solution reaction in accordance with previous observations.[3,4] Even after a short period of anodic attack, the anode surface may become corrugated. This process of roughening of the anode surface due to the uneven dissolution may be called "surface fragmentation."

Discussion

The explanation of the results obtained with a tin anode is possible if we assume that partial surface fragmentation occurs even at low current densities (the surface turns black) and that all of the tin particles present on the tin anode make good electrical contact (Figure 2). In such cases, when no breakdown of the extended tin protrusions occurs, the normal valency number of 2 for the Sn ions is found (*e.g.*, by Rumpel, *et al.*,[18] in sodium chloride, acetate, and tin chloride solutions and with tin amalgam in an arrangement as shown in Figure 1 where no anodic formation of particles and their dispersion in the electrolyte can occur). An apparent valency of less than 2 will be measured if some tin is lost and does not contribute to the faradaic current. Such a loss may occur at specific conditions which further the breakdown of the protrusions shown in Figure 2. For instance, oxidizing agents[18] such as sodium nitrate or potassium chlorate may produce oxide layers on the tin surface (eq 3), so that the protrusions (Figure 2) become thinner and lose their strength and elasticity at their thinnest parts and so that breakdown of the particles can easily occur, especially if intermittent current[18] is used which introduces some oscillations. The touch of the anode with even a soft brush causes breakdown of the protrusions, and, if their weight is not subtracted from the total weight of dissolved tin, the calculated valency of tin ions will be less than 2. The loss of tin particles may be further promoted in oxalate solutions.[4] Application of an external current to a cell having tin amalgam as an anode causes dispersion of the amalgam[18] which reduces the calculated valency of the tin ions.

The particle breakdown or disintegration process may be quite regular because very many particles are involved, and, thus, in a certain electrolyte at a certain current density, the same calculated lower valency of the tin ions (within the error span of several per cent) will be obtained.

Finally, the existence of tin grains with high resistivity toward the action of acids within a less resistive matrix may be explained by the differential concentration of impurities and imperfections within the metal during cooling: the purer and less imperfect areas may produce high-resistivity grains, so that anodic dissolution will nearly undermine them (see Figure 2).

Acknowledgment.—This investigation was sponsored by the Corrosion Research Council and in part by the Office of Naval Research. We thank Dr. W. J. James, Director of the Graduate Center for Materials Research at the University of Missouri, for discussion and assistance with the manuscript.

CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY,
BROOKHAVEN NATIONAL LABORATORY, UPTON, NEW YORK 11973

The Crystal Structure of Dimethyltin Difluoride. An Example of Octahedral Coordination of Tin[1]

BY E. O. SCHLEMPER[2] AND WALTER C. HAMILTON

Received January 28, 1966

As part of an investigation of possible trigonal bipyramidal or octahedral coordination of tin, the structure of $(CH_3)_2SnF_2$ has been determined. Dimethyltin difluoride forms tetragonal crystals in space group I4/mmm with $a = 4.24$ and $c = 14.16$ A and with two formula units per unit cell. The structure consists of an infinite two-dimensional network of tin atoms and bridging fluorine atoms with the methyl groups above and below the plane completing the octahedral coordination of the tin. The interatomic distances are Sn–C (2.08 ± 0.01 A) and Sn–F (2.12 ± 0.01 A). The crystal structure is the same as that of SnF_4 except that the nonbridging fluorines in SnF_4 are replaced by methyl groups in $(CH_3)_2SnF_2$.

Introduction

The physical and chemical properties of R_xSnF_{4-x} ($x = 0$ through 3) compounds are quite different from those of the corresponding bromides and chlorides. For example, the melting point of the fluoride is always much higher than that of the corresponding bromide or chloride, and the chloride and bromide are generally much more soluble in nonpolar organic solvents than is the fluoride. More specifically, $(CH_3)_2SnF_2$ decomposes without melting at ~400° while $(CH_3)_2SnBr_2$ melts at 74–76° and $(CH_3)_2SnCl_2$ at 90°.

It has been predicted[3] that dialkyltin difluorides should have octahedral coordination around tin with bridging fluorines. As a part of a study of possible trigonal bipyramidal or octahedral coordination of tin,

---

[1] Research performed under the auspices of the U. S. Atomic Energy Commission.

[2] Department of Chemistry, University of Missouri, Columbia, Mo.

[3] I. R. Beattie and T. Gilson, *J. Chem. Soc.*, 2585 (1961).

the crystal structure of dimethyltin difluoride has been determined.

Experimental Section

Crystal Preparation.—Dimethyltin difluoride was purchased from Alfa Inorganics, Inc. Single crystals were obtained by slow evaporation of a methanol solution. The crystals so obtained were colorless flat plates with fourfold external symmetry.

X-Ray Data.—Using a crystal with the *a* axis coincident with the spindle axis of a goniometer head, oscillation, Weissenberg, and precession photographs were taken with Mo Kα radiation. The crystals were tetragonal with $a = 4.24 \pm 0.01$ and $c = 14.16 \pm 0.02$ A. A crude flotation measurement in a CCl_4–CBr_4 solution gave a density of 2.42 ± 0.01 g/cc. A calculated density of 2.43 g/cc is obtained if one assumes two formula units per unit cell; the structural analysis confirmed this assumption. The systematic extinctions were for *hkl*, $h + k + l$ odd, indicating that the probable space group is I422, I4mm, I$\bar{4}$2m, I$\bar{4}$m2, or I4/mmm. A reasonable model could be obtained in I4/mmm by placing the atoms in the following positions.[4]

Sn in (2a): (0, 0, 0) + body centering
F in (4c): $(0, {}^1/_2, 0;\ {}^1/_2, 0, 0)$ + body centering
C in (4e): $(0, 0, z;\ 0, 0, \bar{z})$ + body centering

This model was confirmed during the refinement procedure.[5]

Although the only positional parameter with the above structure assumption is the *z* coordinate of the carbon, a complete set of data was collected to confirm the structure and to get accurate thermal parameters and an accurate Sn–C distance. Using Mo Kα radiation, multiple-film Weissenberg data were collected for the *0kl*–*5kl* layers. The crystal fragment was roughly a rectangular parallelepiped with dimensions along *a*, *b*, and *c* of 0.3, 0.2, and 0.07 mm, respectively. There were 379 reflections of measurable intensity in the octant used. These included 243 independent reflections. All intensities were estimated by visual comparison to a series of timed exposures of a selected reflection. The usual Lorentz and polarization corrections were made. Absorption corrections were made[6] for a rectangular parallelepiped of the dimensions given above (μ 40.4 cm^{-1}). The Weissenberg layers were correlated using equivalent reflections occurring on more than one layer, and the average value of F_o^2 for each independent reflection was obtained.

Structure Refinement.—The structure proposed above was taken as the starting point for a least-squares refinement. The *z* coordinate of the methyl carbon was chosen to make the Sn–C distance about 2.15 A. The hydrogen atoms were omitted. The structure was refined by full-matrix least squares. Isotropic refinement[7] converged with

$$R = \sum(|F_o|^2 - |k_oF_c|^2)/\sum|F_o|^2 = 0.27$$

and

$$r = [\sum w(|F_o|^2 - |k_oF_c|^2)^2/(\sum w|F_o|^2)^2]^{1/2} = 0.39$$

where $w = (80)^{-2}$ for $F_o^2 \leq 400$ and $w = (0.20F_o^2)^{-2}$ for $F_o^2 > 400$. The numerator of *r* is minimized in the least-squares refinement. With all atoms anisotropic, the refinement converged with $R = 0.098$ and $r = 0.096$. A difference Fourier at this point had the largest residuals very near the tin atoms, and the next largest residual was a ring of electron density at the position one would expect to find density for freely rotating or disordered methyl hydrogens. Since the three hydrogen atoms are repeated around a fourfold symmetry axis, they must be either freely rotating or at least fourfold disordered. Owing to the small contribution of the hydrogens to the over-all X-ray scattering, it is impossible to distinguish between free rotation and twelve $^1/_4$ hydrogen atoms on a circle of radius 1 A. When a free-rotation model for the hydrogen atoms was included at the proper position to make the C–H distance 1.2 A, the angle Sn–C–H 109° 28′, with B(H) = 5, refinement of the parameters of the remaining atoms converged with $R = 0.095$ and $r = 0.096$. Although no improvement in *r* resulted, there was a decrease in *R* with no increase in the number of refined parameters. No attempt was made to refine the parameters describing the freely rotating hydrogen atoms.

The final parameters from the last described refinement are included in Table I. The observed and calculated squared structure factors are given in Table II. The ellipsoids of vibration are described in Table III.

TABLE I

FINAL PARAMETERS FOR $(CH_3)_2SnF_2$[a]

	x	y	z	U_{11}	U_{22}	U_{33}
Sn (2a)	0	0	0	0.0188 (3)	0.0188 (3)	0.0440 (5)
C (4e)	0	0	0.146 (1)	0.058 (5)	0.058 (5)	0.049 (6)
F (4c)	0	$^1/_2$	0	0.098 (8)	0.0136 (20)	0.067 (4)

[a] Symmetry requires that U_{12}, U_{13}, and U_{23} be zero for all three atoms. The standard deviations are from the least-squares variance–covariance matrix.

TABLE II

OBSERVED AND CALCULATED SQUARED STRUCTURE FACTORS

K	L	ØBS	CALC
H = 0			
0	4	7546	8281
0	6	12546	9912
0	8	5113	4791
0	10	2226	2000
0	12	1310	1366
0	14	965	779
0	16	274	281
0	18	165	122
0	20	113	71
0	22	38	25
0	24	21	7
1	3	4113	3720
1	5	5378	4523
1	7	4404	4088
1	9	1702	1701
1	11	967	955
1	13	793	755
1	15	332	355
1	17	158	126
1	19	92	75
1	21	59	38
1	23	20	11
2	0	7694	9842
2	2	6949	6779
2	4	5515	4816
2	6	5505	4827
2	8	2936	2920
2	10	1363	1342
2	12	905	895
2	14	531	550
2	16	200	207
2	18	105	92
2	20	61	53
2	22	27	20
3	1	2486	2597
3	3	1819	1690
3	5	1672	1672
3	7	1569	1527
3	9	798	810
3	11	487	482
3	13	399	370
3	15	206	188
3	17	81	75
3	19	47	43
3	21	37	21
4	0	1964	2238
4	2	1672	1939
4	4	1307	1465
4	6	1208	1367
4	8	926	944
4	10	484	514
4	12	378	348
4	14	211	214
4	16	104	94
4	18	54	44
4	20	42	24
5	1	637	646
5	3	453	507
5	5	388	468
5	7	381	406
5	9	272	254
5	11	171	162
5	13	126	116
5	15	82	63
5	17	50	29
5	19	24	16
6	0	482	490
6	2	428	439
6	4	344	373
6	6	340	325
6	8	264	236
6	10	164	148
6	12	107	97
6	14	78	59
H = 1			
1	2	2728	2392
1	4	1767	1523
1	6	2606	2438
1	8	1552	1575
1	10	695	681
1	12	498	574
1	14	431	411
1	16	166	153
1	18	86	72
1	20	77	47
1	22	36	18
2	1	4620	5210
2	3	3457	3031
2	5	3099	3052
2	7	2658	2683
2	9	1333	1312
2	11	749	747
2	13	526	567
2	15	250	273
2	17	106	104
2	19	69	59
2	21	42	29
3	0	1800	2041
3	2	1425	1486
3	4	1159	1147
3	6	1270	1287
3	8	999	898
3	10	488	463
3	12	359	349
3	14	249	236
3	16	105	99
3	18	48	47
3	20	35	28
3	22	24	11
4	1	1774	1727
4	3	1184	1303
4	5	1216	1185
4	7	959	1006
4	9	537	585
4	11	328	354
4	13	209	248
4	15	123	128
4	17	65	55
4	19	40	30
5	0	524	614
5	2	581	521
5	4	425	434
5	6	391	413
5	8	334	302
5	10	174	182
5	12	127	128
5	14	91	82
5	16	40	40
5	18	27	20
6	1	428	432
6	3	347	365
6	5	321	320
6	7	260	260
6	9	185	171
6	11	121	110
6	13	93	72
6	15	44	39
H = 2			
2	0	6054	5238
2	2	4924	3940
2	4	3254	2971
2	6	3099	2884
2	8	1930	1876
2	10	911	924
2	12	641	630
2	14	336	385
2	16	126	156
2	18	83	70
2	20	50	40
3	1	1972	1947
3	3	1358	1394
3	5	1457	1302
3	7	1165	1147
3	9	609	652
3	11	356	389
3	13	307	289
3	15	144	149
3	17	63	62
3	19	35	34
3	21	29	17
4	0	1517	1507
4	2	1320	1264
4	4	1011	1028
4	6	945	944
4	8	630	660
4	10	387	379
4	12	207	254
4	14	134	158
4	16	67	71
4	18	46	34
5	1	524	528
5	3	411	428
5	5	396	386
5	7	336	328
5	9	215	211
5	11	141	135
5	13	102	93
5	15	68	52
5	17	39	24
6	0	400	356
6	2	309	322
6	4	277	275
6	6	261	237
6	8	162	174
6	10	115	112
H = 3			
3	0	1281	1170
3	2	1009	946
3	4	716	766
3	6	746	762
3	8	543	544
3	10	293	307
3	12	169	220
3	14	143	143
3	16	70	65
3	18	43	32
3	20	30	18
4	1	957	839
4	3	767	668
4	5	546	602
4	7	487	509
4	9	304	317
4	11	167	198
4	13	121	136
4	15	67	73
4	17	35	34
5	0	434	395
5	2	363	349
5	4	283	296
5	6	264	267
5	8	167	198
5	10	134	124
5	12	100	85
5	14	57	53
6	1	250	236
6	3	188	204
6	5	163	178
6	7	143	145
6	9	96	99
6	11	82	64
H = 4			
4	0	611	566
4	2	562	500
4	4	458	420
4	6	335	373
4	8	247	272
4	10	141	168
4	12	108	112
4	14	65	69
4	16	54	34
5	1	342	268
5	3	253	228
5	5	194	201
5	7	164	165
5	9	113	112
5	11	94	73
5	13	50	48
6	0	199	154
6	2	174	143
6	4	134	124
6	6	115	105
H = 5			
5	0	213	155
5	2	179	143
5	4	152	124
5	6	125	107
5	8	75	80

Discussion

Thermal Motion.—As might be expected, the thermal motion of the tin atom is greatest perpendicular to the plane of tin and fluorine atoms. The thermal motion of the carbon atom is nearly isotropic but is a minimum along the tin–carbon bond. The thermal motion of

---

(4) "International Tables for X-Ray Crystallography," Vol. 1, Kynoch Press, Birmingham, England, 1952.

(5) A structure based on an octahedral model satisfies the symmetry of all of the space groups listed. Under these circumstances, unless there is some evidence to the contrary, the usual practice is to describe the structure in terms of the space group with the most symmetry.

(6) Programs for the 7094 used in this work, in addition to those of local origin, were local modifications of Burnham's GNABS absorption program, A. Zalkin's FORDAP program, W. R. Busing and H. A. Levy's ORFLS least-squares and ORFFE error-function programs, and C. K. Johnson's ORTEP ellipsoid-plot program.

(7) F_o is on the absolute scale (in electrons).

TABLE III

PARAMETERS FOR THE ELLIPSOIDS OF VIBRATION

	Rms amplitude, A	Direction
Sn	0.137 ± 0.002	Along *a*
	0.137 ± 0.002	Along *b*
	0.210 ± 0.001	Along *c*
C	0.22 ± 0.01	Along *c*
	0.24 ± 0.01	Along *a*
	0.24 ± 0.01	Along *b*
F	0.12 ± 0.01	Along *b*
	0.26 ± 0.01	Along *c*
	0.31 ± 0.01	Along *a*

the fluorine atom is highly anisotropic; its minimum amplitude is along the tin–fluorine bond while its maximum is perpendicular to the bond and in the plane of the tin and fluorine atoms. Along the tin–fluorine bonds, the thermal vibrations of the tin and the fluorine atoms are, within experimental error, equal so that, in this plane, the tin–fluorine framework probably vibrates as a unit with the fluorine atoms vibrating much more violently perpendicular to the tin–fluorine bonds. The shapes of the ellipsoids can be seen in Figure 1.[8]

Nature of the Structure.—The tin atoms are in an octahedral environment with four equatorial bridging fluorine atoms and with methyl groups above and below the tin to complete the octahedron (see Figure 1). An interesting fact is that $(CH_3)_2SnF_2$ and SnF_4 (Figure 2[8])[9] both have the same crystal structure. Both crystallize in space group I4/mmm with two formula units in the unit cell. The unit cell dimensions however reflect the substitution of the two methyl groups for two fluorine atoms. SnF_4 has $a = 4.05 \pm 0.01$ and $c = 7.93 \pm 0.01$ A while $(CH_3)_2SnF_2$ has $a = 4.24 \pm 0.01$ and $c = 14.16 \pm 0.02$ A. Both cell constants are longer in $(CH_3)_2SnF_2$, but the elongation of the *c* axis is most dramatic. The *c* translation corresponds to twice the separation of layers of tin and fluorine atoms (see Figures 1 and 2). The layers are able to pack much more closely together in SnF_4 owing to the fact that fluorine has a smaller van der Waals radius than the methyl group. In SnF_4 this results in near coplanarity of the two planes of nonbridging fluorine atoms between the layers of tin and bridging fluorine atoms. However, the two planes of carbon atoms between layers of tin and fluorine atoms in $(CH_3)_2SnF_2$ come nowhere near coplanarity, being separated by 2.95 A along the *c* axis. The carbons from one layer to the next have a total separation of 4.20 A compared to the commonly accepted van der Waals distance[10] of 4.00 A.

The octahedral coordination in $(CH_3)_2SnF_2$ and SnF_4 should be compared with the trigonal bipyramidal coordination in $(CH_3)_3SnF$[11] and in $(CH_3)_3SnCN$,[12] which each have only one bridging group.

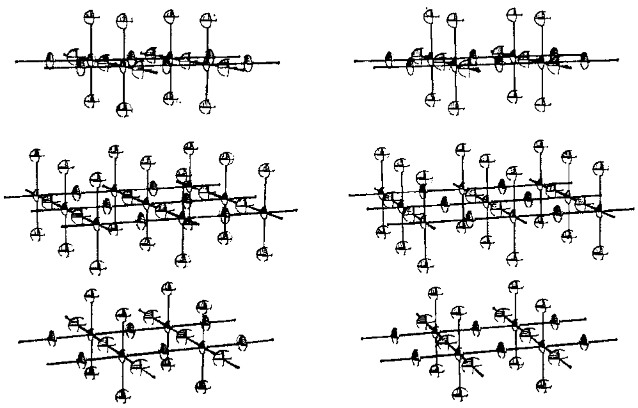

Figure 1.—The crystal structure of $(CH_3)_2SnF_2$. The two views constitute a stereoscopic pair and can be viewed with a small hand stereoscope. The ellipsoid boundaries are at the 50% probability level. The square-planar network is composed of Sn atoms at the square corners and F atoms at the centers of the square edges to give an infinite sheet with formula $(SnF_2)_\infty$. The methyl groups are the vertical appendages.

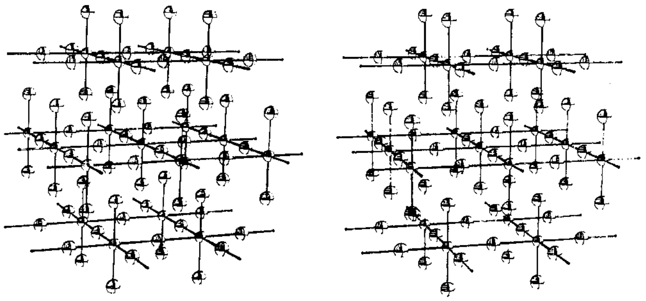

Figure 2.—The crystal structure of SnF_4. The two views constitute a stereoscopic pair and can be viewed with a small hand stereoscope. The atoms are drawn as spheres of arbitrary radius. The Sn atoms are at the square corners; the other atoms are F atoms.

TABLE IV

BOND DISTANCES FOR $(CH_3)_2SnF_2$ AND RELATED COMPOUNDS

Distance	Compd	Value	Ref
Sn–C	$(CH_3)_2SnF_2$	2.06 ± 0.01	*a*
	$(CH_3)_2SnF_2$	2.08 ± 0.01[b]	
Sn–C(CH$_3$)	$(CH_3)_3SnCN$	2.16 ± 0.03	*c*
Sn–C	$(CH_3)_ySnX_{4-y}$ X = I, Br, or Cl *y* = 1–3	2.18 ± 0.03	*d*
Sn–C	CH_3SnH_3	2.143	*d*
Sn–F (bridged)	$(CH_3)_2SnF_2$	2.12 ± 0.01	*a*
Sn–F	SnF_4	1.88 ± 0.01	*e*
Sn–F (bridged)	SnF_4	2.02 ± 0.01	*e*
Sn–F	$K_2Sn_2F_5$	2.08	*f*
Sn–F (bridged)	$K_2Sn_2F_5$	2.22	*f*

[a] This work. [b] Corrected for thermal motion by the method described by W. R. Busing and H. A. Levy, *Acta Cryst.*, **17**, 142 (1964). The carbon atom is assumed to "ride" on the tin atom. [c] E. O. Schlemper and D. Britton, *Inorg. Chem.*, **5**, 507 (1966). [d] L. E. Sutton, Ed., "Interatomic Distances," The Chemical Society, London, 1958. [e] R. Hoppe and W. Dähne, *Naturwissenschaften*, **49**, 254 (1962). [f] R. R. McDonald, A. C. Larson, and D. T. Cromer, *Acta Cryst.*, **17**, 1104 (1964).

Bond Distances.—The bond distances in $(CH_3)_2SnF_2$ are compared with corresponding distances in related compounds in Table IV. To our knowledge, the observed Sn–C distance of 2.08 A is the shortest

---

(8) Figures 1 and 2 each constitute stereoscopic pairs and can be viewed with a small hand stereoscope.

(9) R. Hoppe and W. Dähne, *Naturwissenschaften*, **49**, 254 (1962).

(10) L. Pauling, "The Nature of the Chemical Bond," 3rd ed, Cornell University Press, Ithaca, N. Y., 1960, p 260.

(11) H. C. Clark, R. J. O'Brien, and J. Trotter, *J. Chem. Soc.*, 2332 (1964).

(12) E. O. Schlemper and D. Britton, *Inorg. Chem.*, **5**, 507 (1966).

yet reported and the only Sn–C distance reported for an octahedrally coordinated tin atom. The sum of the covalent radii[13] for Sn and C is 2.17 A which agrees quite well with the average observed distance (2.16 ± 0.02 A) for the other compounds given in Table IV. The Sn–C distance (2.16 A) in the trigonal bipyramidal $(CH_3)_3SnCN$ is within experimental error equal to that (2.14–2.18 A) observed for the tetrahedral tin compounds which constitute the remainder of the examples in Table IV. Although it is possible that the change in the nature of the hybrid orbitals on Sn may cause the octahedral Sn–C bond length to be different from the tetrahedral and trigonal bipyramidal bond lengths, the short bond length observed here for Sn–C is probably due to the highly ionic character of the $(SnF_2)_\infty$ sheet. The resulting positive charge on Sn leads to increased ionic character in the Sn–C bond and thus to a shorter bond length.

It is interesting that the bridging Sn–F distance is 0.1 A shorter in SnF_4 than in $(CH_3)_2SnF_2$. It seems reasonable to describe the structures in the first approximation as $Sn^{4+}[F^-]_4$ and $(CH_3)_2Sn^{2+}[F^{-1}]_2$. Using the univalent crystal radii of Pauling,[14] one can estimate that the crystal radius of Sn^{4+} ought to be about 0.12 A shorter than that of Sn^{2+}. This agrees well with the difference found.

A detailed discussion of the bond lengths and bonding will be deferred until the results of structural studies of $(C_6H_5)_2SnF_2$ and another dialkyltin difluoride are available.

[13] See ref 10, pp 224–229.

[14] See ref 10, p 514.

CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY,
UNIVERSITY OF BRITISH COLUMBIA, VANCOUVER, BRITISH COLUMBIA, CANADA

The Infrared Spectra and Structure of Some Trimethylantimony Derivatives, $(CH_3)_3SbX_2$

BY H. C. CLARK[1] AND R. G. GOEL

Received November 17, 1965

The compounds $(CH_3)_3SbX_2$ where X^- (or X^{2-}) is F^-, NO_3^-, CO_3^{2-}, SO_4^{2-}, CrO_4^{2-}, and $C_2O_4^{2-}$ have been prepared, and their infrared spectra in the 200–4000 cm^{-1} region have been observed. The latter show that, in the anhydrous compounds, free anions are not present, and comparisons are best drawn with the spectra of related covalent species. For example, the infrared spectrum of trimethylantimony carbonate is very similar to that of dimethyl carbonate. Attempts to prepare $(CH_3)_3Sb(BF_4)_2$ and $(CH_3)_3SbSiF_6$ gave impure products containing trimethylantimony difluoride, and BF_3 and SiF_4 were liberated. The evidence thus indicates that strong coordinative interactions occur between $(CH_3)_3Sb$ and X in all these compounds, leading to nonionic, five-coordinate structures, and in some cases to breakdown of the anionic group.

Introduction

Trialkylantimony(V) derivatives, R_3SbX_2, have been investigated by several workers who have established the widespread occurrence of the trigonal bipyramidal structure. Wells[2] showed that trimethylantimony dichloride, dibromide, and diiodide are isomorphous and have a trigonal bipyramidal structure with a planar $(CH_3)_3Sb$ group and the halogens at the apices. Triphenylantimony dibromide has recently been shown to have the same structure,[3] in agreement with the conclusions reached by Jensen,[4] on the basis of dipole moment determinations. More recently, Long, Doak, and Freedman have made infrared spectroscopic studies[5] of some trimethylantimony(V) derivatives and have concluded that trimethylantimony dinitrate is an ionic solid, $[(CH_3)_3Sb]^{2+}$ $(NO_3)_2{}^{2-}$, and that in the analogous sulfate, $(CH_3)_3SbSO_4$, the sulfate groups are covalently bonded.

In view of the numerous studies of triorganotin derivatives which establish the nonionic, solid-state structures of trimethyltin fluoride,[6] trimethyltin nitrate,[7,8] trimethyltin perchlorate,[7] dimethyltin chromate,[9] and related organotin derivatives, the related question of the existence of R_3Sb^{2+} ions in the solid state is an obvious one. There is certainly no apparent reason for the structural difference between trimethylin nitrate[7,8] and trimethylantimony dinitrate, as reported by Doak, *et al.*[5] We have therefore reinvestigated the infrared spectrum of trimethylantimony dinitrate and have similarly examined a number of other trimethylantimony(V) derivatives to provide a more extensive comparison with the analogous organotin compounds.

---

[1] To whom correspondence should be addressed at the Department of Chemistry, University of Western Ontario, London, Ontario, Canada.

[2] A. F. Wells, *Z. Krist.*, **99**, 367 (1938).

[3] T. N. Polynova and M. A. Porai-Koshits, *J. Struct. Chem.* (USSR) **1**, 146 (1960).

[4] K. A. Jensen, *Z. Anorg. Allgem. Chem.*, **250**, 268 (1943).

[5] G. G. Long, G. O. Doak, and L. D. Freedman, *J. Am. Chem. Soc.*, **86**, 209 (1964).

[6] H. C. Clarke, R. J. O'Brien, and J. Trotter, *J. Chem. Soc.*, 2332 (1964).

[7] (a) H. C. Clark and R. J. O'Brien, *Inorg. Chem.*, **2**, 740 (1963); (b) R. Okawara, B. J. Hathaway, and D. E. Webster, *Proc. Chem. Soc.*, 13 (1963).

[8] H. C. Clark, R. J. O'Brien, and A. L. Pickard, *J. Organometal. Chem.*, **4**, 43 (1965).

[9] H. C. Clark and R. G. Goel, *Inorg. Chem.*, **4**, 1428 (1965).