1963

Crystal chemistry of tin (II) compounds

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Recommended Citation

Olson, David Harold, "Crystal chemistry of tin (II) compounds " (1963). *Retrospective Theses and Dissertations*. 2970.
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OLSON, David Harold, 1937–
CRYSTAL CHEMISTRY OF TIN(II) COMPOUNDS.

Iowa State University of Science and Technology
Ph.D., 1963
Chemistry, physical

University Microfilms, Inc., Ann Arbor, Michigan

CRYSTAL CHEMISTRY
OF TIN (II) COMPOUNDS

by

David Harold Olson

A Dissertation Submitted to the
Graduate Faculty in Partial Fulfillment of
The Requirements for the Degree of
DOCTOR OF PHILOSOPHY

Major Subject: Physical Chemistry

Approved:

Signature was redacted for privacy.

In Charge of Major Work

Signature was redacted for privacy.

Head of Major Department

Signature was redacted for privacy.

Dean of Graduate College

Iowa State University
Of Science and Technology
Ames, Iowa

1963

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I. INTRODUCTION

A. Purpose of This Investigation

Until recently, very little was known about the crystal chemistry of divalent tin.[1] As a result, it was not possible to detect any trends or similarities in tin (II) crystal chemistry. Such trends are often important aids in the understanding of an element's chemistry in general and of its coordination chemistry in particular.

This report describes the X-ray study of some tin compounds which show prospect of providing insight into the crystal chemistry of divalent tin.

Stannous chloride was studied first because alkyl derivatives are often isomorphous with the corresponding metal halides. Also in comparison with the alkyl derivatives, the metal halides are easier to prepare and to grow suitable single crystals of. A description of the experimental procedures and a discussion of the structure are given.

A review of the known crystal structures of tin (II) is given and trends are discussed. A covalent radius is calculated for tin (II).

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[1] A divalent tin compound here refers to any compound in which the tin atom uses two of its valence electrons in any type of bonding, ionic or covalent.

Diphenyltin was previously prepared and characterized as a low molecular weight polymer having four different modifications (1). From what was known about inorganic divalent tin compounds, it appeared that a study of one of the modifications of diphenyltin would yield interesting details concerning the coordination of divalent tin to organic ligands. A description of the X-ray study of the hexameric polymer of diphenyltin is given. The results are discussed and related to other known facts about R_2Sn compounds.

B. Literature Review of Known Structures

1. The crystal structure of SnO

Although structural information concerning stannous oxide was reported in 1926, its true structure remained in doubt until 1941. Since this structure is isomorphous with its extensively studied cognate, tetragonal (red) lead oxide, the historical development of this determination will be considered in conjunction with the lead oxide crystal structure determination.

The structure of tetragonal lead oxide was reported in 1924 by Dickinson and Friauf (2). From a study using single crystal Laue photographs, they reported red lead oxide to be tetragonal with $a_0 = 3.99$ A, $c_0 = 5.01$ A, $z_0 = 2$ and atomic positions, 0,0,0; 1/2,1/2,0 for oxygen and 0,1/2,u; 1/2,0,$\bar{u}$ for lead where u = 0.24. This arrangement is similar to that shown in Figure 1. In 1926, Levi and Natta (3) reported the above structure to be in error with respect to the oxygen atom positions. They placed these atoms in the set, 0,1/2,v; 1/2,0,$\bar{v}$ with v = 0.76. This corresponds to a distorted NaCl type structure. They also reported that stannous oxide with a_o = 3.80 A and c_o = 4.81 A is isomorphous with the lead oxide structure. In order to resolve this disagreement, in 1941, Moore and Pauling (4) reexamined these two structures using X-ray powder data. From the relative intensities of certain powder lines, they were able to exclude the Levi and Natta structure and show the Dickenson and Friauf structure to be correct. They obtained the metal atom positions for these two structures by taking intensity ratios of neighboring powder lines and adjusting u to get the best agreement between the calculated and observed ratios. A summary of the results of Moore and Pauling for stannous oxide is shown in Table 1. Since this study, additional X-ray diffraction (5) and neutron diffraction (6) studies on red lead oxide have reaffirmed the Dickinson and Friauf structural determination.

The stannous oxide structure, as shown in Figure 1, can be described in terms of a three layer unit repeating in the c_o direction where each unit consists of a layer of

Figure 1. Illustration of the crystal structure of SnO

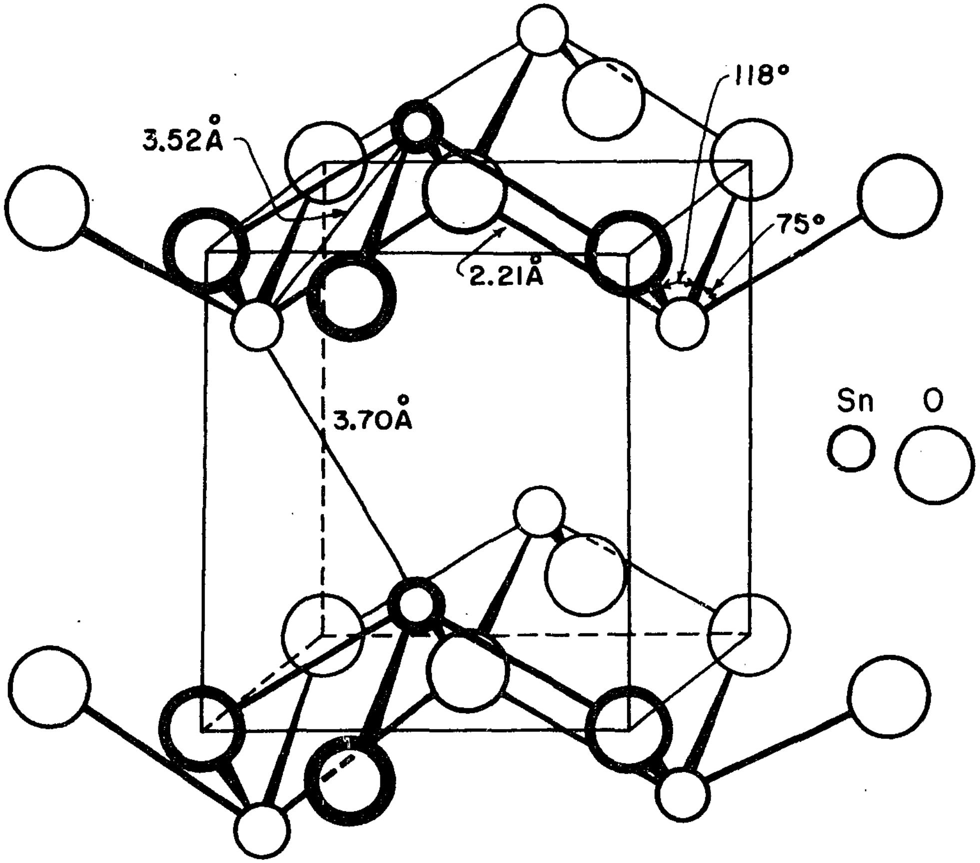

3.52Å
118°
75°
2.21Å
3.70Å
Sn
O

Table 1. Summary of the crystal structure data for SnO

Space group	P4/nmm
a_o	3.796 ± 0.006 A
c_o	4.816 ± 0.008 A
Oxygen atom positions	0,0,0; 1/2,1/2,0
Tin atom positions	1/2,0,z; 0,1/2,$\bar{z}$
z	0.2356 ± 0.0019

oxygen atoms sandwiched between two layers of tin atoms. Closer inspection reveals that each tin atom sits at the apex of a square pyramid of oxygen atoms and each oxygen atom is tetrahedrally coordinated to four tin atoms. The Sn-O distance is 2.21 A. The Sn-Sn distance of 3.70 A between adjacent layers is also of interest. This is considerably shorter than the estimated (7, p. 263) 4.40 A expected for Van der Waals interactions.

2. The crystal structure of SnS

The structure of stannous sulfide was determined by Hofmann using single crystal Weissenberg techniques (8). The cell constants and density are:

a_o = 3.98 ± 0.02 A	z_o = 4
b_o = 4.33 ± 0.02 A	d_c = 5.20 g/cc
c_o = 11.18 ± 0.02 A	d_o = 5.08 g/cc.

Tin (II) sulfide is orthorhombic belonging to the space group Pmcn. All atoms lie on mirror planes in the special four-fold set, 1/4,y,z; 3/4,$\bar{y}$,$\bar{z}$; 1/4,1/2 - y, 1/2 + z; 3/4,1/2 + y,1/2 - z. The positional parameters were found by graphical and trial and error methods; they are:

	x	y	z
Sn	1/4	0.115	0.118
S	1/4	0.428	0.350.

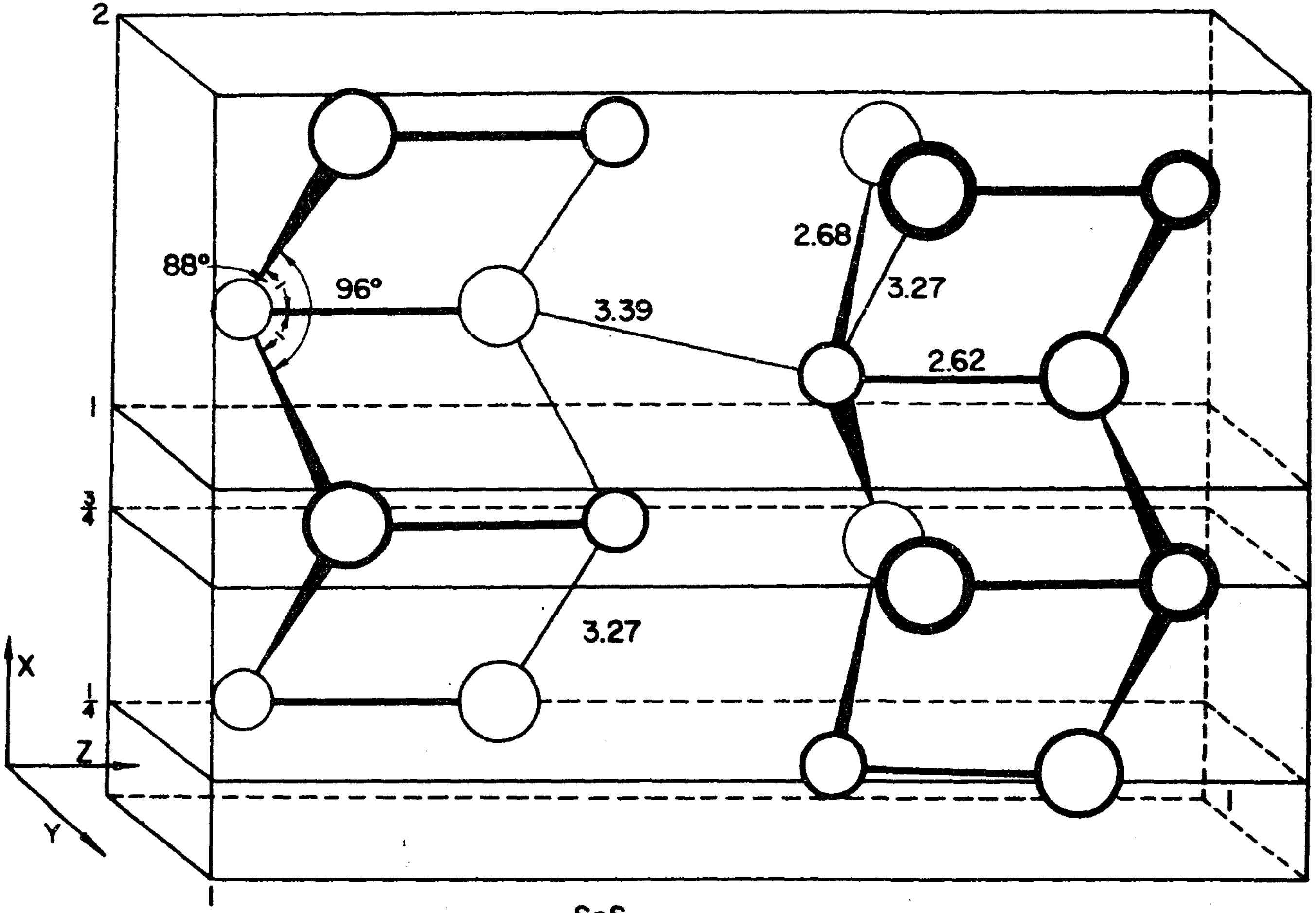
From Figure 2, it can be seen that this is a distorted NaCl type structure. It can also be described as double layer sheets stacked normal to the c_o axis. Each sheet consists of infinite zig-zag S-Sn-S-Sn chains with a Sn-S bond length of 2.68 A. Each chain is connected to an adjacent chain by a short Sn-S bond of length 2.62 A. The three S-Sn-S bond angles formed by the tin atom and the three nearest sulfur atoms are 96° and 88° (two). There are six sulfur atoms within 3.39 A of each tin atom.

3. The crystal structure of SnSe

Stannous selenide crystallizes in two forms, a cubic modification and an orthorhombic modification. Nesterova *et al*. (9) have reported that the cubic modification can be formed by condensing stannous selenide as a thin film and that the cubic modification is apparently a metastable form.

The cubic modification has the NaCl type structure

Figure 2. Illustration of the crystal structure of SnS



SnS

with a_o = 5.99 A and d_c = 6.11 g/cc (10). Each tin atom is octahedrally coordinated to six equidistant selenium atoms. The Sn-Se bond length is 3.00 A.

The crystal structure of orthorhombic tin (II) selenide was determined by Okazaki and Ueda using single crystal oscillation data and two-dimensional Patterson and Fourier techniques (11). This structure is isomorphous with stannous sulfide shown in Figure 2. The cell constants and density are:

a_o = 4.19 A	z_o = 4
b_o = 4.46 A	d_c = 6.07 g/cc
c_o = 11.57 A.	

Thus the space group is Pmcn and all atoms lie on mirror planes in the special four-fold set, 1/4,y,z; 3/4,$\bar{y}$,$\bar{z}$; 1/4,1/2 - y, 1/2 + z; 3/4,1/2 + y,1/2 - z. The positional parameters are:

	x	y	z
Sn	1/4	0.115	0.118
Se	1/4	0.522	0.150

The Sn-Se distances in this structure are 2.77 A, 2.82 A (two), 3.35 A (two) and 3.47 A. The chain Se-Sn-Se angle is 96^o and the Se-Sn-Se angle formed by a chain selenium atom, the adjacent tin atom and a short bonded selenium is 89^o.

4. The crystal structure of SnTe

Crystalline stannous telluride was classified by Goldschmidt (12) as cubic with a NaCl type lattice. The cell constant a_o is 6.298 A. A symmetrical octahedral arrangement of tellurium atoms surrounds the tin atom at a distance of 3.15 A. Stannous telluride is isomorphous with the cubic form of tin (II) selenide.

5. The crystal structure of $SnCl_2 \cdot 2H_2O$

Stannous chloride dihydrate is monoclinic with probable space group $P2_1/c$ (13). The cell constants and density are:

a_o	= 9.38 A	z_o	= 4
b_o	= 7.22 A	d_c	= 2.707 g/cc
c_o	= 9.02 A	d_o	= 2.710 g/cc
β	= 114° 58'.		

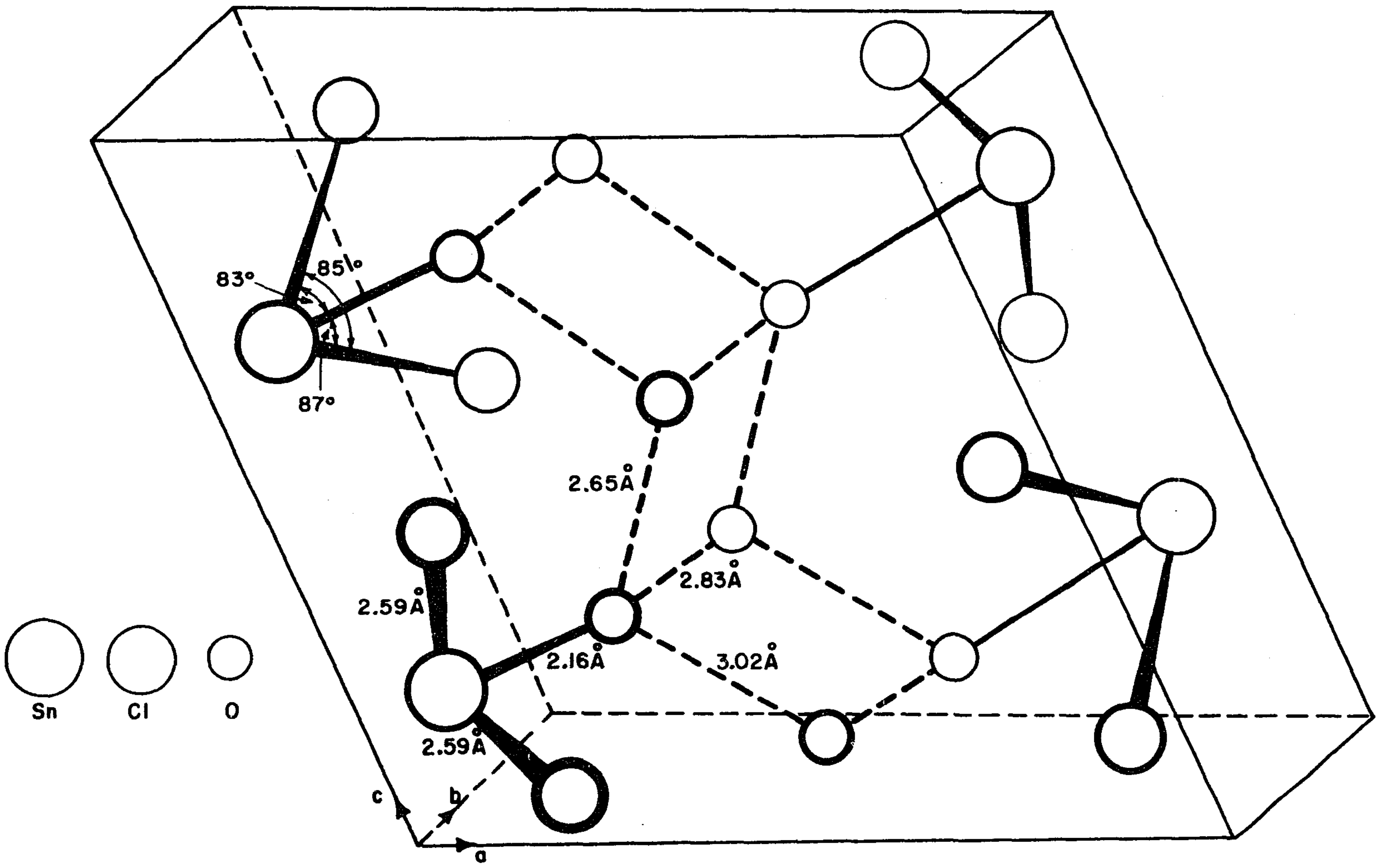
The intensity data for the solution of this structure were collected with an integrating Weissenberg camera and the optical densities were measured with a microdensitometer. Cylindrical absorption corrections were applied to the data. The structure was solved using two dimensional Patterson, Fourier and difference Fourier techniques. The reliability indices for 85 $\{hk0\}$ and 98 $\{h0\ell\}$ reflections are 9.2 % and 12.9 %, respectively. The final parameters and their standard deviations are given in Table 2.

Table 2. Positional parameters and standard deviations for $SnCl_2 \cdot 2H_2O$

	x	y	x	σ(x)(A)	σ(y)(A)	σ(z)(A)
Sn	0.126	0.250	0.217	0.003	0.006	0.004
Cl_I	0.196	0.005	0.448	0.011	0.019	0.014
Cl_{II}	0.213	0.001	0.065	0.014	0.019	0.018
O_I	0.374	0.320	0.327	0.029	0.066	0.040
O_{II}	0.436	0.705	0.358	0.030	0.074	0.032

The crystal structure of dichloroaquotin (II) hydrate, illustrated in Figure 3, can be described in terms of a four layer repeating unit stacked parallel to the b_oc_o plane. The four layer unit consists of a double layer of aquo complex molecules, $SnCl_2OH_2$, sandwiched between two layers of water of crystallization. Thus the order of layers is double layer aquo complex, double layer of water, double layer aquo complex, double layer of water, etc. The aquo complex molecule, Figure 3, is in the shape of a trigonal pyramid with the tin atom at the apex and two chlorine atoms and a water oxygen at the base positions. In the double layer of aquo complex molecules, the apices are facing inward thus exposing the coordinated water molecule to the double layer of water of crystallization. Extensive hydrogen bonding occurs. The coordinated water molecule has an approximately

Figure 3. Illustration of the crystal structure of $SnCl_2 \cdot 2H_2O$
(Hydrogen bonds are indicated by dotted lines)


83°
85°
87°
2.65Å
2.83Å
2.59Å
2.16Å
3.02Å
2.59Å
Sn
Cl
O
c
b
a

tetrahedral bond configuration. In addition to the Sn-O bond, it forms three hydrogen bonds to the neighboring water molecules in the adjacent double layer. (Hydrogen bonds are indicated by dashed lines in Figure 3.) Each water of crystallization molecule interacts with three hydrogen bonds leaving one singly bonded hydrogen with the O-H parallel to the b_o axis.

Evidence concerning the strength of the Sn-O bond is given by the observation of loss of one molecule of water under controlled conditions, 80° C and atmospheric pressure.

6. The crystal structure of $K_2SnCl_4 \cdot H_2O$

From the results of an X-ray study of a single two-dimensional projection, the crystal structure of $K_2SnCl_4 \cdot H_2O$ was reported to contain infinite chains formed by $SnCl_6$ octahedra sharing two opposite edges (14). This structure was redetermined recently by Grdenić and Kamenar and was found to contain trichlorostannite ions, $SnCl_3^-$ (15). The correct formulation for the structure is $KCl \cdot KSnCl_3 \cdot H_2O$. The two zones of two-dimensional intensity data, which were used for the recent structural study, were collected with an integrating Weissenberg camera. The intensities were measured with a microdensitometer and corrected for absorption. The lattice parameters are:

a_o = 8.24 ± 0.02 A　　z_o = 4
b_o = 12.05 ± 0.03 A　　d_c = 2.61 g/cc[1]
c_o = 9.14 ± 0.02 A.

The probable space group is Pbnm. The potassium ion and one of the three crystallographically independent chlorine atoms Cl_{II} are in the general eight-fold set, x,y,z; $\bar{x},\bar{y},\bar{z}$; $1/2-x,1/2+y,z$; $1/2+x,1/2-y,\bar{z}$; $1/2+x,1/2-y,1/2+z$; $1/2-x,1/2+y,1/2-z$; $x,y,1/2-z$; $\bar{x},\bar{y},1/2+z$. The other two crystallographically independent chlorine atoms, Cl_I and Cl_{III}, the tin atom and the water molecule lie on mirror planes at 1/4 and 3/4 z in the special four-fold set, $x,y,1/4$; $\bar{x},\bar{y},3/4$; $1/2-x,1/2+y,1/4$; $1/2+x,1/2-y,3/4$. The structure was solved using trial and error and Fourier methods. The final reliability indices for 129 {hk0} and 75 {0kℓ} reflections are 12 % and 11 %, respectively. The estimated standard errors in the interatomic distances and angles are ± 0.03 A and 1°, respectively. The final coordinates are given in Table 3.

In this structure, the tin atom sits at the apex of a trigonal pyramid formed by three chlorine atoms. The form and dimensions of this trichlorostannite ion can be seen in Figure 4. The next shortest Sn-Cl distance, 3.17 A, is 0.54 A

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[1] For the sake of comparison, the density of K_2SnCl_6 is 2.71 g/cc.

Figure 4. Illustration of the crystal structure of $K_2SnCl_4 \cdot H_2O$

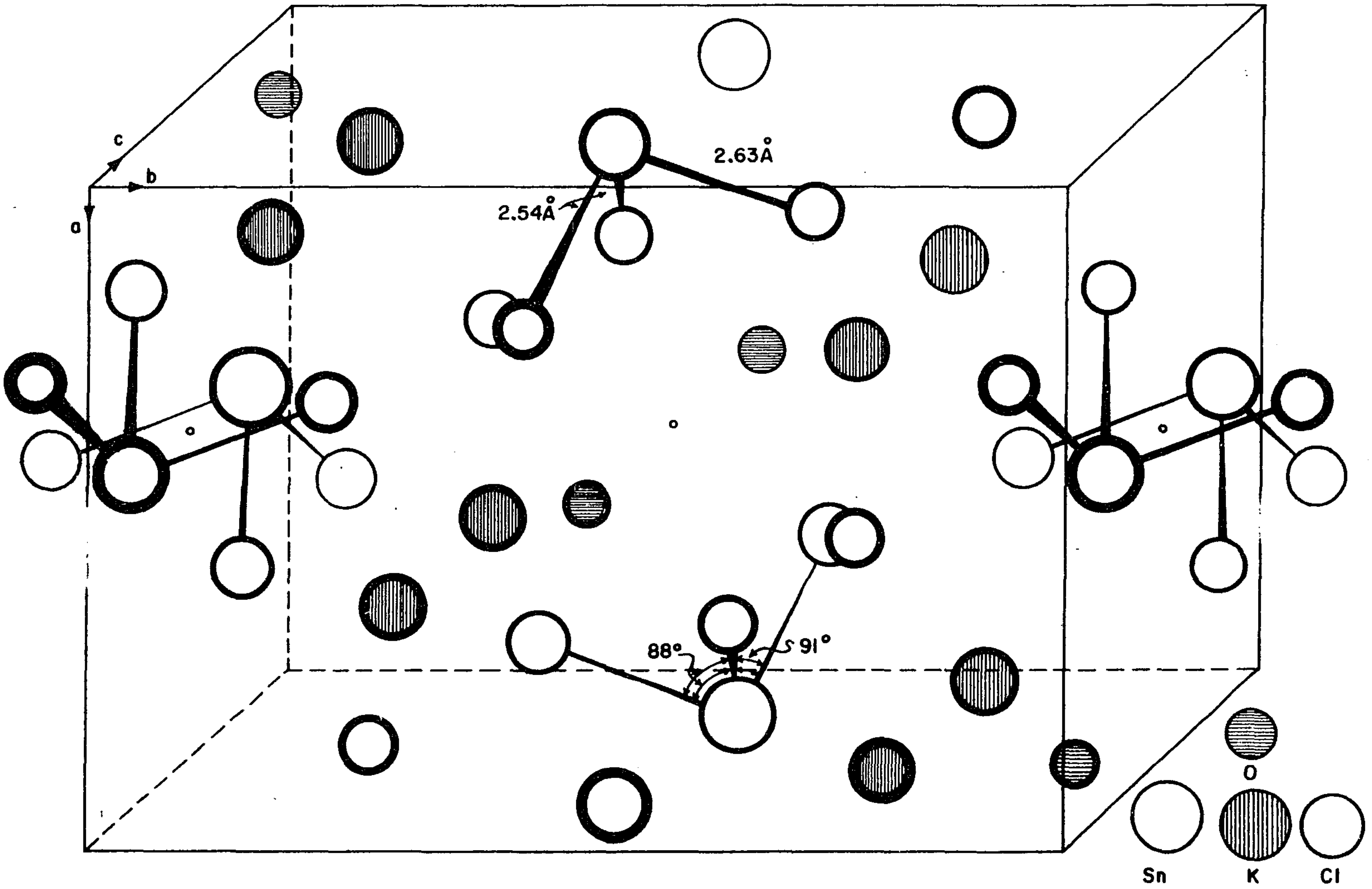
c
b
a
2.54Å
2.63Å
88°
91°
O
Sn
K
Cl

Table 3. Positional parameters for $K_2SnCl_4 \cdot H_2O$

	x	y	z
Sn	0.000	-0.012	1/4
K	0.370	0.185	-0.018
Cl_I	0.112	0.192	1/4
Cl_{II}	0.207	-0.056	0.448
Cl_{III}	-0.099	-0.266	1/4
O	0.559	-0.040	1/4

longer than any Sn-Cl distance within the trichlorostannite ion. The water molecule is not in the primary coordination sphere of the tin atom, but rather is in the polyhedron about the potassium ion which has seven-fold coordination.

7. The crystal structure of $SnSO_4$

The crystal structure of anhydrous $SnSO_4$, a highly hygroscopic, white compound, was determined by Rentzeperis (16). The X-ray data for this study were obtained with a Norelco diffractometer. The cell constants and density are:

$a_o = 8.799 \pm 0.001$ A $\quad z_o = 4$

$b_o = 5.319 \pm 0.001$ A $\quad d_c = 4.185$ g/cc

$c_o = 7.115 \pm 0.001$ A $\quad d_o = 4.15$ g/cc.

The probable space group is Pnma. Two of the oxygen atoms in $SnSO_4$ lie in the general eight-fold set, x,y,z; $\bar{x},\bar{y},\bar{z}$;

$1/2+x, 1/2-y, 1/2-z$; $1/2-x, 1/2+y, 1/2+z$; $\bar{x}, 1/2-y, z$; $1/2-x, \bar{y}, 1/2+z$; $1/2+x, y, 1/2-z$. All other atoms possess m symmetry and occupy the special four-fold set, $x, 1/4, z$; $\bar{x}, 3/4, \bar{z}$; $1/2-x, 3/4, 1/2+z$; $1/2+x, 1/4, 1/2-z$.

The structure was solved by noting the similarity between the X-ray powder pattern of $SnSO_4$ and $BaSO_4$ for which the crystal structure was known. By trial and error methods, it was determined that the tin atom position differed significantly from that of the barium atom; however the positions of the sulfate group atoms were quite similar for both structures. The final reliability index[1] is 7.3 % based upon 58 reflections. The final positional parameters are given in Table 4.

In this structure, the tin atom is surrounded by twelve oxygen atoms with Sn-0 distances ranging from 2.34 A to 3.29 A. It is interesting to note that there are three short Sn-0 distances, 2.34 A and 2.40 A (two). The 0-Sn-0 angles formed by these short Sn-0 bonds are 74° and 76° (two). The next shortest Sn-0 distance is 2.92 A.

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[1] This R value was computed by distributing the observed intensity, for overlapping reflections, in proportion to the corresponding calculated intensities for individual reflections.

Table 4. Positional parameters for $SnSO_4$

	x	y	z
Sn	0.208	1/4	0.222
S	0.069	1/4	0.694
O_I	-0.083	1/4	0.597
O_{II}	0.194	1/4	0.550
O_{III}	0.088	0.020	0.819

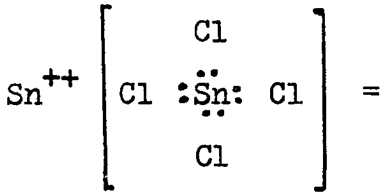
II. STRUCTURAL ANALYSIS

A. Structure of $SnCl_2$

1. Literature review

Stannous chloride is a very common and widely used chemical reagent. A few of its important uses are in qualitative and analytical analysis as a reducing agent and in the textile industry for weighting of fabrics. In spite of its wide usage, until recently very little was known about the structure of this tin (II) compound.

Aiken *et al.* (17) described anhydrous stannous chloride as a hygroscopic, white, glassy solid with melting point 246.8° and observed density 3.90 g/cc. From X-ray powder diagrams, they found $SnCl_2$ to be orthorhombic with lattice constants, $a_o = 6.61 \pm 0.03$ A, $b_o = 9.34 \pm 0.04$ A and $c_o = 9.98 \pm 0.04$ A. For eight molecules per unit cell, $d_c = 4.06$ g/cc. In addition, they reported that the powder pattern of indium dichloride, $InCl_2$, is similar to the powder pattern of $SnCl_2$. They suggested that these structures are very similar and that the structure of $SnCl_2$ is probably related to an intermediate compound, H_2SnCl_4, and is of the type



$$\mathrm{Sn}^{++}\left[\begin{array}{ccc} & \mathrm{Cl} & \\ \mathrm{Cl} & :\ddot{\underset{\cdot\cdot}{\mathrm{Sn}}}: & \mathrm{Cl} \\ & \mathrm{Cl} & \end{array}\right] = \quad .$$

Although the melting point, density, hygroscopic property and crystal description reported above are in agreement with observations of $SnCl_2$ in this laboratory and with other reports (18, 19), the X-ray data, the proposed structure and its similarity to that of $InCl_2$ are not reconcilable with presently known facts. The experimental $\sin^2\theta$ values given by Aiken *et al*. (17) do not agree with those computed from lattice parameters obtained in this experiment. Their proposed structure of $SnCl_2$ does not agree with the experimental results reported below. Other workers (20) investigating the lower halides of indium found no evidence for an $InCl_2$ compound and obtained X-ray patterns unlike those of Aiken *et al*. (17). In view of all the latter evidence, it is difficult to explain the X-ray results reported by Aiken *et al*. (17).

Lister and Sutton (21) did electron diffraction work on a series of metal halides including $SnCl_2$. $SnCl_2$ is monomeric in the gaseous state, at least at pressures less than 5 mm. For $SnCl_2$, they reported a Sn-Cl bond length of 2.42 ± 0.03 A and a Cl-Sn-Cl bond angle of 95°.

In addition to the structural work described above, Van den Berg (19) reported the results of the crystal structure study of $SnCl_2$. Somewhat previous to this time, the author and Dr. Rundle had submitted for publication a similar report as a letter to the editor. This report was not accepted as a letter and in view of Van den Berg's report was not resubmitted. Because of the similarity of the results of the author and Van den Berg, the latter's work will not be discussed.

2. Preparation and properties

Anhydrous stannous chloride was prepared by dehydrating reagent grade $SnCl_2 \cdot 2H_2O$. Dehydration was accomplished by pulling a vacuum on the dihydrate for 40 to 50 hours, the length of time depending upon sample size and particle size. Single crystals suitable for X-ray work were grown by vacuum sublimation. Best results were obtained by placing the sample in an evacuated tube and then placing the tube into a furnace at a temperature 10 - 40° below the melting point of $SnCl_2$. At these temperatures, sublimation occurs slowly and at least one week was required to get crystals of suitable size and quality. Higher temperatures resulted in rapid sublimation and crystals much too small for single crystal X-ray work. The experimental melting point of 246° C agreed with the reported value (18). Due to the hygroscopic tendencies and the instability of the divalent state of tin towards oxidation, special handling techniques were necessary.

The crystals were transferred, in a dry nitrogen atmosphere, from the vacuum sublimation tube into a layer of petroleum jelly on a glass slide. The jelly coated crystals could then be examined with a microscope in the atmosphere. The crystal used for X-ray examination was placed in a thin walled capillary. The capillary opening was fused shut with the flame of a match. The crystal was selected on the basis of size and apparent quality. Some indication of quality could be ascertained by examination with a polarizing microscope; however these observations were obscured by the coating of petroleum jelly on the crystals.

3. X-ray data

Preliminary lattice constants and space group data for stannous chloride were obtained from rotation, Weissenberg and precession photographs. The photographs exhibit orthorhombic symmetry D_{2h}. The systematic extinctions are:

$$k = 2n + 1 \quad \text{for } \{0k\ell\}$$

$$h + \ell = 2n + 1 \quad \text{for } \{h0\ell\} \quad .$$

In addition, alternate levels of the reciprocal lattice along c* are identical in all respects. This latter observation directs the choice of space group Pbnm over that of the other possibility, $Pbn2_1$. The similarity of alternate levels of the reciprocal lattice is indicative of all atoms lying on mirror planes. A cursory proof of this fact follows. The general structure factor is given by

$$F(hk\ell) = \sum_{1}^{n} f_i \exp [2\pi i\ (hx + ky + \ell z)]$$

where h, k and ℓ are the Miller indices, x, y and z are the fractional unit cell coordinates of atom i and f_i, a function of the Bragg angle, is the atomic scattering factor for atom i. The summation is over all n atoms of the unit cell. The complete structure factor for space group Pbnm is

$$F(hk\ell) = \sum_{i=1}^{n/8} f_i\ A(hk\ell)$$

where for $\ell = 2n$

$$A(hk\ell) = 8 \cos 2\pi hx \cos 2\pi ky \cos 2\pi \ell z \qquad h + k = 2n$$

$$A(hk\ell) = -8 \sin 2\pi hx \sin 2\pi ky \cos 2\pi \ell z \qquad h + k = 2n + 1$$

and for $\ell = 2n + 1$

$$A(hk\ell) = -8 \cos 2\pi hx \sin 2\pi ky \sin 2\pi \ell z \qquad h + k = 2n$$

$$A(hk\ell) = -8 \sin 2\pi hx \cos 2\pi ky \sin 2\pi \ell z \qquad h + k = 2n + 1 .$$

The summation is now over all asymmetric atoms. If all asymmetric atoms lie on mirror planes at 1/4z, this reduces to

$$F(hk\ell) = \sum_{i}^{n/4} f_i \, A(hk\ell)$$

where for $\ell = 2n$

$$A(hk\ell) = 4(-1)^{\ell/2} \cos 2\pi hx \cos 2\pi ky \qquad h + k = 2n$$

$$A(hk\ell) = 4(-1)^{\ell/2} \sin 2\pi hx \sin 2\pi ky \qquad h + k = 2n + 1$$

and for $\ell = 2n + 1$

$$A(hk\ell) = 4(-1)^{\frac{\ell+1}{2}} \cos 2\pi hx \sin 2\pi ky \qquad h + k = 2n$$

$$A(hk\ell) = 4(-1)^{\frac{\ell+1}{2}} \sin 2\pi hx \cos 2\pi ky \qquad h + k = 2n + 1 .$$

The observed intensity is proportional to the absolute value of the structure factor squared. Thus it can be seen that the intensities of reflections are equal when ℓ varies by two units.

Final a_o and b_o lattice constants were obtained from back-reflection Weissenberg photographs (22, p. 435) and the c_o lattice constant was obtained by correlating single crystal orienter measurements with the back-reflection measurements for a_o and b_o. The cell constants and the calculated and observed densities are:

$a_o = 9.20 \pm 0.01$ A $\qquad z_o = 4$

$b_o = 7.79 \pm 0.01$ A $\qquad d_o = 3.90$ g/cc (16)

$c_o = 4.43 \pm 0.01$ A $\qquad d_c = 3.97$ g/cc.

Three sets of intensity data were collected and used in the solution and refinement of this structure.

The first set of data, {hk0} data, was taken on a Weissenberg camera with Ni filtered Cu Kα irradiation using multiple film packs and 9, 18 and 36 hour exposures. The relative intensity of each independent reflection was determined visually using a series of standard, timed exposure spots. These data were corrected for Lorentz-polarization, Lp, factors on an IBM 650 with the INCOR IM program written by Walken and Jones.[1]

This first set of data was of quite poor quality. This was mainly due to the very high absorption coefficient of $SnCl_2$ for Cu Kα irradiation. The second set of data was taken with Mo Kα irradiation, a much more favorable situation since for Cu Kα, $\mu = 766.8$ cm^{-1} and for Mo Kα, $\mu = 101.7$ cm^{-1}.[2] This second set of data, {hk0} and {0kℓ}, was collected

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[1] INCOR IM was obtained from Fitzwater, D. R., Ames Laboratory, Iowa State University, Ames, Iowa. INCOR IM. Private communication. 1961.

[2] The attenuation of a beam of X-rays transmitted through a slab of thickness R and with absorption coefficient μ is given by $\exp[-\mu R]$.

on a G. E. XRD-5 single crystal orienter using Zr filtered Mo Kα irradiation. The angle settings were computed on an IBM 650 computor using the SCO-2 program written by Williams.[1] The intensity of each reflection was measured using a 100 second, 1.67° two-theta scan. The background was measured as a function of two-theta and corrections obtained for all data by graphical interpolation. Equatorial Lp corrections were made by the same method used for the first set.

In view of our interest in obtaining accurate bond distances for the two nonequivalent Sn-Cl bonds found in this structure, it was decided to collect a complete set of three-dimensional intensity data. The crystal selected was a rectangular polyhedron with dimensions 0.207 x 0.219 x 0.414 mm. Intensity data set three was collected on a G. E. XRD-5 crystal orienter using Zr filtered Mo Kα irradiation. The angle settings were computed on the Cyclone computor using the SCO-4 program.[2] The 200 second, 3.33° scan method was used. Background measurements and corrections were made and applied as for the previous set. During the collection

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[1] Williams, D. E., Ames Laboratory, Iowa State University, Ames, Iowa. SCO-2. Private communication. 1960.

[2] Burns, L. and Williams, D. E., both of Ames Laboratory, Atomic Energy Commission, Ames, Iowa. SCO-4. Private communication. 1961.

of these data, there was a 28 % drop in the intensity of the standard reflection. This drop was due to electronic drift of the X-ray equipment. The 810 reflections were recorded in a four week period. The data were adjusted for this electronic drift by the relation $I^{o} = (I^{o}_{STD}/I^{x}_{STD}) \cdot I^{x}$. Expressed in words, the intensity at time zero is equal to the quotient of the standard intensity at time zero divided by the standard intensity at time x times the measured intensity at time x. In view of the high absorption coefficient for $SnCl_2$, 101.7 cm^{-1}, absorption corrections were computed on the Cyclone computor using the ABCOR-I program written by Williams.[1] This program evaluates numerically the integral

$$A(\Phi, \chi, 2\theta) = 1/V \int_a^b \int_c^d \int_e^f \exp\left[-\mu(R_p + R_d)\right] dz\, dy\, dx$$

where the transmission factor A to be evaluated is a function of Φ, χ and 2θ, single crystal orienter variables; μ is the absorption coefficient and V is the volume of the crystal. R_p and R_d are the path lengths traveled by the incident and diffracted beams, respectively, and a and b, c and d, e and f are variable limits for the x, y and z coordinates,

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[1] Williams, D. E., Ames Laboratory, Iowa State University, Ames, Iowa. ABCOR-I. Private communication. 1960.

respectively. The integrand is evaluated at 64 points for each reflection condition. The crystal is defined by the equations of its bounding planes.

The data workup program written by Fitzwater[1] in the Fortran language for the IBM 704 was used to apply the transmission factors, compute and apply streak and Lorentz-polarization corrections, test the significance of each reflection and compute a $\sigma(F)$ value. A few words of explanation will be given for the streak correction, significance test and $\sigma(F)$ calculation. The streak correction is an attempt to correct for that part of the intensity of a reflection which is caused by white, noncharacteristic radiation, scattered by a peak with the same prime indices but of lower order than the peak to be corrected. This correction[2] is of the form

$$I_s^n = A_n \cdot 2 \cdot Lp_n / \mathrm{Tan}(\theta_n) \sum_{m=1}^{n-1} F_m{}^2 \left(\frac{n}{m} \lambda_o \cdot K(\lambda)\right)$$

where I_s^n is the contribution to the nth peak of a lattice row of the n - 1 preceding peaks in this row. A_n, Lp_n and

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[1] Fitzwater, D. R., Ames Laboratory, Iowa State University, Ames, Iowa. Data workup program. Private communication. 1961.

[2] This function was derived by D. R. Fitzwater and checked experimentally by J. B. Benson, both of Ames Laboratory, Iowa State University, Ames, Iowa. Streak correction. Private communication. 1960.

θ_n are the absorption factor, Lorentz-polarization factor and the theta angle respectively for the nth peak. F_m is the structure factor of the mth peak and λ_o is the wavelength of the characteristic X-ray. $K(\lambda)$ is an experimentally determined factor which is a function of the wavelength λ of the white radiation. To pass the significance test a reflection must obey the relation

$$I - I_s - 3\sigma(I) > 0$$

where I is the total counts minus background counts, I_s is I_s^n defined above and $\sigma(I)$ is the standard deviation of I. $\sigma(I)$ is defined as

$$\left[\text{total counts} + \text{background counts} + (\text{PCTS}\cdot I_s)^2\right]^{\frac{1}{2}} .$$

Here PCTS is the error in the streak correction, taken as 0.07. A reflection failing this test is called "unobserved". Its structure factor is then given the value

$$F(hk\ell) = \left[\sigma(I)/(3\cdot A\cdot Lp)\right]^{1/2}$$

and $\sigma(F) = 0.4472\ F$. The ideas in this unobserved treatment were set forth in a paper by Hamilton (23).

For an "observed" reflection, $\sigma(F)$ was calculated using the formula

$$\sigma(F) = F\cdot\left[PCTG^2 + \frac{PCTA^2}{4} + \frac{\text{total counts} + \text{background counts} + PCTS\cdot I_s}{4\cdot I^2}\right]^{\frac{1}{2}}$$

where PCTG and PCTA are the estimated errors in the total counts and the absorption correction, respectively. PCTS is the estimated error in the streak correction I_s. For this problem, the estimated values of PCTG and PCTA are 0.05 and 0.01, respectively.

4. Solution of the structure

As stated earlier, the probable space group for $SnCl_2$ is Pbnm and all atoms are assumed to lie on mirror planes at ± 1/4z. This means all atoms occupy the four-fold set, $x,y,1/4;\ \bar{x},\bar{y},3/4;\ 1/2-x,1/2+y,1/4;\ 1/2+x,1/2-y,3/4$. Using the first set of {hk0} data, a Patterson projection onto (001) was computed on an IBM 650 computor using the TDF-2 Fourier program written by Fitzwater and Williams.[1] The Patterson function was derived by A. L. Patterson in

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[1] Fitzwater, D. R. and Williams, D. E., Ames Laboratory, Iowa State University, Ames, Iowa. TDF-2. Private communication. 1960.

1935 (24). The Patterson function for the orthorhombic system is

$$P(u,v,w) =$$

$$\frac{8}{V}\sum_{h=0}^{\infty}\sum_{k=0}^{\infty}\sum_{\ell=0}^{\infty}|F(hk\ell)|^2 \cos 2\pi hu \cos 2\pi kv \cos 2\pi \ell w .$$

Briefly, a Patterson map is a contour plot containing peaks representing interatomic vectors which have been translated to the origin. All possible combinations of interatomic vectors between atoms of the unit cell appear on such a map. Normally, considerable overlapping of vector peaks occurs.

The interatomic vectors and their relative weights for each asymmetric atom are:

Wt.	Vector
4	0,0,0
4	$1/2 + 2x, 1/2, 0$
4	$1/2, 1/2 + 2y, 1/2$
2	$2x, 2y, 1/2$
2	$2\bar{x}, 2y, 1/2$.

The tin atom position, $x = 8/80$, $y = 20/80$, was determined by examination of this (001) map. This position explained the large peaks on the map. Resolution of smaller peaks was very poor and the chlorine atom positions could not be determined.

A Fourier map projected onto (001) was computed using as coefficients in this summation only those structure factors with major contribution from the tin atom. The two groups of data for which the tin atom in this position may control the sign of the structure factor are $k = 2n$ for $h + k = 2n$ and $k = 2n + 1$ for $h + k = 2n + 1$. This may be seen by inspection of the structure factor, which for $\{hk0\}$ data is

$$F(hk0) = 4 \sum_{i=1}^{n/4 \; h+k=2n} f_i \cos 2\pi hx \cos 2\pi ky$$

$$+ 4 \sum_{i=1}^{n/4 \; h+k=2n+1} f_i \sin 2\pi hx \sin 2\pi ky \; .$$

The structure factor calculation, from which the coefficients for the Fourier were prepared, was computed on an IBM 650 computer using the LSII-M program.[1] This Fourier was computed on the same computer using the TDF-2 program. The Fourier function for space group Pbnm is:

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[1] LSII-M is a structure factor and least squares program written by Senko and Templeton. This program was obtained from D. R. Fitzwater, Ames Laboratory, Iowa State University, Ames, Iowa. Private communication. 1960.

$$\begin{aligned} \rho(x,y,z) = \frac{8}{V}\Bigg[& \sum_{0}^{\infty}\sum_{0}^{\infty}\sum_{0}^{\infty}{}^{h+k=2n,\ \ell=2n} F(hk\ell)\cos 2\pi hx \cos 2\pi ky \cos 2\pi \ell z \\ & - \sum_{0}^{\infty}\sum_{0}^{\infty}\sum_{0}^{\infty}{}^{h+k=2n,\ \ell=2n+1} F(hk\ell)\cos 2\pi hx \sin 2\pi ky \sin 2\pi \ell z \\ & - \sum_{0}^{\infty}\sum_{0}^{\infty}\sum_{0}^{\infty}{}^{h+k=2n+1,\ \ell=2n} F(hk\ell)\sin 2\pi hx \sin 2\pi ky \cos 2\pi \ell z \\ & - \sum_{0}^{\infty}\sum_{0}^{\infty}\sum_{0}^{\infty}{}^{h+k=2n+1,\ \ell=2n+1} F(hk\ell)\sin 2\pi hx \cos 2\pi ky \sin 2\pi \ell z \Bigg] . \end{aligned}$$

Other than the tin atom peak, the map had insufficient detail to enable location of the chlorine atom positions. As indicated earlier, the poor quality of these data was mainly due to the high absorption of $SnCl_2$ for Cu Kα irradiation. The problem of high absorption was greatly reduced by collecting the second set of data with Mo Kα irradiation.

A (001) Patterson projection was computed with this set of data. This map is shown in Figure 5. The tin position obtained earlier agreed with the information on this map. No serious attempt was made to find the chlorine atom positions from this Patterson.

A (001) Fourier map was computed as discussed earlier. This Fourier map is shown in Figure 6. Because of the y = 1/4 value of the trial tin atom parameter and the resulting

Figure 5. Patterson projection onto the (001) plane for $SnCl_2$

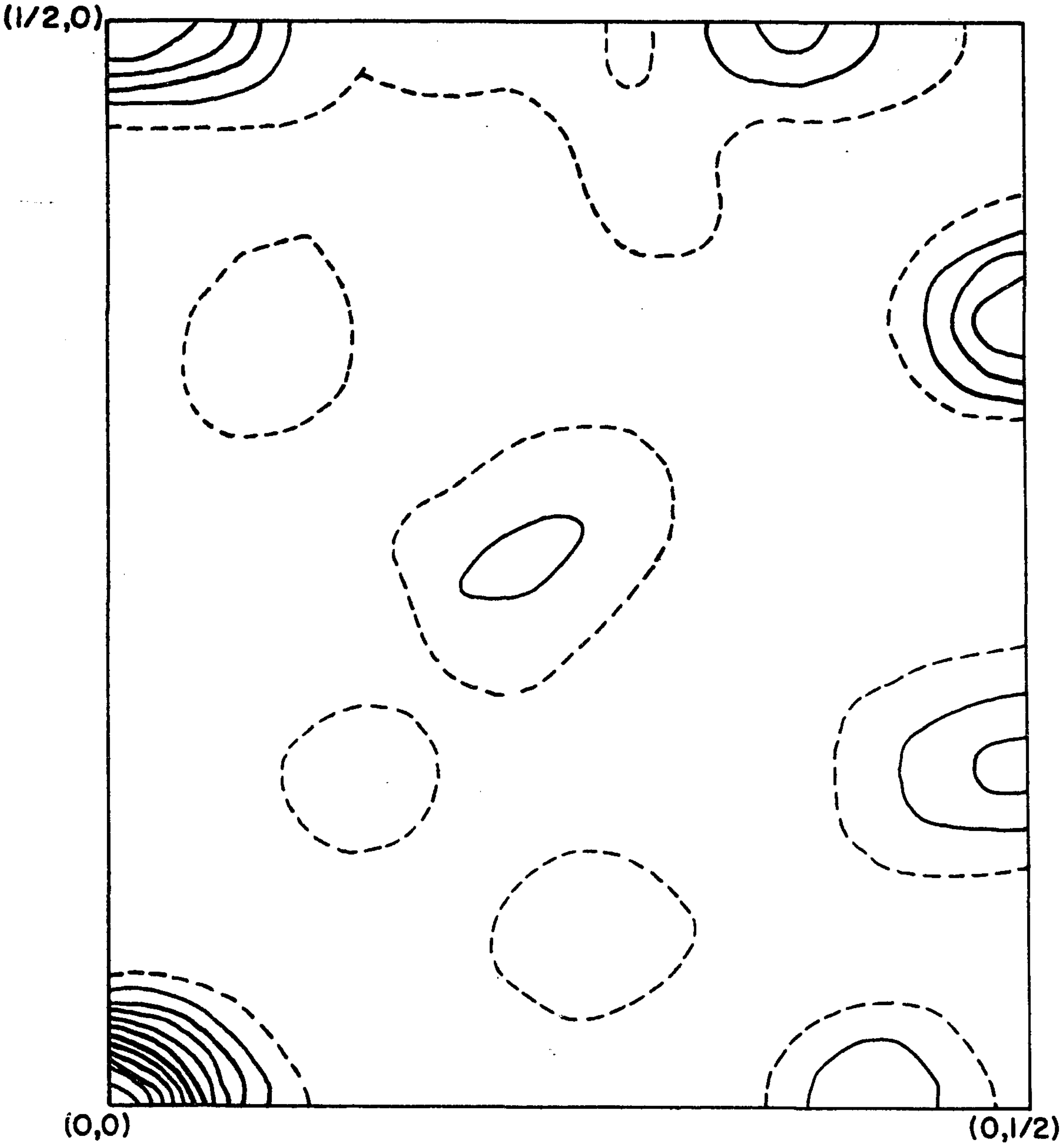
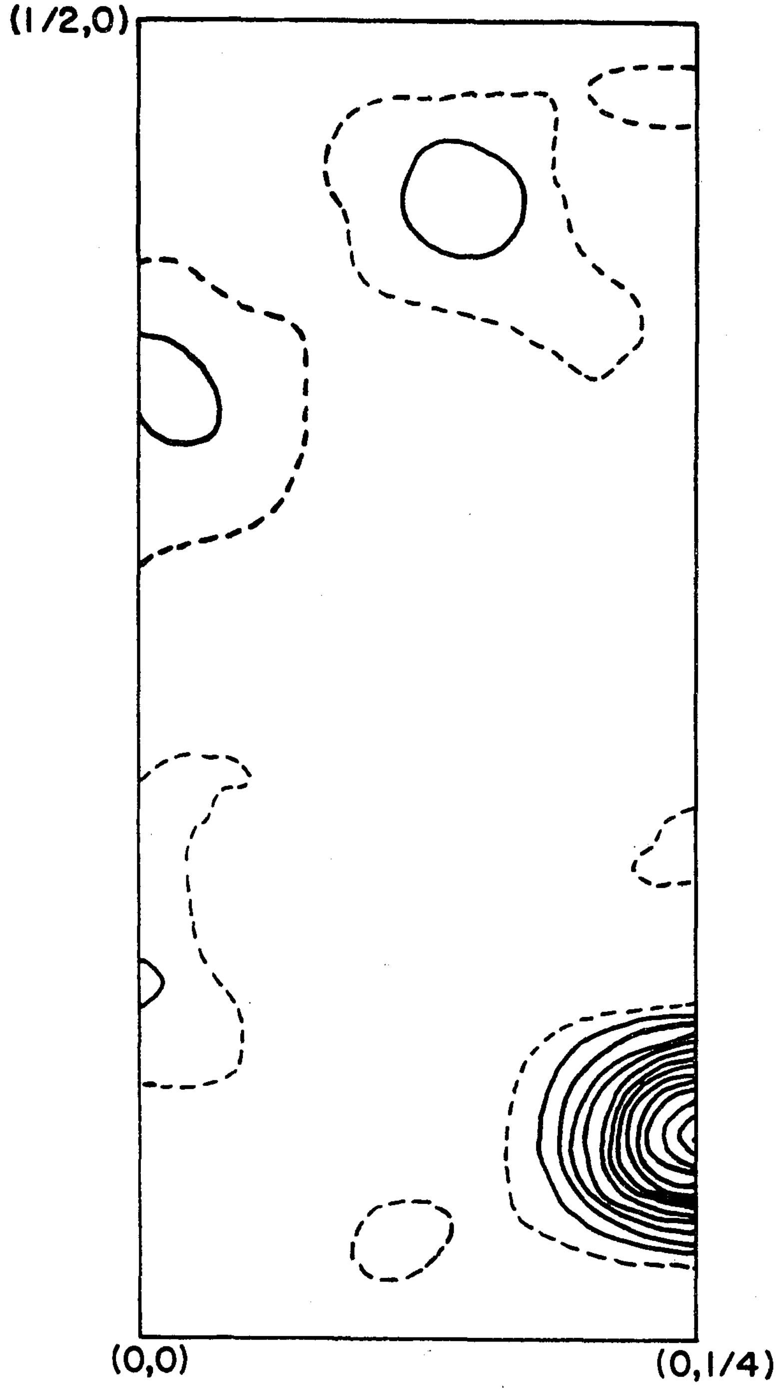

(1/2,0)
(0,0)
(0,1/2)

Figure 6. The first Fourier projection onto the (001) plane for $SnCl_2$


(1/2,0)
(0,0)
(0,1/4)

special set of data used to compute this Fourier, a mirror plane was introduced at y = 1/4. Therefore the map shown includes the area from the origin to x = 1/2, y = 1/4 rather than to x = 1/2, y = 1/2 for the normal asymmetric section. Two chlorine positions were found which explained the Patterson projection and represented a reasonable structure. The tin atom position and the two chlorine atom positions are:

	x	y	z
Sn	0.075	0.250	1/4
Cl_I	0.425	0.150	1/4
Cl_{II}	0.338	0.469	3/4 .

The refinement of these parameters will be discussed next.

5. Refinement

From the above parameters, structure factors were computed using LSII-M and an IBM 650 computer. A (001) Fourier map was computed using as coefficients the observed structure factors having the phases of the computed structure factors. The parameters obtained from this map were refined using LSII-M. After four cycles, the R value was 21.2 %. The calculated and observed structure factors are listed in Table 5. The parameters and standard deviations are given in Table 6. A (001) Fourier map computed following these four cycles is shown in Figure 7.

Table 5. Comparison of observed and calculated structure factors for {hk0} data set two for $SnCl_2$ (* denotes unobserved reflection)

h	k	F_o	F_c	h	k	F_o	F_c	h	k	F_o	F_c
2	0	285	298	5	2	187	-127	3	4	193	186
4	0	275	-231	6	2	323	348	4	4	148	-113
6	0	307	-296	7	2	43	30	5	4	82	73
8	0	260	-232	8	2	216	201	6	4	107	-112
10	0	61	-42	9	2	123	99	7	4*	13	-10
12	0	178	173	10	2	67	-50	8	4	92	-105
1	1	77	51	11	2	65	58	9	4	82	-78
2	1	240	-254	12	2	90	-103	10	4	39	-35
3	1	192	-114	1	3	90	29	11	4	98	-90
4	1	276	-280	2	3	291	354	12	4	63	87
5	1	54	20	3	3	144	-89	1	5	195	192
6	1	93	-77	4	3	272	302	2	5	204	-220
7	1	116	72	5	3	22	28	3	5	40	-31
8	1	160	120	6	3	94	72	4	5	271	-304
9	1	51	-36	7	3	95	77	5	5	113	-98
10	1	148	146	8	3	135	-118	6	5	105	-92
11	1	32	-21	9	3	40	-25	7	5	82	-81
12	1	57	62	10	3	195	-187	8	5	161	157
1	2	106	-75	11	3*	16	-21	9	5	84	-70
2	2	263	-291	12	3	84	-93	10	5	141	165
3	2	208	-180	1	4	157	125	11	5*	17	27
4	2	82	48	2	4	111	105	12	5	45	59

Table 5. (Continued)

h	k	F_o	F_c	h	k	F_o	F_c	h	k	F_o	F_c
1	6	22	-15	9	7	45	44	4	9	24	-66
2	6	98	-109	10	7	70	-98	5	9	68	-100
3	6	71	-98	11	7	48	-61	6	9	22	-29
4	6*	13	12	12	7*	19	-59	7	9	66	-111
5	6	110	-109	1	8	83	90	8	9	29	49
6	6	129	152	2	8	67	79	9	9	26	-55
7	6	29	29	3	8	76	118	10	9*	19	25
8	6	80	89	4	8	41	-63	11	9*	20	38
9	6	54	81	5	8*	15	31	12	9*	20	-1
10	6*	17	-27	6	8	75	-99	1	10*	16	-37
11	6*	18	29	7	8	26	1	2	10	38	-61
12	6	26	-47	8	8	68	-82	3	10	83	-131
1	7	170	-180	9	8	23	-46	4	10*	17	22
2	7	155	176	10	8	21	-12	5	10	95	-123
3	7	67	-66	11	8	49	-74	6	10	47	86
4	7	67	115	12	8*	20	64	7	10*	18	32
5	7	115	141	1	9	106	155	8	10	39	58
6	7	17	21	2	9	55	1	9	10	55	104
7	7	171	188	3	9*	15	7	10	10*	20	-8
8	7*	16	-36								

Table 6. Intermediate parameters and standard deviations for $SnCl_2$

	x	y	z	$\sigma(x)$	$\sigma(y)$
Sn	0.0753	0.2313	1/4	0.0006	0.0007
Cl_I	0.4243	0.1443	1/4	0.0023	0.0029
Cl_{II}	0.3470	0.4793	3/4	0.0022	0.0030

In view of the large estimated error, $3\sigma \geq 0.06$ A for a Sn-Cl bond, it was decided to collect a complete set of three-dimensional data. As stated earlier, the greater accuracy was desired in order to have increased confidence in comparing the two unique Sn-Cl distances.

The 810 reflections of data set three were refined using a full matrix least squares program written for the IBM 704 by Busing and Levy (25). The R value after five cycles of isotropic and five cycles of anisotropic least squares was 10.8 %. A list of the calculated and observed structure factors is given in Figure 8. After the second cycle of anisotropic least squares, 18 low order reflections were removed since it appeared that they were seriously affected by primary extinction. Eight of these were included in the last cycle of refinement. The final positional and thermal parameters and their errors are shown in Table 7. The distances, angles and their errors were computed using

Figure 7. Fourier projection onto the (001) plane for $SnCl_2$

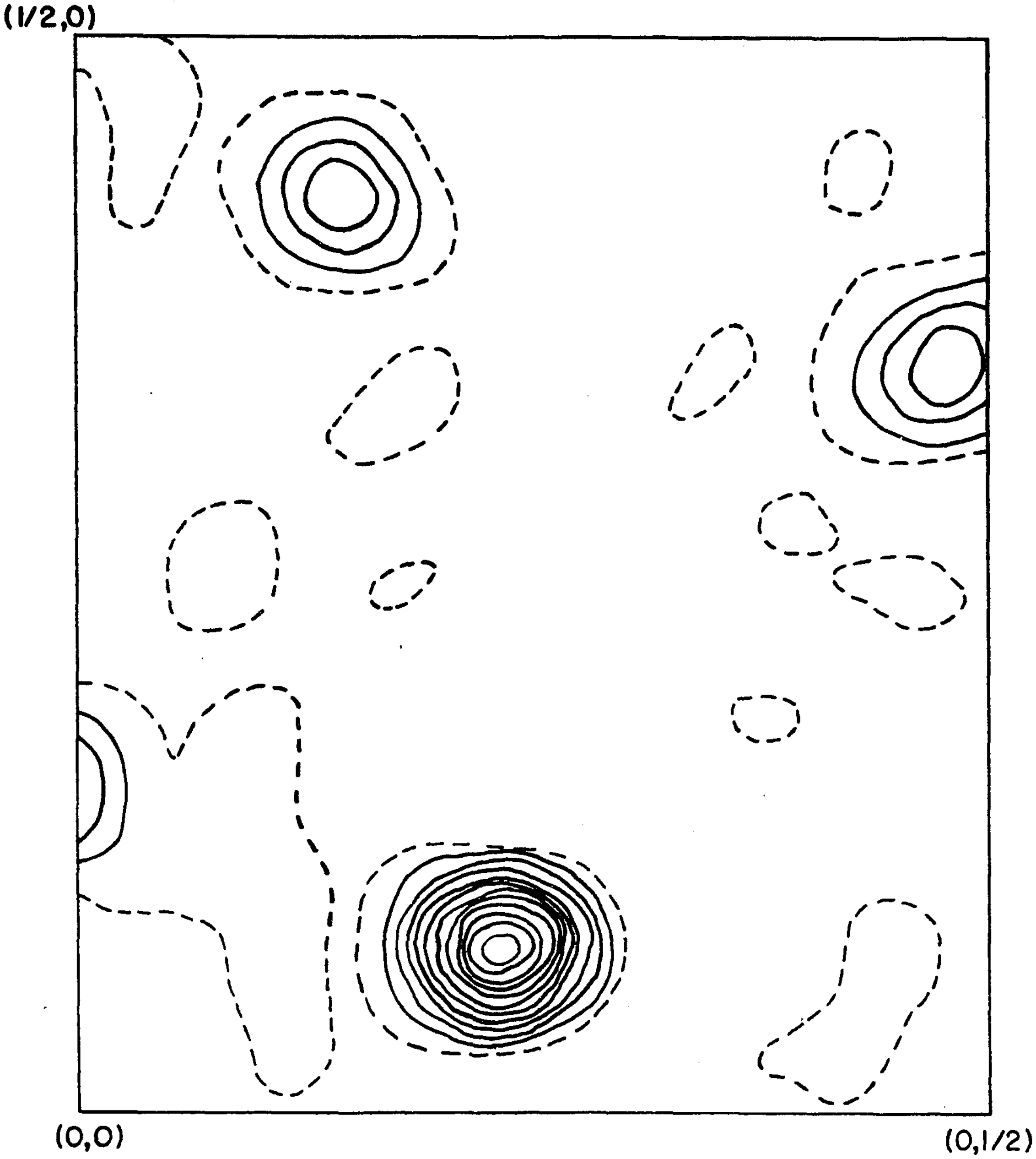

(1/2,0)
(0,0)
(0,1/2)

Figure 8. Comparison of observed and calculated structure factors for $SnCl_2$

(The reflections are grouped into sections of constant k and ℓ. The first column is the running index h; the second column is $5|F_o|$; the third column is $5F_c$. An asterisk following the running index denotes an unobserved reflection.)

```
   H  0  0

 2 435  568
 4 460 -429
 6 491 -514
 8 373 -386
10 109  -69
12 211  227
14 156  159

   H  1  0

 1 110   93
 2 386 -471
 3 270 -223
 4 444 -482
 5  70   51
 6 117 -130
 7 147  132
 8 208  185
 9  66  -63
10 176  187
11• 19  -29
12  61   67
13  38   30
14• 20  -18

   H  2  0

 0 494  -12
 1 152 -131
 2 444 -538
 3 330 -341
 4 112   85
 5 255 -240
 6 518  523
 7  61   52
 8 297  297
 9 148  150
10  84  -77
11  78   79
12 106 -109
13• 18   16
14  83  -85

   H  3  0

 1 127   65
 2 520   24
 3 209 -167
 4 443  484
 5  34   36
 6 120  119
 7 112  110
 8 171 -160
 9  49  -44
10 249 -255
11• 19  -11
12 104 -108
13• 20   37
14  39   48

   H  4  0

 0 479  506
 1 260  228
 2 158  153
 3 299  300
 4 209 -181
 5 104   98
 6 138 -135
 7• 14    6
 8 115 -120
 9 102  -97
10  59  -47
11 109 -116
12  70   83
13• 20   -3
14  56   56

   H  5  0

 1 326  305
 2 338 -323
 3  47  -45
 4 438 -449
 5 159 -132
 6 133 -125
 7 103 -102
 8 210  224
 9 100 -100
10 176  180
11• 20   15
12  58   54
13  69   79

   H  6  0

 0 143 -126
 1  36   23
 2 144 -135
 3  99  -95
 4  27    2
 5 151 -143
 6 180  172
 7  35   44
 8  91   88
 9  59   68
10• 19  -33
11• 21   -5
12  42  -27
13• 20   10

   H  7  0

 1 244 -230
 2 227  229
 3 103  -87
 4  94   93
 5 159  162
 6• 15    4
 7 227  217
 8• 18  -15
 9  50   50
10  79  -89
11  58  -56
12  50  -41

   H  8  0

 0 194  205
 1 105  103
 2  90   91
 3  93   91
 4  61  -71
 5• 16  -13
 6  87 -104
 7  40   29
 8  91  -70
 9• 20   -6
10  39  -12
11  67  -56

   H  9  0

 1 135  128
 2  74   56
 3• 16    6
 4  33  -43
 5  90  -82
 6• 18  -16
 7  77  -74
 8• 20   45
 9  53  -28
10• 22  -12

   H 10  0

 0  83  -90
 1• 17   -3
 2• 16  -49
 3 101  -91
 4• 17   25
 5 118 -113
 6  54   67
 7• 19   26
 8  56   47
 9  55   67

   H 11  0

 1  35  -21
 2• 17   40
 3• 17    1
 4  38  -24
 5• 18    6
 6• 19  -13
 7• 19   13

   H 12  0

 0  40   -6
 1  60   61
 2• 17    3
 3  84   77
 4• 18   -1
 5• 19   16

   H  0  1

 1 318 -290
 3  10 -228
 5 567 -610
 7 182  152
 9 388  406
11 168  176
13  35   27

   H  1  1

 1 457 -563
 2 303  255
 3  60  -53
 4  90  -81
 5 334  299
 6  60  -49
 7 303  297
 8  85   82
 9  95  110
10  54  -44
11  75  -65
12  52  -48
13 100  -98
14  41   35

   H  2  1

 0 458 -458
 1 471  516
 2 206 -186
 3 575   64
 4 135  121
 5 233  236
 6 188  179
 7  38  -17
 8 154  142
 9 213 -209
10• 18   21
11 212 -229
12  76  -81
13• 19  -12
14  55  -57

   H  3  1

 1 523  557
 2 179  178
 3 175  162
 4  79  -52
 5 365 -361
 6  40  -31
 7 390 -406
 8  88   82
 9  90  -89
10• 19  -21
11  92   90
12• 21  -40
13 105  105
14• 20   19

   H  4  1

 0 218  211
 1  43  -44
 2 220  202
 3 266 -265
 4  21  -23
 5 264 -263
 6 245 -246
 7  62   70
 8 116 -119
 9 158  155
10  39   33
11  40   31
12• 22   40
13• 19    4
14  48   40

   H  5  1

 1 498 -485
 2 110 -108
 3  29   20
 4 263 -261
 5 254  255
 6 106  -97
 7 240  242
 8 117  125
 9 133  132
10  97   87
11  59  -73
12• 21   19
13 121 -127

   H  6  1

 0 214 -208
 1 147  141
 2  34  -31
 3 189  183
 4  81   82
 5  41   40
 6• 14    7
 7• 15    0
 8  57   46
 9  36  -42
10  53   39
11  72  -70
12  43  -39
13• 21   -8

   H  7  1

 1 103  107
 2 276  259
 3 108   96
 4 223  203
 5 106 -118
 6  54   42
 7 151 -153
 8  70  -62
 9• 19   10
10 111 -115
11• 21   41
12  70  -64

   H  8  1

 0• 14    3
 1  46  -53
 2  81   75
 3 132 -141
 4  30   32
 5 122 -116
 6 103 -102
 7  33   22
 8  53  -37
 9  80   89
10• 21   25
11  52   43

   H  9  1

 1• 15  -38
 2  95  -87
 3  50   62
 4 118 -108
 5• 17   -6
 6• 19  -36
 7  35  -39
 8  51   53
 9• 21   23
10  73   58

   H 10  1

 0 164 -161
 1• 16   42
 2  54  -43
 3  71   86
 4  57   60
 5• 18   44
 6  51   37
 7• 19  -20
 8  57   51
 9• 22  -38

   H 11  1

 1• 17  -22
 2• 17   12
 3  37   38
 4  35   21
 5• 18   -5
 6• 19    0
 7• 19  -25

   H 12  1

 0  58   41
 1• 17   -8
 2  53   51
 3• 18   -0
 4• 18    0
 5• 19    8

   H  0  2

 0 128 -400
 2 417 -390
 4 351  311
 6 396  388
 8 292  304
10  46   58
12 170 -185
14 120 -130

   H  1  2

 1  64  -59
 2 345  337
 3 188  152
 4 377  375
 5  57  -43
 6  93   95
 7 128 -113
 8 154 -139
 9  47   51
10 139 -141
11• 20   25
12  41  -51
13• 19  -24
14• 20   14

   H  2  2

 0 418  436
 1  89   90
 2 366  381
 3 257  247
 4  76  -59
 5 203  188
 6 447 -457
 7  48  -42
 8 238 -231
 9 123 -124
10  67   64
11  72  -65
12  89   86
13• 19  -13
14  76   67

   H  3  2

 1  81  -47
 2 442 -467
 3 140  125
 4 374 -380
 5• 12  -20
 6  89  -91
 7  83  -81
 8 133  123
 9• 19   40
10 209  203
11• 19    8
12  86   87
13• 19  -34
14• 21  -39

   H  4  2

 0 362 -367
 1 181 -171
 2 109 -105
 3 230 -229
 4 150  137
 5  75  -73
 6  95   90
 7• 16   -6
 8  65   87
 9  81   77
10• 20   40
11  99   95
12  68  -64
13• 19    2

   H  5  2

 1 244 -234
 2 261  246
 3  44   39
 4 369  375
 5 105  102
 6 109  100
 7  76   78
 8 171 -182
 9  87   81
10 149 -145
11• 21  -11
12  47  -43
13  60  -65

   H  6  2

 0  80   85
 1  33  -25
 2 111  101
 3  66   64
 4• 14    3
 5 115  111
 6 129 -132
 7  31  -35
 8  72  -67
 9  50  -52
10• 20   27
11• 21    8
12• 21   19

   H  7  2

 1 190  184
 2 178 -182
 3  82   71
 4  73  -69
 5 127 -131
 6• 16   -1
 7 183 -178
 8• 20    9
 9  42  -41
10  66   71
11  45   46
12• 21   33

   H  8  2

 0 157 -166
 1  81  -82
 2  77  -73
 3  81  -67
 4  44   58
 5• 17   16
 6  74   84
 7  33  -25
 8  60   57
 9• 20    1
10• 21   11
11  46   44

   H  9  2

 1 110 -102
 2  64  -52
 3• 16   -4
 4• 17   32
 5  73   66
 6• 19   12
 7  59   58
 8• 22  -36
 9• 21   22
10• 22   12

   H 10  2

 0  59   75
 1• 16    1
 2  32   40
 3  81   73
 4• 17  -20
 5  99   93
 6  43  -56
 7• 20  -22
 8• 22  -39

   H 11  2

 1• 17   14
 2• 17  -33
 3• 17   -2
 4• 18   21
 5• 19   -3
 6• 20   11

   H 12  2

 0• 17    6
 1  54  -51
 2• 18   -3
 3  73  -64

   H  0  3

 1 175  148
 3 460  475
 5•392  397
 7 103  -98
 9 270 -263
11 108 -111
13• 19  -18

   H  1  3

 1 262  294
 2 158 -140
 3  24   25
 4  39   42
 5 175 -158
 6  34   31
 7 171 -162
 8  57  -54
 9  60  -64
10• 18   35
11  42   37
12• 21   35
13  66   59

   H  2  3

 0 241  247
 1 264 -278
 2 101  105
 3 374 -386
 4  74  -75
 5 125 -124
 6 113 -116
 7• 16    6
 8 100  -96
 9 132  125
10• 19  -14
11 142  149
12  44   57
13• 19    7

   H  3  3

 1 291 -306
 2  80  -94
 3 107  -97
 4  65   53
 5 217  215
 6  31   27
 7 251  252
 8  71  -65
 9  56   54
10• 19    7
11  44  -58
12• 22   25
13  66  -68

   H  4  3

 0 104 -118
 1• 14    8
 2 114 -121
 3 126  132
 4• 13   11
 5 155  154
 6 147  158
 7• 17  -44
 8  76   76
 9  96  -92
10• 20  -23
11• 20  -11
12• 21  -26
13• 20   -2

   H  5  3

 1 289  300
 2  46   57
 3• 14  -17
 4 155  163
 5 155 -161
 6  69   63
 7 154 -154
 8  70  -82
 9  93  -88
10  64  -55
11  42   48
12• 21  -11

   H  6  3

 0 108  119
 1  81  -84
 2• 15    8
 3 101 -102
 4  36  -51
 5• 15  -13
 6• 16    9
 7• 18   -3
 8• 20  -23
 9• 19   20
10• 20  -27
11  47   44

   H  7  3

 1  50  -60
 2 170 -171
 3  72  -65
 4 139 -132
 5  58   74
 6• 16  -27
 7  96   99
 8  41   41
 9• 21  -10
10  77   78
11• 22  -27

   H  8  3

 0• 15   12
 1• 15   33
 2  47  -44
 3  83   92
 4• 17  -26
 5  88   78
 6  54   62
 7• 19  -14
 8• 22   20
 9  51  -60
10• 21  -18

   H  9  3

 1• 16   20
 2  58   54
 3  36  -44
 4  76   69
 5• 18    9
 6• 19   23
 7  36   32
 8• 21  -35

   H 10  3

 0 105  108
 1• 17  -29
 2  38   27
 3  49  -59
 4  42  -40
 5• 18  -30
 6• 20  -22
 7• 20   14

   H 11  3

 1• 17   17
 2• 17   -4
 3  34  -27
 4• 18  -10

   H  0  4

 0 438  451
 2 203  171
 4 198 -155
 6 189 -184
 8 157 -156
10  21  -34
12  99  101

   H  1  4

 1  31   20
 2 139 -126
 3  96  -83
 4 164 -150
 5  38   31
 6  39  -40
 7  79   73
 8  60   61
 9• 20  -28
10  63   61
11• 20  -16
12• 20   22

   H  2  4

 0 179 -174
 1  46  -44
 2 169 -171
 3 125 -129
 4  29   17
 5 109 -103
 6 229  226
 7  33   24
 8 127  114
 9  62   72
10• 20  -38
11• 20   38
12• 23  -42

   H  3  4

 1  57   47
 2 208  221
 3  65  -66
 4 178  175
 5• 14   -1
 6  51   43
 7• 17   33
 8  79  -58
 9• 20  -28
10 106 -106
11• 21   -2
12  53  -47

   H  4  4

 0 154  156
 1  78   88
 2  37   32
 3 100  114
 4  70  -66
 5  31   32
 6• 15  -21
 7• 18    4
 8  45  -33
 9  56  -40
10• 21  -24
11  50  -52

   H  5  4

 1 110  116
 2 115 -120
 3  27  -26
 4 187 -197
 5  39  -49
 6  63  -54
 7• 17  -35
 8  93  100
 9  54  -45
10  87   78
11• 22    4

   H  6  4

 0• 15  -22
 1  35   25
 2  42  -44
 3• 16  -17
 4• 16   -9
 5  50  -53
 6  62   61
 7• 19   19
 8• 21   30
 9• 20   23
10• 21  -15

   H  7  4

 1  91  -97
 2  85   95
 3  37  -40
 4  42   28
 5  55   72
 6• 17   -1
 7  98   99
 8• 21   -1
 9• 21   22

   H  8  4

 0  82   90
 1  36   42
 2  40   39
 3• 17   26
 4  32  -32
 5• 18  -17
 6  38  -45
 7• 20   16
 8  51  -31

   H  9  4

 1  55   53
 2  39   37
 3• 17    1
 4• 18  -12
 5• 19  -35
 6• 20   -6
 7• 19  -30

   H 10  4

 0  33  -43
 1• 17    1
 2• 17  -23
 3  38  -39
 4• 18   11

   H  0  5

 1  56  -49
 3 197 -186
 5 189 -170
 7• 19   44
 9 114  117
11  53   47

   H  1  5

 1  82  -84
 2  75   74
 3• 15   -4
 4• 16  -12
 5  54   47
 6• 17  -13
 7  60   49
 8• 18   24
 9• 20   22
10• 20  -21
11• 21  -12

   H  2  5

 0 100 -113
 1 101  112
 2  38  -47
 3 138  146
 4  40   36
 5  48   37
 6  56   55
 7• 20    1
 8  38   46
 9  48  -47
10• 21    6

   H  3  5

 1 104  114
 2• 15   30
 3  44   42
 4  47  -42
 5  80  -86
 6• 17  -17
 7  97 -105
 8• 22   37
 9  44  -21
10• 23    3

   H  4  5

 0  36   44
 1  31   12
 2  42   51
 3  38  -31
 4• 16   -2
 5  65  -56
 6  62  -71
 7• 18   18
 8• 21  -33
 9• 21   32
10• 22   12

   H  5  5

 1 120 -130
 2• 17  -17
 3• 16   11
 4  58  -69
 5  55   69
 6  37  -28
 7  71   66
 8• 20   37
 9  44   41

   H  6  5

 0• 17  -41
 1  30   32
 2• 17    5
 3• 17   32
 4• 17   21
 5• 17   -3
 6• 18   15
 7• 20    4
 8• 22    4

   H  7  5

 1  39   19
 2  78   77
 3  32   31
 4  53   57
 5  32  -32
 6• 18   11
 7  36  -43

   H  8  5

 0• 17  -13
 1• 17  -14
 2• 17   13
 3  43  -41
 4• 18   16
 5  35  -36

   H  9  5

 1• 17   -4
 2• 18  -22
 3• 18   23

   H  0  6

 0 168 -160
 2  70  -56
 4  67   57
 6  82   61
 8  53   56

   H  1  6

 1• 16   -7
 2  36   25
 3  43   35
 4  50   33
 5• 18  -16
 6• 19   13
 8• 19  -15

   H  2  6

 0  49   45
 1• 17   17
 2  57   56
 3  50   52
 4• 17   -3
 5  45   42
 6  85  -78
 7• 21  -13
 8• 23  -38

   H  3  6

 1  43  -37
 2  70  -75
 3  32   25
 4  53  -56
 5• 18    7
 6• 18  -14
 7• 19   -6

   H  4  6

 0  46  -41
 1• 18  -33
 2• 17   -9
 3  35  -41
 4• 17   21
 5• 17   -9
 6• 18   -5
 7• 22   -2

   H  5  6

 1• 18  -41
 2  45   41
 3• 18   12
 4  68   74
 5• 18   16
 6• 18   21

   H  6  6

 0• 18   -3
 1• 18  -15
 2• 19   11
 3• 18   -2
 4• 18    7
 5• 18   15

   H  7  6

 1  39   37
 2  47  -34
```

Table 7. Final parameters and their standard errors for $SnCl_2$ (The standard errors are in parentheses below the parameter.)

	Sn	Cl_I	Cl_{II}
x	0.07523	-0.07556	-0.15495
	(0.00010)	(0.00027)	(0.00029)
y	0.23203	0.35715	0.02423
	(0.00014)	(0.00034)	(0.00034)
z	1/4	3/4	1/4
Beta (1,1)	0.00643	0.00511	0.00543
	(0.00020)	(0.00030)	(0.00032)
Beta (2,2)	0.01520	0.00776	0.00792
	(0.00021)	(0.00032)	(0.00034)
Beta (3,3)	0.04792	0.02621	0.03232
	(0.00067)	(0.00107)	(0.00124)
Beta (1,2)	0.00047	0.00083	-0.00125
	(0.00009)	(0.00022)	(0.00024)

the function and error program written by Busing and Levy for the IBM 704 (26). These are given in Table 8. The results will be discussed in the next section.

Table 8. Interatomic distances and angles and corresponding standard deviations for $SnCl_2$

Atom 1	NS[a]	Atom 2	NS	D(A)	±	σ(D)(A)
Cl_I	1	Sn	1	2.781		0.004
Cl_{II}	1	Sn	1	2.665		0.004
Cl_{II}	1	Cl_I	1	3.481		0.005
Sn	1	Cl_I	2	3.289		0.004
Sn	1	Cl_{II}	2	3.824		0.004
Cl_I	1	Cl_{II}	2	3.978		0.006
Sn	1	Cl_{II}	3	3.064		0.004
Cl_{II}	1	Cl_{II}	3	3.623		0.006
Cl_I	1	Cl_{II}	4	3.565		0.003

$$Cl_I\text{-}Sn\text{-}Cl_I < \; = 79.44^\circ \pm 0.09^\circ$$

$$Cl_I\text{-}Sn\text{-}Cl_{II} < \; = 104.90^\circ \pm 0.16^\circ$$

[a] NS = 1,2,3,4 represents the symmetry transformation x,y,z; $x,1/2-y,1-z$; $\bar{x},\bar{y},\bar{z}$; $x-1,1/2+y,z$, respectively applied to the basic set.

6. Discussion

In this structure, divalent tin attains three-fold primary coordination. A cursory look at this structure reveals that the tin atom has nine-fold coordination. It sits in the center of a trigonal prism of chlorine atoms with three more chlorine atoms out from each face of the prism at a level one-half of the prism height. See Figure 9. A more careful examination reveals an infinite Cl_I-Sn-Cl_I-Sn chain running parallel to the c_o axis; this chain is shown in Figure 10. The Cl_I-Sn-Cl_I angle is 105° 17' and the Sn-Cl_I bond length is 2.78 A. The third chlorine atom has the same z value as the tin atom and is singly bonded. The Sn-Cl_{II} bond length is 2.67 A and the Cl_{II}-Sn-Cl_I angle is 79° 35'. The next shortest Sn-Cl distance is 3.06 A, nearly 0.3 A longer. In accordance with the nomenclature of Werner (27), we can say that the tin atom has three ligands in its primary coordination sphere and six ligands in its outer coordination sphere. The three-sigma values for the distances and angles are about 0.01 A and 20', respectively.

Divalent tin has two valence electrons and three valence orbitals. Presumably the $5s^2$ electrons remain paired. To the first approximation, the short Sn-Cl_{II} bond is a normal covalent bond with each atom donating one electron. The bonds in the chain can be represented by equal contributions of the two resonating structures,

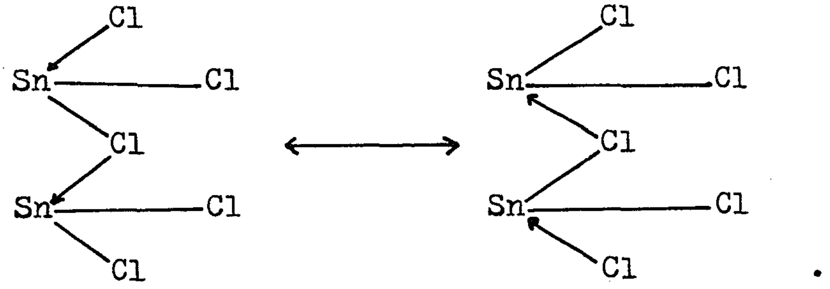
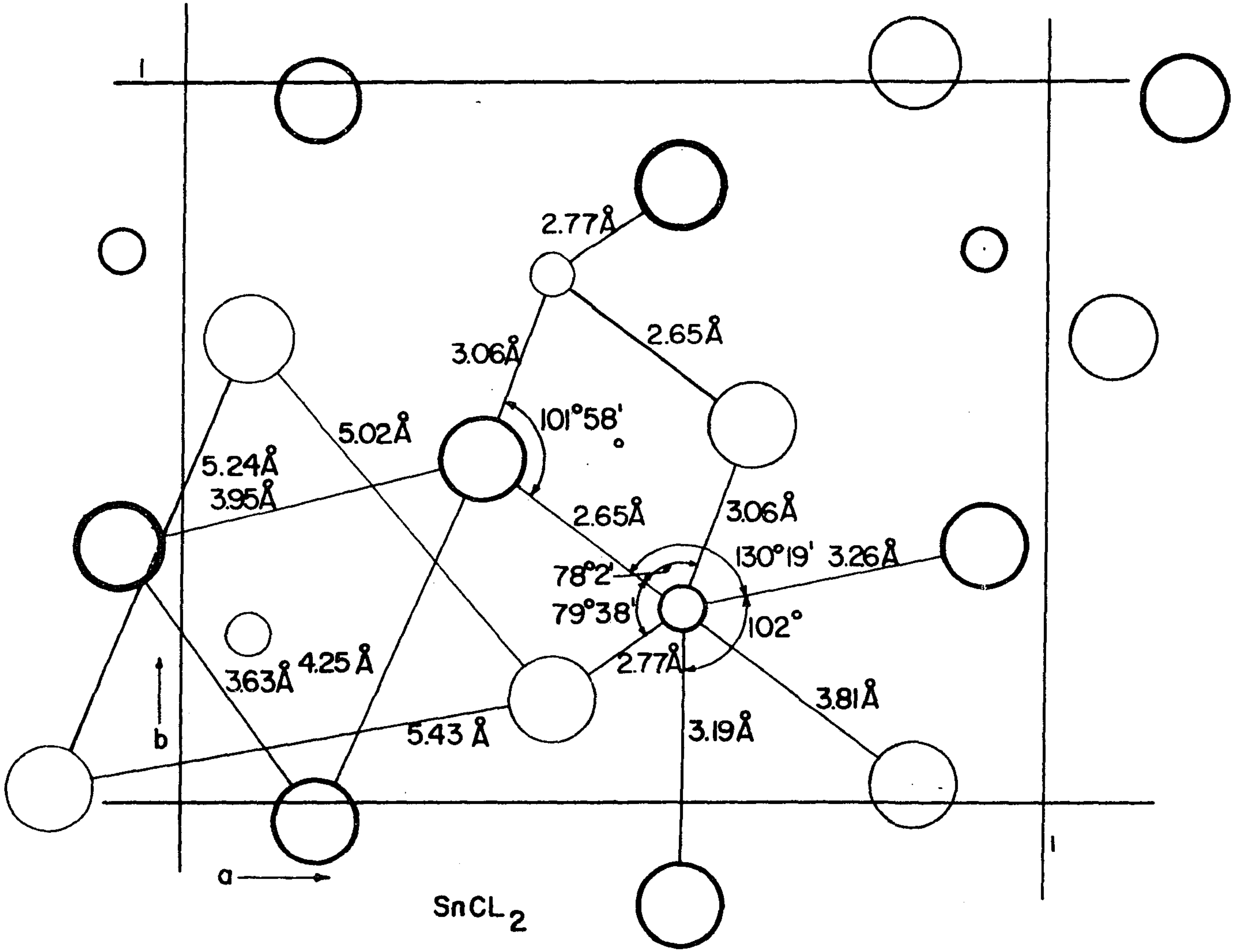


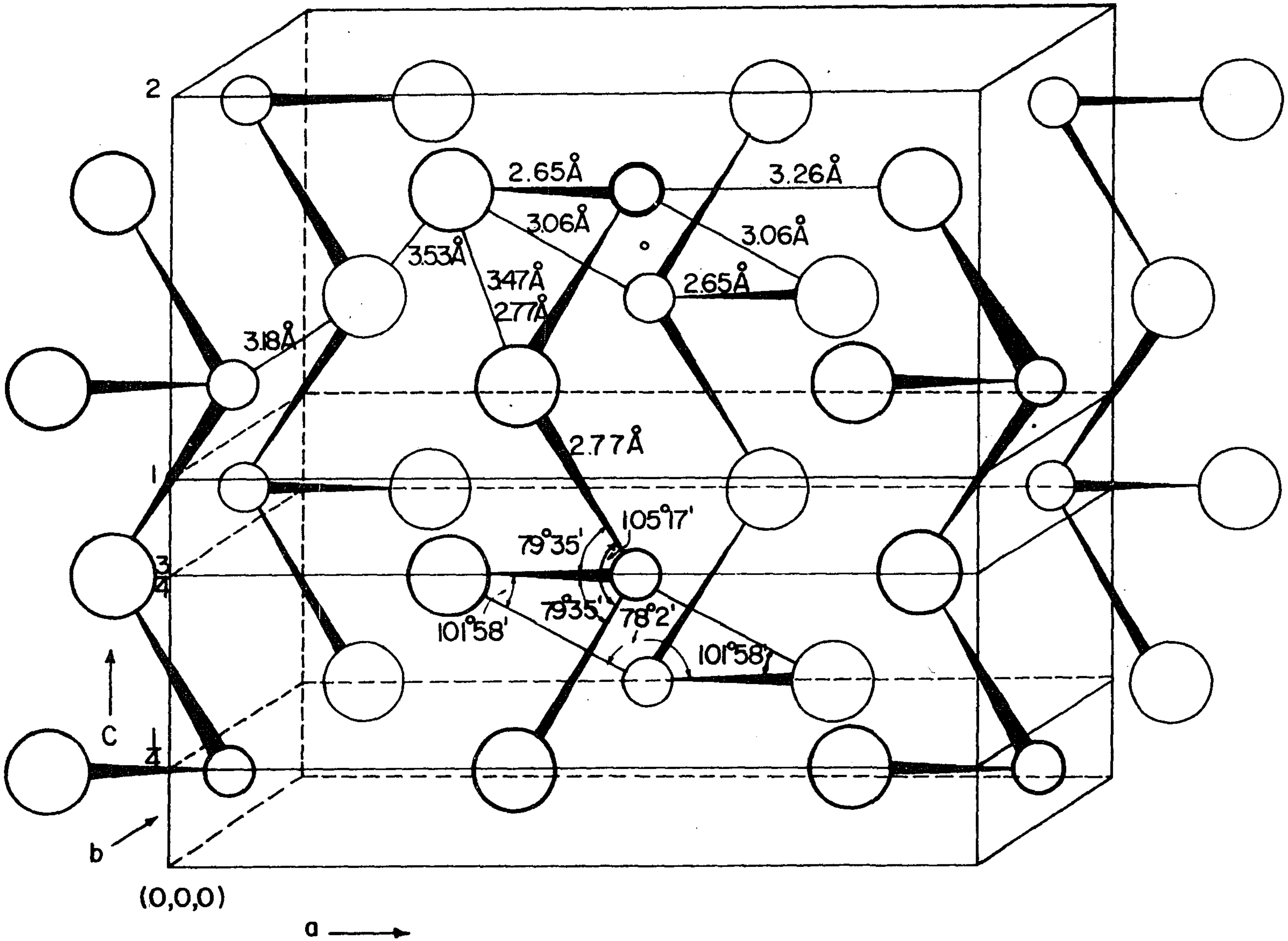
Figure 9. Crystal structure of $SnCl_2$ projected onto the (001) plane (Light-lined and heavy-lined circles represent atoms at 1/4 c and 3/4 c, respectively. For slight corrections to the interatomic distances in this figure see Table 8.)

(001) PROJECTION



$SnCL_2$

Figure 10. Illustration of the crystal structure of $SnCl_2$ (For slight corrections to the interatomic distances in this figure see Table 8.)


2
2.65Å
3.26Å
3.06Å
3.06Å
3.53Å
3.47Å
2.77Å
2.65Å
3.18Å
1
2.77Å
105°17'
79°35'
3/4
79°35'
78°2'
101°58'
101°58'
C
1/4
b
(0,0,0)
a

Sn Cl$_2$

However this scheme puts a negative charge on the tin atom and a positive charge on the chlorine atom which is unreasonable in view of their corresponding electronegativities. Hence there must be considerable ionic character to these bonds to account for redistribution of charge. The Sn-Cl_{II} bond must also have considerable ionic character. At any rate, the influence of the covalent contribution to the bond is seen in the distortion from ideal central positioning of the tin atom among its nine nearest chlorine neighbors.

B. Structure of $[(C_6H_5)_2Sn]_6 \cdot 2C_8H_{10}$

1. Literature review[1]

The physical and chemical properties of diphenyltin are closely related to its method of preparation and its treatment after preparation. This literature review describes briefly various methods of preparation of compounds with composition $(C_6H_5)_2Sn$. Information concerning the structure of various modifications of diphenyltin is also given. When prepared by the reaction of phenylmagnesium bromide with stannous chloride (28),

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[1] For further information concerning organotin chemistry see the comprehensive reviews listed on page 150.

$$2\ C_6H_5MgBr + SnCl_2 \longrightarrow (C_6H_5)_2Sn + 2MgBrCl\ ,$$

it is a bright yellow solid which softens at 126° C and melts to a deep red liquid at 130° C. Although monomeric when freshly prepared, Krause and Becker (28) found that its molecular weight increased by a factor of three in three days and by a factor of five in five months. Chambers and Scherer (29) prepared diphenyltin by the following reactions in liquid ammonia.

$$(C_6H_5)_2SnCl_2 + 4Na \longrightarrow (C_6H_5)_2SnNa_2 + 2NaCl$$

$$(C_6H_5)_2SnNa_2 + 2NH_4Br \longrightarrow 2NH_3 + 2NaBr + (C_6H_5)_2SnH_2$$

$$(C_6H_5)_2SnH_2 \longrightarrow (C_6H_5)_2Sn + H_2$$

The diphenyltin dihydride decomposed to diphenyltin and hydrogen upon the evaporation of ammonia, producing a polymeric product. If ether was added before the ammonia had evaporated, a monomeric product was obtained. The diphenyltin prepared by the latter procedure does not melt below its decomposition temperature of 205°. All of the above diphenyltin products were yellow and they were quite soluble in benzene, chloroform and ethylene chloride.

Kuivila et al. (1) prepared diphenyltin by first preparing diphenyltin dihydride as follows:

$$2(C_6H_5)_2SnCl_2 + LiAlH_4 \xrightarrow{Et_2O} 2(C_6H_5)_2SnH_2 + LiCl + AlCl_3 .$$

If diethylamine was added to the reaction mixture, a product was obtained which had a degree of polymerization of 8.6 ± 1.1. When diethylamine was added to isolated diphenyltin dihydride in ether solution, evolution of hydrogen was quite rapid and a bright yellow, high molecular weight solid precipitate formed. Kuivila *et al.* (1) labeled this form A. When methanol was added to the solution of the hydride, hydrogen evolution was slower and a light yellow solid, form B, resulted. The degree of polymerization was between five and six for form B, which was found to be quite soluble in benzene. When the solid form B was dissolved in dimethylformamide, a new modification, form C, resulted. Form C was much less soluble than form B. Form C may also be prepared by direct addition of dimethylformamide to diphenyltin dihydride in ether solution.

These same workers reported a fourth modification, form D, prepared by the reaction of tin (II) chloride with phenyllithium in a benzene-ether solution,

$$SnCl_2 + 2C_6H_5Li \longrightarrow (C_6H_5)_2Sn + 2LiCl .$$

In addition, form D was slowly converted to form C when in a dimethylformamide solution. The fact that these forms are unique molecular species was shown by the differences in their X-ray powder patterns, solubilities, color and transformations.

Quite recently, Neumann and König (30) reported evidence concerning the structures of some of these modifications. Evidence for their structural proposals was based upon molecular weight studies, degradation experiments and the results of recent structural studies on diethyltin polymers.

By decomposing diethyltin dihydride with amines, Neuman (31) prepared $(Et_2Sn)_n$ where n is in the range 6 - 9 and n = 7 is the main product. From degradation results and analysis of the NMR spectrum, he concluded that the structure of $(Et_2Sn)_7$ is a seven membered ring containing tin-tin bonds.

Concerning diphenyltin, Neumann and König (30) reported that the main product of the reaction of dimethylformamide with diphenyltin dihydride was a pentamer, $[(C_6H_5)_2Sn]_5$ (form C). If pyridine was used in place of dimethylformamide a hexamer, $[(C_6H_5)_2Sn]_6$, was obtained in good yield (form A). Furthermore if methanol was used, a hydride $H\text{-}[(C_6H_5)_2Sn]_6\text{-}H$ (form B) was obtained as the main product. When pyridine or other amines were mixed with it at 20°, the hydride lost hydrogen and was converted to the hexamer. They proposed an open chain structure for the hydride and a five and six membered ring for the pentamer and hexamer, respectively. In addition, diphenyltin prepared by the reaction of stannous chloride with phenylmagnesium bromide or phenyllithium is highly branched and very inhomogeneous. All of the structures presented by Neumann contain tin-tin bonds. The structure of the hexamer, $[(C_6H_5)_2Sn]_6$, has been verified by the X-ray studies described below.

2. Preparation and properties

Several unsuccessful attempts were made to prepare single crystals of diphenyltin. These attempts are discussed only briefly.

Inasmuch as we were mainly interested in the type of linkages in this short chained polymer, it appeared that any of the four forms, A, B, C or D would reveal this information and possibly give insight concerning the others.[1] With the exception of form D, prepared from stannous chloride and phenyllithium, all other forms were prepared as described by Kuivila *et al*. (1). Attempts to recrystallize forms A, B and

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[1] The report by Neumann and König (30) concerning the structure of diphenyltin did not appear until this structural study was well under way.

C from trichloroethylene, acetonitrile and pyridine failed. The transformation of form B to form C by the addition of dimethylformamide or acetonitrile to form B dissolved in benzene always resulted in crystal formation. The crystals resulting from this transformation were large enough for single crystal work but of very poor quality. Serious attempts to control this transformation in order to enhance crystal formation failed.

It was soon learned that diphenyltin in solution is quite sensitive to air oxidation even though in the solid form it is quite stable. In this regard, Kuivila et al. (1) reported that a sample of diphenyltin powder showed no weight change after exposure to the air for several months.

It should be pointed out that several samples of crystalline and amorphous diphenyltin were received from Kuivila.[1] These were used as a guide in the preparation of crystals by the author and eventually led to the procedure by which single crystals suitable for X-ray analysis were obtained. This procedure is described below.

Diphenyltin was prepared using a procedure developed by Kuivila et al. (1). This involved the reaction at 0° C

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[1] These samples were sent to Dr. R. E. Rundle by H. G. Kuivila. The use of these samples is gratefully acknowledged. Kuivila, H. G., Department of Chemistry, University of New Hampshire, Durham, New Hampshire. Private communication. 1961.

of diphenyltin dichloride dissolved in ether with an ether solution of lithium aluminum hydride.[1] The latter solution was added dropwise since this is a rapid exothermic reaction. The reaction is

$$2(C_6H_5)_2SnCl_2 + LiAlH_4 \xrightarrow[0^\circ\ C]{Et_2O} 2(C_6H_5)_2SnH_2 + LiCl + AlCl_3 .$$

The excess $LiAlH_4$ was decomposed by adding ice water to the reaction flask. $(C_6H_5)_2SnH_2$ was separated in the ether layer from the hydrolysis products and other reaction products. The ether solution containing the hydride was then washed with ice water. About four volumes of methanol was added and the mixture allowed to set for at least 24 hours in a flask fitted with a Bunsen valve to allow for escape of hydrogen gas which was evolved slowly. The resulting light yellow solid was separated by filtration from the solution and dried in a vacuum desiccator. Using the designations set forth by Kuivila _et_ _al_. (1) this is diphenyltin form B. From the structural work of Neumann and König (30), the correct formula for this light yellow powder is $H-[(C_6H_5)_2Sn]_6-H$. It is very soluble in benzene but very insoluble in methanol. Thus this form may be purified by

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[1] The author gratefully acknowledges the gift of $(C_6H_5)_2SnCl_2$ from The Metal and Thermit Corporation, Rahway, New Jersey.

repeating the cycle: dissolution in benzene, filtration to separate solid impurities and precipitation by the addition of methanol. Working in a nitrogen atmosphere, this light yellow powder was dissolved in benzene and added to dimethylformamide. Within 24 hours, a crystalline precipitate was present. There were both yellow and colorless crystals present; however all were of poor quality. These crystals were recrystallized from m-xylene in which they are slightly soluble. Approximately 0.01 gram of crystals was dissolved in 150 milliliters of the solvent at about 80° C. The solvent was removed slowly by low pressure evaporation to a cold trap. Clear, colorless, rhombohedral crystals formed in the recrystallization flask. These crystals were analyzed for tin content using the method developed by Gilman and Rosenberg (32) for organotin compounds. This method consists of decomposing the diphenyltin with concentrated sulfuric acid followed by ignition to stannic oxide. The results of the analysis are:

Sample weight	0.0744 g
SnO_2 weight	0.0366 g
Sn weight	0.0288 g
% Sn	38.75
Calc. % Sn for $3(C_6H_5)_2Sn\cdot C_8H_{10}$	38.49
Calc. % Sn for $2(C_6H_5)_2Sn\cdot(C_6H_5)_2SnO\cdot C_8H_{10}$	37.84
Calc. % Sn for $2(C_6H_5)_2Sn\cdot 2(C_6H_5)_2SnO\cdot C_8H_{10}$	38.61

Although the calculated percent tin for $2(C_6H_5)_2Sn \cdot 2(C_6H_5)_2SnO \cdot C_8H_{10}$ was closer to the experimental value than $3(C_6H_5)_2Sn \cdot C_8H_{10}$ was, the former is shown below to be inconsistent with the density data.

The density of the clear, colorless crystals obtained by the recrystallization in m-xylene of form C diphenyltin was determined by the floatation method in a zinc chloride solution. A concentrated zinc chloride solution was diluted with water to the point at which these crystals remain suspended in it. The density of the resulting solution was determined on a Christian Becker Westphal balance. The density found was 1.55 g/cc.

The X-ray diffraction pattern of these crystals was then examined.

3. X-ray data

Preliminary X-ray data on the crystals of diphenyltin form B grown from xylene solution were collected on a Weissenberg camera. Zero and higher level photographs were collected by rotating the crystal about the c_0 and b^* axes. The films show the reciprocal lattice to have C_{2h} symmetry. The systematic extinctions are

$\{00\ell\}$ $\ell = 2n + 1$

$\{hk0\}$ $k = 2n + 1$.

The crystals are monoclinic with probable space group $P2_1/b$.

The a_o, c_o and γ_o lattice parameters were determined using backreflection Weissenberg methods; b_o was determined by correlating the above values with single crystal orienter values. The lattice parameters, calculated and observed density and absorption coefficients for Mo Kα irradiation are:

$a_o = 12.05 \pm 0.01$ A $\quad$ $z_o = 4[3(C_6H_5)_2Sn \cdot C_8H_{10}]$

$b_o = 18.17 \pm 0.01$ A $\quad$ $d_c = 1.55$ g/cc

$c_o = 18.55 \pm 0.01$ A $\quad$ $d_o = 1.55$ g/cc

$\gamma = 102.69 \pm 0.01^o$ $\quad$ $\mu = 20.94$ cm^{-1} .

Previously it was noted that the experimental tin content satisfied, in addition to the unit cell contents above, the composition $2(C_6H_5)_2Sn \cdot 2(C_6H_5)_2SnO \cdot C_8H_{10}$. Moreover three of these units per unit cell gives a calculated density of 1.53 g/cc. However this possibility must be eliminated, since it requires three molecules of xylene per unit cell. Space group $P2_1/b$ has only two-fold and four-fold sets, thus excluding the possibility of having three molecules of m-xylene per unit cell and this chemical composition.

The intensity data used in the solution of this structure was collected on a G. E. XRD-5 crystal orienter using Zr-filtered Mo Kα irradiation. The single crystal orienter angle settings were computed on the Cyclone computer[1]

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[1] Williams, D. E., Ames Laboratory, Iowa State University, Ames, Iowa. SCO-5. Private communication. 1962.

using the SCO-5 program written by Williams. The stationary crystal - stationary counter technique was used with a 40 second peak-count time and 20 second background-count time. Due to the decomposition of the sample in the X-ray beam, four crystals were used in the collection of the data. Standard reflections were recorded at three or four hour intervals during the collection of the data. These standard reflection measurements were used in correcting the data for crystal decomposition. Also an identical set of about fifty reflections was collected with each crystal and used to correlate the data collected with the four crystals. The dimensions of the largest and smallest of the four crystals used were 0.283 x 0.197 x 0.133 mm and 0.248 x 0.146 x 0.136 mm, respectively. The intensities of 6980 unique reflections were collected in a six week period. These data were prepared for use in this structural determination and refinement.

Because the success of this structural determination was strongly dependent upon the accuracy of the data, absorption corrections were computed using the ABCOR program written by Busing[1] for the IBM 704[2]. This program is very

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[1] Busing, W. C., Oak Ridge National Laboratory, Tennessee. ABCOR. Private communication. 1962.

[2] The IBM 704 used for part of this structural study was operated by the Midwestern Universities Research Association and was located at Madison, Wisconsin.

similar to the ABCOR-I program written by Williams and described earlier. A data workup program was written in the Fortran language. This program applied the absorption correction, corrected for streaking, correlated the data collected with the four crystals, calculated and applied the Lp correction, corrected for crystal decomposition, made the significance test and computed F, $\sigma(F)$, $\sigma(F^2)$ and two-theta for future usage. Only $\sigma(F^2)$, $\sigma(F)$ and the significance test need to be discussed since the other data manipulations remain unchanged from previous discussion or need no further explanation. The function for $\sigma(F)$ was derived using the definition of the standard deviation of a function in terms of the standard deviations of its parameters. This function (neglecting cross terms) is

$$\sigma^2(f) = \sum_{i=1}^{n} \left(\frac{\partial f}{\partial x_i}\right)^2 \sigma^2(x_i) .$$

Also used was the fact that

$$\sigma^2(N) = \sigma_{ST}{}^2(N) + \sigma_{SY}{}^2(N)$$

where N is some number representing the value of an experimental measurement. $\sigma_{ST}(N)$ is the statistical error in N and is equal to the square root of N. For lack of any better method, the systematic error, $\sigma_{SY}(N)$, is set equal to an estimated error PCTN times the value N. The standard deviation of F was calculated using the formula

$$\sigma(F) = \frac{1}{2} \cdot F \cdot \left[PCTK + \sigma^2(I)/I^2\right]^{\frac{1}{2}}$$

where

$$\sigma(I) = \left[TC + B + I_s + (PCTI \cdot I)^2 + (PCTB \cdot B)^2 + (PCTI_s \cdot I_s)^2\right]^{\frac{1}{2}} .$$

The constants in this formula were given the values:

$PCTK = 0.0036$
$PCTI_s = 0.1$
$PCTI = 0.0316$
$PCTB = 0.0316$.

The standard deviation of F^2 was calculated by the relation

$$\sigma(F^2) = \frac{1}{A \cdot Lp} \cdot \sigma(I) .$$

The significance test used here is based on the same ideas as discussed earlier for the third set of $SnCl_2$ data and was calculated in the same manner.

Since there is often discussion concerning the merits and demerits of such a significance test, its advantages and the reasons for using it for this problem are discussed below.

The use of such a significance test provides a very useful criterion for eliminating less important reflections when a limited amount of data is desired during certain phases of the structural study. For this structural study, the amount of data was so large that the "unobserved" reflections were never included in the refinement.

In addition to its function in this problem, the results of the significance test may be used in the following situations. The "unobserved" reflection is easily eliminated from Fourier calculations where such less accurately known reflections cannot be weighted. Concerning least squares refinement, in general it is felt that "weak" reflections, called unobserved here, should be included or excluded from the least square process depending upon whether or not they fail or pass the test,

$$|F_o| - |F_c| > 0 ,$$

where F_o and F_c are the observed and calculated structure factors, respectively. Such a test can be made easily during the structure factor calculation.

However in the case where very accurate data is obtained, all F values should be calculated from the observed intensity; as before, an indication of whether it passed or failed the significance test would be useful. In past cases where the significance test was not used, it was felt that the $\sigma(F)$ value calculated for "weak" reflections was too high thus reducing the usefulness or weighting of these reflections in a least squares refinement.[1]

4. Solution of the structure

As pointed out earlier, the probable space group for $3(C_6H_5)_2Sn \cdot C_8H_{10}$ is $P2_1/b$. In the solution of the structure, all asymmetric atoms were assumed to occupy the four-fold set, x,y,z; $\bar{x},\bar{y},\bar{z}$; $x,1/2+y,1/2-z$; $x,1/2-y,1/2+z$.

The solution of an X-ray crystal structure from observed X-ray intensity data is a problem of determining the phase angle of the structure factor F. From the observed intensity, the relative magnitude of F is found but not its phase angle. For a centrosymmetrical structure, the phase angle is either π or 2π and this problem is reduced to determining the sign of all the observed structure factors. Since the intensity of X-radiation scattered by an atom is proportional to the square of the atomic number of the atom, the sign of the structure factor may be controlled by a few relatively heavy atoms.[2] Thus if the positions of the heavy

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[1] The weighting factor for each reflection in least squares refinement is $1/[\sigma(F)]^2$.

[2] The term heavy atom refers to an atom of relatively high atomic number.

atoms in a structure are known, the sign of the structure factor may be computed based upon these positions. A Fourier map computed using as coefficients the observed F with the sign of the computed F will then reveal the positions of most of the light atoms. This procedure is known as the heavy atom method.

The presence of the heavy atoms in the structure made the heavy atom method applicable to the solution of this structure. The determination of the positions of the three asymmetric tin atoms was the first problem faced.

A three-dimensional, sharpened Patterson map was computed on a IBM 704 using the Fourier program, MIFR1, written by Sly and Shoemaker (33). The term sharpened Patterson refers to a Patterson map computed using the usual Fourier summation procedures with modified or sharpened coefficients. This in effect is replacing the Patterson function with some modified function. For this problem, the gradient Patterson function introduced by Jacobson _et_ _al_. (34) was used.

The Patterson function is given by

$$P(u,v,w) \;=\; (1/V) \sum_{h}^{\infty} \sum_{k}^{\infty} \sum_{\ell = -\infty}^{\infty} |F(hk\ell)|^2 \exp 2\pi i(hu + kv + \ell w) \qquad \text{(Eq. 1)}$$

where V is the volume of the unit cell and u, v and w are the fractional points in the unit cell where the function is to be evaluated. The gradient function (34) is given by

$$Q(u,v,w) = (1/V) \int_0^1 \int_0^1 \int_0^1 \overline{V}\rho(x,y,z) \cdot \overline{V}\rho(x+y, y+v, z+w)\,dxdydz \qquad \text{(Eq. 2)}$$

where $\rho(x,y,z)$ is the electron density function,

$$\rho(x,y,z) = (1/V) \sum_{h}^{\infty} \sum_{k}^{\infty} \sum_{\ell=-\infty}^{\infty} F(hk\ell) \exp - 2\pi i(hx + ky + \ell z) \qquad \text{(Eq. 3)}$$

and $\overline{V} = a^*(\partial/\partial x) + b^*(\partial/\partial y) + c^*(\partial/\partial z)$.

Substituting Equation 3 into Equation 2 we have

$$Q(u,v,w) = (16\pi^2/V) \sum_{h=0}^{\infty} \sum_{k}^{\infty} \sum_{l=-\infty}^{\infty} (\sin\theta/\lambda)^2 \, |F(hk\ell)|^2 \cdot$$

$$\cos 2\pi\,(hu + kv + \ell w) \,. \qquad \text{(Eq. 4)}$$

Q(u,v,w) gives a highly peaked maximum in Patterson space but with subsidiary minima on either side. These subsidiary minima may be destroyed by adding some of the original Patterson function to Q(u,v,w). This retains the sharpness of the maximum and improves the resolution of the neighboring peaks. We now have a function of the form

$$P'(u,v,w) = kP(u,v,w) + Q(u,v,w)$$

$$= (16\pi^2/V) \sum_{h=0}^{\infty} \sum_{k}^{\infty} \sum_{\ell=-\infty}^{\infty} \left[k' + (\sin\theta/\lambda)^2\right] |F(hk\ell)|^2 \cdot$$

$$\cos 2\pi(hu + kv + \ell w) \qquad \text{(Eq. 5)}$$

where $k' = k/(16\pi^2)$ and is chosen empirically to be about 1/6 and $|F(hk\ell)|^2$ is modified by the usual sharpening function

$$(1/\hat{f}^2) \exp(-4\pi^2 \sin^2\theta/(\lambda^2 P)) \ . \qquad \text{(Eq. 6)}$$

Here

$$1/\hat{f}^2 = \left(\frac{\Sigma_j Z_j}{\Sigma_j f_j}\right)^2$$

where the sum is over the N atoms of the unit cell and Z_j and f_j[1] are the atomic number and atomic scattering factor

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[1] The scattering factors used in this problem were computed by Thomas and Umeda (35) for tin and Hoerni and Ibers (36) for carbon.

respectively of the jth atom. The gradient Patterson function, Equation 5, gives low weight to low order reflections susceptible to extinction and absorption. It enhances interactions between heavy atoms at the expense of interactions between heavy atoms and light atoms and especially between light atoms and light atoms.

Comparison of Equations 1 and 5 reveals that the gradient Patterson simply required modification of the squared magnitude of the structure factor by

$$\left[k' + (\sin\theta/\lambda)^2\right] (1/\hat{f}^2) \exp -4\pi^2 \sin^2\theta/(\lambda^2 P) \text{ .}$$

This was done using a program written by Stucky and Engebretson for the IBM 704.[1]

Trial positional parameters for the three asymmetric tin atoms were obtained from the sharpened three-dimensional Patterson map. The interpretation of this map was greatly facilitated by making full use of the information contained on the Harker sections.

Harker (37) has pointed out that certain planes or lines of a three-dimensional Patterson map contain much useful information. These planes or lines are called Harker

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[1] Stucky, G. D. and Engebretson, G. R., Department of Chemistry, Iowa State University, Ames, Iowa. PASHAR. Private communication. 1962.

sections. A Harker section contains peaks resulting from interactions of equivalent atoms. The space group $P2_1/b$ has a two-fold screw axis at $x = 0$, $y = 1/4$. Thus for every atom at x,y,z, there is an equivalent atom at $\bar{x}$, $1/2 - y$, $1/2 + z$. The vector between these equivalent atoms is $2x,1/2+2y,1/2$. So peaks resulting from interactions of atoms related by this two-fold screw are found on the Patterson map at $z = 1/2$. The Harker section produced by the b-glide plane is the line at $x = 0$, $y = 1/2$; the corresponding vector is $0,1/2,1/2-2z$. These two Harker sections are shown in Figures 11, 12 and 13.

Due to the large difference in atomic numbers of tin and carbon, the tin-tin interactions were easy to detect on this Patterson map. As can be seen from Figures 11 and 12, there are four peaks of large magnitude on the Harker section at $z = 1/2$. Three of these represent interactions of equivalent tin atoms related by the two-fold screw axes. The line $0,1/2,z$ has two large peaks. These must represent three tin-tin interactions. Figure 13 shows that the larger of these two peaks is spread out indicating the overlapping of two tin-tin interactions. Using the other interactions between equivalent and nonequivalent tin atoms, the following trial parameters were found for the three tin atoms:

Figure 11. Harker section for $[(C_6H_5)_2Sn]_6 \cdot 2C_8H_{10}$ at w = 1/2, part one

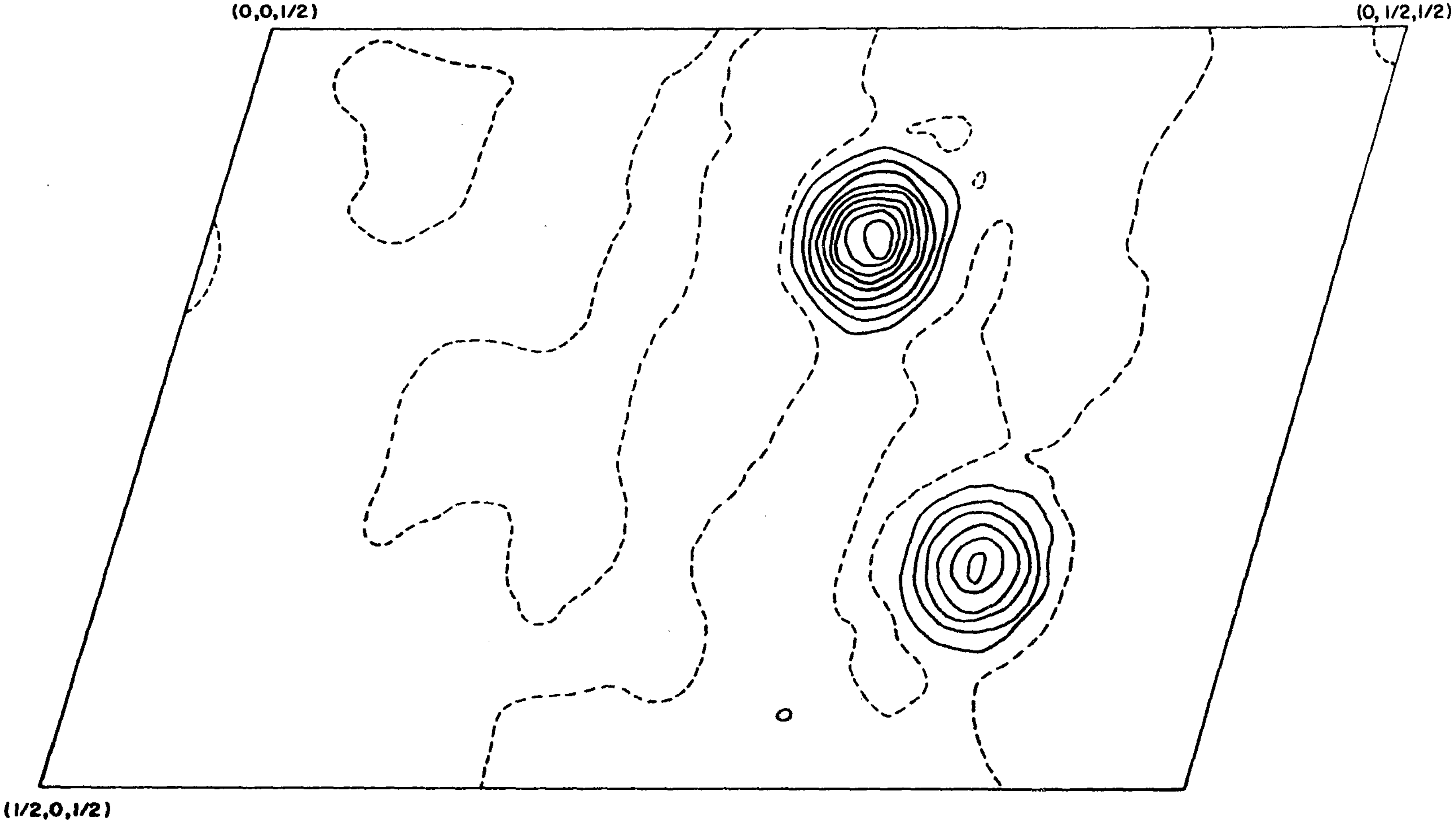

(0,0,1/2)
(0, 1/2,1/2)
(1/2,0,1/2)

Figure 12. Harker section for $[(C_6H_5)_2Sn]_6 \cdot 2C_8H_{10}$ at w = 1/2, part two

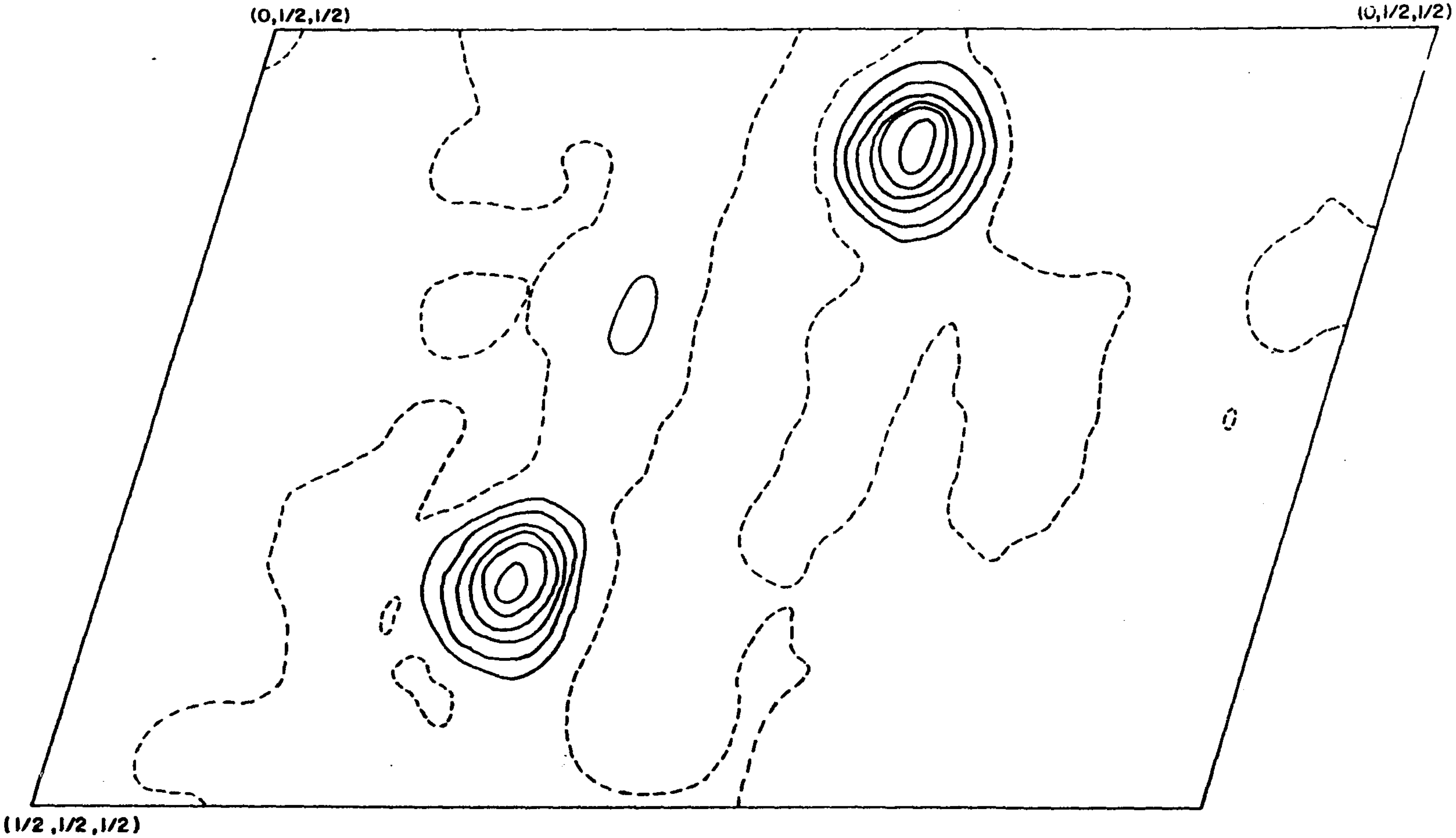
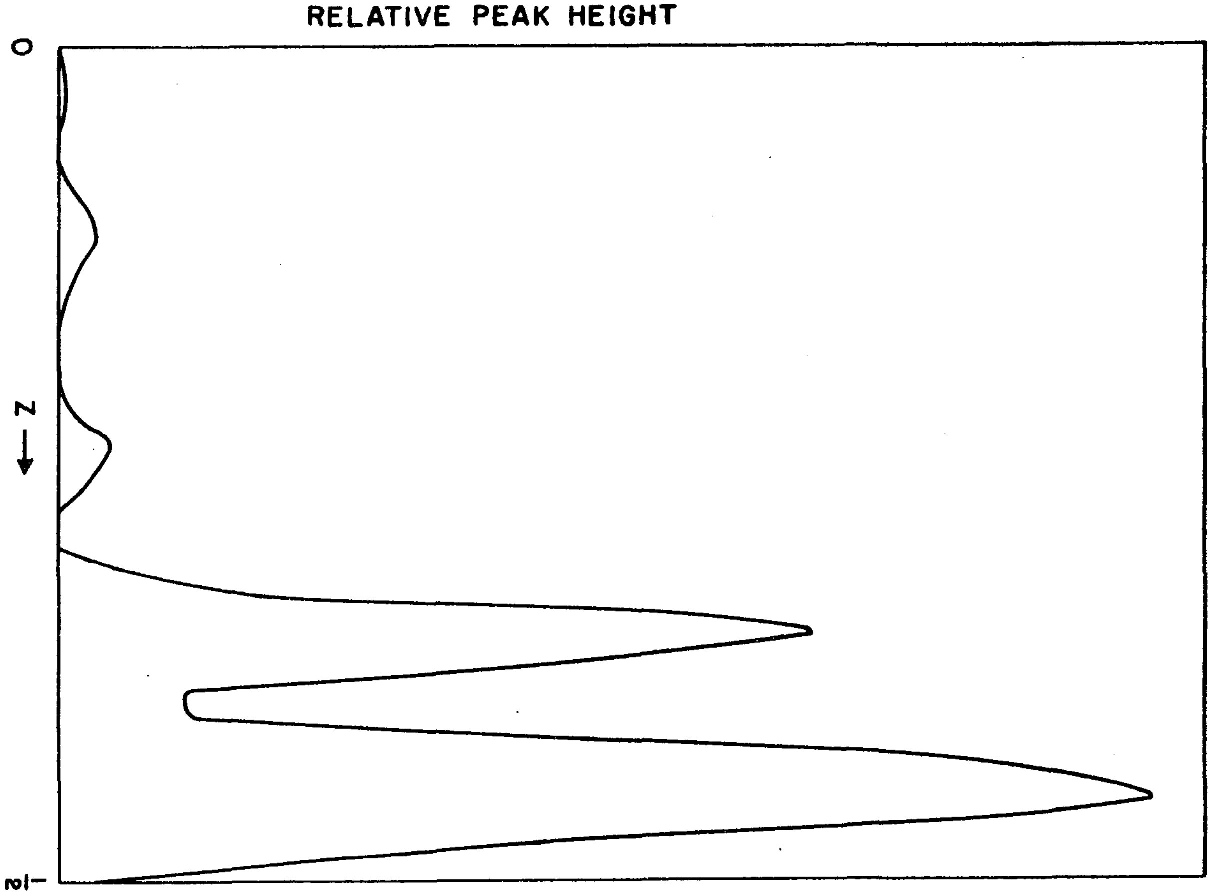

(0,1/2,1/2)
(0,1/2,1/2)
(1/2,1/2,1/2)

Figure 13. Harker section for $[(C_6H_5)_2Sn]_6 \cdot 2C_8H_{10}$ at $u = 0$, $v = 1/2$


RELATIVE PEAK HEIGHT
0
Z
$\frac{1}{2}$

	x	y	z
Sn-1	0.178	-0.058	-0.035
Sn-2	0.180	0.090	-0.072
Sn-3	0.040	0.147	0.020

All interactions between equivalent and nonequivalent tin atoms were accounted for by Patterson peaks with the expected intensity.

These trial parameters were refined using the least squares program written by Busing and Levy (25). For this refinement, 607 reflections with two-theta (Mo Kα) less than 25° were included. After four cycles of refinement, the R factor was 27 %. A three-dimensional Fourier map was computed based upon the tin atom parameters from the above refinement. However due to an error in the selection of octant control cards, a Fourier map of 1/8 of the unit cell was obtained rather than a Fourier map of 1/4 of the unit cell which was required. Attempts to obtain the additional 1/8 of a unit cell always gave maps which had a very high population of spurious peaks. Attempts to find the difficulty were hampered by the constraints imposed by computation via the data link.

From the properly calculated part of the Fourier map, trial parameters were obtained for three of the seven phenyl groups.

At about this time in the structural determination, the computational facilities were changed from the MURA IBM 704 at Madison, Wisconsin to an IBM 7074 on this campus. Since there was no Fourier program available for this computer, a three-dimensional Fourier program was written in the Fortran language for the monoclinic space group $P2_1/a$.[1] This program evaluates the function

$$\rho(x,y,z) \quad =$$

$$(4/V)\sum_{h}^{\infty}\sum_{k}^{\infty}\sum_{\ell=0}^{\infty} \overset{h+k=2n}{\{}[F(hk\ell)+F(\bar{h}k\ell)] \cos 2\pi hx \cos 2\pi \ell z \cos 2\pi ky$$

$$+ [-F(hk\ell)+F(\bar{h}k\ell)] \sin 2\pi hx \sin 2\pi \ell z \cos 2\pi ky\}$$

$$+ (4/V)\sum_{h}^{\infty}\sum_{k}^{\infty}\sum_{l=0}^{\infty} \overset{h+k=2n+1}{\{}-[F(hk\ell)+F(\bar{h}k\ell)] \cos 2\pi hx \sin 2\pi \ell z \sin 2\pi ky$$

$$+ [-F(hk\ell)+F(\bar{h}k\ell)] \sin 2\pi hx \cos 2\pi \ell z \sin 2\pi ky\} \; .$$

The summations are carried out essentially as one-dimensional sums of the Beevers-Lipson type. A three-dimensional Fourier map computed with 4500 coefficients evaluated at 54,000 points required about 12 minutes of computer time. About

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[1] The change in space group was made for compatibility with a structure factor program available for this computer.

two-thirds of this time involved read-write operations.

From a Fourier based upon the three tin atom positions and the carbon atom positions of three phenyl groups, all remaining carbon positional parameters were obtained with the exception of the two methyl carbon atoms. Probable positions for these two atoms were found from a subsequent Fourier map and a difference Fourier map.

5. Refinement

This structure was refined by the least squares method using the HYBL-SFLS2 program written by Hybl.[1] This program was written in the Fortran language and uses the block diagonal approximation. This approximation considers all interactions of the parameters of an individual atom but neglects interactions between atoms. Although convergence is slower than with full matrix least squares, the minimum reached is the same. The fact that this structure has 47 atoms in the asymmetric unit essentially rules out the use of full matrix least squares for at least two reasons. First, there is no least squares program available which will allow all the parameters of this problem to be varied simultaneously. Second, the time required for an individual cycle with all parameters varying

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[1] Hybl, A., Department of Chemistry, Iowa State University, Ames, Iowa. HYBL-SFLS2. Private communication. 1962.

would be more costly in terms of computer time than the extra cycles required for block diagonal least squares. Also, it is known that as the size of the matrix becomes very large, round-off errors become serious.

For the initial refinement of this structure, 1504 of the strongest reflections with two-theta less than 40° were used. Fourteen cycles were computed with this selection of data. The initial conditions and any changes made during this refinement are discussed below.

The carbon positional parameters for cycle one were obtained from the third three-dimensional Fourier and the tin parameters from previous least squares cycles. All temperature factors were computed isotropic. The tin temperature factors, B_{Sn}, were set equal to 2.2 (A^2) and carbon temperature factors, B_C, were given the value 3.6 (A^2). All carbon atoms were included except the methyl carbon atoms. Shifts were computed for all positional parameters, B_{Sn}'s and six B_C's. One-half of the computed shift was applied for the positional parameters and for the three B_{Sn}'s. The reliability index R was

$$R = \frac{\Sigma\left| |F_o| - |F_c| \right|}{\Sigma |F_o|} \times 100\ \% = 12.5\ \% .$$

For cycle two and all additional cycles, all new parameters from the previous cycle were applied except for the following exceptions. During the first few cycles of refinement, carbon atom positions were held constant or corrected if the atom was diverging from the expected C-C distances. In latter cycles, any of the carbon anisotropic temperature factors which were not positive definite were given their previous positive definite value. All B_C's were set at 4.0 (A^2) in response to the indicated shift in B for six carbon atoms. The R factor for cycle two was 10.1 %.

In cycle three, the weighting scheme was switched from unit weights to $1/\sigma(F^2)$ weights. In addition, anisotropic temperature factors were computed for the tin atoms. R = 9.2 %.

For cycle five, the carbon temperature factors were changed as follows: for C1 carbons, $B_C = 4.0$ (A^2) and for all other carbon atoms, $B_C = 4.5$ (A^2).[1] For cycle five, R = 7.4 %.

Before cycle seven was computed, structure factors

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[1] The nomenclature used to refer to particular atoms is as follows: Sn-1, Sn-2 and Sn-3 refer to the three asymmetric tin atoms. Carbon atoms are referred to by Cx or Cx-y where y refers to the phenyl group number, y = 1 through 7. X refers to the carbon atom position in the ring. C1 is the carbon atom attached to a tin atom and the numbering is continued consecutively around the ring. C7-7 and C8-7 are the methyl carbon atoms. Cx refers collectively to the xth carbon atom of all phenyl groups.

were computed using all observed data. Examination of these structure factors revealed that a small percentage were in very poor agreement and that many of these were in the two-theta range 30 - 40°. In this range, there had been some difficulty in making background corrections. In making the background corrections for reflections in this range, the value used for the background was the lowest value found for other reflections in the same region of reciprocal space. It is now apparent that this value of the background was too low for many reflections since $F_c << F_o$. The weight for 136 such reflections was set equal to zero.

In order to obtain additional information concerning the methyl carbon atoms, a Fourier map and difference Fourier map were computed using the results of the structure factor calculation. Very diffuse peaks appeared on both maps in four positions compatible with the known configuration of m-xylene. Although two of these peaks indicated a larger amount of electron density than the other two, the possibility of disordering of the xylene molecule exists.

In cycle eight, two methyl carbons in the two favored positions were included in the least squares refinement. The population parameter for these atoms was set at 0.6 and a shift was calculated. The carbon temperature factors were set as follows: $B_{C1} = 4.0$ (A^2), $B_{Cx} = 5.2$ (A^2), x = 2 through 6 and $B_{C7} = B_{C8} = 6.0$ (A^2). All temperature factors were allowed to vary. The R for this cycle was 5.8 %.

For cycle nine, anisotropic temperature factors were computed for all atoms and R was 5.3 %.

Cycle ten was the first cycle for which all atoms had anisotropic temperature factors. R = 4.8 %. Also the calculated shifts for 51 of the 141 positional parameters were less than ± 0.002 A or were of opposite sign from the previous cycle. During the next cycle, a fixed atom tape was written. In addition to other quantities, this tape contained for each structure factor the sum of the contribution to the structure factor of all atoms except the methyl carbon atoms.

Cycles 12, 13 and 14 were computed using the fixed atom tape as input. This allowed the refinement of the xylene molecule holding all other atoms constant without calculating their contribution to the structure factor. The most interesting information obtained from these three cycles was concerned with the population parameter refinement. For cycle 12, it was set at 1.0. After cycle 14, these parameters were 1.02 and 1.08 for C7-7 and C8-7, respectively. This indicates that the methyl carbon atoms are at these sites with full occupancy.

Two cycles of least squares were computed using all observed data. Following this, structure factors were computed for the entire set of data and were submitted to an

R-factor analysis using the program written by Hybl.[1] The major results of this analysis are:

for 6980 reflections, R = 15.9 %,
for 4264 observed reflections, R = 7.2 %,
for 2580 unobserved reflections, R = 50.5 % and
for 136 rejected reflections, R = 68.4 %.

Figure 14 is a listing of all calculated and observed reflections.

An analysis of the thermal motion was made using a program written by Williams[2] and modified by the author. Results of this analysis are given in Table 9.[3]

The equation of the plane best describing each of the six membered carbon rings was computed using the least squares plane program written by Williams.[4] These equations and the deviation from the plane of each of the atoms defining it are given in Table 10.

A program was written in Fortran to compute inter-

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[1] Hybl, A., Department of Chemistry, Iowa State University, Ames, Iowa. R-factor analysis. Private communication. 1963.

[2] Williams, D. E., Department of Chemistry, Iowa State University, Ames, Iowa. RMS analysis. Private communication. 1963.

[3] In this and subsequent tables, Sn-n refers to the tin atom related to Sn+n by a center of symmetry.

[4] Williams, D. E., Department of Chemistry, Iowa State University, Ames, Iowa. Least squares plane. Private communication. 1963.

Figure 14. Comparison of observed and calculated structure factors for $[(C_6H_5)_2Sn]_6 \cdot 2C_8H_{10}$

(The reflections are grouped into sections of constant h and k. The first column is the running index ℓ; the second column is $|F_o|$; the third column is F_c. An asterisk following the running index denotes an unobserved reflection. An R following the running index denotes a reflection not included in the least squares refinement.)

0	0	L
2	441	414
4	117	112
6	39	-26
8	43	-49
10R	21	1
12	22	-4
14	23	11
16	41	-42
18	92	-92
20	89	-87
22	49	-37
0	1	L
1	457	437
2	27	-19
3	175	174
4	152	141
5	112	106
6	29	-38
7	82	-86
8	164	-170
9	55	-55
10	161	-170
11	50	-36
12	117	-119
13R	40	-9
14	55	-47
15•	24	-3
16•	24	-1
17	56	-56
18R	27	7
19	79	-76
20•	26	-6
21	50	-48
22•	27	-12
0	2	L
0	149	135
1	71	67
2	70	62
3	26	29
4	88	-78
5	169	-163
6	91	-93
7	183	-187
8	131	-136
9	268	-286
10	47	-36
11	200	-208
12	45	-43
13	115	-123
14•	24	-12
15•	24	-22
16•	25	-29
17•	25	25
18•	25	-28
19•	26	12
20•	26	-11
21•	26	-9
0	3	L
1	147	-144
2•	18	-6
3R	25	1
4•	19	-15
5•	20	-16
6	80	-77
7	36	-58
8	216	-225
9	104	-104
10	231	-239
11•	23	-39
12	129	-134
13	54	-44
14	45	-33
15•	24	-10
16	67	57
17•	25	-10
18	59	62
19	38	26
20	28	27
21•	26	26
0	4	L
0	304	-303
1	219	218
2	88	-90
3	47	51
4•	18	-11
5•	20	16
6•	19	-18
7	92	-95
8R	24	-4
9	143	-144
10	38	-36
11	86	-73
12	58	-50
13R	58	4
14•	24	-39

15	70	73
16	32	-18
17	96	96
18•	25	23
19	66	67
20	47	44
21•	26	26
0	5	L
1	186	-192
2	93	92
3	89	-93
4	26	-36
5	54	54
6•	19	20
7	123	124
8	33	-24
9	43	43
10•	21	-17
11	49	-27
12•	22	23
13	45	-43
14	54	50
15•	24	-23
16	103	100
17R	34	7
18	70	74
19	33	33
20	56	54
21•	26	19
0	6	L
0	46	-35
1•	19	3
2	94	-95
3	49	-36
4	55	55
5	50	-53
6	122	119
7	47	-49
8	169	175
9•	21	-21
10	48	58
11	40	41
12•	23	-13
13•	23	26
14•	23	3
15	47	55
16•	24	-12
17•	25	36
18•	25	20
19	45	34
20•	26	16
21	42	38
0	7	L
1	42	-45
2	131	-138
3	39	39
4	161	-169
5	84	93
6	101	-101
7	131	128
8	55	-49
9	115	122
10R	27	-10
11	27	31
12•	23	-11
13	40	39
14	28	-28
15•	24	6
16	42	-39
17•	25	15
18•	25	-19
19•	25	10
20•	26	14
0	8	L
0	61	-59
1	30	-34
2	45	-45
3	160	-165
4	31	-35
5	125	-135
6	27	25
7	96	-93
8	78	64
9	38	-39
10	59	60
11	31	-23
12	53	63
13	57	-61
14	39	40
15	71	-74
16	26	24
17	74	-78
18	30	22
19	42	-38
20•	26	9
0	9	L

1	153	-164
2	121	-125
3	163	-170
4	114	-115
5	111	-107
6	62	-62
7	38	-34
8R	33	0
9R	40	-11
10	27	18
11	41	49
12R	31	-1
13	64	65
14	52	-58
15	58	61
16	72	-73
17	53	41
18	59	-57
19•	26	16
0	10	L
0	127	-126
1	53	-27
2	136	-139
3	59	-48
4	192	-196
5R	24	-1
6	137	-145
7	34	38
8	100	-106
9	74	76
10R	28	-1
11	61	67
12	73	74
13•	25	-6
14	64	69
15•	26	-23
16	76	75
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17• 24  -10
18• 25   -6
19  54  -47
20• 25   -4

  3  6  L

0  301  305
1   90   90
2  216  233
3   86   93
4   58   64
5  192  196
6R  26  -15
7   65   65
8   42  -41
9•  22   11
10R 23   15
11• 23   14
12  29   21
13• 24    2
14• 24   22
15• 24   11
16R 28   -9
17• 25  -15
18  48  -50
19• 26  -20
20  55  -51

  3  7  L

1  196  204
2   97   95
3  103  107
4   95   96
5   54   56
6   31   33
7   29  -18
8   40  -38
9•  22  -16
10  78  -81
11R 37   11
12  59  -59
13• 24  -18
14  56  -36
15• 24  -13
16• 25  -14
17  44  -35
18• 25  -13
19  39  -39

  3  8  L

0  146  131
1   40   40
2   42   44
3   85   90
4   40   39
5   34   34
6•  22   -1
7   28  -37
8   31  -26
9   95  -99
10  29  -38
11  95  -95

12  45  -39
13  65  -64
14  59  -60
15• 25   -9
16  31  -29
17• 25    0
18• 25  -15
19• 26  -12

  3  9  L

1   49  -48
2   56   51
3R  63  -17
4   44   44
5•  22   21
6R  24   13
7•  23   -7
8   37  -36
9   49  -45
10  60  -60
11  65  -71
12• 25  -33
13  82  -81
14• 25  -14
15  61  -59
16  28   24
17• 26  -16
18• 26   10

  3 10  L

0   92  -88
1•  22   14
2   42  -46
3•  23   22
4•  22   -5
5   29   28
6   37   41
7•  23   19
8•  23   -6
9•  24   -7
10  48  -48
11• 25   15
12  89  -88
13  36   18
14  79  -76
15  44   39
16  36  -35
17  45   36
18• 26    3

  3 11  L

1•  23  -21
2•  23  -22
3   42   39
4•  23  -28
5   65   65
6•  23    6
7   77   81
8•  24    9
9•  24   20
10  25   29
11  61  -60
12  54   44
13  72  -65
14  44   40
15  56  -50
16  31   36
17• 26  -19

  3 12  L

0   54   52
1•  23  -13
2   80   83
3  104 -104
4   90   94
5   58  -58
6   90   94
7•  24  -21
8   61   63
9•  24    1
10• 25  -12
11  37   35
12  34  -31
13• 26   33
14  37  -36
15• 26    8
16  36  -27

  3 13  L

1   63   67
2   71  -74
3   66   86
4   94 -100
5R  95   49
6   73  -76
7   28   37
8   36  -42
9•  25   10
10• 25   -7
11• 25    5
12• 25    0

13• 25   -6
14• 26  -17
15• 26  -19

  3 14  L

0•  24   -5
1   44  -40
2   28   28
3   95  -93
4•  25    3
5  110 -114
6•  25   -9
7   75  -75
8•  25    1
9   43  -46
10• 25   -0
11• 25  -23
12• 26   25
13  28  -36
14• 26   13

  3 15  L

1•  25  -17
2   64  -67
3   41  -32
4   84  -87
5   55  -59
6   73  -76
7   49  -50
8   53  -52
9•  25  -17
10• 26  -27
11• 26   24
12• 26  -26
13  41   43

  3 16  L

0   34  -29
1•  25  -15
2   34  -39
3•  25  -24
4   85  -80
5•  26  -27
6•  26  -85
7   28  -25
8   48  -42
9•  26   -9
10• 26    2
11• 26  -14
12• 27   36

  3 17  L

1•  25  -23
2•  25   12
3   64  -62
4•  26   25
5   68  -69
6•  26   16
7   50  -45
8•  26   11
9   34  -12
10• 26    0

  3 18  L

0•  25    3
1•  26    6
2•  26  -13
3   39   45
4•  26  -27
5   48   53
6•  26  -20
7   30   23
8•  26   -7

  3 19  L

1•  26    5
2•  26   32
3•  26    8
4   38   53
5•  26    7

  4  0  L

0   53   -0
1   73  -70
2   36   25
3  232 -237
4   58   53
5  264 -270
6   85   80
7  179 -185
8   63   58
9   87  -91
10  44   33
11  66  -65
12  26   -1
13  65  -60
14  30   -5
15  62  -60
16  25   -5

17  53  -52
18• 26   11
19• 26  -15
20• 26   -1
21• 27   21

  4  1  L

1  169 -130
2  153 -153
3   65  -65
4  239 -241
5   55  -57
6  221 -229
7   44  -39
8  101 -104
9R  25   -2
10  28  -28
11  30   41
12  62  -68
13  52   58
14  87  -92
15  56   52
16  64  -58
17  37   27
18  36  -39
19  27   20
20• 26    2
21• 26    6

  4  2  L

0  121  -79
1   56  -52
2  199 -208
3  151 -160
4R  58 -204
5  171 -184
6  248 -264
7   79  -82
8   91  -95
9•  22    2
10• 22    8
11• 23   -7
12  74   83
13• 24  -32
14 104  110
15  44  -40
16  62   66
17• 26  -26
18  32   35
19• 26   -0
20• 27    9

  4  3  L

1   89  -95
2•  20   -8
3  203 -188
4•  20   13
5  224 -232
6R  43   19
7  176 -184
8   31   21
9   68  -70
10  41   34
11  72   69
12• 24   14
13 129  135
14• 25   19
15  90   91
16• 25   25
17  45   53
18• 26   16
19• 26    0
20• 26   15

  4  4  L

0R  26  -16
1   33   26
2   21  -17
3  111  107
4  122 -111
5  128  131
6   99 -102
7   95   99
8R  52  -41
9   66   64
10  53   56
11  66   67
12 102  106
13  56   48
14 111  111
15  73   74
16  64   64
17  58   50
18• 25    5
19  34   31
20• 26  -19

  4  5  L

1   22   12
2   90   95
3   41   42

4  180  187
5•  20  -16
6  138  139
7•  21    8
8   57   63
9R  41   17
10• 22    0
11  92   96
12• 23   18
13  84   80
14  78   60
15  63   56
16  63   60
17• 25   19
18  35   28
19• 25  -12
20• 26    5

  4  6  L

0   95  -86
1  108  109
2   22   37
3  127  130
4   71   72
5  135  137
6   51   51
7•  22   22
8   45   48
9   37  -38
10  49   47
11  55  -62
12  42   48
13  39  -31
14• 24   19
15• 24   10
16• 25    5
17• 25    5
18• 25    1
19• 26  -12

  4  7  L

1•  23  -66
2  117  127
3•  21    9
4   78   81
5   69   70
6   47   51
7   77   77
8•  23  -21
9   44   47
10  79  -85
11• 24   14
12  56  -60
13• 24  -23
14  39  -32
15• 25  -19
16  26  -23
17• 25    0
18• 26  -22
19• 26   14

  4  8  L

0R123  -89
1   33   40
2  117 -121
3   95  100
4   23  -11
5   51   54
6   44   52
7   31   32
8   52   49
9•  23   -6
10• 24  -10
11• 24  -17
12  46  -47
13• 25  -21
14  38  -50
15• 25  -26
16• 25   -8
17  29  -30
18• 27   27

  4  9  L

1  131 -134
2   31   26
3   81 -102
4   48   50
5•  23   -5
6   46   49
7•  24   30
8   71   76
9•  24   10
10  55   58
11  60  -69
12• 25   40
13  69  -65
14• 25   -3
15  35  -32
16• 25  -20
17• 26   14
18• 27  -22

  4 10  L

0   54  -42
1R  28   -7
2•  25  -37
3•  23   -5
4   25  -30
5   28   28
6•  23   20
7   72   76
8•  24    4
9  123  122
10  34  -29
11  94   91
12  66  -70
13  52   50
14  65  -56
15  30    9
16• 26  -19
17• 26   -9

  4 11  L

1   59   57
2   40  -43
3   79   85
4R  26    0
5   53   53
6   44   39
7   39   40
8  103  101
9•  25   -8
10 113  113
11  49  -42
12  86   76
13  57  -55
14• 26   34
15  53  -38
16• 26   -1

  4 12  L

0  110  114
1R  32  -15
2  141  142
3   58  -62
5•  24  -25
6   47   48
7   31   38
8•  25   11
9   71   74
10• 25  -11
11  72   64
12• 26  -18
13• 26   23
14  30  -34
15• 26   -1

  4 13  L

1  127  130
2   53  -50
3   94   93
4   61  -66
5   65   49
6   37  -38
7•  24   -4
8•  25   -7
9•  25  -16
10• 25    8
11• 25   -7
12• 26  -14
13• 26  -10
14• 26  -19

  4 14  L

0   75   76
1   40  -26
2   68   65
3   50  -50
4   28   19
5   73  -69
6•  25  -26
7   64  -67
8   42  -40
9   54  -52
10• 26  -15
11  35  -40
12• 26   -3
13  37  -39

  4 15  L

1   42   39
2•  25  -10
3•  25   -3
4   40  -36
5   43  -34
6   55  -60
7   63  -60
8   53  -55
9•  26  -30
10  52  -53
11• 26   -8
12  40  -41

  4 16  L

0•  25    3
1•  25   10
2•  25  -15
3•  26   14
4   43  -37
5•  26   -4
6   28  -45
7   28  -33
8   32  -33
9   31  -40
10• 26  -14

  4 17  L

1•  26  -26
2•  26   21
3•  26  -24
4•  26   15
5•  26  -22
6•  26   -0
7•  26  -11
8•  27  -21
9•  26   -7

  4 18  L

0•  26  -17
1•  26   11
2•  26   -8
3•  26   15
4R  30    0
5•  26    5
6•  27   19

  5  0  L

0  239  245
1   98  -95
2  159  165
3  162 -164
4  112   79
5  206 -212
6  102  -29
7  140 -141
8   75  -56
9   88  -76
10  46  -38
11  50  -43
12  27  -17
13  68  -64
14• 24   19
15  67  -75
16  44  -35
17  52  -48
18  49  -46
19• 26  -15
20  37  -38

  5  1  L

1  124  152
2  101  -95
3•  21   -7
4  140 -146
5  129 -130
6  151 -161
7  108 -111
8  150 -155
9   83  -83
10  86  -92
11• 23   -8
12  69  -73
13  25   29
14  56  -58
15  31   33
16  38  -33
17• 26  -15
18• 26   -5
19  31  -23
20• 26   16

  5  2  L

0   46   44
1   27   23
2   51  -45
3•  21  -21
4R  62 -123
5   55  -52
6  169 -172
7  122 -127
8  129 -134
9  120 -123
10  36  -38
11  70  -74
12• 24   28
13  28  -29
14  49   52
15  27   20
16  35   28
17  30   37
18• 26   -3
19  30   38

20• 27   -5

  5  3  L

1   32  -39
2   97   97
3  126 -114
4   58   59
5  114 -114
6•  21   -1
7   85  -92
8  102 -109
9   55  -60
10  67  -68
11• 23   21
12  30  -37
13  37   38
14  48   45
15  28   30
16  83   80
17  29   29
18  70   70
19• 26   -5
20  36   44

  5  4  L

0   41  -36
1   54   57
2   36  -42
3   40   44
4   67  -12
5   85   89
6•  21   14
7   27  -18
8•  22   13
9   87  -82
10  51   55
11  29  -29
12  41   44
13• 24    7
14  27   23
15  98   99
16R 31   16
17  97   93
18• 25    8
19  60   58

  5  5  L

1•  20   -4
2   53   52
3•  21   16
4   49   54
5   80   70
6R  24    5
7   79   80
8   61  -60
9   74   74
10  78  -80
11  61   62
12R 30  -18
13• 24   11
14  40   33
15• 26   13
16  71   71
17R 38    8
18  52   43
19• 26    6

  5  6  L

0   94  -96
1•  21    5
2   59  -67
3•  21    8
4   31   40
5R  28   -8
6  101  102
7   47  -39
8  112  117
9   78  -83
10  41   45
11  66  -72
12• 24   17
13  47  -40
14• 24    3
15• 25  -12
16• 25    7
17• 25   -2
18• 26   21
19• 26   -8

  5  7  L

1  163 -167
2   28  -21
3   73  -79
4   63  -67
5•  22    6
6   53  -42
7   87   91
8   29  -35
9   59   56
10  51  -56
11• 24   21

12  42  -34
13• 24   16
14  57  -56
15• 25   11
16  45  -47
17  32   26
18  29  -33

  5  8  L

0  221 -226
1•  23  -13
2  145 -148
3   52  -42
4   91  -87
5   48  -41
6•  23    1
7•  23    6
8•  24   18
9•  24   11
10• 24   -0
11• 24   20
12  28   19
13  29  -14
14• 25   16
15  42  -41
16  41   32
17  49  -43
18  48   42

  5  9  L

1  149 -152
2•  23   -7
3   97  -94
4•  23   -8
5   82  -67
6   42   45
7•  24  -13
8   67   73
9•  25  -17
10  79   85
11• 24  -13
12  53   56
13  31   23
14• 25   -1
15  29   21
16• 26  -26
17  33   33

  5 10  L

0   65  -59
1•  23   12
2   32  -33
3•  23  -12
4   48  -54
5   78   81
6   45  -44
7  101  104
8•  25  -23
9  121  114
10R 30  -12
11 102  102
12• 25   11
13  56   56
14• 26   23
15• 26    4
16• 26   17

  5 11  L

1   67   67
2•  24   12
3   38   39
4   54   54
5   30  -18
6   78   78
7•  25  -17
8  110  106
9•  25   -2
10 103  103
11• 26   12
12  75   66
13• 26   22
14  29   31
15• 26   13

  5 12  L

0  153  158
1•  25   -6
2  115  115
3•  25   31
4   43   38
5   34   34
6•  24   -7
7   59   53
8•  25    3
9   58   57
10• 25    1
11  41   33
12• 26   26
13• 26   14
14• 26   13
```

Figure 14. (Continued)

```
   5 13  L

 1 125   125
 2  31    19
 3  59    67
 4  42    50
 5  46    13
 6•  25     5
 7•  25   -23
 8•  25     1
 9•  25   -15
10•  25   -14
11•  26     5
12•  26   -22
13•  26     6

   5 14  L

 0  62    49
1R  31     7
 2  41    47
 3  38    42
 4•  25     3
 5•  25    14
 6•  25   -12
 7•  25   -21
 8•  26   -26
 9  27   -29
10•  26   -20
11  55   -50
12•  26   -13

   5 15  L

 1•  25     7
 2•  26    23
 3•  25    -6
 4  34    35
 6•  25     3
 7•  26   -14
 8•  26   -27
 9  27   -27
10  42   -44
11•  27   -25

   5 16  L

0R  38   -16
 1  30    14
 2•  25   -15
 3•  26    32
 4•  26     2
 5  26    32
 6•  26     6
 7•  26    -3
 8•  26    -4
 9•  26   -21

   5 17  L

 1•  26   -16
 2•  26    14
 3•  26     4
 4•  26    13
 5  30    29
 6•  27     8

   5 18  L

 0  32     3
 1•  26    -4
 2•  26    14

   6  0  L

 0 202   206
 1  36     2
 2 174   173
 3  36    53
 4  97    77
 5  50    23
 6  93   -75
 7  69   -43
 8  92   -73
 9  77   -75
10  58   -45
11  81   -76
12R  26    -6
13  51   -54
14•  25    -1
15•  25   -30
16  40   -41
17•  26   -25
18  42   -39
19•  27   -25

   6  1  L

 1  78    46
 2  63    63
 3  55    59
 4  46    45
 5  45   -53
6R  27     7
 7  93   -99
 8  80   -76

 9  79   -84
10  81   -78
11  51   -51
12  79   -82
13•  24   -22
14  28   -26
15  33   -21
16•  25    10
17•  26   -19
18•  26     7
19•  27   -17

   6  2  L

 0  28    15
 1  56    63
 2  30   -29
 3  68    73
 4•  22   -41
 5  52    46
 6  46   -51
 7  34   -22
 8  49   -56
 9  78   -81
10  39   -44
11  56   -56
12  63   -65
13•  24    -5
14  39   -40
15  34    38
16  33   -19
17  38    42
18•  26    -8
19•  27    32

   6  3  L

 1•  21   -19
 2  36    35
 3  35   -14
 4  58    60
 5  44    52
6R  47    -0
 7  32    38
 8  30   -38
 9  27   -21
10  33   -42
11  45   -52
12•  25     3
13  89   -94
14  54    51
15  54   -52
16  66    64
17•  26   -12
18  54    56
19•  26     2

   6  4  L

 0  32    21
 1•  21    10
 2  39    38
 3•  21     0
 4 100    98
 5  25    -7
 6 131   133
 7  39   -32
 8  75    79
 9•  23   -14
10•  23     9
11•  23    10
12  76   -74
13  29    33
14  86   -79
15  58    55
16  41   -42
17  51    50
18•  26    -9
19  31    39

   6  5  L

 1  28    22
 2  27   -29
 3  77    83
 4  84   -83
 5 146   149
 6  90   -88
 7 123   119
 8  35   -37
 9  50    53
10•  23   -10
11•  24     3
12•  23     5
13  60   -56
14•  24     7
15  37   -39
16•  25    14
17•  25   -17
18•  26    21

   6  6  L

 0•  22   -21
 1  41   -42
 2•  23   -15

 3 117  -122
 4  62    65
 5 155  -153
 6  79    69
 7 114  -115
 8  65    65
 9  59   -62
10  28    30
11  43   -39
12•  25    -1
13  45   -47
14•  25   -19
15  48   -40
16•  25    -9
17  31   -21
18•  26     6

   6  7  L

 1  68   -72
 2 113  -111
 3  41   -38
 4 180  -189
5R  25    -9
 6 133  -135
7R  31    -8
 8  78   -81
 9  27    20
10  52   -52
11•  24    15
12  41   -41
13  25    17
14  57   -60
15•  25    10
16  51   -48
17•  26    17

   6  8  L

 0  76   -75
 1  47   -47
 2  85   -91
 3 116  -119
 4  94  -103
 5  91   -91
 6  65   -65
 7  48   -44
 8  46   -40
 9•  24   -11
10•  24     4
11•  24     9
12•  25    26
13•  25   -20
14•  27    42
15  34   -30
16  52    42
17  40   -31

   6  9  L

 1  96   -98
 2  70   -71
 3  77   -84
 4  30   -39
 5 122  -124
 6  27    13
 7  80   -79
 8  44    41
 9  28   -21
10  49    47
11  35    32
12  32    31
13  64    60
14•  26     3
15  65    63
16•  26    -4

   6 10  L

0R  77   -44
 1•  24    18
 2•  25   -29
 3•  24    -5
 4  66   -67
 5  39    23
 6  88   -91
 7  44    46
 8  39   -41
 9  57    53
10•  25    17
11  50    48
12  65    66
13  29    31
14  68    66
15•  27    20

   6 11  L

 1  42    32
 2  35    37
 3•  24    17
 4  53    50
 5  30   -25
 6  50    51
 7  33   -30
 8  29    35

 9•  25    -0
10  32    31
11  51    44
12  27    22
13  63    61
14•  27    20

   6 12  L

 0  47    46
 1  39    33
 2  42    49
 3  78    74
 4•  25    25
 5  58    53
 6•  25    -3
 7  35    31
 8•  25     2
 9•  25    -2
10•  27    26
11•  26    -3
12  44    47
13•  27    -4

   6 13  L

 1•  25    23
 2  50    57
 3•  25    -2
 4  64    68
5R  62     5
 6•  25    24
 7•  25     5
 8•  25    -9
 9•  26    13
10  29   -32
11•  27    18
12•  27   -27

   6 14  L

 0  51   -31
 1•  25    17
 2  33   -28
 3  47    47
 4•  26   -24
 5  46    43
 6•  25     1
 7•  26    -2
 8•  26     5
 9•  26   -21
10•  26    -2
11  35   -31

   6 15  L

 1  52   -45
 2•  26    21
 3•  31   -26
 4  37    36
 5•  26   -11
 6  31    28
 7•  26     6
 8•  26    12
 9•  26    -5

   6 16  L

 0  37   -36
1R  36     6
 2•  26   -20
 3•  26    17
 4•  26     2
 5  30    30
 6•  26    14

   6 17  L

 1•  26     0
 2•  26    -4

   7  0  L

0R  83   -25
 1  74    67
 2  73   -26
 3 120   118
 4  54   -12
 5 116   118
6R  25    -0
 7  36    10
 8•  23     6
 9  58   -52
10R  39     3
11  76   -79
12  25   -20
13  55   -53
14•  25    -5
15•  25   -11
16•  25     1
17•  26   -12
18•  26     3
19•  27   -26

   7  1  L

 1  98   -99
 2  93    94
 3  72   -76
 4 118   120
 5  34   -42
 6  86    93
 7•  23    19
 8•  23   -10
 9  32   -17
10  35   -33
11  52   -52
12  39   -44
13  50   -51
14R  30     3
15  36   -38
16•  26     3
17•  26     9
18•  26   -10

   7  2  L

 0 102  -103
 1  68    68
 2  69   -70
 3  85    85
4R  30     0
 5 100   100
 6  33    34
 7  82    81
 8•  24    25
 9  28    31
10  48   -55
11  32    30
12  84   -81
13R  32    23
14  66   -73
15•  26    30
16•  26   -26
17•  26    15
18•  26    13

   7  3  L

 1•  22   -17
 2  36    32
 3  41    36
 4  55    54
 5  84    85
 6  82    80
 7  77    80
 8 106   108
 9  26   -15
10  73    72
11  56   -61
12  71    71
13  83   -89
14  43    42
15  67   -68
16  38    28
17•  26   -20
18•  26     3

   7  4  L

 0  56    56
 1  24   -24
 2  98    94
 3  32   -24
 4 121   119
 5•  23    -6
 6 109   109
 7  65    60
 8  54    56
 9  86    80
10•  24   -29
11  79    78
12  58   -58
13  31    44
14  71   -66
15•  25    14
16  55   -58
17•  25     6
18•  26   -21

   7  5  L

 1 133   130
 2  66   -64
 3 129   135
 4  88   -90
 5 107   100
 6  48   -49
 7  86    84
 8•  23     1
 9•  24    12
10•  24    19
11  31   -34
12  35    32
13  56   -54
14•  25    -2
15  48   -45
16  32   -26
17  29   -34

   7  6  L

 0 104   102
 1  43   -43
 2 100   105
 3 116  -116
 4  67    67
 5 120  -124
 6  61    45
 7  77   -76
 8•  24    14
 9•  24   -25
10  26   -30
11•  24   -26
12•  25   -29
13  32   -36
14  33   -29
15  60   -50
16•  26   -24
17  43   -40

   7  7  L

 1  50    54
 2  64   -69
 3•  24     6
 4 114  -119
 5  38   -43
 6 115  -111
 7  26   -24
 8  74   -74
 9  25   -26
10  31   -38
11•  25   -21
12  34   -39
13•  25     2
14  44   -48
15•  26    -4
16  39   -44

   7  8  L

 0•  24     8
 1  24   -13
 2•  24   -18
 3  59   -63
 4  56   -64
 5  63   -62
 6  88   -87
 7  80   -69
 8  76   -68
 9  41   -37
10•  25   -14
11  28   -26
12  27    15
13•  26   -23
14•  26    23
15•  26    -2

   7  9  L

 1  32   -16
 2•  24    -6
 3  43   -45
 4•  24     3
 5  78   -79
 6  37   -34
 7  71   -72
 8•  25   -23
 9  33   -28
10•  25   -19
11•  25    14
12•  25   -11
13  41    34
14•  26    22
15  32    30

   7 10  L

 0  24     8
 1•  24    -3
 2•  24    -8
 3•  25    26
 4•  25   -23
 5  29    16
 6•  25   -38
 7•  25    -7
 8•  25   -14
 9•  25   -24
10•  25    12
11  29   -22
12  31    33
13•  26    16
14•  27    29

   7 11  L

 1•  24     4
 2•  25    21
 3•  25     8
 4  47    40
 5  29    20
6R  33    -1
7R  32    19
 8•  25   -17
 9•  26    25
10  29   -37
11  26    29

12•  26   -17
13•  26    26

   7 12  L

 0  31   -27
 1•  25     9
 2•  25    -6
 3  29    27
 4•  25     9
 5•  25     4
 6  50    47
 7•  26   -19
 8  43    44
 9  41   -39
10•  26    31
11  43   -39
12•  27    22

   7 13  L

 1  66   -63
 2•  25    10
 3  33   -32
 4•  25     3
 5•  25    20
 6•  26   -15
 7  46    44
 8  39   -35
 9  28    26
10  43   -36

   7 14  L

 0  97   -96
 1•  26     7
 2  82   -78
 3•  26   -10
 4•  26   -28
 5•  26    -3
 6•  26    10
 7•  26   -11
 8•  26    18

   7 15  L

 1  75   -71
 2•  27    -0
 3  56   -55
 4•  26    -5
 5•  26   -17
 6•  26    13

   7 16  L

 0  42   -28
 1•  27    -3
 2  29   -30

   8  0  L

 0 164  -165
 1  59    39
 2 141  -145
 3  74    17
 4  46   -35
5R  35    -5
 6  48    28
 7  39   -36
8R  34    24
 9  38   -37
10  30    42
11  37   -32
12•  26    -4
13  44   -40
14•  26     4
15  37   -28
16  33    21
17•  26   -23
18•  27    29

   8  1  L

 1 185  -187
 2  46    16
 3 137  -139
 4  38    22
 5  38   -37
 6  45    43
 7•  24    -2
 8  51    55
9R  25    -1
10  41    46
11R  27    -1
12•  25    15
13  29   -23
14•  26    -9
15•  26     9
16•  26   -23
17  37    34

   8  2  L

 0 133  -130
 1  28    -0
 2 125  -129

 3•  24     9
4R  38   -64
 5  70    73
 6•  24   -10
 7 109   118
 8•  25     6
 9  91    98
10•  25     4
11  83    91
12•  28   -11
13  52    54
14•  26   -10
15•  26     9
16•  27    15
17•  27   -20

   8  3  L

 1•  23    -8
 2  30   -21
3R  24    -2
 4  29    28
 5•  24    17
 6  98   103
 7•  25    26
 8 126   128
9R  30    -6
10 115   120
11R  28     1
12  89    95
13•  26   -15
14  37    34
15•  26    -5
16•  26    -1
17•  26     6

   8  4  L

 0 105   102
 1•  23   -13
 2 110   115
3R  27   -14
 4  63    71
 5  45    47
 6•  24    17
 7  94    90
 8•  24    15
 9 105   100
10•  25    -6
11 102    96
12•  25    -6
13  42    39
14•  25   -11
15•  25    -1
16•  26   -19

   8  5  L

 1 153   152
 2•  23     8
 3 124   128
 4  25   -26
 5  49    32
 6  37    36
7R  25    -9
 8  51    50
 9•  24   -15
10  56    51
11•  25    -8
12•  25    21
13•  25    10
14•  25   -22
15•  25   -12
16  27   -28

   8  6  L

 0 132   136
 1•  24     9
 2 103   109
 3•  24   -18
 4  28    34
 5•  24    -8
 6  49   -32
7R  31    -9
 8  39   -39
 9•  24   -15
10•  27   -23
11•  25   -11
12•  25     5
13  36   -36
14•  25     7
15  39   -38

   8  7  L

 1  64    71
 2•  24     2
 3•  24    38
 4•  24    -3
 5  51   -48
 6  30   -22
 7  72   -64
 8  42   -40
 9  49   -50
10  56   -53

11•  25   -17
12  36   -36
13•  25     1
14  28   -27
15•  26    -5

   8  8  L

 0  38    37
 1•  24    15
 2•  24    23
 3  40    34
 4•  25   -25
 5  33    24
 6  36   -47
 7  39   -21
 8  55   -56
 9  45   -44
10  41   -43
11  37   -33
12•  26   -17
13•  26    -6
14•  26   -10

   8  9  L

 1•  24     6
 2  34    32
 3•  24     8
 4  63    63
 5  35   -19
 6•  25    15
 7•  25   -16
 8•  26   -20
 9•  26   -23
10•  26   -24
11•  26   -18
12•  26    -6
13•  26   -21

   8 10  L

 0•  24     3
 1•  25    20
 2•  25    21
 3  42    42
 4  29    30
 5•  25    14
 6•  26    31
 7•  25   -18
 8•  26    23
 9•  26   -30
10•  26    -2
11•  26   -17
12•  26   -15

   8 11  L

 1•  25    19
 2•  25     5
 3  32    33
 4•  25    -0
 5  65    64
 6  41   -29
 7  62    57
 8  38   -39
 9  33    28
10  34   -31
11•  26    -3

   8 12  L

 0•  25    -7
 1•  25   -14
 2•  25     4
 3  29   -33
 4  43    31
 5  39   -39
 6  63    55
 7  47   -47
 8  40    43
 9  40   -38
10•  27    18

   8 13  L

 1  33   -39
 2  42   -44
 3•  26   -18
 4  62   -54
5R  56     9
 6  54   -51
 7•  27    25
 8  44   -37

   8 14  L

 0  71   -64
 1•  27   -23
 2  49   -50
 3  51   -51
 4•  27   -29
 5  43   -45

   9  0  L

 0 143  -149
 1  51   -31
 2 131  -122
 3 101   -95
 4  72   -57
 5 100  -101
6R  29     3
 7  53   -56
 8  41    36
 9  31   -30
10•  25    13
11  28   -26
12•  25    -1
13•  26   -23
14•  26    11
15  32   -36
16  34    28

   9  1  L

 1 166  -158
 2  59   -46
 3 140  -140
 4  74   -71
 5  82   -87
 6  52   -41
 7  34   -20
 8•  25    23
 9•  25   -12
10  36    25
11•  25    18
12•  26    26
13•  26    23
14•  26    -2
15  38    34
16•  27   -22

   9  2  L

 0 103   -98
 1•  25   -19
 2 101  -102
 3  36   -37
 4•  25   -83
 5  39   -27
 6  48   -47
 7  49    44
 8  31   -20
 9  78    77
10•  26     7
11  83    83
12  32    37
13•  26    34
14  31    46
15•  27    -3
16  28    35

   9  3  L

 1•  24     8
 2•  24    -9
 3•  24   -10
 4•  24    -6
 5  33   -22
 6•  25    42
 7  32   -32
 8  84    89
 9•  25    -0
10  96   101
11  41    49
12  61    65
13  40    47
14  27    20
15  46    40

   9  4  L

 0  81    77
1R  27     5
 2  62    55
 3•  23    17
 4•  24     9
 5  50    45
 6  31   -25
 7  55    54
 8  33   -10
 9  76    79
10  43    34
11  43    46
12  60    53
13•  25    15
14  50    48
15•  26     0

   9  5  L

 1  68    67
 2•  24    27
 3  40    36
 4  47    41
5R  37   -19
 6  37    39
 7  35   -21
 8•  24    16
 9•  25    11
10•  25     0

11  37    27
12•  25    -8
13  41    41
14•  26    -7
15•  26    25

   9  6  L

 0  42    49
 1  41    26
 2  24    22
 3  43    45
 4•  24    -9
 5  49    44
 6  26   -33
 7•  24    11
 8•  25   -16
 9  33   -21
10•  25    -0
11  40   -42
12•  26    15
13•  26   -33
14•  26    16

   9  7  L

 1•  24    -5
 2  46    48
 3•  24   -16
 4  68    71
 5  29   -34
 6  30    34
 7  31   -19
 8•  25    -1
 9•  26   -27
10  31   -34
11•  26   -16
12  35   -23
13•  26    -6

   9  8  L

 0  42   -34
 1  30    25
 2•  25   -24
 3  68    69
 4  37   -12
 5  68    62
 6•  25    -5
 7  46    37
 8  28   -16
 9•  25     5
10  27   -25
11•  26    -9
12  37   -29

   9  9  L

 1•  25   -19
 2  32    37
 3•  25     6
 4  54    53
 5  43    28
 6  38    42
 7•  26    20
 8  46    37
 9•  26    -8
10  33    29
11  43   -32

   9 10  L

 0•  25    16
 1•  25     7
 2  30    37
 3•  26     8
 4  57    51
5R  34    11
 6  56    58
 7•  26    23
 8  27    26
 9  32    33
10•  27   -13

   9 11  L

 1  46    50
 2•  26   -15
 3  57    54
 4  30   -27
 5  64    61
 6•  27   -17
 7  48    47
 8•  27     2
 9•  27    11

   9 12  L

 0  39    45
 1•  26   -11
 2  42    36
 3  45   -47
 4  31    38
 5  40   -40
 6  39    37
 7  33    -3
```

Figure 14. (Continued)

```
  9 13  L

 1•  27    17
 2   44   -35
 3•  27     5
 4   50   -55

 10  0  L

 0R  55   -27
 1   41   -30
 2   59   -28
 3  118  -112
 4   50   -30
 5  110  -114
 6   29   -27
 7   61   -63
 8R  25   -14
 9   46   -41
10•  26   -25
11   27   -18
12•  26    -9
13   29   -23
14•  26     4
15•  28   -31

 10  1  L

 1R  77   -39
 2   60   -59
 3   65   -54
 4   78   -73
 5   76   -75
 6   40   -35
 7   56   -58
 8•  25   -17
 9   46   -43
10•  25    -8
11•  25     1
12•  26     6
13   37    24
14•  26    -2
15•  27    20

 10  2  L

 0   44   -31
 1•  25   -13
 2   34   -22
 3   30   -21
 4•  25   -45
 5•  26    -9
 6   69   -68
 7•  26     1
 8   37   -38
 9•  26    -6
10•  26    -0
11•  26    16
12   33    45
13•  27    22
14   36    42

 10  3  L

 1•  25    23
 2•  25    10
 3R  25     7
 4•  25     7
 5   34   -28
 6•  25    10
 7   31   -22
 8•  25   -11
 9•  25     5
10•  26    -1
11   33    44
12•  27    11
13   62    60
14•  27    19

 10  4  L

 0R  34    27
 1   28    24
 2•  24    27
 3   40    37
 4•  24     7
 5   28    21
 6•  24    -8
 7   26   -24
 8•  25    13
 9   27   -28
10   32    35
11•  26   -22
12   35    47
13•  26    -7

 10  5  L

 1•  24   -15
 2   34    43
 3•  24    -9
 4   33    35
 5•  26   -10
 6•  24   -11
 7•  25    19

 8   36   -35
 9   32    26
10   59   -51
11   37    29
12   39   -38
13   30    27

 10  6  L

 0   66   -68
 1R  29    10
 2   61   -65
 3   31    39
 4   32   -36
 5•  25    23
 6•  25     9
 7•  25   -17
 8•  25    22
 9   51   -43
10•  26    17
11   41   -45
12•  26    11

 10  7  L

 1  103  -100
 2•  25     9
 3   66   -62
 4   48    43
 5•  25   -19
 6•  26    19
 7•  26    20
 8•  26     0
 9•  26     9
10•  26   -11
11•  26     2

 10  8  L

 0  106   -93
 1•  26     0
 2   70   -66
 3•  26    21
 4•  26   -28
 5   46    45
 6•  26     9
 7   43    45
 8•  26    11
 9   44    40
10•  27    -6

 10  9  L

 1   29   -36
 2•  25     4
 3R  27   -12
 4   29    27
 5•  26    11
 6   45    50
 7•  27    13
 8   73    63
 9•  27     4

 10 10  L

 0   26    22
 1•  26     1
 2   36    24
 3•  26    -3
 4   29    32
 5   27    14
 6•  27    25
 7   49    42

 10 11  L

 1   68    60
 2•  26   -13
 3   48    47
 4•  26   -11
 5   39    34

 11  0  L

 0   62    70
 1•  25   -13
 2   49    43
 3   59   -39
 4   35   -19
 5   37   -26
 6   73   -69
 7•  26    -7
 8   67   -55
 9   37   -22
10•  26   -30
11•  26   -10
12•  26    -7
13•  26   -10

 11  1  L

 1   58    39
 2   26    11
 3•  26    -8
 4•  26    -2
 5   51   -51

 6•  26     4
 7   66   -69
 8•  26   -15
 9   41   -45
10•  26   -13
11•  26   -10
12•  26    -2
13•  27     3

 11  2  L

 0   44    38
 1•  25    13
 2   37    27
 3   28    22
 4•  26    -0
 5•  26    13
 6•  26   -30
 7•  26   -16
 8   27   -26
 9   28   -21
10   27   -10
11•  26   -12
12•  27    -1

 11  3  L

 1   40    43
 2•  25    29
 3   52    50
 4•  26    18
 5•  26    25
 6•  26   -12
 7•  26    12
 8   41   -36
 9•  26    11
10   38   -37
11•  27     3
12   36   -17

 11  4  L

 0   26    12
 1•  24    13
 2   33    24
 3   26    18
 4   44    40
 5•  25    -8
 6   44    41
 7   50   -46
 8   52    42
 9   64   -56
10•  26    16
11   60   -49

 11  5  L

 1   46   -43
 2R  28    -4
 3•  25    -4
 4•  25   -21
 5•  26    19
 6   47   -46
 7   50    47
 8   58   -59
 9   30    24
10   63   -61

 11  6  L

 0   90   -80
 1•  26   -10
 2   61   -65
 3   38   -28
 4   28   -25
 5   44   -44
 6•  26    15
 7   44   -41
 8•  26    17
 9   50   -42

 11  7  L

 1   92   -87
 2•  26   -22
 3   66   -61
 4   32   -27
 5•  26   -19
 6•  26   -21
 7•  26    -0
 8•  26    -7

 11  8  L

 0   76   -70
 1•  27    -6
 2   58   -60
 3•  27   -19
 4   39   -34
 5•  26    -4
 6•  26   -15
 7•  26    14

 11  9  L

 1•  26   -22

 2•  26    -8
 3   28   -20
 4•  26    -1
 5•  26   -14

 12  0  L

 0   40    18
 1   27    18
 2•  26     4
 3   57    48
 4   42   -17
 5   44    39
 6   49   -42
 7   30    22
 8   33   -36
 9•  27    -5
10•  27   -26
11•  27    -8

 12  1  L

 1   31    10
 2   42    45
 3•  27     2
 4   61    65
 5R  29   -12
 6   51    46
 7•  27   -23
 8•  27    11
 9   27   -28
10•  27    -1

 12  2  L

 0R  27    14
 1•  26    16
 2   32    27
 3   49    47
 4•  27    28
 5   41    46
 6•  27    23
 7•  27    15
 8•  27     6
 9•  27     8
10•  27   -14

 12  3  L

 1   30    43
 2•  26    10
 3   54    55
 4•  27    12
 5   57    52
 6•  27    -0
 7   56    55
 8•  27    -3
 9•  27    15

 12  4  L

 0   28    37
 1•  25    -9
 2   37    39
 3•  26   -21
 4   48    51
 5   29   -27
 6   63    59
 7   32   -26
 8   44    42

 12  5  L

 1•  25     1
 2   42   -40
 3•  26    12
 4   49   -51
 5•  26    25
 6   40   -47
 7   28    32

 12  6  L

 0   35   -25
 1   32   -27
 2•  26   -23
 3   52   -55
 4•  26   -11
 5   55   -53
 6•  27     1

 12  7  L

 1   37   -30
 2   29   -35
 3•  27   -24

 13  0  L

 0   71   -69
 1•  27    21
 2   56   -49
 3   60    46
 4   44   -21
 5   44    50
 6   32   -12

 7   39    31
 8•  27    -1

 13  1  L

 1   47   -52
 2   30    39
 3•  27   -20
 4   53    50
 5•  27     7
 6   47    47
 7•  27    13

 13  2  L

 0•  26   -14
 1•  27     8
 2•  27    -0
 3   32    28
 4•  28    23
 5   32    34
 6   35    34

 13  3  L

 1   33    33
 2•  27    -0
 3   49    44
 4•  27     7
 5   42    38

 13  4  L

 0   42    46
 1•  26    -9
 2   41    42
 3•  27   -17

 14  0  L

 0   85   -80

  1 -1  L

 1  302   286
 2  176   168
 3  149   140
 4  316   301
 5   54   -45
 6  247   238
 7   19     6
 8  150   144
 9   19   -20
10   89    91
11   43    34
12   97    97
13   59    56
14   74    70
15R  25     7
16   28    21
17   28   -28
18•  23    14
19   50   -48
20•  24   -20
21   45   -42

  1 -2  L

 0   60    56
 1   70    62
 2   17    -2
 3  216   205
 4   20   -10
 5  132   143
 6  124  -127
 7  198   197
 8  125  -124
 9  271   275
10•  19   -16
11  138   147
12   72    71
13  112   116
14   98   101
15   51    51
16   31    27
17•  23    17
18•  23   -12
19•  23    -1
20   44   -36
21•  24    -7

  1 -3  L

 1   82   -80
 2  168  -151
 3  156  -145
 4•  16    10
 5  294  -288
 6  144   144
 7  208  -209
 8  207   213
 9   27   -18
10  173   177
11   72    79
12  125   127
13  112   114

14•  22    23
15   88    88
16•  23    -9
17   36    36
18   26   -35
19•  23    10
20•  23   -10
21•  24     4

  1 -4  L

 0  426  -410
 1  159  -152
 2  285  -274
 3  101   -99
 4  230  -214
 5   78   -79
 6  137  -140
 7   48    53
 8   72   -65
 9  145   150
10•  20   -10
11  109   114
12   80    82
13•  22   -15
14   97    97
15   48   -51
16   60    66
17   77   -77
18   43    48
19   52   -56
20•  24    20
21•  24    -6

  1 -5  L

 1  280  -277
 2  151  -150
 3  103  -107
 4  188  -182
 5  101   -75
 6   49   -50
 7   41   -44
 8•  17    -9
 9•  17    21
10   43    37
11   95    92
12   37   -46
13   46    45
14   66   -61
15   28    23
16  105   -98
17   54    48
18   83   -81
19   40    34
20   30   -34
21•  23    18

  1 -6  L

 0  243  -230
 1   50   -48
 2  118  -109
 3  194  -183
 4   48    44
 5  142  -140
 6   98    87
 7  109  -108
 8  147   149
 9   43   -45
10   84    66
11•  19   -24
12   28    29
13   54   -48
14•  20     0
15   82   -84
16   25    22
17   61   -57
18   34    34
19   37   -32
20   23    18
21•  23    -5

  1 -7  L

 1  108  -111
 2   49   -47
 3•  17     8
 4   59   -64
 5  127   127
 6   68   -60
 7  212   206
 8   44   -36
 9  131   135
10•  19    -7
11   35    41
12•  20   -10
13•  20   -10
14•  20   -10
15•  21    -5
16•  21    -6
17   25    20
18   27    23
19•  23    27
20•  23    21

  1 -8  L

 0  109  -109
 1   18    16
 2   85   -85
 3   44    41
 4   50    44
 5   31   -35
 6  148   147
 7   28   -24
 8  156   145
 9   27   -23
10   67    71
11•  20    12
12   30   -38
13   47    54
14R  24   -13
15   40    44
16•  21     7
17   70    65
18   24    21
19   51    50
20•  23    20

  1 -9  L

 1  154  -151
 2   43    47
 3  109  -109
 4   42    42
 5•  18    18
 6•  18     8
 7   35    37
 8   61   -54
 9   69    45
10   55   -51
11   24   -23
12•  20     6
13   30   -30
14   24    25
15•  21   -11
16   80    79
17•  23    16
18   73    73
19   37    32
20   46    47

  1-10  L

 0  111  -105
 1   57    41
 2  111  -110
 3   69    62
 4   80   -74
 5   23   -22
 6   35   -39
 7   59   -56
 8   55   -58
 9  133  -123
10   58   -49
11   92   -85
12•  22   -18
13•  27    -0
14•  22    -6
15   53    45
16•  24    14
17   74    68
18   28    19
19   54    46

  1-11  L

 1   27   -28
 2•  20    20
 3•  20    -6
 4   34   -33
 5   56   -54
 6   63   -62
 7   84   -80
 8  139  -136
 9   51   -53
10  160  -147
11•  23   -12
12  101   -94
13•  22    -9
14•  23   -10
15•  23     4
16   31    26
17•  24     0
18   37    31

  1-12  L

 0  108    94
 1•  20    -7
 2  103   100
 3•  21   -14
 4•  21    17
 5   35   -33
 6   38   -39
 7  121  -115
 8   66   -64
 9  164  -157
10   45   -37
11  140  -130
12•  23    -0

13   73   -66
14•  23    -5
15   27   -16
16•  24    -7
17•  24    -1
18•  25   -24

  1-13  L

 1  211   204
 2   25   -27
 3  135   134
 4•  22   -18
 5•  22    22
 6   61   -58
 7   30   -38
 8   71   -71
 9•  24   -34
10   66   -70
11•  23    -7
12   69   -59
13•  23     5
14   25   -21
15•  23   -11
16•  23   -11
17   35   -29

  1-14  L

 0  226   224
 1R  65   -33
 2  175   176
 3   26   -25
 4   88    93
 5•  22    -6
 6•  22     4
 7•  23    -3
 8   33   -24
 9   38    39
10•  23    -0
11   37    30
12•  23     7
13   26    11
14•  24   -15
15•  24    -0
16•  24   -25

  1-15  L

 1  123   117
 2R  50   -26
 3   72    74
 4•  23   -11
 5   37    37
 6   33    32
 7•  23   -13
 8   86    87
 9•  24   -20
10  104   102
11   29   -17
12   70    67
13   25   -15
14   34    20
15   28   -28

  1-16  L

 0R  58     5
 1   32   -14
 2•  23    17
 3•  23   -15
 4•  23     0
 5•  23     5
 6•  23   -10
 7   65    62
 8   28   -17
 9  103   101
10•  25   -21
11   93    87
12   32   -25
13   47    41
14   27   -26

  1-17  L

 1   60   -51
 2•  23   -15
 3   30   -32
 4•  23    -9
 5•  23   -15
 6•  24    26
 7•  24    -0
 8   55    57
 9•  25    -9
10   54    58
11   27   -25
12   34    33
13   27   -31

  1-18  L

 0   84   -77
 1•  24     1
 2   48   -45
 3•  24    -6
 4•  24   -14

 5•  23     6
 6•  24    12
 7•  24    24
 8•  24    20
 9•  25    19
10•  25   -11
11•  25    14

  1-19  L

 1   44   -39
 2•  24    18
 3•  24    -5
 4   25    19
 5•  24    29
 6•  24    15
 7   42    41
 8•  24     2
 9•  25    24
10•  25    -6

  1-20  L

 0•  24   -14
 1   29    24
 2•  24     0
 3   27    29
 4   37    32
 5   27    27
 6   41    42
 7•  25    13

  1-21  L

 1•  25   -11
 2   32    30
 3•  25     9
 4   39    38

  2 -1  L

 1  106   -99
 2  116   109
 3•  16   -11
 4  131   138
 5•  18    32
 6  211   199
 7   91    88
 8   58    65
 9   86    92
10   36   -31
11   73    69
12•  20   -14
13   58    61
14   50    45
15   38    35
16   68    65
17•  22    18
18   42    43
19   24    -2
20•  23    -4
21•  23   -15

  2 -2  L

 0   55    51
 1  140   136
 2  278  -259
 3  300   293
 4   54   -56
 5  310   298
 6   71   -68
 7  116   117
 8•  18     6
 9•  18    -0
10   82    83
11•  20    21
12   92    98
13   68    68
14  101   101
15   96    95
16   55    50
17  100    97
18   33    23
19   42    41
20•  23    -5
21•  23     1

  2 -3  L

 1   96   -94
 2  313   297
 3  198  -192
 4  108   106
 5  154  -162
 6   53    59
 7  188  -194
 8   30    25
 9   58   -61
10•  19     1
11   75    79
12•  21    11
13  148   153
14   38    43
15  109   112
16  101   101

17   48    46
18   60    64
19•  24    11
20•  24    23
21•  24     1

  2 -4  L

 0   84   -84
 1•  17   -18
 2  185  -176
 3   36    21
 4  262  -258
 5   49   -56
 6  304  -298
 7  112  -115
 8  180  -181
 9   63   -61
10   38    48
11   52   -55
12  111   114
13   32   -37
14  119   123
15•  23    16
16   71    73
17•  23    25
18•  28    27
19•  24    15
20•  24     5
21•  24    18

  2 -5  L

 1•  18     2
 2  111  -112
 3  276  -258
 4  234  -231
 5  159  -142
 6  254  -238
 7  143  -134
 8  174  -171
 9   72   -69
10   79   -75
11   59    57
12   91   -89
13   75    89
14   76   -75
15  112   108
16   62   -57
17   41    37
18   26   -27
19•  22    -6
20•  22     4
21•  22    -9

  2 -6  L

 0   94    86
 1   92   -93
 2•  17    10
 3  259  -251
 4   49   -48
 5  280  -279
 6R  30   -17
 7  151  -147
 8•  17     4
 9   84   -83
10   57    63
11   86   -84
12   38    32
13   72   -74
14•  25    37
15   81   -80
16•  24    23
17   64   -61
18•  22    -9
19•  22   -22
20•  22    -6
21•  23    23

  2 -7  L

 1  177   171
 2  155  -152
 3  142   137
 4  174  -186
 5  128   124
 6  145  -141
 7   97    91
 8   66   -67
 9   48    45
10•  19    -6
11•  19   -19
12•  23    -5
13   35   -36
14•  21   -14
15•  21     7
16   40   -43
17•  22   -16
18•  22    -6
19•  23   -19
20   27    26

  2 -8  L

 0  118   113

 1   74   -73
 2  121   117
 3  119  -123
 4  164   165
 5  145  -145
 6  115   119
 7   45   -45
 8   69    77
 9   36    25
10•  19    12
11   45    47
12   59   -55
13   34    37
14   42   -42
15•  21     7
16   58   -56
17•  22    23
18   25   -25
19   27    33
20•  23    -0

  2 -9  L

 1   58   -53
 2   37   -37
 3   44    46
 4•  17   -17
 5   94    95
 6   35   -39
 7   67    65
 8   23    23
 9   30    16
10   35    28
11   86   -85
12   34    37
13   64   -67
14   41    36
15   75   -72
16   38    43
17   24   -23
18   29    34
19•  23    13
20   33    34

  2-10  L

 0   84   -83
 1•  19   -21
 2   59   -53
 3   70    64
 4   26    26
 5   14    31
 6•  21    27
 7•  20     7
 8•  21    -2
 9•  21     1
10   76   -77
11•  23   -19
12   89   -83
13   28    33
14   80   -79
15   37    33
16   34   -33
17   44    41
18•  24    12
19   30    31

  2-11  L

 1   27   -23
 2   48    44
 3   30   -23
 4   58    57
 5•  20    12
 6•  21     3
 7•  21     3
 8   54   -43
 9   45   -44
10   77   -77
11   89   -86
12   42   -43
13   78   -73
14•  23    12
15   55   -52
16•  23    19
17•  24   -17
18•  24    11

  2-12  L

 0   42    36
 1   30    35
 2   49    51
 3   56    53
 4•  21    13
 5•  21    17
 6   24    23
 7   26   -26
 8•  22   -13
 9   83   -76
10   76   -36
11  113  -114
12   60   -59
13   58   -55
14   60   -57
15•  23   -17
```

Figure 14. (Continued)

```
16• 24   -28
17• 24    -5
18• 24   -11

  2-13   L

 1 140   138
 2  73    71
 3 104   104
 4  73    72
 5  44    43
 6• 22    16
 7• 22    14
 8  40   -41
 9• 23   -15
10  68   -65
11• 23   -10
12  55   -55
13  34   -26
14• 23   -21
15  25   -14
16• 24   -10
17• 24   -23

  2-14   L

 0 181   181
 1  56    40
 2 139   143
 3  84    85
 4  54    57
 5  68    69
 6• 22    18
 7  44    40
 8• 23    -7
 9• 23    25
10• 23     6
11• 23     8
12• 23    24
13• 23    -1
14• 23     0
15• 24     9
16• 24    -6

  2-15   L

 1 111   110
 2  66    55
 3  56    53
 4  81    81
 5• 23    -9
 6  75    78
 7  44   -42
 8  84    87
 9• 23   -16
10  75    72
11  36    23
12  60    60
13  39    34
14  42    34
15• 24     7

  2-16   L

 0  43    26
 1R 27    -3
 2• 22     3
 3  35    37
 4  34   -33
 5  57    59
 6  58   -55
 7  83    78
 8  42   -38
 9  99    97
10• 24    -1
11  82    79
12  35    34
13  55    53
14• 25    34

  2-17   L

 1  54   -46
 2• 23   -12
 3  65   -64
 4• 23     1
 5  67   -61
 6  31    27
 7  51   -50
 8  64    64
 9  31   -16
10  65    65
11• 25    13
12  40    42
13  34    35

  2-18   L

 0  99   -97
 1• 24   -21
 2  76   -74
 3  50   -42
 4  63   -58
 5  26   -25
 6  31   -31
 7• 24    10
```

```
 8• 24    -2
 9  36    34
10• 25    -0
11  28    26
12• 25    13

  2-19   L

 1  70   -70
 2  42   -34
 3  38   -40
 4  39   -37
 5• 24    -9
 6• 25   -21
 7• 25    28
 8• 25    -0
 9• 25    21
10• 25     7

  2-20   L

 0  48   -43
 1• 24    -5
 2• 24   -15
 3• 25   -29
 4• 24     6
 5• 25   -20
 6  35    41
 7• 25   -11
 8  44    47

  2-21   L

 1• 25   -22
 2• 25    -1
 3• 25     8
 4• 25    -2

  3 -1   L

 1 117  -102
 2  39    26
 3R 19     5
 4  34   -29
 5 150   142
 6  34   -26
 7 161   161
 8 107  -109
 9 122   123
10  34   -42
11  58    56
12  31   -29
13• 21   -10
14  22    21
15• 22   -11
16  27    25
17• 22    17
18  42    40
19• 23    19
20• 23    17
21• 23     2

  3 -2   L

 0R 46    23
 1  78    84
 2 142  -137
 3  69    73
 4  35    24
 5  37    34
 6 108   114
 7  43   -41
 8  75    86
 9  82   -72
10  71    76
11  47   -50
12• 20    -0
13  35    38
14• 22   -10
15  75    78
16• 23    26
17 101   103
18  28    24
19  73    75
20• 24    12
21  25    26

  3 -3   L

 1 122  -116
 2  79    73
 3 171  -158
 4  97    99
 5 112  -109
 6• 19     5
 7R 23   -16
 8 117  -110
 9  23    23
10  96   -98
11• 20    18
12• 24    15
13• 21    -8
14  74    78
15  33    33
16  87    91
17  37    24
```

```
18  99   110
19• 24    21
20  52    54
21• 24    16

  3 -4   L

 0  56    39
 1  91   -76
 2 159  -157
 3 104    99
 4 146  -144
 5• 19     9
 6  93  -100
 7 107  -101
 8 136  -133
 9 111  -117
10  53   -57
11 119  -118
12• 21   -19
13  31   -22
14  47    48
15  41    38
16• 23    23
17  77    78
18• 24     6
19  58    60
20• 24     4
21• 25    24

  3 -5   L

 1 136   133
 2  34   -32
 3  41   -11
 4  83   -81
 5  77   -74
 6 199  -190
 7 225  -220
 8 201  -201
 9 115  -110
10 217  -209
11• 19    -2
12 110  -104
13  27    24
14  44   -42
15• 21    20
16• 21     2
17• 21    -8
18• 21     4
19• 22   -20
20• 22    11
21• 23    -7

  3 -6   L

 0 251   251
 1 113  -105
 2 214   200
 3 107  -103
 4  44    44
 5 175  -166
 6 107  -104
 7 180  -185
 8 109  -109
 9 156  -153
10  58   -54
11 126  -118
12  33    33
13  81   -83
14  37    38
15  59   -58
16  23   -16
17  27   -29
18  52   -50
19• 22    -9
20  48   -44

  3 -7   L

 1 353   352
 2 120  -114
 3 244   238
 4 132  -132
 5  39    35
 6  83   -86
 7  53   -46
 8  19   -15
 9  44   -47
10• 19    12
11R 27     9
12  38   -42
13• 20    17
14• 21   -11
15  24   -25
16  41   -43
17  57   -53
18• 23   -24
19  62   -58
20• 23     2

  3 -8   L

 0 257   249
 1  90   -87
 2 290   301
```

```
 3 121  -119
 4 165   164
 5  27   -31
 6  29    34
 7  38    34
 8  32    24
 9 110   109
10• 20   -28
11  99    95
12  49   -48
13  58    58
14  35   -34
15• 21   -10
16  49   -43
17  38   -34
18  52   -52
19• 23   -10
20  50   -47

  3 -9   L

 1 103   108
 2 118  -121
 3 109   112
 4• 18   -13
 5  41    41
 6  50    53
 7  59    55
 8 137   138
 9• 20     0
10 181   182
11  64   -56
12 101   104
13  63   -62
14  37    37
15  64   -72
16• 22   -12
17  36   -39
18• 23   -11
19• 23   -11

  3-10   L

 0  69   -68
 1  35   -37
 2  41   -41
 3• 19    -5
 4• 20    19
 5  46    41
 6  40    38
 7 121   116
 8• 21     7
 9 158   151
10  57   -49
11 124   114
12  73   -70
13  76    73
14  75   -74
15• 23     2
16  30   -32
17• 24   -14
18• 24    12
19• 24    -8

  3-11   L

 1 154  -150
 2• 20     5
 3  67   -64
 4• 20     9
 5• 21     0
 6  54    54
 7• 21     7
 8  86    83
 9• 22    -5
10  74    71
11  54   -49
12  56    52
13  62   -60
14• 23   -16
15  58   -52
16  34   -30
17• 24     0
18  29   -25

  3-12   L

 0 138  -144
 1  27    18
 2  84   -81
 3  45    50
 4• 21   -18
 5  37    36
 6  42    53
 7  39    40
 8• 22    25
 9• 22    -5
10• 23   -20
11• 23   -14
12  35   -37
13  25   -19
14  52   -47
15  50   -49
16• 24   -16
17  34   -36
18• 24    16
```

```
  3-13   L

 1  24   -25
 2  74    73
 3• 21    10
 4  57    56
 5  49    50
 6  28    36
 7  70    71
 8• 22    -9
 9  41    43
10  47   -47
11• 22     3
12  33   -35
13• 23   -20
14  33   -31
15• 24    -7
16• 24   -25
17• 24    -0

  3-14   L

 0  23    17
 1  45    49
 2  23    23
 3  95    97
 4  35    33
 5  89    89
 6  50    53
 7• 23    28
 8  47    51
 9• 23   -24
10  39    39
11• 23   -16
12• 23    23
13• 23    -9
14• 24     8
15• 24     7
16• 24    12

  3-15   L

 1  26    14
 2  68    71
 3• 22    -8
 4  92    89
 5• 23     6
 6  78    76
 7• 23    15
 8  31    34
 9• 23    25
10• 23    17
11  34    34
12• 24    13
13  37    33
14  39    31
15  33    26

  3-16   L

 0  27   -31
 1  37    33
 2  26   -33
 3  60    62
 4  33   -35
 5  64    71
 6  46   -39
 7  42    36
 8• 24   -20
 9• 24    28
10• 24    25
11  28    21
12  50    50
13  26    31
14  46    45

  3-17   L

 1  55   -49
 2• 23    -5
 3  66   -64
 4• 23    18
 5  86   -83
 6  25    20
 7  73   -72
 8• 24     9
 9• 24   -20
10• 24     8
11  32    34
12• 25    12
13  61    56

  3-18   L

 0  50   -53
 1  31   -38
 2  54   -49
 3  43   -42
 4  84   -79
 5  37   -34
 6  81   -80
 7  30   -24
 8  44   -45
 9• 24   -10
10• 24    -2
```

```
11• 24    -5
12  46    40

  3-19   L

 1  30   -19
 2  63   -65
 3  36   -29
 4  69   -63
 5  40   -40
 6  61   -60
 7• 25   -25
 8  34   -33
 9• 25    -4
10• 25   -19

  3-20   L

 0  25    16
 1  29   -24
 2• 25    15
 3  67   -63
 4• 25     6
 5  70   -64
 6  26     8
 7  50   -46
 8• 25    16

  3-21   L

 1• 25    17
 2• 25   -24
 3  27    27
 4  45   -47
 5  35    31

  4 -1   L

 1  93    77
 2 140  -151
 3 103   111
 4 204  -209
 5 161   155
 6 162  -159
 7 160   160
 8  63   -67
 9  80    86
10• 20   -11
11• 20    -2
12• 21   -17
13  69   -66
14• 22   -17
15  40   -40
16  31   -21
17• 23   -24
18• 23     1
19• 23    -7
20• 23    26
21• 24    -7

  4 -2   L

 0  66    58
 1• 18     2
 2  77    68
 3 116  -113
 4 173   174
 5 116  -108
 6 211   221
 7• 19   -14
 8  97   101
 9• 19    -0
10• 20     1
11  37    45
12  93   -94
13  47    48
14  95   -90
15  34    30
16  40   -38
17  34    36
18• 23    -6
19  42    40
20• 24     6
21  39    38

  4 -3   L

 1  87   -81
 2  89    89
 3  71    62
 4  45   -44
 5  81    72
 6• 19    12
 7 108   108
 8• 19     1
 9R 23    10
10  25    27
11  93   -96
12  39    44
13 118  -121
14  60    68
15  76   -75
16  62    68
17  30   -24
18  71    71
19• 24    14
```

```
20  37    41
21• 25    23

  4 -4   L

 0  95  -104
 1  39    42
 2  58   -64
 3 122   120
 4  30    30
 5  34    35
 6• 19   -19
 7  30   -34
 8• 19   -21
 9  57   -57
10  37   -68
11  34   -31
12 111  -113
13R 29     5
14  82   -90
15  44    48
16  48   -51
17  58    61
18• 24     7
19  32    34
20• 25    23

  4 -5   L

 1• 19   -14
 2  50    48
 3• 19   -13
 4  69    70
 5  42    -3
 6• 17     7
 7  19   -23
 8  97   -98
 9  74   -76
10 146  -142
11  97   -94
12  89   -87
13  59   -59
14  37   -36
15  57   -58
16  29    22
17  27   -16
18• 21     6
19• 22     1
20• 22   -13

  4 -6   L

 0 151   147
 1• 16     4
 2 100   101
 3 111   112
 4  50    52
 5  64    60
 6• 18   -20
 7  37   -38
 8  29   -29
 9 115  -114
10  52   -52
11 137  -140
12  35   -30
13  60   -59
14  35   -49
15• 21   -11
16  42   -45
17• 21   -13
18  26   -20
19• 23   -32
20• 23   -12

  4 -7   L

 1 262   270
 2  42    42
 3 158   159
 4 100   102
 5 102    98
 6  90    87
 7  24   -13
 8• 18   -15
 9  35   -29
10  60   -58
11  38   -10
12  55   -58
13• 21   -12
14  34    -9
15• 21   -15
16• 21   -11
17  39   -39
18• 22   -20
19  57   -50
20  25   -23

  4 -8   L

 0 274   288
 1  68    60
 2 235   244
 3  56    58
 4  78    79
 5  61    56
 6• 18    -4
```

```
 7  80    82
 8  45   -37
 9  81    80
10• 20    23
11  50    51
12  46    30
13  22    20
14• 22    22
15• 21     9
16• 22   -24
17• 22    -9
18  54   -50
19  27   -30

  4 -9   L

 1 102    97
 2  46    48
 3  69    73
 4  47    47
 5  26   -14
 6 116   116
 7  50   -46
 8 170   173
 9• 20    -4
10 190   201
11  43    40
12  96    94
13  29    29
14  30    32
15• 22    23
16• 22    -3
17• 22   -14
18• 23   -21
19  26   -27

  4-10   L

 0  68   -21
 1• 19    11
 2  45   -47
 3  30    23
 4  56   -53
 5  67    52
 6  70   -65
 7 152   148
 8  48   -43
 9 204   200
10R 27     7
11 178   174
12• 28    20
13  68    64
14• 24    26
15• 23     0
16  24    20
17• 24   -13
18• 24     6
19• 24   -12

  4-11   L

 1 180  -173
 2• 20   -12
 3 138  -138
 4• 21   -14
 5  78   -69
 6  33    37
 7  47   -41
 8 105   101
 9• 22    -2
10 117   107
11• 23    -6
12  79    74
13• 23    18
14• 23    -2
15• 24    17
16  39   -32
17  41    29
18  40   -34

  4-12   L

 0 191  -186
 1  41   -31
 2 171  -168
 3  43   -42
 4  79   -80
 5  39   -39
 6• 23   -13
 7• 22    15
 8• 24    26
 9  28    27
10• 29    23
11• 23     1
12• 23     2
13• 23    -8
14• 24     9
15  57   -56
16  36    24
17  50   -43

  4-13   L

 1 144  -146
 2  34   -28
 3  61   -65
```

```
 4  35   -32
 5  46    51
 6  27   -24
 7  71    75
 8  28   -27
 9  68    68
10  41   -48
11• 23     8
12  35   -35
13• 23     3
14  48   -40
15• 24    19
16  36   -38
17• 25    19

  4-14   L

 0  78   -76
 1  25     8
 2  36   -44
 3• 22    13
 4• 22    16
 5• 22    -7
 6  80    87
 7• 23   -21
 8  72    80
 9  30   -39
10  31    41
11  46   -46
12• 23     6
13  35   -27
14• 24     2
15• 24    -3
16• 24     9

  4-15   L

 1  38   -36
 2  29    29
 3• 22     5
 4• 23    28
 5  42    48
 6• 23     2
 7  55    61
 8• 23   -20
 9  40    42
10  26   -29
11  26    22
12• 24   -16
13• 24     0
14  27    26
15• 25     7

  4-16   L

 0  24   -25
 1• 22    18
 2• 23   -21
 3  40    38
 4• 24   -12
 5  32    32
 6• 24    16
 7• 24   -13
 8  24    18
 9  26   -23
10• 24    21
11• 24   -23
12• 24    18
13• 24    13
14• 25     8

  4-17   L

 1  32   -28
 2  33    34
 3  43   -41
 4  29    33
 5  45   -46
 6• 24   -12
 7  39   -42
 8  25   -30
 9• 24   -18
10  43   -40
11• 24    10
12• 25   -15
13• 25    16

  4-18   L

 0• 23   -10
 1• 23    -7
 2  27   -21
 3• 23     7
 4  51   -50
 5• 24   -19
 6  67   -72
 7  45   -47
 8  54   -54
 9  54   -50
10• 25   -16
11  38   -40
12• 25     8

  4-19   L

 1  34    31
```

```
 2• 24   -14
 3• 24    -4
 4  33   -25
 5  38   -32
 6  56   -58
 7  44   -47
 8  59   -57
 9  34   -30
10  50   -49

  4-20   L

 0  71    71
 1  29   -14
 2  60    60
 3  43   -39
 4• 25    22
 5  54   -51
 6• 25   -11
 7  48   -43
 8• 25   -17

  4-21   L

 1• 25    68
 2  32   -30
 3  57    55
 4  40   -40

  5 -1   L

 1 313   304
 2 152  -147
 3 218   214
 4 166  -161
 5 131   129
 6  90   -90
 7  44    41
 8• 19    -3
 9• 20    -0
10  21    18
11  38   -35
12• 21    16
13  31   -33
14  44   -46
15  43   -44
16  46   -41
17  68   -63
18  49   -43
19  45   -39
20• 24     4

  5 -2   L

 0 218   223
 1  27   -16
 2 197   194
 3 110  -108
 4 188   193
 5  50   -48
 6 116   123
 7  80    84
 8  56    58
 9  91    95
10  38   -36
11 110   109
12  69   -69
13  54    55
14  63   -65
15• 22     6
16  65   -63
17• 23   -16
18  36   -35
19• 23    -7
20• 24   -20

  5 -3   L

 1• 18    11
 2• 18    -8
 3  92    90
 4• 19     6
 5  71    72
 6 160   154
 7  68    73
 8 137   138
 9• 20    11
10 155   155
11  85   -89
12 112   115
13  94   -96
14  70    83
15  76   -79
16• 23    14
17  34   -37
18• 23     7
19• 24     3
20• 24     4

  5 -4   L

 0  95   -92
 1• 18     4
 2  89   -91
 3  30    32
 4  28     8
```

```
 5 147   142
 6  50    59
 7 131   137
 8  25    24
 9 111   117
10  42   -43
11  88    84
12 114  -121
13  52    54
14  90   -90
15• 23    20
16  42   -45
17• 23     1
18  24    10
19• 24    -7
20  35    32

  5 -5   L

 1 159  -163
 2  75    79
 3  74   -74
 4  75    80
 5  45    36
 6  85    82
 7  28    29
 8• 18     8
 9  28   -23
10• 19     3
11  62   -63
12• 20    -4
13  97   -94
14• 21   -15
15  42   -48
16• 21   -21
17• 21     6
18  23   -23
19  40    41
20  36   -36

  5 -6   L

 0  99   -97
 1  29    19
 2 138  -140
 3 139   139
 4• 17   -12
 5  93    94
 6  54    50
 7• 18     7
 8• 18    17
 9  42   -43
10• 19    10
11  56   -53
12  43   -29
13  54   -47
14  38   -36
15  36   -36
16• 21   -15
17• 22   -19
18• 22    16
19  36   -35
20  29    28

  5 -7   L

 1  55    54
 2 159   170
 3  30   -27
 4 160   162
 5  79    78
 6  74    75
 7  32    28
 8• 19    -0
 9  29    27
10  70   -71
11  36    40
12  48   -51
13• 21     4
14  33   -36
15• 21    -1
16• 21    -7
17• 22    -1
18• 22   -15
19• 23    -0

  5 -8   L

 0 112   108
 1  50    46
 2  82    83
 3 149   157
 4  44    43
 5 126   127
 6  21    26
 7  65    66
 8  25    19
 9• 20   -21
10  51    48
11  31   -33
12  54    48
13  25     9
14  54    54
15• 22    10
16  22    22
17• 22    10
```

Figure 14. (Continued)

```
18• 23   -20
19• 23    -7

  5 -9   L

 1  97   101
 2  82    81
 3  18   -20
 4 156   164
 5  52   -46
 6  97    97
 7  35   -35
 8  44    45
 9  20    24
10  33    32
11  69    70
12  26    25
13  84    89
14  59    58
15  70    72
16  47    44
17• 22     7
18  24    24
19• 23   -20

  5-10   L

0R  42   -15
 1• 20     8
 2  61   -64
 3  59    56
 4 105   -98
 5  72    69
 6 124  -119
 7  71    70
 8  73   -68
 9  72    72
10  40    31
11  51    50
12  82    82
13  57    53
14 112   106
15  54    48
16  65    62
17  28    29
18• 24     4

  5-11   L

 1 111  -105
 2  35   -32
 3 136  -130
 4• 21   -11
 5 167  -161
 6• 22    -4
 7 132  -135
 8  34    25
 9  34   -33
10  62    61
11  38    37
12  42    25
13  97    92
14• 24    16
15  83    74
16• 24     0
17  35    33
18• 24     5

  5-12   L

 0 103  -101
 1  60   -57
 2 100   -96
 3 115  -116
 4  99   -96
 5 112  -108
 6  96   -90
 7  60   -59
 8  54   -52
 9• 22    13
10• 23    -2
11• 23    -3
12  52    50
13  34   -12
14  62    56
15  44   -40
16  48    38
17  31   -31

  5-13   L

 1  58   -59
 2 120  -121
 3  36   -23
 4 148  -147
 5• 23   -26
 6 127  -122
 7• 23     6
 8  60   -60
 9  30    25
10  39   -43
11• 23    15
12  35   -30
13  26    26
14  51   -46
15• 24    17

16  51   -50

  5-14   L

 0  42   -30
 1  71   -76
 2• 22    15
 3  96   -94
 4• 23    14
 5  97   -99
 6  47    52
 7  74   -73
 8  52    54
 9  48   -53
10• 23    13
11  32   -36
12• 23     2
13• 24   -20
14  25   -11
15  33   -34
16• 25    -5

  5-15   L

 1  38    38
 2  44   -49
 3• 24    36
 4  41   -47
 5  58    59
 6  48   -49
 7  77    78
 8• 23   -21
 9  36    33
10• 26   -23
11• 24    -7
12• 24     1
13  31   -28
14• 24     6
15• 25   -25

  5-16   L

 0• 23     8
 1• 23     9
 2• 23    19
 3• 23     2
 4  49    51
 5• 23   -11
 6  71    71
 7• 24   -15
 8  33    42
 9• 24   -12
10• 24    -3
11• 24     1
12  31   -28
13• 25    21
14  40   -38

  5-17   L

1R  24   -10
 2  29    39
 3• 23    -1
 4• 23    21
 5• 24    18
 6• 24     5
 7• 24     9
 8• 24   -19
 9• 24   -13
10• 25   -15
11• 25   -27
12• 25     2
13  35   -35

  5-18   L

 0  26   -24
 1  28    27
 2• 23   -13
 3  33    25
 4• 24    -7
 5• 24    10
 6• 24   -15
 7• 24   -20
 8  26   -31
 9  27   -30
10  34   -33
11• 25   -22

  5-19   L

 1• 24    13
 2• 24    13
 3• 24    11
 4• 24    15
 5• 24    -9
 6• 24    -5
 7  31   -27
 8  34   -35
 9  33   -29

  5-20   L

 0  64    63
 1• 25    -3
 2  56    55

 3• 25    11
 4• 25    17
 5• 26     5
 6• 25    -7
 7• 25   -15

  5-21   L

 1• 25    78
 2• 26    -2
 3  44    45
 4• 25    12

  6 -1   L

 1 291   282
2R  46   -20
 3 213   216
 4  46    41
 5  35    35
 6  24    25
 7  44   -34
 8  25    23
 9  25   -26
10• 21     7
11• 21    15
12• 22   -18
13  34    31
14  36   -33
15• 22   -12
16  30   -29
17  51   -49
18  46   -44
19  57   -54
20  28   -27

  6 -2   L

 0 267   264
 1  55   -53
 2 184   190
 3  25   -18
 4  93    96
 5  64    68
 6• 19     1
 7 139   142
 8  26   -12
 9 140   148
10• 21     1
11  96    99
12  27    28
13  44    49
14• 23     6
15• 22   -12
16• 23   -21
17  35   -35
18• 24   -28
19  25   -26
20  37   -38

  6 -3   L

 1  35    36
 2• 19    -1
 3• 19    18
 4• 19    18
 5• 19     1
 6 123   126
 7• 20    -4
 8 214   223
 9  31     5
10 210   219
11• 22    -5
12 131   143
13• 23    -1
14  46    61
15• 23     8
16• 23   -15
17• 23    -8
18• 27   -20
19• 24    -7
20• 24    -7

  6 -4   L

 0 129  -127
 1  25    28
 2 107  -112
 3• 19     8
 4  75   -70
 5  61    60
 6  23   -25
 7 153   159
 8  32   -15
 9 198   208
10  28    25
11 151   158
12• 23   -18
13  64    64
14• 23    -0
15• 23    -3
16• 24    15
17• 24   -23
18• 24    23
19  31   -23

  6 -5   L

 1 216  -227
 2• 20    14
 3 140  -146
 4  38   -40
 5  86   -76
 6  46    45
 7• 19   -18
 8  82    79
 9• 19     3
10 112   109
11R 29     3
12  51    43
13• 21    -7
14• 21   -18
15  29    22
16  45   -45
17  48    41
18  36   -35
19  47    44

  6 -6   L

 0 255  -257
 1  26   -17
 2 208  -219
 3• 22     3
 4  59   -60
 5  27   -27
 6• 18     8
7R  26    -9
 8  26    27
 9• 19     2
10  34    33
11• 20    -4
12• 22     9
13  35   -35
14• 21     1
15  52   -49
16  26    27
17  38   -42
18  42    47
19  31   -31

  6 -7   L

 1 149  -153
 2• 18    -1
 3  68   -69
 4• 18    -3
 5  55    61
6R  28   -16
 7  66    68
 8  61   -60
 9  63    63
10  84   -87
11  42    39
12  50   -48
13• 21     9
14  38   -42
15• 21     6
16  29   -29
17  30    33
18• 23   -16
19  32    22

  6 -8   L

 0  73   -75
 1• 18    13
 2  40   -38
 3  66    67
 4  44    43
5R  20    14
 6  84    89
 7  53   -46
 8  97    99
 9  75   -79
10  53    55
11  75   -78
12  30    35
13• 21   -13
14• 22    20
15• 22     1
16• 23    16
17• 23    22
18• 23    13
19• 23     6

  6 -9   L

 1• 18    -2
 2  49    49
 3• 19    10
 4  66    70
 5  39    39
 6• 20     9
 7  40    43
 8  26   -26
 9  47    58
10  43   -42
11  37    46
12• 21   -21
13  33    36
14  39    40

15• 24    30
16  58    58
17• 23    12
18  47    46

  6-10   L

0R  31   -23
 1• 20    18
 2• 20   -13
 3  50    52
 4  65   -62
 5  30    28
 6  37   -38
 7• 22   -16
 8• 22    -5
 9  29   -28
10• 23    15
11• 22   -14
12• 25    26
13• 23    18
14  27    42
15  71    66
16  36    29
17  76    71
18• 25     7

  6-11   L

 1  41   -36
 2• 21     0
 3  66   -66
 4• 21     0
 5 124  -122
 6  34   -32
 7  96   -95
 8  54   -51
 9  40   -50
10  29   -27
11• 23    11
12  33   -25
13  44    47
14  35    34
15  40    36
16  51    50
17• 25    18

  6-12   L

 0  32    26
 1  32   -27
 2• 21    -0
 3  66   -64
 4  71   -80
 5  73   -70
 6 124  -129
 7  81   -75
 8  88   -87
 9  60   -59
10• 24   -12
11  55   -47
12  30    35
13• 24   -18
14  30    33
15• 24    -1
16• 25    22
17• 25     0

  6-13   L

 1  60    54
 2  86   -87
 3  33    27
 4 121  -124
 5  51   -41
 6 111  -105
 7  71   -70
 8  71   -71
 9  30   -31
10  47   -47
11• 23    -2
12  32   -34
13  25    23
14  48   -44
15• 24     4
16  32   -35

  6-14   L

 0  88    85
 1  39   -41
 2  78    85
 3  90   -90
 4  47    52
 5 106  -110
 6• 27     5
 7  64   -64
 8• 24   -11
 9• 24   -27
10• 24   -11
11• 24   -14
12• 24    -8
13  29   -24
14  35   -19
15  43   -45

  6-15   L

 1  94    94
 2  49   -50
 3  94    97
 4  78   -82
 5  82    83
 6  45   -46
 7  47    38
 8• 23     5
 9• 23    12
10  29    26
11• 24   -21
12  33    27
13  32   -35
14• 25   -17

  6-16   L

 0• 28    41
 1• 23    -6
 2  52    54
 3  31   -30
 4  48    71
 5  32   -14
 6  73    80
 7  29    30
 8  30    44
 9  49    48
10• 25    -8
11  53    55
12  39   -38
13• 26    24

  6-17   L

 1• 23   -15
 2• 23    14
 3• 24    13
 4• 24    11
 5  39    43
 6  35    40
 7  31    37
 8  50    55
 9• 25    -2
10  49    48
11  37   -34
12  30    35

  6-18   L

 0  56   -49
 1• 24    13
 2  28   -33
 3• 24    20
 4• 24    -5
 5  43    40
 6• 25    15
 7  35    36
 8• 25     1
 9• 29    27
10• 25   -18

  6-19   L

 1  29   -35
 2  25    29
 3• 24   -23
 4  42    40
 5• 24     5
 6• 25    24
 7• 25     4
 8• 25     4
 9• 25    -4

  6-20   L

 0• 25    -2
 1• 25    12
 2• 25    -4
 3  28    40
 4• 25     6
 5  35    31
 6• 25     4

  6-21   L

 1• 25    20
 2• 25    25

  7 -1   L

 1 125   101
 2  95    93
 3  64    56
 4 140   142
 5  22    14
 6  90    91
 7• 20     9
 8  31   -34
 9• 21    19
10  45   -38
11  55    57
12  54   -56
13  42    39
14  29   -31

15• 23    19
16• 23    -4
17• 23    -4
18• 24   -23
19• 24   -13

  7 -2   L

 0 166   161
 1  50    45
 2  91    85
 3 111   111
 4• 20    24
 5  80    87
 6• 20   -19
 7  68    76
 8  28   -29
 9  28    29
10  27    32
11• 22    19
12  89    88
13• 22     5
14  70    74
15• 23    16
16  28    23
17• 23     6
18• 24   -11
19• 24    -8

  7 -3   L

 1  44    45
 2  46    42
 3  39   -17
 4  54    55
 5  75   -80
 6  93    90
 7  76   -73
 8 107   106
 9• 22    -1
10  86    92
11  75    79
12  79    85
13  99   103
14  33    42
15  66    68
16  24    29
17• 23     6
18• 24     6
19• 24   -16

  7 -4   L

 0  34   -41
 1• 19     2
 2  69   -78
 3• 20   -11
 4 122  -126
 5• 21    14
 6 109  -108
 7  76    87
 8  59   -49
 9 101   102
10• 22    15
11  93    96
12  72    78
13  52    57
14  95   100
15  25    11
16  56    54
17• 24     3
18• 24    10
19• 25     4

  7 -5   L

 1 158  -167
 2  60   -65
 3 139  -147
 4 110  -111
 5 140  -136
 6  37   -31
 7  77   -75
 8  30    35
 9• 20    -7
10  66    65
11R 42    15
12  28    29
13  71    64
14• 21    -2
15  76    72
16  28   -26
17  46    43
18• 23   -12
19  26    27

  7 -6   L

 0 179  -172
 1  49   -48
 2 176  -184
 3 122  -124
 4 106  -102
 5 132  -132
 6  44   -41
 7  85   -87

 8• 19    -0
 9• 20   -21
10• 20    15
11• 20    -1
12R 29     5
13  34   -31
14  44    41
15  45   -43
16  39    39
17  41   -35
18  37    37

  7 -7   L

 1 103  -103
 2  82   -79
 3  64   -63
 4 171  -180
 5  40    35
 6 123  -119
 7  56    54
 8  94   -93
 9  41    43
10  47   -49
11• 21     5
12  53   -58
13• 21    -5
14  44   -57
15• 22    -2
16  41   -42
17• 23    17
18  25   -18

  7 -8   L

 0  33   -28
 1  47   -39
 2• 19     6
 3 103  -105
 4  87    84
 5 102  -103
 6 109   111
 7  92   -90
 8  96    95
 9  87   -82
10  26    29
11  55   -61
12• 21   -19
13  33   -30
14  30   -29
15• 22   -19
16• 22   -13
17• 23    -3
18• 23     8

  7 -9   L

 1  28    31
 2  32   -35
 3  54    54
 4  25   -28
 5 113   117
 6  38   -37
 7 104   112
 8  55   -60
 9  55    54
10  53   -54
11• 21    -4
12• 21    -7
13  39   -41
14• 22    16
15  37   -34
16  33    32
17• 23   -11

  7-10   L

 0  21    -4
 1• 20    -7
 2• 21    20
 3  37    36
 4  33    28
 5  26    17
 6  60    63
 7• 22    -6
 8  28    29
 9  45   -45
10• 23    -6
11• 23   -22
12  33   -40
13  27    30
14  47   -45
15  54    53
16  38   -24
17  61    56

  7-11   L

 1• 21    -0
 2  51    52
 3  22   -27
 4  81    80
 5• 22    -0
 6• 22     5
 7• 22   -11
 8  32   -34

 9  23   -26
10  28   -36
11  41   -42
12• 23    -9
13  43   -45
14  36    36
15  28   -26
16  60    56

  7-12   L

 0  62    63
 1  41    43
 2• 22     2
 3  62    63
 4• 22   -12
 5• 22     6
 6  34   -35
 7  42   -39
 8  63   -65
 9  55   -54
10  43   -42
11  43   -50
12• 24   -29
13• 24   -18
14• 24   -15
15• 24    17
16• 25   -10

  7-13   L

 1  57    58
 2  30    32
 3  27    20
 4• 22    -4
 5• 23   -17
 6  34   -30
 7  56   -61
 8  57   -59
 9  36   -36
10  53   -56
11  31   -29
12  47   -52
13• 24    -8
14• 24   -24
15• 24    -6

  7-14   L

 0 101   108
 1• 23     5
 2  82    88
 3• 23     1
 4  33    37
 5• 23     3
 6  26   -23
 7• 25   -19
 8  38   -40
 9• 24   -21
10• 24   -20
11  27   -32
12• 24    -5
13  32   -29
14• 24     2

  7-15   L

 1 129   135
 2• 24   -23
 3  94    93
 4• 24    -4
 5  26    30
 6• 23    20
 7  28   -10
 8  36    36
 9• 24    -5
10• 24    24
11• 24    -1
12• 25    14
13• 25     5

  7-16   L

 0  78    77
 1• 24   -26
 2  76    78
 3• 24    -7
 4  43    42
 5• 24    19
 6• 24    23
 7  63    62
 8• 25    10
 9  77    77
10• 25    -0
11  56    58
12• 25     1

  7-17   L

 1• 24     0
 2• 23    -5
 3• 24    -6
 4• 24    23
 5• 24     2
 6  49    44
 7• 25    12

 8  86    86
 9• 26     2
10  73    73
11• 25    -4

  7-18   L

 0  61   -64
 1• 24     5
 2  58   -56
 3• 24    14
 4  29   -34
 5• 25    25
 6• 25    -0
 7  51    50
 8• 25     6
 9  61    61
10• 26    -4

  7-19   L

 1  74   -77
 2• 25     9
 3  47   -51
 4• 25    16
 5  27   -13
 6• 25    18
 7• 25     9
 8• 25    20

  7-20   L

 0  56   -59
 1• 25     6
 2  45   -46
 3• 25    10
 4• 25   -15
 5• 25     9

  8 -1   L

 1  95   -85
 2  71    70
 3  35   -30
 4  35    38
 5  35    35
 6  27   -22
 7  56    56
 8  96   -97
 9  62    65
10  71   -70
11  51    54
12  80   -77
13  31    26
14  38   -39
15• 23    23
16• 23    -2
17• 24    20
18• 24    -6

  8 -2   L

 0  43     8
 1• 21    16
 2  24    -9
 3  80    81
 4• 21    26
 5• 22    49
 6• 21    14
 7  43   -32
 8• 23    27
 9  80   -80
10  62    64
11  52   -56
12  46    62
13• 23   -20
14  27    41
15  25    20
16• 24    19
17  24    29
18• 24     8

  8 -3   L

 1• 20    21
 2  31    35
3R  30    -0
 4  69    68
 5• 21    -9
 6  35    33
 7• 22   -10
 8  24   -30
 9• 22    17
10  38   -41
11  37    44
12• 23     5
13  59    64
14  32    46
15  43    46
16  56    61
17• 24     8
18  51    48

  8 -4   L

 0  28    23

 1• 20     0
 2• 21    -6
 3• 21    18
 4  52   -56
 5• 22    11
 6  91   -97
 7• 22   -10
 8  37   -39
 9  24   -25
10• 22    16
11  36   -11
12  59    58
13  54    37
14  59    65
15  52    51
16• 25    27
17  50    54
18• 25    -1

  8 -5   L

 1• 21   -19
 2  47   -52
 3  59   -65
 4  58   -62
 5 133  -133
 6  55   -53
 7 109  -112
 8  36   -38
 9  45   -43
10• 21   -18
11• 20     6
12• 21    -4
13  64    59
14• 22    13
15  58    58
16  27    18
17• 23    11
18• 23    25

  8 -6   L

 0• 19    -8
 1  55   -56
 2• 19   -23
 3 130  -129
 4  85   -89
 5 131  -137
 6 113  -108
 7  91   -89
 8  79   -87
 9  30   -37
10• 20   -17
11• 21   -21
12R 23    13
13  34   -35
14  31    35
15  33   -29
16• 23    12
17• 23   -15

  8 -7   L

 1  40    38
 2 113  -114
 3  34    26
 4 166  -171
 5• 20    -9
 6 119  -127
 7  38   -32
 8  80   -79
 9  22   -25
10  32   -32
11• 21   -10
12  36   -37
13• 21   -12
14  51   -59
15• 22   -14
16  59   -55
17• 23    -6

  8 -8   L

 0 102   103
 1  60   -56
 2  89    90
 3 111  -113
 4 100   103
 5 115  -119
 6  63    67
 7  70   -70
 8  23    35
 9  33   -26
10• 20     1
11• 21   -16
12  29   -35
13  26   -27
14  47   -46
15  43   -44
16• 26   -36
17  35   -32

  8 -9   L

 1 106   109
 2  30   -29
```

Figure 14. (Continued)

```
 3 108  112
 4  49  -49
 5 130  137
 6  23  -28
 7  65   69
 8•  21   -1
 9  37   29
10R 26    8
11  22  -24
12•  22   20
13  63  -65
14•  23    0
15  60  -59
16  24  -12
17  29  -28

   8-10   L

 0  37   35
 1•  21   -5
 2  42   47
 3•  22    8
 4  84   83
 5R 30   13
 6  85   86
 7  36   34
 8  39   41
 9  45   42
10•  23  -15
11  51   48
12  69  -68
13  42   39
14  76  -71
15•  25   12
16  46  -41

   8-11   L

 1  31  -32
 2  42   43
 3•  22   -2
 4  79   80
 5  58   60
 6  62   63
 7  30   37
 8  51   49
 9•  23   -9
10  37   39
11  49  -49
12  41   34
13  67  -67
14  29   33
15  50  -46
16  36   30

   8-12   L

 0  39  -41
 1  30   29
 2  30  -24
 3  74   77
 4•  23   -6
 5  74   77
 6•  23   10
 7  40   43
 8•  31  -16
 9•  23    7
10  31  -29
11•  23   -0
12  32  -42
13•  24    9
14  40  -40
15•  24   14

   8-13   L

 1•  22  -27
 2  41   44
 3•  22  -11
 4  68   71
 5•  23  -18
 6  35   35
 7•  23  -18
 8•  23   -7
 9  26  -24
10  28  -29
11•  24  -20
12  32  -38
13•  24  -13
14•  24  -11

   8-14   L

 0  31   24
 1•  23   23
 2•  27   22
 3  57   64
 4•  23   -1
 5  49   52
 6•  23  -20
 7•  23    1
 8•  24  -16
 9  28  -20
10•  24   -0
11  44  -49
12•  24    7

13  29  -29

   8-15   L

 1  68   73
 2  34   37
 3  37   43
 4  52   59
 5•  23   -7
 6  35   29
 7•  24  -20
 8•  24    8
 9•  24   12
10•  24   -7
11•  25   21
12•  25  -13

   8-16   L

 0  63   68
 1•  23    1
 2  44   50
 3  30   33
 4•  24    2
 5  38   41
 6•  24  -24
 7  40   38
 8•  25   -1
 9  36   37
10  27   20
11•  25   27

   8-17   L

 1  29   11
 2•  24   -0
 3•  24   -8
 4•  24   14
 5  31  -35
 6  29   33
 7  26  -28
 8  48   52
 9•  25    0
10  45   48

   8-18   L

 0  53  -50
 1•  24  -12
 2  41  -38
 3  25  -25
 4  44  -48
 5•  25   -3
 6  37  -39
 7  28   28
 8•  25  -15

   8-19   L

 1  66  -68
 2•  25  -27
 3  52  -51
 4  31  -37
 5  34  -36
 6•  25  -14

   8-20   L

 0  57  -60
 1•  25  -13
 2  45  -45

   9 -1   L

 1  90  -83
 2  66  -57
 3•  22   -8
 4  85  -86
 5  52   50
 6 106 -105
 7  80   83
 8  94  -94
 9  54   52
10  68  -65
11•  23    5
12  65  -63
13  25  -12
14  36  -35
15•  24  -10
16  26  -23
17•  24   11

   9 -2   L

 0  26  -26
 1•  22  -10
 2•  22   -8
 3  38  -35
 4  67   71
 5  62  -71
 6 102  105
 7  87  -90
 8  78   76
 9  68  -72
10  31   28
11  68  -67

12  24  -22
13  29  -36
14  31  -28
15•  24    3
16•  24  -15
17•  24   16

   9 -3   L

 1R 24    6
 2  35   31
 3  59   56
 4•  22    2
 5  76   80
 6•  23  -21
 7  65   71
 8  61  -66
 9  31   39
10  43  -38
11•  23   -1
12•  23  -23
13  33  -27
14•  24   25
15•  25  -34
16  48   53
17•  25  -14

   9 -4   L

 0  29   39
 1•  22   21
 2  29   36
 3  46   51
 4  28   24
 5  44   42
 6•  23   20
 7•  22   -5
 8  27   17
 9  44  -43
10  25  -16
11  30  -26
12•  23  -16
13•  24   18
14  26  -25
15  50   60
16•  25  -22
17  61   61

   9 -5   L

 1  38   45
 2  33   35
 3•  22   10
 4  41   41
 5  65  -62
 6•  20    8
 7  48  -51
 8  32  -33
 9  40  -42
10  49  -48
11  29  -29
12•  21  -13
13  23  -16
14  38   28
15•  23   -8
16  54   49
17•  23  -12

   9 -6   L

 0  57   57
 1•  19   -6
 2  36   37
 3•  20   18
 4  45  -53
 5•  20   -2
 6  79  -81
 7  35  -38
 8  77  -78
 9  57  -61
10  50  -51
11  39  -44
12•  21  -19
13  23  -19
14•  22   -0
15•  22    2
16•  23   -4

   9 -7   L

 1 110  116
 2•  20    1
 3  30   26
 4  39  -38
 5  37  -43
 6  44  -45
 7  75  -70
 8  39  -34
 9  54  -54
10  31  -32
11  23  -20
12  35  -37
13•  22    1
14  37  -35
15•  22   -8
16•  23  -28

   9 -8   L

 0 143  148
 1•  21   -2
 2 106  109
 3  34  -37
 4  75   77
 5  42  -45
 6•  21    7
 7•  21   -2
 8  25  -25
 9  24   17
10•  21  -13
11•  21    4
12•  22   -8
13  23  -25
14•  22   -5
15  44  -42
16•  24  -22

   9 -9   L

 1 116  126
 2•  21   -9
 3  98  103
 4  41  -44
 5  77   79
 6•  21   27
 7•  21   14
 8  56   54
 9•  22    1
10  69   67
11  22  -10
12•  23   28
13•  23  -11
14•  23  -14
15•  24  -23

   9-10   L

 0  21   27
 1R 34   -3
 2  48   49
 3  24  -21
 4  65   64
 5  32   26
 6  37   40
 7  83   85
 8  30   20
 9 110  105
10  31   -4
11  88   84
12  26  -22
13  46   42
14  28  -23
15•  25   -2

   9-11   L

 1  39  -40
 2•  22   -1
 3•  22  -17
 4  41   40
 5•  23   21
 6  79   80
 7  33   27
 8 106  104
 9•  24   13
10 102   98
11•  24  -14
12  68   66
13  29  -28
14•  25   25

   9-12   L

 0  91  -92
 1•  23    3
 2  77  -77
 3  35   39
 4  43  -49
 5  44   46
 6•  23   11
 7  58   65
 8•  24   12
 9  58   58
10•  24   -5
11  45   44
12•  25  -16
13•  25   22
14•  25  -12

   9-13   L

 1 104 -104
 2•  23   20
 3  89  -85
 4  30   33
 5  24  -30
 6•  23   14
 7•  23   12
 8•  23   15
 9•  23   11
10•  24    0
11•  24   -5

12•  24   -9
13•  24   -9

   9-14   L

 0  69  -73
 1•  23   18
 2  64  -67
 3  32   36
 4  31  -26
 5  24   20
 6•  23   12
 7•  24  -13
 8•  24   21
 9  26  -31
10•  24   16
11  36  -39
12•  25    5

   9-15   L

 1•  23  -13
 2•  23   28
 3•  23   -0
 4•  24   27
 5•  24   11
 6•  24    3
 7•  24   17
 8  28  -32
 9  33   30
10  40  -40
11  26   22

   9-16   L

 0•  23    9
 1•  23   11
 2•  23    9
 3•  24   24
 4•  24   11
 5•  24   19
 6•  24    4
 7•  24  -10
 8•  25   13
 9•  25  -18
10•  25   25

   9-17   L

 1  26   10
 2•  24   12
 3•  24   -6
 4•  24   12
 5•  24  -16
 6•  25    3
 7•  25  -20
 8•  25   -1

   9-18   L

 0•  25   -4
 1•  24   -0
 2•  25  -12
 3•  25   -5
 4  31  -35
 5•  25  -14
 6•  28  -41

   9-19   L

 1•  25  -13
 2•  25  -21
 3  30  -26

  10 -1   L

 1R 40   14
 2  75  -71
 3•  23   21
 4 114 -115
 5•  23   20
 6  92  -97
 7  42   42
 8  68  -67
 9•  23    5
10  35  -33
11  27  -21
12  34  -37
13  34  -30
14  43  -44
15•  24  -15

  10 -2   L

 0  67   70
 1•  22  -21
 2  58   70
 3  65  -66
 4  63   72
 5  55  -64
 6  69   85
 7  48  -53
 8  34   36
 9  28  -33
10•  23    0
11•  23  -14

12  45  -45
13•  24  -25
14  53  -52
15•  25  -21

  10 -3   L

 1  71   73
 2•  23  -19
 3  87   91
 4•  23  -18
 5 104  109
 6•  23    8
 7  72   80
 8•  23  -11
 9•  23   18
10•  23   11
11•  24  -30
12•  24    9
13  65  -65
14•  24   11
15  59  -60

  10 -4   L

 0  37   38
 1•  22   17
 2  34   34
 3  52   55
 4  80   85
 5  54   54
 6  67   74
 7  28   33
 8•  23   29
 9•  23   12
10•  23  -10
11•  24   22
12  47  -51
13•  25   36
14  66  -66
15•  26   39

  10 -5   L

 1•  23  -17
 2  60   64
 3•  23    1
 4  86   93
 5  37   31
 6  75   78
 7•  21    3
 8•  21   19
 9•  21  -11
10•  22    5
11  37  -43
12•  22   17
13  59  -50
14  31   31
15  35  -32

  10 -6   L

 0•  20  -14
 1  46   48
 2  21  -26
 3 100  102
 4  25  -23
 5  82   65
 6  38  -29
 7•  21   26
 8  37  -38
 9•  21  -14
10  35  -38
11•  22  -16
12  25  -35
13•  22   -6
14•  22  -16
15•  23   13

  10 -7   L

 1•  20   17
 2  48   53
 3•  21   -2
 4  62   65
 5  28  -31
 6  39   40
 7  53  -50
 8•  21  -14
 9  37  -36
10  28  -31
11•  22  -16
12  29  -30
13•  22    3
14•  23  -17
15•  23    8

  10 -8   L

 0  75   81
 1  32   16
 2  56   58
 3  41   40
 4•  21   -0
 5  39   40
 6  23  -24

 7•  21   15
 8  27  -24
 9•  21  -12
10•  22    4
11•  22  -20
12•  22   26
13•  23  -24
14  34   31
15•  23  -20

  10 -9   L

 1  81   90
 2•  21   16
 3  47   50
 4•  21   18
 5R 32   -6
 6  35   42
 7•  22  -26
 8  28   33
 9•  22   -0
10  24   17
11  28   32
12•  22    1
13  43   47
14•  23   -6

  10-10   L

 0  51   61
 1  28  -12
 2  27   37
 3•  23   -5
 4•  22   -2
 5•  25   25
 6  35  -32
 7  55   59
 8•  23   -5
 9  78   74
10  35   25
11  58   55
12  37   37
13  36   32
14  46   41

  10-11   L

 1  29  -26
 2  23  -18
 3  31  -38
 4•  23    0
 5•  23  -13
 6  48   39
 7•  24  -14
 8  76   79
 9•  24   15
10  91   84
11  36   21
12  63   61
13  33   30

  10-12   L

 0  81  -86
 1•  23  -16
 2  83  -84
 3•  24  -20
 4  46  -52
 5•  23   -4
 6•  24  -23
 7•  24   31
 8•  24   -1
 9  48   47
10•  24   13
11  57   51
12•  25   14

  10-13   L

 1 116 -116
 2  41  -34
 3  84  -86
 4  35  -35
 5  30  -26
 6  27  -28
 7•  24   -2
 8•  24   -7
 9•  24    5
10•  24    7
11•  24    7
12•  25    7

  10-14   L

 0  86  -94
 1•  24  -12
 2  70  -74
 3  30  -34
 4•  25  -28
 5  45  -45
 6•  24   20
 7  40  -42
 8  31   18
 9  33  -36
10•  25   13
11  27  -28

  10-15   L

 1  33  -35
 2•  24  -12
 3•  24   -4
 4•  24  -23
 5•  25   27
 6  44  -44
 7  35   40
 8  46  -51
 9•  25   25

  10-16   L

 0•  24    7
 1•  24   -1
 2•  24   10
 3•  24    3
 4  26   28
 5•  25   -9
 6  38   41
 7  34  -36
 8  26   28

  10-17   L

 1•  24   18
 2•  24   12
 3•  25   20
 4•  25   13
 5•  25   24
 6•  25   -6

  10-18   L

 0•  24   10
 1•  24    4
 2•  25    9
 3•  25   19

  11 -1   L

 1  84   84
 2  46  -42
 3  45   43
 4  56  -51
 5•  23  -11
 6  34  -38
 7  35  -28
 8•  23  -16
 9  42  -36
10•  23  -10
11•  23  -13
12•  23  -18
13•  24  -13

  11 -2   L

 0  95   98
 1•  23  -16
 2  82   79
 3  40  -40
 4  55   51
 5  37  -42
 6•  23   23
 7•  23   -4
 8•  23  -10
 9•  23   21
10•  23   -8
11•  24   12
12•  24  -19
13•  24  -14
14•  24  -20

  11 -3   L

 1  65   73
 2•  23    0
 3  66   69
 4•  23   -5
 5  63   72
 6•  23   13
 7  28   37
 8  47   53
 9•  24    4
10  68   71
11•  24  -12
12  34   35
13•  25  -27
14•  24   -2

  11 -4   L

 0•  23   32
 1•  23    8
 2•  23   21
 3•  23   20
 4  56   59
 5  35   35
 6  47   57
 7  61   73
 8•  24   20
 9  87   94
10•  24   -0
11  70   73

12•  25  -25
13  34   42
14  25  -29

  11 -5   L

 1  57  -59
 2•  24   23
 3•  23  -17
 4  30   35
 5•  21   21
 6  69   71
 7•  22   17
 8  76   72
 9•  22   13
10  79   73
11•  23  -17
12  49   46
13  29  -25
14  25   30

  11 -6   L

 0 124 -127
 1•  21    3
 2  84  -90
 3  33   35
 4  30  -39
 5  58   64
 6•  22  -11
 7  46   49
 8•  21    7
 9  35   33
10•  22   -5
11•  22   14
12•  22  -16
13•  23   11

  11 -7   L

 1  91  -90
 2  32   32
 3  61  -66
 4  55   57
 5  39  -39
 6  26   27
 7R 25   -1
 8•  21   -0
 9•  22    9
10•  22  -18
11•  22    3
12•  23  -19
13•  23    3

  11 -8   L

 0  38  -42
 1•  21   13
 2  27  -27
 3  43   49
 4  31  -27
 5  24   22
 6•  21   -2
 7•  22  -11
 8  29   15
 9  29  -36
10•  23   23
11  37  -43
12•  23   24
13  28  -31

  11 -9   L

 1•  21   18
 2  35   41
 3•  21    0
 4  23   30
 5•  21  -11
 6•  22   -6
 7•  22    4
 8  30  -33
 9•  23   21
10  43  -42
11  33   35
12  27  -31

  11-10   L

 0  36   28
 1  24   15
 2•  23   21
 3•  23   17
 4•  23   -2
 5•  23    0
 6•  23  -23
 7•  23   -5
 8•  24   -2
 9•  24   -7
10  33   27
11•  24   -6
12  47   47

  11-11   L

 1•  23    8
 2•  23   -5

 3•  23  -10
 4R 24  -11
 5  30  -30
 6•  24    8
 7  29  -33
 8•  24   10
 9•  24    7
10•  24   22
11  30   33

  11-12   L

 0•  23  -18
 1•  23  -14
 2•  24  -27
 3  41  -39
 4  41  -40
 5•  24  -29
 6  46  -45
 7•  24   -2
 8  26  -28
 9•  24   17
10•  25    6
11  30   22

  11-13   L

 1  40  -36
 2  53  -48
 3  42  -47
 4  66  -66
 5  41  -36
 6  41  -46
 7  30  -34
 8•  24  -10
 9•  25  -14
10•  24    2

  11-14   L

 0  25  -30
 1  33  -23
 2•  24  -25
 3  58  -61
 4•  25  -12
 5  70  -71
 6•  25    1
 7  43  -47
 8•  25   -7
 9•  25  -25

  11-15   L

 1•  24   -6
 2  31  -32
 3•  25   18
 4  50  -51
 5•  26   29
 6  46  -51
 7•  26   24

  11-16   L

 0•  24   19
 1•  24   -7
 2  25   32
 3•  25  -17
 4  44   48
 5•  25  -20

  11-17   L

 1  33   29

  12 -1   L

 1  40   34
 2  27   19
 3•  23    2
 4  30   22
 5  33  -29
 6•  23   17
 7  36  -34
 8•  23    2
 9  25  -23
10•  24  -11
11•  24   -4

  12 -2   L

 0  64   63
 1•  23    4
 2  43   41
 3•  23   -2
 4•  23    2
 5•  23    7
 6•  25  -16
 7•  24   18
 8•  24  -13
 9•  24   11
10•  24   -0
11•  24    5

  12 -3   L

 1  50   47

 2•  23  -16
 3  37   34
 4•  23   -9
 5•  23    5
 6•  24   30
 7•  24    7
 8  45   46
 9•  24    1
10  43   48
11  25   21
12•  26   20

  12 -4   L

 0•  24   -3
 1•  23   -7
 2•  24    9
 3•  24  -14
 4•  24    7
 5•  24   15
 6•  24   16
 7  51   58
 8•  24    9
 9  72   74
10•  25   17
11  57   62
12  32   25

  12 -5   L

 1  60  -61
 2•  24  -11
 3  35  -35
 4•  24   -7
 5•  22  -13
 6  34   37
 7•  22   11
 8  64   63
 9•  23   12
10  72   63
11•  23   14
12  47   45

  12 -6   L

 0 105 -107
 1•  22   -3
 2  94  -98
 3•  22   -6
 4  51  -54
 5•  22   -1
 6•  22  -13
 7•  22   15
 8•  22   14
 9•  23   32
10•  23    5
11  31   27

  12 -7   L

 1 105 -113
 2•  22  -13
 3  77  -81
 4•  22  -17
 5•  22  -22
 6  30  -23
 7•  22    9
 8•  22  -22
 9•  23   21
10•  23   -9
11•  23   -3

  12 -8   L

 0  72  -73
 1•  22   -5
 2  59  -63
 3•  22  -14
 4•  22  -22
 5•  22  -27
 6  25   26
 7  48  -49
 8  34   36
 9  60  -55
10•  24   13
11  30  -36

  12 -9   L

 1  29  -24
 2•  21    6
 3•  22    0
 4•  22   -9
 5  24    3
 6•  23  -31
 7  41   38
 8  63  -63
 9  30   25
10  63  -64

  12-10   L

 0•  22   18
 1•  23   11
 2  26   23
 3•  23   12
```

Figure 14. (Continued)

```
 4  26   25
 5• 24   -2
 6  30   25
 7  35  -33
 8  25   18
 9  59  -54
10• 25   13

 12-11   L

 1  34   34
 2• 24   20
 3  26   23
 4• 24   10
 5• 24   10
 6• 24    0
 7• 24   -2
 8  28  -22
 9• 25   -4

 12-12   L

 0  35   34
 1• 23    7
```

```
2• 24   19
3• 24   10
4• 24   -7
5• 24    4
6  25  -32
7• 25   -6
8• 25  -31

12-13   L

1  24   26
2• 24   -8
3• 24   -9
4• 24  -13
5• 25  -29
6• 25  -11
7  47  -49

12-14   L

0  34   36
1• 24  -12
2• 24   16
```

```
3• 25  -32
4• 25   -8
5  27  -32

12-15   L

1  37   36
2• 25  -29
3• 25   22

13 -1   L

1  60  -45
2  31   29
3  36  -29
4  39   33
5  32  -26
6• 24   25
7• 24  -16
8• 24  -14

13 -2   L
```

```
0• 24   -9
1• 24   13
2• 24   -7
3  25   20
4  31  -20
5• 24   15
6• 24  -28
7• 24   -5
8• 24   -1
9  29  -34

13 -3   L

1• 24   27
2• 24   11
3• 24    4
4• 24   13
5• 24  -21
6• 24    1
7• 24   -6
8• 25  -10
9• 25   12
```

```
13 -4   L

0  37   40
1• 24    3
2• 24   17
3• 24    0
4• 25  -10
5• 24   -5
6• 24  -11
7• 24    5
8• 25   -4
9  27   10

13 -5   L

1• 24   -6
2• 24  -13
3  28  -18
4  27  -28
5  35  -23
6• 22   -4
7• 23  -18
8• 23   10
```

```
9• 23    2

13 -6   L

0  43  -48
1• 22  -17
2  44  -47
3  42  -39
4  28  -30
5  30  -29
6• 23  -26
7• 22   -6
8• 23  -19
9• 23   11

13 -7   L

1  51  -51
2  37  -36
3  35  -36
4  49  -52
5• 23  -14
6  41  -35
```

```
7• 23  -18
8• 23  -23

13 -8   L

0  25  -27
1  26  -22
2• 22  -21
3  45  -48
4• 23    2
5  52  -55
6• 23   12
7  47  -50
8• 23    5

13 -9   L

1• 22   10
2  23  -19
3• 23   17
4  27  -34
5  39   39
6  44  -49
```

```
7• 24   30

13-10   L

1• 24    1
2  43   38
3• 24    2
4  51   46
5• 25  -14
6  46   44
7• 25  -23

13-11   L

1  32   34
2• 24   20
3  49   44
4  28   25
5  36   36
6• 25   14

13-12   L
```

```
0• 24   21
1• 24   13
2  30   26
3  38   36
4• 25   18

13-13   L

1• 24   18

14 -1   L

1  72  -67
2• 25    1
3  48  -44

14 -2   L

0  31  -35
1• 25    2
2  26  -30
3• 25    7
```

```
14 -3   L

1• 24    9
2• 25    9
3• 25    3
4• 25    4

14 -4   L

0  35   43
1• 25    9
2  27   30
3• 25    8
4• 25   15

14 -5   L

1  41   39
2• 25    3
3• 25   19
4• 25   -8

14 -6   L
```

```

0  34   26
1• 22   -4
2• 22   15
3• 22  -15
4• 22  -14

14 -7   L

1• 23   14
2• 23  -25
3• 23   -4
4  34  -34

14 -8   L

0• 23   21
1  30  -21
2• 23   12
3  35  -39

14 -9   L

1  29   29
```

Figure 14. (Continued)

Table 9. Analysis of thermal motion for $[(C_6H_5)_2Sn]_6 \cdot 2C_8H_{10}$ (RMS is root mean square amplitude of vibration in angstrom units)

Atom 1	Atom 2	RMS(1)	RMS(2)
The RMS component of thermal displacement of two atoms in the direction of a vector which they define			
Sn+1	Sn+2	0.20	0.20
Sn+1	Sn-3	0.21	0.21
Sn+2	Sn+3	0.21	0.21
Sn+1	C1-3	0.21	0.23
C1-3	C2-3	0.24	0.22
C2-3	C3-3	0.22	0.24
C3-3	C4-3	0.28	0.25
C4-3	C5-3	0.25	0.29
C5-3	C6-3	0.25	0.26
C6-3	C1-3	0.22	0.22
Sn+1	C1-4	0.21	0.21
C1-4	C2-4	0.23	0.25
C2-4	C3-4	0.23	0.24
C3-4	C4-4	0.30	0.32
C4-4	C5-4	0.31	0.28
C5-4	C6-4	0.26	0.22
C6-4	C1-4	0.29	0.23
Sn+2	C1-1	0.21	0.22

Table 9. (Continued)

Atom 1	Atom 2	RMS(1)	RMS(2)
C1-1	C2-1	0.21	0.22
C2-1	C3-1	0.26	0.21
C3-1	C4-1	0.29	0.26
C4-1	C5-1	0.30	0.29
C5-1	C6-1	0.24	0.28
C6-1	C1-1	0.26	0.23
Sn+2	C1-2	0.22	0.19
C1-2	C2-2	0.20	0.24
C2-2	C3-2	0.25	0.23
C3-2	C4-2	0.28	0.25
C4-2	C5-2	0.25	0.28
C5-2	C6-2	0.25	0.22
C6-2	C1-2	0.23	0.18
Sn+3	C1-5	0.21	0.20
C1-5	C2-5	0.21	0.27
C2-5	C3-5	0.23	0.29
C3-5	C4-5	0.34	0.37
C4-5	C5-5	0.37	0.38
C5-5	C6-5	0.28	0.21
C6-5	C1-5	0.25	0.23
Sn+3	C1-6	0.21	0.24
C1-6	C2-6	0.18	0.28

Table 9. (Continued)

Atom 1	Atom 2	RMS(1)	RMS(2)
C2-6	C3-6	0.22	0.24
C3-6	C4-6	0.34	0.32
C4-6	C5-6	0.35	0.34
C5-6	C6-6	0.30	0.23
C6-6	C1-6	0.31	0.21
C1-7	C2-7	0.39	0.37
C2-7	C3-7	0.28	0.38
C3-7	C4-7	0.33	0.33
C4-7	C5-7	0.34	0.38
C5-7	C6-7	0.46	0.47
C6-7	C1-7		
C1-7	C7-7	0.33	0.35
C3-7	C8-7	0.33	0.37

Atom 1	RMS(1)	Atom 1	RMS(1)
RMS amplitude of vibration of atom 1 normal to the plane of the six membered carbon ring of which it is a part or to which it is attached[a]			
Sn+1 (Ring 3)	0.21	C1-3	0.17
C2-3	0.25	C3-3	0.27
C4-3	0.25	C5-3	0.25

---

[a] In the direction of the vector defined by C2-7 and C6-7.

Table 9. (Continued)

Atom 1	RMS(1)	Atom 1	RMS(1)
C6-3	0.28	Sn+1 (Ring 4)	0.21
C1-4	0.22	C2-4	0.21
C3-4	0.26	C4-4	0.29
C5-4	0.28	C6-4	0.23
Sn+2 (Ring 1)	0.21	C1-1	0.20
C2-1	0.31	C3-1	0.35
C4-1	0.33	C5-1	0.33
C6-1	0.27	Sn+2 (Ring 2)	0.21
C1-2	0.22	C2-2	0.25
C3-2	0.29	C4-2	0.30
C5-2	0.26	C6-2	0.23
Sn+3 (Ring 5)	0.21	C1-5	0.21
C2-5	0.26	C3-5	0.23
C4-5	0.24	C5-5	0.24
C6-5	0.29	Sn+3 (Ring 6)	0.21
C1-6	0.20	C2-6	0.35
C3-6	0.33	C4-6	0.29
C5-6	0.30	C6-6	0.21
C1-7	0.25	C2-7	0.26
C3-7	0.23	C4-7	0.26
C5-7	0.30	C6-7	0.23
C7-7	0.39	C8-7	0.46

Table 9. (Continued)

Atom 1	RMS(1)	Atom 1	RMS(1)
RMS amplitude of vibration of atom 1 in a direction defined by C2-C6 of the ring of which it is a part or to which it is attached			
Sn+1 (Ring 3)	0.20	C1-3	0.23
C2-3	0.22	C3-3	0.27
C4-3	0.25	C5-3	0.30
C6-3	0.29	Sn+1 (Ring 4)	0.21
C1-4	0.23	C2-4	0.27
C3-4	0.28	C4-4	0.34
C5-4	0.27	C6-4	0.32
Sn+2 (Ring 1)	0.21	C1-1	0.22
C2-1	0.25	C3-1	0.31
C4-1	0.27	C5-1	0.28
C6-1	0.29	Sn+1 (Ring 2)	0.21
C1-2	0.19	C2-2	0.24
C3-2	0.29	C4-2	0.26
C5-2	0.28	C6-2	0.27
Sn+3 (Ring 5)	0.21	C1-5	0.23
C2-5	0.29	C3-5	0.33
C4-5	0.39	C5-5	0.35
C6-5	0.26	C7-7[a]	0.49
C7-8[b]	0.57		

[b] In the direction of the vector defined by C2-1 and C4-7.

Table 10. Least squares plane for the six membered carbon ring in $[(C_6H_5)_2Sn]_6 \cdot 2C_8H_{10}$ (The deviation from the plane of the six carbons defining it are given below the equation. The plane is computed for a Cartesian coordinate system.[a])

Atom	Deviation
Ring 1	
$-0.599X + 0.7171Y + 0.3547Z + 0.8227 = 0$	
Sn+2[b]	-0.11
C1-1	-0.01
C2-1	-0.02
C3-1	0.02
C4-1	0.00
C5-1	-0.02
C6-1	0.03
Ring 2	
$-0.6516X - 0.3010Y + 0.6964 - 1.12 = 0$	
Sn+1[b]	0.01
C1-2	0.00
C2-2	0.00
C3-2	0.01
C4-2	-0.02

---

[a] The Y and Z axes are coincident with b_o and c* and X is in the a_ob_o plane perpendicular to b_o and c*.

[b] Atom not used in defining the plane.

Table 10. (Continued)

Atom	Deviation
C5-2	0.02
C6-2	-0.01
Ring 3	
$0.8988X + 0.1976Y = 0.3912Z + 2.43 = 0$	
Sn+1[b]	0.12
C1-3	0.02
C2-3	0.00
C3-3	-0.02
C4-3	0.02
C5-3	0.01
C6-3	-0.03
Ring 4	
$0.6978X - 0.6031Y + 0.7137Z - 0.4962 = 0$	
Sn+1[b]	-0.03
C1-4	0.00
C2-4	0.02
C3-4	-0.02
C4-4	0.00
C5-4	0.02
C6-4	-0.02

Table 10. (Continued)

Atom	Deviation
Ring 5	
$0.7091X - 0.2696Y + 0.6516Z - 2.15 = 0$	
Sn+3[b]	-0.11
C1-5	-0.03
C2-5	0.00
C3-5	0.02
C4-5	-0.01
C5-5	-0.02
C6-5	0.04
Ring 6	
$0.3136X + 0.7703Y + 0.5553Z - 1.24 = 0$	
Sn+3[b]	0.10
C1-6	0.01
C2-6	0.03
C3-6	-0.06
C4-6	0.05
C5-6	-0.01
C6-6	-0.03
Ring 7	
$-0.3993X + 0.8991Y + 0.1795Z - 8.29 = 0$	
C1-7	-0.01

Table 10. (Continued)

Atom	Deviation
C2-7	0.01
C3-7	0.01
C4-7	-0.02
C5-7	0.01
C6-7	0.01
C7-7[b]	-0.21
C8-7[b]	-0.03

atomic distances and angles and their errors. This program is compatible with the information calculated by the HYBL-SFLS2 program. This information includes the positional parameters, their standard deviations and the correlation coefficients for the off-diagonal elements of the inverted matrix. To make the program applicable to all crystal systems, the cell parameters and computed covariance matrix, COV, is orthogonalized[1] by the relations

$$X = Dx$$

$$\text{and } COV = D \text{ cov } D^t$$

---

[1] Y and Z axes are coincident with b and c* and X is in ab, the plane perpendicular to b and c*.

where D is the orthogonalization matrix,

$$D = \begin{pmatrix} a \sin\gamma & 0 & c(\cos\beta - \cos\alpha \cos\gamma)/\sin\gamma \\ c \cos\gamma & b & c \cos\alpha \\ 0 & 0 & V/(ab \sin\gamma) \end{pmatrix} .$$

In the orthogonalized system all distances can be computed by the simple equation

$$\ell = \sqrt{(\Delta X)^2 + (\Delta Y)^2 + (\Delta Z)^2} .$$

Neglecting cell parameter errors and assuming the parameters of one atom independent of those of any other atom, we have for the variance of a bond of length ℓ between atom 1 and 2 (38)

$$\sigma^2(\ell) = \sigma^2(1) + \sigma^2(2)$$

where $\sigma(1)$ and $\sigma(2)$ are the standard deviations in the direction of the bond ℓ of atoms 1 and 2, respectively. $\sigma(n)$ is given by

$$\sigma^2(n) = \sigma^2(X_n) \cos^2\alpha + \sigma^2(Y_n) \cos^2\beta + \sigma^2(Z_n) \cos^2\gamma$$

$$+ 2 \cos\alpha \cos\beta \, \mathrm{COV}(X_n, Y_n)$$

$$+ 2 \cos\alpha \cos\gamma \, \mathrm{COV}(X_n, Z_n)$$

$$+ 2 \cos\beta \cos\gamma \, \mathrm{COV}(Y_n, Z_n)$$

where α, β, and γ are the direction angles of ℓ. Let θ be the angle between the two bonds, 1-2 and 2-3, formed by the three atoms, 1, 2 and 3. The angle θ is given by

$$\theta = \cos^{-1}\left[\cos\alpha_1 \cos\alpha_3 + \cos\beta_1 \cos\beta_3 + \cos\gamma_1 \cos\gamma_3\right]$$

where α_1, β_1 and γ_1 are the direction angles of bond 1-2 and α_3, β_3 and γ_3 have similar relationships to bond 2-3. The variance of θ is given by (39, p. 331)

$$\sigma^2(\theta) = \frac{\sigma^2(1)}{\ell_{1-2}^2} + \sigma^2(2)\left[\frac{1}{\ell_{1-2}^2} - \frac{2\cos\theta}{\ell_{1-2}\ell_{2-3}} + \frac{1}{\ell_{2-3}^2}\right] + \frac{\sigma^2(3)}{\ell_{2-3}^2}$$

where $\sigma(1)$ and $\sigma(3)$ are the standard deviations of atoms 1 and 3 in the directions at right angles to ℓ_{1-2} and ℓ_{1-3}, respectively. $\sigma(2)$ is the standard deviation of atom 2 in the direction of the center of the circle passing through atoms 1, 2 and 3. The interatomic distances and angles and their errors calculated with this program are given in Table 11. The errors listed in this table do not include cell constant errors. With the possible exception of the

Table 11. Interatomic distances and angles for $[(C_6H_5)_2Sn]_6 \cdot 2C_8H_{10}$

Atom 1	Atom 2	Distance (A)	Error (A)
Sn+1	Sn+2	2.781	0.002
Sn+1	Sn-3	2.775	0.002
Sn+2	Sn+3	2.782	0.002
C1-1	Sn+2	2.10	0.01
C1-1	C2-1	1.40	0.02
C1-1	C3-1	2.47	0.02
C1-1	C4-1	2.87	0.02
C1-1	C5-1	2.50	0.02
C1-1	C6-1	1.45	0.03
C2-1	C3-1	1.47	0.02
C2-1	C4-1	2.47	0.03
C2-1	C5-1	2.85	0.03
C2-1	C6-1	2.43	0.03
C3-1	C4-1	1.37	0.03
C3-1	C5-1	2.42	0.03
C3-1	C6-1	2.78	0.03
C4-1	C5-1	1.42	0.03
C4-1	C6-1	2.42	0.03
C5-1	C6-1	1.39	0.03
C1-2	Sn+2	2.14	0.01
C1-2	C2-2	1.40	0.02

Table 11. (Continued)

Atom 1	Atom 2	Distance (A)	Error (A)
C1-2	C3-2	2.43	0.02
C1-2	C4-2	2.78	0.02
C1-2	C5-2	2.49	0.02
C1-2	C6-2	1.41	0.02
C2-2	C3-2	1.41	0.02
C2-2	C4-2	2.39	0.02
C2-2	C5-2	2.83	0.02
C2-2	C6-2	2.43	0.02
C3-2	C4-2	1.37	0.03
C3-2	C5-2	2.41	0.03
C3-2	C6-2	2.80	0.02
C4-2	C5-2	1.36	0.02
C4-2	C6-2	2.42	0.02
C5-2	C6-2	1.47	0.02
C1-3	Sn+1	2.15	0.02
C1-3	C2-3	1.37	0.02
C1-3	C3-3	2.41	0.02
C1-3	C4-3	2.79	0.02
C1-3	C5-3	2.42	0.02
C1-3	C6-3	1.43	0.02
C2-3	C3-3	1.43	0.02
C2-3	C4-3	2.44	0.02

Table 11. (Continued)

Atom 1	Atom 2	Distance (A)	Error (A)
C2-3	C5-3	2.80	0.02
C2-3	C6-3	2.46	0.02
C3-3	C4-3	1.38	0.03
C3-3	C5-3	2.40	0.03
C3-3	C6-3	2.83	0.03
C4-3	C5-3	1.40	0.02
C4-3	C6-3	2.46	0.03
C5-3	C6-3	1.42	0.03
C1-4	Sn+1	2.19	0.02
C1-4	C2-4	1.43	0.02
C1-4	C3-4	2.41	0.02
C1-4	C4-4	2.78	0.02
C1-4	C5-4	2.44	0.02
C1-4	C6-4	1.40	0.03
C2-4	C3-4	1.43	0.03
C2-4	C4-4	2.50	0.03
C2-4	C5-4	2.88	0.03
C2-4	C6-4	2.51	0.03
C3-4	C4-4	1.44	0.03
C3-4	C5-4	2.42	0.03
C3-4	C6-4	2.83	0.03
C4-4	C5-4	1.33	0.03

Table 11. (Continued)

Atom 1	Atom 2	Distance (A)	Error (A)
C4-4	C6-4	2.43	0.03
C5-4	C6-4	1.46	0.03
C1-5	Sn+3	2.16	0.01
C1-5	C2-5	1.43	0.03
C1-5	C3-5	2.46	0.02
C1-5	C4-5	2.77	0.02
C1-5	C5-5	2.40	0.02
C1-5	C6-5	1.34	0.02
C2-5	C3-5	1.43	0.03
C2-5	C4-5	2.45	0.03
C2-5	C5-5	2.86	0.03
C2-5	C6-5	2.41	0.03
C3-5	C4-5	1.44	0.03
C3-5	C5-5	2.51	0.03
C3-5	C6-5	2.81	0.03
C4-5	C5-5	1.42	0.04
C4-5	C6-5	2.38	0.03
C5-5	C6-5	1.38	0.03
C1-6	Sn+3	2.14	0.02
C1-6	C2-6	1.36	0.02
C1-6	C3-6	2.43	0.02
C1-6	C4-6	2.78	0.03

Table 11. (Continued)

Atom 1	Atom 2	Distance (A)	Error (A)
C1-6	C5-6	2.41	0.03
C1-6	C6-6	1.42	0.03
C2-6	C3-6	1.41	0.03
C2-6	C4-6	2.37	0.03
C2-6	C5-6	2.72	0.03
C2-6	C6-6	2.39	0.03
C3-6	C4-6	1.38	0.03
C3-6	C5-6	2.38	0.03
C3-6	C6-6	2.80	0.03
C4-6	C5-6	1.36	0.03
C4-6	C6-6	2.40	0.03
C5-6	C6-6	1.38	0.03
C1-7	C2-7	1.30	0.03
C1-7	C3-7	2.36	0.04
C1-7	C4-7	2.69	0.03
C1-7	C5-7	2.43	0.04
C1-7	C6-7	1.42	0.04
C1-7	C7-7	1.58	0.04
C2-7	C3-7	1.39	0.04
C2-7	C4-7	2.35	0.03
C2-7	C5-7	2.73	0.03
C2-7	C6-7	2.42	0.04

Table 11. (Continued)

Atom 1	Atom 2	Distance (A)	Error (A)
C2-7	C7-7	2.54	0.04
C2-7	C8-7	2.44	0.05
C3-7	C4-7	1.38	0.03
C3-7	C5-7	2.36	0.03
C3-7	C6-7	2.87	0.04
C3-7	C8-7	1.42	0.04
C4-7	C5-7	1.26	0.04
C4-7	C6-7	2.45	0.04
C4-7	C8-7	2.49	0.04
C5-7	C6-7	1.55	0.05
C6-7	C7-7	2.48	0.05

Atom 1	Atom 2 (Apex)	Atom 3	Angle (°)	Error (°)
Sn+2	Sn+1	Sn-3	113.91	0.05
Sn+2	Sn+1	Cl-3	106.3	0.4
Sn+2	Sn+1	Cl-4	112.1	0.4
Sn-3	Sn+1	Cl-3	107.4	0.4
Sn-3	Sn+1	Cl-4	109.1	0.3
Cl-3	Sn+1	Cl-4	107.7	0.6
Sn+1	Sn+2	Sn+3	109.26	0.05

Table 11. (Continued)

Atom 1	Atom 2 (Apex)	Atom 3	Angle (°)	Error (°)
Sn+1	Sn+2	C1-1	115.5	0.4
Sn+1	Sn+2	C1-2	108.6	0.4
Sn+3	Sn+2	C1-1	108.2	0.4
Sn+3	Sn+2	C1-2	110.0	0.4
C1-1	Sn+2	C1-2	105.1	0.5
Sn+2	Sn+3	Sn-1	114.22	0.05
Sn+2	Sn+3	C1-5	112.5	0.4
Sn+2	Sn+3	C1-6	112.0	0.4
Sn-1	Sn+3	C1-5	105.4	0.4
Sn-1	Sn+3	C1-6	104.6	0.3
C1-5	Sn+3	C1-6	107.5	0.6
C1-1	C2-1	C3-1	119.3	1.6
C2-1	C3-1	C4-1	121.3	1.6
C3-1	C4-1	C5-1	120.3	1.9
C4-1	C5-1	C6-1	118.4	1.7
C5-1	C6-1	C1-1	123.3	1.6
C6-1	C1-1	C2-1	117.1	1.4
Sn+2	C1-1	C2-1	121.7	1.1
Sn+2	C1-1	C6-1	121.1	1.1
Sn+2	C1-1	C4-1	176.9	0.7
C1-2	C2-2	C3-2	120.2	1.4

Table 11. (Continued)

Atom 1	Atom 2 (Apex)	Atom 3	Angle (°)	Error (°)
C2-2	C3-2	C4-2	119.2	1.5
C3-2	C4-2	C5-2	124.3	1.6
C4-2	C5-2	C6-2	117.1	1.5
C5-2	C6-2	C1-2	119.3	1.4
C6-2	C1-2	C2-2	119.7	1.2
Sn+2	C1-2	C2-2	121.6	0.9
Sn+2	C1-2	C6-2	118.7	0.9
Sn+2	C1-2	C4-2	178.9	0.5
C1-3	C2-3	C3-3	118.8	1.3
C2-3	C3-3	C4-3	120.3	1.5
C3-3	C4-3	C5-3	119.9	1.5
C4-3	C5-3	C6-3	121.9	1.6
C5-3	C6-3	C1-3	116.1	1.5
C6-3	C1-3	C2-3	112.7	1.4
Sn+1	C1-3	C2-3	118.1	1.1
Sn+1	C1-3	C6-3	119.2	1.1
Sn+1	C1-3	C4-3	177.3	0.7
C1-4	C2-4	C3-4	114.9	1.4
C3-4	C4-4	C5-4	121.8	1.6
C4-4	C5-4	C6-4	120.6	1.7
C5-4	C6-4	C1-4	116.9	1.6

Table 11. (Continued)

Atom 1	Atom 2 (Apex)	Atom 3	Angle (°)	Error (°)
C2-4	C3-4	C4-4	121.2	1.6
C6-4	C1-4	C2-4	124.6	1.4
Sn+1	C1-4	C2-4	116.5	1.0
Sn+1	C1-4	C6-4	118.9	1.0
Sn+1	C1-4	C4-4	179.1	0.7
C1-5	C2-5	C3-5	118.6	1.5
C2-5	C3-5	C4-5	117.0	1.6
C3-5	C4-5	C5-5	122.6	1.6
C4-5	C5-5	C6-5	116.1	1.6
C5-5	C6-5	C1-5	124.4	1.6
C6-5	C1-5	C2-5	120.8	1.4
Sn+1	C1-5	C2-5	119.7	1.1
Sn+3	C1-5	C6-5	119.4	1.1
Sn+3	C1-5	C4-5	177.8	0.7
C1-6	C2-6	C3-6	122.8	1.7
C2-6	C3-6	C4-6	116.7	1.7
C3-6	C4-6	C5-6	120.9	1.8
C4-6	C5-6	C6-6	121.6	1.9
C5-6	C6-6	C1-6	118.5	1.6
C6-6	C1-6	C2-6	118.3	1.4
Sn+3	C1-6	C2-6	122.5	1.1

Table 11. (Continued)

Atom 1	Atom 2 (Apex)	Atom 3	Angle (°)	Error (°)
Sn+3	C1-6	C6-6	119.2	1.0
Sn+3	C1-6	C4-6	176.7	0.6
C1-7	C2-7	C3-7	122.3	2.1
C2-7	C3-7	C4-7	115.6	2.0
C3-7	C4-7	C5-7	125.8	2.1
C4-7	C5-7	C6-7	120.7	2.0
C5-7	C6-7	C1-7	109.9	1.9
C6-7	C1-7	C2-7	125.6	2.2
C6-7	C1-7	C7-7	111.5	2.3
C2-7	C1-7	C7-7	122.7	2.1
C4-7	C1-7	C7-7	171.8	1.3
C2-7	C3-7	C8-7	119.6	2.4
C4-7	C3-7	C8-7	124.7	2.4
C6-7	C3-7	C8-7	176.5	1.3

Table 12. Final parameters and standard errors for $[(C_6H_5)_2Sn]_6 \cdot 2C_8H_{10}$ (The thermal parameters and errors are X10^5. The standard errors are in parentheses below the parameter.)

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Sn+1	.17744 (00009)	-.05816 (00006)	-.03472 (00006)	613 (8)	269 (4)	253 (3)	174 (8)	15 (10)	31 (7)
Sn+2	.17892 (00009)	.08974 (00006)	-.07412 (00006)	664 (8)	267 (4)	265 (3)	179 (9)	54 (10)	37 (7)
Sn+3	.03887 (00009)	.14828 (00006)	.01887 (00006)	654 (8)	259 (3)	255 (3)	165 (9)	17 (10)	0 (6)
C1-1	0.3393 (0012)	0.1635 (0009)	-0.0752 (0008)	594 (128)	347 (63)	244 (52)	269 (146)	-270 (141)	-65 (102)
C2-1	0.3630 (0016)	0.2285 (0010)	-0.0328 (0011)	1089 (182)	388 (73)	465 (77)	95 (189)	-105 (206)	-250 (135)
C3-1	0.4761 (0015)	0.2791 (0011)	-0.0371 (0012)	837 (164)	581 (98)	561 (87)	-286 (206)	76 (207)	-123 (159)
C4-1	0.5663 (0018)	0.2658 (0012)	-0.0847 (0011)	1307 (217)	646 (106)	431 (81)	236 (246)	419 (220)	-51 (152)
C5-1	0.5332 (0016)	0.2007 (0012)	-0.1296 (0012)	792 (174)	549 (92)	588 (93)	110 205	230 205	-112 153
C6-1	0.4293 (0016)	0.1501 (0011)	-0.1221 (0011)	1049 (198)	486 (86)	520 (85)	508 (210)	268 (208)	230 (142)

Table 12. (Continued)

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
C1-2	0.1175 (0012)	0.0900 (0008)	-0.1821 (0008)	737 (135)	195 (51)	202 (48)	113 (135)	-24 (129)	35 (84)
C2-2	0.0324 (0015)	0.0311 (0010)	-0.2087 (0009)	915 (167)	374 (73)	326 (65)	180 (177)	66 (168)	-50 (114)
C3-2	-0.0053 (0016)	0.0326 (0011)	-0.2804 (0010)	1007 (180)	564 (94)	342 (68)	334 (209)	-158 (172)	45 (130)
C4-2	0.0396 (0016)	0.0932 (0010)	-0.3230 (0009)	1064 181	434 (78)	341 (66)	161 (195)	-337 (177)	75 (121)
C5-2	0.1255 (0016)	0.1514 (0011)	-0.3024 (0010)	1091 (188)	500 (88)	319 (66)	399 (205)	74 (179)	1 (127)
C6-2	0.1645 (0015)	0.1515 (0010)	-0.2274 (0008)	1077 (174)	345 (69)	269 (57)	569 (179)	-53 (160)	78 (103)
C1-3	0.2599 (0013)	-0.0523 (0008)	0.0688 (0009)	779 (136)	215 (54)	307 (55)	395 (139)	57 (147)	86 (96)
C2-3	0.3677 (0013)	-0.0087 (0009)	0.0749 (0009)	743 (142)	323 (65)	310 (56)	92 (156)	-107 (155)	139 (107)
C3-3	0.4225 (0015)	-0.0025 (0010)	0.1437 (0010)	887 (172)	426 (80)	428 (75)	200 (188)	-303 (179)	-4 (127)
C2-3	0.3677 (0013)	-0.0087 (0009)	0.0749 (0009)	743 (142)	323 (65)	310 (56)	92 (156)	-107 (155)	139 (107)
C3-3	0.4225 (0015)	-0.0025 (0010)	0.1437 (0010)	887 (172)	426 (80)	428 (75)	200 (188)	-303 (179)	-4 (127)

Table 12. (Continued)

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
C4-3	0.3658 (0015)	-0.0375 (0010)	0.2032 (0009)	969 (176)	353 (69)	326 (63)	173 (177)	-11 (171)	-56 (109)
C5-3	0.2567 (0017)	-0.0828 (0010)	0.1957 (0010)	1437 (221)	313 (69)	388 (73)	319 (203)	-281 (203)	-17 (119)
C6-3	0.2008 (0018)	-0.0937 (0011)	0.1281 (0009)	1740 (251)	422 (81)	209 (57)	470 (230)	224 (192)	126 (113)
C1-4	0.2745 (0012)	-0.1123 (0008)	-0.1099 (0008)	615 (128)	182 (52)	306 (56)	27 (131)	5 (134)	-79 (88)
C2-4	0.3554 (0014)	-0.1502 (0009)	-0.0788 (0010)	859 (150)	289 (61)	394 (66)	212 (155)	113 (172)	-33 (113)
C3-4	0.4135 (0016)	-0.1878 (0011)	-0.1294 (0011)	1024 (183)	441 (80)	491 (81)	612 (194)	199 (194)	210 (135)
C4-4	0.3948 (0016)	-0.1832 (0010)	-0.2056 (0011)	1020 (186)	370 (75)	549 (86)	99 (193)	391 (208)	-101 (134)
C5-4	0.3207 (0017)	-0.1456 (0011)	-0.2323 (0010)	1259 (210)	506 (88)	352 (69)	419 (224)	-98 (194)	-157 (126)
C6-4	0.2525 (0016)	-0.1094 (0010)	-0.1841 (0011)	1081 (183)	397 (77)	439 (78)	206 (194)	196 (194)	-286 (130)
C1-5	0.1074 (0012)	0.1650 (0009)	0.1267 (0008)	401 (116)	382 (68)	257 (52)	25 (141)	-40 (124)	-20 (97)
C2-5	0.2014 (0017)	0.1331 (0010)	0.1466 (0010)	1385 (220)	355 (74)	322 (66)	360 (205)	-228 (191)	195 (115)

Table 12. (Continued)

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
C3-5	0.2437 (0017)	0.1435 (0010)	0.2189 (0011)	1157 (199)	356 (77)	541 (88)	-111 (203)	-314 (213)	160 (134)
C4-5	0.1868 (0019)	0.1855 (0012)	0.2670 (0010)	1354 (221)	602 (102)	384 (80)	-487 (248)	-153 (214)	96 (145)
C5-5	0.0947 (0019)	0.2176 (0012)	0.2445 (0010)	1626 (253)	473 (91)	387 (77)	-163 (251)	392 (224)	38 (137)
C6-5	0.0645 (0014)	0.2082 (0011)	0.1729 (0009)	803 (159)	528 (85)	300 (60)	243 (187)	-267 (158)	-211 (120)
C1-6	0.0106 (0011)	0.2545 (0008)	-0.0182 (0008)	475 (107)	299 (56)	248 (50)	-59 (126)	103 (126)	-6 (94)
C2-6	0.0830 (0017)	0.2997 (0011)	-0.0642 (0012)	1137 (202)	408 (79)	643 (93)	612 (207)	170 (221)	257 (145)
C3-6	0.0634 (0018)	0.3688 (0011)	-0.0903 (0012)	1293 (221)	481 (90)	533 (92)	204 (232)	25 (232)	480 (150)
C4-6	-0.2031 (0018)	0.3953 (0011)	-0.0582 (0012)	1455 (230)	386 (78)	641 (101)	542 (219)	-433 (240)	-85 (148)
C5-6	-0.0995 (0020)	0.3503 (0013)	-0.0141 (0010)	2005 (280)	670 (109)	294 (69)	934 (286)	7 (226)	-60 (147)
C6-6	-0.0856 (0017)	0.2799 (0010)	0.0073 (0009)	1377 (203)	328 (68)	350 (68)	387 (193)	-503 (185)	-108 (111)
C1-7	0.3694 (0022)	-0.0424 (0014)	0.4080 (0010)	2091 (312)	770 (121)	303 (70)	888 (309)	-130 (232)	-3 (146)

Table 12. (Continued)

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
C2-7	0.4784 (0020)	-0.0158 (0014)	0.4003 (0011)	1759 (275)	868 (128)	322 (74)	119 (302)	-162 (220)	-164 (155)
C3-7	0.5495 (0020)	-0.0575 (0014)	0.3686 (0011)	1556 (256)	720 (117)	403 (81)	296 (282)	-483 (232)	203 (164)
C4-7	0.4972 (0020)	-0.1293 (0013)	0.3460 (0012)	1534 (255)	608 (109)	456 (87)	779 (267)	133 (232)	162 (155)
C5-7	0.3933 (0025)	-0.1599 (0012)	0.3538 (0013)	2488 (370)	343 (88)	663 (112)	-491 (299)	-631 (330)	187 (160)
C6-7	0.3101 (0027)	-0.1158 (0017)	0.3885 (0012)	2497 (384)	1037 (162)	393 (88)	93 (408)	-144 (302)	610 (199)
C7-7	0.2852 (0030)	0.0079 (0020)	0.4338 (0019)	2905 (494)	1094 (195)	929 (170)	1737 (500)	654 (474)	331 (307)
C8-7	0.6669 (0031)	-0.0240 (0025)	0.3585 (0025)	1776 (404)	1353 (275)	1648 (295)	422 (560)	-493 (565)	830 (479)

tin-tin distances which are quite accurately known, the cell constant errors are small in comparison with the standard deviations listed. The final positional and thermal parameters are given in Table 12.

6. Discussion

In this structure, the configuration of the tin atoms is the main point of interest. The three asymmetric tin atoms are related to three additional equivalent tin atoms by a center of symmetry forming a six membered ring. The average tin-tin distance of 2.78 A is not unlike the tin-tin distance of 2.80 A found in the diamond form of tin (40). The tin atoms are linked to each other by normal covalent bonds. The six membered ring is in the favored chair configuration as shown in Figure 15. Also of interest is the C_{2h} symmetry exhibited by the six membered ring since the space group requires only $\bar{1}$ symmetry. The extent to which this ring approaches ideal C_{2h} symmetry is shown by the following list of bond lengths, angles and standard deviations.

2.7813 ± 0.0016 A

2.7817 ± 0.0016 A

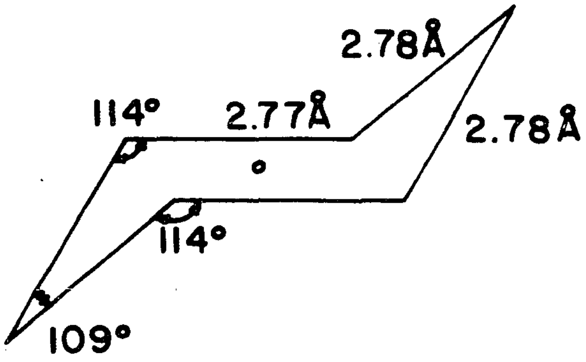
2.7748 ± 0.0014 A

109.26 ± 0.04°

114.21 ± 0.05°

113.90 ± 0.05°

Figure 15. Configuration of tin atoms in $[(C_6H_5)_2Sn]_6 \cdot 2C_8H_{10}$


2.78Å
114°
2.77Å
2.78Å
114°
109°

Since the cell parameter errors and the covariances between atoms were neglected in the standard deviation calculation, the estimated errors are not correct on an absolute basis. However it is felt that these values are significant relative to each other since cell constant errors are likely to be systematic and standard errors computed with the mentioned approximation are known to be quite good.

Two phenyl groups are attached to each tin atom at approximately tetrahedral angles. The photograph in Figure 16 shows six phenyl groups in the axial positions and six in the equatorial positions. The analysis of the thermal motion of these groups indicates a general increase in the amount of thermal vibration as the distance to the bonded tin increases. This indicates a flapping or rotation of the phenyl group about the Sn-C bond. The slight decrease in RMS amplitude of vibration in a direction normal to the ring for the C4 carbon atom with respect to C3 and C5 is evidence for this rotational motion. This difference, however, is not outside the limits of experimental error.

The relative position of the xylene molecule is shown in the projection of this structure in Figure 17. There are no tin atoms close enough to it to indicate any π bonding. The xylene molecule acts in the capacity of a solvent of crystallization.

Figure 16. Photograph of a model of the unit cell contents of $[(C_6H_5)_2Sn]_6 \cdot 2C_8H_{10}$

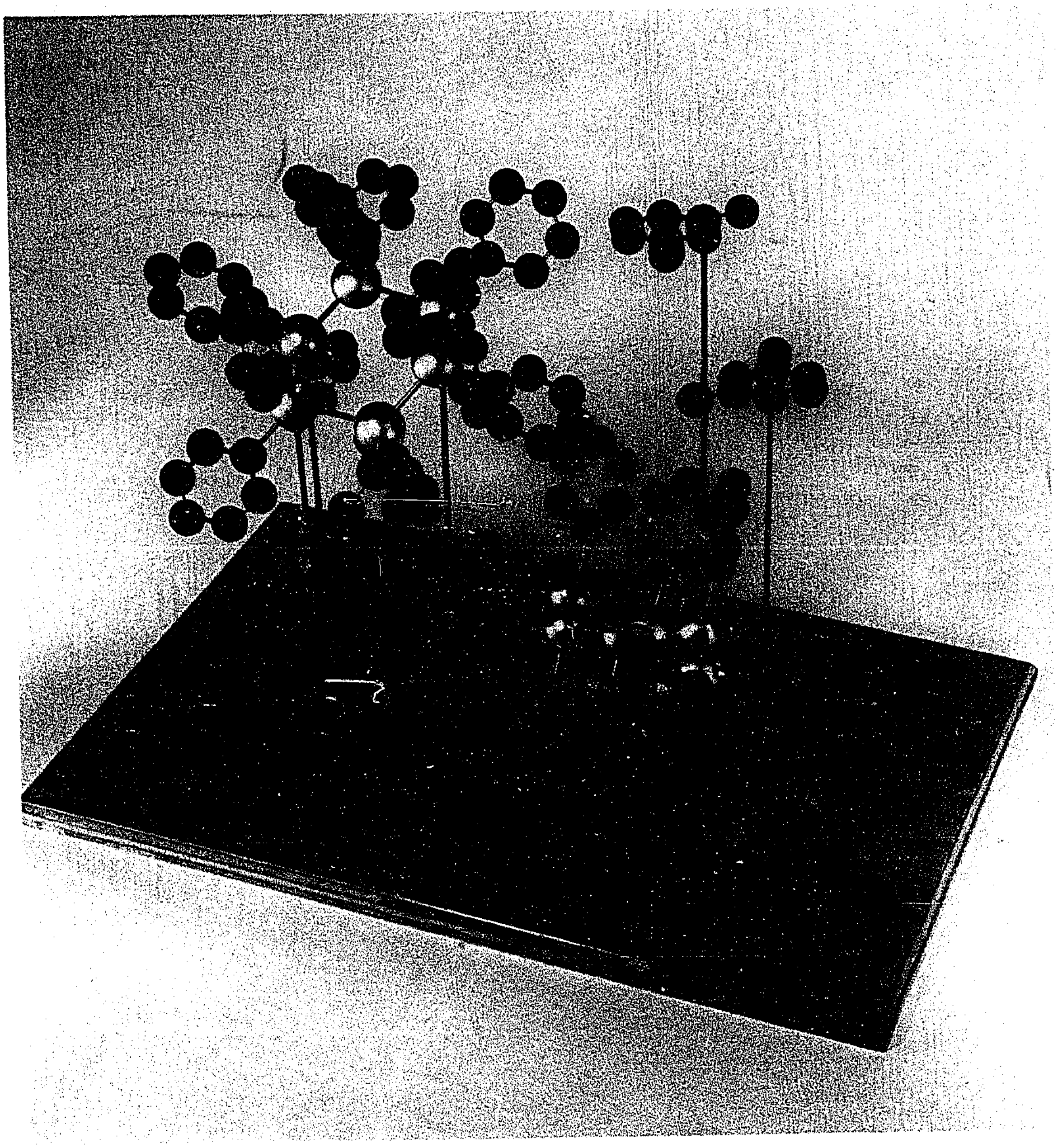
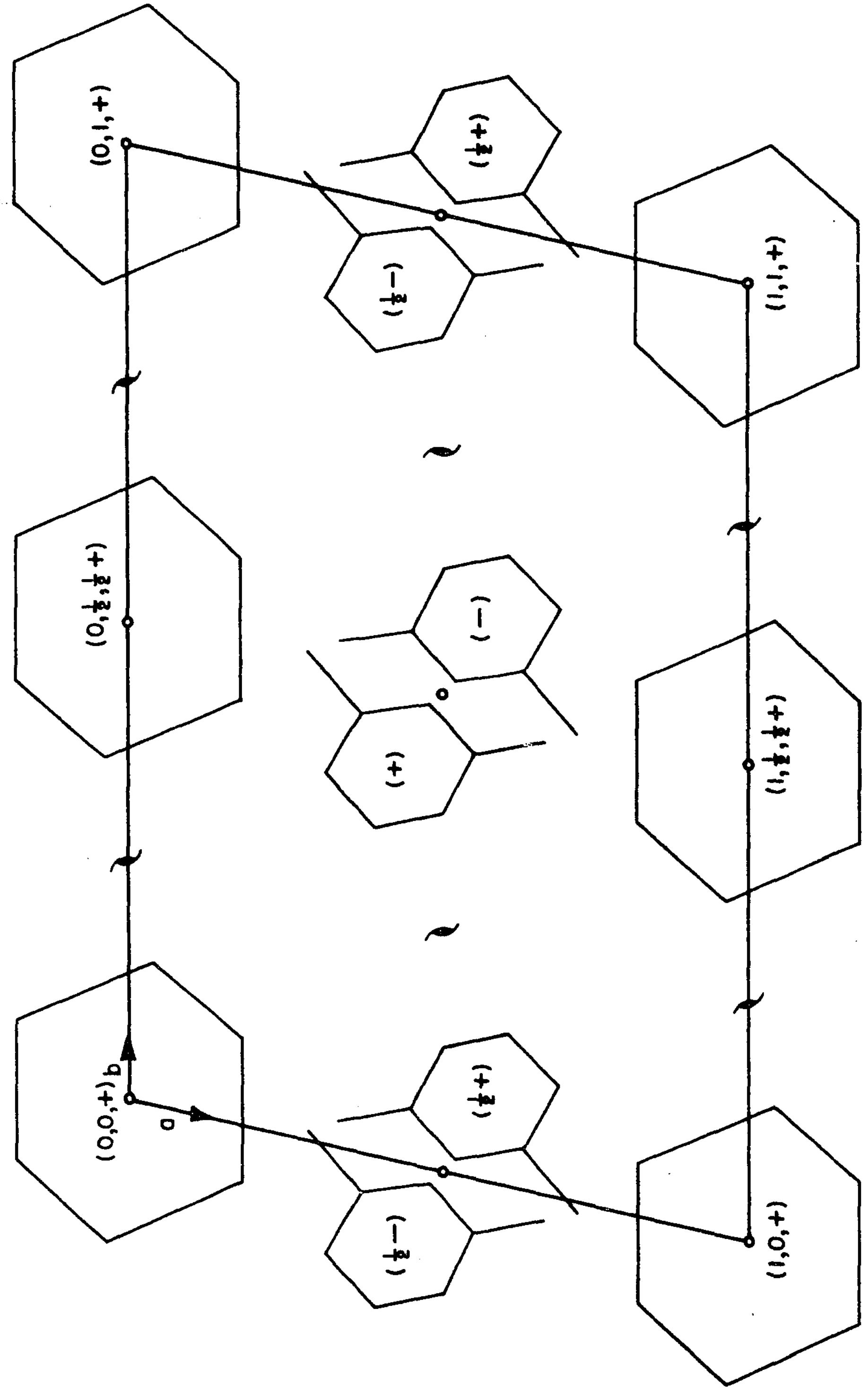


Figure 17. Projection onto (001) of $[(C_6H_5)_2Sn]_6 \cdot 2C_8H_{10}$ showing the six membered tin rings and m-xylene molecules


(0,1,+)
(½+)
(½−)
(1,1,+)
(0,½,½+)
(−)
(+)
(1,½,½+)
(0,0,+)
b
a
(½+)
(½−)
(1,0,+)

The discovery of tin-tin bonds in this structure has proven that polymerization of diphenyltin results in the formation of tin-tin bonds. In this structure, each tin atom forms two tin-tin bonds. Recently similar bonding was found in diethyltin (31) and dimethyltin (41). In addition, evidence was recently presented indicating that extensive sidebranching occurs in some of these polymers (30, 31) suggesting that tin forms up to three cationic bonds.

III. ANALYSIS OF THE PRESENT KNOWLEDGE CONCERNING TIN (II) CRYSTAL CHEMISTRY

A. Trends in Tin (II) Crystal Chemistry

1. Classification

A cursory review of the crystal chemistry of tin (II) reveals that in almost all compounds the tin atom has one of two types of coordination, three-coordinate covalent and six-coordinate covalent.[1] Conspicuously absent from the above classification are ionic compounds. With the KrO $4d^{10}$ $5s^2$ configuration, one would expect Sn^{++} to form typical ionic compounds with a symmetrical arrangement of ligands about the tin ion. There are no known compounds of this type. A recent X-ray study of SnF_2 (42), the compound most likely to form an ionic structure, has failed to reveal the fluorine positions in the presence of the heavy tin atoms. However this study has shown that the structure is complex, contrary to what is expected for a typical ionic compound. In the SnO structure, there are Sn-O distances of 2.21 A and adjacent layers of tin atoms with Sn-Sn distances of 3.70 A. Although the bonding in this compound is not clear,

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[1] Although the compounds described here have considerable ionic character, the configurations about the tin atom appear to be dictated by the covalent bond character.

this rather short Sn-Sn distance may be indicative of some degree of tin-tin bonding. (The estimated distance for van der Waals interaction is 4.4 A.) Hence this may not be a truly tin (II) compound according to the definition given originally.

2. Three-coordinate covalent tin (II)

In five of the compounds described in the literature review and in $SnCl_2$, the tin atom has three atoms in its first coordination sphere or in other words has a coordination number of three. A summary of the interatomic distances and angles of three coordinate tin is given in Table 13. In all of these cases, the tin atom sits at the apex of a trigonal pyramid with the three ligands forming the base. The average X-Sn-Y angle is 87°.[1] This is very close indeed to the theoretical angle of 90° which is predicted for the use of the 3 5p-orbitals of the tin atom. The $5s^2$ electrons then remain as a nonbonding pair. Kamenar and Grdenić contend that the angles in $K_2SnCl_4 \cdot H_2O$ (13) and $SnCl_2 \cdot 2H_2O$ (15) are really the result of deviations from the tetrahedral angle of 109° 28' and that the deviations are caused by strong lone pair - bond pair repulsions. In view of the fact that the average angle of 87° requires a 22° deviation

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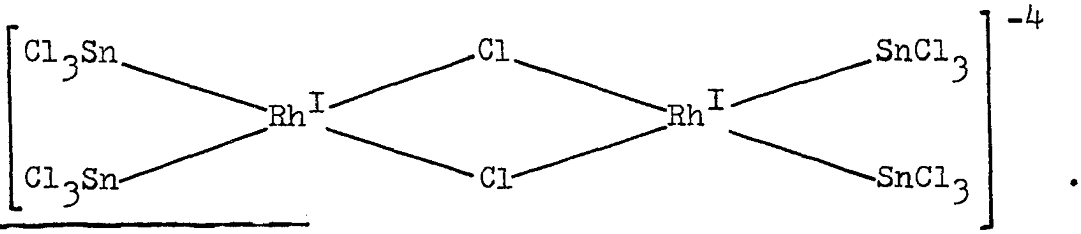
[1] The average angle not including the $SnSO_4$ data, which is probably the least accurate, is 89°.

Table 13. Interatomic distances and angles of three coordinate tin

Compound	Complex	Distance (A)			Angles (^{o})		
	A-Sn-C B	Sn-A	Sn-B	Sn-C	A-Sn-B	A-Sn-C	B-Sn-C
SnS	S-Sn-S S	2.68	2.62	2.68	88	96	88
SnSe	Se-Sn-Se Se	2.82	2.77	2.82	89	96	89
$SnCl_2$	Cl-Sn-Cl Cl	2.78	2.67	2.78	80	105	80
$SnCl_2 \cdot 2H_2O$	Cl-Sn-Cl OH_2	2.59	2.16	2.59	87	85	83
$K_2SnCl_4 \cdot H_2O$	Cl-Sn-Cl Cl	2.54	2.63	2.54	88	91	88
$SnSO_4$	O-Sn-O O	2.40	2.33	2.40	76	74	76

from tetrahedral bond angles but only 3° deviation from p-orbital bond angles, the use of tetrahedral hybrid orbitals for bonding does not seem likely. In $SnCl_2$ there is a Cl-Sn-Cl angle of 105° in the Cl-Sn chain and $SnCl_2$ is very hygroscopic indicating that the Sn-Cl bond in the chain is very weak. In $SnCl_2 \cdot 2H_2O$ where all bond angles are about 90°, the water molecule attached to the tin atom is not removed until the temperature is increased to 80° C at atmospheric pressure indicating a fairly strong $Sn\text{-}OH_2$ bond.[1] These two experimental facts indicate stronger bond formation as the angle approaches 90°.

Although to the first approximation, it appears that the tin atom is using p-orbitals for bonding in these compounds, this does not imply that tetrahedral hybridization is never energetically feasible or never occurs in divalent tin compounds. Very recently, Davies <u>et al</u>. (43) isolated and identified a tetramethyl ammonium salt containing the $[Rh_2Sn_4Cl_{14}]^{-4}$ anion. They proposed the following structure for this anion:



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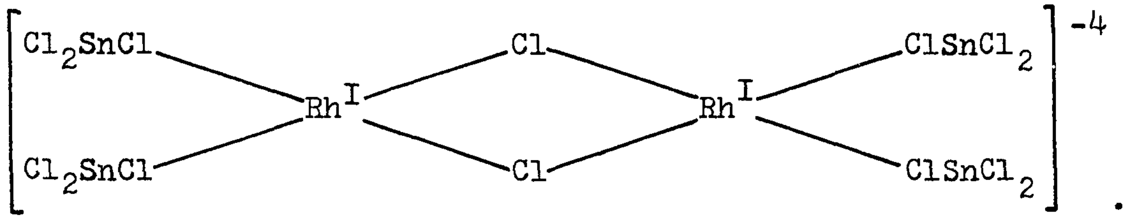
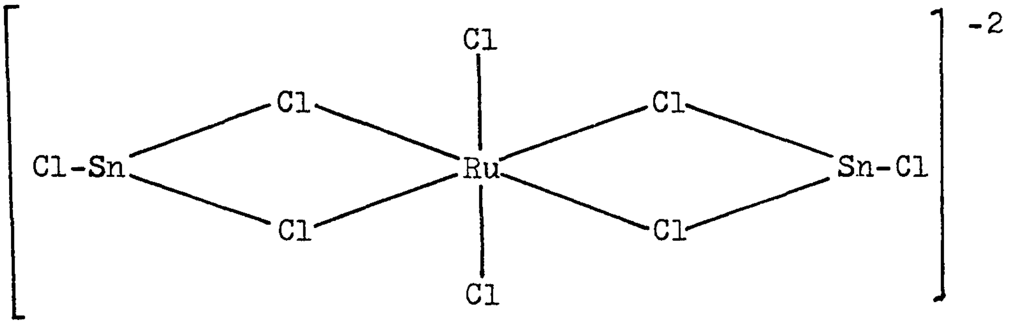
[1] Also note that in $K_2SnCl_4 \cdot H_2O$ where there is an abundance of ligand ions, the tin (II) is bonded to a chloride ion rather than a water molecule.

If correct, it verifies the tetrahedral state for divalent tin. However it seems equally likely that the structure of this anion is



This structure is supported by further evidence given by Davis et al. (43). They identified the $[Ru(SnCl_3)_2Cl_2]^{-2}$ anion which probably has the structure



rather than a structure containing Ru-Sn bonds, since Ru (II) is known to have octahedral coordination in most of its complex formations. An X-ray investigation of compounds containing these anions would be very interesting.

3. Six-coordinate covalent tin (II)

SnTe and cubic SnSe are six-coordinate and have the NaCl type structure. However these compounds must be mainly covalent, since the electronegativity of these elements are 1.8, 2.4 and 2.1 for Sn, Se and Te, respectively (7, p. 93). A bonding scheme has been proposed for these compounds by Dr. Rundle[1] which is similar to that proposed earlier for interstitial compounds (44).

Starting with Sn^{++} and Se^{+4} or Te^{+4}, there will be three empty valence p-orbitals on each atom directed along the three cube directions. In a molecular orbital scheme, the bond orbitals will be just filled by the six electrons per SnSe (or SnTe) pair. (The s-orbitals could be brought into this scheme, but bonding and anti-bonding orbitals would both be filled.) In a valence bond description, there will be resonance among the two forms

$$\text{Sn - X}\quad\text{Sn - X}\quad\text{Sn - X} \qquad\qquad \text{I}$$

$$\text{- Sn}\quad\text{X - Sn}\quad\text{X - Sn}\quad\text{X -} \qquad\qquad \text{II}$$

in all three cube directions. Such a resonating system of covalent bonds would place a formal charge of -1 on tin and

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[1] Rundle, R. E., Department of Chemistry, Iowa State University, Ames, Iowa. Bonding in six-coordinate tin (II). Private communication. 1963.

+1 on the chalconide and hence we may expect more than 33 % ionic character in each bond to redistribute the charge making the charge on tin somewhat positive in agreement with its lower electronegativity.

B. Calculation of a Covalent Radius of Tin (II)[1]

From the available structural information, a covalent radius can be derived for tin (II). In making this calculation, Sn-Cl distances will be used which appear to represent normal single bonds. In the $SnCl_2$ structure, this eliminates the Sn-Cl distance of 2.78 A found in the zig-zag chain but includes the singly bonded Sn-Cl distance of 2.67 A. This value will be weighted doubly since the values found in $SnCl_2 \cdot H_2O$ and $K_2SnCl_4 \cdot H_2O$ have higher standard deviations. The values used in this calculation are

2 x 2.67 A from $SnCl_2$
2 x 2.59 A from $SnCl_2 \cdot 2H_2O$
1 x 2.63 A from $K_2SnCl_4 \cdot H_2O$
1 x 2.54 A from $K_2SnCl_4 \cdot H_2O$.[2]

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[1] Most of the ideas presented here resulted from discussions with Dr. Rundle. Rundle, R. E., Department of Chemistry, Iowa State University, Ames, Iowa. Covalent radius for tin (II). Private communication. 1963.

[2] Used only once since the two chlorines are crystallographically equivalent.

The average Sn-Cl distance is 2.62 A. Taking the covalent radius of chlorine to be 0.99 A, the covalent radius of tin (II) is 1.63 A (7, p. 224). This value may now be tested using the remainder of the known structural data on divalent tin compounds. For Sn-O in $SnCl_2 \cdot 2H_2O$, $\Sigma r = 2.29$ _vs_. 2.16 ± 0.04 A (obsd.). Since the electron pair in this bond is "donated" by the oxygen atom, the bond must have considerable ionic character possibly shortening it. For Sn-S in SnS, $\Sigma r = 2.66$ _vs_. 2.68 (two) and 2.62 A observed. Also for Sn-Se in SnSe $\Sigma r = 2.80$ _vs_. 2.82 (two) and 2.77 A observed.

In addition, this tin (II) radius can be tested for SnTe and cubic SnSe where Sn-Se and Sn-Te bonds have bond number one-half. The calculated bond distance will be increased by 0.18 A using Pauling's rule for fractional bonds (7, p. 255). Thus for SnSe, $\Sigma r = 2.80 + 0.18 = 2.98$ _vs_. 3.00 A (obsd.) and for SnTe, $\Sigma r = 3.00 + 0.18 = 3.18$ _vs_. 3.14 A (obsd.). Although this value is only approximate, it is much larger than the accepted value of 1.40 A for tin (IV) and the value of 1.55 A calculated by Moore and Pauling for tin (II) from the SnO structure analysis (4).

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VI. ACKNOWLEDGMENTS

The author wishes to express deep appreciation to Dr. Robert E. Rundle for his continued interest in and helpful suggestions concerning this work.

The author is grateful to Dr. Albert Hybl and Donald E. Williams for many helpful discussions concerning this work and for the use of computer programs which they each wrote.

The able technical assistance of Mr. Fred Hollenbeck is greatly appreciated.

Lastly, the author is grateful to his wife, Kathy, for her assistance in preparing this manuscript.