to F_{hkl}. All refinements were on the basis of F. An empirical weighting scheme was derived by plotting ΔF *vs.* F and in final refinement $\sigma F_o = 1.20 + 0.052F_o$. The form factors used are those given in ref 8. The thermal ellipsoids found for the iridium atoms are restricted by symmetry so that the axes within the *ab* plane are equal, and the short, unique axis is parallel to *c*. The axial ratio is 0.59.

Results and Discussion

The crystal structure of $IrSi_3$ is illustrated in Figure 1. The silicon atoms are arranged in infinite planar layers perpendicular to the *c* axis, with two such layers per unit cell. These layers are of the four-connected planar net type consisting of one-third hexagons and two-thirds triangles,[9] which is well known in alloy structures, *e.g.*, $CaCu_5$.[10] In addition to having four near neighbors within the layer, each silicon atom is in contact with three iridium atoms, two on one side and one on the other side of the layer. Each iridium atom, between two silicon layers, is in contact with nine silicon atoms. Of these, six form a hexagon in the nearer layer with Ir–Si = 2.44 ± 0.05 Å while three are in a triangle in the further layer with Ir–Si = 2.49 ± 0.04 Å.

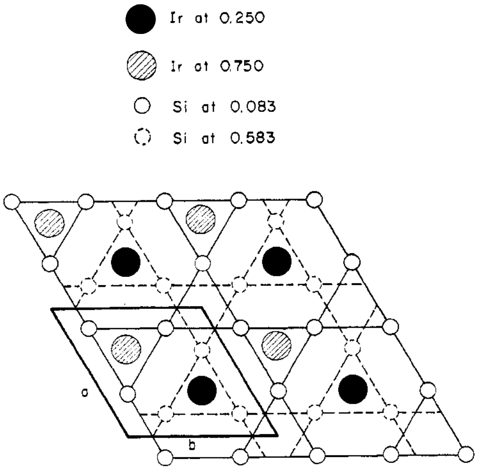

Figure 1.—Positions of the atoms in $IrSi_3$. The *z* coordinates (height above the plane of the diagram) are given in fractions of the *c* axis.

For the "ideal" value, $^5/_6$, for the silicon parameter all the close silicon–silicon distances would be equal. Taking the refined x value these distances would fall into two groups, with the distances in triangles in contact with iridium atoms being 1.98 ± 0.16 Å and the others 2.37 ± 0.16 Å. However the differences between the two sets is less than 3σ and may not be real. Regardless of the exact value of x it is clear that the Si–Si distances must average $a/2$ or 2.175 ± 0.001 Å which is considerably shorter than the elemental single-bond distance of 2.3516 ± 0.0001 Å for silicon.[11] In order to explain the higher observed bond order in $IrSi_3$ it is necessary to postulate electron transfer from the iridium atoms to the silicon layers. The 60° angles at each silicon atom would seem to favor a resonating structure giving partial double-bond character to essentially equal bonds rather than a nonresonating structure with bonds of appreciably different character.

The structure here described for $IrSi_3$ has identical iridium positions but quite different silicon positions from the structure proposed by Finnie[3] on the basis of powder data. He noted good agreement between the powder patterns of $IrSi_3$ and $HgMg_3$ (Na_3As type). However this agreement is due to domination of the X-ray intensities by the heavy atom in both cases. The three-dimensional Patterson map derived from the single-crystal data is quite incompatible with a structure similar to that of Na_3As and can only be interpreted by a structure containing planar networks of silicon atoms.

(8) "International Tables for X-Ray Crystallography," Vol. III, Kynoch Press, Birmingham, England, 1962, pp 202–204.

(9) A. F. Wells, "Structural Inorganic Chemistry," Clarendon Press, Oxford, 1962, p 103.

(10) H. Haucke, *Z. Anorg. Allg. Chem.*, **244**, 17 (1940).

(11) A. Smakula and J. Kalnajs, *Phys. Rev.*, **99**, 1737 (1955).

CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY,
UNIVERSITY OF ARKANSAS, FAYETTEVILLE, ARKANSAS 72701

A New Isomer of Diiodotetraphosphorus Trisulfide. The Preparation and Crystal Structure of β-$P_4S_3I_2$[1]

BY GARY W. HUNT AND A. W. CORDES*

Received December 29, 1970

β-$P_4S_3I_2$ is prepared by the reaction of P_4S_3 with I_2 in solution. The structure was determined from three-dimensional X-ray data collected by standard Weissenberg multiple-film techniques and refined with a data set collected by counter methods using Mo radiation. The compound crystallizes in the orthorhombic space group *Pnma* with $a = 9.399$ (2) Å, $b = 16.343$ (4) Å, $c = 6.657$ (9) Å, and four molecules per unit cell. The structure was refined with 259 observed reflections to a conventional residual of $R = 0.037$. The molecules lie on a crystallographic mirror plane, with one sulfur and two phosphorus atoms on the mirror. The P–S bond lengths range from 2.091 (16) to 2.145 (10) Å, the P–P bond length is 2.221 (10) Å, and the P–I bond length is 2.471 (8) Å. Evidence is presented to support the reaction sequence $P_4S_3 + I_2 \rightarrow \beta\text{-}P_4S_3I_2 \rightarrow \alpha\text{-}P_4S_3I_2$, where α-$P_4S_3I_2$ is a previously reported $P_4S_3I_2$ structure which has a P–S framework different from that of β-$P_4S_3I_2$.

Introduction

The phosphorus sulfides (P_4S_n, n = 3, 5, 7, 9, 10) constitute a series of molecules with cagelike structures unique in inorganic stereochemistry.[2,3] The compound $P_4S_3I_2$ is closely related to this series, and the

(1) This work was supported by the National Science Foundation and by a NASA traineeship to G. W. H.

(2) A. W. Cowley, *J. Chem. Educ.*, **41**, 530 (1964).

(3) W. Hilmer, *Acta Crystallogr., Sect. B*, **25**, 1229 (1969).

structure of $P_4S_3I_2$ bears an interesting relationship to that of the phosphorus sulfides.

Two methods were originally reported[4] for the preparation of $P_4S_3I_2$: the reaction of P_4S_3 with I_2 in solution and the direct reaction of the elements in the absence of a solvent. A difference in melting points (120.5° *vs.* 108°) and a difference in the ease of purification was reported for the products obtained from the two different reactions, but these differences seem to have been largely ignored.[2]

The cagelike structure of P_4S_3[5] is shown in Figure 1a, and the structure of $P_4S_3I_2$[6] is shown in Figure 1c (hereafter referred to as α-$P_4S_3I_2$). The crystal used for the α-$P_4S_3I_2$ structural determination was prepared by direct combination of the elements.

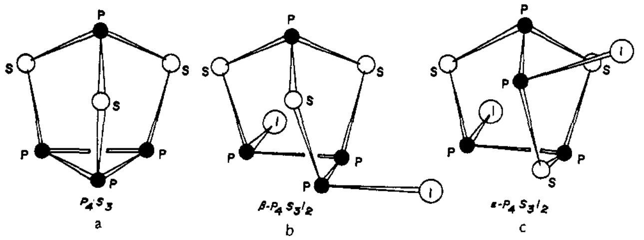

Figure 1.—The structures of (a) P_4S_3, (b) β-$P_4S_3I_2$, and (c) α-$P_4S_3I_2$.

We wish to report the isolation of a second structural isomer of $P_4S_3I_2$ (hereafter referred to as β-$P_4S_3I_2$), which is shown in Figure 1b. It appears that β-$P_4S_3I_2$ is the initial product of the reaction of P_4S_3 with I_2 in an inert solvent; thus the initial addition of the iodine does not involve a rearrangement of the P-S bonding of P_4S_3. On the other hand, we also wish to report that β-$P_4S_3I_2$ can be easily converted to α-$P_4S_3I_2$, a process which *does* involve the breaking and re-forming of P–S bonds.

Experimental Section

The preparation of β-$P_4S_3I_2$ was carried out by the direct reaction of equimolar quantities of P_4S_3 and I_2. In a typical reaction 12.7 g of I_2, dissolved in 300 ml of dry benzene, was slowly added to a stirred solution of 11 g of P_4S_3 in 50 ml of benzene under a N_2 atmosphere at room temperature. The 3-hr addition was dropwise at a rate which maintained a light orange color for the solution, which color indicated a lack of any appreciable excess of I_2. The solution was stirred for 1 hr at room temperature after all the I_2 solution had been added. The product was obtained by cooling the solution with an ice bath, followed by filtration. The light orange product was washed quickly with dry ether and dried under vacuum. The filtering and drying operations were done under a N_2 atmosphere or in a dried-atmosphere glove box. The yield is about 18 g or 76%; mp 111–112°. *Anal.* Calcd for $P_4S_3I_2$: P, 26.14; S, 20.30; I, 53.56. Found: P, 25.91; S, 20.00; I, 53.61. Suitable crystals were obtained from benzene solution by slow cooling. Since β-$P_4S_3I_2$ is sensitive to atmospheric moisture the crystal used for data collection was sealed in a 0.2-mm Lindemann glass capillary.

The approximately parallelpiped crystal used for the diffractometer data collection measured 0.031 × 0.171 × 0.186 mm (all ±0.002 mm) in the [010], [101], and [100] directions. The crystal was mounted with a^* approximately parallel to the ϕ axis. The unit cell parameters at 23° are a = 9.399 (2), b = 16.343 (4), and c = 6.657 (9) Å, as determined by a least-squares refinement[7] of the setting angles of 23 carefully centered reflections. The standard deviations reflect the variance in unit cell parameters determined for two different crystals and are roughly twice the values calculated by the least-squares program.

Reflection data were obtained using a GE quarter-circle manually operated XRD-5 diffractometer system. The Mo Kα (λ 0.71069 Å) beam was filtered with 1-mil zirconium foil. Each reflection was scanned 1.5° in 2θ at a scan rate of 2°/min with a takeoff angle of 3°. Stationary-crystal, stationary-counter background readings were taken for 10 sec at each end of the scans. All peaks with $0° \leq 2\theta < 60°$ were scanned. Only a few reflections with $2\theta > 45°$ had intensities above background. There are 702 reflections in the Mo Kα sphere bounded by $2\theta <$ 45°. We found 420 reflections had intensities greater than one standard deviation and 260 of these had intensities greater than 1.8 standard deviations. The latter reflections were used for the structure determination and refinement. The standard deviations were calculated by $\sigma_I = (S + BT^2 + 0.0016I^2)^{1/2}$ (where S is the total scan count, B is the sum of the background counts, T is the scan time per total background time, and $I = S - BT$), except for a number of reflections for which the two background readings differed by more than twice the calculated σ due to tailing of the reflection peak. For the latter reflections the standard deviations were set equal to the deviation of the background readings from the mean background value.

Since absorption by the crystal is appreciable (the linear absorption coefficient for Mo Kα is 75.19 cm^{-1}), a correction was applied to each reflection with an intensity greater than its background using the program DEAR.[8] A 4 × 10 × 10 summation was employed. The range of transmission coefficients (for I) was 0.37–0.79. No extinction corrections were made.

The crystal density, measured by flotation in an aqueous solution of K_2HgI_4, was 3.06 (2) g cm^{-3}, compared to a calculated density for four molecules per unit cell of 3.08 g cm^{-3}. The systematic absences ($0kl$, $k + l$ odd; $hk0$, h odd) imply either the noncentrosymmetric space group $Pna2_1$ ($C_{2v}{}^9$, no. 33) or the centrosymmetric space group $Pnma$ ($D_{2h}{}^{16}$, no. 62). The structure was solved and refined in the centrosymmetric space group. A least-squares refinement in the noncentrosymmetric space group did not better the fit between F_o and F_c values (as monitored by the R factors defined below) in spite of the increase (27 to 47) in the number of parameters used to describe the structure; consequently use of a noncentrosymmetric space group is not warranted. Use of the centrosymmetric space group with the restriction of four molecules per unit cell requires the molecules be positioned on inversion centers or on mirror planes.

Neutral atom scattering factors were calculated using the analytical coefficients given by Cromer and Waber[9] and real and imaginary corrections for anomalous dispersion were applied to all atoms.[10]

The structure was solved by analysis of a three-dimensional Patterson map obtained with a preliminary set of Weissenberg film data. The vector map revealed the molecular structure and the fact that the molecular centers lie on a crystallographic mirror plane, with one sulfur atom and two phosphorus atoms in the mirror. Fourier and full-matrix least-squares refinement techniques with the diffractometer data led to an unweighted residual R_1 $(\Sigma||F_o| - |F_c||/\Sigma|F_o|)$ of 0.081 and a weighted residual R_2 $(\{\Sigma w|F_o| - |F_c|)^2/\Sigma w|F_o|^2\}^{1/2}$, where $w = 1/\sigma$) of 0.080. The function minimized in the least squares was $\Sigma w(|F_o| - |F_c|)^2$. At this point all atoms had isotropic temperature factors (22 parameters total). When the iodine atom was refined with anisotropic thermal parameters R_1 = 0.039 and R_2 = 0.039 (27 parameters) and when all atoms were refined with anisotropic thermal parameters R_1 = 0.037 and R_2 = 0.035 (46 parameters).[11] Use of Hamilton's R factor ratio test[12] showed the use of anisotropic thermal parameters for the iodine was highly significant, but anisotropic thermal motion for the phosphorus

---

(4) R. D. Topsom and C. J. Wilkins, *J. Inorg. Nucl. Chem.*, **3**, 187 (1956).

(5) U. C. Lueng, J. Waser, S. van Houten, A. Vos, G. A. Weigers, and E. H. Wiebenga, *Acta Crystallogr.*, **10**, 574 (1957).

(6) D. A. Wright and B. R. Penfold, *ibid.*, **12**, 455 (1959).

(7) Program ANGSET, University of Wisconsin.

(8) A general absorption program from the University of Wisconsin which utilizes the method of W. R. Busing and H. A. Levi, *Acta Crystallogr.*, **10**, 180 (1957).

(9) D. T. Cromer and J. T. Waber, Los Alamos Scientific Laboratory, private communication.

(10) D. T. Cromer, *Acta Crystallogr.*, **18**, 17 (1965).

(11) A listing of structure factor amplitudes will appear immediately following this article in the microfilm edition of this volume of the journal. Single copies may be obtained from the Reprint Department, ACS Publications, 1155 Sixteenth St., N.W., Washington, D. C. 20036, by referring to author, title of article, volume, and page number. Remit $3.00 for photocopy or $2.00 for microfiche.

(12) W. C. Hamilton, *Acta Crystallogr.*, **18**, 502 (1965).

and sulfur atoms could not be included at a 75% level of confidence. Accordingly, the structural parameters reported here are those from the refinement which included anisotropic thermal motion for the iodine atoms only. The final distances and angles of the $P_4S_3I_2$ molecule obtained by each of the three refinements just discussed were within less than two standard deviations of the values in Table II.

A final inspection of the $\Delta F/\sigma$ values did not show any apparent dependence on θ or F values. No reflection in the least squares had a $\Delta F/\sigma$ value of greater than 1.4, and the standard deviation of a reflection of a unit weight was 0.50. The 0,18,0 reflection was omitted because of an obvious error in recording its intensity. None of the reflections whose intensities were less than 1.8σ had F_o values more than 1σ greater than F_o In the final cycle of refinement no parameter shifted more than 0.07 times its estimated standard deviation. A final difference Fourier showed no peaks greater than 0.66 e^- Å^{-3} (2.5% of the peak values for the phosphorus atoms).

Discussion

The final atomic parameters are given in Table I.

TABLE I

POSITIONAL PARAMETERS AND ISOTROPIC THERMAL PARAMETERS IN β-$P_4S_3I_2$[a]

	X	Y	Z	B, Å^2
P(1)	0.1312 (10)	0.25	0.3005 (13)	2.4 (2)
P(2)	0.2489 (9)	0.25	0.7510 (20)	3.0 (2)
P(3)	0.0152 (7)	0.1486 (5)	0.4502 (9)	2.3 (1)
S(1)	0.3251 (11)	0.25	0.4562 (14)	2.7 (2)
S(2)	0.1091 (7)	0.1485 (4)	0.7436 (13)	3.1 (1)
I	0.1476 (2)	0.0394 (1)	0.2744 (3)	

[a] Numbers in parentheses are estimated standard deviations in the least significant digit(s) of the preceding number.

The distinction betweeen phosphorus and sulfur atoms could not be made on the basis of the X-ray data. All noniodine atom positions were refined as sulfur atoms and later as phosphorus atoms, but while the temperature factors of the incorrectly labeled atoms changed in a logical manner, the changes were not large enough to constitute a basis for the assignments. There are seven possible assignments of the P and S atoms, excluding the possibility of a disordered crystal structure. The assignments made in Table I are the only ones consistent with normal covalencies and bond lengths.

TABLE II

ANISOTROPIC THERMAL PARAMETERS FOR IODINE IN β-$P_4S_3I_2$[a,b]

$10^4\beta(1,1)$	$10^4\beta(2,2)$	$10^4\beta(3,3)$	$10^4\beta(1,2)$	$10^4\beta(1,3)$	$10^4\beta(2,3)$
148 (3)	23 (1)	195 (5)	8 (2)	38 (7)	−9 (2)

[a] Numbers in parentheses are estimated standard deviations in the least significant digit. [b] The form of the anisotropic parameter is $\exp\{-[h^2\beta(1,1) + k^2\beta(2,2) + l^2\beta(3,3) + 2hk\beta(1,2) + 2hl\beta(1,3) + 2kl\beta(2,3)]\}$.

TABLE III

ROOT-MEAN-SQUARE AMPLITUDES OF VIBRATION (IN Å) OF IODINE IN β-$P_4S_3I_2$

Min	Intermed	Max
0.162	0.207	0.268

Tables II and III give the final anisotropic temperature factors and the shape of the thermal ellipsoid. The bond distances and angles are shown in Figure 2, and Figure 3 is an ORTEP drawing illustrating the thermal ellipsoids.

The Structure of β-$P_4S_3I_2$.—The molecular structure of β-$P_4S_3I_2$ is directly related to that of P_4S_3, with the only change being one basal P–P bond broken for the iodine addition. The lengths of the remaining P–P bonds in β-$P_4S_3I_2$ are not appreciably affected by the addition of iodine (2.235 (5) Å for P_4S_3 *vs.* 2.221 (9) Å for β-$P_4S_3I_2$). There is a noticeable change in P–S distances however: in P_4S_3 all P–S distances are 2.090 (5) Å, but in β-$P_4S_3I_2$ they range from 2.09 (16) and 2.097 (13) Å for the P–S bonds in the mirror plane to 2.116 (8) and 2.145 (10) Å for the other P–S lengths.[12a] The S2–P2–S2′ angle is 103.2 (5)°, indicating a spreading effect as the P–P bond is ruptured, while the remaining S2–P2–S1 bond is 101.0 (3)°, close to the 99.4 (5)° average in P_4S_3.

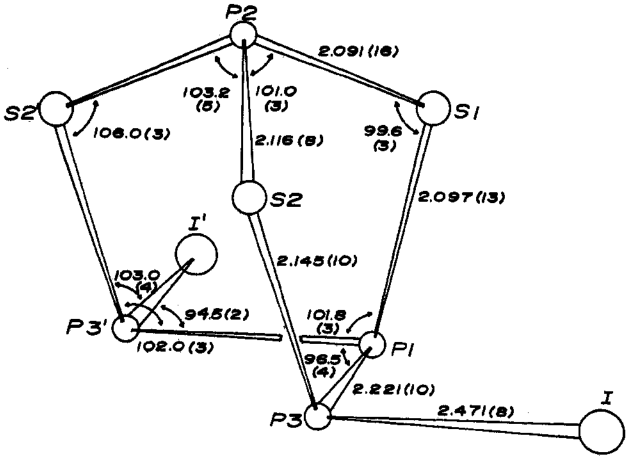

Figure 2.—The bond distances and angles in β-$P_4S_3I_2$.

Figure 3.—ORTEP stereodiagram of β-$P_4S_3I_2$ showing the orientation of the ellipsoids of thermal motion of the iodine atoms.

Comparison of α-$P_4S_3I_2$ with β-$P_4S_3I_2$ shows that, despite the difference in bonding, both forms have bond lengths and angles within the deviations reported for the α form.

Four molecules are packed in a unit cell as two pairs related by the center of symmetry. The closest approach of one nonbonded iodine atom to another is 4.058 (5) Å, which is shorter than Pauling's[13] van der Waals distance of 4.30 Å but longer than the nonbonded I–I distance of 3.90 (3) Å found by Wright and Penfold in α-$P_4S_3I_2$. Similarly, the shortest P–S nonbonded distance of 3.34 (1) Å is 0.4 Å shorter than the sum of the van der Waals radii[13] but is longer than the 3.1 (4) Å P–S distance found in α-$P_4S_3I_2$. This short distance is between the sulfur atom on the mirror and the apical phosphorus atom of the molecule nearest it. This short distance is also accompanied by an S–S distance of 3.73 (1) Å (just slightly greater than the sum of the van der Waals radii of 3.70 Å). In general, no special significance is given to the short distances noted and the molecular packing appears to be rather simple, as shown in Figure 4.

The Conversion of and Comparison of β-$P_4S_3I_2$ to α-$P_4S_3I_2$.—While the reaction of stoichiometric amounts of P_4S_3 with I_2 first gives β-$P_4S_3I_2$ with the structure

---

[12a] NOTE ADDED IN PROOF.—These differences are also seen in the less accurate structural study just reported by G. J. Penny and G. M. Sheldrick, *J. Chem. Soc.*, 1100 (1971).

[13] L. Pauling, "The Nature of the Chemical Bond," 3rd ed, Cornell University Press, Ithaca, N. Y., 1960.

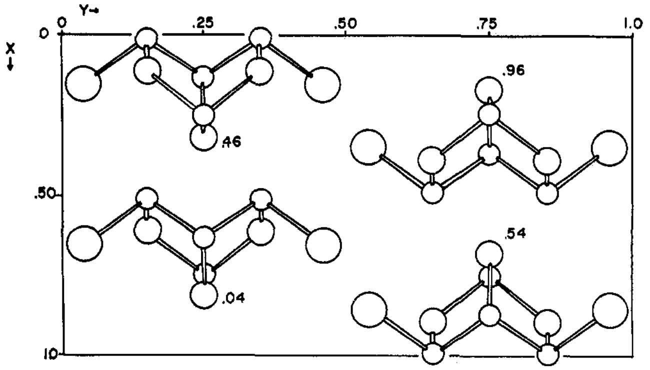

Figure 4.—The crystal packing of β-$P_4S_3I_2$ as viewed down *c*. The relative heights (*z* values) of the molecular centers are indicated.

just described, β-$P_4S_3I_2$ can be converted to α-$P_4S_3I_2$. If a benzene solution of β-$P_4S_3I_2$ is allowed to stand at room temperature for several days, crystals of the less soluble α-$P_4S_3I_2$ are slowly formed. The conversion is also carried out, with almost quantitative yields, when dry β-$P_4S_3I_2$ is held in the molten state at 122° for 10 min. The cooled product is a fused mass of α-$P_4S_3I_2$, which is soluble in warm benzene and can be obtained in a crystalline form by slow cooling of the benzene solution.

There are several means of readily differentiating between the two forms of $P_4S_3I_2$. The α form melts at 122–124° to a light orange liquid which is essentially the same color as the crystal, while the β form melts to a deep red liquid which seems to indicate some decomposition to elemental iodine. The α form usually crystallizes in a short prismatic habit, while the β form gives thin, tabular crystals frequently twinned on the large faces. Both crystals are sensitive to atmospheric moisture; the β form seems to be the most sensitive to hydrolysis. The infrared spectra (in KBr) are similar (480 (m), 460 (s), 445 (w), 410–430 (vs), 400 cm^{-1} (s)) but the β form has a broad peak at 660–700 cm^{-1} (m), a weak shoulder at 540 cm^{-1}, and an additional peak at 520 cm^{-1} (w). Initial attempts to compare the ^{31}P spectra of the two forms failed because sufficiently concentrated solutions (in CS_2, the best solvent we have found) could not be prepared.

Acknowledgment.—We are grateful to the University of Arkansas for providing computing facilities for this work.

CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY,
UNIVERSITY OF MINNESOTA, MINNEAPOLIS, MINNESOTA 55455

The Crystal Structures of Dimethyltin(IV) Bisdicyanamide and Trimethyltin(IV) Dicyanamide

BY YEH MEI CHOW

Received September 25, 1970

The crystal structures of dimethyltin(IV) bisdicyanamide, $(CH_3)_2Sn[N(CN)_2]_2$, and trimethyltin(IV) dicyanamide, $(CH_3)_3Sn[N(CN)_2]$, have been determined from three-dimensional X-ray data collected by counter methods. Dimethyltin(IV) bisdicyanamide crystallizes in the monoclinic space group $P2_1/c$ with a = 6.178 (3) Å, b = 11.265 (10) Å, c = 6.860 (6) Å, and β = 99.56 (5)°; $Z = 2$; d_{obsd} = 1.94 (3) g/cm³ and d_{calcd} = 1.98 g/cm³. Trimethyltin(IV) dicyanamide crystallizes in the orthorhombic space group *Pnam* with a = 17.644 (9) Å, b = 6.565 (4) Å, and c = 7.684 (3) Å; $Z = 4$; d_{obsd} = 1.69 (3) g/cm³ and d_{calcd} = 1.71 g/cm³. A least-squares refinement gave final conventional R values of 0.036 and 0.029 for dimethyltin(IV) bisdicyanamide and trimethyltin(IV) dicyanamide, respectively. Both structures are composed of planar $N(CN)_2$ groups symmetrically disposed on either side of the organotin groups. The average distances and angles of the $N(CN)_2$ groups in $(CH_3)_2Sn[N(CN)_2]_2$ and $(CH_3)_3Sn[N(CN)_2]$, respectively, are as follows: central C–N, 1.289 (10), 1.263 (15) Å, which are shorter than the normal C–N single covalent bond; cyano C–N, 1.132 (9), 1.116 (14) Å, which are no longer than the C–N triple bond; N–C–N, 172.3 (8), 173.2 (13)°; and C–N–C, 124.2 (8), 129.6 (11)°. The structure of dimethyltin(IV) bisdicyanamide consists of an infinite two-dimensional network of tin atoms and bridging dicyanamide groups with methyl groups above and below completing the octahedral coordination of the tin. The interatomic distances are $Sn-CH_3$ 2.091 (8) Å and Sn–N 2.289 (6) Å. The network is similar to that of dimethyltin difluoride. The crystal structure of trimethyltin(IV) dicyanamide consists of infinite chains. The tin atoms are in a trigonal-bipyramidal environment with methyl groups in the equatorial plane and dicyanamide groups in the axial positions. The interatomic distances are $Sn-CH_3$ 2.130 (11) Å and Sn–N 2.335 (9) Å. The structure is similar to that of trimethyltin cyanide.

Introduction

In most of the known crystal structures of $(CH_3)_2SnX_2$ and $(CH_3)_3SnX$ (X = halide, pseudohalide), X is a bridging ligand.[1-7] Many dicyanamides have been prepared and their infrared spectra reported,[8-10] but no crystal structures have been reported. In isolated organotin dicyanamides the tin atom conceivably may bond either to the amide part (I) or to the cyano part (II) of the ligand. From infrared spectral evidence type II structures have been suggested for dimethyltin(IV) bisdicyanamide.[10] By comparison of the infrared spectrum of trimethyltin(IV) dicyanamide to that of

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

(2) R. A. Forder and G. M. Sheldrick, *J. Organometal. Chem.*, **21**, 115 (1970).

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

(4) E. O. Schlemper and W. C. Hamilton, *ibid.*, **5**, 995 (1966).

(5) A. G. Davis, H. J. Milledge, D. C. Puxley, and P. J. Smith, *J. Chem. Soc. A*, 2862 (1970).

(6) Y. M. Chow, *Inorg. Chem.*, **9**, 794 (1970).

(7) J. H. Konnert, Ph.D. Thesis, University of Minnesota, 1967.

(8) M. Kuhn and R. Mecke, *Chem. Ber.*, **94**, 3010 (1961).

(9) H. Kohler and S. Beck, *Z. Anorg. Allg. Chem.*, **359**, 241 (1968).

(10) H. Kohler and B. Seifert, *J. Organometal. Chem.*, **12**, 253 (1968).