

# A Note on the Crystal Structure of Ti<sub>3</sub>P and V<sub>3</sub>P

TORSTEN LUNDSTRÖM

*Institute of Chemistry, University of Uppsala,  
Uppsala, Sweden*

A metal-rich phase in the Ti-P-system has been reported by Biltz *et al*<sup>1</sup>. In the V-P-system a metal rich phase has been prepared both by Zumbusch and Biltz<sup>2</sup> and by Chêne<sup>3</sup>. The only crystallographic constants reported, however, are the unit cell dimensions of Ti<sub>3</sub>P<sup>4</sup>. During investigations on Me<sub>3</sub>P-phases<sup>5</sup> made at this Institute it was also found desirable to include an examination of Ti<sub>3</sub>P and V<sub>3</sub>P.

Ti<sub>3</sub>P was prepared from titanium turnings (analyzed purity 99.8 % Ti, 0.1 % Fe, 0.066 % O, 0.006 % N) and red phosphorus (better than 99 % purity) as follows. Stoichiometric amounts of the reactants were sealed under vacuum in a silica capsule and simultaneously heated and shaken in a furnace, constructed by Dr A. Brown of this Institute. After heating at 800°C for 48 h no unreacted phosphorus was visible. In order to complete the preparation, the inhomogeneous product was arc-melted in a purified argon atmosphere with only an inappreciable loss of phosphorus. V<sub>3</sub>P was prepared in a similar way from 99.7 % V and red phosphorus. For the present purpose it was not considered necessary to analyze the products. Powder diffraction patterns were taken with Guinier focussing cameras using CuK $\alpha$  and CrK $\alpha$  radiation and with silicon ( $a = 5.4305 \text{ \AA}$ ).

Table 1. X-Ray powder data for V<sub>3</sub>P (CrK $\alpha$  radiation,  $\lambda = 2.2909 \text{ \AA}$ ).

<i>hkl</i>	$\sin^2\Theta_o \times 10^5$	$\sin^2\Theta_c \times 10^5$	$I_o$	$p F ^2$ for $\epsilon_1$ Ref. <sup>6</sup>
201	11754	11745	v.w.	20.4
310 } 130 } 031 } 311 } 131 }	14881	14871	w.	7.9
400 } 321 } 231 }	19185	19183	w.	0.8
112 } 330 } 202 } 420 } 240 }	20680	20670	m.	36.8
212 } 122 } 411 } 141 }	23804	23800	w.	18.1
331 } 222 } 302 } 312 } 132 }	25129	25133	v.st.	136.2
510 } 150 } 431 } 341 } 501 }	26157	26155	v.st.	20.9
511 } 402 }	26770	26775	st.	349.2
	29138	29130	m.	176.4
	29748	29750	st.	327.4
	30624	30618	v.w.	155.6
	31082	31083	v.st.	89.5
	36567	36568	w.	231.2
	35080	35080	st.	0.1
	36567	36568	w.	0.5
	38045	38055	m +	9.9
	38657	38675	m +	75.7
	42977	42983	v.w.	365.7
	44458	44470	m.	36.9
	46977	46980	m.	132.7
				16.9
				210.0
				0.0
				4.3
				148.8
				14.4
				1.7
				4.3

Table 2. Unit cell dimensions at room temperature. (Estimated accuracy  $\pm 0.05\%$ ).

Phase	a-axis (Å)	c-axis (Å)	c/a
Ti <sub>3</sub> P	9.956	4.988	0.5005
V <sub>3</sub> P	9.387	4.756	0.5067

Table 1 gives the X-ray powder data for V<sub>3</sub>P. There are a further three weak reflections which can be identified as oxide lines. The agreement of the unit cell dimensions and X-ray intensities for V<sub>3</sub>P and  $\epsilon_1$  (FeP<sub>0.37</sub>B<sub>0.63</sub>)<sup>6</sup> shows that V<sub>3</sub>P belongs to the  $\epsilon_1$  structure type, space group  $P4_2/n-C_{4h}^2$ . The presence of lines  $h + k + l = 2n + 1$  demonstrates that V<sub>3</sub>P is not isostructural with Fe<sub>3</sub>P, which has the space group  $I4^2$ .

The powder pattern and Weissenberg photographs of Ti<sub>3</sub>P show that this phase is also of the  $\epsilon_1$ -type. A single crystal investigation of Ti<sub>3</sub>P is currently being undertaken and will be published in this journal.

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1. Biltz, W., Rink, A. and Wiechmann, F. *Z. anorg. Chem.* **238** (1938) 395.
2. Zumbusch, M. and Biltz, W. *Z. anorg. Chem.* **249** (1942) 1.
3. Chêne, M. *Ann. Chim.* **15** (1941) 187.
4. Schönberg, N. *Acta Chem. Scand.* **8** (1954) 1460.
5. Rundqvist, S. *Arkiv Kemi* **20** (1962) 67.
6. Rundqvist, S. *Acta Chem. Scand.* **16** (1962) 1.

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## Constituents of the Umbelliferous Plants

### II\*. A Note on the Isolation of *O*- $\beta$ -D-Glucosyl- $\beta$ -sitosterol from the Root of *Levisticum officinale* L.

BENT EICHSTEDT NIELSEN and  
HELMER KOFOD

*Chemical Laboratory B, The Royal Danish School of Pharmacy, Copenhagen, Denmark*

Naturally occurring sterol glycosides (sterolins), were first reported in 1913 by Power and Salway<sup>1</sup>, and since compounds of this type have been isolated from a variety of plant species<sup>2-4</sup>.

In continuation of our attempts to find lignans in the plant family Umbelliferae<sup>5</sup> a sterol glycoside was isolated from the root of *Levisticum officinale* L. and identified as *O*- $\beta$ -D-glucosyl- $\beta$ -sitosterol.

The root also afforded angelic acid as well as an unidentified glucoside, m.p. 228–232°\*\*.

In the initial steps of the investigation the method described in the U.S. Pharmacopoeia XI for the preparation of podophyllin resin (*Resina podophylli*) was followed. The resin prepared in this way was fractionated according to the method of Hartwell and Detty<sup>6</sup> starting with a chloroform extraction. The alcohol-benzene solution prepared in this manner was chromatographed on alumina (Alcoa). The results are presented in Table 1.

*Fraction 7.* On evaporation this fraction left a yellow waxy solid, which upon washing with diethyl ether yielded a colourless powder. A total of 190 mg was obtained.

The product recrystallized from pyridine-ethanol yielded a colourless compound positive to the Liebermann-Burchard as well as the Molisch and Withby<sup>7</sup> tests, m.p. 283–286° (decomp.),  $[\alpha]_D^{26} = -41.5^\circ$  (*c* 0.397, pyridine).

\* Part I: *Acta Chem. Scand.* **17** (1963) 1161.

\*\* Melting points are uncorrected and determined in capillary tubes.