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Semecarpuflavanone—a new biflavanone from *Semecarpus anacardium* Linn.

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Abstract. A new biflavanone designated as semecarpuflavanone has been recently isolated from the alcoholic extract of the nut shells of *Semecarpus anacardium* Linn. It was dehydrogenated with iodine and potassium acetate in glacial acetic acid to the corresponding, relatively more stable biflavone known as SA4. The structure of semecarpuflavanone has been established through chemical and spectroscopic studies.

Keywords. *Semecarpus anacardium*; Anacardiaceae; biflavanones; semecarpuflavanone; biflavone SA4; PMR and mass spectra.

1. Introduction

Three new biflavanones, named as galluflavanone, jeediflavanone and semecarpuflavanone, besides the three known biflavanones (Prakasa Rao *et al* 1973), 1–3 have been recently isolated from the acetone soluble fraction of the ethanolic extract of the defatted nut shells of *Semecarpus anacardium* Linn. On the basis of spectral and chemical data, structures 4 and 5 have been already assigned to galluflavanone (Murthy 1983) and jeediflavanone (Murthy 1985), respectively. The present paper deals with the isolation and structure determination of semecarpuflavanone.

2. Results and discussion

Semecarpuflavanone (6) appeared as a fine microcrystalline pale yellow powder from acetone, $C_{30}H_{22}O_{10}$, m.p. 248–49°. Preliminary characterisation showed it to be a polyhydroxybiflavanone. It exhibited UV maxima in ethanol at 291 nm which underwent a bathochromic shift (291 → 312 nm) on addition of sodium acetate to the test solution, while with aluminium chloride no such shift was observed, indicating the absence of chelated hydroxyl groups in the biflavonoid. Further, in its PMR spectrum, no low field proton could be noticed. The above observation clearly revealed that there was at least one 7-hydroxyflavanone system (Horowitz and Jurd 1961; Mabry *et al* 1970) in the molecule.

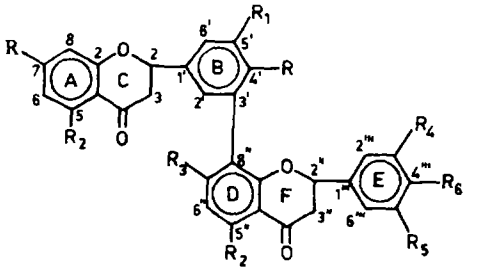
The PMR spectrum (270 MHz, acetone-d_6) of semecarpuflavanone displayed signals due to six non-chelated D_2O exchangeable hydroxylic protons at τ 1·50 (*s*, 1H), 2·24 (*s*, 1H), 2·36 (*s*, 2H) and 2·74 (*s*, 2H). Hence, the biflavonoid linkage must only be through a C–C linkage since all the ten oxygen atoms in the biflavanone (6) are accounted for by the six non-chelated hydroxyl groups and four pyranone oxygen

atoms. Oxidation of semecarpuflavanone with neutral permanganate afforded only one mole of gallic acid suggesting that one of the side-phenyls is involved in the interflavonoid linkage.

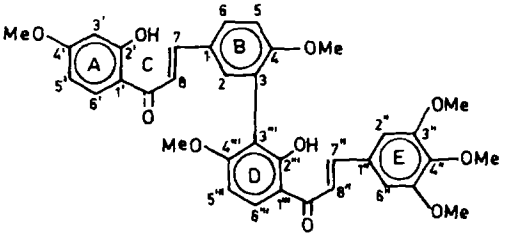
On methylation with diazomethane, semecarpuflavanone gave a pentamethyl ether (7), $C_{35}H_{32}O_{10}$, m.p. 161–62°, whose PMR spectrum ($CDCl_3$) displayed a singlet signal at τ 1·48(1H) corresponding to the non-chelated D_2O exchangeable hydroxylic proton. Further, the biflavonoid (6) on acetylation with acetic anhydride and pyridine afforded a hexaacetate (8), $C_{42}H_{34}O_{16}$, m.p. 174–76°, while with dimethyl sulphate and K_2CO_3 in dry acetone, it gave a bichalcone hexamethyl ether (9), $C_{36}H_{34}O_{10}$, m.p. 199–201°. The PMR spectrum of compound 9 showed the presence of two chelated hydroxylic protons at τ -4·20 (*s*, 1H) and -4·30 (*s*, 1H).

When semecarpuflavanone pentamethyl ether was oxidised with neutral permanganate, both syringic acid and 2-hydroxy-4-methoxybenzoic acid (identified by mixed m.p. and IR) were obtained. Hence the diaryl system must contain the remaining two methoxyl groups which are placed by analogy and PMR spectral data at the B-4′ and D-7″ positions. Consequently the biflavonoid linkage must be either at the B-3′-D-8″ position or at the B-3′-D-6″ position. Since the parent compound contains no chelated hydroxyl groups, the positions at A-5 and D-5″ are free. Further, the PMR spectra of semecarpuflavanone and its three derivatives (7–9) clearly indicated the presence of two *ortho*-coupled protons which must correspond to ring D (see §3). On the basis of this observation, the biphenyl linkage at the B-3′-D-6″ position is eliminated from consideration and hence, semecarpuflavanone must have a C–C linkage at the B-3′-D-8″ position.

Semecarpuflavanone was dehydrogenated (Pelter *et al* 1971; Murthy *et al* 1981) with iodine and potassium acetate in glacial acetic acid to the corresponding, relatively more stable biflavone designated as SA4 which appeared as a light yellow powder from acetone, $C_{30}H_{18}O_{10}$, m.p. > 300°. Preliminary characterisation showed it to be a



1 R=R₂=R₄=R₆=OH, R₁=R₃=R₅=H
2 R=R₂=R₃=R₆=OH, R₁=R₄=R₅=H
3 R=R₃=R₆=OH, R₁=R₂=R₄=R₅=H
4 R=R₁=R₃=R₄=R₅=R₆=OH, R₂=H
5 R=R₂=R₃=R₄=R₆=OH, R₁=R₅=H
6 R=R₃=R₄=R₅=R₆=OH, R₁=R₂=H
7 R=R₃=R₄=R₅=OMe, R₆=OH, R₁=R₂=H
8 R=R₃=R₄=R₅=R₆=OAc, R₁=R₂=H



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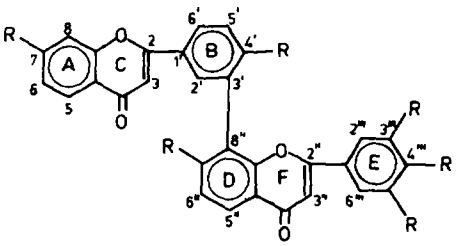
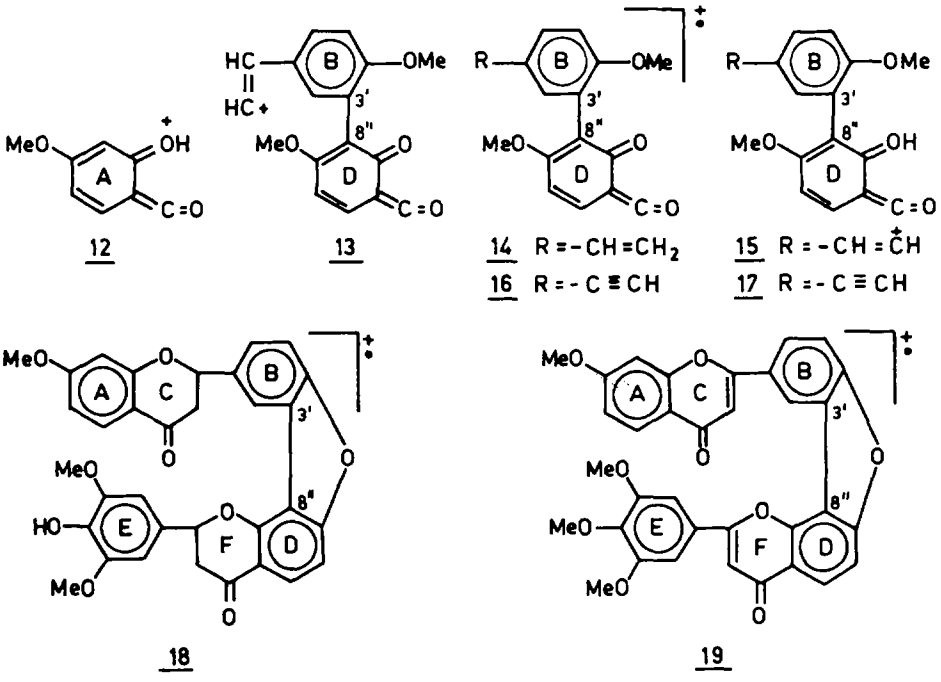


10 R=OH, 11 R=OMe

polyhydroxybiflavone. The PMR spectrum of SA4 (10) in acetone-d_6 indicated the presence of six non-chelated D_2O exchangeable hydroxyl groups at τ 1·54 (*s*, 1H), 2·14 (*s*, 2H), and 2·39 (*s*, 3H). On methylation with dimethyl sulphate and K_2CO_3, SA4 furnished a hexamethyl ether (11), $C_{36}H_{30}O_{10}$, m.p. 190–91°. Oxidation of 11 with neutral permanganate afforded only one mole of gallic acid tri-methyl ether suggesting that one of the side-phenyls is involved in the diaryl linkage.

The chemical shifts and multiplicity of protons in the PMR spectra of semecarpuflavanone (6), its three derivatives (7–9), compound SA4 (10) and its hexamethyl ether (11), fully supported the proposed structure of semecarpuflavanone (6) (see § 3).

Semecarpuflavanone pentamethyl ether (7) and SA4 hexamethyl ether (11) in their respective mass spectra showed the molecular ions (M^+) at *m/z* 612 and *m/z* 622. The peak at *m/z* 179 [3,5-$(H_3CO)_2$-4-OH-C_6H_2-CH=C^+H] arising from rings E and F in 7 not only revealed that these rings are not involved in the interflavonoid linkage but also indicated that the hydroxyl groups at positions 3‴ and 5‴ in ring E are methylated. The ion at *m/z* 151 corresponding to the fragment (12) appeared in the mass spectra of both the methyl ethers (7 and 11) indicating that the hydroxyl group at the A-7 position is methylated. The peak at *m/z* 281 corresponding to the fragment (13) in the pentamethyl ether could arise after two RDA fragmentations. There is another peak at *m/z* 282 corresponding to either of the ions (14) and (15) and these two are closely related to the fragment (13). Compound (11) also afforded the two central fragments at *m/z* 280 and *m/z* 281 corresponding to the ions (16) and (17). Similar crucial fragments were already reported in (−) succedaneaflavanone tetramethyl ether (Chen and Lin 1975) and morelloflavone heptamethyl ether (Karanjgaokar *et al* 1967). Compounds (7) and (11) showed the peaks, respectively, at *m/z* 566 and *m/z* 576, which are formed by the loss of 46 mass units. These fragments can be formulated respectively as (18) and (19) in which



the methoxyl groups *ortho* to the biphenyl linkage cyclise to a furan ring (Natarajan *et al* 1969). Perhaps the most characteristic feature is the formation of the two fragments (13) and (18) in the pentamethyl ether (7) and the two peaks (16) and (19) in the hexamethyl ether (11) which could be sufficiently indicative or diagnostic of a C–C linkage. It may be mentioned here that similar fragments have been reported in (±) fukugetin heptamethyl ether (Konoshima *et al* 1969) and rhusflavanone (Lin and Chen 1973). Overall the mass spectral (MS) fragmentation pattern of semecarpuflavanone pentamethyl ether exhibits a close similarity to that of 2,3-dihydroamentoflavone hexamethyl ether (Varshney *et al* 1973) and GB-2 (Jackson *et al* 1971). Similarly, the MS fragmentation of SA4 hexamethyl ether showed a very close resemblance to that of amentoflavone hexamethyl ether (Natarajan *et al* 1969).

Based on the foregoing chemical and spectral evidences, structure (6) has been assigned to semecarpuflavanone with the biflavonoid linkage at the B-3′-D-8″ position.

3. Experimental

Equipment and methods used for UV, IR, PMR, MS, m.p., C and H analysis, TLC and PLC have been described elsewhere (Murthy 1985).

Isolation: On removal of solvent under reduced pressure, fraction FI (Murthy 1985) yielded a dark coloured mass from which the gummy material was separated by thorough maceration with *n*-hexane (50 ml × 3). The insoluble deep yellow residue (~ 18 gms) showed only one major spot on TLC besides a large tailing (chloroform:methanol, 5:1) and was adsorbed on silica gel (40 gms) and transferred over to a column of silica gel (110 gms, 100–200 mesh) set with chloroform. The column was eluted successively with chloroform, ether and acetone. The ether eluate afforded a deep pink colour with Mg-HCl. Hence, on removal of solvent under vacuum, the combined ether fractions furnished a yellow compound which contained two close running minor components on TLC (chloroform: methanol, 5:1) besides a small streak. The major component was separated by preparative TLC using chloroform: methanol (5:1) as the solvent for development. The compound so obtained still showed minor impurities on a thin layer chromatogram and hence it was filtered through a small column of silica gel with chloroform: acetone (5:2) as the eluent. The column fractions, on concentration under vacuum, yielded a pale yellow compound, semecarpuflavanone, which was found to be pure on a thin layer chromatoplate.

Semecarpuflavanone (6): It resisted crystallisation from normal organic solvents. On drying in a vacuum oven for six hours at 60°, semecarpuflavanone appeared as a pale yellow powder, $C_{30}H_{22}O_{10}$, m.p. 248–49°, yield: 525 mg. It gives a greenish-violet ferric reaction, a pinkish-red colour with Mg-HCl, an orange colour with $NaBH_4$-HCl and is readily soluble in aqueous NaOH giving a deep orange-yellow solution; UV (EtOH): nm, 221, 291, 333; + NaOAc, 223, 312, 335; + $AlCl_3$, 221, 292, 332; IR ν(nujol): 3520–3470 (*br*, OH), 3225 (OH), 1680 (flavanone carbonyl), 1600, 1580 (aromatic) cm^{-1}; Found: C, 66·04; H, 4·06; $C_{30}H_{22}O_{10}$ requires C, 66·42 and H, 4·09%; PMR (270 MHz, acetone-d_6, τ values): 2·54 (*dd*, 1H, J = 2, 8·5 Hz, B-6′), 2·63 (*d*, 1H, J = 2 Hz, B-2′), 2·85 (*d*, 1H, J = 8·5 Hz, B-5′), 3·08 (*d*, 1H, J = 2 Hz, E-6‴), 3·16 (*d*, 1H, J = 2 Hz, E-2‴), 3·28 [(*d*, 1H, J = 8 Hz, D-5″) (Prakasa Rao *et al* 1973)], 3·48 (*d*, 1H, J = 8 Hz, D-6″), 3·66 [(*d*, 1H, J = 8 Hz, A-5) (Prakasa Rao

et al 1973)], 3·86 (*dd*, 1H, J = 2, 8 Hz, A-6), 3·78 (*d*, 1H, J = 2 Hz, A-8), 4·58 (*dd*, 2H, J = 4, 12 Hz, C-2, F-2″), 6·92 (*m*, 2H, *trans*-protons, C-3, F-3″), 7·27 (*dd*, 2H, J = 3, 17 Hz, *cis*-protons, C-3, F-3″), 1·50 (*s*, 1H), 2·24 (*s*, 1H), 2·36 (*s*, 2H), 2·74 (*s*, 2H) [non-chelated D_2O exchangeable hydroxylic protons, A-7, B-4′, D-7″, E-3‴, 4‴, 5‴].

Semecarpuflavanone pentamethyl ether (7): Compound 6 (175 mg) in acetone (10 ml) was added to an ethereal solution of diazomethane till the yellow colour persisted. After the usual work-up, 7 appeared as yellow crystals from a chloroform and acetone mixture, $C_{35}H_{32}O_{10}$, m.p. 161–62°, UV (EtOH): nm, 220, 289, 332; IR ν(nujol): 3450 (OH), 2830 (OCH_3), 1685 (flavanone carbonyl), 1600, 1585 (aromatic) cm^{-1}; Found: C, 68·19; H, 5·22; OCH_3, 25·10; $C_{35}H_{32}O_{10}$ requires C, 68·62; H, 5·26 and OCH_3, 25·32%; PMR (80 MHz, $CDCl_3$, τ values), 7·24 (*dd*, 2H, J = 3, 16 Hz, *cis*-protons, C-3, F-3″), 6·90 (*m*, 2H, *trans*-protons, C-3, F-3″), 4·60 (*q*, 2H, J = 4, 12 Hz, C-2, F-2″), 2·60–2·82 (*m*, 3H, B-2′, 5′, 6′), 3·10 (*s*, 2H, E-2‴, 6‴), 3·31 (*d*, 1H, J = 8 Hz, D-5″), 3·49 (*d*, 1H, J = 8 Hz, D-6″), 3·62 (*d*, 1H, J = 8 Hz, A-5), 3·88 (*dd*, 1H, J = 2, 8 Hz, A-6), 3·78 (*d*, 1H, J = 2 Hz, A-8), 1·48 (*s*, 1H, non-chelated D_2O exchangeable hydroxylic proton, E-4‴), 6·16 (*s*, 2 × 3H), 6·25 (*s*, 3 × 3H) [methoxyl groups, A-7, B-4′, D-7″, E-3‴, 5‴]; m/z (relative intensity) 612 $[M]^+$ (42), 597 $[M-15]^+$ (15), 582 $[M-2\times15]^+$ (7·6), 581 $[M-31]^+$ (8·5), 566 $[M-46]^+$ (8), 461 (11), 432 (10), 329 (9·5), 283 (6·8), 281 (29), 181 (57·5), 179 (64·6), 151 (100), 150 (72·5).

Semecarpuflavanone hexaacetate (8): To 6 (30 mg), a mixture of freshly distilled acetic anhydride (1 ml) and dry pyridine (1 ml) was added, heated on a steam-bath for four hours and worked-up in the usual manner. The hexaacetate (8) appeared as pale yellow crystals from a chloroform and methanol mixture, $C_{42}H_{34}O_{16}$, m.p. 174–76°, UV (EtOH): nm, 222, 288, 329; IR ν(nujol): 1755, 1745 ($OCOCH_3$), 1685 (flavanone carbonyl), 1600, 1580 (aromatic) cm^{-1}; Found: C, 63·06; H, 4·25; $C_{42}H_{34}O_{16}$ requires C, 63·48 and H, 4·28%; PMR (80 MHz, $CDCl_3$, τ values), 7·26 (*dd*, 2H, J = 3, 16 Hz, *cis*-protons, C-3, F-3″), 6·86 (*m*, 2H, *trans*-protons, C-3, F-3″), 4·60 (*q*, 2H, J = 4, 12 Hz, C-2, F-2″), 2·65–2·83 (*m*, 3H, B-2′, 5′, 6′), 3·12 (*s*, 2H, E-2‴, 6‴), 3·30 (*d*, 1H, J = 8 Hz, D-5″), 3·48 (*d*, 1H, J = 8 Hz, D-6″), 3·64 (*d*, 1H, J = 8 Hz, A-5), 3·88 (*dd*, 1H, J = 2, 8 Hz, A-6), 3·80 (*d*, 1H, J = 2 Hz, A-8), 7·72 (*s*, 2 × 3H), 7·76 (*s*, 3 × 3H), 7·78 (*s*, 3H) [acetoxyl groups, A-7, B-4′, D-7″, E-3‴, 4‴, 5‴].

Bichalcone hexamethyl ether (9): A mixture of semecarpuflavanone (30 mg), freshly ignited potassium carbonate (0·5 gm) and dimethyl sulphate (0·3 ml) was refluxed in dry acetone (10 ml) on a water bath for several hours with occasional shaking. The inorganic salts were filtered off and washed well with hot acetone. The filtrate after solvent removal gave a residue which resisted crystallization from common organic solvents. On drying in a vacuum oven for six hours at 70°, 9 appeared as a pale yellow powder, $C_{36}H_{34}O_{10}$, m.p. 199–201°, yield: 25 mg. It furnished a red precipitate with antimony trichloride in carbon tetrachloride, a brown colour with neutral ferric chloride solution and was readily soluble in aqueous sodium hydroxide giving an orange-red solution. UV (EtOH): nm, 225, 381; + $AlCl_3$, 224, 436; + NaOAc, 223, 380; IR ν(nujol): 3460 (*br*, OH), 2830 (OCH_3), 1630 (chalcone carbonyl), 1595, 1580 (aromatic) cm^{-1}; Found: C, 68·67; H, 5·43; OCH_3, 29·52; $C_{36}H_{34}O_{10}$ requires C, 69·00; H, 5·47 and OCH_3, 29·71%; PMR (80 MHz, $CDCl_3$, τ values), 2·62–2·86 (*m*, 3H, B-2, 5, 6), 3·10 (*s*, 2H, -E-2″, 6″), 3·20 (*d*, 1H, J = 8 Hz, D-6‴), 3·36 (*d*, 1H, J = 8 Hz, D-5‴), 3·62 (*d*, 1H, J = 8 Hz, A-6′), 3·84 (*dd*, 1H, J = 2, 8 Hz, A-5′), 3·78 (*d*, 1H, J = 2 Hz, A-3′), 2·22

(*d*, 2H, J = 16 Hz, C-7, F-7″), 3·46 (*d*, 2H, J = 16 Hz, C-8, F-8″), −4·20 (*s*, 1H, chelated hydroxylic proton, A-2′), −4·30 (*s*, 1H, chelated hydroxylic proton, D-2‴), 6·20 (*s*, 2 × 3H), 6·26 (*s*, 4 × 3H) [methoxyl groups, A-4′, B-4, D-4‴, E-3″, 4″, 5″]; *m/z* (relative intensity) 626 $[M]^+$ (41·5), 611 $[M-15]^+$ (11·5), 596 $[M-2\times15]^+$ (21), 595 $[M-31]^+$ (7), 580 $[M-46]^+$ (7·5), 475 (10·5), 443 (9), 343 (10), 283 (9), 282 (8·5), 281 (26·5), 221 (14), 193 (50·5), 192 (44), 151 (100), 150 (76).

Dehydrogenation of semecarpuflavanone (6): Semecarpuflavanone (140 mg), potassium acetate (1·5 gm) and iodine (0·3 gm) in glacial acetic acid (10 ml) were heated at reflux temperature for three hours. The cooled mixture was poured into water and the reaction product was extracted with ethyl acetate. The solvent was removed under reduced pressure and a saturated solution of sodium metabisulphite (12 ml) was added to the residue to destroy excess iodine and the whole filtered. The filtrate was concentrated under vacuum when a yellow compound separated out, which has been designated as SA4.

Compound SA4 (10): It did not crystallize from normal organic solvents. Hence on drying in a vacuum oven for six hours at 80°, SA4 appeared as a light yellow powder, $C_{30}H_{18}O_{10}$, m.p. > 300°, yield: 105 mg. It gave a violet colour with neutral ferric chloride solution, a deep pink colour with magnesium-hydrochloric acid and is readily soluble in aqueous sodium hydroxide giving a deep orange-yellow solution; UV (EtOH): nm, 255, 302, 378; + NaOAc, 256, 327, 431; + $AlCl_3$, 255, 301, 379; IR ν(nujol): 3530–3290 (*br*, OH), 3170 (OH), 1685, 1680 (flavone carbonyls), 1600, 1585 (aromatic) cm^{-1}; Found: C, 66·65; H, 3·34; $C_{30}H_{18}O_{10}$, requires C, 66·92 and H, 3·37%; PMR (270 MHz, acetone-d_6, τ values), 2·28 (*q*, 1H, J = 2, 8 Hz, B-6′), 2·43 (*d*, 1H, J = 2Hz, B-2′), 2·84 (*d*, 1H, J = 8 Hz, B-5′), 2·90 (*d*, 1H, J = 2 Hz, E-6‴), 2·98 (*d*, 1H, J = 2 Hz, E-2‴), 3·14 (*d*, 1H, J = 8·5 Hz, D-5″), 3·34 (*d*, 1H, J = 8·5 Hz, D-6″), 3·48 (*s*, 2H, C-3, F-3″), 3·62 (*d*, 1H, J = 8 Hz, A-5), 3·74 (*d*, 1H, J = 2 Hz, A-8), 3·90 (*dd*, 1H, J = 2, 8 Hz, A-6), 1·54 (*s*, 1H), 2·14 (*s*, 2H), 2·39 (*s*, 3H) [non-chelated D_2O exchangeable hydroxylic protons, A-7, B-4′, D-7″, E-3‴, 4‴, 5‴].

SA4 hexamethyl ether (11): SA4 (80 mg) was dissolved in dry acetone (10 ml). Freshly ignited potassium carbonate (0·8 gm) and dimethyl sulphate (0·5 ml) were introduced and the mixture was refluxed on a water bath for several hours till the reaction product did not respond to the ferric reaction and then it was worked up as usual. The hexamethyl ether (11) appeared as light yellow crystals from a chloroform and methanol mixture, $C_{36}H_{30}O_{10}$, m.p. 190–91°, UV (EtOH): nm, 252, 301, 374; IR ν(nujol): 2830 (OCH_3), 1690, 1680 (flavone carbonyls), 1595, 1580 (aromatic) cm^{-1}; Found: C, 69·20; H, 4·83; OCH_3, 29·64; $C_{36}H_{30}O_{10}$ requires C, 69·45; H, 4·86 and OCH_3 29·90%; PMR (80 MHz, $CDCl_3$, τ values), 2·38–2·65 (*m*, 3H, B-2′, 5′, 6′), 2·92 (*s*, 2H, E-2‴, 6‴), 3·21 (*d*, 1H, J = 8 Hz, D-5″), 3·42 (*d*, 1H, J = 8 Hz, D-6″), 3·50 (*s*, 2H, C-3, F-3″), 3·59 (*d*, 1H, J = 8·5 Hz, A-5), 3·76 (*d*, 1H, J = 2 Hz, A-8), 3·88 (*dd*, 1H, J = 2, 8·5 Hz, A-6), 6·22 (*s*, 2 × 3H), 6·30 (*s*, 4 × 3H) [methoxyl groups, A-7, B-4′, D-7″, E-3‴, 4‴, 5‴]; *m/z* (relative intensity) 622 $[M]^+$ (82), 607 $[M-15]^+$ (38), 592 $[M-2\times15]^+$ (19·5), 591 $[M-31]^+$ (24), 576 $[M-46]^+$ (9), 560 $[M-2\times31]^+$ (6·5), 472 (11), 430 (10·5), 341 (8·5), 281 (7), 280 (30), 195 (70·5), 192 (78), 151 (100), 150 (47·5).

Oxidation of semecarpuflavanone (6) *with neutral permanganate*: A mixture of semecarpuflavanone (100 mg) and potassium permanganate (100 mg) in dry acetone (15 ml) was left at room temperature for one hour. The solvent was then evaporated and the

residue diluted with water. The manganous salts were decomposed with sulphur dioxide and extracted with ether. The ether extract was shaken with 1% aqueous sodium bicarbonate solution, acidified with dilute hydrochloric acid and again extracted with ether, dried and evaporated. The residue on crystallization from water afforded colourless needles, m.p. 250–51° (decomp.), yield: 14 mg; identical with an authentic sample of gallic acid (mixed m.p. and IR).

Oxidation of semecarpuflavanone pentamethyl ether (7) *with neutral permanganate*: A mixture of 7 (100 mg) and potassium permanganate (100 mg) in dry acetone (15 ml) was left at room temperature for one hour and worked up in the usual manner. The residue was extracted with hot benzene and concentrated, when a solid substance separated out. It appeared as colourless crystals from a chloroform and benzene mixture, m.p. 204°, yield: 12 mg; identical with authentic syringic acid (mixed m.p. and IR). The residual solid was crystallised from chloroform and methanol mixture as colourless needles, m.p. 155–56°, yield: 10 mg; identical with an authentic sample of 2-hydroxy-4-methoxybenzoic acid (mixed m.p. and IR).

Oxidation of SA4 hexamethyl ether (11) *with neutral permanganate*: Compound 11 (50 mg) was oxidised with neutral permanganate (50 mg) in dry acetone (10 ml) for one hour. After the usual working up of the reaction mixture, the residue on crystallization from a chloroform and methanol mixture appeared as colourless prisms, m.p. 172–73°, yield: 5 mg; identical with an authentic sample of gallic acid trimethyl ether (mixed m.p. and IR).

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