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Structure of a Te–Methylene–Te Bridged Double-Butterfly Complex

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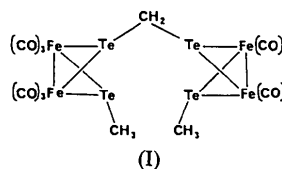
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Abstract. *Te,Te'*-Methylenebis[hexacarbonyl- μ -methyltellurio- μ -tellurio-diiron(*Fe—Fe*)], [Fe₂(μ -CH₃-Te)(CO)₆]₂(μ_4 -CH₂Te₂), *M_r* = 1090.01, monoclinic, *C2/c*, *a* = 28.865 (10), *b* = 13.787 (7), *c* = 14.733 (4) Å, β = 104.67 (2)°, *V* = 5671.86 Å³, *Z* = 8, *D_x* = 2.637 Mg m⁻³, $\lambda(\text{Mo } K\alpha)$ = 0.7107 Å, μ = 5.83 mm⁻¹, *F*(000) = 4096, *T* = 296 K. Final *R* and *wR* are 0.0632 and 0.0598, respectively, for 2591 observed reflections. The crystal is stacked with two isomers occupying alternative lattice points. Each lattice point is associated with two identical structures with occupation probabilities of 80 and 20%. One Fe atom [Fe(1)/Fe(3)], and a CO group attached to it, are common in both. For the remaining atoms, a symmetry relationship $1 - x, y, \frac{1}{2} - z$ exists between the positions with unequal probabilities. Each molecule contains two butterfly shaped [(CO)₆Fe₂Te(CH₃)Te] units bridged through a CH₂ group.

Introduction. The dinuclear compound Fe₂(CO)₆(μ -Te₂) is reported as a favourable starting material for the preparation of various mixed-metal complexes (Day, Lesch & Rauchfuss, 1982; Mathur, Mavunkal & Rheingold, 1989). Similarly, the compound Fe₃(CO)₉(μ_3 -Te)₂ also coordinates with the unsaturated metal fragments but it is relatively more inert towards such addition than the diiron compound. The title compound (I), an unusual product, was obtained from the reaction of Fe₃(CO)₉(μ_3 -Te)₂ with excess diazomethane (Mathur, Reddy, Das & Sinha,

1991) at room temperature. Black brick-shaped crystals were obtained from a hexane solution of the compound at 268 K.



Experimental. A crystal of dimensions 0.18 × 0.14 × 0.40 mm was used for the intensity-data collection on a Nicolet *R3m/V* four-circle diffractometer with graphite-monochromated Mo *K*α radiation. The unit-cell parameters were refined from 20 reflections in the range 6 < θ < 13°. A total of 7484 intensities were scanned in the range 2 ≤ 2 θ ≤ 45°, 0 ≤ *h* ≤ 32, 0 ≤ *k* ≤ 15 and -16 ≤ *l* ≤ 16, assuming a primitive monoclinic cell. Two reflections (2,0,10 and 811), checked every 3600 s, indicated no significant variation in intensities. An empirical correction (North, Phillips & Mathews, 1968) for absorption and extinction was applied to all data; minimum and maximum transmission factors were 0.298 and 0.371 respectively. Lp corrections were applied. After averaging the equivalent reflections, 3373 unique reflections (*R*_{int} = 0.076) were obtained which were used in structure determination. The systematic absences (*hkl*: *h* + *k* = 2*n*; *h0l*: *l* = 2*n* + 1 and *h* = 2*n* + 1)

Table 1. Fractional coordinates ($\times 10^5$ for Te and Fe with s.o.f. 1.00 and 0.8 and $\times 10^4$ for the remaining atoms), isotropic temperature factors for C and O atoms with s.o.f. 0.2 and equivalent isotropic temperature factors for the remaining atoms

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}/U_{iso}
Te(1)	43829 (4)	44960 (12)	19434 (9)	46 (2)
Te(2)	33350 (4)	53576 (6)	19692 (10)	49 (2)
Fe(1)†	40647 (7)	49947 (21)	33292 (14)	43 (2)
Fe(2)	36441 (9)	36312 (25)	21825 (21)	47 (4)
Te(3)	43837 (4)	-5133 (13)	24395 (9)	46 (2)
Te(4)	33373 (4)	3633 (16)	13609 (10)	50 (2)
Fe(3)†	40631 (7)	112 (22)	7357 (14)	43 (2)
Fe(4)	36420 (9)	-13630 (24)	14412 (21)	46 (4)
C(1)	5000‡	3591 (19)	2500‡	54 (15)
C(2)	2711 (7)	5463 (21)	2553 (17)	93 (15)
C(3)	5000‡	8480 (23)	2500‡	54 (15)
C(4)	2726 (8)	10513 (22)	160 (17)	88 (15)
C(11)†	3699 (5)	4926 (16)	4093 (10)	64 (13)
O(11)†	3469 (4)	4928 (16)	4637 (9)	117 (12)
C(12)	4501 (7)	4321 (16)	4108 (14)	54 (14)
O(12)	4814 (5)	3871 (13)	4625 (11)	79 (13)
C(13)	4297 (7)	6179 (17)	3572 (15)	71 (15)
O(13)	4447 (7)	6984 (14)	3690 (13)	105 (14)
C(21)	3986 (7)	2655 (14)	2766 (14)	62 (15)
O(21)	4223 (6)	1988 (12)	3154 (12)	93 (14)
C(22)	3413 (8)	3220 (18)	1027 (15)	76 (15)
O(22)	3292 (7)	2824 (15)	281 (11)	109 (14)
C(23)	3166 (8)	3326 (20)	2638 (17)	80 (15)
O(23)	2845 (7)	3075 (19)	2959 (17)	157 (15)
C(31)†	3703 (5)	9974 (20)	-393 (11)	83 (14)
O(31)†	3446 (6)	9926 (18)	-1171 (9)	149 (14)
C(32)	4308 (7)	11181 (17)	756 (16)	71 (15)
O(32)	4454 (6)	11983 (14)	783 (14)	101 (14)
C(33)	4521 (7)	9346 (17)	415 (13)	62 (14)
O(33)	4815 (6)	8894 (13)	162 (12)	83 (13)
C(41)	3993 (8)	7633 (17)	1232 (17)	75 (15)
O(41)	4225 (6)	6987 (13)	1024 (12)	91 (14)
C(42)	3410 (8)	8154 (16)	2338 (15)	63 (15)
O(42)	3278 (6)	7859 (14)	2980 (13)	96 (14)
C(43)	3152 (8)	8326 (21)	504 (16)	87 (15)
O(43)	2853 (7)	8130 (19)	-191 (14)	149 (15)
Te(1')	4385 (2)	5559 (4)	1943 (4)	34 (6)
Te(2')	3358 (2)	4533 (5)	2007 (4)	44 (7)
Fe(2')	3634 (4)	6331 (9)	2245 (8)	42 (10)
Te(3')	4383 (2)	484 (5)	2436 (4)	41 (6)
Te(4')	3315 (2)	9594 (5)	1361 (5)	51 (6)
Fe(4')	3648 (4)	1395 (8)	1524 (9)	42 (11)
C(1')	5000‡	6434 (60)	2500‡	13 (9)
C(2')	2748 (30)	4431 (61)	2570 (53)	23 (9)
C(3')	5000‡	1598 (75)	2500‡	35 (10)
C(4')	2683 (25)	-463 (54)	174 (47)	16 (9)
C(12')	5460 (24)	4350 (48)	5932 (42)	15 (9)
O(12')	5190 (19)	3876 (39)	5329 (36)	21 (9)
C(13')	4377 (23)	3934 (42)	3444 (45)	13 (9)
O(13')	4449 (22)	3080 (41)	3660 (40)	29 (9)
C(21')	4024 (37)	7334 (67)	2611 (61)	35 (10)
O(21')	4186 (27)	7933 (53)	3218 (48)	42 (9)
C(22')	3440 (32)	6812 (59)	1128 (45)	29 (10)
O(22')	3268 (23)	7155 (47)	352 (40)	33 (9)
C(23')	3251 (26)	6619 (61)	2803 (53)	27 (9)
O(23')	2939 (22)	6685 (48)	3219 (43)	34 (9)
C(32')	4203 (30)	8842 (47)	455 (53)	25 (9)
O(32')	4404 (21)	8061 (42)	590 (38)	27 (9)
C(33')	4469 (22)	626 (50)	195 (48)	17 (9)
O(33')	4836 (20)	1055 (43)	195 (38)	25 (9)
C(41')	3967 (24)	2227 (45)	1270 (47)	19 (9)
O(41')	4243 (19)	2903 (39)	1314 (36)	24 (9)
C(42')	3521 (29)	1807 (58)	2583 (50)	26 (9)
O(42')	3303 (26)	2127 (52)	3127 (46)	41 (9)
C(43')	3207 (28)	1732 (55)	556 (49)	19 (9)
O(43')	2848 (29)	2097 (57)	119 (51)	23 (9)

† Atoms with s.o.f. 1.00. Those atoms labelled with a prime (') have s.o.f. 0.2; the s.o.f. for each of the remaining atoms is 0.8.

‡ Coordinate fixed on a twofold axis (y axis).

indicated the space group to be either Cc or $C2/c$. Hypercentric distribution of intensities favours the centrosymmetric space group with the existence of

Table 2. Important bond distances (Å) and bond angles ($^\circ$) with e.s.d.'s in parentheses

Fe(1)—Te(1)	2.537 (2)	Fe(4)—Fe(3)	2.605 (4)
Fe(2)—Te(1)	2.544 (3)	Fe(2)—Fe(1)	2.612 (4)
C(1)—Te(1)	2.159 (15)	Fe(3)—Te(3)	2.550 (2)
Fe(1)—Te(2)	2.562 (2)	Fe(4)—Te(3)	2.554 (3)
Fe(2)—Te(2)	2.533 (4)	C(3)—Te(3)	2.240 (19)
C(2)—Te(2)	2.189 (20)	C(4)—Te(4)	2.170 (21)
Fe(3)—Te(4)	2.540 (2)	Fe(4)—Te(4)	2.530 (4)
Fe(1)—Te(1')	2.567 (6)	Fe(1)—Te(2')	2.520 (6)
Fe(2)—Te(1')	2.554 (12)	Fe(4')—Te(4')	2.651 (13)
C(1')—Te(1')	2.130 (47)	C(4')—Te(4')	2.184 (67)
Fe(2')—Te(2')	2.600 (13)	Fe(2')—Fe(1)	2.547 (11)
C(2')—Te(2')	2.132 (85)	C(3')—Te(3')	2.335 (68)
Fe(3)—Te(3')	2.526 (6)	Fe(4')—Te(3')	2.538 (13)
Te(4')—Fe(3)	2.618 (7)	Fe(4')—Fe(3)	2.670 (11)
Fe(2)—Te(1)—Fe(1)	61.9 (1)	Te(4)—Fe(3)—Te(3)	79.9 (1)
C(1)—Te(1)—Fe(1)	106.4 (1)	Fe(4)—Fe(3)—Te(3)	59.4 (1)
C(1)—Te(1)—Fe(2)	108.4 (5)	Fe(4)—Fe(3)—Te(4)	58.9 (1)
Fe(2)—Te(2)—Fe(1)	61.7 (1)	Te(4)—Fe(4)—Te(3)	80.0 (1)
C(2)—Te(2)—Fe(1)	107.5 (7)	Fe(3)—Fe(4)—Te(3)	59.2 (1)
C(2)—Te(2)—Fe(2)	108.2 (8)	Fe(3)—Fe(4)—Te(4)	59.3 (1)
Te(2)—Fe(1)—Te(1)	79.5 (1)	Te(2)—Fe(2)—Te(1)	79.8 (1)
Fe(2)—Fe(1)—Te(1)	59.2 (1)	Fe(2)—Fe(1)—Te(2)	58.6 (1)
Fe(1)—Fe(2)—Te(1)	58.9 (1)	Fe(1)—Fe(2)—Te(2)	59.7 (1)
Fe(4)—Te(3)—Fe(3)	61.4 (1)	Fe(4)—Te(4)—Fe(3)	61.8 (1)
C(3)—Te(3)—Fe(3)	107.5 (1)	C(3)—Te(3)—Fe(4)	106.3 (5)
C(4)—Te(4)—Fe(3)	107.3 (7)	C(4)—Te(4)—Fe(4)	109.2 (8)
Fe(2')—Te(1')—Fe(1)	59.6 (3)	Fe(4')—Te(4')—Fe(3)	60.9 (3)
C(4')—Te(4')—Fe(1)	107.3 (4)	C(4')—Te(4')—Fe(3)	108.8 (19)
C(1')—Te(1')—Fe(2')	111.0 (15)	C(4')—Te(4')—Fe(4')	108.4 (20)
Fe(2')—Te(2')—Fe(1)	59.6 (3)	Fe(3)—Fe(4')—Te(3')	58.0 (3)
C(2')—Te(2')—Fe(1)	107.6 (20)	C(3')—Te(3')—Fe(3)	107.1 (3)
C(2')—Te(2')—Fe(2')	105.4 (23)	C(3')—Te(3')—Fe(4')	102.9 (17)
Te(2')—Fe(1')—Te(1')	81.1 (2)	Te(4')—Fe(4')—Te(3')	79.6 (4)
Fe(2')—Fe(1')—Te(1')	59.9 (3)	Te(4')—Fe(4')—Fe(3)	59.0 (3)
Fe(4')—Fe(1')—Te(2')	61.7 (3)	Fe(4')—Te(3')—Fe(3)	63.6 (3)
Te(2')—Fe(2')—Te(1')	79.9 (4)	Te(4')—Fe(3)—Te(3')	80.5 (2)
Fe(1')—Fe(2')—Te(1')	60.4 (3)	Fe(4')—Fe(3)—Te(3')	58.4 (3)
Fe(1')—Fe(2')—Te(2')	58.6 (3)	Fe(4')—Fe(3)—Te(4')	60.2 (3)

some additional centres of symmetry at general positions. Finally, the space group $C2/c$ was confirmed by structure solution.

At first, the structure was solved using *MULTAN88* (Debaerdemaeker, Germain, Main, Refaat, Tate & Woolfson, 1988). While refining the atoms, high Fourier peaks appeared near Te and Fe atoms (Mathur, Reddy, Das & Sinha, 1991). Further investigation indicated probable symmetry relationships among the heavy atoms and the nearby peaks.

The structure solution was re-attempted by *SIR88* (Burla, Camalli, Cascarano, Giacovazzo, Polidori, Spagna & Viterbo, 1989) using the detected pseudo-translation symmetry condition $2h + 2k + 3l = 6n$ (for 1888 reflections with 20% mean fractional scattering power). The triplets were estimated by a Cochran type $P-3$ formula. The phase set with CFOM = 0.882 [$M(\text{ABS}) = 1.019$; $\text{ALCOMB} = 0.998$; $\text{CPHASE} = 1.000$; $\psi\text{-COMB} = 0.583$] was accepted as the solution. Even in the presence of pseudo-translation symmetry the solution showed excellent FOM (figures of merit) except the $\psi\text{-FOM}$. Out of the 12 strongest peaks in the E map, derived from the above phase set, eight peaks were assigned as the positions of Te and Fe atoms and each of the remaining four peaks was close to a Te atom with probable symmetry relation $(x, 1 - y, z)$. Conse-

quently, the crystal was found to be a superposition of two isomers with occupation probabilities 80 and 20%. All the light atoms (C and O) with site occupancy factor (s.o.f.) 0.8 were located from subsequent Fourier maps and those with s.o.f. 0.2 from difference Fourier maps. Later, atoms were refined with isotropic thermal parameters only. *SHELX76* (Sheldrick, 1976) was used for structure refinement.

Instead of a complete molecule, two half units [each with type I and type II units] are present in the asymmetric unit; each is bridged to another half unit at $1-x, y, \frac{1}{2}-z$ through a CH₂ unit on a crystallographic twofold axis. The C and O atoms with 20% occupancy were refined with isotropic thermal parameters. In the final cycle of least-squares refinement of the structure, 470 independent parameters were refined against 2591 observed reflections, suppressing those with $|F_c| < 3.0\sigma(F)$; (shift/e.s.d.)_{max} = 0.252. Final $R = 0.0632$ and $wR = 0.0598$ {where $w = 1/[\sigma^2(F) + 0.00016F^2]$ }. The atomic scattering factors for Te and Fe were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149) and those of other atoms from *SHELX76*. In the final difference map, high electron-density peaks appeared near Te atoms with $(\Delta\rho)_{\text{max}} = 1.444$ and $(\Delta\rho)_{\text{min}} = -1.440 \text{ e } \text{Å}^{-3}$; H atoms could not be located. A CYBER-180/840 computer was used for all computations.

Discussion. The positional parameters for all non-H atoms are listed in Table 1* along with their respective equivalent isotropic or isotropic temperature factors. Important bond lengths and bond angles are given in Table 2. In Fig. 1, (a) and (b) (Motherwell & Clegg, 1978) represent the molecules (type I) with respective occupation probabilities 80% (molecule A) and 20% (molecule B), viewed in the same projection. The positions of Fe(1)/Fe(3) and a CO group [C(11)/C(31) and O(11)/O(31)] attached to it are common in both isomers. As a consequence, each has s.o.f. 1.00. Each molecule consists of two Fe₂Te₂ butterfly units in which the Te atoms are at the 'wing tips'. Two Te atoms in both units are bridged through a CH₂ group forming a double-butterfly conformation of the molecule. Each of the two remaining Te atoms [Te(2)] are bonded to a methyl group. The methyl groups attached to the outer Te atoms [Te(2), Te(4)] are at similar Te—C distances with an average of 2.184 (20) Å. In contrast, the C—Te bond lengths associated with the bridging

methyls [C(1) and C(3)] are significantly different from each other: C(1)—Te(1) 2.159 (15) and C(3)—Te(3) 2.240 (19) Å. The Fe—Fe bond, the bridging Te atoms and the three terminally bonded carbonyl groups form a distorted octahedral conformation around each Fe atom. As a consequence, Fe—Fe—Te and Fe—Te—Fe angles are about 60° with maximum deviation 3.6°. In the butterfly cores, the average Te—Fe distance is 2.544 (3)/2.579 (9) Å and the average Fe—Fe distance is 2.608 (4)/2.608 (13) Å for molecule A/molecule B. The average Te...Te non-bonding distance, 3.524 (5) Å, is larger than that observed in (CO)₉Fe₃(μ-TeCH₂Te) (Mathur, Reddy & Bohra, 1991) resulting in a greater opening of the Fe₂Te₂ butterflies. This is also reflected in the higher Te—Fe—Te bond angles, average 79.98 (2)° compared to 75° in (CO)₆Fe₂(μ-TeCH₂Te) (Day, Lesch & Rauchfuss, 1982). The arrangement of the molecules (with s.o.f. 0.8) along the *b* axis (range $-b/2$ to $b/2$) in Fig. 2 shows the

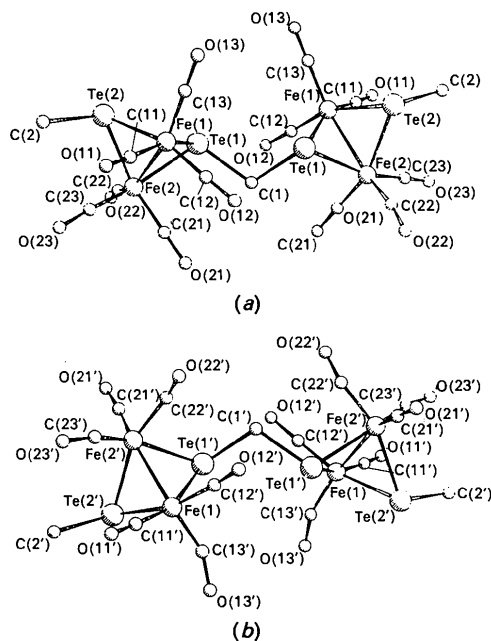


Fig. 1. Perspective view of the molecule with (a) 80% occupancy and (b) 20% occupancy.

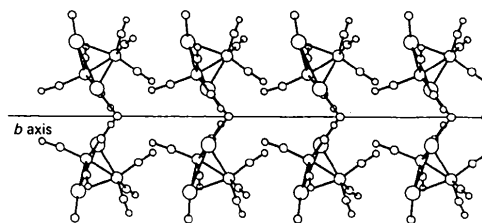


Fig. 2. Stacking of the molecules (occupancy 80%) along the *b* axis.

* Lists of structure factors, anisotropic thermal parameters and bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54938 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0505]

presence of additional centres of symmetry apart from the space group symmetries. No short intermolecular distances were observed.

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Structures of Partially Deuterated Sodium Hydrogen Succinate at 295 and 120 K and Rubidium Hydrogen Succinate at 120 K

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Abstract. $\text{Na}^+ \cdot \text{C}_4(\text{H,D})\text{H}_4\text{O}_4^-$, $M_r = 140.07$ (undeuterated), monoclinic, $C2/c$, $Z = 4$, $F(000) = 288$; at $T = 295$ K: $a = 9.217$ (1), $b = 9.998$ (1), $c = 7.365$ (1) Å, $\beta = 117.07$ (1)°, $V = 604.3$ (3) Å³, $D_x = 1.539$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54184$ Å, $\mu = 17.667$ cm⁻¹, $R = 0.036$ for 572 observed reflections; at $T = 120$ K: $a = 8.844$ (2), $b = 10.284$ (3), $c = 7.349$ (2) Å, $\beta = 117.33$ (2)°, $V = 593.8$ (6) Å³, $D_x = 1.567$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 1.886$ cm⁻¹, $R = 0.028$ for 1891 observed reflections. $\text{Rb}^+ \cdot \text{C}_4\text{H}_5\text{O}_4^-$, $M_r = 202.55$, monoclinic, $C2/c$, $a = 16.927$ (3), $b = 6.1050$ (6), $c = 6.3343$ (9) Å, $\beta = 109.17$ (1)°, $V = 618.3$ (3) Å³, $Z = 4$, $D_x = 2.176$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 77.299$ cm⁻¹, $F(000) = 392$, $T = 120$ K, $R = 0.031$ for 1692 observed reflections. No significant discrepancies are observed in the crystal structure of partially deuterated sodium hydrogen succinate at 295 and 120 K. In this salt the hydrogen succinate ions are linked by short crystallographically symmetric hydrogen bonds across an inversion centre and in rubidium hydrogen succinate across a twofold axis forming infinite chains in the structure. The length of the short hydrogen bond is 2.4272 (9) Å at 295 K

and 2.4295 (5) Å at 120 K in partially deuterated sodium hydrogen succinate and 2.4406 (15) Å in rubidium hydrogen succinate. In both salts the H atom in the short hydrogen bond is best described by two equally populated sites on each side of the centre of the hydrogen bond. The Na⁺ ion is coordinated by six O atoms with four short (2.37–2.40 Å) and two long (2.58 Å) Na—O distances forming an irregular octahedron. The coordination polyhedron for the Rb⁺ ion is a square antiprism formed by eight O atoms with Rb—O distances in the range 2.92–3.02 Å.

Introduction. The crystal structure determinations of partially deuterated sodium hydrogen succinate (1) and rubidium hydrogen succinate (2) were performed as a continuation of earlier studies of acid salts of malonic acid and succinic acid with special interest in the hydrogen bonding (Kalsbeek & Larsen, 1991; Kalsbeek, 1991, 1992). Acid salts of carboxylic acids and dicarboxylic acids have been extensively investigated, both structurally (Speakman, 1972) and spectroscopically (Hadži, 1965).