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Synthesis and structural characterisation of a new methylene bridged double butterfly shaped complex $[(\mu\text{-}CH_3Te)Fe_2(CO)_6]_2[\mu\text{-}Te(CH_2)Te\text{-}\mu]$

Pradeep Mathur *[a], V. Dayal Reddy [a], Kalyan Das [b] and Umesh C. Sinha [b]

[a] *Department of Chemistry and* [b] *Department of Physics, Indian Institute of Technology, Powai, Bombay 400 076 (India)*

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Abstract

The new methylene bridged compound $[(\mu\text{-}CH_3Te)Fe_2(CO)_6]_2[\mu\text{-}Te(CH_2)Te\text{-}\mu]$ has been prepared from the reaction at room temperature of $Fe_3(CO)_9(\mu_3\text{-}Te)_2$ and excess diazomethane. The compound was subjected to X-ray structural analysis. The crystals were monoclinic, space group $C2/c$, $a = 28.865(10)$, $b = 13.787(7)$, $c = 14.733(4)$ A, $\beta = 104.67(2)°$, $U = 5672$ A^3, $Z = 8$, $D_c = 2.637$ g cm^{-3}. The structure consists of two '$(CO)_6Fe_2Te(CH_3)Te$' units connected by a CH_2 group.

Introduction

Bridging single atom ligands derived from the main groups of the Periodic Table play an important role in the synthesis and stabilisation of transition metal complexes [1]. The dinuclear compound $Fe_2(CO)_6(\mu\text{-}Te_2)$ (I) has been used as a convenient starting material for the preparation of several mixed metal complexes [2–5]. Addition across the Te–Te bond of I occurs readily, thereby relieving strain in the FeTeTeFe ring system of I. For instance, I reacts with $Ru(CO)_4(C_2H_4)$ to form $Fe_2Ru(CO)_9(\mu_3\text{-}Te)_2$ and $Fe_2Ru_2(CO)_{11}(\mu_4\text{-}Te)_2$, and with $Fe(CO)_6$ to form $Fe_3(CO)_9(\mu_3\text{-}Te)_2$ (II) and $Fe_4(CO)_{11}(\mu_4\text{-}Te)_2$ [6]. Although II has also been shown to add coordinatively unsaturated metal fragments, eg. with $Ru(CO)_4(C_2H_4)$ it forms $Fe_3Ru(CO)_{11}(\mu_4\text{-}Te)_2$, it is a far more inert molecule towards such additions than I. With diazomethane I readily inserts a methylene group into the Te–Te bond to give $(CO)_6Fe_2(\mu\text{-}TeCH_2Te)$ [7]. When $Fe_3(CO)_9(\mu_3\text{-}Te)_2$ is treated with diazomethane, $(CO)_6Fe_2(\mu\text{-}TeCH_2Te)$ and $Fe_2(CO)_6Te_2(CH_2)_2$ are formed [8]. Here, we report an unusual product obtained when II was reacted with a large excess of diazomethane.

Results and discussion

Room temperature stirring of a diethylether solution containing II and a large excess of diazomethane for 2 hours formed a new compound, the bridged double

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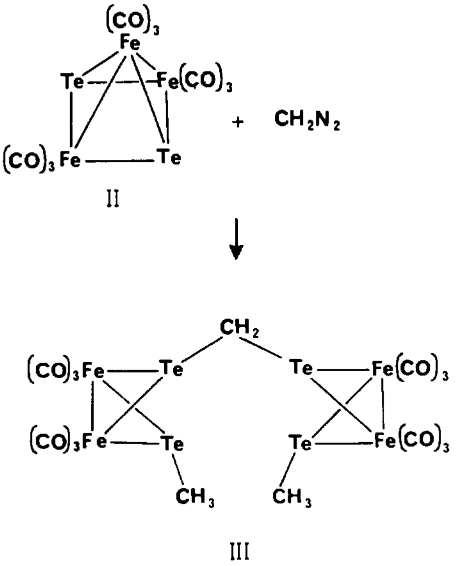
Table 1

Atomic coordinates ($\times 10^4$) and equivalent temperature factors U_{eq} (Å^2) for non-H atoms with esd's in parentheses

Atom	*x*	*y*	*z*	U_{eq} ^{a}
Te(1)	9383(1)	500(2)	1943(2)	42(3)
Te(2)	8334(1)	−371(2)	1969(2)	46(3)
Fe(1)	9064(1)	5(3)	3329(2)	22(4)
Fe(2)	8643(2)	1375(4)	2181(3)	44(6)
C(1)	10000(0)	−8591(33)	2500(0)	39(23)
C(2)	7721(12)	−455(36)	2551(26)	77(22)
C(11)	8704(9)	60(31)	4088(17)	49(21)
O(11)	8454(8)	82(31)	4630(16)	97(20)
C(12)	9507(10)	641(24)	4097(21)	47(21)
O(12)	9824(9)	1063(21)	4654(18)	72(21)
C(13)	9288(12)	−1177(15)	3547(26)	82(23)
O(13)	9420(12)	−2011(16)	3669(22)	92(22)
C(21)	8994(12)	2331(24)	2764(26)	60(23)
O(21)	9218(10)	2992(22)	3199(21)	86(22)
C(22)	8434(12)	1805(25)	1026(15)	55(22)
O(22)	8277(13)	2155(23)	258(17)	108(23)
C(23)	8152(16)	1710(35)	2647(29)	77(23)
O(23)	7837(12)	1959(34)	2945(32)	153(23)
Te(3)	9383(1)	−4479(2)	2441(2)	41(3)
Te(4)	8338(1)	−5383(3)	1362(2)	46(3)
Fe(3)	9064(1)	−5014(3)	734(2)	21(4)
Fe(4)	8642(2)	−3637(4)	1438(3)	45(6)
C(3)	10000(0)	−3578(36)	2500(0)	41(23)
C(4)	7730(12)	−5548(35)	183(25)	71(22)
C(31)	9504(15)	−4308(36)	407(22)	79(23)
O(31)	9813(9)	−3872(22)	164(19)	74(21)
C(32)	9324(12)	−6165(35)	756(27)	71(22)
O(32)	9447(11)	−6985(26)	774(23)	98(22)
C(33)	8697(9)	−5008(30)	−386(17)	49(21)
O(33)	8446(9)	−4941(28)	−1174(15)	87(20)
C(41)	8992(11)	−2669(19)	1215(24)	51(22)
O(41)	9213(9)	−2007(18)	1005(21)	77(21)
C(42)	8166(10)	−3357(32)	465(21)	70(23)
O(42)	7847(11)	−3121(33)	−207(23)	140(24)
C(43)	8394(15)	−3217(29)	2337(23)	77(23)
O(43)	8265(11)	−2833(24)	2964(20)	96(22)

butterfly Fe–Te complex, $[(\mu\text{-CH}_3\text{Te})\text{Fe}_2(\text{CO})_6]_2[\mu\text{-Te}(\text{CH}_2)\text{Te-}\mu]$ (III), in addition to trace amounts of the previously reported $(\text{CO})_6\text{Fe}_2(\mu\text{-TeCH}_2\text{Te})$ and $\text{Fe}_2(\text{CO})_6\text{Te}_2(\text{CH}_2)_2$ (Scheme 1). Due to their novel structures, unique properties and their potential use as model compounds of the iron–molybdenum cofactor of nitrogenase, bridged double cluster core complexes have attracted much attention in recent times, and have generally been prepared from the reactions of the monoanions $(\text{CO})_6\text{Fe}_2(\mu\text{-SR})(\mu\text{-S})^-$ with diacid chlorides or from the reaction of $(\text{CO})_6\text{Fe}_2(\mu\text{-S}_2)$ with BrMgRMgBr reagents [9]. Compound III has been characterised by IR and NMR spectroscopy and by elemental analysis. The IR spectrum in hexane exhibits only terminal CO stretching frequencies at 2059(m), 2053(m), 2027(vs), 1988(s) and 1981(s) cm^{-1}. ^{1}H NMR spectrum in CDCl_3 shows signals at δ



Scheme 1

2.2 (s, 6H, 2CH$_3$, ^2J(H–Te) 7 Hz) and δ 3.5 (s, 2H, CH$_2$, ^2J(H–Te) 12 Hz). The methylene protons observed at δ 3.5 are outside the δ 1–3 region given by Herrmann for the ^{1}H NMR chemical shift of μ-C(H)R moieties that are unsupported by metal–metal bonds [10]. However, recently compounds containing methylene groups unsupported by metal–metal bonds and having chemical shifts outside the Herrmann range have been reported; δ 3.5 for (MeTe)$_2$CH$_2$ [11], δ 3.5 for [Pt$_2$-μ-{o-C$_6$H$_4$P(Ph)CH$_2$CH$_2$PPh$_2$}$_2$(μ-CH$_2$)] [12] and at δ 3.7 for (C$_5$Me$_5$)$_2$Mo$_2$(CO)$_4$(μ-CH$_2$)(CH$_2$N$_2$) [13]. Elemental analysis confirms the molecular formula [(μ-CH$_3$Te)Fe$_2$(CO)$_6$]$_2$[μ-Te(CH$_2$)Te-μ]. Found: C, 16.12; H, 0.72. $C_{15}H_8O_{12}Fe_4Te_4$ calc.: C, 16.25; H, 0.94%. Overall, the formation of III involves the loss of a ‘Fe(CO)$_3$’ group from II, addition of a methyl group to one of the Te atoms and coupling of two resulting ‘Fe$_2$(CO)$_6$Te(CH$_3$)Te’ units through a CH$_2$ group. The relatively large size of tellurium prevents any metal–metal bond formation between the two ‘Fe$_2$Te$_2$ cores.

Black, brick shaped crystals of III were obtained by slow evaporation of hexane solution at −5 °C, and an X-ray analysis was undertaken. The molecular structure of III is shown in Fig. 1. The core geometry of III consists of two Fe$_2$Te$_2$ butterfly units; in each butterfly unit, the tellurium atoms are at the ‘wing tips’. One tellurium ligand on each unit is bonded to a methyl group, whereas the second tellurium ligand on each unit is bonded to a methyne group, which serves as a bridge between the two ‘Fe$_2$Te$_2$’ butterfly cores. The Fe–Fe bond, the bridging tellurium atoms and the three terminally bonded carbonyl groups define a distorted octahedral geometry around each iron atom. The average Te–Te distance in III, 3.523(3) Å is considerably longer than the average Te–Te nonbonding distance of 3.114(1) Å observed in (CO)$_6$Fe$_2$(μ-TeCH$_2$Te) [8], indicating a greater opening of the Fe$_2$Te$_2$ butterfly in III. This is also reflected in the higher average Te–Fe–Te bond angle in III, ca. 79°, than in (CO)$_6$Fe$_2$(μ-TeCH$_2$Te) and in (CO)$_6$Fe$_2$(μ$_3$-

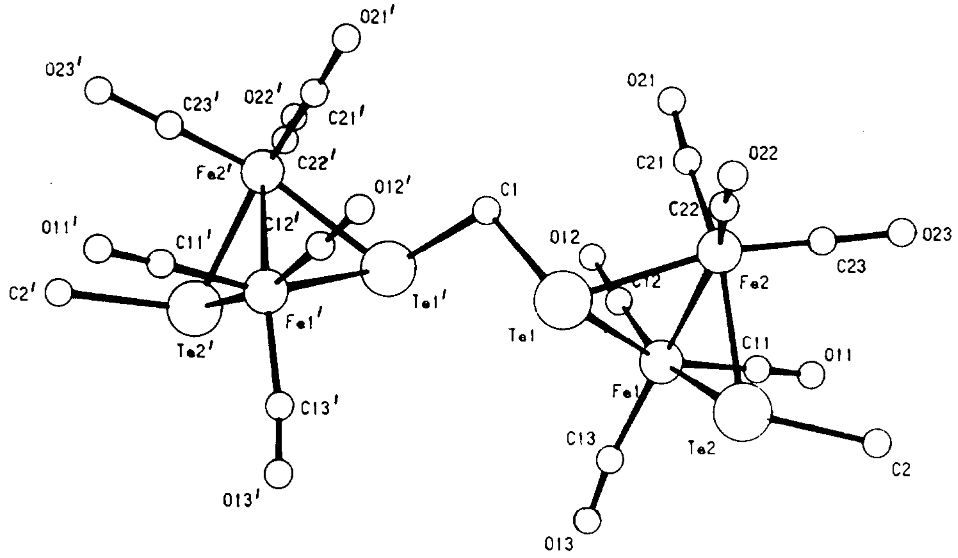


Fig. 1. Crystal structure of $[(\mu\text{-}CH_3Te)Fe_2(CO)_6]_2[\mu\text{-}Te(CH_2)Te\text{-}\mu]$.

$Te)_2Fe(CO)_3(PPh_3)$, ca. 75° [5]. In fact, the extent of opening of the Fe_2Te_2 butterfly in III is similar to that observed in $Fe_2Ru_3(CO)_{17}(\mu_3\text{-}Te)(\mu_4\text{-}Te)$, ca. 79°, which has been obtained by the insertion of '$Ru_3(CO)_{11}$' into the Te–Te bond of $Fe_2(CO)_6(\mu\text{-}Te_2)$ [2], but is less than the average S–Fe–S angle in the related molecule, $[(\mu\text{-}CH_3S)Fe_2(CO)_6]_2[\mu\text{-}S(m\text{-}CH_2C_6H_4CH_2)S\text{-}\mu]$, ca. 81° [9]. The average Fe–Fe bond distance in III, 2.613(7) Å is slightly more than the Fe–Fe bond distances of 2.587(2) and 2.585(1) Å in $(CO)_6Fe_2(\mu\text{-}TeCH_2Te)$ and $(CO)_6Fe_2(\mu_3\text{-}Te)_2Fe(CO)_3(PPh_3)$, respectively, but shorter than the Fe–Fe bond distance of 2.650(2) Å in $Fe_2Ru_3(CO)_{17}(\mu_3\text{-}Te)(\mu_4\text{-}Te)$. It is considerably longer than the average Fe–Fe bond distance in $[(\mu\text{-}CH_3S)Fe_2(CO)_6]_2[\mu\text{-}S(m\text{-}CH_2C_6H_4CH_2)S\text{-}\mu]$, 2.510(10 Å.

Assuming that the tellurium ligands contribute three electrons to each 'Fe_2' unit, compound III satisfies the eighteen electron rule. $Fe_3(CO)_9(\mu_3\text{-}Te)_2$ is known to react with '$M(PPh_3)_2$' fragments in polar solvents to form $(CO)_6Fe_2(\mu_3\text{-}Te)_2M(PPh_3)_2$ via an initial formation of $Fe_2(CO)_6(\mu\text{-}Te_2)$. In the presence of triphenylphosphine, it is known to form the adduct, $(CO)_6Fe_2(\mu_34\text{-}Te)_2Fe(CO)_3(PPh_3)$ which readily undergoes substitution of '$Fe(CO)_3PPh_3$' group by '$M(PPh_3)_2$' groups to form $(CO)_6Fe_2(\mu_3\text{-}Te)_2M(PPh_3)_2$ [4]. The formation of III is unusual in that it does not involve an initial formation of $Fe_2(CO)_6(\mu\text{-}Te)_2$ or $(CO)_6Fe_2(\mu_3\text{-}Te)_2Fe(CO)_3(PPh_3)$, because when these compounds were treated with diazomethane, formation of III was not observed.

Experimental

All operations were carried out under pure argon with the use of standard Schlenk techniques. The solvents were dried, and distilled under inert atmosphere immediately before use. The compound $Fe_3(CO)_9(\mu_3\text{-}Te)_2$ was prepared as previously reported [14]. Diazomethane was prepared according to standard procedures

Table 2

Bond distances (Å) with esd's in parenthesis

Fe(1)–Te(1)	2.536(4)	C(3)–Te(3)	2.158(29)
Fe(2)–Te(1)	2.556(5)	C(22)–Fe(2)	1.759(17)
C(1)–Te(1)	2.156(27)	Fe(3)–Te(4)	2.548(4)
Fe(4)–Te(4)	2.556(6)	C(41)–Fe(4)	1.755(16)
Fe(1)–Te(2)	2.567(4)	C(4)–Te(4)	2.150(34)
Fe(2)–Te(2)	2.556(6)	C(42)–Fe(4)	1.759(17)
C(2)–Te(2)	2.157(31)	O(32)–C(32)	1.203(48)
Fe(4)–Fe(3)	2.607(6)	Fe(4)–Te(3)	2.550(5)
C(31)–Fe(3)	1.760(37)	Fe(3)–Te(3)	2.558(4)
Fe(2)–Fe(1)	2.619(7)	C(32)–Fe(3)	1.738(40)
C(11)–Fe(1)	1.711(24)	C(33)–Fe(3)	1.719(25)
C(12)–Fe(1)	1.719(26)	C(13)–Fe(1)	1.754(17)
C(21)–Fe(2)	1.748(31)	C(43)–Fe(4)	1.756(16)
C(23)–Fe(2)	1.787(40)	O(31)–C(31)	1.201(40)
O(11)–C(11)	1.205(18)	O(33)–C(33)	1.207(18)
O(12)–C(12)	1.213(18)	O(41)–C(41)	1.198(17)
O(13)–C(13)	1.207(17)	O(42)–C(42)	1.210(70)
O(21)–C(21)	1.204(18)	O(23)–C(23)	1.155(45)
O(22)–C(22)	1.210(17)	O(43)–C(43)	1.204(17)

[15] using *N*-methyl-*N*-nitroso-*p*-toluenesulfonamide (Aldrich Chemical Co.). Infrared spectra were recorded on a Nicolet 5-DXB FT infrared spectrometer using sodium chloride solution cells of 0.1 mm path length. NMR spectra were recorded on a Bruker-80 FT-NMR spectrometer as $CDCl_3$ solutions using TMS as internal standard. Microanalysis was performed on a Carlo–Erba automatic analyser.

Crystallographic analysis

A black crystal, 0.8 × 0.14 × 0.4 mm, was used for intensity data collection using Nicolet *R*3*m*/*V* four-circle diffractometer and graphite monochromated Mo-K_α ($\lambda = 0.7107$ Å) radiation. The cell parameters, refined from 25 reflections, are $a = 28.865(10)$, $b = 13.787(7)$, $c = 14.733(4)$ Å, $\beta = 104.67(2)°$ and $V = 5671.82$ Å^3. The compound crystallised according to the monoclinic crystal system, space group = $C2/c$, $Z = 8$, $F(000) = 4096$, μ(Mo-K_α) = 58.3 cm^{-1}. $D_x = 2.637$. Empirical absorption correction was applied with minimum and maximum transmission factors 0.371 and 0.298 respectively. The structure was solved by a combination of direct methods using MULTAN88 [16] and Fourier recycling using SHELX76 [17]. Near Te and Fe atoms high peaks appeared and were considered as scattering while refining the structure. All the atoms were refined anisotropically and in the final refinement 345 parameters were refined using 2601 observed reflections. Final $R = 0.0907$, unit weights. Since the final difference map shows high positive and negative peaks, with $(\Delta\rho)_{max} = 2.18$ and $(\Delta\rho)_{min} = -1.58$ e Å^{-3}, H atoms could not be located. The atomic scattering factors for Te and Fe have been taken from *International Tables for X-ray Crystallography*, Vol. IV and those of others from SHELX76. Instead of a complete molecule, two half molecules appear per asymmetric unit which are connected to the other half molecules at symmetry positions $2 - x$, y, $\frac{1}{2} - z$ with C(1) and C(3) on the *y*-axis and site occupation factor 0.5 each.

Table 3

Bond angles (°) with esd's in parentheses

Fe(2)–Te(1)–Fe(1)	61.9(2)	C(31)–Fe(3)–Te(3)	90.5(11)
C(1)–Te(1)–Fe(1)	106.4(2)	C(31)–Fe(3)–Te(4)	158.3(16)
C(1)–Te(1)–Fe(2)	108.3(8)	C(31)–Fe(3)–Fe(4)	99.0(16)
Fe(2)–Te(2)–Fe(1)	61.5(2)	C(32)–Fe(3)–C(31)	99.1(22)
C(2)–Te(2)–Fe(1)	107.2(10)	C(33)–Fe(3)–Te(3)	157.4(12)
C(2)–Te(2)–Fe(2)	107.6(12)	C(33)–Fe(3)–Te(4)	89.3(8)
Te(2)–Fe(1)–Te(1)	79.6(1)	C(33)–Fe(3)–Fe(4)	98.3(11)
Fe(2)–Fe(1)–Te(1)	59.4(1)	C(33)–Fe(3)–C(31)	92.5(15)
Fe(2)–Fe(1)–Te(2)	59.0(1)	C(33)–Fe(3)–C(32)	100.2(17)
C(11)–Fe(1)–Te(1)	156.7(12)	C(32)–Fe(3)–Te(3)	101.4(12)
C(11)–Fe(1)–Te(2)	90.2(8)	C(32)–Fe(3)–Te(4)	101.8(13)
C(11)–Fe(1)–Fe(2)	97.4(12)	C(32)–Fe(3)–Fe(4)	153.4(13)
C(12)–Fe(1)–Te(1)	91.7(11)	Te(4)–Fe(4)–Te(3)	79.9(2)
C(12)–Fe(1)–Te(2)	160.7(12)	Fe(3)–Fe(4)–Te(3)	59.5(1)
C(12)–Fe(1)–Fe(2)	101.7(12)	Fe(3)–Fe(4)–Te(4)	59.1(2)
C(12)–Fe(1)–C(11)	91.2(15)	C(41)–Fe(4)–Te(3)	90.8(12)
C(13)–Fe(1)–Te(1)	102.0(12)	C(41)–Fe(4)–Te(4)	156.4(11)
C(13)–Fe(1)–Te(2)	98.5(12)	C(41)–Fe(4)–Fe(3)	97.5(11)
C(13)–Fe(1)–Fe(2)	151.5(12)	C(42)–Fe(4)–Te(3)	160.6(16)
C(13)–Fe(1)–C(11)	100.2(18)	C(42)–Fe(4)–Te(4)	89.2(13)
C(13)–Fe(1)–C(12)	100.2(16)	C(42)–Fe(4)–Fe(3)	101.2(16)
Te(2)–Fe(2)–Te(1)	79.5(2)	C(43)–Fe(4)–C(41)	104.2(17)
Fe(1)–Fe(2)–Te(1)	58.7(1)	C(43)–Fe(4)–C(42)	98.3(20)
Fe(1)–Fe(2)–Te(2)	59.5(2)	O(31)–C(31)–Fe(3)	177.3(41)
C(21)–Fe(2)–Te(1)	91.4(12)	O(32)–C(32)–Fe(3)	171.3(37)
C(21)–Fe(2)–Te(2)	155.4(12)	O(33)–C(33)–Fe(3)	175.7(37)
C(21)–Fe(2)–Fe(1)	96.2(12)	O(41)–C(41)–Fe(4)	174.9(34)
C(22)–Fe(2)–Te(1)	96.7(12)	O(42)–C(42)–Fe(4)	177.7(37)
C(22)–Fe(2)–Te(2)	99.9(12)	C(42)–Fe(4)–C(41)	93.0(17)
C(22)–Fe(2)–Fe(1)	148.7(12)	C(43)–Fe(4)–Te(3)	99.2(13)
C(22)–Fe(2)–C(21)	103.9(17)	O(43)–C(43)–Fe(4)	173.0(39)
C(23)–Fe(2)–Te(1)	161.0(16)	C(43)–Fe(4)–Te(4)	98.7(13)
C(23)–Fe(2)–Te(2)	89.9(16)	C(43)–Fe(4)–Fe(3)	150.0(13)
C(23)–Fe(2)–Fe(1)	102.4(16)	Fe(4)–Fe(3)–Te(3)	59.2(1)
C(23)–Fe(2)–C(21)	91.9(18)	C(23)–Fe(2)–C(22)	100.6(20)
O(11)–C(11)–Fe(1)	178.3(39)	O(12)–C(12)–Fe(1)	177.8(30)
O(13)–C(13)–Fe(1)	176.5(32)	O(21)–C(21)–Fe(2)	177.2(35)
O(22)–C(22)–Fe(2)	175.1(39)	O(23)–C(23)–Fe(2)	175.9(48)
Fe(4)–Te(3)–Fe(3)	61.4(2)	Fe(4)–Fe(3)–Te(4)	59.4(2)
C(3)–Te(3)–Fe(3)	107.1(2)	C(3)–Te(3)–Fe(4)	108.8(8)
Fe(4)–Te(4)–Fe(3)	61.4(2)	C(4)–Te(4)–Fe(3)	107.8(10)
C(4)–Te(4)–Fe(4)	109.4(12)	Te(4)–Fe(3)–Te(3)	79.9(1)

Preparation of $[\{(CO)_6Fe_2TeCH_3Te\}_2CH_2]$

To a diethylether solution (100 mL) of $Fe_3(CO)_9(\mu_3\text{-}Te)_2$ (0.15 g, 0.22 mmol) was added a large excess of freshly prepared diazomethane (20 mmol) in diethylether at room temperature. The mixture was stirred for 3 hours, after which it was filtered through a Celite pad. The solvent was removed *in vacuo* and the residue was chromatographed on silica gel TLC plates. Elution with hexane separated trace amounts of orange coloured bands of $Fe_2(CO)_6Te_2(CH_2)_2$ and $Fe_2(CO)_6(TeCH_2Te)$ from the major red band of $[\{(CO)_6Fe_2TeCH_3Te\}_2CH_2]$ (yield 0.047 g, 39%).

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