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* $C_4H_8SSe.2I_2$ *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 −			
170	37·0	37·5 −	530	21·0*	0·1 −
180	21·7	23·9 −	540	52·2	47·7
200	28·3	16·6 −	550	43·5	30·1 −
210	119·6	137·7 −	560	20·0*	3·9
220	32·6	30·1 −	600	71·8	61·9
230	17·0*	13·2	610	52·2	48·7 −
240	106·6	106·9 −	620	37·0	40·2 −
250	28·3	24·6	630	22·0*	11·9 −
260	22·0*	0·4 −	640	37·0	23·8 −
			650	21·0*	15·1
270	30·4	21·1 −			
300	246·5	266·2 −	700	45·7	39·6 −
310	78·3	76·5	710	22·0*	2·4 −
320	56·5	64·7	720	22·0*	6·5 −
330	18·0*	13·2	730	21·0*	18·2
340	54·4	48·7	740	20·0*	5·4
350	21·0*	12·8 −	800	21·0*	3·1 −
360	56·5	47·7 −	810	21·7	17·0 −
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.

Table 1. *X-ray diffraction pattern of* VSb

Indices	$d_{\text{calc.}}$	$d_{\text{obs.}}$	Relative line intensities Calc.	Relative line intensities Obs.
101	3·059 Å	3·045 Å	112	*vs*
002	2·723	2·729	6·3	*vw*
102	2·193	2·190	46	*s*
110	2·135	2·125	52	*s*
201	1·751	1·750	20	*mw*
112	1·681	1·684	9	*mw*
103	1·630	1·626	16	*mw*
202	1·530	1·531	14	*mw*
004	1·361	1·356	4·2	*w*
203	1·295	1·295	9	*w*
212	1·244	1·244	18	*m*
300	1·233	1·231	11	*m*
114	1·148	1·148	23	*ms*
302	1·123	1·122	4·7	*vw*
213	1·105	1·107	10	*m*
220	1·068	1·067	14	*m*
105	1·045	1·045	12	*m*
311	1·008	1·008	34	*m*

Alloy specimens near the equiatomic composition were prepared from electrolytic vanadium chips and laboratory grade antimony by arc melting under Argon atmosphere in a water cooled copper crucible. After a homogenizing anneal of 72 hours at 1000 °C. such a specimen was found by metallographic examination to consist almost entirely of a single phase. The X-ray powder pattern, taken with filtered Fe $K\alpha$ radiation, is given in Table 1. All diffraction lines observed could be satisfactorily indexed on a NiAs-type unit cell with $c_0 = 5{\cdot}447$ and $a_0 = 4{\cdot}270$ Å. The calculated relative line intensities were based on Lorentz factor, structure factor and multiplicity factor. The variation of the atomic scattering factors with $\sin \theta/\lambda$ was taken into consideration.

The observed $c/a = 1{\cdot}276$ is rather low; among the NiAs-type phases formed by each first long period transition element the antimonide generally has the lowest c/a value. Work is continuing on the physical properties of VSb.

The authors are indebted to Mr R. K. Malik for preparing some of the specimens, and to Dr W. Pearson for making available a prepublication copy of his detailed review of NiAs-type phases. The work was supported by a grant from U.S. Army Research Office (Durham).

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A new calcium tungstate: Ca_6WO_9.

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In the course of the preparation of $CaWO_4$ crystals from the flux (Uitert & Soden, 1960), a melt consisting of 13·4 g. CaO, 164·5 g. Na_2WO_4, 185·5 g. WO_3 and 11·8 g. $K_2Cr_2O_7$ contained in a platinum crucible was heated to 1250 °C., held at this temperature for five hours and cooled at a rate of 9°/hour. On leaching the resultant product with NaOH, in addition to the expected crystals of $CaWO_4$, some unusual emerald green crystals were obtained. These were generally needles up to 1 mm. in diameter and up to 5 mm. long, having hexagonal crosssection and frequently a hexagonally shaped central cavity extending the full length of the crystal. Parts of this cavity were filled with $CaWO_4$ and one end was occasionally embedded in $CaWO_4$, indicating growth before, or simultaneously with, the $CaWO_4$. Several attempted duplications were unsuccessful, indicating that this may represent a metastable phase.

Results of emission spectroscopic examination indicated only Ca and W as major detectable components as shown in Table 1. It is believed that the chromium, probably present in the trivalent state, gives the green color.

Table 1. *Results of emission spectroscopic examination*

Major components (> 10%)	Ca, W
Impurities (< 1·0%)	Cr, Si, Mg
Traces or absent (< 0·03%)	All other detectable elements

The X-ray diffraction powder pattern of the new crystal was compared with the two other known calcium tungstates; Ca_3WO_6, prepared by the method of Steward & Rooksby (1951) and Scheelite–$CaWO_4$ (Swanson *et al.*, 1956). The patterns were clearly different and single crystal oscillation and Weissenberg patterns were taken to assist with the indexing. In addition to showing the presence of twinning, the results indicate a hexagonal structure, and systematic absences show this to be rhombohedral, the probable space groups being $R\bar{3}m\text{–}D^5_{3d}$, $R3m\text{–}C^5_{3v}$ or $R32\text{–}D^7_3$. In Table 2 are listed both the hexagonal and rhombohedral lattice parameters.

Table 2. *Lattice parameters*

Rhombohedral unit cell	Hexagonal unit cell
$a = 9{\cdot}99$ Å	$a = 15{\cdot}05$ Å
$\alpha = 97°\ 47'$	$c = 14{\cdot}77$ Å
$V = 966$ Å^3	$c/a = 0{\cdot}981$

Table 3 gives the hexagonal indexing of a powder pattern taken with Cr $K\alpha$ radiation in a 114·6 mm. diameter Straumanis type Norelco camera.

In view of the small quantity of material available, the only analyses performed were those for tungsten. X-ray fluorescence spectroscopy gave 33·0% W and volumetric analysis (lead reduction and $K_2Cr_2O_7$ titration) 28·3% W. The density determined by flotation in thallium malonate–formate solution, was 3·95 g.cm.$^{-3}$, giving a molecular weight per rhombohedral unit cell of 2298. Based on a full complement of oxygen, the only formula consistent with the above results is Ca_6WO_9. This gives a calculated density of 3·99, calculated W