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CHEMICAL CONSTITUENTS FROM THE ROOTS OF *Synsepalum dulcificum*

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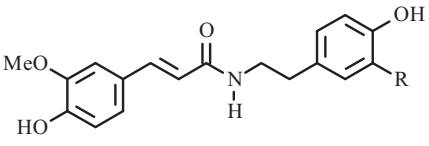
Botanical data and some properties of *Synsepalum dulcificum* Daniell (Sapotaceae) are discussed in our article published in the journal number [1].

The MeOH extract of its roots was subjected to solvent partitioning and chromatographic separation to afford five fractions. The chemical constituents in the roots of *S. dulcificum* were separated with column chromatography. Nine compounds, including *N-trans*-feruloyltyramine (**1**) [2], *N-cis*-feruloyltyramine (**2**) [3], *N-trans*-feruloylmethoxytyramine (**3**), *N-cis*-feruloylmethoxytyramine (**4**) [4], *p*-hydroxybenzoic acid (**5**), methylparaben (**6**), vanillic acid (**7**), isovanillic acid (**8**), and syringic acid (**9**) [5], were isolated from the roots of *S. dulcificum*. All of these compounds were found for the first time from this plant.

The specimen of *S. dulcificum* was collected from Kaohsiung County, Taiwan, October 2007. A voucher specimen was identified by Dr. Fu-Yuan Lu (Department of Forestry and Natural Resources College of Agriculture, National Chiayi University) and deposited in the School of Medical and Health Science, The Fooyin University, Kaohsiung County, Taiwan. The roots (5.2 kg) of *S. dulcificum* were extracted repeatedly with MeOH at room temperature for 24–48 hrs. The MeOH extract was dried and evaporated to leave a viscous residue (43.8 g). The residue was placed on a silica gel column and eluted with $CHCl_3$ gradually enriched with MeOH to afford five fractions. Fraction 2 (5.11 g) eluted with *n*-hexane–EtOAc (1:1) was further purified by silica gel CC using the same solvent system to obtain *N-trans*-feruloyltyramine (**1**) (8 mg) and *N-cis*-feruloyltyramine (**2**) (5 mg). Fraction 3 (7.81 g) eluted with *n*-hexane–Me_2CO (6:1) was further separated using silica gel CC and preparative TLC (*n*-hexane–EtOAc (1:2)) to give *N-trans*-feruloylmethoxy-tyramine (**3**) (18 mg) and *N-cis*-feruloylmethoxytyramine (**4**) (12 mg). Fraction 4 (3.12 g) was purified by silica gel chromatography ($CHCl_3$–MeOH, 80:1) to give *p*-hydroxybenzoic acid (**5**) (4 mg) and methylparaben (**6**) (6 mg). Fraction 5 (5.91 g) was purified by silica gel chromatography ($CHCl_3$–MeOH, 50:1) to give colorless needles of vanillic acid (**7**) (15 mg), isovanillic acid (**8**) (10 mg), and syringic acid (**9**) (22 mg).

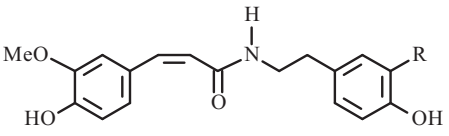
***N-trans*-Feruloyltyramine (1)** as in [2], colorless crystals ($CHCl_3$), UV (λ_{max}, nm): 220, 293, 319. IR (ν_{max}, cm^{-1}): 3300 (OH), 1650 (C=O). ^1H NMR (400 MHz, $CDCl_3$, δ, ppm, J/Hz): 2.75 (2H, t, J = 6.8, H-2), 3.54 (2H, t, J = 6.8, H-1), 3.87 (3H, s, OCH_3), 6.15 (1H, d, J = 15.6, H-2′), 6.75 (2H, d, J = 8.8, H-5 and H-7), 6.83 (1H, d, J = 8.0, H-8′), 7.00 (1H, dd, J = 8.0, 2.0, H-9′), 7.02 (2H, d, J = 8.8, H-4 and H-8), 7.46 (1H, d, J = 15.6, H-3′), EI-MS *m/z*: 313 $[M]^+$.

***N-cis*-Feruloyltyramine (2)** as in [3], yellow oil, UV (λ_{max}, nm): 220, 293, 318. IR (ν_{max}, cm^{-1}): 3350 (OH), 1650 (C=O). ^1H NMR (400 MHz, CD_3OD, δ, ppm, J/Hz): 2.69 (2H, t, J = 7.6, H-2), 3.40 (2H, t, J = 7.6, H-1), 3.83 (3H, s, OCH_3), 5.81 (1H, d, J = 12.8, H-2′), 6.61 (1H, d, J = 12.8, H-3′), 6.68 (2H, d, J = 8.4, H-5 and H-7), 6.73 (1H, d, J = 8.4, H-8′), 6.92 (1H, dd, J = 8.4, 2.0, H-9′), 6.99 (2H, d, J = 8.4, H-4 and H-8), 7.35 (1H, d, J = 2.0, H-5′), EI-MS *m/z*: 313 $[M]^+$.



1, 3

1: R = H; **3:** R = OMe



2, 4

2: R = H; **4:** R = OMe

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***N-trans*-Feruloylmethoxytyramine (3)** as in [4], white needles (MeOH), UV (λ_{max}, nm): 220, 290, 319. IR (ν_{max}, cm^{-1}): 3350 (OH), 1650 (C=O). ^1H NMR (400 MHz, $CDCl_3$, δ, ppm, J/Hz): 2.74 (2H, t, J = 6.8, H-2), 3.51 (2H, t, J = 6.8, H-1), 3.80 (3H, s, OCH_3), 3.83 (3H, s, OCH_3), 6.16 (1H, d, J = 15.6, H-2′), 6.63 (1H, dd, J = 8.0, 2.0, H-9′), 6.68 (1H, d, J = 2.0, H-5′), 6.77 (1H, d, J = 8.0, H-8′), 6.80 (1H, d, J = 8.0, H-7), 6.93 (1H, d, J = 2.0, H-4), 6.96 (1H, dd, J = 8.0, 2.0, H-8), 7.43 (1H, d, J = 15.6, H-3′), EI-MS *m/z*: 343 $[M]^+$.

***N-cis*-Feruloylmethoxytyramine (4)** as in [4], white needles (MeOH), UV (λ_{max}, nm): 221, 290, 319. IR (ν_{max}, cm^{-1}): 3350 (OH), 1650 (C=O). ^1H NMR (400 MHz, CD_3OD, δ, ppm, J/Hz): 2.71 (2H, t, J = 7.2, H-2), 3.42 (2H, t, J = 7.2, H-1), 3.80 (3H, s, OCH_3), 3.83 (3H, s, OCH_3), 5.82 (1H, d, J = 12.8, H-2′), 6.61 (1H, d, J = 12.8, H-3′), 6.61 (1H, dd, J = 8.0, 2.0, H-8), 6.70 (1H, d, J = 8.0, H-7), 6.73 (1H, d, J = 8.4, H-8′), 6.77 (1H, d, J = 2.0, H-4), 6.93 (1H, dd, J = 8.4, 2.0, H-9′), 7.36 (1H, d, J = 2.0, H-5′), EI-MS *m/z*: 343 $[M]^+$.

***p*-Hydroxybenzoic acid (5)** as in [5], brown powder ($CHCl_3$), UV (λ_{max}, nm): 250, 285, 290. IR (ν_{max}, cm^{-1}): 3500, 1660, 1590, 1280, 1165. ^1H NMR (400 MHz, $CDCl_3$, δ, ppm, J/Hz): 6.85 (2H, d, J = 8.6, H-3 and H-5), 7.96 (2H, d, J = 8.6, H-2 and H-6), EI-MS *m/z*: 138 $[M]^+$.

Methylparaben (6) as in [5], colorless needles ($CHCl_3$), UV (λ_{max}, nm): 225, 258, 300. IR (ν_{max}, cm^{-1}): 3400, 2960, 2850, 1690, 1610. ^1H NMR (400 MHz, $CDCl_3$, δ, ppm, J/Hz): 3.89 (3H, s, $COOCH_3$), 6.86 (2H, d, J = 8.7, H-3 and H-5), 7.96 (2H, d, J = 8.7, H-2 and H-6), EI-MS *m/z*: 152 $[M]^+$.

Vanillic acid (7) as in [5], colorless needles (MeOH), UV (λ_{max}, nm): 220, 265, 300. IR (ν_{max}, cm^{-1}): 3550, 1680, 1510, 1280. ^1H NMR (400 MHz, $CDCl_3$, δ, ppm, J/Hz): 3.89 (3H, s, OCH_3), 6.80 (1H, d, J = 8.0, H-5), 7.50 (1H, dd, J = 8.0, 1.8, H-6), 7.57 (1H, d, J = 1.8, H-2), EI-MS *m/z*: 168 $[M]^+$.

Isovanillic acid (8) as in [5], colorless needles (MeOH), UV (λ_{max}, nm): 220, 260, 290. IR (ν_{max}, cm^{-1}): 3500, 2900, 1690, 1560, 1280. ^1H NMR (400 MHz, CD_3OD, δ, ppm, J/Hz): 3.92 (3H, s, OCH_3), 7.27 (1H, dd, J = 8.8, 2.8, H-6), 8.02 (1H, d, J = 8.8, H-5), 8.17 (1H, d, J = 2.8, H-2), EI-MS *m/z*: 168 $[M]^+$.

Syringic acid (9) as in [5], brown needles ($CHCl_3$), UV (λ_{max}, nm): 212, 235, 310. IR (ν_{max}, cm^{-1}): 3250, 1670, 1590, 1515. ^1H NMR (400 MHz, $CDCl_3$, δ, ppm, J/Hz): 3.81 (6H, s, 2 × OCH_3), 7.38 (2H, s, H-2 and H-6), EI-MS *m/z*: 198 $[M]^+$.

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