# On the Active Components of the Roots of Citrus genus in Taiwan
## V. The Components of the roots of Citrus maximus form. buntan , C. maximus form. hakuyu , C. maximus form. hounyu , and C. maximus form. yayu

by

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In previous reports[1)2)3)4)], authors have been studied the chemical components of the roots of 8 kinds of *Citrus* genus. Lately, the authors have continue to study on *Citrus maximus,* those classified as four kinds. Those are (1) *Citrus maximus* form. *buntan* , (2) *C. maximus* form. *hakuyu,* (3) *C. maximus* form. *hounyu* , (4) *C. maximms* form. *yayu.*

The above 4 kinds were treated under the general procedure of extraction and concentraction. As from the ethereal extract were separated and isolated by eluting through silica gel chromatography. However, evidence from their indentical melting point, gas liquid chromatography, these are exhibit each contained the mixed phytosterol and results were submitted as following:

1) *C. maximus* form. *buntan* contains a mixture of stigmasterol and β -sitosterol. (fig.1)
2) *C. maximus* form. *hakuyu* contains a mixture of stigmasterol and β -sitosterol. (Fig.2)
3) *C. maximus* form. *hounyu* contains xanthyletin and a mixture of stigmastrol and β-sitosterol. (Fig.3)
4) C. maximus form. *yayu* contains a mixture of stigmasterol and β -sitosterol. (Fig.4)

The roots all contain a mixture of stigmasterol and β -sitosterol. But the different view only in volumn (Show as Fig.1,2,3,4). Thus, it is interesting fact as view from the point of chemotaxonomy.

## Experimental

1) *Citrus maximus* form. *buntan* (CMB)

2.8 Kg of dried roots were collected from Tainan county in September 1974. They were firstly frashmented and macerated three

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times with ether. The ethereal extract was then concentrated to a dark brown viscous oil (150 ml). It was chromatographed through silica gel column using n-hexane as eluting solvent. The n-hexane eluting was concentrated to give yellowish scales, which decolorized with actived carbon and recrystallized with ethanol to give a colorless scale, CMB-I, mp.133-4°. It gives a positive Lieberman Burchard's reaction (red-violet-blue). However, according to gas liquid chromatographic retention time, it is exhibited to be a mixture of stigmasterol and β-sitosterol. (Fig.1)

2) C. *maximus* form. *hakuyu* (CMH)

5.3 Kg of dried roots were collected from Tainan county in November 1974. The ethereal extract was concentrated to a dark brown oilly like substance (200 ml) and a precipitate obtained. Removing the precipitate and recrystallized with ethanol to give a yellowish powder, CMH-I, mp.135-7°. The oil like substance was treated as 1). The extract was chromatographed through silica gel column with *n*-hexane as eluting solvent while each successive fraction were collected in order. The n-hexane eluting was concentrated to give yellowish scales, which recrystallized with ethanol to give a colorless scale, CMH-II, mp.147-8° and a positive Lieberman Burchard's reaction (red-violet-blue). According to gas liquid chromatograph analysis, it is suggested to be a mixture of stigmasterol and β-sitosterol. (Fig. 2)

3) *Citrus maximus* form. "*hounyu*" (CH)

3.7 Kg of dried roots were collected from Tainan county in September, 1974 and treated as in 1).

Fr.1 : An oilly substance was obtained.

Fr.2 : The *n*-hexane eluting was concentrated to give a yellowish powder. Which decolorized and recrystallized to give a white scale, CH-I, mp.68-72°.

Fr.3 : Colorless scales, CH-II, mp.136-8°, which give a positive Lieberman Burchard's reaction (red-violet-blue). According to gas liquid chromatograph analysis, it is a mixture of stigmasterol and β-sitosterol. (Fig. 3)

Fr.4 : The n-hexane eluting was concentrated to give a solid crystalline which recrystallized with ethanol to give a colorless prism, CH-III, mp. 126-8°, $C_{14}H_{12}O_3$,

*Anal.* Calcd: C, 73.67; H, 5.30

Found: C, 74.01; H, 5.38

It was verified to be xanthyletin. By mixed melting point with authentic sample, IR spectrum and Co-TLC were identical.

4) *Citrus maximus* form. *yaya* (CMY)

3.9 Kg of dried roots were collected from Tainan County in September 1974, and treated as in 1).

Concentrated the ethyl acetate eluting portion and re-column chromatographed through silica gel, n-hexane as the eluting solvent

to give a solid crystalline. It recrystallized and recolumn to give a colorless scale, CMY-1 mp.152-4°. It gives a positive result in Lieberman Burchard's reaction (red-violet-blue), a mixture of stigmasterol and β -sitosterol confirm by gas liquid chromatography. (Fig. 4)

**Shimadzu GC-3AF**

Column: 1.5% SE-30 on Chromosorb -W (60-80 mesh)

Glass column: 1.5m x 0.4 cmϕ, Temp.: 230°

Carrier gas: $N_2$ 0.9 Kg/cm², 16 sec/10ml; $H_2$ 0.6 Kg/cm², 12 sec/10ml;
Air 0.45 Kg/cm²; Range 3.2 V; Sens $10^3$ MΩ

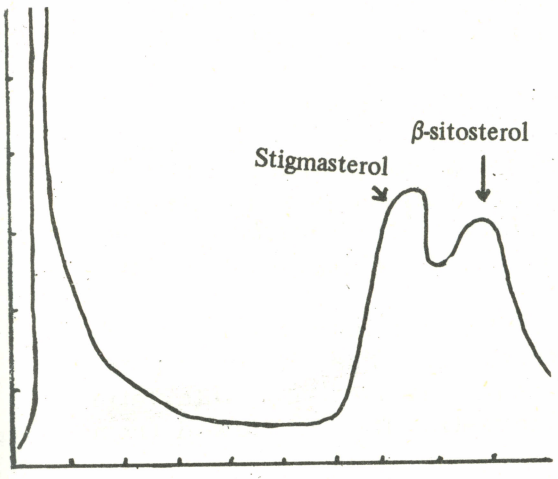


Fig. 1 GLC of mixed phytosterol isolated from *C. maximus* form *buntan*

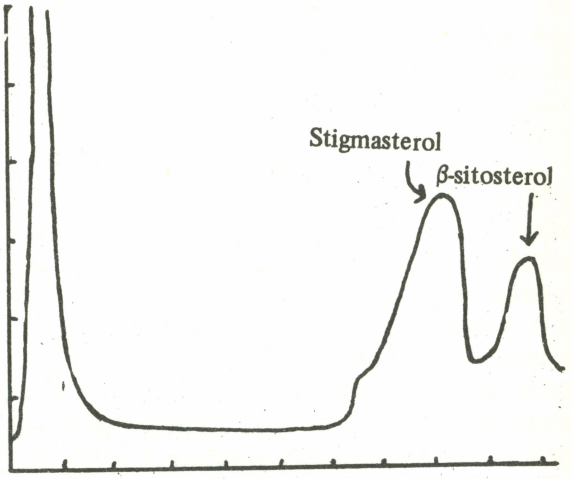


Fig. 2 GLC of mixed phytosterol isolated from *C. maximus* form *hakuyu*

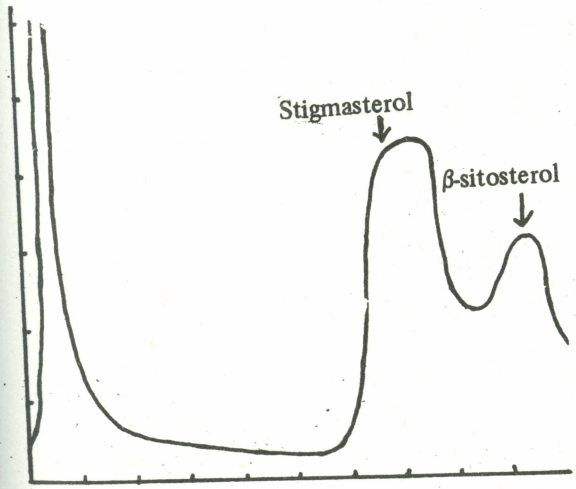


Fig. 3 GLC of mixed phytosterol isolated from *C. maximus* form *hounyu*

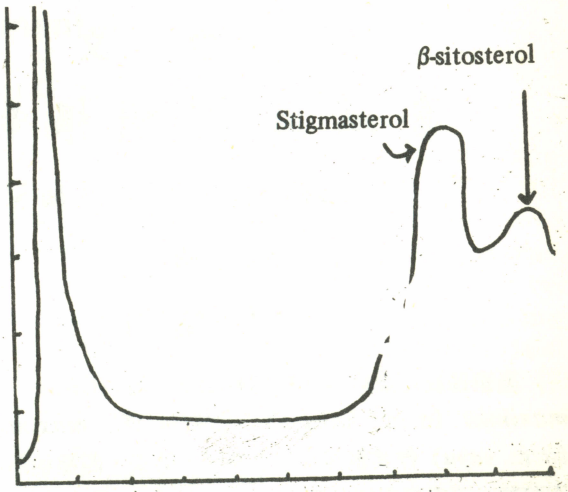


Fig. 4 GLC of mixed phytosterol isolated from *C. maximus* form *yayu*

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## Summary

In this paper, we investigated the chemical components of following 4 kinds of *Citrus maximus* species, both indigenous and cultivated in Taiwan. (1) *Citrus maximus* form. *buntan*, (2) (*C. maximus*; form. *hakuyu*, (3) *C. maximus* form. *hounyu*, (4) *C. maximus* form. *yayu*.

In view of the result, the 4 species, all contained the mixture phytosterol of stigmasterol and β-sitosterol. The different point only in the 2 mixture volume, and the coumarin derivatives-xanthyletin only containing in (3). Thus, it is an interesting fact as viewed from the point of chemotaxonomy.

## 中 文 摘 要

# 臺灣產柑類根部之活性成分

### 第五報：文旦柚、白柚、紅柚及野柚根之成分

楊 玲 玲　　顏 焜 熒

臺北醫學院　　生藥化學科

臺南縣栽培及野生柚類四種(1)文旦柚 (*Citus maximus* form. *buntan*)，(2)白柚 (*C. maximus* form. *hakuyu*)，(3)紅柚 (*C. maximus* form. *hounyu*)，(4)野柚 (*C. maximus* form. *yayu*) 作其根部成分之探討，結果，四種柚類均含 Stigmasterol 及 β-Sitosterol 之混合固醇類，而香豆素衍化物 -xanthyletin，僅含於紅柚之根部。

本論文內容已以簡報形式投稿於臺灣藥學雜誌，第二十九卷，第一期 (1977)