M.p. 236–238°, $[\alpha]_D^{20}$ + 76° ($CHCl_3$); $C_{32}H_{52}O_2$ (M^+ by MS was 468). On hydrolysis it gave needles (β-amyrin), m.p. 197°. The compound was confirmed by superimposable IR, NMR, UV spectra with authentic acetyl derivative of β-amyrin.

β-Amyrenone (2·8% yield) crystallized from MeOH. M.p. 177°, $[\alpha]_D$ + 107·2° ($CHCl_3$). $C_{30}H_{48}O$ (M^+ 424 by MS). Superimposable IR, NMR, UV spectra with authentic specimen prepared from oxidation of β-amyrin. *Lupeol* (1·35% yield) crystallized as needles from MeOH. M.p. 216°, $[\alpha]_D^{20}$ + 27·2° ($CHCl_3$). The MS showed a MW of 426 ($C_{30}H_{50}O$). Its acetate melted at 215–216°, and its benzoate at 258–259°. When hydrogenated it produced lupanol, m.p. 201°; and the MS had a molecular ion peak at *m/e* 428. Its oxidation product (lupen-3-one), m.p. 170°, $[\alpha]_D^{20}$ + 63·5°. *Sitosterol* (1·9% yield). M.p. 137–138°, [α] −37° ($CHCl_3$). It had an MS characteristic of 3 β-hydroxy steroid (M^+ 414). Molecular formula $C_{29}H_{50}O$. Its acetate melted at 127°. This was confirmed by IR, NMR, UV and co-TLC with an authentic sample of sitosterol.

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LIGNAN DIGLUCOSIDES FROM *TRACHELOSPERMUM ASIATICUM*

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Key Word Index—*Trachelospermum asiaticum* var. *intermedium;* Apocynaceae; lignans; matairesinol-4,4′-di-*O*-β-D-glucopyranoside and nortrachelogenin-4,4′di-*O*-β-D-glucopyranoside.

We report the isolation of two new lignan glucosides; matairesinol-4,4′-di-*O*-β-D-glucopyranoside(I) and nortrachelogenin-4,4′-di-*O*-β-D-glucopyranoside(IV), from the stems of *Trachelospermum asiaticum* Nakai var. *intermedium* Nakai.

The stems (25 kg) were extracted with hot MeOH and the MeOH solution evaporated to small volume, diluted with H_2O and filtered. The filtrate was extracted with successive, light petrol., Et_2O and $CHCl_3$. The aqueous layer was concentrated to syrup and extracted with hot EtOAc. The residue was extracted with $CHCl_3$–MeOH (2:1). The $CHCl_3$–MeOH extractive was column chromatographed on activated charcoal and eluted by MeOH–H_2O (49:1), MeOH–H_2O (1:1) and MeOH. The MeOH eluate was concentrated, chromatographed on silica gel column and eluted by $CHCl_3$–EtOH (3:2). The fraction showing R_f 0·16 spot on TLC [Merk silica gel G, $CHCl_3$–MeOH (3:1)] was evaporated to give I (51·4 mg).

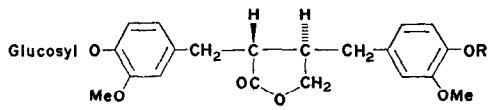
The residue after $CHCl_3$–MeOH extraction was column chromatographed on activated charcoal, followed by silica gel column chromatography in a similar manner as $CHCl_3$–MeOH extractive. The fraction showing R_f 0·09 spot on TLC gave IV (41·7 mg).

I is colorless powder, $C_{32}H_{42}O_{16}.H_2O$, m.p. 104–106°, $[\alpha]_D^{14}$ — 24·0° (*c* 0·5 in EtOH), λ_{max}^{EtOH} nm (log ϵ) 225 (4·09) 279 (3·65), no shift on alkaline addition, ν_{max}^{KBr} cm^{-1} 1765 (γ-lactone), and gave octaacetate, $C_{46}H_{56}O_{23}$, m.p. 88–89°, λ_{max}^{EtOH} nm (log ϵ) 227 sh (4·11) 279 (3·66), ν_{max}^{KBr} cm^{-1} 1760(CO), the NMR spectrum ($CDCl_3$) of which showed signals attributable to two aromatic methoxyls(δ 3·75s) and eight acetyls(δ 2·02s and 2·05s) attached to the glucosyl groups.

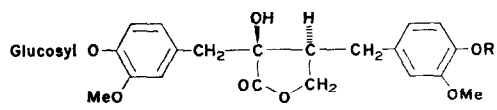
Hydrolysis of I with 10% H_2SO_4 solution or emulsin gave matairesinol[1] and D-glucose. The permethyl ether prepared by the methylation of I with NaH, DMSO and Me I (Hakomori's method[2]) afforded only methyl 2,3,4,6-tetra-*O*-methyl-D-glucopyranoside on methanolysis with 3% methanolic —HCl, which was detected by GLC [column; 15% poly-butanediol glycol succinate on Celite 545 (2 m × 3 mm); column temp., 175°; carrier gas: N_2 (30 ml/min), on JEOL-JGC-1100 with flame ionization detector].

IV is colorless powder, $C_{32}H_{42}O_{17}.1\frac{1}{2}H_2O$, m.p. 137–140°, $[\alpha]_D^{14}$ — 15·9° (*c* 0·83 in EtOH), λ_{max}^{EtOH} nm (log ϵ) 224(4·19) 279(3·75), no shift on alkaline addition, ν_{max}^{KBr} cm^{-1} 1770(γ-lactone), and gave octaacetate, $C_{46}H_{56}O_{24}$, m.p. 124–125°, λ_{max}^{EtOH} nm (log ϵ) 226sh(4·15) 279(3·76), ν_{max}^{KBr} cm^{-1} 1760(CO), and the NMR spectrum($CDCl_3$) of which showed signals attributable to two aromatic methoxyls(δ 3·85s) and eight acetyls(δ 2·00s and 2·05s) attached to the glucosyl groups.

Hydrolysis of IV with 10% H_2SO_4 solution or emulsin gave nortrachelogenin[1] and D-glucose. The permethyl ether of IV afforded only methyl 2,3,4,6-tetra-O-methyl-D-glucopyranoside on methanolysis.



(I) R = glucosyl
(II) R = H
(III) R = Me



(IV) R = glucosyl
(V) R = H
(VI) R = Me

The co-occurrence of I and IV with four lignan glucosides; matairesinoside(II), arctini (III), nortracheloside(V) and tracheloside(VI) in the same plant is interested from the biogenetic point of view.

[1] S. NISHIBE, S. HISADA and I. INAGAKI, *Phytochem.* **10**, 2231 (1971).
[2] S. HAKOMORI, *J. Biochem.* **55**, 205 (1964).