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Addition of methylene groups to $Fe_3(CO)_9(\mu_3\text{-}Te)_2$

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Abstract

At room temperature, $Fe_3(CO)_9(\mu\text{-}Te)_2$ reacts with diazomethane to form $(CO)_6Fe_2(\mu\text{-}TeCH_2Te)$ (I) and $(CO)_6Fe_2Te_2(CH_2)_2$ (II). Complexes I and II have been separated by chromatography and characterised by IR and NMR spectroscopy, mass spectrometry and microanalysis. Complex I was also subjected to an X-ray diffraction study. The crystals are monoclinic, space group $P2_1/c$ with $a = 6.864(2)$, $b = 13.926(3)$, $c = 14.159(3)$ Å, $\beta = 104.43(2)°$, $V = 1310.6(5)$ Å, $Z = 4$, $D_c = 2.79$ g cm^{-3} and $R = 0.043$.

Introduction

Currently there is great interest in the synthesis and characterisation of di- and polynuclear complexes containing CH_2 groups [1]. Reactions of these species which result in the carbon–carbon and carbon–hydrogen bond formation are of particular interest [2]. Metal alkylidene complexes have been implicated as intermediates in many catalytic reactions such as Fischer–Tropsch synthesis [3], olefin metathesis [4], alkene [5], and alkyne polymerisation [6], cyclopropanation of olefins [7] and methylenation of carbonyl compounds [8]. Many compounds have been characterised in which one or more CH_2 groups bridge between homonuclear [9], heteronuclear [10] and between transition metal and main group elements [11]. Here, we report the synthesis and characterisation of two $\mu\text{-}CH_2$ containing complexes, $Fe_2(CO)_6(\mu\text{-}TeCH_2Te)$ and $Fe_2(CO)_6Te_2(CH_2)_2$, obtained from the reaction of $Fe_3(CO)_9(\mu\text{-}Te)_2$ and diazomethane. The results of the X-ray diffraction study of $Fe_2(CO)_6(\mu\text{-}TeCH_2Te)$ are also reported.

Results and discussion

Addition of diazomethane to a solution containing $Fe_3(CO)_9(\mu_3\text{-}Te)_2$ with stirring at room temperature yields two compounds which could be separated by

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chromatography on a silica gel column. The compounds were characterised by infrared and NMR spectroscopy, mass spectrometry and elemental analysis, as $(CO)_6Fe_2(\mu\text{-}TeCH_2Te)$ (I) and $(CO)_6Fe_2Te_2(CH_2)_2$ (II) (eq. 1). The synthesis and

$$Fe_3(CO)_9(\mu_3\text{-}Te)_2 + CH_2N_2 \rightarrow (CO)_6Fe_2(\mu\text{-}TeCH_2Te) + (CO)_6Fe_2Te_2(CH_2)_2 \quad (1)$$

characterisation of I, obtained from the reaction of $(CO)_6Fe_2(\mu\text{-}Te_2)$ and diazomethane have been described previously [12].

Dark red irregular crystals of I were obtained from hexane solution at −10 °C and an X-ray diffraction study was undertaken. An ORTEP diagram of the molecular structure of I is shown in Fig. 1. Different views of the molecule are shown in Figs. 2a–c. The core geometry of I can best be described as open Fe_2Te_2 tetrahedron or a butterfly in which the tellurium atoms are located at the 'wing tips'. The open edge of the Fe_2Te_2 tetrahedron is bridged symmetrically by a CH_2 group. Three terminally bonded carbonyl groups, the bridging tellurium atoms and the Fe–Fe bond define the distorted octahedral geometry of each iron atom. The Te–Te distance of 3.114 Å is within the known range of 3.06 to 3.14 Å for strong Te–Te bonding interactions [15]. The Te–Te distance of 2.712 Å in Ph_2Te_2 is considered typical of a single bond distance [13]. In I, the Te–Te distance is close to the Te–Te distances between triangular faces in $[Te_6]^{4+}$ which average 3.133 Å [15], and is slightly more than the Te–Te bond distances in Cs_2Te_5, which average 3.046 Å [14]. Valence bond analysis suggests that these intertriangular bonds have a bond order of 2/3. In the related compound, $Cp_2Mo_2FeTe_2(CO)_7$ the average Te–Te distance of 3.142 Å suggests that here also there is significant Te–Te bonding interaction [15]. When $Fe_3Te_2(CO)_9$ forms an adduct with PPh_3, $(CO)_6Fe_2(\mu_3\text{-}Te)_2Fe(CO)_3PPh_3$, the Te–Te distance contracts from 3.36 to 3.14 Å. A similar magnitude of contraction (3.30 to 3.06 Å) is observed when $Co_4Te_2(CO)_{10}$ forms an adduct with CO. An alternate view of the bonding in I is suggested by the reported compound $Rh(P_4)(PPh_3)_2Cl$ [14]. In this compound, the 14-electron $Rh(PPh_3)_2Cl$ fragment binds in an η^2-fashion to the P_4 tetrahedron. The coordinated P atoms remain

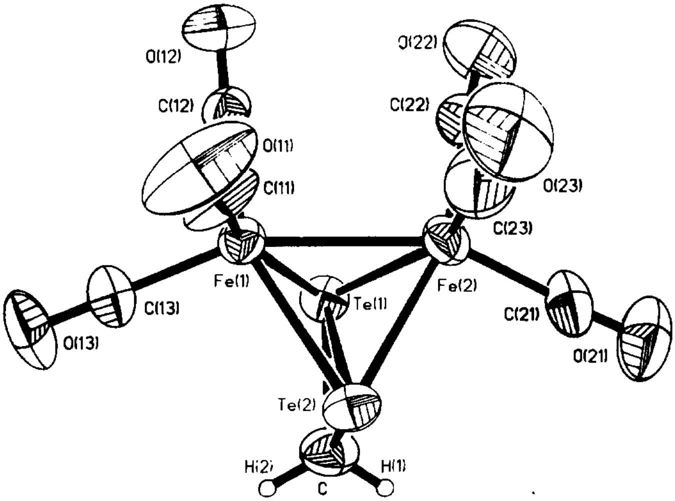

Fig. 1.

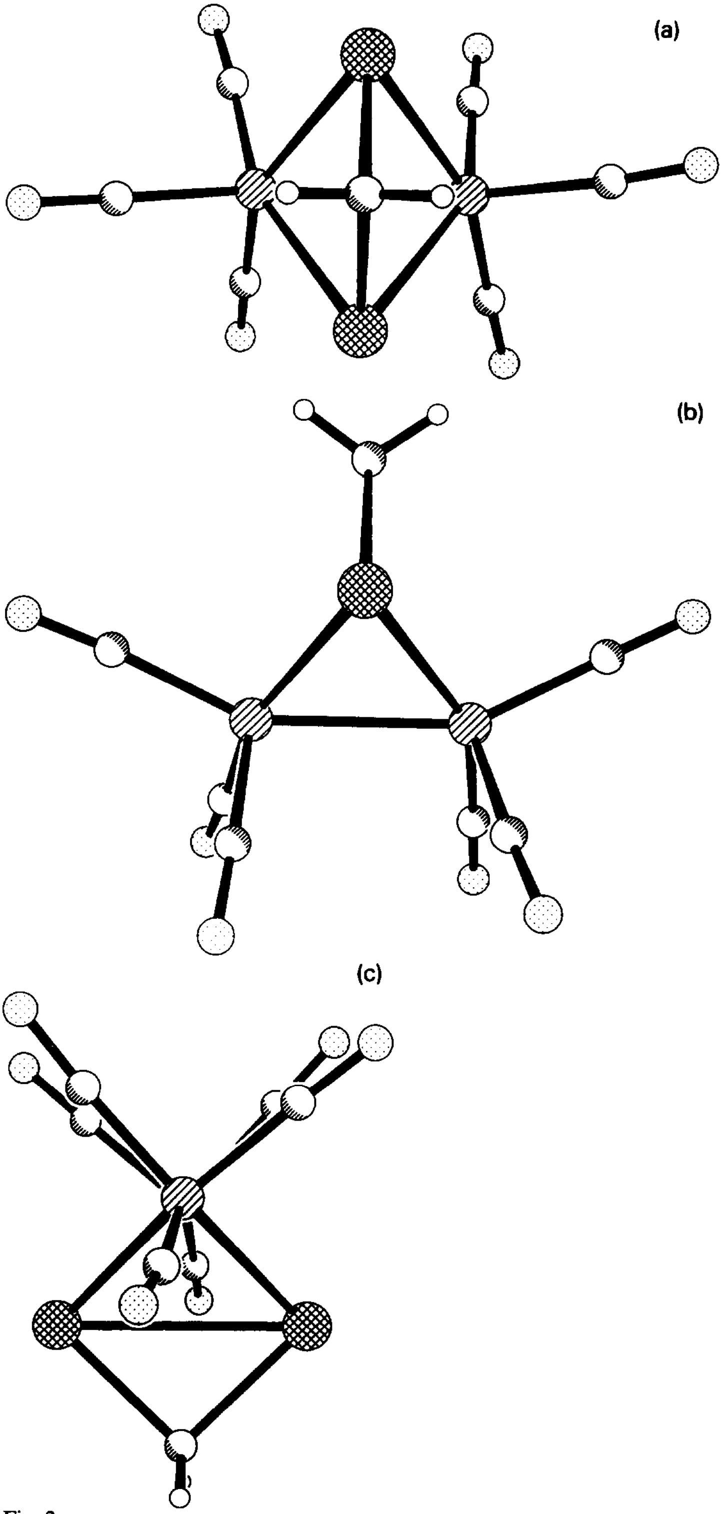

Fig. 2.

mutually bonded and the elongation of the P–P bond is analogous to that observed for olefin and acetylene coordination. In $(CO)_6Fe_2(\mu\text{-}Te_2)$, the Fe_2Te_2 core is similar structurally and electronically to P_4, and from this perspective the addition of $Fe_2(CO)_6(\mu\text{-}Te_2)$ and $Fe(CO)_3PPh_3$ may be more appropriately described as coordination of a largely-intact Te–Te bond to the $Fe(CO)_3PPh_3$ fragment. Similarly, compound I may be regarded as being derived from the coordination of a largely-intact Te–Te bond to CH_2 group.

Although the gross structural features are similar to the other members of the class of compounds of the general form $(CO)_6Fe_2(\mu\text{-EXE})$, (E = chalcogen atom, X = inserting unit) the presence of bridging methylene has some interesting consequences. For example, the Te–CH_2–Te bridging angle is only 92.1° which is slightly less than the S–CH_2–S angle of 94.6° reported for $Fe_2(CO)_6(\mu\text{-}SCH_2S)$ [16]. The Fe–Fe bond distance of 2.591 Å in I is slightly less than the Fe–Fe bond distance of 2.650 Å in the related molecule, $Fe_2Te_2(CO)_6Ru_3(CO)_{11}$ [17], but considerably less than the Fe–Fe bond distance of 2.867 Å observed in $Fe_2Ru_2(CO)_{10}(\mu\text{-}CO)(\mu_4\text{-}Te)_2$ [18]. The average Te–Fe–Te bond angle of ca 75° in I is less than the average Te–Fe–Te bond angle of ca. 79° observed in $Fe_2Te_2(CO)_6Ru_3(CO)_{11}$, which is consistent with a lesser degree of opening of the $TeFe_2Te$ 'butterfly' to accomodate the much smaller CH_2 group.

The infrared spectrum of II shows carbonyl stretching frequencies at 2055 (m), 2022 (vs) and 1984 (s) cm^{-1} in a pattern typically found for compounds containing the '$(CO)_3FeFe(CO)_3$' moiety. Its mass spectrum shows a molecular ion peak centred at *m/e* 566 and peaks at *m/e* 538, 510, 482, 454, 426 and 398 corresponding to a loss of six carbonyl groups. The spectrum also showed peaks at *m/e* 398 ($Fe_2Te_2(CH_2)_2^+$), 368 ($Fe_2Te_2^+$), 312 ($FeTe_2^+$), 240 (Fe_2Te^+), 186 ($FeTe^+$), 130 (Te^+), 112 (Fe_2^+) and 56 (Fe^+). Elemental analysis (calculated for $C_8H_4Fe_2O_6Te_2$: C 17.02%, H 0.71; found: C 17.24%, H 0.98%) confirms the molecular formula of II as $Fe_2Te_2(CO)_6(CH_2)_2$. Proton NMR spectrum shows a peak at δ 2.1 (s, 2H) with long and short range ^{125}Te satellites. Herrmann has suggested that the chemical shifts of μ-C(H)R moieties that are unsupported by a metal–metal bond fall in the δ 1–3 range [19]; therefore, it is likely that in II, the CH_2 groups are unsupported by metal–metal bonds. Though II is stable, it has not been possible to obtain X-ray quality crystals, because only thin fibres are isolated.

Experimental

All operations were carried out under pure argon or nitrogen with the use of standard Schlenk techniques. The solvents were dried, and distilled under inert atmosphere immediately before use. $Fe_3(CO)_9(\mu\text{-}Te)_2$ was prepared as previously described [20]. Diazomethane was prepared according to standard procedures [21] using *N*-methyl-*N*-*p*-toluenesulfonamide (Aldrich Chemical Co.). Infrared spectra were recorded with a Nicolet 5-DXB Fourier Transform spectrometer in sodium chloride solution cells of 0.1 mm path length. NMR spectra were recorded with a Bruker-80 FT-NMR spectrometer as $CDCl_3$ solutions using TMS as internal standard. EI mass spectra were recorded on a Kratos MS 890/DGDG-30 instrument by use of the modified direct insertion system ('in beam method') operated at an ionising voltage of 70 eV and at 200 °C. Microanalyses were performed with a Carlo-Erba automatic analyser.

Table 1

Structure determination

Crystal data	
Empirical formula	$Te_2(CH_2)Fe_2(CO)_6$
Color; habit	dark red, irregular
Crystal size (mm)	0.42 × 0.22 × 0.18
Crystal system	monoclinic
Space group	$P2_1/n$
Unit cell dimensions	$a = 6.864(2)$ Å $b = 13.926(3)$ Å $c = 14.159(3)$ Å $\beta = 104.43(2)°$
Volume	1310.6(5) Å^3
Z	4
Formula weight	549.0
Density (calc.)	2.782 cm^{-3}
Absorption coefficient	6.602 mm^{-1}
$F(000)$	992
Data collection	
Diffractometer used	Nicolet $R3m/v$
Radiation	Mo-K_α ($\lambda = 0.71073$ Å)
Temperature (K)	296
Monochromator	Highly-oriented graphite crystal
2θ range	5.0 to 50.0°
Scan type	ω
Scan speed	Variable; 14.65 to 29.30°/min in ω
Scan range (ω)	1.10°
Background measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 25.0% of total scan time
Standard reflections	3 measured every 300 reflections
Index ranges	$-8 \leqslant h \leqslant 7$; $0 \leqslant k \leqslant 16$; $0 \leqslant l \leqslant 16$
Reflections collected	2304
Independent reflections	2304 ($R_{int} = 0.00\%$)
Observed reflections	1938 ($F > 6.0\sigma(F)$)
Absorption correction	–
Solution and refinement	
System used	Nicolet SHELXTL PLUS (MicroVAX II)
Solution	Direct methods
Refinement method	Full-matrix least-squares
Quantity minimized	$\Sigma w(F_o - F_c)^2$
Absolute configuration	–
Extinction correction	$\chi = 0.00001(11)$, where $F^* = F[1 + 0.002\chi F^2/\sin(2\theta)]^{-1/4}$
Hydrogen atoms	Riding mode, fixed isotropic U
Weighting scheme	$w^{-1} = \sigma^2(F) + 0.0050F^2$
Final R indices (obs. data)	$R = 4.30\%$, $wR = 7.70\%$
R indices (all data)	$R = 5.20\%$, $wR = 9.03\%$
Goodness-of-fit	1.02
Largest and mean Δ/σ	0.004, 0.000
Data-to-parameter ratio	12.5 : 1
Largest difference peak	0.98 eÅ^{-3}
Largest difference hole	−0.96 eÅ^{-3}

Table 2

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($Å^2 \times 10^3$)

	x	y	z	U (eq) [a]
Te(1)	618(1)	8058(1)	3319(1)	41(1)
Te(2)	4698(1)	8657(1)	2906(1)	44(1)
C	3776(14)	7776(8)	3970(8)	53(4)
Fe(1)	1620(2)	9705(1)	2805(1)	38(1)
C(11)	2557(16)	10610(8)	2167(11)	66(5)
O(11)	3157(15)	11232(6)	1783(10)	107(6)
C(12)	−899(13)	10076(6)	2301(7)	40(3)
O(12)	−2510(11)	10307(5)	1962(6)	64(3)
C(13)	2151(19)	10261(8)	3991(10)	67(4)
O(13)	2497(18)	10622(8)	4720(7)	107(5)
Fe(2)	1434(2)	8235(1)	1669(1)	44(1)
C(21)	2187(20)	7039(7)	1377(8)	57(4)
O(21)	2695(19)	6365(6)	1140(8)	88(4)
C(22)	−1160(20)	8196(9)	1053(10)	74(5)
O(22)	−2797(16)	8130(8)	672(9)	111(5)
C(23)	2026(28)	8911(9)	734(11)	89(6)
O(23)	2319(22)	9340(9)	84(8)	122(6)
H(1)	4134	7113	3942	80
H(2)	4225	8018	4624	80

[a] Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

X-Ray crystal structure determination of I

Single crystal X-ray data were collected on a Nicolet $R3m/V$ diffractometer. The Fe and Te positions were located by direct methods and subsequent difference Fourier maps revealed the CO groups and the carbon of the CH_2 group. At this point an absorption correction was made with DIFABS [22]. All non-hydrogen atoms were anisotropically refined, and the hydrogen atoms were included as idealized contributions. X-Ray crystal data are given in Table 1, while the final fractional coordinates and selected bond distances and angles are listed in Tables 2, 3 and 4 respectively. Structure solution, refinement, and calculation of derived results were performed by use of the SHELXT PLUS [23] package of computer programs.

Table 3

Bond lengths (Å)

Te(1)–Te(2)	3.114(1)	Te(1)–C	2.169(9)
Te(1)–Fe(1)	2.551(2)	Te(1)–Fe(2)	2.546(2)
Te(2)–C	2.157(12)	Te(2)–Fe(1)	2.542(2)
Te(2)–Fe(2)	2.544(2)	Fe(1)–C(11)	1.763(13)
Fe(1)–C(12)	1.776(9)	Fe(1)–C(13)	1.802(13)
Fe(1)–Fe(2)	2.587(2)	C(11)–O(11)	1.153(17)
C(12)–O(12)	1.136(11)	C(13)–O(13)	1.119(16)
Fe(2)–C(21)	1.822(11)	Fe(2)–C(22)	1.778(12)
Fe(2)–C(23)	1.752(16)	C(21)–O(21)	1.083(15)
C(22)–O(22)	1.122(16)	C(23)–O(23)	1.156(21)

Table 4

Bond angles (°)

Te(2)–Te(1)–C	43.8(3)	Te(2)–Te(1)–Fe(1)	52.2(1)
C–Te(1)–Fe(1)	88.4(3)	Te(2)–Te(1)–Fe(2)	52.3(1)
C–Te(1)–Fe(2)	89.0(3)	Fe(1)–Te(1)–Fe(2)	61.0(1)
Te(1)–Te(2)–C	44.1(2)	Te(1)–Te(2)–Fe(1)	52.4(1)
C–Te(2)–Fe(1)	88.9(3)	Te(1)–Te(2)–Fe(2)	52.3(1)
C–Te(2)–Fe(2)	89.3(2)	Fe(1)–Te(2)–Fe(2)	61.1(1)
Te(1)–C–Te(2)	92.1(4)	Te(1)–Fe(1)–Te(2)	75.4(1)
Te(1)–Fe(1)–C(11)	161.3(4)	Te(2)–Fe(1)–C(11)	92.0(4)
Te(1)–Fe(1)–C(12)	94.1(3)	Te(2)–Fe(1)–C(12)	154.4(3)
C(11)–Fe(1)–C(12)	91.6(5)	Te(1)–Fe(1)–C(13)	97.3(4)
Te(2)–Fe(1)–C(13)	102.9(4)	C(11)–Fe(1)–C(13)	99.0(6)
C(12–Fe(1)–C(13)	101.5(5)	Te(1)–Fe(1)–Fe(2)	59.4(1)
Te(2)–Fe(1)–Fe(2)	59.5(1)	C(11)–Fe(1)–Fe(2)	102.4(4)
C(12)–Fe(1)–Fe(2)	95.1(3)	C(13)–Fe(1)–Fe(2)	152.5(4)
Fe(1)–C(11)–O(11)	176.8(13)	Fe(1)–C(12)–O(12)	178.8(8)
Fe(1)–C(13)–O(13)	178.5(13)	Te(1)–Fe(2)–Te(2)	75.4(1)
Te(1)–Fe(2)–Fe(1)	59.6(1)	Te(2)–Fe(2)–Fe(1)	59.4(1)
Te(1)–Fe(2)–C(21)	105.0(4)	Te(2)–Fe(2)–C(21)	96.7(4)
Fe(1)–Fe(2)–C(21)	153.1(3)	Te(1)–Fe(2)–C(22)	91.4(5)
Te(2)–Fe(2)–C(22)	161.3(5)	Fe(1)–Fe(2)–C(22)	102.6(5)
C(21)–Fe(2)–C(22)	99.6(6)	Te(1)–Fe(2)–C(23)	152.9(5)
Te(2)–Fe(2)–C(23)	93.8(5)	Fe(1)–Fe(2)–C(23)	93.5(5)
C(21)–Fe(2)–C(23)	100.8(6)	C(22)–Fe(2)–C(23)	92.2(7)
Fe(2)–C(21)–O(21)	173.6(12)	Fe(2)–C(22)–O(22)	177.1(13)
Fe(2)–C(23)–O(23)	176.3(13)		

Reaction between $Fe_3(CO)_9(\mu_3\text{-}Te)_2$ and diazomethane

An excess of diazomethane (6 mmol) in diethyl ether was added to an Et_2O (100 ml) solution of $Fe_3(CO)_9(\mu_3\text{-}Te)_2$ (0.170 g, 0.25 mmol) at room temperature. The colour of the solution changed from black to brownish-red. The reaction mixture was stirred for 2 hours, after which it was filtered through a Celite pad. Solvent was removed in vacuo and the residue subjected to chromatographic work-up on silica gel tlc plates. Elution with petroleum ether gave two orange red bands.

First band: $Fe_2Te_2(CO)_6(CH_2)_2$, yield: 13 mg (15% based on $Fe_3Te_2(CO)_9$); second band: $Fe_2Te_2(CO)_6(CH_2)$, yield: 29 mg (35% based on $Fe_3Te_2(CO)_9$), m.p.: 121–122°C.

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