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

Indices	$d_{\text{calc.}}$	$d_{\text{obs.}}$	Relative line intensities Calc.	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 cross-section 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 $R\bar{3}m$–D_{3d}^5, $R3m$–C_{3v}^5 or $R32$–D_3^7. 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

Table 3. *X-ray data*

h,k,l	calc. d	obs. d	obs. int.
110	7.52	7.45	s
-	-	6.39	vw
003;202	4.92,4.89	4.87	vw
300	4.34	4.32	s
113	4.12	4.11	vvw
220	3.76	3.74	mw
104	3.55	3.56	mw
303;312	3.26,3.25	3.26	m
401	3.18	3.22	mw
223	2.99	2.99	m
214	2.95	2.96	m
410	2.84	2.84	ms
-	-	2.59	mw
330	2.51	2.50	mw
413;006;502	2.46	2.46	mw
404	2.44	2.45	vvw
116;422	2.34	2.35	w
324;511	2.32,2.31	2.33	w
333	2.23	2.23	vvw
600	2.17	2.17	vvw
306;431	2.14,2.12	2.13	vvvw
520	2.09	2.08	w
107	2.08	2.07	vw
226	2.06	2.05	vw
514	1.977	1.978	vw
523;612	1.922,1.919	1.922	vw
416	1.861	1.867	ms
434	1.853	1.856	vvw
532	1.805	1.806	vvvw
407	1.771	1.763	w
443;336	1.757	1.752	vw
710;327	1.726,1.724	1.724	vw
704	1.663	1.664	vw
541;615	1.658,1.649	1.652	vvvw
318;630;009	1.644,1.642,1.641	1.641	m
713	1.629	1.631	vw
606	1.629	1.628	vvw
119;526	1.603,1.592	1.596	vw
633;722	1.558,1.557	1.559	vvvw
544	1.521	1.522	vvw
811	1.517	1.516	vvvw
446	1.495	1.492	vvvw
428	1.477	1.479	vvvw
642	1.465	1.464	vvw
900	1.448	1.450	vvw
805	1.427	1.429	vvw
820;419	1.422,1.421	1.423	w
2,1,10	1.415	1.418	vvw
716;814	1.413,1.410	1.411	vvvw
903	1.389	1.389	vw
339;627	1.373	1.377	vw
823;636	1.366	1.369	w
618;740	1.353,1.352	1.352	vw
4,0,10;912	1.345,1.344	1.344	vvvw
645	1.334	1.334	vvvw
3,2,10;2,0,11	1.324,1.315	1.319	vvvw
743;832	1.303	1.305	vvvw
10,0,1	1.298	1.296	vvvw
556;654	1.284,1.281	1.283	vw
921	1.279	1.275	vw
3,1,11	1.259	1.258	vvw
660	1.254	1.255	vvw
5,1,10;906	1.249;1.248	1.248	vvvw
915	1.240	1.242	vvvw
10,1,0;449;817	1.237,1.237,1.236	1.234	w
841;808	1.227,1.222	1.224	vw
4,3,10	1.216	1.217	vvw
663;1,1,12	1.215	1.215	vvw
728;930	1.206,1.205	1.206	vw
10,1,3	1.200	1.201	vvw
-	-	1.195	vw
5,0,11	1.194	1.194	vw
719	1.189	1.188	w
746;3,0,12	1.185,1.184	1.184	vw

content of 32·35% and a molecular weight of 2273 per rhombohedral unit cell containing four Ca_6WO_9 molecules.

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On the crystal structure of the chloranil-hexamethylbenzene complex.*

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The crystal structure of the complex between chloranil and hexamethylbenzene—$C_6Cl_4O_2 . C_6(CH_3)_6$—was recently investigated by Harding & Wallwork (HW) (1955). They reported extreme non-planarity for the chloranil and hexamethylbenzene molecules and explained the puckering in terms of steric interactions and polarization bonding between the two molecules. Since then, Donohue & Trueblood (1956) and Nyburg (1961) have expressed surprise at this and other features of the reported structure and have suggested that, in view of the small number of observed reflections (185), the reported non-planarity might not be of significance. We have now shown that the observed intensities are indeed consistent with entirely coplanar structures for the two molecules.

We have carried out a least-squares refinement of the positional parameters, based on the observed structure factors and atomic coordinates of HW. The quantity minimized was $\Sigma w(F_o^2-F_c^2)^2$ and the weights w were taken inversely proportional to the square of the form factor of carbon.† Individual isotropic temperature parameters were adjusted only approximately, with the restriction that chemically equivalent atoms have identical parameters. In order to place the data of HW (their Table 2) on an absolute scale we found it necessary to multiply their observed structure factors by 4·25.

Our final parameters and their estimated standard

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† A number of pronounced and persistent discrepancies between calculated and observed structure factors among the weak reflections discouraged us from the usual scheme of assigning weights inversely proportional to the observed structure factors. In addition, the 100 reflection was given zero weight.