Sesquiterpene Phenylpropanoid and Sesquiterpene Chromone Derivatives from *Ferula pallida*

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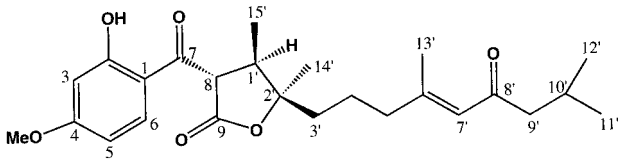
Four novel compounds—two sesquiterpene phenylpropanoid derivatives, pallidones G (**1**) and H (**2**), and two sesquiterpene chromone derivatives, pallidones I (**3**) and J (**4**)—have been isolated from the roots of *Ferula pallida*. Their structures were determined on the basis of NMR and HREIMS evidence.

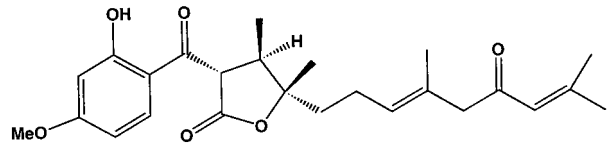
Sesquiterpene coumarins and sesquiterpene phenylpropanoid derivatives, pallidones A−F, and the two known compounds, feselol and conferol, have been reported from *Ferula pallida* (Umbelliferae).[1] In a continued study of the chemical constituents of the same plant, we report here the isolation and structure elucidation of four additional novel compounds, two sesquiterpene phenylpropanoid derivatives, pallidones G (**1**) and H (**2**), and two sesquiterpene chromone derivatives, pallidones I (**3**) and J (**4**).

An EtOAc extract of the roots of *F. pallida* was fractioned using repeated Si gel column chromatography, HPLC, and gel permeation chromatography (GPC) to afford compounds **1**−**4**. Compound **1** was obtained as a colorless oil, and its HREIMS indicated a molecular formula of $C_{25}H_{34}O_6$. ^{1}H and ^{13}C NMR data of **1** were very similar to those of pallidones C−F.[1] The double bond and the carbonyl group of the sesquiterpene unit were conjugated according to their NMR spectral data (δ_H 6.07, br s, H-7′; δ_C 201.5, C-8′; δ_C 157.0, C-6′; δ_C 124.2, C-7′). This was confirmed by the correlations of H-7′ with C-8′, C-9′, C-6′, and C-13′ and of H-9′ with C-8′, C-7′, C-11′, and C-12′ in the HMBC spectrum of **1**. The double bond at C-6′ has an *E* configuration based on the chemical shifts of C-5′ (δ_C 41.3) and C-13′ (δ_C 19.4).[2−4] The relative configuration was determined as 8*S**, 1′*R**, and 2′*R** by comparison of the ^{13}C NMR data with those of pallidones C−F[1] and confirmed by the correlations of H-3′ with H-15′ and H-8 and of H-1′ with H-14′ in the NOESY spectrum. All of the ^{1}H and ^{13}C NMR assignments of **1** were made on the basis of ^{1}H−^{1}H COSY, HSQC, HMBC, and NOESY data.

The HREIMS of **2** gave an intense parent ion peak, corresponding to a molecular formula of $C_{25}H_{32}O_6$. Its ^{1}H and ^{13}C NMR data were also very similar to those of pallidones C−F.[1] Both the ^{1}H and ^{13}C NMR data of **2** indicated two double bonds. In the ^{1}H NMR spectrum of **2**, 11′-CH_3 and 12′-CH_3 were singlets, indicating that C-10′ was a quaternary carbon. In the HMBC spectrum of **2**, the correlations of H-11′ with C-9′, C-10′, and C-12′; H-12′ with C-9′, C-10′, and C-11′; H-9′ with C-10′, C-11′, C-12′, C-8′, and C-7′; and H-7′ with C-9′, C-8′, C-6′, C-5′, and C-13′ indicated that the two double bonds were between C-5′,C-6′ and C-9′,C-10′, respectively. The C-5′,C-6′ double bond was 5′*E*, based on the chemical shifts of C-4′ (δ_C 22.6) and C-13′ (δ_C 16.6).[2−4] The relative configuration was determined as 8*S**, 1′*R**, and 2′*S** by comparison of the ^{13}C NMR data with those of pallidones C−F′ and confirmed by the correlations of H-8 with H-15′ and H-14′ and of H-1′ with H-3′ in the NOESY spectrum. All of the ^{1}H and ^{13}C NMR signals of **2** were assigned on the basis of ^{1}H−^{1}H COSY, HSQC, HMBC, and NOESY experiments.



1



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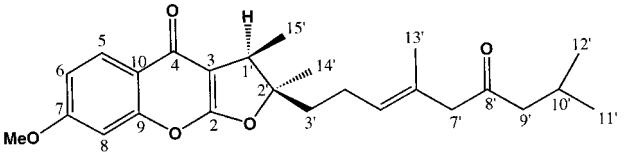

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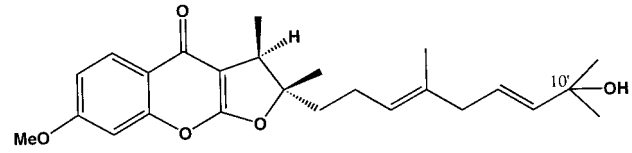

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The HREIMS of **3** indicated a molecular formula of $C_{25}H_{32}O_5$. Its ^{1}H NMR spectrum displayed an ABX system typical of an aromatic ring, a methoxy group, and other signals similar to those of the sesquiterpene unit of pallidone E.[1] The chemical shift of H-5 (δ 8.12) showed a

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significant downfield shift when compared to the chemical shift of H-5 in pallidones A (δ 7.63) and B (δ 7.54).[1] This suggested that **3** was a chromone derivative.[5−7] Its ^{13}C NMR data were consistent with the above analysis, and the carbonyl signal at δ 175.3 (C-4) confirmed that **3** was a chromone-type derivative. In the HMBC spectrum of **3**, the correlations of H-5 with C-4, C-7, and C-9; OMe with C-6, C-7, and C-8; H-15′ with C-1′, C-2′, and C-3; and H-1′ with C-4, C-2, C-14′, and C-3′ confirmed that C-1′ of the sesquiterpene unit was attached to C-3 of the chromone and that the methoxy group was attached to C-7. On the basis of the above analysis, and combined with the ^{13}C NMR spectral data ($\delta_{C-2'}$ 95.2, δ_{C-2} 167.1, DEPT spectrum indicated both C-2′ and C-2 were quaternary carbons) and the molecular formula of **3**, it was deduced that C-2 and C-2′ were linked by an oxygen atom.

The configuration of the double bond of the sesquiterpene unit was determined to be 5′*E* based on the ^{13}C NMR spectral data (δ_C 16.6, C-13′).[2−4] In the NOESY spectrum of **3**, the correlations of H-5′ with H-7′; H-13′ with H-4′; H-1′ with H-14′; and H-15′ with H-3′ suggested not only that the double bond was 5′*E*, but also that the relative stereochemistry of **3** was 1′*R** and 2′*R**. All of the ^{1}H and ^{13}C NMR signals of **3** were assigned according to the correlations observed in ^{1}H−^{1}H COSY, HSQC, HMBC, and NOESY spectra. Thus, the structure of pallidone I was determined as shown.

The NMR data of **4** were very similar to those of **3**, and its HREIMS suggested a molecular formula of $C_{25}H_{32}O_5$. The evident differences between compounds **3** and **4** were in the sesquiterpene units. ^{1}H and ^{13}C NMR indicated that the sesquiterpene unit of **4** bore two double bonds. ^{1}H NMR spectrum of **4** showed a signal for the terminal methyls (11′ and 12′), while a corresponding doublet was observed for compound **3**. This suggested that C-10′ of **4** was a quaternary carbon, in agreement with the ^{13}C NMR data. In the HMBC spectrum, the correlations of H-5 with C-4, C-7, and C-9; OMe with C-6, C-7, and C-8; H-15′ with C-1′, C-2′, and C-3; H-1′ with C-4, C-2, C-14′, and C-3′; and H-11′ and H-12′ with C-10′ and C-9′ suggested that C-1′ of the sesquiterpene unit was attached to C-3 of the chromone, and the methoxy group was attached to C-7. Furthermore, C-10′ was oxygenated, by a hydroxyl group, and the second double bond was C-8′ and C-9′. The existence of a hydroxyl group at C-10′ was suggested further by the fragments at *m*/*z* 394 and 354 in the EIMS. The configuration of the double bond between C-5′ and C-6′ was determined to be 5′*E*, based on the ^{13}C NMR data (δ_C 16.2, C-13′).[2−4] The other double bond should be 8′*E*, considering the coupling constant of H-9′ ($J_{9',8'}$ = 15.6 Hz). In the NOESY spectrum of **4**, correlations of H-7′ with H-5′ and H-9′; H-1′ with H-3′; and H-15′ with H-14′ confirmed the configurations of the double bonds and indicated that the relative stereochemistry of **4** was 1′*R** and 2′*S**. All of the ^{1}H and ^{13}C NMR signals of **4** were assigned using ^{1}H−^{1}H COSY, HSQC, HMBC, and NOESY spectra. Thus, the structure of **4** is as shown.

The known sesquiterpene coumarins, conferone,[8,9] badrakemin,[10−12] samarcandin acetate,[13] and isosamarcandin angelate,[14] were also isolated and identified by comparison of their spectral data with those published in the literature. The structures of these known compounds were also confirmed by 2D NMR (^{1}H−^{1}H COSY, HSQC, HMBC, and NOESY).

Experimental Section

General Experimental Procedures and Plant Material. These were as described previously.[1]

Extraction and Isolation. Powdered, air-dried roots (1.6 kg) of *F. pallida* were extracted with *n*-hexane (7 L), EtOAc (7 L), and MeOH (7 L) at 60 °C, three times, using a Soxhlet extraction apparatus, 24 h each time. The EtOAc extract (55 g) was chromatographed over a Si gel column (10 × 50 cm, Merck Si gel 60, 1.2 kg) eluted with *n*-hexane−EtOAc (10:1 to 1:1, then EtOAc). Ten fractions were obtained. Fraction 5 (300 mg) was chromatographed using HPLC (silica, hexane−EtOAc, 6:1), and 14 fractions were obtained (fractions 5.01−5.14). Compounds **1** (11 mg), **2** (11 mg), and conferone (57 mg) were obtained after further fractionation of fractions 5.03, 5.06, and 5.14 by GPC ($CHCl_3$), respectively. Fraction 7 (400 mg) separated by HPLC (silica, hexane−EtOAc, 3:1), yielded 13 fractions from which **3** (12 mg), badrakemin (9 mg), and isosamarcandin angelate (4 mg) were obtained. Fraction 9 (400 mg) was further separated by HPLC (silica, hexane−EtOAc, 2:1) to give samarcandin acetate (172 mg). Fraction 10 (300 mg) was further purified by HPLC (silica, hexane−EtOAc, 2:1), to give **4** (10 mg).

Pallidone G (1): $[\alpha]^{24}_D$ +10.5° (*c* 0.96, $CHCl_3$); UV ($CHCl_3$) λ_{max} (log ϵ) 241.6 (4.12), 285.2 (4.03), 319.6 (3.83) nm; IR (KBr) ν_{max} 3457, 2961, 1769, 1626, 1513, 1459, 1377, 1215, 620 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 12.44 (1H, s, Ar−OH), 7.68 (1H, d, *J* = 9.1 Hz, H-6), 6.52 (1H, dd, *J* = 9.1, 2.4 Hz, H-5), 6.46 (1H, d, *J* = 2.4 Hz, H-3), 6.07 (1H, br s, H-7′), 4.24 (1H, d, *J* = 12.0 Hz, H-8), 3.87 (3H, s, OMe), 3.12 (1H, dq, *J* = 12.0, 6.8 Hz, H-1′), 2.31 (2H, d, *J* = 6.8 Hz, H-9′), 2.17 (1H, m, H-10′), 2.14 (3H, br s, H-13′), 1.62−1.68 (2H, m, H-4′), 1.59 (2H, m, H-3′), 1.52 (3H, s, H-14′), 1.08 (3H, d, *J* = 6.8 Hz, H-15′), 0.93 (6H, d, *J* = 6.8 Hz, H-11′ and H-12′); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 201.5 (s, C-8′), 195.9 (s, C-7), 171.2 (s, C-9), 167.2 (s, C-4), 166.5 (s, C-2), 157.0 (s, C-6′), 133.1 (d, C-6), 124.2 (d, C-7′), 114.1 (s, C-1), 108.6 (d, C-5), 101.1 (d, C-3), 87.5 (s, C-2′), 55.9 (q, OMe), 54.6 (d, C-8), 53.7 (t, C-9′), 44.1 (d, C-1′), 41.3 (t, C-5′), 34.9 (t, C-3′), 25.4 (d, C-10′), 24.0 (q, C-14′), 22.8 (q, C-11′ and C-12′), 21.4 (t, C-4′), 19.4 (q, C-13′), 12.9 (q, C-15′); EIMS *m*/*z* 430 [M]$^+$ (32.2), 373 (11.4), 263 (7.4), 221 (6.8), 220 (5.2), 210 (17.4), 193 (7.6), 192 (13.4), 177 (7.7), 152 (10.8), 151 (100), 95 (21.2), 85 (7.0), 83 (5.0), 69 (8.0), 67 (7.1), 57 (8.0), 43 (10.8), 41 (10.6); HREIMS *m*/*z* 430.2352 (calcd for $C_{25}H_{34}O_6$, 430.2355).

Pallidone H (2): $[\alpha]^{24}_D$ +7.0° (*c* 0.65, $CHCl_3$); UV ($CHCl_3$) λ_{max} (log ϵ) 241.4 (4.10), 285.0 (4.04), 319.0 (3.79) nm; IR (KBr) ν_{max} 3438, 2982, 2915, 2363, 1768, 1690, 1629, 1513, 1447, 1380, 1282, 1231, 1051, 962, 805, 619 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 12.47 (1H, s, Ar−OH), 7.69 (1H, d, *J* = 9.0 Hz, H-6), 6.52 (1H, dd, *J* = 9.0, 2.4 Hz, H-5), 6.46 (1H, d, *J* = 2.4 Hz, H-3), 6.11 (1H, s, H-9′), 5.26 (1H, t, *J* = 6.7 Hz, H-5′), 4.24 (1H, d, *J* = 12.0 Hz, H-8), 3.87 (3H, s, OMe), 3.13 (1H, dq, *J* = 12.0, 6.8 Hz, H-1′), 3.06 (2H, s, H-7′), 2.26 (1H, m, H-2′a), 2.15 (3H, s, H-11′), 1.86 (3H, s, H-12′), 1.82 (2H, m, H-3′), 1.66 (3H, s, H-13′), 1.36 (3H, s, H-14′), 1.08 (3H, d, *J* = 6.8 Hz, H-15′), 0.94 (1H, m, H-2′b); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 199.2 (s, C-8′), 196.0 (s, C-7), 171.1 (s, C-9), 167.1 (s, C-4), 166.4 (s, C-2), 156.0 (s, C-10′), 133.0 (d, C-6), 131.0 (s, C-6′), 127.8 (d, C-5′), 123.0 (d, C-9′), 114.0 (s, C-1), 108.4 (d, C-5), 101.0 (d, C-3), 87.7 (s, C-2′), 55.8 (q, OMe), 55.3 (t, C-7′), 54.6 (d, C-8), 41.3 (d, C-1′), 39.5 (t, C-3′), 27.8 (q, C-12′), 22.6 (t, C-4′), 20.8 (q, C-11′), 20.6 (q, C-14′), 16.6 (q, C-13′), 13.5 (q, C-15′); EIMS *m*/*z* 428 [M]$^+$ (41.0), 346 (5.5), 345 (11.1), 262 (7.7), 245 (16.4), 219 (9.2), 192 (5.1), 177 (7.1), 152 (11.3), 151 (100), 136 (6.1), 135 (7.9), 108 (8.7), 107 (6.2), 95 (10.1), 85 (5.7), 84 (12.4), 83 (137.5), 69 (7.7), 67 (5.9), 57 (5.6), 55 (29.0), 43 (11.9), 41 (10.9), 39 (6.2); HREIMS *m*/*z* 428.2171 (calcd for $C_{25}H_{32}O_6$, 428.2199).

Pallidone I (3): $[\alpha]^{24}_D$ +3.1° (*c* 0.97, $CHCl_3$); UV ($CHCl_3$) λ_{max} (log ϵ) 243.8 (4.28), 285.6 (4.41) nm; IR (KBr) ν_{max} 2354, 1715, 1626, 1556, 1446, 1353, 1253, 1151, 1098, 984, 845, 680, 476 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.12 (1H, d, *J* = 8.9 Hz, H-5), 6.97 (1H, dd, *J* = 8.9, 2.2 Hz, H-6), 6.84 (1H, d, *J* = 2.2 Hz, H-8), 5.29 (1H, t, *J* = 6.9 Hz, H-5′), 3.89 (3H, s, OMe), 3.31 (1H, q, *J* = 6.9 Hz, H-1′), 3.05 (2H, s, H-7′), 2.31 (2H, d, *J* = 6.9 Hz, H-9′), 2.27 (1H, m, H-4′a), 2.14 (1H, m, H-10′), 1.83−1.97 (2H, m, H-3′), 1.70 (1H, m, H-4′b), 1.67 (3H, s, H-13′), 1.52 (3H, s, H-14′), 1.33 (3H, d, *J* = 6.9 Hz, H-15′), 0.91 (6H, d, *J* = 6.6 Hz, H-11′ and H-12′); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 209.3 (s, C-8′), 175.3 (s, C-4), 167.1 (s, C-2), 163.1

(s, C-7), 155.0 (s, C-9), 130.1 (s, C-6′), 128.5 (d, C-5′), 126.9 (d, C-5), 117.9 (s, C-10), 113.3 (d, C-6), 101.0 (d, C-8), 99.1 (s, C-3), 95.2 (s, C-2′), 55.9 (q, OMe), 54.3 (t, C-7′), 50.9 (t, C-9′), 43.4 (d, C-1′), 34.8 (t, C-3′), 25.4 (q, C-14′), 24.5 (d, C-10′), 22.9 (t, C-4′), 22.6 (q, C-11′ and C-12′), 16.6 (q, C-13′), 14.1 (q, C-15′); EIMS *m*/*z* 412 $[M]^+$ (42.3), 342 (6.3), 341 (6.6), 328 (21.5), 327 (66.8), 313 (12.3), 285 (9.3), 273 (23.0), 271 (12.2), 260 (23.4), 259 (59.1), 257 (20.5), 245 (86.5), 244 (81.3), 220 (42.0), 219 (97.1), 205 (25.9), 193 (19.0), 151 (100), 136 (20.8), 135 (43.2), 122 (19.0), 121 (15.9), 120 (18.4), 109 (24.1), 107 (17.2), 95 (15.2), 85 (63.4), 79 (14.5), 69 (13.2), 57 (79.9), 43 (40.4), 41 (44.4), 39 (12.9); HREIMS *m*/*z* 412.2246 (calcd for $C_{25}H_{32}O_5$, 412.2250).

Pallidone J (4): $[\alpha]^{24}_D$ +4.1° (*c* 1.13, $CHCl_3$); UV ($CHCl_3$) λ_{max} (log ϵ) 243.4 (4.24), 285.8 (4.29) nm; IR (KBr) ν_{max} 3412, 2979, 2931, 2363, 1716, 1626, 1556, 1446, 1355, 1253, 1153, 1097, 1033, 845, 775, 716, 605, 475 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 8.11 (1H, d, J = 8.8 Hz, H-5), 6.96 (1H, dd, J = 8.8, 2.0 Hz, H-6), 6.84 (IH, d, J = 2.0 Hz, H-8), 5.64 (1H, d, J = 15.6 Hz, H-9′), 5.57 (1H, dt, J = 15.6, 5.9 Hz, H-8′), 5.13 (1H, t, J = 6.7 Hz, H-5′), 3.88 (3H, s, OMe), 3.41 (1H, q, J = 6.9 Hz, H-1′), 2.66 (2H, d, J = 5.9 Hz, H-7′), 2.15 (2H, m, H-4′), 1.84 (2H, m, H-3′), 1.57 (3H, s, H-13′), 1.46 (3H, s, H-14′), 1.36 (3H, d, J = 6.9 Hz, H-15′), 1.31 (6H, s, H-11′ and H-12′); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 175.3 (s, C-4), 167.3 (s, C-2), 163.1 (s, C-7), 154.9 (s, C-9), 139.7 (d, C-9′), 135.0 (s, C-6′), 126.9 (d, C-5), 124.9 (d, C-8′), 124.0 (d, C-5′), 117.9 (s, C-10), 113.3 (d, C-6), 101.0 (d, C-8), 98.7 (s, C-3), 96.0 (s, C-2′), 70.7 (s, C-10′), 55.9 (q, OMe), 42.3 (t, C-7′), 41.5 (t, C-3′), 41.1 (d, C-1′), 29.9 (q, C-11′ and C-12′), 22.2 (t, C-4′), 20.5 (q, C-14′), 16.2 (q, C-13′), 14.6 (q, C-15′); EIMS *m*/*z* 412 $[M]^+$ (11.6), 394 $[M - H_2O]^+$ (69.3), 328 (13.6), 313 (5.3), 285 (8.5), 283 (9.2), 273 (11.4), 260 (21.8), 259 (81.6), 257 (31.2), 245 (73.3), 244 (100), 219 (65.0), 203 (10.0), 193 (10.2), 175 (17.4), 162 (14.1), 151 (75.8), 135 (24.2), 121 (36.1), 109 (23.1), 107 (18.2), 95 (23.0), 93 (20.3), 79 (20.4), 67 (18.9), 59 (11.3), 55 (22.7), 43 (62.9), 41 (27.3), 39 (7.9); HREIMS *m*/*z* 412.2263 (calcd for $C_{25}H_{32}O_5$, 412.2250).

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