On the Active Components of the root of Citrus genus in Taiwan

II. The Components of the root of *Citrus tankan* Hayata

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According to Tsukamoto[1] studies on the chemical constituent on *Citrus tankan* Hayata, flavanone glycoside——hesperidin was isolated from its pericarps. But the root part had never been researched before. So that the authors studied on the components of the root of this species. The investigation is described as below.

The ethereal extract of the dried roots of *Citrus tankan* is treated on silica gel column chromatography as shown in Chart 1. Two crystalline compounds were isolated.

Compound(I) is a colorless scales, m.p. 139–142°. It gives a positive with Liebermann Burchard's reaction. From the result of gas liquid chromatography analysis, it is a mixture of stigmasterol and β-sitosterol(4 : 6).(Table 1, Fig. 1,2)

Compound(II) is a colorless prisms, m.p. 126–128°, $C_{14}H_{12}O_5$. From the melting point, composition, to be (II) is xanthyletin. The melting point was not depressed on admixture with an authentic sample.

From the view of the above facts, the authors not only isolated a mixture of phytosterol, but also obtained a macro quantity of xanthyletin(0.04%).

Chart 1.

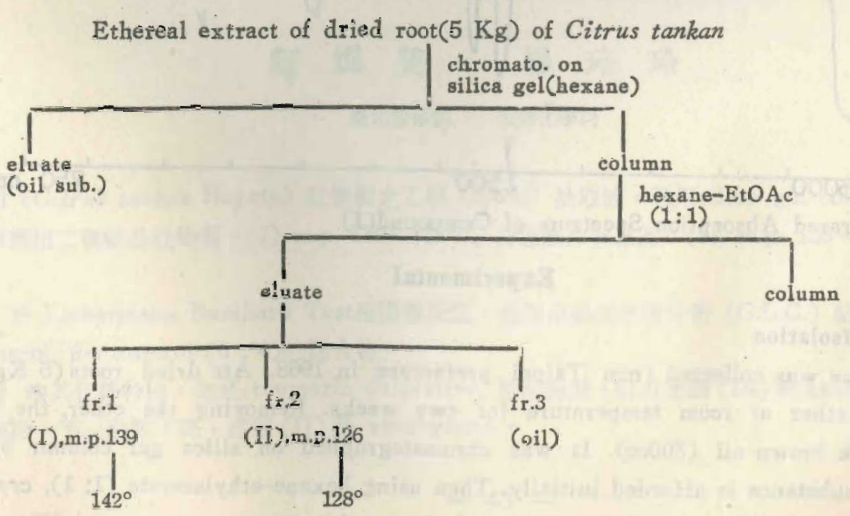

Table 1.

Relative Retention time of Steroid derivatives
Isolated from *Citrus tankan*

Compd.	Retention time (min) Column Temp. 269°
Stigmasterol	5.80
β-sitosterol	6.50

Shimazu GC-3AF

1.5%-SE-30 on Chromosorb-W(60-80 mesh)
Glass column: 1.8m × 0.4cmϕ, Column Temp. 269°
Carrier gas: N_2, 0.8Kg/cm², 16sec/10ml
H_2, 0.65Kg/cm², 12sec/10ml
sens $10^8 M\Omega$, range 3.2 V.

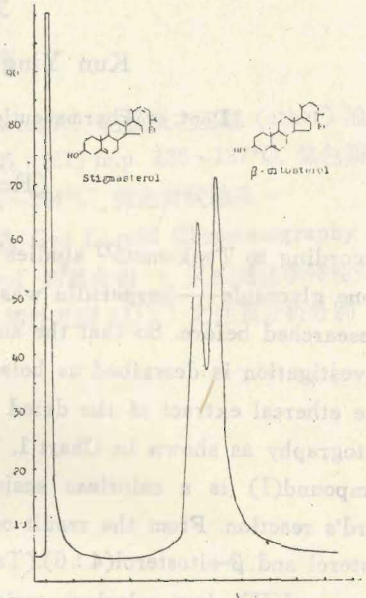

Fig. 1. Gas liquid chromatography of Compound (I)
Isolated from *Citrus tankan*

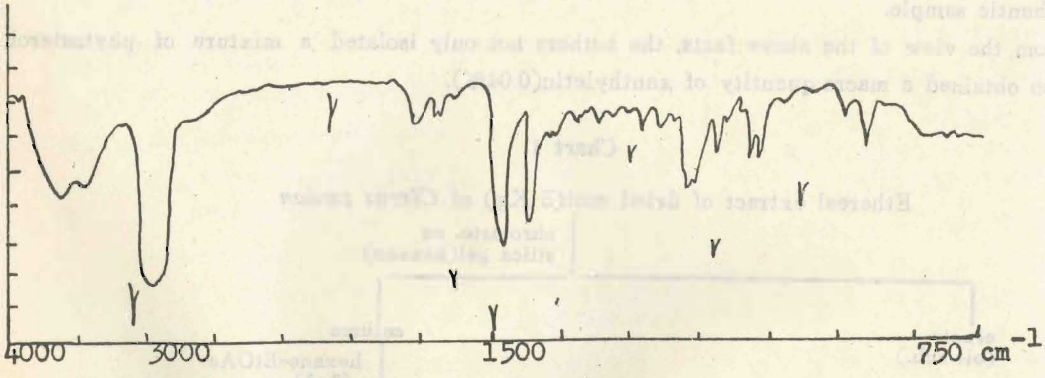

Fig. 2. Infrared Absorption Spectrum of Compound(I)

Experimental

Extraction and Isolation

Citrus tankan was collected from Taipei prefecture in 1968. Air dried roots (5 Kg) were extracted with ether at room temperature for two weeks. Removing the ether, the ethereal extract is a dark brown oil (300cc). It was chromatographed on silica gel column by using hexane. An oil substance is afforded initially. Then using hexane-ethylacetate (1: 1), crystals(I) and crystals(II) were afforded and recrystallized from ethanol to pure; crystals(I): colorless

scales and crystals(II): colorless prisms.

Mixed sterols (I): m.p. 139–142°, colorless scales, which gave a positive Liebermann Burchard's reaction. By carring out gas liquid chromatography analysis there are two kinds of mixed compounds from the relative retention times were exhibited. They are stigmasterol and β-sitosterol (4 : 6). (Fig.1, 2; Table I).

Xanthyletin(II): m.p. 126–128°, colorless prisms

Anal. Calcd. for $C_{14}H_{12}O_3$: C, 73.67; H, 5.30

Found: C, 73.60; H, 5.6

The IR-spectrum is entirely identical to xanthyletin and the mixed melting point with an authentic sample did not show any depression.

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Reference

1). T.Tsukamoto: Yakugaku Zasshi(Tokyo), 67, 45(1947)

中 文 摘 要

臺灣產柑類根部之活性成分

第二報 桶柑根之成分

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桶柑 (*Citrus tankan* Hayata) 乾燥根之乙醚 (ether) 抽取液，利用silica gel column chromatography，單離出二種結晶性物質。(I) m.p. 139～142°C, 白色鱗片狀結晶；(II) m.p. 126～128°C, 無色板狀結晶。

(I) 於 Liebermann Burchard Test呈陽性反應，進而由氣相色層分析 (G.L.C.) 結果，確認 (I) 爲 stigmasterol, β-sitosterol (6 : 4)的混合物。

(II) 由其化學特性，知是 coumarin derivative, 再觀融點，紅外光譜 (IR) 與 xanthyletin 一致。經與標品混融，不呈融點下降，確認 (II) 是 xanthyletin.。