Evaluation of this integral leads to:

$$\overline{A} = 6f \sin 2\pi c/(2\pi c) .$$

This is the transform of a spherical shell multiplied by $6f$. The function $\overline{A}/f$ is shown in curve C, Fig. 1, and selected values are listed in column 4, Table 1.

The expression for the mean square value is:

$$\overline{A}^2 = 8f^2/\pi \int_0^{\pi/2}\int_0^{\pi/2} \{\cos(2\pi c \sin\theta \cos\varphi) + \cos(2\pi c \sin\theta \sin\varphi) + \cos(2\pi c \cos\theta)\}^2 \sin\theta d\theta d\varphi$$

or

$$\overline{A}^2/f = 8\left\{\frac{3}{4} + \frac{3}{4}\,\frac{\sin 4\pi c}{4\pi c} + \frac{\sin 2\sqrt{(2)}\pi c}{2\sqrt{(2)}\pi c} + 2\int_0^{\pi/2} \cos(2\pi c \cos\theta) J_0(2\pi c \sin\theta) \sin\theta d\theta\right\} .$$

The integral was evaluated by Simpson's rule. The function $\sqrt{(\overline{A}^2)}/f$ is shown in curve B, Fig. 1 and selected values are listed in column 3, Table 1.

Since the function A has many relative maxima and minima, the most feasible method for obtaining the absolute maximum and absolute minimum seemed to be the evaluation of A for many closely spaced values of h and k. These calculations were made and the functions $A_{\max}/f$ and $A_{\min}/f$ are shown in curves A and D, Fig. 1; selected values are listed in columns 2 and 5, Table 1.

All of the calculations for A/f, $\sqrt{(\overline{A}^2)}/f$, $A_{\max}/f$, and $A_{\min}/f$ were done on a Burrough's 220 computer. The absolute error in $\overline{A}/f$ and $\sqrt{(\overline{A}^2)}/f$ is less than 0·001; the absolute error in $A_{\max}/f$ and $A_{\min}/f$ is less than 0·01.

Wilson (1942) has shown that for a random distribution of nearly equal atoms

$$\overline{A}^2 = \Sigma f_i^2 .$$

If this relation is applied to the present structure, even though the atoms are hardly distributed in a random manner, the expected value of $\sqrt{(\overline{A}^2)}/f = \sqrt{6} = 2{\cdot}449$. From curve B, Fig. 1 and the expression for the root mean square value it is clear that the value $\sqrt{6}$ is approached quite closely, particularly for large values of c. When one applies Wilson's relation, one normally operates in discrete ranges of values of c (or $\sin\theta/\lambda$). Since the number of reflections one may expect for a given value of c is proportional to c^2, a weighted average has been computed over ranges of c using the values, ci, in column 1, Table 1 and the corresponding values $(\sqrt{(\overline{A}^2)}/f)_i$, in column 3, Table 1.

The results are given in Table 2.

Table 2. *Average value of* $\sqrt{(\overline{A}^2)}/f = \Sigma c_i^2 \sqrt{(\overline{A}^2)}/f_i/\Sigma c_i^2$.

Range of c	Average value of $\sqrt{(\overline{A}^2)}/f$
0·0–1·0	2·515
1·0–2·0	2·374
2·0–3·0	2·456
3·0–4·0	2·456
4·0–5·0	2·428
0·0–5·0	2·439

These values fall quite close to the predicted value of 2·449.

The diameter of the tantalum and niobium octahedra is 4·21 Å. For reflections out to a minimum spacing of 5 Å, c has a maximum value of 0·421. From Fig. 1 it may be seen that for work at this low resolution the orientation of the complex has little effect on its Fourier transform.

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The structure of the iodine complex of 1,4-selenothiane, $C_4H_8SSe.2I_2$.

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Structural studies of the iodine complexes of 1,4-dithiane (Chao & McCullough, 1960) and of 1,4-diselenane (Chao & McCullough, 1961) have shown that these substances have similar (nearly isomorphous) crystal structures even though their molecular structures are quite different. In the dithiane complex, the iodine molecules are bonded to sulfur atoms in the six-membered ring in equatorial positions while the bonding in the diselenane complex is axial. This immediately raises the question of the configuration of the iodine complex of 1,4-selenothiane which contains one selenium atom and one sulfur atom in the ring. Several possibilities are suggested by analogy, one axial–axial, one equatorial–equatorial and two axial–equatorial configurations. The present study was undertaken in order to answer this question.

The preparation and properties of 1,4-selenothiane and its derivatives will be described elsewhere. Crystals of the iodine complex are garnet-red needles elongated on b. Precession and Weissenberg photographs about the b axis indicated the following unit cell dimensions based on Mo $K\alpha = 0{\cdot}7107$ Å:

$$a = 6{\cdot}80 \pm 0{\cdot}03,\ b = 6{\cdot}38 \pm 0{\cdot}03,\ c = 16{\cdot}69 \pm 0{\cdot}08\ \text{Å};\ \beta = 114{\cdot}1 \pm 0{\cdot}4^\circ .$$

The measured density was found to be 3·33 g.cm.$^{-3}$ while that calculated on the basis of $2(C_4H_8SeSI_4)$ in the unit cell is 3·39 g.cm.$^{-3}$. The only systematic extinctions

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noted were those for $h0l$ with l odd and $0k0$ with k odd. The presence of only two molecules in the unit cell in $P2_1/c$ requires that these be centrosymmetric, hence it appears that the structure of the 1,4-selenothiane complex is randomly disordered. For this reason, a precise structural study was not attempted and the work was carried only far enough to establish the molecular configuration.

Comparison of the precession photographs of the $hk0$ nets of the three complexes showed a strong resemblance between the 1,4-selenothiane and the 1,4-dithiane complexes. Since the greatest differences between the structures of the iodine complexes of 1,4-dithiane and 1,4-diselenane are in the y parameters, the intensities from the $hk0$ precession photographs were considered adequate for the present objective.

Table 1. *Positional and isotropic vibrational parameters for the iodine complex of* 1,4-*selenothiane*

Atom	x	y	B
I_1	0·818	0·810	4·2
I_2	0·521	0·141	3·5
S, Se	0·221	0·477	3·5
C_1	0·234	0·585	4·0
C_2	0·972	0·291	4·0

The observed $F(hk0)$ values for the selenothiane complex were compared with sets of F_c values based on the positional parameters found in the diselenane and dithiane complexes, respectively. The selenium and sulfur positions were filled by $\frac{1}{2}$ Se $+ \frac{1}{2}$ S and the isotropic temperature factors in Table 1 were applied in these calculations. The resulting R values for the observed reflections were 50% for the diselenane (axial) structure and 20% for the dithiane (equatorial) structure. One cycle of Fourier refinement of the dithiane structure shifted the heavy atoms to the positions shown in Table 1. The carbon parameters and the isotropic temperature factors given in Table 1 were derived from the dithiane structure without further refinement. The structure factors computed on the basis of the parameters in Table 1 are compared with the observed values in Table 2. The corresponding value of R is 13%.

Even stronger evidence in favor of the dithiane structure is found in the behavior of the calculated F values of the unobserved reflections. The F values corresponding to the minimum observable intensity were never over 22. On the basis of the structure of the diselenane complex, 7 of the F values for the 20 unobserved reflections have calculated values over 40 and one, (330), has a calculated value of 105. On the other hand, 18 of the F values for unobserved reflections have calculated values less than 22 on the basis of the structure of the dithiane complex, and all have calculated values less than 24. Furthermore, all calculated F values for unobserved reflections are less than 22 when based on the 'refined' parameters.

Table 2. *Comparison of structure factors observed for* $C_4H_8SSe \cdot 2I_2$ *with those calculated on basis of the parameters in Table* 1

Unobserved reflections are indicated by * and the values given correspond to the minimum observable intensities

$hk0$	F_o	F_c	$hk0$	F_o	F_c
020	80·5	87·4 –	400	117·4	115·7
040	63·1	52·6 –	410	52·2	51·2
060	67·4	72·6	420	18·0*	18·1
080	20·0*	1·6	430	30·4	40·3 –
100	52·2	74·9 –	440	21·7	32·7 –
110	104·4	136·3 –	450	28·3	19·0
120	17·4	14·4 –	460	21·0*	19·3
130	39·1	40·9	470	26·1	22·4
140	97·9	102·4	500	37·0	26·3
150	20·0*	8·9 –	510	67·4	67·2
			520	21·0*	5·3 –
160	20·0*	15·6 –	530	21·0*	0·1 –
170	37·0	37·5 –	540	52·2	47·7
180	21·7	23·9 –	550	43·5	30·1 –
200	28·3	16·6 –	560	20·0*	3·9
210	119·6	137·7 –	600	71·8	61·9
220	32·6	30·1 –	610	52·2	48·7 –
230	17·0*	13·2	620	37·0	40·2 –
240	106·6	106·9 –	630	22·0*	11·9 –
250	28·3	24·6	640	37·0	23·8 –
260	22·0*	0·4 –	650	21·0*	15·1
270	30·4	21·1 –	700	45·7	39·6 –
300	246·5	266·2 –	710	22·0*	2·4 –
310	78·3	76·5	720	22·0*	6·5 –
320	56·5	64·7	730	21·0*	18·2
330	18·0*	13·2	740	20·0*	5·4
340	54·4	48·7	800	21·0*	3·1 –
350	21·0*	12·8 –	810	21·7	17·0 –
360	56·5	47·7 –			
370	18·0*	14·4			

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The crystal structure of VSb.

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In view of the known occurrence of the series of NiAs-type phases TiSb (Nowotny & Pesl, 1951), CrSb (Oftedal, 1927; Willis, 1953), MnSb (Oftedal, 1927, 1928; Willis, 1953) etc., it seemed very probable that an intermediate phase of the same structure occurs also at the composition VSb, although no such phase appears to have beey reported. The present work was undertaken to clarifn this question.