{77}Se-NMR spectra of double clusters **8**, **9**, **11** displayed one singlet at 249.63, 248.04 or 249.52 ppm for their bridged Se atoms in RE groups. Furthermore, the ^{1}H-NMR spectra of multiple clusters **12** and **13** also showed one singlet at 1.42 or 1.43 ppm for their t-Bu groups attached to bridged S atoms, whereas the ^{1}H-NMR spectrum of **12** displayed only one quartet of AB pattern for its CH$_2$ groups bound to bridged S atoms. Obviously, such NMR spectral patterns for clusters **7**–**13** are completely consistent with the fact that Fe/E clusters containing a μ_4-S or μ_4-Se atom with a general formula [(μ-RE) Fe$_2$(CO)$_6$]$_2$(μ_4-S) or (μ_4-Se) could exist, on the basis of the steric repulsion grounds, as only one isomer in which the two substituents R are bonded to the bridged E (E = S, Se, Te) atoms with an equatorial type of bond [16,18,24,26].

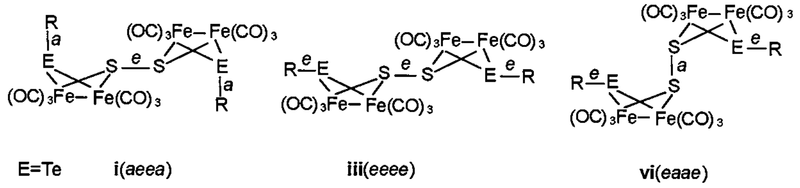
2.2. Reactions of [(μ-RTe)(μ-CO)Fe$_2$(CO)$_6$]$^-$ (3) with S$_2$Cl$_2$ leading to [(μ-RTe)Fe$_2$(CO)$_6$]$_2$(μ-S–S-μ) (14–17; R = Et, i-Bu, Ph, p-MeC$_6$H$_4$)

We previously reported [12] that anions [(μ-RE)(μ-CO)Fe$_2$(CO)$_6$]$^-$ (**1**, **2**; E = S, Se) as their [Et$_3$NH]$^+$ salts could react with S$_2$Cl$_2$ to give double clusters [(μ-RE) Fe$_2$(CO)$_6$]$_2$ (μ-S–S-μ) (E = S, Se). In order to systematically develop the chemistry of anions **1**–**3**, we further studied this type of reactions by using anions **3** (E = Te) to see if similar results could be obtained. In fact, it was found that anions **3** (R = Et, i-Bu, Ph, p-MeC$_6$H$_4$) as their [MgBr]$^+$ salts, prepared by an insertion reaction of elemental Te with Grignard reagents RMgBr followed by treatment of the intermediates RTeMgBr with Fe$_3$(CO)$_{12}$ [20], were indeed reacted with S$_2$Cl$_2$ under similar conditions to give a series of corresponding RTe-containing double butterfly clusters **14**–**17** (Scheme 2).

It follows that anion **3**, as compared with anions **1** and **2**, shows similar chemical behavior towards electrophile S$_2$Cl$_2$. Apparently, double clusters **14**–**17**, just like their S/Se analogs [12] might be rationalized as produced via intermediates *n* formed by nucleophilic attack of the negatively charged Fe atom in **3** at two S atoms in S$_2$Cl$_2$, followed by the intramolecular displacement of two μ-CO ligands by the S–S moiety in *n* (Scheme 2).

It is worth noting that in the preparations of **14**–**17** the corresponding single clusters of type (μ-RE)$_2$Fe$_2$(CO)$_6$ (E = Te) were also yielded along with double clusters **14**–**17**. In fact, single clusters of type (μ-RE)$_2$Fe$_2$(CO)$_6$ (E = S, Se, Te) were often observed as by-products in other reactions involved anions **1**–**3** [2,12,13,16]. In the reactions of **3**·[MgBr]$^+$ with S$_2$Cl$_2$, **14**–**17** were produced in 11%–14% yields along with (μ-RTe)$_2$Fe$_2$(CO)$_6$ (R = Et, i-Bu, Ph p-MeC$_6$H$_4$) in 17%–44% yields. The quite high yields for (μ-RTe)$_2$Fe$_2$(CO)$_6$ are apparently due to serious decomposition of anion **3** to give fragments (μ-RTe)Fe(CO)$_3$ and subsequent dimerization [3,12,13,16].

While these single clusters (μ-RTe)$_2$Fe$_2$(CO)$_6$(R = Et, i-Bu, Ph, p-MeC$_6$H$_4$) are known [3,20], clusters **14**–**17** are the first S–S bonded double Fe$_2$STe butterfly clusters, which have been characterized by combustion analysis, IR, ^{1}H-NMR and ^{125}Te-NMR spectroscopies. It should be pointed out that although double clusters of [(μ-RE)Fe$_2$(CO)$_6$]$_2$(μ-S–S-μ)(E = S, Se, Te) may have ten conformers, namely i(*aeea*), ii(*aeee*), iii(*eeee*), iv(*eaea*), v(*eaee*), vi(*eaae*), vii(*aaaa*), viii(*aaae*), ix(*aaee*) and x(*aaea*), according to the axial and/or equatorial orientations of the S–S and R–E bonds to the butterfly Fe$_2$SE subcluster core [12], only six conformers of i(*aeea*)–vi(*eaae*) could actually exist since conformers of vii(*aaaa*)–x(*aaea*) all involve the strong steric repulsions between two axially bonded R and subcluster core (μ-RE)(μ-S)Fe$_2$(CO)$_6$ [25,26]. In fact, double clusters **14**–**17** have proved by ^{1}H-NMR and ^{125}Te-NMR spectroscopies to be present as only one of the three conformers i(*aeea*), iii(*eeee*) or vi(*eaae*), as shown in Scheme 3. This is because all these three conformers have their two R groups and two Te atoms located in



Scheme 3.

the same chemical environment and the ^{1}H-NMR spectra of **14**, **15** and **17** showed only one quartet at 2.76 ppm, one doublet at 2.75 ppm and one quartet of AA′BB′ pattern at 7.22 ppm, respectively, for the two CH_2 of two Et groups, two CH_2 of two *i*-Bu groups and two *para*-substituted benzene rings attached to bridged Te atoms. In addition, it is also because that the ^{125}Te-NMR spectra of **14**–**17** each exhibited only one signal, a singlet, respectively, at 182.52, 96.11, 309.14 or 306.17 ppm for the two bridged Te atoms attached to R groups. However, since no comparable ^{1}H- or ^{125}Te-NMR spectral data are available so far for assignment of the spatial orientations of Et, *i*-Bu, Ph and *para*-substituted benzene groups, we cannot distinguish the three conformers of double clusters **14**–**17**. Furthermore, it should be also noted that although the conformers for similar double clusters [(μ-PhTe)$Fe_2(CO)_6]_2$(μ-Te–Te-μ) (conformer *eaae*) [3] and [(μ-PhS)$Fe_2(CO)_6]_2$(μ-S–S-μ) (conformer *aeea*) [27] were characterized by X-ray diffraction techniques, the conformers for **14**–**17** have not been established by X-ray diffractions, owing to lack of the suitable single crystals.

2.3. Crystal structures of **9** and **12**

The X-ray crystal diffraction analyses have confirmed the structures of products **9** and **12**. While ORTEP plots of **9** and **12** are shown in Figs. 1 and 2, selected bond lengths and angles are given in Tables 1 and 2, respectively. As seen from Fig. 1, product **9** is a chiral double cluster complex which consists of the two different butterfly subcluster cores Fe(1)Fe(2)S(1)S(2) and Fe(3)Fe(4)S(1)Se joined to a spiro type of μ_4-S, i.e. the S(1) atom. In addition, it can be seen that each of the three COs attached to Fe atom is terminal and the two substituents Me and *p*-MeC_6H_4 on the subcluster cores are bonded to S(2) and Se atoms by an equatorial bond. Therefore, this structure is consistent with its spectroscopic data and the conformer analysis. It is noteworthy that the corresponding bond lengths and angles related to the two different subcluster cores are very similar. For example, the bond length of Fe(3)–Fe(4) (2.544(2)Å) and bond angle of Fe(3)–S(1)–Fe(4) (69.60(8)°) are only slightly longer than that of Fe(1)–Fe(2) (2.515(2) Å) and Fe(1)–S(1)–Fe(2) (68.10(8)°), respectively. In addition, the geometrical parameters for this S/Se mixed cluster **9** are also very similar to corresponding those of Fe_4S_3 and Fe_4Se_3 clusters, such as [(μ-MeS)$Fe_2(CO)_6]_2$(μ_4-S) [28] and [(μ-*p*-MeC_6H_4Se)-$Fe_2(CO)_6]_2$(μ_4-Se) [17]. More interestingly, Fig. 2 shows that the molecule of product **12** comprises two identical double butterfly structural units{(μ-*t*-BuS)(μ-S)[Fe_2-$(CO)_6]_2$(μ_4-S)}, which are bridged together by a *para*-disubstituted benzene moiety *p*-$CH_2C_6H_4CH_2$-*p*. Similar to **9**, the two substituents *t*-Bu and two CH_2 groups in the bridging *p*-$CH_2C_6H_4CH_2$-*p* group on the two double cluster cores are bound to S_3/S_3^* and S_1/S_1^* atoms by an equatorial bond and each of the three COs bonded to Fe atom is terminal. So, this structure is also in accordance with its spectroscopic data and the conformer analysis. Although the quadruple Fe/S butterfly clusters {(μ-RS)[$Fe_2(CO)_6]_2$(μ_4-S)}$_2$(μ-S–S-μ)} (R = Et, *t*-Bu) are known[23], to our knowledge, **12** is the first organic group-bridged quadruple butterfly Fe/S cluster complex prepared and crystallographically characterized so far. The geometrical parameters of **12** involved in each of the four butterfly subcluster cores Fe_2S_2 are very close to each other. For instance, in subcluster core Fe1Fe2S1S2 the bond lengths and angles are Fe1–Fe2 = 2.538(1) Å, Fe1–S2 = 2.246(2) Å, Fe2–S2 = 2.241(2) Å, Fe1–S2–Fe2 = 68.87(5)° and Fe1–S1–Fe2 = 68.24(5)°, whereas in subcluster core Fe3Fe4S2S3 those are Fe3–Fe4 = 2.523(1) Å, Fe3–S2 = 2.244(2) Å, Fe4–S2 = 2.251(2) Å, Fe3–S2–Fe4 = 68.31(5)° and Fe3–S3–Fe4 = 67.42(5)°, respectively. In fact, these geometrical parameters are also very close to those found in the single, double, triple and non-bridged quadruple

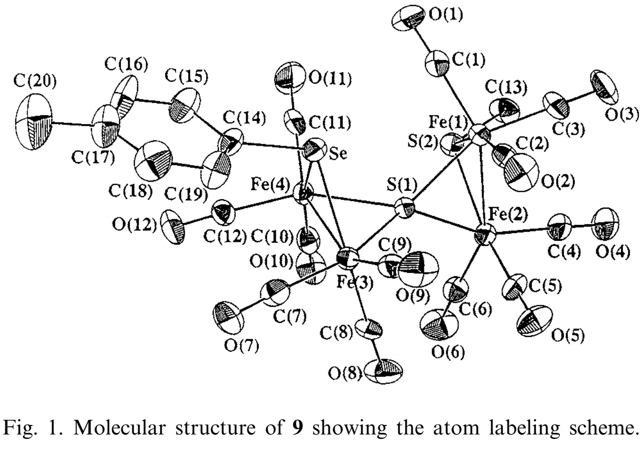


Fig. 1. Molecular structure of **9** showing the atom labeling scheme.

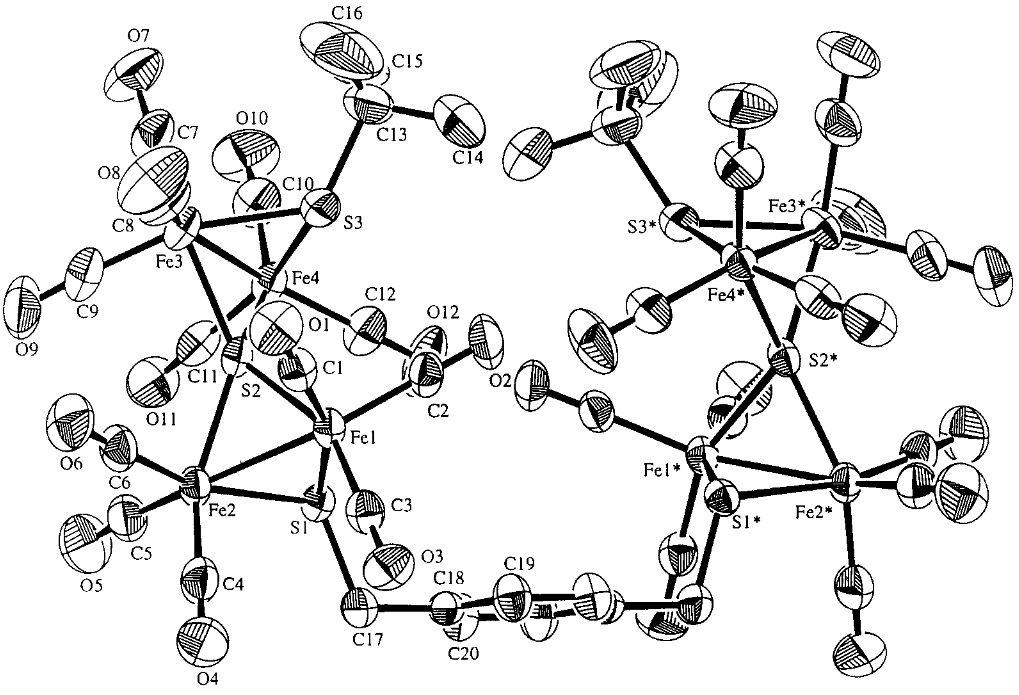


Fig. 2. Molecular structure of **12** showing the atom labeling scheme.

butterfly Fe/S cluster complexes, such as (μ-EtS)$_2$Fe$_2$(CO)$_6$ [29], [(μ-MeS)Fe$_2$(CO)$_6$]$_2$(μ$_4$-S) [28], [(μ-PhS)Fe$_2$(CO)$_6$]$_2$(μ-S–S-μ) [27], [(μ-MeS)Fe$_2$(CO)$_6$]$_2$-(μ-S–(*m*-CH$_2$C$_6$H$_4$CH$_2$-*m*)–S-μ) [30], [(μ-*t*-BuS)Fe$_2$-(CO)$_6$]$_2$(μ$_4$-S)Fe$_2$(CO)$_6$–S-μ$_4$] [22] and {(μ-*t*-BuS)[Fe$_2$-(CO)$_6$]$_2$ (μ$_4$-S)}$_2$(μ-S–S-μ)} [23].

3. Experimental

All reactions were carried out under an atmosphere of highly purified nitrogen using standard Schlenk and vacuum-line techniques. Tetrahydrofuran(THF) was distilled under nitrogen from sodium/benzophenone ketyl, triethylamine from potassium hydroxide. While S$_2$Cl$_2$ commercially available was redistilled from sulfur powder, tellurium powder, EtSH, *t*-BuSH, MeI and PhCH$_2$Br were commercially available and used without further purification. Fe$_3$(CO)$_{12}$ [31], μ-S$_2$Fe$_2$(CO)$_6$ [26], PhSeH [32], *p*-MeC$_6$H$_4$SeH [32], Grignard reagents RMgX (R = Et, *i*-Bu, Ph, *p*-MeC$_6$H$_4$) [33], *p*-(BrCH$_2$)$_2$C$_6$H$_4$ [34] and 1,3,5-(BrCH$_2$)$_3$C$_6$H$_3$ [34] were prepared according to the literature. The products were separated by TLC (20 × 25 × 0.25 cm, silica gel G) and further purified by recrystallization from mixed CH$_2$Cl$_2$–hexane solvent. IR spectra were recorded on a Nicolet 170 SX FTIR spectrophotometer. ^{1}H-NMR spectra were recorded on a Bruker AC-P200 NMR spectrometer.^{77}Se-NMR and ^{125}Te-NMR spectra were recorded on a UNITY plus-400 spectrometer with Ph$_2$Se$_2$ and Ph$_2$Te$_2$ as external standards and the chemical shifts are referenced to Me$_2$Se($\delta = 0$) and Me$_2$Te-($\delta = 0$), respectively. C/H analyses were performed on a Yanaco CHN Corder MT-3 analyzer. Melting points were determined on a Yanaco MP-500 apparatus.

Table 1
Selected bond lengths (Å) and angles (°) for **9**

Fe(1)–Fe(2)	2.515(2)	Fe(1)–S(1)	2.247(3)
Fe(1)–S(2)	2.261(3)	Fe(2)–S(1)	2.245(3)
Fe(2)–S(2)	2.265(3)	Fe(3)–S(1)	2.226(3)
Fe(3)–Fe(4)	2.544(2)	Fe(4)–S(1)	2.231(3)
Se–Fe(3)	2.391(2)	Se–Fe(4)	2.397(2)
S(1)–Fe(1)–S(2)	76.5(1)	S(1)–Fe(1)–Fe(2)	55.92(7)
Fe(3)–Se–Fe(4)	64.19(5)	S(2)–Fe(1)–Fe(2)	56.32(8)
S(1)–Fe(2)–S(2)	76.4(1)	S(1)–Fe(2)–Fe(1)	55.98(7)
S(2)–Fe(2)–Fe(1)	56.16(8)	Se–Fe(3)–Fe(4)	58.02(5)
S(1)–Fe(4)–Fe(3)	55.12(7)	S(1)–Fe(4)–Se	77.47(8)
Fe(3)–S(1)–Fe(4)	69.60(8)	S(2)–Fe(2)–Fe(1)	56.16(8)
Fe(2)–S(1)–Fe(1)	68.10(8)	Fe(1)–S(2)–Fe(2)	67.52(9)

Table 2
Selected bond lengths (Å) and angles (°) for **12**

Fe(1)–Fe(2)	2.538(1)	Fe(1)–S(2)	2.246(2)
Fe(2)–S(1)	2.255(2)	Fe(3)–S(2)	2.244(2)
Fe(4)–S(3)	2.279(2)	Fe(1)–S(1)	2.269(2)
Fe(2)–S(2)	2.241(2)	Fe(3)–Fe(4)	2.523(1)
Fe(3)–S(3)	2.268(2)	Fe(4)–S(2)	2.251(2)
Fe(1)–S(1)–Fe(2)	68.24(5)	Fe(1)–S(2)–Fe(3)	134.86(7)
Fe(2)–S(2)–Fe(3)	137.27(7)	Fe(3)–S(2)–Fe(4)	68.31(5)
Fe(1)–S(2)–Fe(2)	68.87(5)	Fe(1)–S(2)–Fe(4)	124.42(6)
Fe(2)–S(2)–Fe(4)	134.76(7)	Fe(3)–S(3)–Fe(4)	67.42(5)

3.1. Preparation of (μ-*t*-BuS)(μ-MeS))[$Fe_2(CO)_6$]$_2$(μ_4-S) (**7**)

A 100 ml two-necked flask equipped with a magnetic stir-bar, a N_2 inlet tube and a serum cap was charged with 0.504 g (1.0 mmol) of $Fe_3(CO)_{12}$, 10 ml of THF, 0.11 ml (1.0 mmol) of *t*-BuSH and 0.14 ml (1.0 mmol) of Et_3N. The mixture was stirred at room temperature (r.t.) for 0.5 h to give a yellow–brown solution. To the solution was added 0.344 g (1.0 mmol) of μ-$S_2Fe_2(CO)_6$ and the mixture was stirred at r.t. for 2 h to give a brown–green solution. To this solution was added 0.13 ml (2.0 mmol) of MeI and then the new mixture was stirred at r.t. for an additional 12 h. The resulting mixture was filtered and condensed under reduced pressure. The residue was subjected to TLC separation using petroleum ether as eluent. From the major red band was obtained 0.668 g (92%) of **7** as a red solid. M.p. 150°C (dec.). Anal. Found: C, 28.06; H, 1.67. Calc. for $C_{17}H_{12}Fe_4O_{12}S_3$: C, 28.04; H, 1.65%. IR (KBr disk): terminal C≡O, 2082vs, 2052vs, 2034vs, 1984s, 1979s, 1972s, 1967m cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.44 (s, 9H, $C(CH_3)_3$), 2.14 (s, 3H, CH_3) ppm.

3.2. Preparation of (μ-PhSe)(μ-MeS)[$Fe_2(CO)_6$]$_2$(μ_4-S) (**8**)

The flask described above was charged with 0.760 g (1.5 mmol) of $Fe_3(CO)_{12}$, 15 ml of THF, 0.16 ml (1.1 mmol) of PhSeH and 0.23 ml (1.6 mmol) of Et_3N. The mixture was stirred at r.t. for 0.5 h to give a brown solution. Upon cooling the solution to −78°C by a dry ice–acetone bath 0.344 g (1.0 mmol) of μ-$S_2Fe_2(CO)_6$ and 0.13 ml (2.0 mmol) of MeI were added. After the mixture was warmed naturally to r.t. by removal of the cooling bath, the mixture was stirred at this temperature for an additional 12 h. The resulting mixture was filtered and condensed under reduced pressure. The residue was subjected to TLC separation using CH_2Cl_2–petroleum ether (v/v, 1:20) as eluent. The first major band gave 0.236 g (53%) of (μ-PhSe)$_2$$Fe_2(CO)_6$, which was identified by comparison of its melting point and ^{1}H-NMR spectrum with those of an authentic sample [35]. The second major band afforded 0.268 g (34%) of **8** as a red solid. M.p. 150°C (dec.). Anal. Found: C, 28.80; H, 1.20. Calc. for $C_{19}H_8Fe_4O_{12}S_2Se$: C, 28.70; H, 1.01%. IR (KBr disk): terminal C≡O, 2085m, 2050s, 2035vs, 1991vs, 1969s cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 2.17 (s, 3H, CH_3), 7.22–7.38 (m, 5H, C_6H_5) ppm. ^{77}Se-NMR ($CDCl_3$, Me_2Se): δ 249.63 (s) ppm.

3.3. Preparation of (μ-*p*-MeC_6H_4Se)(μ-MeS)[$Fe_2(CO)_6$]$_2$(μ_4-S) (**9**)

The procedure for preparation of **9** is similar to that of **8**, but using 0.256 g (1.5 mmol) of μ-*p*-MeC_6H_4SeH instead of PhSeH. The first major band afforded 0.228 g (49%) of (μ-*p*-MeC_6H_4Se)$_2$$Fe_2(CO)_6$, which was identified by comparison of its melting point and ^{1}H-NMR spectrum with those of an authentic sample [12]. The second major band gave 0.380 g (47%) of **9** as a red solid. M.p. 154°C (dec.). Anal. Found: C, 29.50; H, 1.66. Calc. for $C_{20}H_{10}Fe_4O_{12}S_2Se$: C, 29.68; H, 1.24%. IR (KBr disk): terminal C≡O, 2082m, 2052s, 2032vs, 1993vs, 1970s cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 2.17 (s, 3H, SCH_3), 2.29 (s, 3H, $ArCH_3$), 7.12 (q, AA′BB′, $J = 7.5$ Hz, 4H, C_6H_4) ppm. ^{77}Se-NMR ($CDCl_3$, Me_2Se): δ 248.04 (s) ppm.

3.4. Preparation of (μ-*p*-MeC_6H_4Te)(μ-MeS)[$Fe_2(CO)_6$]$_2$(μ_4-S) (**10**)

A 100 ml two-necked flask equipped with a magnetic stir-bar, a serum cap and a reflux condenser topped with a N_2 inlet tube was charged with 0.255 g (2.0 mmol) of tellurium powder, 20 ml of THF and ca. 2.0 mmol of *p*-MeC_6H_4MgBr in THF. The mixture was stirred at reflux for 0.5 h to give a yellowish solution. After the solution was cooled to r.t., 1.00 g (2.0 mmol) of $Fe_3(CO)_{12}$ was added and the reaction mixture was stirred for 0.5 h to produce a brown solution. The solution was cooled to −78°C by a dry-ice–acetone bath and then 0.344 g (1.0 mmol) of μ-$S_2Fe_2(CO)_6$ and 0.13 ml (2.0 mmol) of MeI were added. The new mixture was warmed naturally to r.t. by removal of the cooling bath and then was stirred for an additional 12 h. The resulting mixture was filtered and condensed under reduced pressure. The residue was subjected to TLC separation using CH_2Cl_2–petroleum ether (v/v, 1:10) as eluent. The first major band afforded 0.298 g (42%) of (μ-*p*-MeC_6H_4Te)$_2$$Fe_2(CO)_6$, which was identified by comparison of its melting point and ^{1}H-NMR spectrum with those of an authentic sample [3]. The second major band gave 0.100 g (12%) of **10** as a red solid. M.p. 130°C (dec.). Anal. Found: C, 28.17; H, 1.05. Calc. for $C_{20}H_{10}Fe_4O_{12}S_2Te$: C, 28.02; H, 1.17%. IR (KBr disk): terminal C≡O, 2080m, 2048s, 2030vs, 1991s cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 2.16 (s, 3H, SCH_3), 2.31 (s, 3H, $ArCH_3$), 7.16 (q, AA′BB′, $J = 7.5$ Hz, 4H, C_6H_4) ppm.

3.5. Preparation of (μ-*p*-MeC_6H_4Se)(μ-$PhCH_2$S)[$Fe_2(CO)_6$]$_2$(μ_4-S) (**11**)

The procedure for the preparation of **11** is similar to that of **8**, but using 0.256 g (1.5 mmol) of μ-*p*-MeC_6H_4SeH and 0.24 ml (1.9 mmol) of $PhCH_2Br$ instead of PhSeH and MeI, respectively. The first major band separated by TLC using CH_2Cl_2–petroleum ether (v/v, 1:10) as eluent afforded 0.234 g (50%) of known compound (μ-*p*-MeC_6H_4Se)$_2$$Fe_2(CO)_6$ [12]. The second major band gave 0.392 g (44%) of **11** as a red solid.

M.p. 180°C (dec.). Anal. Found: C, 35.25; H, 1.86. Calc. for $C_{26}H_{14}Fe_4O_{12}S_2Se$: C, 35.28; H, 1.58%. IR (KBr disk): terminal C≡O, 2081m, 2055s, 2035vs, 2005s, 1991s, 1970s cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 2.28 (s, 3H, CH_3), 3.64 (q, AB pattern, $J = 12.5$ Hz, 2H, CH_2), 7.11 (q, AA′BB′, $J = 8.3$ Hz, 4H, C_6H_4), 7.34 (s, 5H, C_6H_5) ppm. ^{77}Se-NMR ($CDCl_3$, Me_2Se): δ 249.52 (s) ppm.

3.6. Preparation of $\{(\mu\text{-}t\text{-BuS})Fe_2(CO)_6]_2(\mu_4\text{-}S)\}_2$-$[\mu\text{-}p\text{-}(SCH_2)_2C_6H_4]$ (*12*)

The procedure for the preparation of **12** is similar to that of **7**, except that 0.106 g (0.104 mmol) of *p*-$(BrCH_2)_2C_6H_4$ was used instead of MeI and the reaction mixture after adding *p*-$(BrCH_2)_2C_6H_4$ was stirred for 24 h. CH_2Cl_2–petroleum ether (v/v, 1:20) was used as eluent. From the major band 0.330 g (54%) of **12** was obtained as a red solid. M.p. 172°C (dec.). Anal. Found: C, 31.21; H, 1.78. Calc. for $C_{40}H_{26}Fe_8O_{24}S_6$: C, 31.40; H, 1.71%. IR (KBr disk): terminal C≡O, 2083s, 2054vs, 2033vs, 1990vs, 1976vs cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.42 (s, 18H, 2 $C(CH_3)_3$), 3.60 (q, AB pattern, $J = 13.0$ Hz, 4H, $2CH_2$), 7.32 (s, 4H, C_6H_4) ppm.

3.7. Preparation of $\{(\mu\text{-}t\text{-BuS})[Fe_2(CO)_6]_2(\mu_4\text{-}S)\}_3$-$[(\mu\text{-}1,3,5\text{-}(SCH_2)_3C_6H_3]$ (*13*)

The procedure for the preparation of **13** is similar to that of **7**, except that 0.107 g (0.30 mmol) of 1,3,5-$(BrCH_2)_3C_6H_3$ was used instead of MeI and the reaction mixture after adding 1,3,5-$(BrCH_2)_3C_6H_3$ was stirred for 24 h. CH_2Cl_2–petroleum ether (v/v, 1:10) was used as eluent. From the major band 0.253 g (37%) of **13** was obtained as a red solid. M.p. 113°C (dec.). Anal. Found: C, 30.18; H, 1.49. Calc. for $C_{57}H_{36}Fe_{12}O_{36}S_9$: C, 30.35; H, 1.60%. IR (KBr disk): terminal C≡O, 2082s, 2058vs, 2032vs, 1981vs cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.43 (s, 27H, $3C(CH_3)_3$), 3.48–3.70 (m, 6H, $3CH_2$), 7.28 (s, 3H, C_6H_3) ppm.

3.8. Preparation of $[(\mu\text{-EtTe})Fe_2(CO)_6]_2(\mu\text{-}S\text{–}S\text{-}\mu)$ (*14*)

A 100 ml two-necked flask equipped with a magnetic stir-bar, a serum cap and a reflux condenser topped with a N_2 inlet tube was charged with 0.255 g (2.0 mmol) of tellurium powder, 20 ml of THF and ca. 2.0 mmol of EtMgBr in THF. The mixture was stirred at reflux for 8 h to give a light gray solution. After the solution was cooled to r.t., 1.00 g (2.0 mmol) of $Fe_3(CO)_{12}$ was added and the reaction mixture was stirred for 0.5 h to produce a brown solution. The solution was cooled to −78°C by a dry-ice–acetone bath and then 0.08 ml (1.0 mmol) of S_2Cl_2 was added. After the mixture was stirred at −78°C for 10 min, the cooling bath was removed and the mixture was naturally warmed to r.t. The mixture was continuously stirred at this temperature for 2 h and then was filtered through a ca. 5cm high bed of 200–300 mesh silica gel to remove insoluble materials. The filtrate was evaporated to dryness at reduced pressure and the residue was subjected to TLC separation using petroleum ether as eluent. The first main band gave 0.116 g (20%) of $(\mu\text{-EtTe})_2Fe_2(CO)_6$, which was identified by comparison of its IR and ^{1}H-NMR spectra with those of an authentic sample [20]. The second main band afforded 0.113 g (12%) of **14** as a red solid. M.p. 167°C (dec.). Anal. Found: C, 20.74; H, 1.17. Calc. for $C_{16}H_{10}Fe_4O_{12}S_2Te_2$: C, 20.50; H, 1.07%. IR (KBr disk): terminal C≡O, 2076s, 2049s, 2022vs, 2002s, 1982s, 1968vs cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.53 (t, $J = 6.7$ Hz, 6H, $2CH_3$), 2.76 (q, $J = 6.7$ Hz, 4H, $2CH_2$) ppm. ^{125}Te-NMR ($CDCl_3$, Me_2Te): δ 182.52 (s) ppm.

3.9. Preparation of $[(\mu\text{-}i\text{-BuTe})Fe_2(CO)_6]_2(\mu\text{-}S\text{–}S\text{-}\mu)$ (*15*)

The same procedure as that for **14** was followed, but *i*-BuMgBr was used instead of EtMgBr. From the first main band was obtained 0.107 g (16%) of $(\mu\text{-}i\text{-BuTe})_2Fe_2(CO)_6$, which was identified by comparison of its IR and ^{1}H-NMR spectra with those of an authentic sample [20]. From the second main band was obtained 0.126 g (13%) of **15** as a red solid. M.p. 157°C (dec.). Anal. Found: C, 24.45; H, 2.00%. Calc. for $C_{20}H_{18}Fe_4O_{12}S_2Te_2$: C, 24.18; H, 1.81%. IR (KBr disk): terminal C≡O, 2072s, 2046s, 2023vs, 2002s, 1980vs cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.06 (d, $J = 6.5$ Hz, 12H, $4CH_3$), 1.81–2.01 (m, 2H, 2CH), 2.75 (d, $J = 6.5$ Hz, 4H, $2CH_2$) ppm. ^{125}Te-NMR ($CDCl_3$, Me_2Te): δ 96.11 (s) ppm.

3.10. Preparation of $[(\mu\text{-PhTe})Fe_2(CO)_6]_2(\mu\text{-}S\text{–}S\text{-}\mu)$ (*16*)

The same procedure as that for **14** was followed, except that PhMgBr was used instead of EtMgBr and the mixture of PhMgBr and Te powder was refluxed for 0.5 h. The first main band gave 0.293 g (43%) of $(\mu\text{-PhTe})_2Fe_2(CO)_6$, which was identified by comparison of its IR and ^{1}H-NMR spectra with those of an authentic sample [3]. The second main band gave 0.118 g (11%) of **16** as a red solid. M.p. 188°C (dec.). Anal. Found: C, 28.07; H, 1.08%. Calc. for $C_{24}H_{10}Fe_4O_{12}S_2Te_2$: C, 27.89; H, 0.97%. IR (KBr disk): terminal C≡O, 2075s, 2052s, 2025vs, 1990vs, 1968s cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 7.24–7.43 (m, 10H, $2C_6H_5$) ppm. ^{125}Te-NMR ($CDCl_3$, Me_2Te): δ 309.14 (s) ppm.

3.11. Preparation of $[(\mu\text{-}p\text{-}MeC_6H_4Te)Fe_2(CO)_6]_2(\mu\text{-}S\text{–}S\text{-}\mu)$ (*17*)

The same procedure as that for **14** was followed, except that *p*-MeC_6H_4MgBr was used instead of

Table 3
Crystal data and structural refinements details for **9** and **12**

	9	**12**
Formula	$C_{20}H_{10}Fe_4O_{12}S_2Se$	$C_{40}H_{26}Fe_8O_{24}S_6$
Formula weight	808.76	1529.77
Crystal system	Ticlinic	Monoclinic
Space group	*P* (no. 2)	*C*2/*c* (no. 15)
Unit cell dimensions		
a (Å)	8.782(2)	33.755(7)
b (Å)	9.093(3)	12.492(2)
c (Å)	19.514(7)	15.539(3)
α (°)	86.98(2)	
β (°)	86.42(2)	116.49(1)
γ (°)	68.61(2)	
V (Å^3)	1447.5(8)	5864(2)
Z	2	4
D_{calc} (g cm^{-3})	1.855	1.733
F(000)	792	3048
μ(Mo–K$_\alpha$) (cm^{-1})	34.03	22.06
Temperature (K)	296	293
Wavelength (Å)	0.71069	0.71069
Scan type	ω–2θ	ω–2θ
2θ_{max} (°)	52.2	49.9
No. of observations (*n*)	3194	2966
No. of variables (*p*)	352	352
R	0.058	0.036
Rw	0.067	0.043
Goodness-of-fit indicator	1.60	1.43
Largest peak (e Å^{-3})	0.81	0.46

EtMgBr and the mixture of *p*-MeC_6H_4MgBr and Te powder was refluxed for 0.5 h. From the first main band was obtained 0.316 g (44%) of known compound (μ-*p*-$MeC_6H_4Te)_2Fe_2(CO)_6$ [3]. From the second main band was obtained 0.148 g (11%) of **17** as a red solid. M.p. 196°C (dec.). Anal. Found: C, 29.32; H, 1.09%. Calc. for $C_{26}H_{14}Fe_4O_{12}S_2Te_2$: C, 29.42; H, 1.32%. IR (KBr disk): terminal $C\equiv O$, 2076s, 2053s, 2026vs, 1988vs cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 2.36 (s, 6H, 2CH_3), 7.22 (q, AA′BB′, $J = 9.0$ Hz, 8H, 2C_6H_4) ppm. ^{125}Te-NMR ($CDCl_3$, Me_2Te): δ 306.17 (s) ppm.

*3.12. Single crystal structure determinations of **9** and **12***

Single crystals of **9** and **12** suitable for X-ray diffraction analyses were grown by slow evaporation of their CH_2Cl_2–hexane solutions at about 4°C. Each crystal was mounted on an Enraf-Nonius CAD4 or a Rigaku AFC5R diffractometer with a graphite monochromator with Mo–K$_\alpha$ radiation ($\lambda = 0.71069$ Å). Details of the crystal data, data collections and structure refinements are summarized in Table 3. The structures were solved by direct methods and expanded by Fourier techniques. The final refinements were accomplished by the full-matrix least-squares method with anisotropic thermal parameters for non-hydrogen atoms. The calculations for **9** and **12** were performed using the TEXSAN crystallographic software package of the Molecular Structure Corporation.

4. Supplementary material

Crystallographic data for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre, CCDC no. 144605 for **9** and CCDC no. 144606 for **12**. Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: + 44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

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