Table 2. Unit cell dimensions at room temperature. (Estimated accuracy ± 0.05%).

<table>
<thead>
<tr>
<th>Phase</th>
<th>a-axis (Å)</th>
<th>c-axis (Å)</th>
<th>c/a</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti₃P</td>
<td>9.956</td>
<td>4.988</td>
<td>0.5005</td>
</tr>
<tr>
<td>V₃P</td>
<td>9.387</td>
<td>4.756</td>
<td>0.5067</td>
</tr>
</tbody>
</table>

Table 1 gives the X-ray powder data for V₃P. 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 V₃P and $e_i$ (FeP$_{3.37}$B$_{9.66}$)$^6$ shows that V₃P belongs to the $e_i$ structure type, space group $P4_2_1/n-C4-a$. The presence of lines $h + k + l = 2n + 1$ demonstrates that V₃P is not isostructural with Fe₃P, which has the space group $I4^4$.

The powder pattern and Weissenberg photographs of Ti₃P show that this phase is also of the $e_i$-type. A single crystal investigation of Ti₃P is currently being undertaken and will be published in this journal.

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The author wishes to thank Prof. G. Hägg for his kind interest and Dr. S. Rundqvist for valuable discussions.


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Constituents of the Umbelliferous Plants

II*. A Note on the Isolation of O-β-D-Glucosyl-β-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$^1$, and since compounds of this type have been isolated from a variety of plant species.$^2$-$^4$

In continuation of our attempts to find lignans in the plant family Umbelliferae$^5$ a sterol glycoside was isolated from the root of Levisticum officinale L. and identified as O-β-D-glucosyl-β-sitosterol.

The root also afforded angelic acid as well as an unidentified glucoside, m.p. 228–232$^o$$^o$.

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$^6$ starting with a chloroform extraction. The alcohol-benzene solution prepared in this manner was chromatographed on alumina (Aleco). 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$^7$ tests, m.p. 283–288$^o$ (decomp.), [α]$_D^{26}$ = -41.5$^o$ (c 0.397, pyridine).


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

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

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

These values are in agreement with those reported by other workers for a compound named β-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 glucoside by the method of Thornton et al. 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 β-sitosterol.

On enzymic hydrolysis with emulsin, paper chromatography revealed glucose. Hence the compound is considered to be O-β-D-glucosyl-β-sitosterol.


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