Reactions of diferrocenyl dichalcogenides with [W(CO)$_5$(THF)]: X-ray crystal structures of Fc$_2$Te$_2$ and [W$_2$(μ-SeFc)$_2$(CO)$_8$] (Fc = [Fe(η^5-C$_5$H$_5$)(η^5-C$_5$H$_4$)])

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Abstract

Treatment of [W(CO)$_5$THF] with diferrocenyl diselenide, Fc$_2$Se$_2$, yielded the novel metal–metal bonded tungsten(I) complex, [W$_2$(μ-SeFc)$_2$(CO)$_8$] (**1**: Fc = ferrocenyl, [Fe(η^5-C$_5$H$_5$)(η^5-C$_5$H$_4$)]), which was characterised by NMR and IR spectroscopy, mass spectrometry, and X-ray crystallography. The corresponding tellurium derivative could not be prepared by an analogous route. The X-ray crystal structure of Fc$_2$Te$_2$ has also been determined.
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1. Introduction

In simple cases, oxidative addition to M(0) carbonyl complexes of the Group 6 metals leads to products in which the metal has the 2+ oxidation state. Examples are the preparations of [WI$_2$(μ-I)$_2$(CO)$_8$] [1] and [WI$_2$(CO)$_3$(MeCN)$_2$] [2]. By careful control of the reaction conditions, it is, however, also possible to obtain M(I) complexes by this route. The reaction of [W(CO)$_6$] and I$_2$ has been performed using UV irradiation by Schmidt et al. in 1975. The product of the reaction was the diamagnetic complex [W$_2$(μ-I)$_2$(CO)$_8$]. Although the structure of this compound was not determined, the structure of the analogous molybdenum compound [Mo$_2$(μ-I)$_2$(CO)$_8$], prepared from the reaction of [Mo(CO)$_6$] with SiI$_4$ using UV irradiation, was

elucidated, and it was found that two-coordinate iodine atoms bridge the two metal atoms. A metal–metal bond length of 3.161 Å was observed [3].

Dinuclear M(I) compounds having other bridging ligands have also been prepared, with thiolate derivatives being best known for M = Mo due to their relevance to the active site of nitrogenase and other molybdoenzymes. Kamata et al., for example, have studied the reaction of [Mo(SBu^t)$_4$] with CO, which led to the compound [Mo$_2$(μ-SBu^t)$_2$(CO)$_8$]. This was characterised by X-ray crystallography, and found to have a metal-metal bond and Mo–S bonds of 2.923 and 2.48 Å, respectively, with distorted octahedral geometry around the Mo(I) centres [4]. The bond lengths were found to be of similar magnitude to those observed in the related compound [Mo$_2$(μ-SEt)$_2$(η-C$_5$H$_5$)$_2$(NO)$_2$] [5].

Thiolate-bridged dinuclear complexes of formula [Et$_4$N]$_2$[M$_2$(μ-SR)$_2$(CO)$_8$] (M = Mo, W; R = Ph, Bu^t, CH$_2$Ph, CH$_2$CO$_2$Et) can be synthesised by refluxing the metal hexacarbonyl with [Et$_4$N]SR in acetonitrile. These M(0) compounds were found to oxidise both

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electrochemically, in a single step to the M(I) species $[M_2(\mu\text{-}SR)_2(CO)_8]$, and also by treatment with a mild oxidant, to the solvated species $[M_2(\mu\text{-}SR)_2(CO)_6(MeCN)_2]$ [6–8]. The complex $[Mo_2(\mu\text{-}SPh)_2(CO)_6(MeCN)_2]$ can also be prepared by treatment of $[Mo(CO)_3(MeCN)_3]$ with Ph_2S_2 [9].

Other $[M_2(\mu\text{-}ER)_2(CO)_8]$ structures (with, for example, M = W, R = Me or Bu^t, E = S) have been reported [10–13], but only one compound of this type having E = Se appears to have been structurally characterised: $[Cr_2(\mu\text{-}SePh)_2(CO)_8]$ was isolated from the reaction of Ph_2Se_2 with $[Cr(CO)_6]$ [14]. The low yields obtained in this and other examples illustrate the fact that such oxidative addition reactions of R_2E_2 are notoriously complex and unreliable. Analogous compounds containing bridging tellurolate ligands seem to be unknown, although the related triply bridged species $[Mo_2(\mu\text{-}TePh)_2(\eta^5\text{-}C_5H_4Me)(CO)_4]$ was obtained from the reaction of $[Mo_2(\eta^5\text{-}C_5H_4Me)_2(CO)_4]$ with Ph_2Te_2 [15].

The incorporation of the redox-active ferrocenyl group into chalcogen-containing ligands provides a useful probe of the coordination environment of soft Lewis acids, and may potentially lead to the development of electrochemical sensors. We have previously reported the preparation and coordination chemistry of a range of neutral ferrocenyl selenide and telluride ligands [16–18]. Oxidative addition of diferrocenyl diselenide and ditelluride, Fc_2E_2 (or their substituted derivatives) to low-valent metal centres has been shown to be a viable route to transition metal complexes of ferrocenyl selenolate and tellurolate anions [19–22]. We now describe the use of this approach to synthesise the dinuclear tungsten(I) derivative $[W_2(\mu\text{-}SeFc)_2(CO)_8]$, its spectroscopic and structural characterisation, and the failure of our attempts to prepare the analogous telluride complex. The X-ray crystal structure of Fc_2Te_2 is also reported.

2. Results and discussion

A solution of $[W(CO)_5THF]$ was prepared by irradiation of $[W(CO)_6]$ in THF. After addition of diferrocenyl diselenide, Fc_2Se_2 ($Fc = [Fe(\eta^5\text{-}C_5H_5)(\eta^5\text{-}C_5H_4)]$), the solution darkened immediately to a deep green colour. After stirring at room temperature overnight, and removal of the solvent by evaporation under reduced pressure, the product was extracted using a hexane/toluene mixture, leaving a sticky black insoluble residue. Thin layer chromatography showed that only one compound was present, having an R_f value similar to that of the diselenide starting material. Recrystallisation of the product from hexane yielded dark green crystals, which were shown to be $[W_2(\mu\text{-}SeFc)_2(CO)_8]$, **1**, by ^1H and ^{13}C NMR spectroscopy, IR spectroscopy, FAB mass spectrometry and X-ray diffraction. Compound **1** is slightly air-sensitive in solution, but appears air-stable in the solid state. The preparation of **1** is summarised in Eq. (1).

(1)
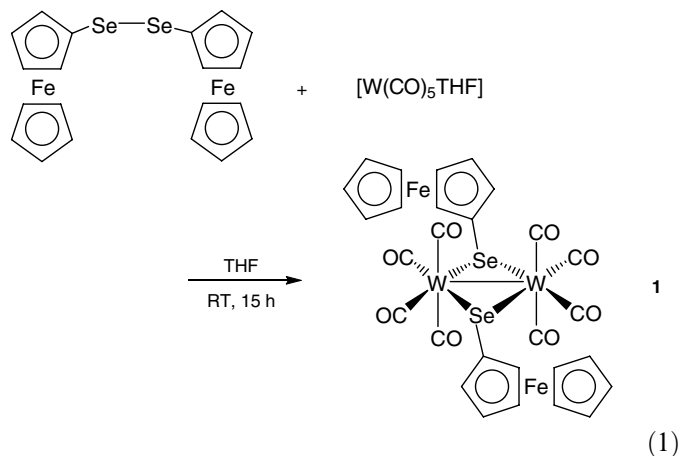
The NMR spectra of **1** were recorded in C_6D_6 solution, as the compound reacts rapidly with chlorinated solvents. The ^1H NMR spectra of complexes of this type present problems in assignment of the resonances due to the two pairs of protons on the substituted cyclopentadienyl ring. These occur as apparent triplets with chemical shifts of 4.40 and 3.80 ppm that place them either side of the singlet corresponding to the unsubstituted ring ($\delta = 4.17$ ppm). The spectrum of Fc_2Se_2 in C_6D_6 consists of apparent triplets at 4.20 ppm (H-2,5) and 3.82 ppm (H-3,4), and a singlet at 3.74 ppm. If in the spectrum of **1** we continue to assign the lowest field signal to H-2,5, this leads to the unlikely conclusion that the unsubstituted ring is most affected by the change of chemical environment. We therefore tentatively suggest the alternative assignment, i.e. that the resonances for the C_5H_5 ring, and for H-3,4 are shifted downfield with respect to Fc_2Se_2, whilst the resonance for H-2,5 is shifted *upfield*. Incidentally, it should be noted that the spectrum of Fc_2Se_2 in $CDCl_3$ is quite different [23], highlighting the importance of solvent effects in the NMR spectroscopy of this class of compound.

The same problem occurs in assigning the ^{13}C NMR spectrum. Based on the same argument, we have assigned the highest field resonance to C-2,5. No signal corresponding to C-1 (bound to the selenium atom) was observed, as is often the case in compounds of this type [16]. There are two CO resonances as expected.

The molecular ion is observed in the FAB mass spectrum, together with a succession of clusters associated with the loss of up to eight carbonyl ligands. The infrared spectrum contains two prominent bands in the C–O stretching region, one of which is very broad. A compound with *cis*-$[M(CO)_4X_2]$ geometry (C_{2v} symmetry) is expected to display four C–O bands, so two or more absorptions are presumed to overlap.

The molecular structure of **1** has been confirmed by X-ray crystallography and is shown in Fig. 1. Table 1 lists selected bond lengths and angles. The W–W distance (3.027(9) Å) is less than the sum of the van der Waals' radii, and indicative of a single bond. The comparable distances in the compounds $[W_2(\mu\text{-}SR)_2(CO)_8]$ (R = Me: 2.970 Å; R = Bu^t: 2.988 Å) [10,12] are slightly shorter, but this can
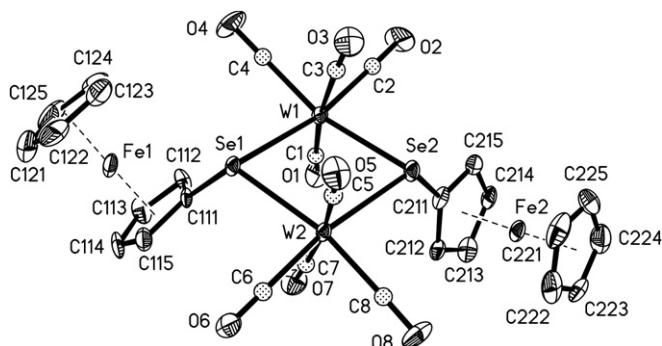
Fig. 1. Molecular structure of **1**. Hydrogen atoms are omitted for clarity.

Table 1
Selected bond lengths (Å) and angles (°) in the structure of **1**

W(1)–W(2)	3.0209(7)	W(2)–C(7)	2.013(12)
W(1)–C(1)	2.036(12)	W(2)–C(8)	2.011(9)
W(1)–C(2)	1.988(12)	W(2)–C(5)	2.021(12)
W(1)–C(3)	2.019(13)	W(2)–C(6)	2.038(11)
W(1)–C(4)	2.023(8)	W(2)–Se(1)	2.5890(10)
W(1)–Se(1)	2.5792(13)	W(2)–Se(2)	2.5800(13)
W(1)–Se(2)	2.5926(10)	Se(2)–C(211)	1.905(10)
Se(1)–C(111)	1.925(10)	Fe(2)–Cp‴	1.6423(42)
Fe(1)–Cp′	1.6489(47)	Fe(2)–Cp⁗	1.6459(39)
Fe(1)–Cp″	1.6375(42)		
C(1)–W(1)–Se(1)	96.9(3)	C(7)–W(2)–Se(1)	89.5(3)
C(2)–W(1)–Se(1)	168.3(3)	C(8)–W(2)–Se(1)	167.6(3)
C(3)–W(1)–Se(1)	90.1(3)	C(5)–W(2)–Se(1)	85.3(2)
C(4)–W(1)–Se(1)	84.1(3)	C(6)–W(2)–Se(1)	82.4(2)
C(1)–W(1)–Se(2)	91.2(2)	C(7)–W(2)–Se(2)	98.x(3)
C(2)–W(1)–Se(2)	83.8(3)	C(8)–W(2)–Se(2)	84.6(3)
C(3)–W(1)–Se(2)	83.2(3)	C(5)–W(2)–Se(2)	86.2(3)
C(4)–W(1)–Se(2)	165.9(3)	C(6)–W(2)–Se(2)	168.6(3)
Se(1)–W(1)–Se(2)	107.62(3)	Se(1)–W(2)–Se(2)	107.71(3)
C(111)–Se(1)–W(1)	111.6(3)	C(211)–Se(2)–W(1)	108.8(2)
C(111)–Se(1)–W(2)	109.4(3)	C(211)–Se(2)–W(2)	112.2(3)
W(1)–Se(1)–W(2)	71.54(3)	W(1)–Se(2)–W(1)	71.47(3)
C(112)–C(111)–Se(1)	128.5(8)	C(212)–C(211)–Se(2)	129.7(8)
C(115)–C(111)–Se(1)	121.7(8)	C(215)–C(211)–Se(2)	121.9(8)

be ascribed to the smaller size of the sulfur bridging atoms. The W–Se distance found in the cycloheptatrienyl complex [W(SePh)(η^7-C₇H₇)(CO)₂] (2.588(2) Å) [24] is almost identical to those observed in **1**, but in the mono- and dinuclear cyclopentadienyl complexes [W(SeCH₂Ph)(η^5-C₅H₅)(CO)₃] (2.623(1) Å) [25] and [W₂(μ-Se₃)(η^5-C₅H₅)₂(CO)₆] (av. 2.642 Å) [26] the W–Se distances are slightly longer. Jones and Thöne [25] have pointed out that the similarity of the W–Se bond lengths in the tungsten carbonyl complexes reported thus far, which is continued in the case of **1**, is in marked contrast to the wide range of bond lengths observed in tungsten selenide anions. The W–C bond lengths do not show any systematic variation which might reflect the different *trans*-influence of CO and SeFc ligands, and all lie within the typical range for complexes of this type. The Se–C distances (av. 1.910 Å) are typical of those for selenium bound to an sp² hybridised carbon atom; similar values are found in the ferrocenophane [Fe{η^5,η^5-(C₅H₄)₂Se₃}] (1.909(9), 1.897(9) Å) [27].

Consideration of bond angles provides further evidence of the influence of metal–metal bonding on the structure of **1**. Although few comparable ditungsten complexes have been structurally characterised, data from analogous molybdenum compounds show clearly that the M–E–M bond angles contract considerably when a metal-metal bond is present. This is accompanied by expansion of the E–M–E angles. In the Mo(I) dimer [Mo₂(μ-I)₂(CO)₈] [1], $\angle$Mo–I–Mo = 69.56°, $\angle$I–Mo–I = 110.44°; in the Mo(II) dimer [Mo₂(μ-SeCH₂C(CH₃)=CH₂)₂(η^5-C₅H₅)₂(CO)₄] [28], $\angle$Mo–Se–Mo = 99.31° (av.), $\angle$Se–Mo–Se = 69.49° (av.). The W–Se–W and Se–W–Se angles in **1** are obviously much closer to the former than the latter.

The reaction of Fc₂Te₂ with [W(CO)₅THF] appeared at first sight to proceed in analogous fashion. After stirring overnight the mixture was again a very dark colour, but thin layer chromatography revealed the presence of unreacted diferrocenyl ditelluride in addition to a new purple band assigned to the product. These could not be separated by column chromatography. Furthermore, NMR spectroscopy and mass spectrometry of the crude product showed that it was a complex mixture, and suggested that Te–C, as well as Te–Te bond cleavage had taken place. As was noted in Section 1, such observations are common in this area. Carrying out the reaction at low temperature did not significantly change the outcome.

Although the diferrocenyl dichalcogenides have been known for some time [23], the crystal structures of Fc₂S₂ and Fc₂Se₂ were only recently reported [29], and that of Fc₂Te₂ has apparently not previously been determined. Crystals of Fc₂Te₂ suitable for X-ray crystallography were grown by slow evaporation of the solvent from a solution in hexane/toluene (9:1). Like its lighter congeners, Fc₂Te₂ crystallises in the acentric space group P2₁. One of the four independent molecules in the unit cell is shown in Fig. 2.
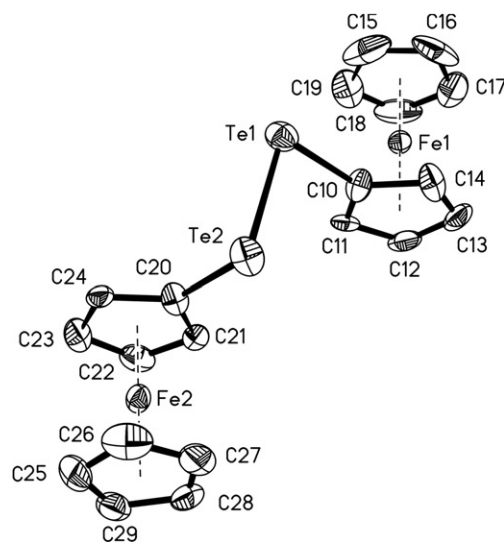

Fig. 2. Structure of one of the four independent molecules of Fc₂Te₂ in the unit cell. Hydrogen atoms are omitted for clarity.

Table 2
Selected bond lengths (Å) and angles (°) in the structure of Fc$_2$Te$_2$

Te(1)–Te(2)	2.7086(10)	Te(3)–Te(4)	2.7207(10)
Te(5)–Te(6)	2.7104(11)	Te(7)–Te(8)	2.7124(11)
Te(1)–C(10)	2.100(10)	Te(2)–C(20)	2.087(10)
Te(3)–C(30)	2.089(10)	Te(4)–C(40)	2.088(9)
Te(5)–C(50)	2.086(10)	Te(6)–C(60)	2.096(10)
Te(7)–C(70)	2.103(10)	Te(8)–C(80)	2.097(10)
C(10)–Te(1)–Te(2)	95.5(3)	C(20)–Te(2)–Te(1)	98.7(3)
C(30)–Te(3)–Te(4)	98.6(3)	C(40)–Te(4)–Te(3)	99.9(2)
C(50)–Te(5)–Te(6)	96.2(3)	C(60)–Te(6)–Te(5)	99.2(3)
C(70)–Te(7)–Te(8)	96.0(2)	C(80)–Te(8)–Te(7)	99.1(3)
C(11)–C(10)–Te(1)	125.6(8)	C(14)–C(10)–Te(1)	127.0(8)
C(21)–C(20)–Te(2)	126.2(7)	C(24)–C(20)–Te(2)	125.6(8)
C(31)–C(30)–Te(3)	123.9(8)	C(34)–C(30)–Te(3)	128.8(8)
C(41)–C(40)–Te(4)	126.4(7)	C(44)–C(40)–Te(4)	124.9(7)
C(51)–C(50)–Te(5)	124.9(8)	C(54)–C(50)–Te(5)	128.5(8)
C(61)–C(60)–Te(6)	127.0(8)	C(64)–C(60)–Te(6)	124.2(8)
C(71)–C(70)–Te(7)	126.1(8)	C(74)–C(70)–Te(7)	126.7(7)
C(81)–C(80)–Te(8)	126.8(8)	C(84)–C(80)–Te(8)	125.5(8)

Selected bond lengths and angles are listed in Table 2. The average Te–Te and Te–C bond lengths (2.713 and 2.094 Å, respectively), and C–Te–Te angles (97.9°) in Fc$_2$Te$_2$ are very similar to those in other diaryl ditellurides, Ar$_2$Te$_2$ (Ar = C$_6$H$_5$, 4-C$_6$H$_4$OMe, 2-C$_{10}$H$_7$) [30–32].

3. Experimental

All reactions were performed using standard Schlenk techniques and pre-dried solvents under an atmosphere of dinitrogen. ^{1}H and ^{13}C NMR spectra: C$_6$D$_6$ solutions; Bruker AC400; tetramethylsilane as internal standard. IR spectra: Perkin–Elmer 1725X. Mass spectra were recorded by the EPSRC Mass Spectrometry Centre, using Fast Atom Bombardment (FAB). [Fe(η^5-C$_5$H$_5$)(η^5-C$_5$H$_4$E)]$_2$ (E = Se, Te) were prepared from [Fe(η^5-C$_5$H$_5$)$_2$] via lithiation in THF with 0.9 equiv of LiBu^t, treatment with excess selenium or tellurium, and air oxidation, in a minor modification of a literature procedure [23,33]. [W(CO)$_5$(THF)] was prepared as described in the literature [34].

3.1. Preparation of [W$_2$(μ-SeFc)$_2$(CO)$_8$] (1)

Addition of Fc$_2$E$_2$ (90 mg, 0.17 mmol) to a solution of [W(CO)$_5$(THF)] (130 mg, 0.36 mmol) in THF (50 cm^3) at room temperature led immediately to a deepening of the colour from orange to an apparent black, which upon closer inspection was revealed to be dark green. After stirring overnight, the solution was reduced to dryness by evaporation of the solvent under reduced pressure, and the residue then extracted with hexane/toluene (1:1). The solution was

Table 3
Crystal data and details of data collection and refinement for 1 and Fc$_2$Te$_2$

Compound	1	Fc$_2$Te$_2$
Empirical formula	C$_{28}$H$_{18}$Fe$_2$O$_8$Se$_2$W$_2$	C$_{20}$H$_{18}$Fe$_2$Te$_2$
Formula weight	1119.74	625.24
Temperature	173(2) K	173(2) K
Wavelength	0.71073 Å	0.71073 Å
Crystal system	Orthorhombic	Monoclinic
Space group	Pbca	P2$_1$
Unit cell dimensions	a = 11.011(4) Å	a = 18.579(2) Å
	b = 14.212(3) Å	b = 10.3413(16) Å
	c = 38.469(9) Å	c = 20.037(2) Å
	β = 90°	β = 92.722(8)°
Volume	6020(3) Å^3	3845.4(9) Å^3
Z	8	8
Density (calculated)	2.471 Mg m^{-3}	2.160 Mg m^{-3}
Absorption coefficient (mm^{-1})	11.029	4.481
F(0 0 0)	4144	2352
Crystal size (mm^3)	0.38 × 0.20 × 0.10	0.32 × 0.16 × 0.10
θ Range for data collection	3.06–25.00°	3.00–25.00°
Index ranges	−13 ⩽ h ⩽ 13	−22 ⩽ h ⩽ 13
	−16 ⩽ k ⩽ 8	−12 ⩽ k ⩽ 11
	−45 ⩽ l ⩽ 5	−23 ⩽ l ⩽ 23
Reflections collected	14,441	13,969
Independent reflections	5299 [R_{int} = 0.0967]	12,358 [R_{int} = 0.0471]
Completeness to θ = 25.00°	99.8%	99.7%
Absorption correction	Ψ-Scans	Ψ-Scans
Maximum and minimum transmission	0.961 and 0.378	0.91 and 0.66
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	5299/160/339	12,358/641/865
Goodness-of-fit on F^2	0.854	0.782
Final R indices [$I > 2\sigma(I)$]	R_1 = 0.0406, wR_2 = 0.0660	R_1 = 0.0406, wR_2 = 0.057
R Indices (all data)	R_1 = 0.0774, wR_2 = 0.0729	R_1 = 0.0701, wR_2 = 0.0629
Absolute structure parameter	–	−0.05(3)
Largest difference in peak and hole	1.741 and −2.006 e Å^{-3}	0.694 and −0.587 e Å^{-3}

filtered to remove a sticky black insoluble residue, and the solvent removed in vacuo. The product was recrystallised from hexane at −10 °C.

Yield: 70 mg (37%, based on Fc_2Se_2). Mp: 85–90 °C (dec.). ^{1}H NMR (C_6D_6): $\delta = 4.40$ (at, 2H, C_5H_4, H-3,4), 4.17 (s, 5H, C_5H_5), 3.80 (at, 2H, C_5H_4, H-2,5) ppm. ^{13}C NMR (C_6D_6): $\delta = 204.1$ (CO), 191.2 (CO), 75.6 (C_5H_4, H-3,4), 71.8 (C_5H_5), 70.0 (C_5H_4, H-2,5) ppm. MS (FAB, [80]Se, [184]W): $m/e = 1122$ (M^+, 67%), 1010 ($M^+ − 4CO$, 25%), 982 ($M^+ − 5CO$, 100%), 954 ($M^+ − 6CO$, 35%), 898 ($M^+ − 8CO$, 63%), 833 ($M^+ − 8CO, C_5H_5$, 45%). IR (KBr disk): $v(CO) = 2033$ (s), 1950 (br, vs) cm^{-1}.

3.2. X-ray crystallography

Details of the data collections and refinements are summarised in Table 3.

Crystallographic data (excluding structure factors) for the structures of **1** and Fc_2Te_2 have been deposited with the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk), and are available on request quoting the deposition numbers CCDC 610278 and 610279.

Acknowledgements

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