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ARYLALKANONES FROM *MYRISTICA DACTYLOIDES*

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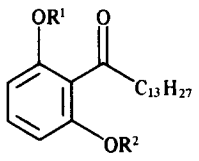
Key Word Index—*Myristica dactyloides*; Myristicaceae; malabaricones; arylalkanones.

Abstract—Four new arylalkanones isolated from the stem bark of *Myristica dactyloides* were identified as 1-(2-methoxy-6-hydroxyphenyl)tetradecan-1-one, 1-(2-methoxy-6-hydroxyphenyl)-9-(3′,4′-methylenedioxyphenyl)-nonan-1-one, 1-(2,6-dihydroxyphenyl)tetradecan-1-one and 1-(2-methoxy-6-hydroxyphenyl)-9-(4′-hydroxyphenyl)-nonan-1-one.

INTRODUCTION

Three species of *Myristica*; *M. ceylanica*, *M. dactyloides* and *M. fragrans* are found in Sri Lanka. *M. fragrans* is the nutmeg of commerce while *M. ceylanica* is endemic to Sri Lanka [1]. *M. fragrans* [3] and *M. malabarica* [4] have been used for many years in the folk medicine of several countries. *M. dactyloides* is a large tree found in the montane forests of Sri Lanka [1, 5]; decoctions of the bark and leaves are used in traditional medicine [5]. In this paper we report the isolation of seven arylalkanones **1–7** from this plant. The arylalkanones, 1-(2-methoxy-6-hydroxyphenyl)tetradecan-1-one **1**, 1-(2-methoxy-6-hydroxyphenyl)-9-(3′,4′-methylenedioxyphenyl)nonan-1-one **2**, 1-(2,6-dihydroxyphenyl) tetradecan-1-one **3**, and 1-(2-methoxy-6-hydroxyphenyl)-9-(4′-hydroxyphenyl) nonan-1-one **6** are new compounds while **4**, **5** and **7** have previously been isolated as malabaricones A, D and B



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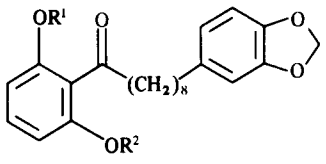
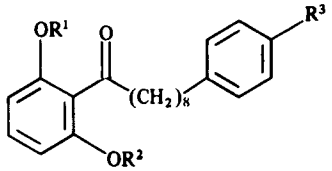
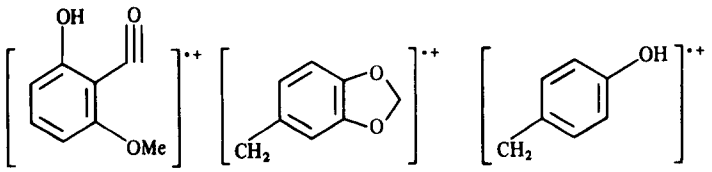
11 R^1 = R^2 = Ac



2 R^1 = H, R^2 = Me
5 R^1 = R^2 = H
13 R^1 = R^2 = Me



4 R^1 = R^2 = R^3 = H
6 R^1 = H, R^2 = Me, R^3 = OH
7 R^1 = R^2 = H, R^3 = OH
14 R^1 = H, R^2 = Me, R^3 = OMe
15 R^1 = R^2 = Me, R^3 = OMe



m/z 151 **8**

m/z 135 **10**

m/z 107 **11**

from the fruit rind of *M. malabarica* [4]. The only previous report on *M. dactyloides* describes the isolation of myo-inositol from a methanol extract of the stem bark [6]. Structurally related compounds which have been isolated previously include arylpropanoids from *M. fragrans* [7–10], diarylheptanoids such as curcuminoids from *Curcuma longa* [11] and gingerols from *Zingiber officinalis* [12] (Zingiberaceae) and lignans from *Horsfieldia iryaghedhi* [13, 14] (Myristicaceae).

RESULTS

Dried stem bark of *M. dactyloides* was collected at Hewaheta in the Central Province of Sri Lanka. The seven compounds **1–7** isolated by combined CC and prep. TLC from the hot dichloromethane extract of the stem bark were closely related and had a common 2,6-disubstituted acetophenone moiety with a polymethylene residue. In compounds **2**, **4–7**, a second benzene ring was linked to the 2,6-disubstituted acetophenone residue by the polymethylene chain.

The IR and ^{1}H NMR spectra of **1** ($C_{21}H_{34}O_3$) showed the presence of a conjugated carbonyl group (ν_{max} 1620 cm^{-1}), a single hydrogen bonded phenolic hydroxy group [δ_H 13.70 (1H, *s*) exchangeable with D_2O], a 1,2,3-trisubstituted benzene ring [δ_H 7.32 (1H, *t*, J = 8 Hz), 6.56 (1H, *d*, J = 8 Hz), 6.38 (1H, *d*, J = 8 Hz)], a methoxy group [δ_H 3.83 (3H, *s*)], an extended polymethylene side chain with one methylene group flanking the carbonyl group [δ_H 3.04 (2H, *t*, J = 7.5 Hz), δ_C 45.20 (*t*)] and a $C_{12}H_{25}$ alkyl residue [δ_H 1.60 (2H, *m*), 1.25 (23H, *br s*)]. A quartet at δ_C 14.3 indicated the unbranched nature of the polymethylene moiety. Hence it was deduced that compound **1** had the structure 1-(2-methoxy-6-hydroxyphenyl)tetradecan-1-one. Additional support for this structure was obtained from the mass spectrum which showed a base peak at m/z 151, attributed to the fragment ion **8** resulting from cleavage α-to the carbonyl group. Treatment of compound **1** with diazomethane gave a dimethyl derivative **9** ($C_{22}H_{36}O_3$), [δ_H 3.83 (3H, *s*), 3.72 (3H, *s*)].

Compound **2** ($C_{23}H_{28}O_5$) showed the presence of a phenolic hydroxy group [ν_{max} 3500–3510 cm^{-1}, δ_H 13.10, exchangeable with D_2O], a conjugated carbonyl group (ν_{max} 1620 cm^{-1}), a methoxy group [δ_H 3.83 (3H, *s*)], a –CO–CH_2 residue [δ_H 3.03 (2H, *t*, J = 7.5 Hz) δ_C 45.01 (*t*)], and a benzylic methylene group [δ_H 2.51 (2H, *t*, J = 7.5 Hz), δ_C 35.67 (*t*)]. NMR data also indicated the presence of a methylenedioxy residue [δ_H 5.90 (2H, *s*), δ_C 100.66 (*t*)] and six aromatic protons [δ_H 7.32 (1H), 6.66 (3H), 6.56 (1H), 6.38 (1H)]. This suggested the presence of two trisubstituted benzene rings, one of which was a 2,6-disubstituted acetophenone moiety as in compound **1** (Table 1). Mass spectral evidence permitted the assignment of the methylenedioxy residue to the aromatic ring, which underwent benzylic cleavage to give a very intense peak at m/z 135 probably due to ion **10**. The base peak at m/z 151 arose from α-cleavage of the carbonyl group as in compound **1**. Hence, the data available suggested that the two benzene rings were linked by the polymethylene residue. Spectral data and biogenetic considerations suggested that compound **2** had the structure, 1-(2-methoxy-6-hydroxyphenyl)-9-(3′,4′-methylenedioxyphenyl)nonan-1-one, which corresponds to the structure of the monomethyl ether of malabaricone D [4] previously isolated from *M. malabarica*.

^{1}H NMR and ^{13}C NMR data (Table 1) for compound **3** ($C_{20}H_{32}O_3$) were essentially the same as those for compound **1**, the only difference being the absence of a methoxy signal and the presence of two phenolic OH signals [δ_H 9.40 (2H, *s*, exchangeable with D_2O] in compound **3**. Acetylation yielded a diacetate **11** [($C_{24}H_{36}O_5$) δ_H 2.20 (6H, *s*)]. Hence compound **3** was assigned the structure 1-(2,6-dihydroxyphenyl)-tetradecan-1-one. The base peak at m/z 137 in its mass spectrum, analogous to the peak at m/z 151 in compound

Table 1. ^{13}C NMR chemical shifts (δ) of arylalkanones **1–7***

Carbon	1	2	3	4†	5	6	7
1	111.43 (*s*)	110.70 (*s*)	110.80 (*s*)	110.11 (*s*)	110.73 (*s*)	111.38 (*s*)	110.75 (*s*)
2	161.43 (*s*)	161.27 (*s*)	162.82 (*s*)	161.15 (*s*)	161.24 (*s*)	161.47 (*s*)	161.47 (*s*)
3	101.34 (*d*)	101.16 (*d*)	108.00 (*d*)	108.50 (*d*)	108.58 (*d*)	101.45 (*d*)	106.95 (*d*)
4	135.80 (*d*)	135.67 (*d*)	136.39 (*d*)	135.72 (*d*)	135.73 (*d*)	135.91 (*d*)	134.97 (*d*)
5	111.04 (*d*)	110.84 (*d*)	108.01 (*d*)	108.50 (*d*)	108.58 (*d*)	110.98 (*d*)	106.95 (*d*)
6	164.85 (*s*)	164.65 (*s*)	162.82 (*s*)	161.15 (*s*)	161.24 (*s*)	161.69 (*s*)	161.47 (*s*)
1′	—	147.42 (*s*)	—	142.90 (*s*)	147.55 (*s*)	135.15 (*s*)	132.86 (*s*)
2′	—	107.99 (*d*)	—	128.25 (*d*)	108.17 (*d*)	129.53 (*d*)	128.48 (*d*)
3′	—	136.75 (*s*)	—	128.43 (*d*)	136.96 (*s*)	115.25 (*d*)	114.38 (*d*)
4′	—	136.75 (*s*)	—	125.57 (*d*)	136.96 (*s*)	153.63 (*s*)	154.33 (*s*)
5′	—	108.83 (*d*)	—	128.43 (*d*)	109.00 (*d*)	115.25 (*d*)	114.38 (*d*)
6′	—	121.00 (*d*)	—	128.25 (*d*)	121.22 (*d*)	129.53 (*d*)	128.48 (*d*)
$CH_2\underline{Me}$	14.25 (*q*)	—	14.30 (*q*)	—	—	—	—
OCH_2O	—	100.66 (*t*)	—	—	100.80 (*t*)	—	—
CO	208.61 (*s*)	207.90 (*s*)	209.50 (*s*)	208.12 (*s*)	207.98 (*s*)	208.27 (*s*)	207.34 (*s*)
$CO\underline{CH_2}$	45.20 (*t*)	45.01 (*t*)	45.35 (*t*)	44.80 (*t*)	44.88 (*t*)	45.17 (*t*)	43.94 (*t*)
$Ar\underline{CH_2}$	—	35.67 (*t*)	—	35.98 (*t*)	35.75 (*t*)	35.16 (*t*)	34.29 (*t*)
OMe	55.72 (*q*)	55.61 (*q*)	—	—	—	55.76 (*q*)	—

*Relative to TMS (int. standard).

†Reported in ref. [4].

1 provided further evidence for the structure of compound **3**. Methylation of compound **3** with CH_2N_2 gave a mixture of **1** and the dimethyl ether **9**.

IR, ^1H NMR and ^{13}C NMR spectra of compound **6** ($C_{22}H_{28}O_4$) suggested that it contained a conjugated carbonyl group (ν_{max} 1620 cm^{-1}), two phenolic OH groups, one of which was chelated (ν_{max} 3600–3100 cm^{-1}), δ_H 13.35 and 5.18 (each 1H, *s*, exchangeable with D_2O)], a methoxy group [δ_H 3.87 (3H, *s*), δ_C 55.76 (*q*)], eight methylene groups with one flanking the carbonyl group [δ_H 3.04 (2H, *t*, J = 7.5 Hz), δ_C 45.17 (*t*)], and one benzylic methylene group [δ_H 2.52 (2H, *t*, J = 7.5 Hz), δ_C 35.16 (*t*)]. The presence of two benzene rings, one of which was a 2,6-di-*O*-substituted acetophenone residue was also indicated (Table 1). The second benzene ring was deduced to be 1-alkyl-4-hydroxy substituted [δ_H 7.02 and 6.74 (both 2H, *d*, J = 8.0 Hz), δ_C 153.63 (*s*), 135.15 (*s*), 129.53 (*d*), 115.25 (*d*)]. This suggested that the two benzene rings were linked by the polymethylene chain as in compound **4**, and that the arylalkanone **6** had the structure, 1-(2-methoxy-6-hydroxyphenyl)-9-(4′-hydroxyphenyl)nonan-1-one. The presence of the fragment ions at m/z 151 (100%) due to α-cleavage of the carbonyl group and m/z 107 (29%) due to benzylic cleavage in the mass spectrum provided additional support for this structure.

Comparisons of the physical and spectroscopic data showed that compounds **4**, **5** and **7** were identical to malabaricones A, D and B previously isolated from *M. malabarica* [4]. The structural relationships between the different arylalkanones were clearly indicated by the analyses of the products of methylation with diazomethane (Table 2).

EXPERIMENTAL

Mps: uncorr. Identities of compounds were established by mmp, co-TLC, IR and ^1H NMR comparison. Petrol refers to the fraction having bp 60–80°, Prep. TLC and medium pressure column chromatography (MPCC) were carried out on Merck Kieselgel 60 $PF_{254+366}$ and Merck Kieselgel ASTM 200, respectively. IR spectra were recorded in KBr discs. ^1H NMR spectra were recorded at 100 MHz in $CDCl_3$ with TMS as int st unless otherwise stated. MS were recorded at the Research School of Chemistry, The Australian National University. Methylations were carried out with CH_2N_2–Et_2O at 27° for 12 hr followed by separation by prep. TLC.

Extraction and isolation. Dried and powdered stem bark of *M. dactyloides* (1.2 kg) was extd successively with hot petrol, hot CH_2Cl_2 and hot MeOH. The CH_2Cl_2 ext (48 g) was washed with 5% NaOH, acidified with 2M HCl and extd × 3 with Et_2O. The dried Et_2O ext (15.2 g) was chromatographed on silica gel using petrol–EtOAc mixts as eluents.

Table 2. Methylation products of arylalkanones **1**–**7** (CH_2N_2–ether)

Arylalkanone	Product(s)
1	**9**
3	**1** + **9**
2	**13**
5	**2** + **13**
6	**14** + **15**
7	**14** + **15**

Elution with petrol–EtOAc (17:3) gave a yellow solid (380 mg), which on separation by MPCC and prep. TLC gave the less polar 1-(2-methoxy-6-hydroxyphenyl)tetradecan-1-one (**1**: 230 mg), needles from MeOH, mp 51–52°; $[M]^+$ 334.2510. $C_{21}H_{34}O_3$ requires $[M]^+$ 334.2508; IR ν_{max} 2910, 2850, 1620, 1590, 1450, 1375, 1235, 1180, 1090, 825, 780 and 710 cm^{-1}; δ_H 13.70 (1H, *s*, D_2O exchangeable), 7.32 (1H, *t*, J = 8 Hz), 6.56 and 6.38 (each 1H, *d*, J = 8 Hz), 3.83 (3H, *s*) 3.04 (2H, *t*, J = 7.5 Hz), 1.60 (2H, *m*), 1.26 (23H, *br s*); MS m/z (rel. int.): 334 (9) $[M]^+$, 316 (7),179 (28), 166 (21), 151 (100) and 137 (5), and the polar 1-(2-methoxy-6-hydroxyphenyl)-9-(3′,4′-methylenedioxyphenyl)nonan-1-one (**2**: 70 mg), yellow crystls from $CHCl_3$–MeOH, mp 51–52°; $[M]^+$ 384.1936. $C_{23}H_{28}O_5$ requires $[M]^+$ 384.1937; IR ν_{max} 3500–3100, 2900, 2800, 1620, 1580, 1500, 1450, 1380, 1240, 1180, 1090, 1030, 935, 790 and 715 cm^{-1}; δ_H (300 MHz) 13.10 (1H, *s*, D_2O exchangeable), 7.32 (1H, *t*, J = 8 Hz), 6.71 (1H, *d*, J = 8 Hz), 6.67 (1H, *s*), 6.61, 6.56 and 6.38 (each 1H, *d*, J = 8 Hz), 5.90 (2H, *s*), 3.88 (3H, *s*), 3.03 and 2.51 (each 2H, *t*, J = 7.5 Hz), 1.81 (4H, *m*), 1.33 (8H, *br s*); MS m/z (rel. int.): 384 (88) $[M]^+$, 218 (14), 192 (12), 179 (44), 151 (100), 137 (11), 136 (30) and 135 (83).

Elution with petrol–EtOAc (7:3) gave a yellow solid (1.6 g) which was separated by MPCC and prep. TLC into the least polar 1-(2,6-dihydroxyphenyl)-tetradecan-1-one (**3**: 540 mg), pale yellow needles from CH_2Cl_2–petrol, mp 91–92°; $[M]^+$ 320.2334. $C_{20}H_{32}O_3$ requires $[M]^+$ 320.2351; IR ν_{max} 3500–3000, 2930, 2850, 1630, 1600, 1450, 1245, 1035, 960, 790 and 715 cm^{-1}; δ_H 9.40 (2H, *s*, D_2O exchangeable), 7.18 (1H, *t*, J = 8 Hz), 6.32 (2H, *d*, J = 8 Hz), 3.12 (2H, *t*, J = 7.5 Hz), 1.60 (2H, *m*), 1.25 (23H, *br s*); MS m/z (rel. int.): 320 (11) $[M]^+$, 302 (13), 189 (11), 165 (28), 152 (29), and 137 (100), 1-(2,6-dihydroxyphenyl)-9-phenylnonan-1-one (malabaricone A) (**4**: 480 mg) bright yellow crystals from Et_2O–hexane, mp 80–81° (lit. [4] 81–82°), and the most polar 1-(2,6-dihydroxyphenyl)-9-(3′,4′-methylenedioxyphenyl)nonan-1-one (malabaricone D) (**5**: 86 mg), pale yellow crystals from CH_2Cl_2–hexane, mp 86–87° (lit. [4] 90–91°).

Elution with petrol–EtOAc (11:9) gave on purification by prep. TLC and recrystallization from Me_2CO–hexane, pale yellow crystals of 1-(2-methoxy-6-hydroxyphenyl)-9-(4′-hydroxyphenyl)nonan-1-one (**6**: 1.6 g), mp 65–66°; $[M]^+$ 356.1987. $C_{22}H_{28}O_4$ requires $[M]^+$ 356.1988; IR ν_{max} 3600–3100, 2895, 2850, 1620, 1590, 1510, 1460, 1240, 1090, 830, 780 and 720 cm^{-1}; δ_H 13.35 (1H, *s*, D_2O exchangeable), 7.32 (1H, *t*, J = 8 Hz), 7.02 and 6.74 (each 2H, *d*, J = 8 Hz), 6.58 and 6.38 (each 1H, *d*, J = 8 Hz), *s*, 5.18 (1H, *s*, D_2O exchangeable), 3.87 (3H, *s*), 3.04 and 2.52 (each 2H, *t*, J = 7.5 Hz), 1.60 (4H, *m*), 1.32 (8H, *br s*); MS m/z (rel. int.): 356 (6) $[M]^+$, 179 (24), 166 (12), 151 (100), 137 (6) and 107 (29).

Elution with petrol–EtOAc (1:1) gave, on purification by prep. TLC and crystallization from Me_2CO–hexane, 1-(2,6-dihydroxyphenyl)-9-(4′-hydroxyphenyl)nonan-1-one (malabaricone B) (**7**: 240 mg), mp 105–106° (lit. [4] 102°).

Methylation of (**1**). Compound **1** (30 mg) on methylation gave 1-(2,6-dimethoxyphenyl)tetradecan-1-one (**9**: 11 mg $[M]^+$ 348.2665. $C_{22}H_{36}O_3$ requires $[M]^+$ 348.2664; IR ν_{max} 2900, 1710, 1600, 1470, 1110, and 770 cm^{-1}; δ_H (60 MHz) 7.08 (1H, *t*, J = 8 Hz) 6.51 and 6.18 (each 1H, *d*, J = 8 Hz), 3.83 (3H, *s*), 3.72 (3H, *s*), 2.54 (2H, *t*, J = 7.5 Hz), 1.65 (2H, *m*), 1.25 (23H, *br s*); MS m/z (rel. int.): 348 (19) $[M]^+$, 211 (14), 153 (21), 151 (67), 138 (79), 137 (100) and 107 (25).

Methylation of (**2**). Compound **2** (25 mg) on methylation gave 1-(2,6-dimethoxyphenyl)-9-(3′,4′-methylenedioxyphenyl)nonan-1-one (**13**: 8.2 mg); IR ν_{max} (film) 2925, 1725, 1595, 1475, 1350, 1220, 1190, 1015, 920, 800, 760 and 710 cm^{-1}; δ_H 7.00 (1H, *t*, J = 8 Hz), 6.57 (3H, *m*), 6.33 (2H, *d*, J = 8 Hz), 5.85 (2H, *s*), 3.81 (3H,

s), 3.67 (3H, *s*), 2.46 (4H, *m*), 1.48 (4H, *m*), 1.25 (8H, *br s*); MS *m/z* (rel. int.): 398 (69) $[M]^+$, 218 (16), 193 (28), 179 (11), 165 (100), 151 (96), 135 (91), 121 (38), and 107 (21),

Methylation of (**3**). Compound **3** (50 mg) on methylation gave two products identical with compounds (**1**) (21 mg) and (**9**) (16 mg).

Acetylation of (**3**). Compound **3** (30 mg) on reaction with pyridine (0.5 ml) and Ac_2O (1 ml) at 70° for 12 hr gave 1-(2,6-diacetoxyphenyl)tetradecan-1-one (**11**: 28 mg), $[M]^+$ 404.2564 $C_{24}H_{36}O_5$ requires $[M]^+$ 404.2563; IR ν_{max} 2950, 1770, 1700, 1605, 1460, 1365, 1180, and 1030 cm^{-1}; δ_H 7.18 (3H, *m*) 2.64 (2H, *t*, $J = 7.5$ Hz), 2.20 (6H, *s*), 1.65 (2H, *m*), 1.25 (23H, *br s*); MS *m/z* (rel. int.): 403 (3) $[M]^+$, 302 (14), 189 (17), 165 (67), 152 (59), and 137 (100).

Methylation of (**5**). Compound **5** (50 mg) on methylation gave two products identical with compounds (**2**) (21 mg) and (**13**) (12 mg).

Methylation of (**6**). Compound **6** (50 mg) on methylation gave 1-(2-methoxy-6-hydroxyphenyl)-9-(4′-methoxyphenyl)nonan-1-one (**14**: 23 mg); $[M]^+$ 370.2142. $C_{23}H_{30}O_4$ requires $[M]^+$ 370.2144; IR ν_{max}(film) 2925, 1730, 1625, 1600, 1510, 1460, 1375, 1235, 1185, 1090, 1045 and 715 cm^{-1}; δ_H 13.20 (1H, *s*), 7.31 (1H, *t*, $J = 8$ Hz), 7.08 and 6.80 (each 2H, *d*, $J = 8$ Hz), 6.57 and 6.38 (each 1H, *d*, $J = 8$ Hz), 3.88 and 3.78 (each 3H, *s*), 3.03 and 2.55 (each 2H, *t*, $J = 7.5$ Hz), 1.65 (4H, *m*), 1.32 (8H, *br s*); MS *m/z* (rel. int.): 370 (23) $[M]^+$, 179 (27), 151 (100), 149 (55), 147 (10), and 121 (87), and 1-(2,6-dimethoxyphenyl)-9-(4′-methoxyphenyl)nonan-1-one (**15**: 16 mg); $[M]^+$ 384.2297. $C_{24}H_{32}O_4$ requires $[M]^+$ 384.2301; IR ν_{max} (film) 2900, 1700, 1595, 1510, 1460, 1375, 1245, 1110 and 720 cm^{-1}; δ_H 7.08 (3H, *m*), 6.80 and 6.57 (each 2H, *d*, $J = 8$ Hz), 3.78 (9H, *s*), 2.56 and 1.64 (each 4H, *m*), 1.32 (8H, *br s*); MS *m/z* (rel. int.): 384 (9) $[M]^+$, 274 (10), 166 (15), 165 (93), 152 (50), 151 (100), 137 (15), 121 (87), and 107 (11).

Methylation of compound (**7**). Compound **7** (40 mg) on methylation gave two products identical with the Me ethers (**14**) (23 mg) and (**15**) (12 mg).

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