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Molecular and crystal structure of $[Et_4N][Cl] \cdot 2[Fe_2(CO)_6Te_2]$

Robert E. Bachman and Kenton H. Whitmire

Department of Chemistry, Rice University, PO Box 1892, Houston, TX 77251 (USA)

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Abstract

$[Et_4N]_2[Fe_2(CO)_8]$ reacts with $TeCl_4$ to yield $Fe_2(CO)_6Te_2$. The product crystallizes in the space group $Pca2_1$ (no. 29) with two tetrahedral cluster molecules co-crystallized with one molecule of $[Et_4N]Cl$ per asymmetric unit. The presence of the chloride stabilizes the lattice and helps to create an intricate packing array through a variety of intermolecular contacts.

Key words: Iron; Tellurium; Metal clusters; Carbonyl; Chalcogenide; Group 16

1. Introduction

$Fe_2(CO)_6Te_2$, (I), was first observed by Rauchfuss and co-workers [1] while reinvestigating the earlier work of Hieber [2]. Rauchfuss was able to isolate small quantities of this material by careful column chromatography. It was found to be moderately stable in solution but decomposition was observed upon solvent removal. While the chemistry of this fundamental compound has been probed by several researchers [3-9], its structural characterization has long proved elusive. We wish to report the preparation of I via a new synthetic route and its structural characterization in a chloride ion stabilized lattice.

2. Experimental details

2.1. General

All manipulations were carried out in a nitrogen atmosphere using standard Schlenk line and glove box techniques. The solvents were dried over appropriate drying agents and distilled under nitrogen prior to use: THF (Na/Ph_2CO), hexane ($LiAlH_4$), MeCN (CaH_2), and CH_2Cl_2(CaH_2). The $[Et_4N]_2[Fe_2(CO)_8]$ was prepared according to literature procedures [10] and the $TeCl_4$ (Strem) was used as received.

2.2. Synthesis of $Fe_2(CO)_6Te_2$

2.98 g (5.0 mmol) of $[Et_4N]_2[Fe_2(CO)_8]$ was dissolved in 40 ml MeCN. A solution of 1.35 g (5.0 mmol) of $TeCl_4$ in 10 ml MeCN was added in 2 ml portions. The resulting solution was stirred for 3-4 h. The solvent was removed *in vacuo* and the residue was extracted into 50 ml CH_2Cl_2 with stirring for 30 min. The solution was filtered and reduced to half volume. An equal volume of hexane was added and the resulting mixture was cooled to -20°C for 2-4 days. A few small red plates formed along with a fine black powder. The yield of the black powder was approximately 0.7 g (46% based on tellurium). Upon standing the red crystalline material decomposed to black powder. No analytic data were obtained due to the low yield and limited stability of the crystalline material.

2.3. Structure of $[Et_4N][Cl] \cdot 2[Fe_2(CO)_6Te_2]^{(II)}$

A thin red plate of approximate dimensions $0.05 \times 0.10 \times 0.15$ mm^3 was mounted on a glass fiber with epoxy cement. The data were collected on a Rigaku AFC5 four circle automated diffractometer at a temperature of -50°C. Final unit cell parameters were based on least-squares analysis of 25 carefully centered reflections ($6.90° \leq 2\theta \leq 17.43°$). The crystallographic parameters are summarized in Table 1. The crystal was shown to be orthorhombic and the space group was chosen as $Pca2_1$ (no. 29) on the basis of systematic absences and intensity statistics. The data were cor-

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Correspondence to: Dr. K.H. Whitmire.

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TABLE 1. Crystallographic Data Collection Parameters for compound **II**

Empirical formula	$[C_8H_{20}N]Cl\text{-}2[Fe_2Te_2C_6O_6]$
FW	1215.46
Crystal system	Orthorhombic
Space group (No.)	$Pca2_1$ (no. 29)
a (Å)	20.170(9)
b (Å)	12.273(4)
c (Å)	14.109(6)
V (Å^3)	3492.5(4.6)
Z	4
D (calc) (g cm^{-3})	2.31
μ (cm^{-1}) (Mo Kα)	50.42
T(max)/T(min)(Ψ-scans)	1.00/0.552
λ (Å) (Mo Kα)	0.7107
T (°C)	−50
N(obs); N(var)	1472, 274
R; R_w	0.055, 0.058
GOF	1.79

rected for absorption (Ψ-scans), Lorentz and polarization factors. Crystal and instrument stability were checked by measuring three standard reflections every 150 observations. Only small random fluctuations were observed, therefore no decay correction was applied. The analytic form of the scattering factors for the appropriate neutral atoms were corrected for both the real (Δf) and imaginary ($\Delta f'$) components of anomalous dispersion. The structure was solved by direct methods using SHELX-86 [12] which located the metal atoms and the chloride ion. The light atoms were located by successive least-square refinements. Structure refinement was carried out using the TEXSAN 5.0 structure analysis package [12]. The metal atoms, the oxygen atoms, and the chloride ion were refined anisotropically. All the carbon atoms were refined isotropically and the contribution of the hydrogen atoms on the tetraethylammonium cation was ignored. During the final stages of refinement the correct enantiomorph was chosen by refining both enantiomorphic structures first without and then with the anomalous dispersion terms included. Full matrix least squares refinement converged to final residuals of $R = 0.055$, $R_w = 0.058$, and $S = 1.792$ for 274 refined parameters and 1472 observed ($I > 3\sigma(I)$) reflections. Final positional and displacement parameters are given in Table 2 and the bond metricals are listed in Table 3.

3. Results and discussion

The reaction of the dinuclear iron cluster $[Fe_2(CO)_8]^{2-}$ with one equivalent of $TeCl_4$ yields **I** as the only carbonyl containing compound detectable by IR spectroscopy. It crystallizes in the acentric space group $Pca2_1$ (no. 29) with two independent cluster molecules in the asymmetric unit. Each asymmetric unit also contains one molecule of $[Et_4N]Cl$ as a co-crystallite. The molecular structure of **II** is, as expected, a tetrahedron consisting of an $Fe_2(CO)_6$ fragment bridged by a ditelluride moiety. It is isostructural with the related sulfur and selenium analogs. Figure 1 shows the molecular geometry of both of the two independent molecules and the related chloride ion, as well as the atom labeling scheme.

The Fe–Fe distance, 2.645 Å (avg.), is similar to other compounds containing an $Fe_2(CO)_6$ fragment bridged by tellurium such as $Fe_2(CO)_6(TeCH_3)_2$,

TABLE 2. Positional parameters and B_{eq} for **II** $[Fe_2(CO)_6(Te_2)]\cdot[(C_2H_5)_4N]Cl$

Atom	x	y	z	B_{eq}
Te(1)	0.0810(1)	0.0366(2)	0.4260	3.7(1)
Te(2)	0.0620(1)	0.2536(2)	0.4103(3)	4.3(1)
Fe(1)	0.1731(3)	0.1710(4)	0.4611(5)	3.5(3)
Fe(2)	0.0669(3)	0.1565(4)	0.5706(5)	4.1(3)
O(11)	0.251(2)	0.187(2)	0.288(2)	6(2)
O(12)	0.261(2)	0.018(2)	0.560(2)	6(2)
O(13)	0.224(2)	0.359(2)	0.562(3)	8(2)
O(21)	−0.074(2)	0.130(3)	0.619(4)	10(3)
O(22)	0.127(2)	0.013(2)	0.708(2)	6(2)
O(23)	0.097(2)	0.342(3)	0.681(3)	10(3)
C(11)	0.218(2)	0.175(3)	0.354(3)	3.0(8)
C(12)	0.226(2)	0.079(4)	0.530(4)	5(1)
C(13)	0.207(2)	0.291(4)	0.519(4)	5(1)
C(21)	−0.020(3)	0.141(3)	0.600(4)	6(1)
C(22)	0.107(2)	0.067(4)	0.645(4)	5(1)
C(23)	0.082(3)	0.257(6)	0.643(5)	10(1)
Te(3)	0.1178(1)	0.2716(2)	0.1362(3)	4.2(1)
Te(4)	−0.0067(1)	0.1971(2)	0.1009(3)	3.5(1)
Fe(3)	0.0684(3)	0.2604(5)	−0.0306(5)	3.7(3)
Fe(4)	0.0205(3)	0.3975(4)	0.0966(5)	3.7(3)
O(31)	0.124(2)	0.056(3)	−0.085(3)	9(2)
O(32)	−0.030(1)	0.282(2)	−0.176(2)	6(2)
O(33)	0.164(2)	0.410(3)	−0.112(3)	10(3)
O(41)	0.100(1)	0.588(3)	0.041(3)	10(3)
O(42)	−0.092(1)	0.468(2)	−0.017(2)	5(2)
O(43)	−0.025(2)	0.463(2)	0.279(3)	7(2)
C(31)	0.101(2)	0.140(4)	−0.068(4)	5(1)
C(32)	0.014(3)	0.262(4)	−0.114(4)	7(1)
C(33)	0.125(2)	0.347(4)	−0.085(4)	5(1)
C(41)	0.068(3)	0.520(4)	0.069(3)	6(1)
C(42)	−0.048(2)	0.440(4)	0.024(3)	4(1)
C(43)	−0.007(2)	0.436(4)	0.201(4)	5(1)
Cl(1)	0.6333(4)	0.1754(7)	0.5060(8)	3.2(5)
N(1)	0.755(2)	0.301(2)	0.259(2)	3.6(7)
C(1A)	0.745(2)	0.183(3)	0.209(3)	4.0(8)
C(1B)	0.708(3)	0.107(4)	0.276(4)	8(1)
C(2A)	0.688(2)	0.356(4)	0.272(4)	6(1)
C(2B)	0.690(2)	0.474(3)	0.295(3)	5(1)
C(3A)	0.790(2)	0.295(4)	0.358(3)	5(1)
C(3B)	0.858(3)	0.241(4)	0.353(4)	8(1)
C(4A)	0.795(2)	0.364(3)	0.183(3)	4.2(9)
C(4B)	0.768(2)	0.392(3)	0.099(4)	6(1)

2.643(5) Å [13], and $Fe_2(CO)_6Te_2CH_2$, 2.587(2) Å [14]. However, this distance is significantly longer than that found in the corresponding disulfide [15] and diselenide clusters [16], 2.552(2) and 2.575(2) Å respectively. This progressive lengthening of the M–M bond with the increasing size of the bridging atom is also found in the $Fe_2(CO)_6(ER)_2$ (E = S, Se, Te) series [13,17,18]. The Fe–Te bonds, 2.543 Å (avg.), compare favorably with other compounds such as $Fe_2(CO)_6(TeCH_3)_2$, 2.548(2) and 2.551(3) Å [13], and $Fe_2(CO)_6Te_2CH_2$, 2.551(2) and 2.542(2) Å [14]. The Te–Te distances, 2.700(4) and 2.719(4) Å, are normal for a ditelluride single bond. These bond distances are close to that of diphenyl ditelluride, 2.712(2) [19], and slightly shorter than that found in hexagonal tellurium, 2.835(2) [20]. The observed bond distance also compares well with that observed for other transition metal clusters, such as 2.828(1) in $[Fe_8Te_{10}(CO)_{20}]^{2-}$ [21], and 2.705(3) in $[Fe_2(CO)_6(Te)(Te_2)]^{2-}$ [22].

The chloride ion of the co-crystallized organic salt is situated so as to make two long contacts of 2.90(1) and 3.027(9) Å with the two independent cluster molecules. These distances are long but well within the sum of the van der Waal's radii of 3.8 Å. Although the experimental errors in the Te–Te bond distances make definitive conclusions impossible, it appears that the longer contact is to the cluster molecule with shorter Te–Te bond (Tel–Te2) while the shorter contact is made to the molecule with the longer Te–Te (Te3–Te4) bond. The length, and therefore the strength, of these contacts appears to be inversely correlated to the Te–Te single bond distances observed. This lengthening of the tellurium–tellurium bond is consistent with that seen in the clusters $CpMoFe(CO)_5(Te_2Br)$ and $CpMoFe(CO)_5(Te_2S_2CNEt_2)$ [23]. The Te–Te distance in the former is 2.807(1) Å while this distance in the latter is 2.954(1) Å. The lengthening of the Te–Te bond in this case is thought to be caused by a secondary interaction between the thiocarbonyl sulfur atom and one of the tellurium atoms. The solution infrared spectrum of this material does not differ from samples of $Fe_2(CO)_6Te_2$ prepared via other synthetic routes. Therefore, it does not appear that the tellurium–chloride interactions persist in solution.

In addition to the tellurium chloride contacts, several other intermolecular contacts both between the tellurium atoms, and between the telluriums and the carbonyl oxygens are present (Table 4). The Te–Te distances of 4.071(4) and 4.034(5) Å are comparable to the Te–Se contact in $Fe_3(CO)_9SeTe$ (4.00 Å) [24], as well as the Te–Te contact found in $Fe_3(CO)_9(Te)_2$ (4.043(1) Å) [25]. However, it is a much longer contact than that found in $Fe_2(CO)_{10}(Te)_2$ (3.596(1) Å) [25]. The O_{CO}–Te contacts range from a relatively short

TABLE 3. Bond distances and angles for II $[Fe_2(CO)_6(Te_2) \cdot [(C_2H_5)_4N]Cl$

Distances (Å):			
Te(1)–Te(2)	2.700(4)	Te(3)–Te(4)	2.719(4)
Te(1)–Fe(1)	2.533(6)	Te(3)–Fe(3)	2.559(7)
Te(1)–Fe(2)	2.531(7)	Te(3)–Fe(4)	2.560(6)
Te(2)–Fe(1)	2.562(6)	Te(4)–Fe(3)	2.518(6)
Te(2)–Fe(2)	2.559(7)	Te(4)–Fe(4)	2.521(5)
Fe(1)–Fe(2)	2.647(8)	Fe(3)–Fe(4)	2.643(8)
Fe(1)–C(11)	1.76(4)	Fe(3)–C(31)	1.71(5)
Fe(1)–C(12)	1.82(5)	Fe(3)–C(32)	1.60(6)
Fe(1)–C(13)	1.81(5)	Fe(3)–C(33)	1.74(5)
Fe(2)–C(21)	1.82(5)	Fe(4)–C(41)	1.82(5)
Fe(2)–C(22)	1.72(5)	Fe(4)–C(42)	1.80(5)
Fe(2)–C(23)	1.63(7)	Fe(4)–C(43)	1.64(5)
O(11)–C(11)	1.16(4)	O(31)–C(31)	1.15(5)
O(12)–C(12)	1.12(5)	O(32)–C(32)	1.29(6)
O(13)–C(13)	1.10(5)	O(33)–C(33)	1.16(5)
O(21)–C(21)	1.12(5)	O(41)–C(41)	1.13(5)
O(22)–C(22)	1.18(5)	O(42)–C(42)	1.11(4)
O(23)–C(23)	1.22(7)	O(43)–C(43)	1.21(5)
N(1)–C(1A)	1.63(4)	C(1A)–C(1B)	1.53(6)
N(1)–C(2A)	1.53(6)	C(2A)–C(2B)	1.49(6)
N(1)–C(3A)	1.57(5)	C(3A)–C(3B)	1.52(6)
N(1)–C(4A)	1.54(5)	C(4A)–C(4B)	1.35(6)
Te(2)–Te(1)–Fe(1)	58.5(1)	Te(1)–Fe(2)–C(23)	157(2)
Te(2)–Te(1)–Fe(2)	58.5(2)	Te(2)–Fe(2)–Fe(1)	58.9(2)
Fe(1)–Te(1)–Fe(2)	63.0(2)	Te(2)–Fe(2)–C(21)	102(2)
Te(1)–Te(2)–Fe(1)	57.5(1)	Te(2)–Fe(2)–C(22)	149(2)
Te(1)–Te(2)–Fe(2)	57.5(2)	Te(2)–Fe(2)–C(23)	102(3)
Fe(1)–Te(2)–Fe(2)	62.3(2)	Fe(1)–Fe(2)–C(21)	158(2)
Te(1)–Fe(1)–Te(2)	64.0(2)	Fe(1)–Fe(2)–C(22)	91(2)
Te(1)–Fe(1)–Fe(2)	58.5(2)	Fe(1)–Fe(2)–C(23)	99(2)
Te(1)–Fe(1)–C(11)	103(1)	C(21)–Fe(2)–C(22)	104(2)
Te(1)–Fe(1)–C(12)	97(1)	C(21)–Fe(2)–C(23)	97(3)
Te(1)–Fe(1)–C(13)	153(2)	C(22)–Fe(2)–C(23)	91(3)
Te(2)–Fe(1)–Fe(2)	58.8(2)	Fe(1)–C(11)–O(11)	172(4)
Te(2)–Fe(1)–C(11)	102(1)	Fe(1)–C(12)–O(12)	170(5)
Te(2)–Fe(1)–C(12)	154(2)	Fe(1)–C(13)–O(13)	172(5)
Te(2)–Fe(1)–C(13)	97(1)	Fe(2)–C(21)–O(21)	179(4)
Fe(2)–Fe(1)–C(11)	157(1)	Fe(2)–C(22)–O(22)	168(4)
Fe(2)–Fe(1)–C(12)	97(2)	Fe(2)–C(23)–O(23)	168(7)
Fe(2)–Fe(1)–C(13)	95(2)	Te(4)–Te(3)–Fe(3)	56.9(2)
C(11)–Fe(1)–C(12)	100(2)	Te(4)–Te(3)–Fe(4)	56.9(1)
C(11)–Fe(1)–C(13)	100(2)	Fe(3)–Te(3)–Fe(4)	62.2(2)
C(12)–Fe(1)–C(13)	93(2)	Te(3)–Te(4)–Fe(3)	58.3(2)
Te(1)–Fe(2)–Te(2)	64.1(2)	Te(3)–Te(4)–Fe(4)	58.4(1)
Te(1)–Fe(2)–Fe(1)	58.5(2)	Fe(3)–Te(4)–Fe(4)	63.3(2)
Te(1)–Fe(2)–C(21)	104(2)	Te(3)–Fe(3)–Te(4)	64.7(2)
Te(1)–Fe(2)–C(22)	94(2)	Te(3)–Fe(3)–Fe(4)	58.9(2)
Te(3)–Fe(3)–C(31)	100(2)	C(41)–Fe(4)–C(42)	93(2)
Te(3)–Fe(3)–C(32)	160(2)	C(41)–Fe(4)–C(43)	97(2)
Te(3)–Fe(3)–C(33)	97(2)	C(42)–Fe(4)–C(43)	99(2)
Te(4)–Fe(3)–Fe(4)	58.4(2)	Fe(3)–Fe(4)–C(43)	156(2)
Te(4)–Fe(3)–C(31)	101(2)	Fe(3)–C(31)–O(31)	175(5)
Te(4)–Fe(3)–C(32)	98(2)	Fe(3)–C(32)–O(32)	169(5)
Te(4)–Fe(3)–C(33)	155(2)	Fe(3)–C(33)–O(33)	172(5)
Fe(4)–Fe(3)–C(31)	155(2)	Fe(4)–C(41)–O(41)	170(5)
Fe(4)–Fe(3)–C(32)	104(2)	Fe(4)–C(42)–O(42)	177(4)
Fe(4)–Fe(3)–C(33)	99(2)	Fe(4)–C(43)–O(43)	178(4)
C(31)–Fe(3)–C(32)	93(3)	C(1A)–N(1)–C(2A)	109(3)
C(31)–Fe(3)–C(33)	98(2)	C(1A)–N(1)–C(3A)	113(3)
C(32)–Fe(3)–C(33)	97(3)	C(1A)–N(1)–C(4A)	102(3)

TABLE 3 (continued)

Te(3)–Fe(4)–Te(4)	64.7(2)	C(2A)–N(1)–C(3A)	108(3)
Te(3)–Fe(4)–Fe(3)	58.9(2)	C(2A)–N(1)–C(4A)	109(3)
Te(3)–Fe(4)–C(41)	98(2)	C(3A)–N(1)–C(4A)	115(3)
Te(3)–Fe(4)–C(42)	153(1)	N(1)–C(1A)–C(1B)	110(4)
Te(3)–Fe(4)–C(43)	104(2)	N(1)–C(2A)–C(2B)	115(4)
Te(4)–Fe(4)–Fe(3)	58.3(2)	N(1)–C(3A)–C(3B)	112(4)
Te(4)–Fe(4)–C(41)	157(2)	N(1)–C(4A)–C(4B)	122(4)
Te(4)–Fe(4)–C(42)	97(1)		
Te(4)–Fe(4)–C(43)	101(2)		
Fe(3)–Fe(4)–C(41)	101(2)		
Fe(3)–Fe(4)–C(42)	95(1)		

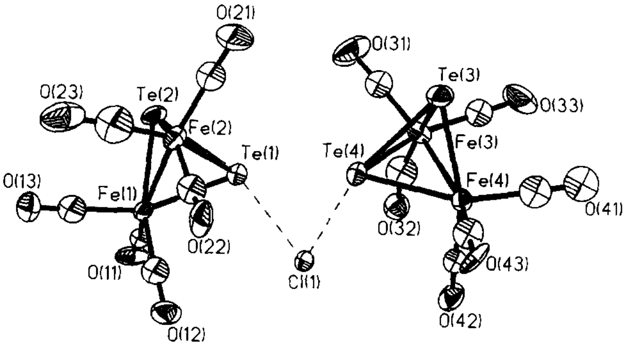


Fig. 1. View of $Fe_2(CO)_6Te_2$ (displacement ellipsoids at 35% probability) illustrating the molecular geometry and the atom labeling scheme.

3.53(3) to 3.86(3) Å. These distances are also similar to those found in the $Fe_3(CO)_9(Te)_2$ and $Fe_3(CO)_{10}(Te)_2$ clusters [25].

The intermolecular contacts create an elaborate three dimensional network consisting of a complex spiral structure containing channels in two directions (Fig. 2). Along the c direction the intermolecular contacts between the tellurium atoms, the tellurium atoms and the chloride ion, and between the tellurium atoms and the oxygen atoms create a series of interlocking helices. These helices leave an infinite array of roughly circular channels which share edges. The tetraethylammonium cations reside in these large channels. The spiral nature of the network also creates a series of channels in the b direction. These channels are built from the tellurium–tellurium contacts as well as the tellurium–oxygen contacts. This channel can be viewed as oval in shape with a single contact between an oxygen and a tellurium bisecting it along the minor axis of the ellipse.

Based on the large number of intermolecular contacts seen in the solid state structure of II and the

TABLE 4. Intermolecular Contacts (Å) for II $[Fe_2(CO)_6(Te_2)] \cdot [(C_2H_5)_4N]Cl$

Te(1)–Cl(1)	3.027(9)	Te(2)–O(43)	3.63(3)
Te(4)–Cl(1)	2.90(1)	Te(3)–O(11)	3.59(3)
Te(1)–Te(4)	4.071(4)	Te(3)–O(13)	3.53(3)
Te(2)–Te(3)	4.034(5)	Te(4)–O(22)	3.86(3)
Te(2)–O(42)	3.62(3)		

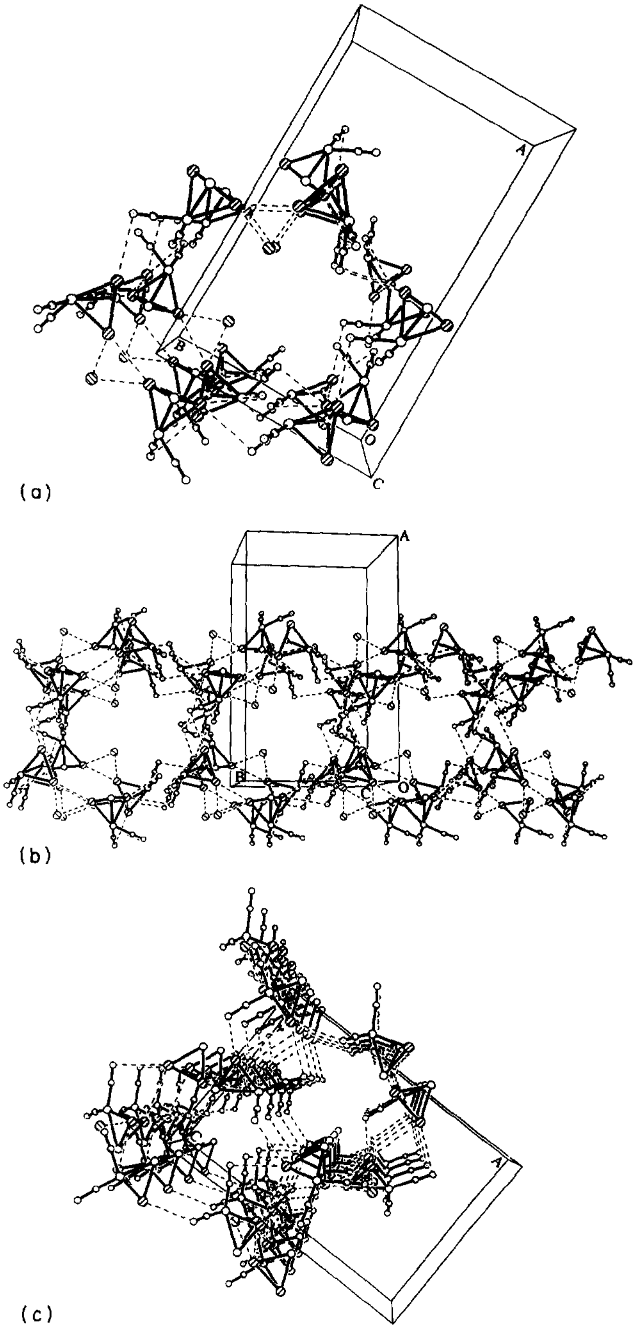
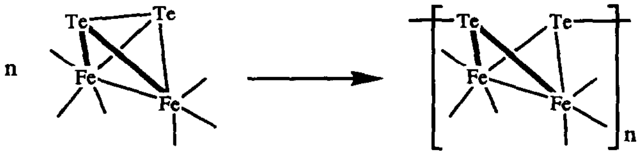


Fig. 2. Packing diagram projections along ((a) and (b)) the c axis and (c) along the b axis showing the Cl–Te, Te–Te, and Te–O contacts. The $[Et_4N]^+$ cation and those CO groups not involved in the contacts have been omitted for clarity.

related $Fe_3(CO)_9(Te)_2$ and $Fe_3(CO)_{10}(Te)_2$, it is interesting to consider the possible fate of **I** upon decomposition. It has been postulated that **I** decomposes by dimerizing to $Fe_4Te_4(CO)_{12}$ with a cubane-like structure on the basis of infrared and analytic data [1,26]. However, the presence of the tellurium–tellurium as well as the tellurium–chloride contacts suggest that an alternative decomposition pathway such as polymerization via the rupture and subsequent reforming of tellurium–tellurium bonds could be possible (eqn. 1).



This result would give material which would show the same analytic results as $Fe_4Te_4(CO)_{12}$ and could be expected to have a similar IR spectrum. This process should also be energetically favorable as it will make as many bonds as it breaks and will also relieve the strain imposed on the Fe–Te–Te and Te–Fe–Te angles by the tetrahedral geometry of the cluster. Further study of this highly reactive species and its decomposition products will be necessary to resolve this question.

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Supplementary Material Available

Listings of the anisotropic displacement parameters and structure factor tables for **I**, have been deposited with the Cambridge Crystallographic Data Centre.

References

1 D.A. Lesch and T.B. Rauchfuss, *Inorg. Chem., 20*, (1981) 3583.
2 W. Hieber and J. Gruber, *Z. Anorg. Allg. Chem., 296* (1958) 91.
3 D.A. Lesch and T.B. Rauchfuss, *J. Organomet. Chem., 199* (1980) C6.
4 V.W. Day, D.A. Lesch and T.B. Rauchfuss, *J. Am. Chem. Soc., 104* (1982) 1290.
5 P. Mathur, I.J. Mavunkal and A.L. Rheingold, *J. Chem. Soc., Chem. Commun.*, (1989) 382.
6 P. Mathur, I.J. Mavunkal and V. Rugmini, *J. Organomet. Chem., 367* (1989) 243.
7 P. Mathur and D. Reddy, *J. Organomet. Chem., 385* (1990) 363.
8 P. Mathur, I.J. Mavunkal and V. Rugmini, *Inorg. Chem., 29* (1990) 3616.
9 P. Mathur, V.D. Reddy, K. Das and U.C. Sinha, *J. Organomet. Chem., 409* (1991) 255.
10 H. Strong, P.J. Krusic and J. San Filippo, Jr., *Inorg. Synth., 28* (1990) 203.
11 G.M. Sheldrick, in G.M. Sheldrick, C. Kruger and R. Goddard (eds.), Crystallographic Computing 3, Oxford University Press, Oxford, 1985, pp. 175–189.
12 TEXSAN: Single Crystal Structure Analysis Software, v. 5.0. Molecular Structure Corporation, Woodlands, TX, 1989.
13 R.E. Bachman and K.H. Whitmire, *Organometallics, 12* (1993) 1988.
14 P. Mathur, V.D. Reddy and R. Bohra, *J. Organomet. Chem., 401* (1991) 339.
15 C.H. Wei and L.F. Dahl, *Inorg. Chem., 4* (1965) 1.
16 C.F. Campana, F.Y.K. Lo and L.F. Dahl, *Inorg. Chem., 18* (1979) 3060.
17 C.H. Wei and L.F. Dahl, *Inorg. Chem., 2* (1963) 328.
18 P. Mathur and M.M. Hossain, *Organometallics, 12* (1993) 2398.
19 G. Llabres, O. Dideberg and L. Dupont, *Acta Crystallogr., Sect. B., 28* (1972) 2438.
20 P. Cherin and P. Unger, *Acta Crystallogr., 23* (1967) 670.
21 L.C. Roof, W.T. Pennington and J.W. Kolis, *Angew. Chem. Int. Ed. Engl., 31* (1992) 913.
22 B.W. Eichhorn, R.C. Haushalter and J.S. Merola, *Inorg. Chem., 29* (1990) 728.
23 L.E. Bogan, Jr., T.B. Rauchfuss and A.L. Rheingold, *Inorg Chem., 24* (1985) 3720.
24 G. Gervasio, *J. Organomet. Chem., 445* (1993) 147.
25 G. Gervasio, *J. Organomet. Chem., 441* (1993) 271.
26 L.E. Bogan, Jr., D.A. Lesch and T.B. Rauchfuss, *J. Organomet. Chem., 250* (1983) 429.