Roots. Alcoholic extract of the ground root was further extracted with $CHCl_3$. This extract (I) was chromatographed on a column of neutral alumina, whereas the insoluble portion was redissolved in EtOH (II).

Sitosterol. $C_{29}H_{50}O$ (identified by m.p., m.m.p., analyses and IR of the sterol and its acetate), from the alumina column of the $CHCl_3$ extract (I).

3,4,3'-*Tri*-O-*methylellagic acid.* $C_{17}H_{12}O_8$ (identified by m.p., analysis, UV, IR and MS), from the subsequent fraction of the alumina column.

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PIPERACEAE

CONSTITUENTS OF *PIPER METHYSTICUM*

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Key Word Index—*Piper methysticum*; Piperaceae; yangonin; methysticin; kawain.

Plant. Piper methysticum, Forst., *Use. Medicinal.*[1] *Previous work.* On roots.[2–6]

Roots. Extracted with light petroleum (b.p. 60–80°), benzene, $CHCl_3$ and MeOH. *Petrol extract.* The extract upon concentration and cooling deposited yellow solid which was found to be a mixture of at least two components (TLC). $CHCl_3$ solution of the crude solid was chromatographed over silica gel. Elution with benzene–$CHCl_3$ (9:1) yielded *yangonin* $C_{15}H_{14}O_4$ (M^+ 258), m.p. 152–54° (lit.[7] m.p. 153–154°. Found: C, 69·71; H, 5·84, $C_{15}H_{14}O_4$; required: C, 69·76; H, 5·42%). Characteristic NMR bands at 3·90 δ

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(3H, singlet OCH_3), 3·92 δ (3H, singlet, aromatic OCH_3), 5·22δ and 5·94 δ (each 1H, doublet, $J = 2$ Hz, olefinic protons of α-pyrone ring), 6·18 δ and 7·22 δ (2H, each, doublet, $J = 8$ Hz aromatic proton), 6·20 δ and 7·25 δ (1H, each, doublet, $J = 16$ Hz, *trans* >CH =CH<); MS *m/e* (rel. intensity) M^+ 258 (100), 230 (30), 215 (8), 187 (25), 115 (7). Benzene $CHCl_3$ (3:1) mixture yielded *methysticin*, $C_{15}H_{14}O_5$ (M^+ 274), m.p. 134–136°. (lit.[8] m.p. 136–137°. Found: C, 65·23, H, 5·17. $C_{15}H_{14}O_5$; required; C, 65·69, H, 5·10%). Characteristics NMR bands 2·60 δ (2H, triplet, $-CH_2-$ of α-pyrone ring), 3·75 δ (3H, singlet, OCH_3), 5·00 δ (1H, multiplet > C*H*–CH_2–) 5·20 δ (1H, singlet, olefinic proton), 5·98 δ (2H, singlet, –O–CH_2–O–), 6·20 δ and 6·88 δ (1H, each doublet, $J = 16$ Hz *trans* >CH = CH<); MS *m/e* (rel. intensity) M^+ 274 (50), 228 (28), 176 (10), 175 (12), 148(75), 135(100), 115(14), 98(14), 68(15).

Mother liquor was chromatographed over silica gel. The oil obtained from light petroleum (b.p. 60–80°) eluates upon cooling deposited *kawain* $C_{14}H_{14}O_3$ (M^+ 230), m.p 110° (lit.[2] 106–5–108°. Found: C, 72·88; H, 5·93. $C_{14}H_{14}O_3$; required: C, 73·04; H, 6·09%). Characteristic NMR bands at 2·60 δ (2H, multiplet–CH_2–CH< of α-pyrone ring), 3·80 δ (3H, singlet OCH_3), 5·00 δ (1H, multiplet, >CH–CH_2–), 5·22 δ (1H, singlet, olefinic proton); 7·38 δ (5H, broad singlet, aromatic protons), MS *m/e* (rel. intensity) M^+ 230(38), 202(22), 104(25), 98(100), 90(40), 70(33), 69(85).

Benzene extract. Fractionated into acidic and neutral fractions. Neutral fraction upon chromatographic separation over alumina yielded yangonin. Acidic fraction was chromatographed over silica gel. Benzene–$CHCl_3$ (1:1) eluates afforded slightly orange coloured solids, designated as *flavokawain C*. $C_{17}H_{16}O_5$ (M^+ 300), m.p. 194–195° (*new compound*); ν_{max} (KBr) 3380 cm^{-1} (broad, bonded-OH); 1640 cm^{-1} (chelated > C = O); λ_{max}^{EtOH} 245, 265, 370 nm; MS *m/e* (rel. intensity). M^+ 300(74), 299(45), 272(52), 207(52), 194(22), 181(100), 152(45), 138(37), 137(45), 119(37), 107(30), 95(30), 91(75), 77(22), 69(60), 65(70).

Further elution with $CHCl_3$ yielded another compound, $C_{17}H_{16}O_5$ (M^+ 300) m.p. 210–212° (*new compound*) MS *m/e* (rel. intensity) M^+ 300(9), 299(5), 270(59), 193(22), 166(100), 138(63), 123(27), 95(22), 85(27), 83(45), 78(68), 77(32), 69(30), 52(22), 51(32), 50(27).

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