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# The Molecular and Crystal Structure of Dichlorobis(dicarbonyl-π-cyclopentadienyliron)tin(IV), $Cl_2Sn[\pi\text{-}C_5H_5Fe(CO)_2]_2$

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The crystal and molecular structure of $Cl_2Sn[\pi\text{-}C_5H_5Fe(CO)_2]_2$ has been determined by X-ray diffraction. Three-dimensional isotropic least-squares refinement resulted in an unweighted discrepancy index of 11.2%. The compound crystallizes in the monoclinic space group C2/c with $a$ = 14.98 A, $b$ = 7.63 A, $c$ = 15.18 A, and $\beta$ = 94° 27′. The discrete molecules possess crystallographic $C_2$ symmetry. The two chlorine atoms and two iron atoms are bonded to the central tin atom in a distorted tetrahedral arrangement; the Fe–Sn–Fe′ and Cl–Sn–Cl′ bond angles are 128.6 and 94.1°, respectively. The Sn–Fe and Sn–Cl bond lengths are 2.492 and 2.43 A, respectively. Each iron atom exhibits an approximately octahedral environment with the cyclopentadienyl moiety occupying three coordination sites; the tin and two carbonyls complete the octahedral coordination. The molecular features are compared to those of related molecules and are shown to be in agreement with the observed infrared spectrum.

## Introduction

Although a large number of metal cluster compounds that contain tin atoms have been prepared,[3] relatively few solid-state structures have been reported.[4-10] We have undertaken the crystal structures of two closely related compounds, $[\pi\text{-}C_5H_5Mo(CO)_3]_2Sn[\pi\text{-}C_5H_5Fe(CO)_2]_2$[11] and $Cl_2Sn[\pi\text{-}C_5H_5Fe(CO)_2]_2$.[12] This paper describes results obtained for the latter compound.

## Experimental Section

Crystals of $Cl_2Sn[\pi\text{-}C_5H_5Fe(CO)_2]_2$ were supplied by Dr. S. Dighe of Professor M. Orchin's laboratory at the University of Cincinnati.

**Single Crystal X-Ray Data.**—Multiple film equiinclination Weissenberg data were collected for the reciprocal levels $h0l$ through $h8l$ with Mo K$\alpha$ radiation ($\lambda$ 0.7107 A). The intensities of 908 independent diffraction maxima were estimated visually and were corrected for Lorentz-polarization effects. The crystal chosen for data collection was 0.74 mm in length and 0.20 × 0.19 mm in width. Because the maximum value of $\mu R$ is 0.34 ($\mu$ = 34.4 cm$^{-1}$), absorption corrections were neglected. Lattice constants were determined from $hk0$ and $0kl$ precession photographs which were calibrated by superposition of a sodium chloride diffraction pattern ($a$ = 5.627 A). The angle $\beta$ was determined from the $h0l$ Weissenberg photograph.

**Unit Cell and Space Group.**—The crystals of $Cl_2Sn[\pi\text{-}C_5H_5Fe(CO)_2]_2$ are monoclinic with lattice parameters $a$ = 14.98 ± 0.03 A, $b$ = 7.63 ± 0.02 A, $c$ = 15.18 ± 0.03 A, and $\beta$ = 94° 27′ ± 20′. The volume of the unit cell is 1729 A$^3$. The experimental density of 2.02 g/cc agrees well with the calculated density of 2.08 g/cc based on four molecules per unit cell. Systematic extinctions of $hkl$ reflections for $h + k$ odd and $h0l$ reflections for $l$ odd indicate the probable space group $C_c$ ($C_s^4$) or C2/c ($C_{2h}^6$). The latter space group was confirmed by the solution of the structure. The tin atoms are located on twofold axes corresponding to sets of fourfold positions (4e) and the remaining atoms occupy general eightfold positions (8f). The positions are: 4e (0, 0, 0; $^1/_2$, $^1/_2$, 0) ± (0, $y$, $^1/_4$) and 8f (0, 0, 0; $^1/_2$, $^1/_2$, 0) ± ($x$, $y$, $z$; $x$, $-y$, $^1/_2 + z$).[13] The total number of electrons per unit cell, $F(000)$, is 1048.

**Determination of the Structure.**—A three-dimensional Patterson function[14] computed on the IBM 1620 computer provided trial coordinates for the tin and iron atoms. A structure factor calculation with these trial coordinates resulted in an unweighted discrepancy factor, $R_1 = [\Sigma||F_o| - |F_c||/\Sigma|F_o|] \times 100$ of 27%. The phases from this structure factor calculation were used to calculate a three-dimensional electron density synthesis. All other atoms (except hydrogen atoms) were located readily from peaks appearing in the synthesis. Addition of these atoms to the structure factor calculation lowered the discrepancy index, $R_1$, to 21%. A full-matrix isotropic least-squares refinement[15] of the twelve nonhydrogen atoms in the asymmetric unit gave a final discrepancy index, $R_1$, of 11.2% and a weighted discrepancy index, $R_2 = [\Sigma w||F_o| - |F_c||^2/\Sigma w|F_o|^2]^{1/2} \times 100$, of 13.2%. All parameter shifts on the last cycle were less than 2% of the corresponding standard deviation. Unit weights were used in the final refinement although two different variable weight schemes were tested. Variable weights in the first scheme were assigned as follows: $\sqrt{w} = 20/F_o$ if $I_o > \sqrt{20}I_{\min}$; $\sqrt{w} = I_o^2/F_oI_{\min}^2$ if $I_o \leqslant \sqrt{20}I_{\min}$. This scheme resulted in $R_1$ = 14.5% and $R_2$ = 12.7%. Variable weights in the second scheme were assigned as follows: $\sqrt{w} = 5/F_o$ if $I_o \leqslant 2I_{\min}$; $\sqrt{w} = 10/F_o$ if $I_o \geqslant (2I_{\min})^2$; and $\sqrt{w} = 15/F_o$ if $2I_{\min} < I_o < (2I_{\min})^2$. This scheme gave $R_1$ = 13.1% and $R_2$ = 14.7%. Thermal parameters for Sn, Fe, and Cl were 0.14, 0.96, and 4.1 with the variable weights of the first scheme and 0.42, 1.2, and 3.9, respectively, with the weights of the second scheme. Carbonyl carbon atoms ($C_1$ and $C_2$) yielded values of 2.0 and 6.7 in the first scheme and 1.4 and 4.3 in the second scheme; the cyclopentadienyl carbons ($C_3$–$C_7$) had thermal parameters that ranged from 1.0 to 3.7 and 1.0 to 5.3 in the first and second schemes, respectively. Carbonyl oxygen atoms ($O_1$ and $O_2$) gave thermal parameters of 3.9 and 7.5 in the first scheme and 4.1 and 6.7 in the second scheme. Positional parameters changed only slightly. Bond lengths and interatomic angles calculated from the refined models of the three weighting schemes were identical within the standard deviations assigned. The refinement with unit weights has

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been reported here since the final thermal parameters for atoms in similar chemical environments were most nearly the same with this scheme of weights (Table I).

TABLE I

ATOMIC PARAMETERS WITH STANDARD DEVIATIONS[a]

| Atom | x | y | z | B, A² |
|---|---|---|---|---|
| Sn | 0.0000 (—) | 0.0971 (4) | 0.2500 (—) | 0.26 (6) |
| Fe | 0.1127 (3) | 0.2388 (6) | 0.3572 (3) | 1.3 (1) |
| Cl | 0.0785 (7) | −0.1203 (16) | 0.1682 (7) | 3.9 (2) |
| $C_1$ | 0.140 (2) | 0.031 (5) | 0.402 (2) | 2.7 (7) |
| $O_1$ | 0.158 (2) | −0.098 (5) | 0.427 (2) | 5.2 (7) |
| $C_2$ | 0.189 (2) | 0.241 (5) | 0.274 (2) | 2.8 (7) |
| $O_2$ | 0.232 (2) | 0.244 (5) | 0.220 (2) | 6.4 (9) |
| $C_3$ | 0.079 (2) | 0.506 (5) | 0.357 (2) | 2.0 (6) |
| $C_4$ | 0.010 (2) | 0.414 (6) | 0.383 (2) | 3.2 (7) |
| $C_5$ | 0.037 (2) | 0.316 (5) | 0.459 (2) | 2.6 (6) |
| $C_6$ | 0.133 (2) | 0.354 (5) | 0.478 (2) | 3.4 (8) |
| $C_7$ | 0.161 (2) | 0.475 (5) | 0.416 (2) | 2.2 (6) |

[a] Numbers in parentheses here and in succeeding tables are standard deviations in the least significant digits. (—) indicates a fixed parameter.

Scattering factors for carbon and oxygen were from Berghuis, *et al.*,[16] for chlorine from Dawson,[17] for iron from Freeman and Watson,[18] and for tin from Thomas and Umeda.[19] Final positional and thermal parameters with their standard deviations are given in Table I. The output from the final least-squares cycle was used to calculate a three-dimensional difference Fourier map. Appreciable anisotropic thermal motion was observed around the tin, iron, chlorine, and oxygen atoms but no other anomalies were observed. Anisotropic thermal parameters would undoubtedly reduce the discrepancy index but this further refinement was not performed since multiple scale factors were used in the isotropic refinement. Table II lists the observed and calculated structure factors; the eleven most intense reflections, indicated by asterisks, were removed from the refinement because of extinction. None of the unobserved maxima accessible on the films had a calculated intensity greater than the minimum observed intensity. Unobserved data were not included in the structure refinement. Least-squares refinement[15] and bond distances and interatomic angles[20] were calculated on an IBM 7040 16K computer.

## Results and Discussion

Dichlorobis(dicarbonyl-π-cyclopentadienyliron)tin(IV) consists of discrete molecules (Figure 1) of crystallographically required point group symmetry 2 ($C_2$). The two chlorine atoms and two iron atoms are bonded to the central tin atom in a distorted tetrahedral arrangement. Each iron atom exhibits an approximately octahedral environment with the cyclopentadienyl moiety occupying three coordination sites; the tin and two carbonyls are located at the three remaining sites.

The most unusual features of the molecule are the bond lengths and interatomic angles associated with the coordination environment of the tin atom (bond lengths and interatomic angles are given in Tables III and IV, respectively). The Sn–Fe bond length of 2.492 A is *shorter* than any previously reported tin to transition metal atom bond. Bond distances of 2.53 and 2.536 A are found in $Sn[Fe(CO)_4]_4$[7] and $\pi\text{-}C_5H_5Fe(CO)_2Sn(C_6H_5)_3$;[5] in the compound $(CH_3)_4Sn_3[Fe(CO)_4]_4$,[8] two different Sn–Fe bond lengths of 2.747 and 2.625 A are observed. The Sn–Fe bond distance is also shorter than the tin to transition metal bond lengths of 2.674, 2.70, 2.63, and 2.54 A reported in $(C_6H_5)_3SnMn(CO)_5$,[9]

TABLE II

OBSERVED AND CALCULATED STRUCTURE FACTORS SCALED TO $F(000)$

| H,K= | L | FOBS | FCAL |
|---|---|---|---|
| 0, 0 | 2 | 441 | -501 |
| | 4 | 148 | -88 |
| | 6 | 159 | -136 |
| | 8 | 391 | 398 |
| | 10 | 475 | -482 |
| | 12 | 279 | 248 |
| | 18 | 204 | -249 |
| | 20 | 200 | 189 |
| 2, 0 | 2 | 289 | 239 |
| | 4 | 333 | 304 |
| | 6 | 324 | -303 |
| | 8 | 404 | 392 |
| | 10 | 202 | -182 |
| | 14 | 230 | -226 |
| | 16 | 326 | 357 |
| | 18 | 197 | -225 |
| -2, 0 | 0 | 472 | 509 |
| | 2 | 646 | -839* |
| | 4 | 140 | -104 |
| | 6 | 181 | 121 |
| | 8 | 254 | 231 |
| | 10 | 476 | -521 |
| | 12 | 409 | 412 |
| | 14 | 265 | -250 |
| | 20 | 210 | 262 |
| 4, 0 | 2 | 470 | -481 |
| | 4 | 631 | 692* |
| | 6 | 517 | -590* |
| | 8 | 271 | 252 |
| | 10 | 121 | 59 |
| | 12 | 156 | 115 |
| | 14 | 367 | -406 |
| | 16 | 311 | 286 |
| -4, 0 | 0 | 168 | -139 |
| | 2 | 324 | -319 |
| | 4 | 548 | 682* |
| | 6 | 425 | -391 |
| | 8 | 135 | 102 |
| | 10 | 234 | -228 |
| | 12 | 249 | 240 |
| | 14 | 278 | -276 |
| | 16 | 181 | 173 |
| | 22 | 214 | -216 |
| 6, 0 | 2 | 392 | -393 |
| | 4 | 493 | 524 |
| | 6 | 422 | -389 |
| | 10 | 164 | -129 |
| | 12 | 293 | 302 |
| | 14 | 286 | -271 |
| -6, 0 | 0 | 200 | 167 |
| | 2 | 306 | 248 |
| | 4 | 336 | 332 |
| | 6 | 585 | -780* |
| | 8 | 382 | 360 |
| | 14 | 228 | -219 |
| | 16 | 298 | 321 |
| 8, 0 | 2 | 241 | -203 |
| | 4 | 240 | 218 |
| | 8 | 334 | 284 |
| | 10 | 375 | -365 |
| | 12 | 249 | 243 |
| -8, 0 | 0 | 433 | 445 |
| | 2 | 314 | -293 |
| | 6 | 392 | -394 |
| | 8 | 463 | 509 |
| | 10 | 230 | -195 |
| | 18 | 213 | -261 |
| 10, 0 | 2 | 168 | -167 |
| | 4 | 243 | -202 |
| | 8 | 469 | 482 |
| | 10 | 290 | -290 |
| -10, 0 | 0 | 402 | 362 |
| | 2 | 493 | -577* |
| | 4 | 185 | 197 |
| | 10 | 315 | -343 |
| | 12 | 204 | 198 |
| 12, 0 | 6 | 303 | -310 |
| | 8 | 264 | 219 |
| | 14 | 205 | -206 |
| | 16 | 206 | 199 |
| -12, 0 | 0 | 247 | 218 |
| | 2 | 389 | -406 |
| | 4 | 377 | 362 |
| | 6 | 193 | -172 |
| | 10 | 196 | -213 |
| | 12 | 336 | 364 |
| 14, 0 | 2 | 281 | -248 |
| | 4 | 323 | 327 |
| | 6 | 211 | -214 |
| | 12 | 205 | 238 |
| | 14 | 247 | -233 |
| -14, 0 | [illegible] | 165 | 145 |
| | 4 | 191 | 180 |
| | 6 | 246 | -249 |
| | 8 | 177 | 195 |
| | 14 | 213 | -233 |
| 16, 0 | 2 | 296 | -324 |
| | 4 | 229 | 226 |
| | 12 | 229 | 205 |
| -16, 0 | 0 | 222 | 217 |
| | 6 | 235 | -210 |
| | 8 | 322 | 327 |
| | 10 | 257 | -195 |
| 18, 0 | 2 | 189 | -172 |
| -18, 0 | | 206 | 222 |
| 1, 1 | 1 | 189 | -181 |
| | 2 | 394 | -462 |
| | 3 | 207 | -173 |
| | 4 | 320 | 344 |
| | 5 | 103 | -104 |
| | 6 | 234 | -216 |
| | 7 | 431 | 470 |
| | 9 | 425 | -433 |
| | 10 | 227 | -208 |
| | 12 | 261 | 249 |
| | 13 | 168 | 65 |
| | 14 | 227 | -210 |
| -1, 1 | 2 | 313 | -363 |
| | 3 | 258 | 264 |
| | 4 | 194 | 162 |
| | 5 | 126 | -101 |
| | 6 | 206 | -191 |
| | 8 | 251 | 216 |
| | 9 | 339 | -323 |
| | 10 | 246 | -227 |
| | 11 | 246 | 231 |
| | 12 | 259 | 266 |
| | 14 | 159 | -169 |
| 3, 1 | 1 | 356 | 358 |
| | 2 | 426 | -477 |
| | 3 | 276 | -245 |
| | 4 | 496 | 483 |
| | 5 | 268 | -262 |
| | 6 | 244 | -192 |
| | 7 | 407 | 389 |
| | 9 | 162 | -116 |
| | 10 | 205 | -170 |
| | 12 | 222 | 214 |
| | 14 | 187 | -183 |
| | 15 | 202 | 222 |
| -3, 1 | 0 | 198 | 163 |
| | 1 | 334 | -345 |
| | 2 | 157 | -143 |
| | 3 | 445 | 502 |
| | 4 | 252 | 236 |
| | 5 | 392 | -394 |
| | 6 | 215 | -181 |
| | 7 | 138 | -87 |
| | 8 | 321 | 332 |
| | 10 | 290 | -239 |
| | 11 | 248 | 245 |
| | 12 | 240 | 212 |
| | 13 | 274 | -264 |
| | 15 | 167 | 165 |
| 5, 1 | 1 | 194 | -165 |
| | 2 | 368 | -397 |
| | 3 | 409 | 436 |
| | 4 | 358 | 363 |
| | 5 | 370 | -397 |
| | 6 | 259 | -233 |
| | 8 | 226 | 200 |
| | 10 | 179 | -175 |
| | 11 | 176 | 152 |
| | 12 | 170 | 163 |
| | 13 | 262 | -273 |
| | 15 | 176 | 157 |
| -5, 1 | 0 | 170 | 139 |
| | 1 | 158 | -137 |
| | 3 | 286 | 286 |
| | 4 | 311 | 308 |
| | 5 | 366 | -378 |
| | 6 | 304 | -303 |
| | 7 | 234 | 204 |
| | 8 | 287 | 275 |
| | 9 | 191 | 129 |
| | 10 | 215 | -235 |
| | 12 | 164 | 138 |
| | 13 | 178 | -193 |
| | 15 | 222 | 249 |
| 7, 1 | 1 | 398 | -404 |
| | 2 | 204 | -209 |
| | 3 | 329 | 310 |
| | 4 | 211 | 182 |
| | 5 | 134 | 71 |
| | 6 | 291 | -267 |
| | 7 | 218 | -161 |
| | 8 | 255 | 244 |
| | 9 | 164 | -114 |
| | 10 | 218 | -201 |
| | 11 | 238 | 238 |
| | 13 | 201 | -214 |
| -7, 1 | 0 | 198 | 187 |
| | 1 | 151 | -105 |
| | 2 | 232 | -197 |
| | 3 | 191 | -148 |
| | 4 | 305 | 261 |
| | 5 | 133 | -90 |
| | 6 | 303 | -325 |
| | 7 | 361 | 355 |
| | 8 | 200 | 211 |
| | 9 | 288 | -291 |
| | 10 | 192 | -176 |
| | 15 | 207 | -178 |
| 9, 1 | 1 | 357 | -359 |
| | 2 | 132 | -118 |
| | 3 | 173 | 141 |
| | 4 | 150 | 143 |
| | 6 | 238 | -296 |
| | 7 | 165 | 166 |
| | 8 | 236 | 216 |
| | 9 | 230 | -255 |
| | 10 | 215 | -223 |
| | 11 | 166 | 154 |
| | 12 | 163 | 148 |
| -9, 1 | 0 | 224 | 205 |
| | 1 | 376 | -406 |
| | 2 | 301 | -320 |
| | 4 | 286 | 282 |
| | 5 | 172 | 125 |
| | 6 | 203 | -175 |
| | 8 | 162 | 169 |
| | 9 | 275 | -309 |
| | 10 | 152 | -136 |
| | 11 | 179 | 214 |
| | 12 | 178 | 184 |
| 11, 1 | 2 | 205 | -151 |
| | 4 | 166 | 175 |
| | 5 | 200 | -170 |
| | 6 | 153 | -151 |
| | 7 | 290 | 309 |
| | 8 | 177 | 173 |
| | 10 | 203 | -201 |
| -11, 1 | 0 | 232 | 198 |
| | 1 | 255 | -249 |
| | 2 | 248 | -259 |
| | 3 | 273 | 278 |
| | 4 | 279 | 297 |
| | 6 | 146 | -114 |
| | 8 | 153 | 107 |
| | 9 | 173 | -119 |
| | 11 | 248 | 264 |
| | 12 | 172 | 191 |
| 13, 1 | 2 | 203 | -216 |
| | 3 | 158 | 143 |
| | 4 | 160 | 184 |
| | 5 | 253 | -265 |
| -13, 1 | 0 | 206 | 192 |
| | 2 | 168 | -150 |
| | 3 | 244 | 250 |
| | 4 | 170 | 205 |
| | 5 | 234 | -258 |
| | 6 | 160 | -173 |
| | 7 | 178 | 135 |
| | 10 | 173 | -165 |
| 15, 1 | 2 | 171 | -208 |
| | 3 | 173 | 207 |
| | 4 | 175 | 181 |
| -15, 1 | 0 | 168 | 177 |
| | 5 | 172 | -187 |
| | 6 | 174 | -192 |
| | 7 | 176 | 213 |
| 0, 2 | 2 | 103 | 101 |
| | 3 | 410 | 475 |
| | 4 | 96 | 105 |
| | 5 | 288 | -289 |
| | 6 | 306 | -299 |
| | 7 | 409 | 414 |
| | 9 | 276 | -269 |
| | 10 | 138 | 117 |
| | 11 | 196 | 188 |
| | 12 | 239 | 169 |
| | 14 | 215 | -203 |
| | 15 | 177 | 162 |
| 2, 2 | 1 | 226 | -233 |
| | 2 | 415 | -450 |
| | 4 | 364 | 380 |
| | 5 | 296 | -300 |
| | 7 | 398 | 419 |
| | 8 | 159 | -103 |
| | 9 | 267 | -283 |
| | 10 | 195 | -177 |
| | 11 | 236 | 230 |
| | 12 | 258 | 221 |
| | 13 | 164 | -148 |
| -2, 2 | 0 | 170 | 186 |
| | 1 | 137 | -151 |
| | 2 | 133 | -123 |
| | 3 | 491 | 604* |
| | 5 | 391 | -421 |
| | 6 | 280 | -257 |
| | 7 | 214 | 211 |
| | 8 | 358 | 354 |
| | 9 | 180 | -157 |
| | 11 | 212 | 212 |
| | 12 | 155 | -100 |
| | 13 | 185 | -201 |
| | 15 | 177 | 184 |
| | 16 | 185 | 151 |
| | 17 | 183 | -202 |
| 4, 2 | 1 | 226 | -207 |
| | 2 | 304 | -267 |
| | 3 | 194 | 160 |
| | 4 | 207 | -180 |
| | 5 | 309 | -280 |
| | 6 | 186 | 137 |
| | 7 | 220 | 208 |
| | 8 | 207 | 211 |
| | 9 | 296 | -279 |
| | 10 | 242 | -227 |
| | 11 | 281 | 285 |
| | 13 | 193 | -191 |
| -4, 2 | 0 | 244 | 229 |
| | 1 | 399 | -432 |
| | 3 | 451 | 469 |
| | 4 | 125 | -75 |
| | 5 | 361 | -370 |
| | 6 | 112 | 100 |
| | 7 | 186 | 151 |
| | 8 | 312 | 278 |
| | 9 | 134 | -105 |
| | 10 | 273 | -282 |
| | 11 | 206 | 197 |
| | 13 | 275 | -292 |
| | 15 | 179 | 189 |
| | 16 | 150 | 92 |
| | 17 | 195 | -178 |
| 6, 2 | 1 | 313 | -334 |
| | 2 | 166 | 100 |
| | 3 | 368 | 392 |
| | 4 | 204 | -161 |
| | 6 | 286 | -275 |
| | 8 | 273 | 269 |
| | 9 | 230 | -214 |
| | 11 | 271 | 277 |
| | 13 | 208 | -222 |
| -6, 2 | 0 | 274 | 278 |
| | 1 | 322 | -327 |
| | 2 | 372 | -294 |
| | 3 | 325 | -341 |
| | 4 | 193 | 133 |
| | 5 | 218 | -197 |
| | 6 | 173 | 60 |
| | 7 | 147 | 164 |
| | 9 | 210 | -211 |
| | 10 | 175 | -177 |
| | 11 | 196 | 217 |
| | 12 | 185 | 216 |
| | 13 | 220 | -260 |
| | 15 | 183 | 198 |
| 8, 2 | 1 | 448 | -456 |
| | 2 | 168 | 101 |
| | 3 | 317 | 307 |
| | 4 | 245 | 247 |
| | 5 | 189 | -180 |
| | 6 | 331 | -334 |
| | 7 | 203 | 157 |
| | 9 | 159 | -141 |
| | 11 | 236 | 240 |
| -8, 2 | 0 | 150 | -92 |
| | 1 | 219 | -197 |
| | 2 | 234 | -204 |
| | 3 | 210 | 189 |
| | 4 | 272 | 263 |
| | 5 | 155 | -157 |
| | 6 | 128 | -106 |
| | 7 | 203 | 202 |
| | 9 | 253 | -274 |
| | 11 | 242 | 266 |
| | 12 | 160 | 146 |
| | 14 | 191 | -198 |
| 10, 2 | 1 | 266 | -284 |
| | 2 | 234 | -203 |
| | 3 | 246 | 240 |
| | 4 | 252 | 230 |
| | 5 | 292 | -309 |
| | 7 | 160 | 160 |
| | 8 | 165 | -108 |
| | 9 | 179 | -170 |
| -10, 2 | 0 | 139 | -78 |
| | 1 | 186 | -188 |
| | 2 | 139 | 99 |
| | 3 | 161 | 155 |
| | 4 | 197 | 191 |
| | 5 | 172 | -193 |
| | 6 | 235 | -230 |
| | 7 | 199 | 231 |
| | 8 | 179 | 154 |
| | 9 | 240 | -261 |
| | 11 | 196 | 221 |
| 12, 2 | 1 | 148 | -133 |
| | 2 | 250 | -242 |
| | 3 | 243 | 240 |
| | 5 | 246 | -262 |
| | 7 | 174 | 197 |
| | 9 | 174 | -189 |
| -12, 2 | 0 | 214 | 236 |
| | 1 | 155 | -142 |
| | 2 | 155 | 87 |
| | 3 | 191 | 209 |
| | 4 | 153 | -89 |
| | 5 | 243 | -274 |
| | 7 | 189 | 206 |
| | 8 | 178 | 174 |
| | 9 | 198 | -213 |
| 14, 2 | 1 | 172 | -146 |
| | 3 | 184 | 184 |
| | 5 | 181 | -191 |
| | 7 | 188 | 205 |
| -14, 2 | 0 | 217 | 242 |
| | 1 | 171 | -140 |
| | 2 | 195 | -196 |
| | 3 | 172 | 200 |
| | 5 | 219 | -278 |
| | 10 | 209 | -186 |
| -16, 2 | 5 | 190 | -194 |
| 1, 3 | 1 | 131 | -127 |
| | 2 | 182 | 186 |
| | 3 | 403 | 435 |
| | 5 | 416 | -413 |
| | 6 | 113 | 37 |
| | 7 | 282 | 236 |
| | 10 | 141 | 112 |
| | 11 | 217 | 228 |
| | 13 | 301 | -332 |
| | 15 | 190 | 190 |
| -1, 3 | 1 | 104 | 57 |
| | 2 | 257 | 233 |
| | 3 | 252 | 252 |
| | 4 | 118 | 82 |
| | 5 | 538 | -578* |
| | 6 | 122 | -68 |
| | 7 | 470 | 484 |
| | 8 | 130 | -81 |
| | 9 | 158 | -126 |
| | 10 | 147 | 119 |
| | 12 | 187 | -159 |
| | 13 | 218 | -209 |
| | 15 | 311 | 326 |
| | 17 | 206 | -203 |
| 3, 3 | 1 | 474 | -588* |
| | 2 | 138 | 97 |
| | 3 | 344 | 357 |
| | 4 | 150 | -94 |
| | 5 | 132 | -111 |
| | 6 | 183 | 124 |
| | 7 | 187 | 162 |
| | 9 | 353 | -346 |
| | 11 | 400 | 412 |
| -3, 3 | 1 | 378 | -399 |
| | 2 | 136 | 69 |
| | 3 | 135 | 120 |
| | 4 | 160 | -104 |
| | 5 | 243 | -246 |
| | 6 | 127 | -49 |
| | 7 | 408 | 433 |
| | 8 | 139 | -103 |
| | 9 | 313 | -316 |
| | 12 | 189 | -128 |
| | 15 | 225 | 237 |
| | 17 | 244 | -278 |
| 5, 3 | 1 | 408 | -389 |
| | 2 | 148 | 78 |
| | 3 | 112 | -57 |
| | 4 | 215 | -172 |
| | 5 | 153 | 77 |
| | 6 | 228 | 194 |
| | 7 | 274 | 261 |
| | 9 | 415 | -417 |
| | 11 | 261 | 229 |
| -5, 3 | 1 | 560 | -591* |
| | 3 | 304 | 294 |
| | 4 | 199 | -156 |
| | 5 | 90 | 38 |
| | 6 | 143 | 141 |
| | 7 | 157 | 156 |
| | 8 | 132 | -77 |
| | 9 | 346 | -358 |
| | 11 | 363 | 360 |
| 7, 3 | 1 | 294 | -240 |
| | 2 | 126 | 66 |
| | 4 | 147 | -101 |
| | 5 | 320 | -326 |
| | 6 | 165 | 105 |
| | 7 | 402 | 417 |
| | 9 | 220 | -210 |
| | 13 | 192 | -179 |
| | 15 | 237 | 236 |
| -7, 3 | 1 | 431 | -446 |
| | 3 | 422 | 461 |
| | 5 | 152 | -141 |
| | 6 | 218 | 168 |
| | 8 | 150 | -110 |
| | 9 | 171 | -155 |
| | 11 | 283 | 300 |
| | 13 | 266 | -306 |
| 9, 3 | 1 | 154 | -125 |
| | 2 | 143 | 79 |
| | 3 | 349 | 321 |
| | 5 | 425 | -397 |
| | 7 | 187 | 162 |
| | 8 | 157 | -82 |
| | 10 | 199 | 133 |
| | 13 | 256 | -260 |
| | 15 | 216 | 235 |
| -9, 3 | 3 | 258 | 237 |
| | 4 | 144 | -109 |
| | 5 | 380 | -379 |
| | 7 | 257 | 258 |
| | 13 | 218 | -265 |
| 11, 3 | 3 | 375 | 387 |
| | 5 | 263 | -230 |
| | 11 | 219 | 240 |
| | 13 | 202 | -216 |
| -11, 3 | 5 | 258 | -239 |
| | 7 | 323 | 376 |
| | 9 | 204 | -237 |
| 13, 3 | 1 | 272 | -304 |
| | 7 | 211 | 196 |
| | 9 | 222 | -270 |
| -13, 3 | 1 | 240 | -264 |
| | 7 | 202 | 249 |
| | 9 | 228 | -299 |
| 15, 3 | 7 | 247 | 262 |
| -15, 3 | 1 | 263 | -302 |
| | 3 | 235 | 280 |
| 0, 4 | 2 | 116 | 107 |
| | 3 | 209 | 214 |
| | 4 | 344 | -379 |
| | 5 | 252 | -258 |
| | 6 | 246 | 199 |
| | 7 | 138 | 121 |
| | 12 | 215 | -231 |
| | 13 | 220 | -163 |
| | 14 | 288 | 312 |
| | 15 | 180 | 145 |
| | 16 | 199 | -170 |
| 2, 4 | 1 | 181 | -205 |
| | 2 | 453 | 654* |
| | 3 | 226 | 240 |
| | 4 | 383 | -447 |
| | 5 | 218 | -193 |
| | 7 | 164 | 108 |
| | 9 | 163 | -130 |
| | 10 | 166 | 159 |
| | 11 | 202 | 179 |
| | 12 | 291 | -320 |
| | 13 | 176 | -162 |
| | 14 | 218 | 213 |
| -2, 4 | 0 | 244 | -316 |
| | 1 | 152 | -152 |
| | 2 | 110 | -65 |
| | 3 | 148 | 113 |
| | 5 | 274 | -280 |
| | 6 | 372 | 415 |
| | 7 | 150 | 153 |
| | 8 | 317 | -317 |
| | 9 | 153 | -157 |
| | 10 | 156 | 116 |
| | 11 | 149 | 114 |
| | 13 | 145 | -84 |
| | 14 | 182 | 142 |
| | 15 | 199 | 150 |
| | 16 | 208 | -198 |
| 4, 4 | 1 | 170 | -173 |
| | 2 | 352 | 387 |
| | 3 | 158 | 169 |
| | 4 | 122 | -65 |
| | 5 | 140 | -131 |
| | 7 | 134 | 85 |
| | 8 | 206 | -173 |
| | 9 | 232 | -200 |
| | 10 | 249 | 248 |
| | 11 | 196 | 182 |
| | 12 | 173 | -157 |
| -4, 4 | 0 | 320 | -377 |
| | 1 | 248 | -250 |
| | 3 | 134 | 121 |
| | 4 | 216 | 169 |
| | 5 | 161 | -144 |
| | 6 | 184 | 178 |
| | 7 | 178 | 163 |
| | 8 | 383 | -458 |
| | 9 | 177 | -155 |
| | 10 | 297 | 316 |
| | 11 | 203 | 182 |
| | 13 | 167 | -91 |
| 6, 4 | 1 | 181 | -131 |
| | 3 | 196 | 155 |
| | 5 | 162 | -129 |
| | 6 | 323 | 316 |
| | 7 | 195 | 152 |
| | 8 | 301 | -339 |
| | 9 | 236 | -204 |
| | 10 | 190 | 169 |
| -6, 4 | 0 | 239 | -247 |
| | 1 | 211 | -210 |
| | 2 | 328 | 353 |
| | 3 | 196 | 223 |
| | 4 | 184 | -195 |
| | 5 | 176 | -127 |
| | 8 | 195 | -189 |
| | 9 | 180 | -132 |
| | 10 | 257 | 277 |
| | 11 | 241 | 187 |
| | 12 | 198 | -178 |
| 8, 4 | 1 | 152 | -109 |
| | 2 | 113 | -34 |
| | 3 | 201 | 171 |
| | 4 | 263 | -243 |
| | 5 | 186 | -178 |
| | 6 | 338 | 354 |
| | 7 | 180 | 166 |
| | 8 | 254 | -258 |
| -8, 4 | 1 | 150 | -142 |
| | 2 | 255 | 284 |
| | 3 | 170 | 172 |
| | 4 | 377 | -427 |
| | 5 | 209 | -213 |
| | 6 | 226 | 205 |
| | 11 | 183 | 153 |
| | 12 | 207 | -209 |
| 10, 4 | 2 | 205 | 173 |
| | 3 | 240 | 217 |
| | 4 | 230 | -209 |
| | 5 | 192 | -157 |
| | 11 | 185 | 142 |
| | 12 | 202 | -212 |
| -10, 4 | 4 | 247 | -252 |
| | 5 | 226 | -176 |
| | 6 | 299 | 324 |
| | 7 | 238 | 188 |
| 12, 4 | 1 | 167 | -155 |
| | 2 | 268 | 279 |
| | 3 | 196 | 192 |
| | 10 | 221 | 235 |
| | 12 | 213 | -202 |
| -12, 4 | 0 | 253 | -260 |
| | 7 | 178 | 166 |
| | 8 | 207 | -221 |
| 14, 4 | 1 | 184 | -152 |
| -14, 4 | 0 | 253 | -272 |
| | 2 | 232 | 212 |
| | 5 | 164 | -101 |
| | 8 | 196 | -171 |
| | 10 | 204 | 245 |
| 1, 5 | 1 | 113 | -139 |
| | 2 | 226 | 287 |
| | 3 | 171 | -153 |
| | 4 | 308 | -348 |
| | 5 | 118 | 76 |
| | 6 | 173 | 171 |
| | 7 | 146 | 110 |
| | 10 | 252 | 238 |
| | 12 | 253 | -239 |
| | 14 | 207 | 212 |
| -1, 5 | 1 | 223 | -286 |
| | 2 | 156 | 212 |
| | 3 | 102 | 66 |
| | 4 | 179 | -198 |
| | 5 | 251 | 242 |
| | 6 | 231 | 248 |
| | 8 | 193 | -193 |
| | 10 | 238 | 212 |
| | 11 | 203 | 152 |
| | 12 | 251 | -223 |
| | 14 | 206 | 197 |
| 3, 5 | 1 | 181 | 167 |
| | 2 | 318 | 367 |
| | 4 | 295 | -329 |
| | 5 | 198 | -152 |
| | 6 | 157 | 138 |
| | 7 | 167 | 91 |
| | 8 | 227 | -193 |
| | 10 | 214 | 217 |
| | 11 | 186 | -115 |
| | 12 | 193 | -183 |
| -3, 5 | 0 | 210 | -269 |
| | 2 | 161 | 186 |
| | 3 | 214 | 190 |
| | 4 | 168 | -164 |
| | 6 | 226 | 214 |
| | 7 | 191 | -112 |
| | 8 | 276 | -294 |
| | 10 | 253 | 271 |
| 5, 5 | 2 | 209 | 212 |
| | 3 | 237 | 225 |
| | 4 | 230 | -225 |
| | 5 | 206 | -188 |
| | 6 | 277 | 258 |
| | 8 | 258 | -253 |
| | 9 | 175 | 107 |
| | 13 | 177 | -108 |
| -5, 5 | 0 | 236 | -235 |
| | 1 | 202 | 186 |
| | 2 | 184 | 194 |
| | 4 | 213 | -230 |
| | 5 | 236 | -236 |
| | 6 | 236 | 231 |
| | 8 | 237 | -212 |
| | 10 | 285 | 284 |
| 7, 5 | 1 | 172 | -102 |
| | 4 | 278 | -289 |
| | 6 | 286 | 297 |
| | 8 | 212 | -228 |
| -7, 5 | 0 | 152 | -144 |
| | 2 | 255 | 277 |
| | 3 | 212 | -155 |
| | 4 | 275 | -290 |
| | 6 | 244 | 278 |
| | 7 | 249 | 229 |
| 9, 5 | 2 | 186 | 202 |
| | 4 | 248 | -260 |
| | 6 | 181 | 175 |
| | 8 | 221 | -190 |
| | 9 | 200 | -156 |
| -9, 5 | 1 | 182 | -150 |
| | 2 | 243 | 232 |
| | 4 | 281 | -301 |
| | 6 | 251 | 272 |
| 11, 5 | 2 | 265 | 269 |
| | 3 | 185 | -121 |
| | 8 | 209 | -168 |
| -11, 5 | 0 | 212 | -222 |
| | 1 | 170 | -121 |
| | 2 | 170 | 164 |
| | 3 | 206 | 163 |
| | 4 | 183 | -186 |
| | 6 | 207 | 220 |
| | 8 | 198 | -181 |
| 13, 5 | 2 | 221 | 215 |
| -13, 5 | 0 | 226 | -264 |
| | 2 | 198 | 213 |
| 0, 6 | 2 | 213 | 298 |
| | 3 | 144 | -144 |
| | 5 | 203 | 230 |
| | 6 | 126 | 123 |
| | 7 | 171 | -140 |
| | 8 | 246 | -260 |
| | 10 | 268 | 259 |
| 2, 6 | 1 | 122 | 119 |
| | 2 | 105 | 67 |
| | 3 | 144 | -128 |
| | 5 | 140 | 122 |
| | 6 | 224 | 268 |
| | 8 | 253 | -268 |
| | 9 | 186 | 136 |
| | 11 | 199 | -127 |
| -2, 6 | 0 | 137 | -196 |
| | 2 | 247 | 312 |
| | 3 | 119 | -108 |
| | 4 | 174 | -203 |
| | 5 | 208 | 208 |
| | 7 | 201 | -171 |
| | 8 | 164 | -128 |
| | 10 | 217 | 212 |
| | 12 | 264 | -275 |
| 4, 6 | 1 | 173 | 168 |
| | 2 | 161 | 153 |
| | 4 | 233 | -296 |
| | 6 | 269 | 313 |
| | 8 | 177 | -136 |
| | 9 | 225 | 183 |
| | 11 | 197 | -129 |
| | 12 | 206 | -166 |
| | 14 | 224 | 219 |
| -4, 6 | 1 | 183 | 218 |
| | 2 | 151 | 187 |
| | 3 | 119 | -86 |
| | 4 | 257 | -312 |
| | 5 | 142 | 80 |
| | 6 | 233 | 227 |
| | 8 | 192 | -123 |
| | 9 | 172 | 171 |
| 6, 6 | 1 | 143 | 108 |
| | 2 | 238 | 299 |
| | 4 | 248 | -266 |
| | 6 | 195 | 134 |
| | 7 | 173 | -110 |
| | 10 | 207 | 160 |
| | 11 | 173 | -96 |
| | 12 | 245 | -224 |
| | 14 | 220 | 149 |
| -6, 6 | 0 | 159 | -141 |
| | 1 | 225 | 251 |
| | 3 | 182 | -140 |
| | 4 | 189 | -167 |
| | 6 | 273 | 300 |
| | 8 | 244 | -261 |
| | 9 | 171 | 96 |
| | 11 | 187 | -162 |
| 8, 6 | 2 | 233 | 254 |
| | 3 | 188 | -145 |
| | 5 | 175 | 130 |
| | 8 | 195 | -175 |
| | 10 | 248 | 246 |
| -8, 6 | 0 | 238 | -287 |
| | 1 | 148 | 98 |
| | 2 | 180 | 140 |
| | 3 | 182 | -137 |
| | 8 | 227 | -276 |
| | 10 | 218 | 212 |
| | 11 | 207 | -136 |
| 10, 6 | 3 | 202 | -170 |
| | 5 | 183 | 125 |
| | 6 | 208 | 213 |
| | 8 | 282 | -256 |
| -10, 6 | 0 | 276 | -278 |
| | 2 | 258 | 277 |
| | 10 | 232 | 214 |
| 12, 6 | 6 | 255 | 235 |
| -12, 6 | 2 | 242 | 230 |
| | 4 | 229 | -264 |
| 14, 6 | 4 | 237 | -235 |
| 1, 7 | 1 | 130 | 204 |
| | 7 | 287 | -311 |
| | 8 | 229 | -162 |
| | 9 | 282 | 280 |
| -1, 7 | 1 | 174 | 264 |
| | 2 | 133 | 160 |
| | 3 | 225 | -278 |
| | 9 | 250 | 230 |
| | 10 | 220 | 129 |
| | 11 | 243 | -235 |
| 3, 7 | 5 | 232 | 231 |
| | 6 | 210 | 142 |
| | 7 | 302 | -316 |
| -3, 7 | 1 | 175 | 213 |
| | 2 | 170 | 190 |
| | 3 | 233 | -314 |
| | 4 | 195 | -169 |
| | 5 | 243 | 239 |
| 5, 7 | 3 | 248 | -253 |
| | 4 | 169 | -123 |
| | 5 | 270 | 273 |
| -5, 7 | 1 | 166 | 142 |
| | 3 | 218 | -236 |
| | 4 | 210 | -166 |
| | 5 | 240 | 239 |
| | 6 | 188 | 114 |
| | 7 | 231 | -205 |
| 7, 7 | 1 | 278 | 292 |
| | 2 | 220 | 143 |
| | 3 | 280 | -265 |
| | 4 | 191 | -113 |
| -7, 7 | 7 | 233 | -227 |
| | 8 | 213 | -119 |
| 9, 7 | 1 | 291 | 300 |
| | 2 | 204 | 120 |
| -9, 7 | 1 | 218 | 230 |
| | 9 | 236 | 234 |
| 0, 8 | 2 | 86 | -95 |
| | 3 | 137 | -175 |
| | 4 | 122 | -76 |
| | 5 | 152 | 150 |
| | 6 | 138 | 57 |
| | 7 | 170 | -165 |
| | 9 | 208 | 221 |
| | 11 | 184 | -178 |
| 2, 8 | 1 | 93 | 133 |
| | 3 | 114 | -134 |
| | 5 | 160 | 174 |
| | 7 | 215 | -223 |
| | 9 | 192 | 189 |
| -2, 8 | 1 | 136 | 222 |
| | 2 | 137 | -136 |
| | 3 | 170 | -235 |
| | 5 | 160 | 186 |
| | 6 | 159 | 87 |
| | 9 | 188 | 157 |
| | 11 | 218 | -204 |
| | 12 | 203 | 120 |
| 4, 8 | 1 | 126 | 131 |
| | 3 | 170 | -191 |
| | 4 | 155 | 172 |
| | 5 | 208 | 227 |
| | 6 | 182 | -140 |
| | 7 | 180 | -190 |
| | 9 | 193 | 124 |
| -4, 8 | 0 | 132 | -97 |
| | 1 | 137 | 152 |
| | 3 | 171 | -228 |
| | 4 | 149 | 124 |
| | 5 | 185 | 231 |
| | 11 | 206 | -156 |
| | 13 | 206 | 180 |
| 6, 8 | 1 | 176 | 197 |
| | 2 | 158 | -94 |
| | 3 | 203 | -230 |
| | 4 | 156 | 115 |
| | 5 | 185 | 190 |
| | 9 | 187 | 129 |
| | 11 | 222 | -187 |
| -6, 8 | 1 | 146 | 132 |
| | 2 | 165 | 88 |
| | 3 | 151 | -154 |
| | 5 | 163 | 200 |
| | 6 | 185 | -141 |
| | 7 | 193 | -213 |
| | 9 | 187 | 151 |
| 8, 8 | 1 | 204 | 217 |
| | 3 | 200 | -193 |
| | 9 | 209 | 191 |
| -8, 8 | 1 | 170 | 181 |
| | 7 | 177 | -172 |
| | 9 | 233 | 206 |
| -10, 8 | 1 | 192 | 204 |
| | 3 | 178 | -172 |

* Reflections omitted from least-squares refinement because of extinction.

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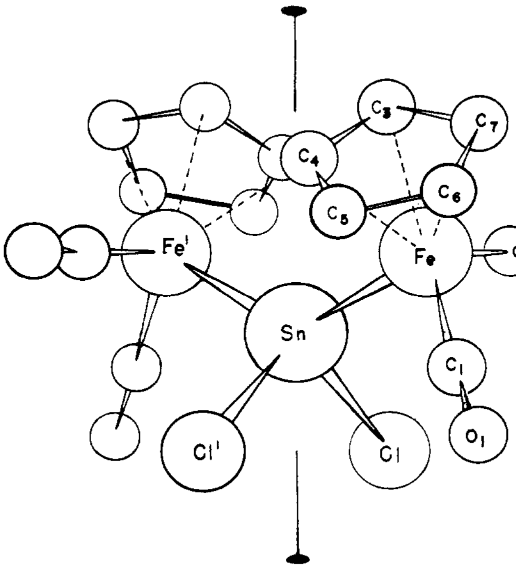

Figure 1.—The molecular configuration of $Cl_2Sn[\pi\text{-}C_5H_5Fe(CO)_2]_2$.

Table IV

Bond Angles in $Cl_2Sn[\pi\text{-}C_5H_5Fe(CO)_2]_2$ with Their Estimated Standard Deviations

| Atoms | Angle, deg | Atoms | Angle, deg |
|---|---|---|---|
| Cl–Sn–Cl′ | 94.1 (0.6) | $C_3$–$C_4$–$C_5$ | 110.2 (3.2) |
| Fe–Sn–Fe′ | 128.6 (0.3) | $C_4$–$C_5$–$C_6$ | 105.7 (3.2) |
| Fe–Sn–Cl | 107.4 (0.3) | $C_5$–$C_6$–$C_7$ | 109.0 (3.1) |
| Fe–Sn–Cl′ | 107.0 (0.3) | $C_6$–$C_7$–$C_3$ | 103.8 (2.9) |
| | | $C_7$–$C_3$–$C_4$ | 111.3 (3.1) |
| Sn–Fe–$C_1$ | 89.0 (1.2) | | |
| Sn–Fe–$C_2$ | 89.0 (1.2) | Sn–Fe–$C_3$ | 105.8 (0.9) |
| $C_1$–Fe–$C_2$ | 98.4 (1.7) | Sn–Fe–$C_4$ | 86.2 (1.0) |
| | | Sn–Fe–$C_5$ | 103.2 (1.0) |
| Fe–$C_1$–$O_1$ | 177.5 (3.6) | Sn–Fe–$C_6$ | 145.2 (1.1) |
| Fe–$C_2$–$O_2$ | 176.0 (3.5) | Sn–Fe–$C_7$ | 146.7 (0.9) |

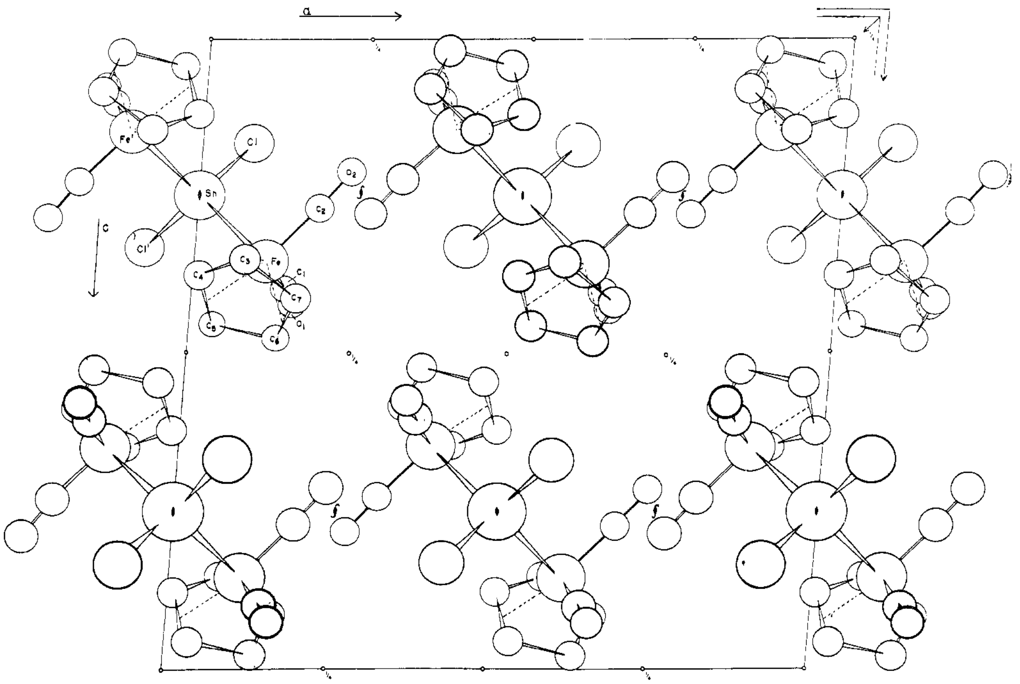

Figure 2.—[010] projection of the unit cell.

Table III

Bond Lengths in $Cl_2Sn[\pi\text{-}C_5H_5Fe(CO)_2]_2$ with Their Estimated Standard Deviations

| Bond | Distance, A | Bond | Distance, A |
|---|---|---|---|
| Sn–Fe | 2.492 (8) | Fe–$C_7$ | 2.11 (4) |
| Sn–Cl | 2.43 (1) | $C_1$–$O_1$ | 1.08 (5) |
| Fe–$C_1$ | 1.76 (4) | $C_2$–$O_2$ | 1.09 (4) |
| Fe–$C_2$ | 1.76 (4) | $C_3$–$C_4$ | 1.33 (5) |
| Fe–$C_3$ | 2.09 (4) | $C_4$–$C_5$ | 1.40 (5) |
| Fe–$C_4$ | 2.10 (4) | $C_5$–$C_6$ | 1.48 (5) |
| Fe–$C_5$ | 2.08 (4) | $C_6$–$C_7$ | 1.41 (5) |
| Fe–$C_6$ | 2.04 (4) | $C_7$–$C_3$ | 1.47 (5) |

$(C_6H_5)_2Sn[Mn(CO)_5]_2$,[4] $(C_6H_5)_3SnMn(CO)_4P(C_6H_5)_3$,[10] and $[Pt(SnCl_3)_5]^{3-}$.[6] The Sn–Cl bond length of 2.43 A is *longer* than the Sn–Cl distances of 2.31, 2.32, 2.34, and 2.37 A found in the series of compounds $SnCl_4$,[21] $CH_3SnCl_3$,[22] $(CH_3)_2SnCl_2$,[22] and $(CH_3)_3SnCl$.[22] The Fe–Sn–Fe′ angle of 128.6° and the Cl–Sn–Cl′ angle of 94.1° show the largest deviation from ideal tetrahedral coordination yet observed for a tin atom. An Mn–Sn–Mn angle of 117° has been observed in the compound $(C_6H_5)_2Sn[Mn(CO)_5]_2$.[4]

The above observations concerning bond distances and angles in $Cl_2Sn[\pi\text{-}C_5H_5Fe(CO)_2]_2$ are consistent with increased s character in the tetrahedral hybrid orbitals used in Sn–Fe bond formation and increased p character in the orbitals utilized in Sn–Cl bond formation. The short Sn–Fe bond length and long Sn–Cl bond length are in agreement with this interpretation.[23]

Figure 2 shows the [010] projection of the unit cell. The minimum intermolecular bond distance of 3.13 A between $O_1$ and $O_2'$ is consistent with normal van der Waals interactions.

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The solid-state structure of $Cl_2Sn[\pi\text{-}C_5H_5Fe(CO)_2]_2$ is in agreement with the infrared spectral data for the carbonyl region[3] although Flitcroft, *et al.*, did not consider the molecular point group symmetry found in the solid state. Point groups $C_{2v}$, $C_s$, and $C_1$ were considered; group theoretical calculations predict three bands ($A_1$, $B_1$, and $B_2$) for $C_{2v}$, four bands (2 A′ and 2 A″) for $C_s$, and four bands (4 A) for $C_1$ in the carbonyl region. The $C_{2v}$ model may be rejected since four frequencies are observed. Similar calculations for the point group $C_2$ show that four bands (2 A and 2 B) are expected in the carbonyl region. The infrared data have been measured in solution and the point group symmetries $C_1$, $C_s$, and $C_2$ are all possible, but the point group symmetry of $C_2$ has been established conclusively for $Cl_2Sn[\pi\text{-}C_5H_5Fe(CO)_2]_2$ in the solid state.

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# Intensities of CO Stretching Modes in the Infrared Spectra of Adsorbed CO and Metal Carbonyls[1]

BY THEODORE L. BROWN AND D. J. DARENSBOURG

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The presently available data relating to the infrared intensity of CO stretching motion in adsorbed CO and in metal carbonyls are reviewed. For adsorbed CO which exhibits higher CO stretching frequency than gaseous CO, it is proposed that the intensity is predominantly controlled by the strength of σ-bond interaction. A reversal in sign of $\partial\mu/\partial r_{CO}$ at an intermediate CO–adsorbate bond strength accounts for the relationship between frequency and intensity. Adsorbed CO with lower frequency than gaseous CO, and metal carbonyls, exhibit enormously enhanced infrared intensity in the CO stretching mode. This enhancement arises from extensive π-bond interaction with the adsorbate or central metal. It is possible to derive certain rules for the ratios of intensities of CO modes of different symmetry species in metal carbonyls, assuming local MCO bond moment derivatives, and assuming separability of the CO stretches from other normal modes. These rules are tested and found inadequate for substituted octahedral carbonyl systems. Estimation of bond angles in monosubstituted carbonyl compounds from intensity ratios is therefore an uncertain procedure. An alternative approach is proposed which makes use of data in a series such as $M(CO)_6$, $M(CO)_5L$, *trans*-$M(CO)_4L_2$.

Infrared spectroscopy of metal carbonyls has progressed from attempts to determine molecular point group symmetries *via* application of selection rules to an emphasis on utilization of vibrational data to learn about valence electron distribution. Jones[2] has outlined valence bond and molecular orbital considerations which suggest relationships which should obtain among the force constants which define the valence force field. Cotton and co-workers[3–5] have developed a simplified force field model which is related to that of Jones, but which considers only the CO stretching modes. The force field is assumed to consist entirely of CO stretching and CO–CO stretch–stretch interaction force constants. Metal–carbon bonding is neglected; internal coordinate space is spanned entirely by the CO bond displacements. The model is premised upon the overriding importance of π bonding between metal and CO in determining variations in the force constants and on a negligible mixing of the CO stretching coordinates into other normal modes of the molecule. Variations in CO force constants which result from substitution of one or more carbonyl groups by other ligands arise predominantly from variation in the extent of π bonding.

It is well known that carbonyl stretching frequencies in substituted carbonyls are sensitive to the nature of the substituent; it is not as widely recognized that the intensities of the CO modes are also subject to sizable variation. A number of studies of intensities in metal carbonyl compounds have already been reported in the literature,[6–10] and we can expect that more work will be done in this area in the near future. The purpose of the present contribution is to outline some considerations which are of value in interpreting the intensity results and to interpret certain aspects of the extant data.

## Dipole Moment Function for CO and σ-Bonded CO.

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