

& Dulac, 1955) mentioned in I. All other parts of I are correct.

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Crystallographic data for tin monophosphide. By G. KATZ, J. A. KOHN and J. D. BRODER, *U. S. Army Signal Engineering Laboratories, Fort Monmouth, N. J., U.S.A.*

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According to Hansen (1936, pp. 973-4), the phosphides of tin, SnP_3 , Sn_4P_3 , and Sn_3P_4 , may be formed in the binary system Sn-P. An attempt to prepare SnP_3 was made by the high-temperature reaction of a 1:3 mixture of tin and red phosphorus. The constituent elements were heated to 700° C. in an evacuated (10^{-6} mm. of Hg) vycor tube; afterwards slow-cooled at 40° C.hr.⁻¹ to 380° C. and then allowed to reach room temperature. The reaction product was a solidified mass with a silicon-like dull metallic luster. The presence of unreacted phosphorus indicated a deviation from the stoichiometry SnP_3 . The product was graphitic in character, i.e., quite soft with an easy cleavage parallel to (00*l*). The entire product appeared to be one large single crystal. It was found to be insoluble in concentrated HNO_3 at 300° C. For chemical analysis*, it was fused with K_2CO_3 and sulphur in a platinum crucible and then acidified. The excess sulphur was oxidized and the phosphorus was determined as phosphomolybdate. For the tin determination, the sample was acidified with HCl, reduced with aluminum foil and titrated iodometrically. The results, which are averaged from two analyses, checked closely with the stoichiometry of tin monophosphide, SnP :

| | Analysis (wt. %) | Calculated (wt. %) |
|----|---------------------|-----------------------|
| Sn | 78.86 | 79.30 |
| P | 20.47 | 20.70 |

This formulation is not in agreement with Hansen (1936, p. 974), who indicated that a monophosphide of tin appeared unlikely.

Rotation and precession patterns were obtained, using a thin cleaved section of the crystal with unfiltered Mo radiation ($\lambda = 0.7107$ Å). A hexagonal unit cell was established with the dimensions:

$$a_0 = 8.78 \pm 0.01 \text{ \AA}, \quad c_0 = 5.98 \pm 0.01 \text{ \AA}, \quad c/a = 0.682.$$

The unit-cell dimensions were determined from the spacings of high-order reflections: a_0 from the fourteenth order of (*h*0.0) and c_0 from the twelfth order of (00*l*) on zero-level precession photographs. The density of a 77 mg. sample was measured pycnometrically in methanol and found to be 5.01 g.cm.⁻³ as compared to a calculated density of 4.98 g.cm.⁻³ for $Z = 8$.

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Table 1. *Principal lines of the powder pattern of SnP*

| <i>hkl</i> | d_c (Å) | d_o (Å) | <i>I</i> |
|------------|-----------|-----------|------------|
| 00.1 | 5.98 | 5.97 | <i>vwv</i> |
| 20.0 | 3.80 | 3.77 | <i>m</i> |
| 20.1 | 3.21 | 3.19 | <i>vs</i> |
| 00.2 | 2.99 | 2.99 | <i>m-</i> |
| 21.0 | 2.87 | 2.91 | <i>vwv</i> |
| 10.2 | 2.78 | 2.82 | <i>m-</i> |
| 20.2 | 2.35 | 2.35 | <i>s</i> |
| 30.1 | 2.33 | | |
| 22.0 | 2.20 | 2.18 | <i>s</i> |
| 31.0 | 2.11 | 2.09 | <i>w</i> |
| 00.3 | 1.993 | 2.001 | <i>w</i> |
| 31.1 | 1.989 | 1.973 | <i>vw</i> |
| 40.0 | 1.901 | 1.890 | <i>w</i> |
| 40.1 | 1.812 | 1.804 | <i>m+</i> |
| 22.2 | 1.769 | 1.768 | <i>m</i> |
| 41.1 | 1.599 | 1.598 | <i>w</i> |
| 22.3 | 1.476 | 1.477 | <i>m-</i> |
| 42.0 | 1.437 | 1.433 | <i>vw</i> |
| 42.1 | 1.397 | 1.394 | <i>m+</i> |
| 20.4 | 1.391 | | |
| 42.2 | 1.295 | 1.296 | <i>w</i> |
| 60.0 | 1.267 | 1.269 | <i>vwv</i> |
| 40.4 | 1.175 | 1.177 | <i>w</i> |
| 42.3 | 1.166 | 1.166 | <i>w</i> |

In Table 1 the observed d spacings of the powder pattern are compared with those calculated from the lattice constants obtained using single crystal data. The powder pattern was obtained with Ni-filtered Cu radiation ($\lambda = 1.5418$ Å); relative intensities were estimated visually. Owing to the soft, graphitic nature of SnP , the diffraction lines for $2\theta > 90^\circ$ were broad and diffuse and not usable for a more accurate determination of the unit-cell dimensions. No systematic absences were observed. The reciprocal-lattice-level symmetry for c axis rotation was characterized by C_{6l} for the zero level and C_{3l} for the upper levels. The diffraction symmetry is $D_{3d}^2-\bar{3}m$ with the following possible space groups: D_{3d}^2-P321 ; C_{3v}^1-P3m1 ; $D_{3d}^3-P\bar{3}m1$, the last being centrosymmetric. A structure analysis is planned.

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