

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  $P4_2/n-C_{4h}^1$ . 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  $I4^1$ .

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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## 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.

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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.

Table 1. Column-chromatographic fractionation on alumina of resin from *Levisticum officinale* L.

Fraction	Eluent	ml	Substance
1	benzene 1 abs. ethanol 1	200	colourless, crystalline m.p. 45° (angelic acid) 20 mg
2	benzene 1 abs. ethanol 1	500	colourless, crystalline m.p. 228–232° (unidentified glucoside) 50 mg
3	benzene 47.5 abs. ethanol 47.5 water 5.0	250	no residue
4	benzene 47.5 abs. ethanol 47.5 water 5.0	500	gummy resin, unidentified
5	abs. ethanol	300	gummy resin, unidentified
6	96 % ethanol	700	no residue
7	96 % ethanol	700	colourless, crystalline <i>O</i> - $\beta$ -D-glucosyl- $\beta$ -sitosterol 190 mg

These values are in agreement with those reported by other workers<sup>8,9</sup> for a compound named  $\beta$ -sitosterol-D-glucoside. The identity of the compound was confirmed partly through the IR-spectrum (KBr) and partly by identification of the sterol and sugar components separately after hydrolysis of the glycoside by the method of Thornton *et al.*<sup>10</sup> Paper chromatography revealed glucose as the only sugar component. Recrystallization of the diethyl ether-soluble product from the hydrolysis yielded a sterol, m.p. 134–135°. By means of the IR-spectrum (KBr) it was identified as  $\beta$ -sitosterol.

On enzymic hydrolysis with emulsin, paper chromatography revealed glucose.

Hence the compound is considered to be *O*- $\beta$ -D-glucosyl- $\beta$ -sitosterol.

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