against both on theoretical and practical grounds. All the parameters measured, however, indicate a considerable difference in effective electronegativity and basicity between silicon and germanium homologs.

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CONTRIBUTION[1] FROM THE CENTRAL RESEARCH DEPARTMENT, EXPERIMENTAL STATION
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Synthesis and Structure of $(CH_3)_4Sn_3Fe_4(CO)_{16}$[2]

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The reaction of CH_3SnCl_3 and $Na_2Fe(CO)_4$ involves a facile redistribution of methyl groups to give as isolable products $[(CH_3)_2SnFe(CO)_4]_2$ and the new compound $(CH_3)_4Sn_3Fe_4(CO)_{16}$. The crystal structure of $(CH_3)_4Sn_3Fe_4(CO)_{16}$ has (monoclinic) symmetry $P2_1/c$ and a unit cell of dimensions $a = 11.04$ A, $b = 16.36$ A, $c = 19.09$ A, and $\beta = 108.5°$. There are four molecules per unit cell. The structure has been refined by least squares, using counter-measured Mo $K\alpha$ diffraction data. Each $(CH_3)_4Sn_3Fe_4(CO)_{16}$ molecule contains a row of three tetrahedrally coordinated tin atoms, the central tin atom being bonded to four $Fe(CO)_4$ groups and each terminal tin atom being bonded to two $Fe(CO)_4$ groups and two methyl groups. The coordination of each iron is octahedral. The presence of almost exactly planar Sn_2Fe_2 rings causes compression of the average SnFeSn angle to 77.9° and of the FeSnFe angles within a ring to 105.2° at the terminal tin atom and 98.8° at the central tin atom. The average interatomic distances are: Fe–(terminal)Sn = 2.625 A, Fe–(central)Sn = 2.747 A, Fe–C = 1.75 A, C–O = 1.17 A, and Sn–C = 2.22 A.

Introduction

A number of compounds containing tin–transition metal bonds have been described in the literature.[4] One such compound is $[(CH_3)_2SnFe(CO)_4]_2$, which can be obtained[5] by the reaction of $(CH_3)_2SnCl_2$ with solutions of the $HFe(CO)_4^-$ anion. Since Fe–Sn bonds are quite stable and a compound, $[Fe(CO)_3NO]_4Sn$, is known,[6] in which the four iron atoms are bonded to one tin atom, the reaction of CH_3SnCl_3 and $Na_2Fe(CO)_4$ was investigated in hopes of obtaining a closed polynuclear metal cluster of iron and tin atoms. However, because of a facile redistribution of the methyl groups bonded to tin, the product was not the expected adamantane-type metal cluster, but a mixture of the known $[(CH_3)_2SnFe(CO)_4]_2$ and the new compound, $(CH_3)_4Sn_3Fe_4(CO)_{16}$. The crystal structure of the latter compound was determined to establish its geometry, in a continuing study of the bonding in polynuclear complexes.

Experimental Section

All reactions were performed in a dry, oxygen-free nitrogen atmosphere in either glass apparatus or a polyethylene dry bag.

Reagents.—$Fe_3(CO)_{12}$ was obtained from Alfa Inorganics and purified by thorough washing with 0.1 *M* HCl, methanol, and petroleum ether (bp 30–60°), followed by overnight Soxhlet extraction with *n*-hexane in a nitrogen atmosphere. The mixture was cooled to 25° and the dark green crystals were collected under nitrogen, vacuum dried, and stored in sealed glass ampoules. Pure $Fe_3(CO)_{12}$ was recovered in 90% yield. Ammonia (Matheson Co.) was distilled from sodium. Anhydrous $SnCl_2$, $(CH_3)_2SnCl_2$, and $(CH_3)_3SnCl$ were used as obtained from the Metal and Thermit Co. Tetrahydrofuran was distilled from $LiAlH_4$ in a nitrogen atmosphere and used immediately.

Preparation of CH_3SnCl_3.—The CH_3SnCl_3 was prepared[7] from $SnCl_2$ and CH_3Cl at 360°. The product was purified by recrystallization from petroleum ether at −78° followed by sublimation at 60° (1 μ). Gas chromatographic analysis showed that the purified product contained 98.2% CH_3SnCl_3 and 1.7% $(CH_3)_2SnCl_2$. The gas chromatographic analyses were made using a 1-m 20% fluorosilicon (FS 1265) on 60–80 mesh NAWFB column with a helium flow rate of 10 cc/7.1 min. The column was maintained at 100° with the vaporizer at 150°. Under these conditions the observed retention times were: CH_3SnCl_3, 5.3 min; $(CH_3)_2SnCl_2$, 8.4 min; and $(CH_3)_3SnCl$, 3.3 min. The CH_3SnCl_3 samples contained no $(CH_3)_3SnCl$.

Preparation of $Na_2Fe(CO)_4$.—The preparation of $Na_2Fe(CO)_4$ from sodium and $Fe_3(CO)_{12}$ in tetrahydrofuran[8] produces a dark red solution which contains uncertain quantities of polynuclear iron carbonyl anions as well as $Na_2Fe(CO)_4$. However, the reaction of $Fe_3(CO)_{12}$ with sodium in anhydrous liquid ammonia produces almost pure $Na_2Fe(CO)_4$.[9] The following procedure is convenient for a large-scale preparation. A 1-l. flask equipped with a Dry Ice condenser, nitrogen inlet, and magnetic stirrer was filled with dry nitrogen. $Fe_3(CO)_{12}$ (16.9 g, 0.0336 mole) and 600 ml of sodium-dried ammonia were charged into the flask, which was immersed in a Dry Ice–acetone bath. Freshly cut sodium (4.7 g, 0.20 g-atom) was added in 0.1–0.2-g portions over 0.5 hr, the sodium being maintained in a nitrogen atmosphere before addition. The reaction mixture initially became deep red, but, upon complete addition of the sodium, a white precipitate and a pale yellow supernatant liquid were obtained. The mixture was stirred at −78° for 15 min; the ammonia was then removed at 0°, and the gray residue was dried at 90° (0.1 μ)

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overnight. The yield of $Na_2Fe(CO)_4$ was assumed to be quantitative.[10]

Preparation of $(CH_3)_4Sn_3Fe_4(CO)_{16}$.—The entire procedure, including purification, was performed in a dry nitrogen atmosphere. A slurry of $Na_2Fe(CO)_4$ (0.101 mole, prepared as above) in 300 ml of tetrahydrofuran was cooled to 0° and a solution of 16.1 g (0.067 mole) of CH_3SnCl_3 in 75 ml of tetrahydrofuran was added over 0.5 hr. No gas evolution was observed. The reaction mixture, initially pale yellow, rapidly became orange and then deep red. After being stirred at 0° for 0.5 hr and refluxed for 4.0 hr, the solution was filtered through Celite and the solvent was removed under vacuum. The resulting red residue was extracted overnight with benzene in a Soxhlet extractor. Evaporation of the benzene extract left 12.8 g of a red crystalline solid which had a complex infrared spectrum (hexane solution) in the metal carbonyl stretching region, consisting of nine bands between 1900 and 2100 cm^{-1}.

The crude product was extracted with a total of 4.2 l. of petroleum ether and chromatographed in a nitrogen atmosphere in 400-ml portions on grade 1 acid alumina (Woelm) using 2.5 × 50 cm columns. A 2:1 petroleum ether–benzene mixture eluted a red fraction which was evaporated to give a red crystalline solid. A second fraction was collected on elution with dichloromethane. Acetone then slowly eluted a yellow fraction which was identified as $[(CH_3)_2SnFe(CO)_4]_2$ by its infrared spectrum (see below).

All of the second (dichloromethane) eluates were combined and evaporated, and the residue was chromatographed as before. The residue from evaporation of the first fraction was combined with that obtained from the previous first fraction to give 6.6 g of a dark red crystalline solid. The infrared spectrum (hexane solution) now showed four strong bands from 1900 to 2100 cm^{-1}, as well as three weak bands corresponding to $[(CH_3)_2SnFe(CO)_4]_2$. This product was again chromatographed; the first fraction gave 3.6 g of red crystalline solid upon solvent removal. Finally, this residue was crystallized from 30 ml of oxygen-free benzene to give 2.8 g of $(CH_3)_4Sn_3Fe_4(CO)_{16}$ as small, red, acicular crystals, mp 198–200° dec. *Anal.* Calcd for $C_{20}H_{12}Fe_4O_{16}Sn_3$: C, 22.1; H, 1.1; Sn, 32.7; Fe, 20.5; mol wt, 1088. Found: C, 22.4; H, 1.2; Sn, 32.5; Fe, 19.4; mol wt, 1181, 1107, 1127. The molecular weight was measured in dichloromethane solution using a vapor pressure osmometer. Recrystallization from *n*-heptane gave larger crystals suitable for the X-ray study. This recrystallized material was identical, in other respects, with the product from benzene.

The compound is moderately stable to moist air when crystalline but becomes orange after 1–2 days of exposure. The powdered solid decomposes in a few minutes. The compound is soluble in common organic solvents. These solutions rapidly deposit orange solids on air exposure.

The infrared spectrum of $(CH_3)_4Sn_3Fe_4(CO)_{16}$ in *n*-hexane shows metal–carbonyl stretching frequencies at 2062 (s), 2037 (m), 2000 (m), and 1988 (s) cm^{-1}. The proton resonance spectrum shows a single slightly asymmetric line at τ 8.92 in $CDCl_3$ and at τ 9.78 in C_6H_6 with $(CH_3)_4Si$ as an external standard. The sharp nmr spectra show the compound to be diamagnetic.

The yield of $[(CH_3)_2SnFe(CO)_4]_2$ was not determined since it could not be removed completely from the column after chromatography. However, in one reaction of $Na_2Fe(CO)_4$ (0.141 mole) and CH_3SnCl_3 (0.067 mole), water (200 ml) was added after the addition of CH_3SnCl_3, and the tetrahydrofuran was removed under vacuum. Extraction of the remaining red slurry with 650 ml of benzene, separation of the layers, and evaporation of the benzene gave a red-orange solid (10.8 g).

A 3.8-g portion of this product was crystallized twice from *n*-hexane at −78° to give 2.7 g (0.0085 mole) of bright yellow crystalline $[(CH_3)_2SnFe(CO)_4]_2$. *Anal.* Calcd for $C_6H_6FeO_4Sn$: C, 22.8; H, 1.9; Sn, 37.6; Fe, 17.7. Found: C, 23.5; H, 2.2; Sn, 37.1; Fe, 17.7. The infrared spectrum in *n*-hexane showed carbonyl bands at 2049 (s), 2004 (s), 1996 (s), 1984 (sh), 1969 (w), and 1946 (w) cm^{-1}. A sample of $[(CH_3)_2SnFe(CO)_4]_2$ prepared from $Na_2Fe(CO)_4$ and $(CH_3)_2SnCl_2$ had an identical infrared spectrum.

X-Ray Data for $(CH_3)_4Sn_3Fe_4(CO)_{16}$.—Precession and Weissenberg photographs indicated a monoclinic structure belonging to symmetry class $P2_1/c$. An *h*0*l* precession photograph taken with Mo Kα radiation was used to measure β, and an 0*kl* precision Weissenberg photograph[11] taken with Co Kα radiation, with the Straumanis film mounting, was used in measuring *b* and *c*. The value of *c* thus obtained was used to correct the measurement of *a* from the *h*0*l* precession photograph for film shrinkage. The values of the unit cell parameters are: $a = 11.04 \pm 0.02$ A, $b = 16.36 \pm 0.01$ A, $c = 19.09 \pm 0.01$ A, and $\beta = 108.5 \pm 0.2°$. The calculated density is 2.21 g/cm^3 if four molecules are assumed to occupy each unit cell. The wavelengths used in the calculations are: Co Kα_1 = 1.78890 A, Co Kα_{av} = 1.7902 A, and Mo Kα_{av} = 0.7107 A.

Because of slow disintegration of the crystals in air, and in an effort to reduce the very considerable absorption errors that would result in some reflections if a glass capillary were used, each crystal was mounted in a nitrogen-filled drybox and was then coated with a thin layer of Krylon acrylic ester resin. A polyhedral crystal with dimensions of approximately 0.015 × 0.007 × 0.008 cm was chosen for intensity measurement. The resin coating of this crystal reduced decomposition to the point where several standard reflections decayed by only about 50% during the 2 weeks of intensity measurement.

Intensity data were measured by a Picker card-controlled diffractometer with goniostat-setting cards produced with a program written by C. T. Prewitt of this laboratory. A zirconium filter and a pulse-height analyzer, set to admit some 90% of the Mo Kα peak distribution, were used to reduce the effect of Kβ and white background radiation. The moving crystal–moving counter scanning technique described by Furnas[12] was used. The counter angle, 2θ, was scanned over 2° at a speed of 1°/min. Background counts of 20 sec were taken at each end of the 2θ scan. Because of rather large thermal motion of the molecules, reflections having 2θ greater than 35° were in virtually all cases unobservable. A total of 1925 independent reflections having smaller scattering angles was measured. Each integrated reflection intensity was assigned a standard deviation (σ_I) by consideration of counting statistics in the integrated scan and the background measurements and of a 4% uncertainty in the relative scale factor applicable to *I*. If σ_I exceeded 0.5*I*, the reflection was considered unobserved and was assigned $2\sigma_I$ as a threshold value. Of the measured reflections, 941 were unobserved. Structure factor magnitudes, $|F_o|$, and their standard deviations were derived by application of the Lorentz and polarization corrections, using a program written by C. J. F.

Determination and Refinement of the Structure

Because of early uncertainty about the molecular formula, the three-dimensional Patterson map was first interpreted in terms of a trial structure for tin and iron atoms involving one square-planar and two tetrahedral tin atoms. After a cycle of least squares and a trial F_o Fourier map, however, this structure was proven to be wrong. Considerable study of the Patterson map then led to a trial structure including only the three tin atoms. Study of a Fourier map calculated after a cycle of least-squares refinement of the tin positions indicated the location of one of the iron atoms. Subsequently, the other three iron atoms were added to the model as a (distorted) tetrahedron about the central tin atom and refinement was continued. After nine least-squares cycles with three tin and four iron

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[11] C. J. Fritchie, Jr., *Acta Cryst.*, **20**, 107 (1966).

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atoms present, the R factor had been reduced from 64 to 33%. A Fourier map calculated at this time indicated the positions of 13 of the carbon and 11 of the oxygen atoms in the structure. Further cycles of least-squares and Fourier calculations established the positions of the remaining carbon and oxygen atoms. After several iterations of least-squares refinement of the complete structure, during which positional parameters were allowed to shift and in which isotropic temperature factors of $B = 3.0$ for iron and tin, 4.0 for carbon, and 4.5 for oxygen were assumed, R was reduced to 14%. Introduction and refinement of ellipsoidal temperature factors for iron and tin atoms at this stage then allowed reduction of R to 11%.

The linear absorption coefficient, μ, of this compound for Mo Kα radiation is 41.2 cm^{-1}. The crystal used in data collection was of such a shape that the transmission factor varied by about 50% for different orientations, so absorption corrections were made at this point in the refinement using a program written by C. T. Prewitt. The isotropic temperature factors of carbon and oxygen were then allowed to shift, bringing R to 8.5%. Considerable effort was expended in attempting full anisotropic refinement of the structure, because of the rather large thermal motion. The extreme thermal motion together with the large difference in scattering factors between the light carbon and oxygen atoms and the heavier tin and iron atoms had made convergence slow and had made considerable manual readjustment of parameters necessary throughout the refinement. These factors remained major difficulties, continually producing very elongated and unrealistically shaped thermal ellipsoids and, in fact, consistently causing some of the thermal quadratic forms to become nonpositive definite. Although the estimated standard deviations for positional parameters were at their lowest values and R was reduced to 6.4%, this method of refinement was abandoned. All positional parameters, anisotropic temperature factors for tin and iron atoms, and isotropic temperature factors for carbon and oxygen atoms were allowed to shift for several cycles. Convergence was reached with R = 6.9%. On the final cycle, each positional parameter's shift averaged 0.04 times its own σ and only three were as large as 0.25σ.

Atomic positional parameters and light-atom thermal parameters at the conclusion of refinement are given in Table I. Anisotropic temperature parameters are in Table II. The observed and the final calculated structure factors appear in Table III.

For the refinement, a modification of the block-diagonal Gantzel–Sparks–Trueblood least-squares program[13] was used, with refinement based on minimization of $\Sigma w||F_o| - |F_c||^2$. Each $|F_o|$ was weighted with the squared reciprocal of its assigned standard deviation: $w = 1/\sigma_F^2$. Unobserved reflections were omitted from the least-squares calculations and from R on each cycle in which $|F_o|$ exceeded $|F_c|$. On the final cycle, only 61 of the 941 unobserved reflections had calculated magnitudes exceeding their observational thresholds. The final value of the "goodness of fit" index, $(\Sigma w|\Delta F|^2/(n - s))^{1/2}$, is 1.26. Atomic scattering factors used in

TABLE I

FINAL ATOMIC POSITIONAL AND ISOTROPIC THERMAL PARAMETERS[a]

Atom	x	y	z	
Sn(1)	0.4199 (3)	0.0614 (2)	0.1331 (2)	
Sn(2)	0.2601 (3)	0.0728 (2)	0.2554 (2)	
Sn(3)	0.1062 (3)	0.0812 (2)	0.3843 (2)	
Fe(4)	0.4843 (6)	0.1436 (4)	0.2566 (3)	
Fe(5)	0.2003 (6)	−0.0088 (3)	0.1224 (3)	
Fe(6)	0.2745 (5)	−0.0272 (4)	0.3748 (3)	
Fe(7)	0.0770 (6)	0.1773 (4)	0.2694 (3)	
Atom	x	y	z	B
C(8)	0.406 (4)	0.153 (3)	0.039 (2)	5.4 (12)
C(9)	0.566 (4)	−0.030 (3)	0.136 (2)	6.5 (13)
C(10)	0.177 (4)	0.144 (3)	0.490 (2)	5.0 (11)
C(11)	−0.070 (4)	0.019 (3)	0.384 (2)	6.2 (12)
C(12)	0.384 (4)	0.223 (2)	0.204 (2)	3.9 (10)
O(13)	0.311 (3)	0.275 (2)	0.176 (2)	7.1 (10)
C(14)	0.549 (5)	0.200 (3)	0.341 (3)	7.6 (13)
O(15)	0.576 (3)	0.236 (2)	0.393 (2)	8.0 (9)
C(16)	0.542 (4)	0.058 (3)	0.295 (2)	6.4 (12)
O(17)	0.588 (3)	−0.011 (2)	0.322 (2)	8.2 (9)
C(18)	0.611 (4)	0.168 (3)	0.229 (2)	5.0 (11)
O(19)	0.692 (3)	0.191 (2)	0.204 (2)	8.1 (9)
C(20)	0.299 (4)	−0.083 (3)	0.181 (2)	4.8 (11)
O(21)	0.360 (2)	−0.132 (2)	0.216 (1)	4.6 (7)
C(22)	0.045 (4)	−0.055 (3)	0.109 (2)	6.1 (12)
O(23)	−0.050 (2)	−0.083 (2)	0.102 (1)	5.2 (7)
C(24)	0.051 (3)	0.086 (3)	0.087 (2)	6.4 (12)
O(25)	0.095 (3)	0.147 (2)	0.063 (2)	6.5 (8)
C(26)	0.206 (4)	−0.052 (3)	0.040 (2)	4.7 (11)
O(27)	0.228 (3)	−0.073 (2)	−0.013 (2)	8.0 (9)
C(28)	0.145 (4)	−0.077 (3)	0.307 (2)	4.9 (10)
O(29)	0.057 (2)	−0.099 (2)	0.264 (1)	5.8 (8)
C(30)	0.390 (5)	−0.109 (3)	0.370 (3)	7.3 (13)
O(31)	0.473 (3)	−0.149 (2)	0.377 (1)	6.5 (8)
C(32)	0.381 (4)	0.050 (3)	0.420 (2)	5.0 (11)
O(33)	0.460 (3)	0.098 (2)	0.453 (2)	6.4 (8)
C(34)	0.246 (4)	−0.072 (3)	0.452 (2)	5.6 (12)
O(35)	0.237 (3)	−0.102 (2)	0.504 (2)	7.4 (9)
C(36)	0.204 (4)	0.226 (3)	0.328 (2)	6.3 (12)
O(37)	0.290 (3)	0.266 (2)	0.364 (1)	6.6 (8)
C(38)	0.040 (5)	0.239 (3)	0.191 (3)	7.5 (13)
O(39)	0.021 (3)	0.282 (3)	0.141 (2)	8.1 (9)
C(40)	−0.024 (5)	0.106 (3)	0.222 (2)	6.9 (13)
O(41)	−0.097 (3)	0.052 (2)	0.189 (1)	6.8 (8)
C(42)	−0.037 (5)	0.219 (3)	0.305 (2)	7.0 (13)
O(43)	−0.103 (3)	0.258 (2)	0.333 (2)	7.0 (8)

[a] Figures are in fractions of the unit cell edge. Standard deviations are in parentheses.

TABLE II

TIN AND IRON ANISOTROPIC THERMAL PARAMETERS[a]

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Sn(1)	132 (3)	043 (2)	034 (1)	−005 (5)	092 (3)	−009 (2)
Sn(2)	070 (3)	022 (1)	019 (1)	−002 (4)	017 (2)	−006 (2)
Sn(3)	103 (3)	028 (1)	026 (1)	010 (4)	047 (3)	−003 (2)
Fe(4)	076 (7)	037 (3)	037 (2)	−017 (8)	045 (6)	002 (5)
Fe(5)	119 (8)	025 (3)	022 (2)	026 (9)	016 (7)	−005 (4)
Fe(6)	091 (7)	037 (3)	020 (2)	−005 (9)	040 (6)	002 (4)
Fe(7)	122 (7)	028 (3)	028 (2)	020 (8)	059 (6)	−006 (4)

[a] The temperature expression is of the form $\exp[-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{12}hk + B_{13}hl + B_{23}kl)]$. Figures have been multiplied by 10^4. Standard deviations are in parentheses.

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(13) International Union of Crystallography, World List of Crystallographic Computer Programs, 1st ed, Sept 1962, Program 384.

TABLE III

OBSERVED AND CALCULATED STRUCTURE FACTORS[a]

k	l	$|F_o|$	F_c
H = 0			
0	6	208	-204
0	8	558	558
0	10	111	-108
0	12	134	-126
0	14	122	-120
-1	5	-43	-18
-1	6	-43	2
-1	7	83	90
-1	8	105	101
-1	9	112	-117
-1	10	69	-70
-1	11	-48	7
-1	12	-49	13
-1	13	-51	-9
-1	14	72	-72
-1	15	84	95
-2	5	233	216
-2	6	98	-98
-2	7	262	-260
-2	8	321	317
-2	9	219	210
-2	10	-47	-25
-2	11	60	-46
-2	12	129	-126
-2	13	61	53
-2	14	61	-58
-3	4	55	-69
-3	5	-44	-14
-3	6	-46	-55
-3	7	95	93
-3	8	149	149
-3	9	75	76
-3	10	-47	-32
-3	11	89	-71
-3	12	69	-65
-3	13	-53	33
-3	14	-71	-36
-4	3	125	131
-4	4	362	-335
-4	5	116	-111
-4	6	210	206
-4	7	194	-182
-4	8	77	-58
-4	9	157	152
-4	10	-49	45
-4	11	-49	-1
-4	12	80	-88
-4	13	-52	21
-4	14	-55	39
-5	1	51	58
-5	2	-46	-7
-5	3	46	38
-5	4	137	-127
-5	5	69	-77
-5	6	-46	11
-5	7	-47	16
-5	8	60	56
-5	9	88	86
-5	10	57	53
-5	11	-52	-18
-5	12	79	-69
-5	13	79	-66
-6	0	307	-313
-6	1	108	107
-6	2	-46	28
-6	3	84	-95
-6	4	99	99
-6	5	-47	-4
-6	6	-50	26
-6	7	-48	-35
-6	8	110	-108
-6	9	-50	-3
-6	10	-50	10
-6	11	-52	-12
-6	12	-53	-24
-6	13	-55	-2
-7	1	-48	38
-7	2	131	142
-7	3	-47	-12
-7	4	-47	20
-7	5	54	-52
-7	6	142	-139
-7	7	87	-68
-7	8	-50	57
-7	9	62	60
-7	10	-52	30
-7	11	-54	-1
-7	12	-56	40
-8	0	236	-236
-8	1	102	-86
-8	2	117	117
-8	3	149	-141
-8	4	-59	-31
-8	5	108	92
-8	6	110	102
-8	7	137	117
-8	8	141	-136
-8	9	74	-57
-8	10	81	71
-8	11	113	-98
-8	12	-58	-12
-9	1	67	-52
-9	2	83	95
-9	3	-58	12
-9	4	-58	28
-9	5	-64	20
-9	6	-61	-65
-9	7	95	-103
-9	8	-64	-56
-9	9	110	78
-9	10	72	65
-9	11	-65	-14
-10	0	-65	-59
-10	1	312	-304
-10	2	-61	48
-10	3	-60	-17
-10	4	-60	-45
-10	5	-60	19
-10	6	-71	7
-10	7	248	234
-10	8	-64	-12
-10	9	202	-189
-10	10	86	69
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-2	-3	-60	1
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-2	-5	-53	-49
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-4	5	-58	-27
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-4	-11	-55	-8
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-5	0	-52	-25
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-6	3	75	66
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-6	4	-62	6
-6	-4	-55	-35
-6	5	-60	11
-6	-5	86	70
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-6	-11	-57	5
-6	-12	-60	-14
-7	0	61	60
-7	1	-58	-48
-7	-1	70	64
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-7	4	-57	-29
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-8	-5	101	106
-8	-6	-63	-25
-8	-7	96	-99
-8	-8	-67	-36
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0	-6	156	-153
0	-8	-63	48
0	-10	-63	23
-1	0	-61	-17
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-1	-1	74	-58
-1	2	-63	2
-1	-2	-60	36
-1	3	74	80
-1	-3	73	63
-1	-4	-57	-24
-1	-5	75	57
-1	-6	-68	21
-1	-7	69	-69
-1	-8	-62	-12
-1	-9	-64	-25
-1	-10	-64	0
-1	-11	79	44
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-2	1	133	127
-2	-1	-60	-4
-2	2	-64	-36
-2	-2	-58	-32
-2	-3	-59	-17
-2	-4	102	78
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-2	-6	106	-88
-2	-7	157	139
-2	-8	-62	44
-2	-9	-63	42
-2	-10	-65	-24
-2	-11	-69	-28
-3	0	-61	8
-3	1	109	-108
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-3	-7	-64	-83
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-4	-5	189	-193
-4	-6	116	106
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-6	-4	-63	-64
-6	-5	69	-58
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-6	-7	-64	43
H = 9			
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0	-6	142	126
0	-8	-75	-63
-1	-2	-72	3
-1	-3	127	-113
-1	-4	-73	50
-1	-5	136	141
-1	-6	94	-65
-1	-7	-75	-5
-1	-8	-75	3
-2	-3	83	-90
-2	-4	-73	13
-2	-5	120	102
-2	-6	-75	44
-2	-7	-75	-11
-3	-5	-74	6

[a] Each group of four columns contains *k*, *l*, $|F_o|$, and F_c. Negative figures in the $|F_o|$ column are threshold values for unobserved reflections. Missing low-order reflections were too close to the direct beam to be measured.

the refinement are those given in "International Tables for X-ray Crystallography," Vol. III, 2nd ed, with the real anomalous scattering correction added.

Discussion

The schematic molecular structure and the crystallographic numbering system of $(CH_3)_4Sn_3Fe_4(CO)_{16}$ are shown in Figure 1. The molecule may be described as having a spiro structure of idealized $\bar{4}2m$ (D_{2d}) symmetry. Figure 2 shows the structure in perspective. The three tin atoms fall amost in a line and are bridged by four $Fe(CO)_4$ groups in such a manner that the central tin is surrounded by a somewhat elongated tetrahedron of iron atoms. Two methyl groups complete the tetrahedral coordination sphere of each outer tin. The bond angles about these outer tin atoms deviate only slightly from the regular tetrahedral value. Each iron is in an irregular octahedral environment of two tin atoms and four carbonyl groups. The groups of four atoms—Sn(1), Fe(4), Sn(2), and Fe(5), and Sn(2), Fe(6), Sn(3), and Fe(7)—form nearly perpendicular planes. The angle between the least-squares

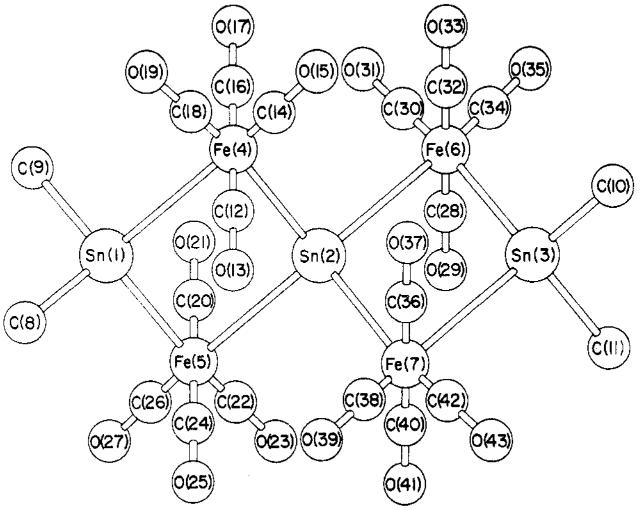

Figure 1.—Schematic molecular structure and crystallographic numbering system.

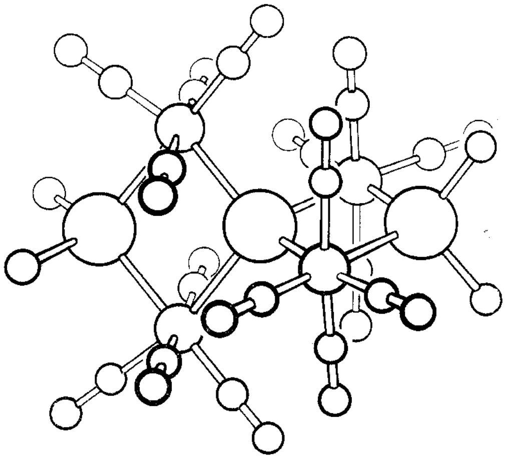

Figure 2.—The $(CH_3)_4Sn_3Fe_4(CO)_{16}$ molecule. In order of increasing size, the atoms are carbon and oxygen, iron, and tin.

planes is 89.6° and the maximum deviation of an atom from its plane is 0.03 A. Bond distances and angles in the tin–iron framework are shown in Figure 3. Those involving lighter atoms are listed in Tables IV and V, respectively.

The average molecular structure is compared in

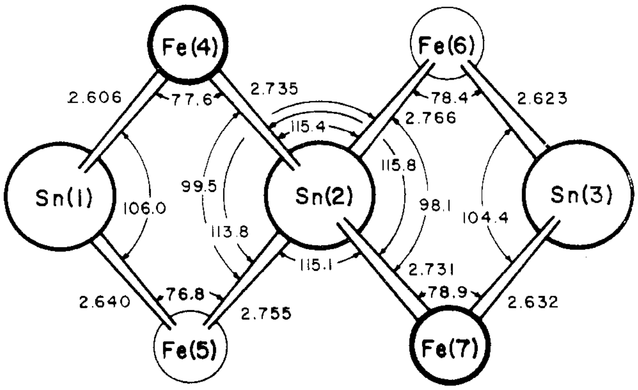

Figure 3 —Bond distances and angles in the Sn_3Fe_4 framework. Average standard deviations are 0.010 A and 0.5°.

TABLE IV

BOND DISTANCES INVOLVING LIGHT ATOMS[a]

Bond	Length, A	Bond	Length, A
Sn(1)–C(8)	2.31	Fe(7)–C(40)	1.68
Sn(1)–C(9)	2.20	Fe(7)–C(42)	1.75
Sn(3)–C(10)	2.18	C(12)–O(13)	1.18
Sn(3)–C(11)	2.19	C(14)–O(15)	1.11
Fe(4)–C(12)	1.79	C(16)–O(17)	1.28
Fe(4)–C(14)	1.80	C(18)–O(19)	1.20
Fe(4)–C(16)	1.61	C(20)–O(21)	1.12
Fe(4)–C(18)	1.70	C(22)–O(23)	1.11
Fe(5)–C(20)	1.77	C(24)–O(25)	1.18
Fe(5)–C(22)	1.83	C(26)–O(27)	1.17
Fe(5)–C(24)	1.71	C(28)–O(29)	1.11
Fe(5)–C(26)	1.74	C(30)–O(31)	1.10
Fe(6)–C(28)	1.80	C(32)–O(33)	1.20
Fe(6)–C(30)	1.87	C(34)–O(35)	1.15
Fe(6)–C(32)	1.76	C(36)–O(37)	1.19
Fe(6)–C(34)	1.77	C(38)–O(39)	1.15
Fe(7)–C(36)	1.69	C(40)–O(41)	1.22
Fe(7)–C(38)	1.75	C(42)–O(43)	1.21

[a] Average standard deviations are 0.05 A for Fe–C and Sn–C bonds and 0.07 A for C–O bonds.

TABLE V

BOND ANGLES INVOLVING LIGHT ATOMS[a]

Atoms	Angle, deg	Atoms	Angle, deg
Fe(4)–Sn(1)–C(8)	107	C(28)–Fe(6)–C(32)	160
Fe(4)–Sn(1)–C(9)	110	C(28)–Fe(6)–C(34)	96
Fe(5)–Sn(1)–C(8)	113	C(30)–Fe(6)–Sn(2)	105
Fe(5)–Sn(1)–C(9)	111	C(30)–Fe(6)–Sn(3)	176
Fe(8)–Sn(1)–C(9)	109	C(30)–Fe(6)–C(32)	100
Fe(6)–Sn(3)–C(10)	109	C(30)–Fe(6)–C(34)	93
Fe(6)–Sn(3)–C(11)	110	C(32)–Fe(6)–Sn(2)	80
Fe(7)–Sn(3)–C(10)	114	C(32)–Fe(6)–Sn(3)	82
Fe(7)–Sn(3)–C(11)	114	C(32)–Fe(6)–C(34)	99
C(10)–Sn(3)–C(11)	106	C(34)–Fe(6)–Sn(2)	163
C(12)–Fe(4)–Sn(1)	86	C(34)–Fe(6)–Sn(3)	84
C(12)–Fe(4)–Sn(2)	85	C(36)–Fe(7)–Sn(2)	83
C(12)–Fe(4)–C(14)	97	C(36)–Fe(7)–Sn(3)	83
C(12)–Fe(4)–C(16)	165	C(36)–Fe(7)–C(38)	102
C(12)–Fe(4)–C(18)	95	C(36)–Fe(7)–C(40)	164
C(14)–Fe(4)–Sn(1)	173	C(36)–Fe(7)–C(42)	96
C(14)–Fe(4)–Sn(2)	109	C(38)–Fe(7)–Sn(2)	105
C(14)–Fe(4)–C(16)	93	C(38)–Fe(7)–Sn(3)	174
C(14)–Fe(4)–C(18)	92	C(38)–Fe(7)–C(40)	90
C(16)–Fe(4)–Sn(1)	85	C(38)–Fe(7)–C(42)	96
C(16)–Fe(4)–Sn(2)	82	C(40)–Fe(7)–Sn(2)	84
C(16)–Fe(4)–C(18)	96	C(40)–Fe(7)–Sn(3)	85
C(18)–Fe(4)–Sn(1)	82	C(40)–Fe(7)–C(42)	93
C(18)–Fe(4)–Sn(2)	159	C(42)–Fe(7)–Sn(2)	159
C(20)–Fe(5)–Sn(1)	83	C(42)–Fe(7)–Sn(3)	80
C(20)–Fe(5)–Sn(2)	80	Fe(4)–C(12)–O(13)	171
C(20)–Fe(5)–C(22)	100	Fe(4)–C(14)–O(15)	173
C(20)–Fe(5)–C(24)	158	Fe(4)–C(16)–O(17)	176
C(20)–Fe(5)–C(26)	96	Fe(4)–C(18)–O(19)	173
C(22)–Fe(5)–Sn(1)	177	Fe(5)–C(20)–O(21)	177
C(22)–Fe(5)–Sn(2)	106	Fe(5)–C(22)–O(23)	180
C(22)–Fe(5)–C(24)	99	Fe(5)–C(24)–O(25)	167
C(22)–Fe(5)–C(26)	91	Fe(5)–C(26)–O(27)	169
C(24)–Fe(5)–Sn(1)	79	Fe(6)–C(28)–O(29)	171
C(24)–Fe(5)–Sn(2)	83	Fe(6)–C(30)–O(31)	167
C(24)–Fe(5)–C(26)	96	Fe(6)–C(32)–O(33)	175
C(26)–Fe(5)–Sn(1)	87	Fe(6)–C(34)–O(35)	175
C(26)–Fe(5)–Sn(2)	164	Fe(7)–C(36)–O(37)	173
C(28)–Fe(6)–Sn(2)	81	Fe(7)–C(38)–O(39)	176
C(28)–Fe(6)–Sn(3)	86	Fe(7)–C(40)–O(41)	178
C(28)–Fe(6)–C(30)	92	Fe(7)–C(42)–O(43)	170

[a] The average standard deviation is about 2° for angles involving one light atom and 4° for those involving two.

Figure 4 with a hypothetical, more regular structure obtained by averaging appropriate distances and angles to give each half of the $Sn_3Fe_4(CO)_{16}$ framework D_{2h} symmetry. A number of significant deviations from regularity are evident. The interior Fe–Sn bond of 2.747 ± 0.008 A is much longer than the exterior bond of 2.625 ± 0.008 A. The interior Fe–Sn–Fe angle within an iron–tin ring is 98.8°, compared with the corresponding terminal angle of 105.2°. If the carbonyl groups are considered to be equatorial and axial in regard to their respective Fe_2Sn_2 planes, each equatorial carbonyl group is seen not only to be displaced with its reference iron atom parallel to the molecular axis, but to be rotated about the iron atom toward the terminal tin as well. The interior and terminal tin–iron–equatorial carbon angles are 106 and 84°, respectively. The axial carbonyl groups above and those below each Fe_2Sn_2 plane also bend directly toward each other, reducing the corresponding C–Fe–C angle from 180 to 162°.

For the sake of mnemonic and rationalizing purposes, the observed molecular configuration can be regarded as a balance between a tendency to maintain regular octahedral and tetrahedral bond angles and "normal" bond lengths and a tendency for nonbonded atoms to achieve "normal" van der Waals contacts. In the Fe_2Sn_2 ring, some compromise with the ideal angles of 90 and 109.5° is obviously necessary. The average regular structure in Figure 4 has an SnFeSn angle of 77.9°, an FeSnFe angle of 102.0°, and an Fe–Sn bond length of 2.69 A. The octahedral angle seems thus to accommodate more distortion (12.1°) than the tetrahedral angle (7.5°) in this molecule. Further deviations from the regular structure appear primarily to be associated with relief of steric strain between the axial carbonyl groups on one side of the central tin atom and the equatorial ones on the opposite side. The $O \cdots O$ contact, even with the observed distortion, is 2.98 A, much shorter than the minimal intermolecular values of 3.11–3.15 A usually found in carbonyl compounds (see below). The inward bending of axial carbonyl groups can be similarly understood as an attempt to reach an ideal van der Waals contact. Exactly parallel carbonyl groups would be separated by 4.17 A. Bending reduces the average $C \cdots O$ distance to 3.63 A and the average $O \cdots O$ distance to 3.32 A.

The values of 2.625 and 2.747 A are among the first determinations of the iron–tin bond length and can be compared with the distance of 2.53 A recently reported in $Sn[Fe(CO)_4]_4$.[14] The latter value, like the length of the Mn–Sn bond in $(C_6H_5)_3SnMn(CO)_4P(C_6H_5)_3$,[15] is about 0.1 A shorter than the sum of covalent radii.[16] Both steric strain of the type discussed above and also possible cross-ring repulsion are absent in $Sn[Fe(CO)_4]_4$ and $(C_6H_5)_3SnMn(CO)_4P(C_6H_5)_3$. Thus the shorter lengths may be closer to "normal" single-bond values.

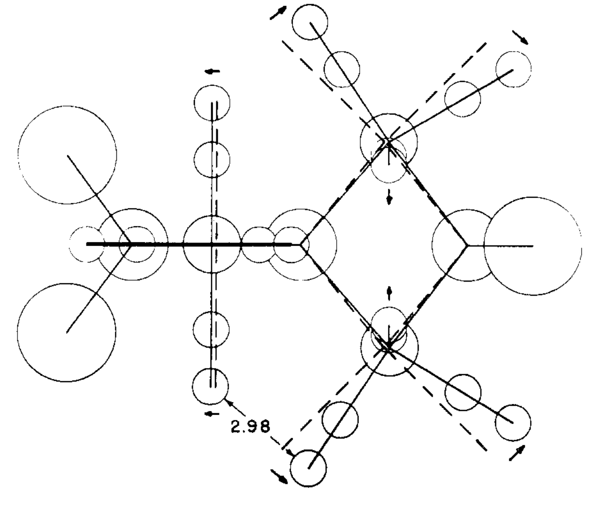

Figure 4.—Average molecular structure (solid lines) compared with a hypothetical structure of greater regularity (dashed lines). The dashed structure contains $Fe_2Sn_2(CO)_8$ fragments of mmm (D_{2h}) symmetry. Heavy arrows indicate displacement of the $Fe(CO)_4$ groups (left) and rotation of equatorial carbonyl groups and inward motion of axial carbonyl groups (right). One of the short axial–equatorial $O \cdots O$ contacts of 2.98 A is shown. In order of increasing size, the circles are carbonyl carbon and oxygen, iron, tin, and methyl carbon.

The poorly determined average Sn–C bond length of 2.22 ± 0.03 A is about the same as that of 2.18 A found in halogen-substituted stannanes[17] and the value of 2.143 A found in H_3SnCH_3.[17] The iron–carbon bonds average 1.75 ± 0.015 A in length, and the carbon–oxygen bond lengths average 1.17 ± 0.012 A, slightly shorter and longer, respectively, than the corresponding averages of 1.79 ± 0.02 and 1.12 ± 0.02 A in $Fe(CO)_5$[18] and of 1.76 ± 0.02 and 1.17 ± 0.02 A in $S_2Fe_2(CO)_6$.[19]

Most intermolecular contacts are of the type $O \cdots O$, with the shortest in the range 3.14–3.30 A. Similar contacts in $Fe(CO)_5$,[18] $S_2Fe_2(CO)_6$,[19] and $Fe_2(CO)_9$[20] fall in the range 3.11–3.15 A. The shortest intermolecular $C \cdots O$ contact is 3.24 A.

The compound $[(CH_3)_2SnFe(CO)_4]_2$ probably has a molecular structure related to that of $(CH_3)_4Sn_3Fe_4(CO)_{16}$, consisting of two tin tetrahedra sharing a common edge with a planar Fe–Sn–Fe–Sn ring and equal Fe–Sn bond lengths. Another closely related tin–iron compound is $Sn[Fe(CO)_4]_4$.[14,21] A butyl analog of $(CH_3)_4Sn_3Fe_4(CO)_{16}$ has been prepared from $(n\text{-}C_4H_9)_3SnCl$ and $Fe(CO)_5$.[14,21]

The preparation of $(CH_3)_4Sn_3Fe_4(CO)_{16}$ from CH_3SnCl_3 demonstrates the lability of alkyl groups bonded to tin. This lability has long been recognized in the preparation of alkyltin halides from R_4Sn and $SnCl_4$.[22] The mechanism of methyl group transfer in the reaction of CH_3SnCl_3 and $Na_2Fe(CO)_4$ is unknown.

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