SUPPLEMENTARY MATERIAL

A new isoflavanone from the trunk of *Horsfieldia pandurifolia*

Qin Ma, Yujiao Liu, Rui Zhan, Yegao Chen*

*School of Chemistry and Chemical Engineering, Yunnan Normal University, Kunming
650500, China*

*Corresponding author. Tel: 86-871-65941089; Email: ygchen48@126.com

Abstract: A new isoflavanone, 2,2'-epoxy-4'-methoxy-3,7-dihydroxyisoflavanone (**1**), and a new natural coumaranone, 2-hydroxy-2-(4'-methoxybenzyl)-6-methoxy-3-coumaranone (**2**), along with twenty-six known compounds, were first isolated from the trunk of *Horsfieldia pandurifolia*. Their structures were elucidated by the means of spectroscopic analysis. **1** was assessed for its cytotoxicity against five human tumor lines (HL-60, SMMC-7721, A-549, MCF-7 and SW-480), and the result showed that it has no activity.

Keywords: *Horsfieldia pandurifolia*; Myristicaceae; isoflavanone

Supporting information

Figure S1. HMBC correlations of compound **1**

Spectroscopic data of the known compounds **3-28**.

Original spectra of compound **1**.

^1H NMR Spectrum (500 MHz, CD_3COCD_3)

^{13}C NMR Spectrum (125 MHz, CD_3COCD_3)

HSQC Spectrum

HMBC Spectrum

IR Spectrum

HREIMS Spectrum

Figure S1. HMBC correlations of compound **1**

Spectroscopic data of the known compounds **3-28**.

α ,2'-dihydroxy-4,4'-dimethoxydihydrochalcone (3)

Colorless needles; MF: C₁₇H₁₈O₅, MW: 302; ¹H NMR (500 MHz, CD₃COCD₃): δ 2.93 (1H, dd, J = 14.0, 6.0 Hz, β -H), 3.13 (1H, dd, J = 14.0, 4.0 Hz, β -H), 3.77 (3H, s, 4-OMe), 3.91 (3H, s, 4'-OMe), 4.36 (1H, d, α -OH), 5.24 (1H, m, α -H), 6.49 (1H, d, J = 2.5 Hz, H-3'), 6.55 (1H, dd, J = 9.0, 2.5 Hz, H-5'), 6.82 (2H, d, J = 8.5 Hz, H-3, 5), 7.17 (2H, d, J = 8.5 Hz, H-2, 6), 7.99 (1H, d, J = 9.0 Hz, H-6'), 12.43 (1H, s, 2'-OH); ¹³C NMR (125 MHz, CD₃COCD₃): δ 204.5 (s, C=O), 166.6 (s, C-4'), 165.8 (s, C-4), 158.6 (s, C-2'), 132.4 (d, C-6'), 130.5 (d, C-2, 6), 129.3 (s, C-1), 113.5 (d, C-3, 5), 111.4 (s, C-1'), 107.6 (d, C-5'), 100.9 (d, C-3'), 73.4 (d, α -C), 55.3 (q, 4'-OMe), 54.5 (q, 4-OMe), 40.9 (t, β -C).

α ,4,2',4'-tetrahydroxydihydrochalcone (4)

Colorless needles; MF: C₁₅H₁₄O₅, MW: 274; ¹H NMR (500 MHz, CD₃COCD₃): δ 7.89 (1H, d, J = 9.0 Hz, H-6'), 7.06 (2H, d, J = 8.5 Hz, H-2, 6), 6.73 (2H, d, J = 8.5 Hz, H-3, 5), 6.47 (1H, dd, J = 9.0, 2.5 Hz, H-5'), 6.37 (1H, d, J = 2.0 Hz, H-3'), 5.19 (1H, dd, J = 7.5, 4.5 Hz, α -H), 3.06 (1H, dd, J = 14.0, 4.5 Hz, β -H), 2.87 (1H, dd, J = 14.0, 7.5 Hz, β -H); ¹³C NMR (125 MHz, CD₃COCD₃): δ 204.0 (s, C=O), 165.9 (s, C-4'), 165.9 (s, C-2'), 156.1 (s, C-4), 132.8 (d, C-6'), 130.5 (d, C-2, 6), 128.0 (s, C-1), 114.9 (d, C-3, 5), 110.6 (s, C-1'), 108.6 (d, C-5'), 102.9 (d, C-3'), 73.9 (d, α -C), 41.2 (t, β -C).

rhusopolyphenol E (5)

Light-yellow crystal; MF: C₁₅H₁₄O₆, MW: 290; ¹H NMR (500 MHz, CD₃COCD₃): δ 7.73 (1H, d, J = 8.5 Hz, H-6'), 7.44 (2H, d, J = 8.5 Hz, H-2, 6), 6.91 (2H, d, J = 8.5 Hz, H-3, 5), 6.63 (1H, dd, J = 8.5, 2.0 Hz, H-5'), 6.41 (1H, d, J = 2.0 Hz, H-3'), 5.06 (1H, d, J = 12.0 Hz, β -H), 4.58 (1H, d, J = 12.0 Hz, α -H); ¹³C NMR (125 MHz, CD₃COCD₃): δ 192.4 (s, C=O), 165.2 (s, C-4'), 163.7 (s, C-2'), 158.0 (s, C-4), 129.4

(d, C-2, 6), 128.9 (d, C-6'), 128.4 (s, C-1), 115.0 (d, C-3, 5), 112.1 (s, C-1'), 110.9 (d, C-5'), 102.7 (d, C-3'), 84.0 (d, β -C), 73.1 (d, α -C).

1-(2-hydroxy-4-methoxyphenyl)-3-(3,4-methylenedioxyphenyl)-propan-2-ol (6)

Light-yellow crystal; MF: C₁₇H₁₈O₅, MW: 302; ¹H NMR (500 MHz, CD₃COCD₃): δ 6.78 (1H, d, J = 9.0, H-6'), 6.71 (3H, m, H-2'', 5'', 6''), 6.43 (1H, d, J = 2.5, H-3'), 6.37 (1H, dd, J = 8.5, 2.5, H-5'), 5.95 (2H, s, CH₂O₂), 4.14 (1H, m, H-2), 3.73 (3H, s, 4'-OMe), 3.05 (1H, br, 2-OH), 2.71-2.81 (4H, m, H-1, 3); ¹³C NMR (125 MHz, CD₃COCD₃): δ 159.8 (s, C-4'), 157.0 (s, C-2'), 147.6 (s, C-3''), 146.0 (s, C-4''), 132.9 (s, C-1''), 132.0 (d, C-6'), 122.4 (d, C-6''), 118.0 (s, C-1'), 109.7 (d, C-2''), 107.8 (d, C-5''), 105.0 (d, C-5'), 102.2 (d, C-3'), 100.8 (t, CH₂O₂), 74.0 (d, C-2), 54.5, (q, 4'-OMe), 42.9 (t, C-1), 38.1 (t, C-3).

1-(2-hydroxy-4-methoxyphenyl)-3-(3-methoxy-4-hydroxyphenyl)-propan-2-ol(7)

Colorless oil; MF: C₁₇H₂₀O₅, MW: 304; ¹H NMR (500 MHz, CD₃COCD₃): δ 6.92 (1H, d, J = 8.5, H-6'), 6.85 (1H, d, J = 2.0, H-2''), 6.75 (1H, d, J = 8.0, H-5''), 6.69 (1H, dd, J = 8.0, 2.0, H-6''), 6.42 (1H, d, J = 2.5, H-3'), 6.37 (1H, dd, J = 8.5, 2.5, H-5'), 4.16 (1H, m, H-2), 3.83 (3H, s, 4''-OMe), 3.73 (3H, s, 4'-OMe), 2.68-2.83 (4H, m, H-1, 3); ¹³C NMR (125 MHz, CD₃COCD₃): δ 159.8 (s, C-4'), 157.1 (s, C-2'), 147.2 (s, C-4''), 145.0 (s, C-3''), 132.0 (d, C-6'), 130.2 (s, C-1''), 121.9 (d, C-6''), 118.1 (s, C-1'), 114.7 (d, C-5''), 113.0 (d, C-2''), 104.9 (d, C-5'), 102.2 (d, C-3'), 74.2 (d, C-2), 55.3 (q, 3''-OMe), 54.5 (q, 4'-OMe), 42.9 (t, C-1), 38.0 (t, C-3).

isoliquiritigenin (8)

Yellow amorphous powder; MF: C₁₅H₁₂O₄, MW: 256; ¹H NMR (500 MHz, CD₃COCD₃): δ 8.12 (1H, d, J = 9.0 Hz, H-6'), 7.85 (1H, d, J = 15.5 Hz, β -H), 7.77 (1H, d, J = 15.5 Hz, α -H), 7.74 (2H, d, J = 9.0 Hz, H-2, 6), 6.94 (2H, d, J = 9.0 Hz, H-3, 5), 6.48 (1H, dd, J = 8.5, 2.0 Hz, H-5'), 6.38 (1H, d, J = 2.0 Hz, H-3'); ¹³C NMR (125 MHz, CD₃COCD₃): δ 192.0 (s, C=O), 166.7 (s, C-4'), 164.9 (s, C-2'), 160.3 (s, C-4), 144.3 (d, β -C), 132.4 (d, C-5'), 130.9 (d, C-2, 6), 126.6 (s, C-1), 117.4 (d, α -C),

115.9 (d, C-3, 5), 113.6 (s, C-1'), 107.9 (d, C-5'), 102.9 (d, C-3').

butein (9)

Yellow needles; MF: C₁₅H₁₂O₅, MW: 272; ¹H NMR (125 MHz, CD₃COCD₃): δ 13.65 (1H, br, 5-OH), 8.11 (1H, d, *J* = 9.0 Hz, H-6'), 7.77 (1H, d, *J* = 15.0 Hz, β-H), 7.69 (1H, d, *J* = 15.0 Hz, α-H), 7.36 (1H, d, *J* = 2.0 Hz, H-2), 7.22 (1H, dd, *J* = 8.0, 2.0 Hz, H-6), 6.91 (1H, d, *J* = 8.0 Hz, H-5), 6.47 (1H, dd, *J* = 8.5, 2.0 Hz, H-5'), 6.37 (1H, d, *J* = 2.0 Hz, H-3'); ¹³C NMR (125 MHz, CD₃COCD₃): δ 191.9 (s, C=O), 166.7 (s, C-4'), 165.0 (s, C-2'), 148.6 (s, C-3), 145.7 (s, C-4), 144.7 (d, β-C), 132.4 (d, C-6'), 127.2 (s, C-1), 122.5 (d, α-C), 117.4 (d, C-6), 115.5 (d, C-5), 115.0 (d, C-2), 113.5 (s, C-1'), 107.9 (d, C-5'), 102.9 (d, C-3').

sulfuretin (10)

Yellow amorphous powder; MF: C₁₅H₁₀O₅, MW: 270; ¹H NMR (500 MHz, CD₃COCD₃): δ 7.62 (1H, d, *J* = 8.0 Hz, H-4), 7.53 (1H, s, =CH), 7.24 (1H, d, *J* = 8.0 Hz, H-6'), 6.69 (3H, H-5, H-7, H-2'), 6.85 (1H, d, *J* = 8.0 Hz, H-5'). ¹³C NMR (125 MHz, CD₃COCD₃) δ 181.5 (s, C-3), 168.2 (s, C-6), 166.7 (s, C-7a), 147.5 (s, C-4'), 146.5 (s, C-2), 145.3 (s, C-3'), 125.5 (d, C-4), 124.7 (d, C-6'), 124.5 (s, C-1'), 117.7 (d, C-2'), 115.6 (d, C-5'), 113.6 (s, C-3a), 112.9 (d, =CH), 111.3 (d, C-7), 98.5 (d, C-5).

7-hydroxyflavonone (11)

Light-yellow crystal; MF: C₁₅H₁₂O₃, MW: 240; ¹H NMR (500 MHz, CD₃COCD₃): δ 7.75 (1H, d, *J* = 8.5 Hz, H-5), 7.60-7.40 (5H, m, H-2'~6'), 6.61 (1H, dd, *J* = 8.5, 2.5 Hz, H-6), 6.48 (1H, d, *J* = 2.5 Hz, H-8), 5.58 (1H, dd, *J* = 13.0, 3.0 Hz, H-2), 3.05 (1H, dd, *J* = 17.0, 13.0 Hz, H-3a), 2.77 (1H, dd, *J* = 17.0, 3.0 Hz, H-3b); ¹³C NMR (125 MHz, CD₃COCD₃): δ 189.3 (s, C-4), 164.4 (s, C-7), 163.5 (s, C-9), 139.6 (s, C-1'), 128.7 (d, C-4'), 128.6 (d, C-3', 5'), 128.4 (d, C-5), 126.4 (d, C-2', 6'), 114.4 (s, C-10), 110.5 (d, C-6), 102.8 (d, C-8), 79.7 (d, C-2), 43.9 (t, C-3).

liquiritigenin (12)

White crystals; MF: C₁₅H₁₂O₄, MW: 256; ¹H NMR (500 MHz, CD₃COCD₃): δ 7.74 (1H, d, *J* = 8.5 Hz, H-5), 7.41 (2H, d, *J* = 8.5 Hz, H-2', 6'), 6.91 (2H, d, *J* = 8.5 Hz, H-3', 5'), 6.58 (1H, dd, *J* = 8.5, 2.5 Hz, H-6), 6.43 (1H, d, *J* = 2.5 Hz, H-8), 5.45 (1H, dd, *J* = 13.0, 3.0 Hz, H-2), 3.06 (1H, dd, *J* = 17.0, 13.0 Hz, H-3a), 2.68 (1H, dd, *J* = 17.0, 3.0 Hz, H-3b); ¹³C NMR (125 MHz, CD₃COCD₃): δ 189.9 (s, C-4), 164.6 (s, C-7), 163.7 (s, C-9), 157.8 (s, C-4'), 130.3 (s, C-1'), 128.6 (d, C-5), 128.1 (d, C-2', 6'), 115.3 (d, C-3', 5'), 114.2 (s, C-10), 110.4 (d, C-6), 102.8 (d, C-8), 79.6 (d, C-2), 43.8 (t, C-3).

butin (13)

Light-yellow crystal; MF: C₁₅H₁₂O₅, MW: 272; ¹H NMR (500 MHz, CD₃COCD₃): δ 7.73 (1H, d, *J* = 8.5 Hz, H-5), 7.06 (1H, d, *J* = 1.5 Hz, H-2'), 6.89 (1H, dd, *J* = 8.5, 1.5 Hz, H-6'), 6.88 (1H, d, *J* = 8.5 Hz, H-5'), 6.58 (1H, dd, *J* = 8.5, 2.2 Hz, H-6), 6.43 (1H, d, *J* = 2.2 Hz, H-8), 5.39 (1H, dd, *J* = 13.0, 3.0 Hz, H-2), 3.02 (1H, dd, *J* = 16.5, 13.0 Hz, H-3a), 2.68 (1H, dd, *J* = 16.5, 3.0 Hz, H-3b); ¹³C NMR (125 MHz, CD₃COCD₃) δ 190.0 (s, C-4), 164.7 (s, C-7), 163.7 (s, C-9), 145.4 (s, C-3'), 145.1 (s, C-4'), 131.1 (s, C-1'), 128.6 (d, C-5), 118.3 (d, C-6'), 115.1 (d, C-5'), 114.1 (s, C-10), 113.8 (d, C-2'), 110.4 (d, C-6), 102.8 (d, C-8), 79.7 (d, C-2), 43.8 (t, C-3).

fustin (14)

White needles; MF: C₁₅H₁₂O₆, MW: 288; ¹H NMR (500 MHz, CD₃COCD₃): δ 7.73 (1H, d, *J* = 8.5 Hz, H-5), 7.09 (1H, d, *J* = 8.5 Hz, H-6'), 6.90 (2H, m, H-2', 5'), 6.62 (1H, d, *J* = 8.0 Hz, H-6), 6.39 (1H, s, H-8), 4.99 (1H, d, *J* = 11.5 Hz, H-2), 4.55 (1H, d, *J* = 11.5 Hz, H-3), 4.53 (1H, br, 3-OH); ¹³C NMR (125 MHz, CD₃COCD₃) δ 192.4 (s, C-4), 165.1 (s, C-7), 163.6 (s, C-9), 145.7 (s, C-3'), 144.9 (s, C-4'), 129.1 (s, C-1'), 128.9 (d, C-5), 120.0 (d, C-6'), 115.0 (d, C-2'), 114.9 (d, C-5'), 112.1 (s, C-10), 110.9 (d, C-6), 102.7 (d, C-8), 84.1 (d, C-2), 73.1 (d, C-3).

(-)-festidinol (15)

Colorless crystals; MF: C₁₅H₁₄O₅, MW: 274; ¹H NMR (500 MHz, CD₃COCD₃): δ 6.89 (1H, d, *J* = 9.0 Hz, H-5), 6.81 (1H, d, *J* = 1.5 Hz, H-2'), 6.80 (1H, d, *J* = 8.0 Hz, H-5'), 6.75 (1H, dd, *J* = 8.0, 1.5 Hz, H-6'), 6.39 (1H, dd, *J* = 9.0, 2.0 Hz, H-6), 6.31 (1H, d, *J* = 2.0 Hz, H-8), 4.62 (1H, d, *J* = 7.5 Hz, H-2), 4.03 (1H, m, H-3), 4.01 (1H, br, 3-OH), 2.90 (1H, dd, *J* = 15.0, 5.0 Hz, H-4a), 2.71 (1H, dd, *J* = 15.0, 8.0 Hz, H-4b); ¹³C NMR (125 MHz, CD₃COCD₃) δ 156.9 (s, C-9), 155.2 (s, C-7), 145.0 (s, C-4'), 144.9 (s, C-3'), 131.1 (s, C-1'), 130.2 (d, C-5), 119.0 (d, C-6'), 114.8 (d, C-5'), 114.3 (d, C-2'), 111.6 (s, C-10), 108.2 (d, C-6), 102.5 (d, C-8), 82.0 (d, C-2), 67.4 (d, C-3), 32.6 (t, C-3).

formononetin (16)

Colorless needles; MF: C₁₆H₁₂O₄, MW: 268; ¹H NMR (500 MHz, Pyr): δ 8.45 (1H, d, *J* = 8.5 Hz, H-5), 8.16 (1H, s, H-2), 7.78 (2H, d, *J* = 8.5 Hz, H-2', 6'), 7.23 (1H, dd, *J* = 8.5, 2.0 Hz, H-6), 7.13 (1H, d, *J* = 2.0 Hz, H-8), 7.07 (2H, d, *J* = 8.5 Hz, H-3', 5'), 3.69 (3H, s, 4'-OMe); ¹³C NMR (125 MHz, Pyr): δ 175.4 (s, C-4), 163.9 (s, C-7), 159.8 (s, C-4'), 158.3 (s, C-9), 152.5 (d, C-2), 130.6 (d, C-2', 6'), 128.0 (d, C-5), 125.1 (s, C-1'), 124.4 (s, C-3), 117.7 (s, C-10), 115.7 (d, C-6), 114.0 (d, C-3', 5'), 102.9 (d, C-8), 55.0 (q, 4'-OMe).

iristectorigenin A (17)

Light-yellow needles; MF: C₁₇H₁₄O₇, MW: 330; ¹H NMR (500 MHz, Pyr): δ 13.20 (1H, br, 5-OH), 8.17 (1H, s, H-2), 7.49 (1H, d, *J* = 1.0 Hz, H-6'), 7.31 (2H, m, H-2', 5'), 6.77 (1H, s, H-8), 3.97 (3H, s, 6-OMe), 3.84 (3H, s, 4'-OMe); ¹³C NMR (125 MHz, Pyr): δ 181.6 (s, C-4), 159.0 (s, C-7), 154.6 (s, C-5), 153.9 (s, C-9), 153.7 (d, C-2), 148.6 (s, C-4'), 148.5 (s, C-3'), 132.6 (s, C-6), 123.3 (s, C-6), 122.7 (s, C-1'), 122.6 (d, C-6'), 116.6 (d, C-5'), 114.0 (d, C-2'), 106.1 (s, C-10), 94.9 (d, C-8), 60.3 (q, 6-OMe), 59.8 (q, 6-OMe).

2'-hydroxyformononetin (18)

Colorless needles; MF: C₁₆H₁₂O₅, MW: 284; ¹H NMR (500 MHz, CD₃COCD₃): δ

8.31 (1H, s, H-2), 8.14 (1H, d, $J = 8.5$ Hz, H-5), 7.26 (1H, d, $J = 8.5$ Hz, H-6'), 7.12 (1H, dd, $J = 8.5, 2.0$ Hz, H-6), 7.02 (1H, d, $J = 2.0$ Hz, H-8), 6.55 (2H, m, H-3', 5'), 3.82 (3H, s, 4'-OMe); ^{13}C NMR (125 MHz, CD_3COCD_3): δ 177.8 (s, C-4), 163.3 (s, C-7), 161.7 (s, C-4'), 158.0 (s, C-9), 157.7 (s, C-2'), 155.4 (d, C-2), 131.1 (d, C-6'), 127.7 (d, C-5), 123.4 (s, C-3), 116.4 (s, C-10), 115.8 (d, C-6), 112.8 (s, C-1'), 106.3 (d, C-5'), 103.4 (d, C-3'), 102.1 (d, C-8), 54.7 (q, 4'-OMe).

tectorigenin (19)

Light-yellow needles; MF: $\text{C}_{16}\text{H}_{12}\text{O}_6$, MW: 300; ^1H NMR (500 MHz, CD_3COCD_3): δ 13.20 (1H, br, 5-OH), 8.17 (1H, s, H-2), 7.46 (2H, d, $J = 9.0$ Hz, H-2', 6'), 6.91 (2H, d, $J = 9.0$ Hz, H-3', 5'), 6.50 (1H, s, H-8), 3.88 (3H, s, 6-OMe); ^{13}C NMR (125 MHz, CD_3COCD_3): δ 181.2 (s, C-4), 157.6 (s, C-4'), 157.1 (s, C-7), 153.6 (s, C-9), 153.6 (d, C-2), 153.4 (s, C-5), 131.4 (s, C-6), 130.3 (d, C-2', 6'), 122.6 (s, C-1'), 122.1 (s, C-3), 115.1 (d, C-3', 5'), 105.6 (s, C-10), 93.6 (d, C-8), 59.8 (q, 6-OMe).

genistein (20)

White crystals; MF: $\text{C}_{15}\text{H}_{10}\text{O}_5$, MW: 270; ^1H NMR (500 MHz, CD_3COCD_3): δ 13.03 (1H, br, 5-OH), 8.16 (1H, s, H-2), 7.45 (2H, d, $J = 8.5$ Hz, H-2', 6'), 6.91 (2H, d, $J = 8.5$ Hz, H-3', 5'), 6.42 (1H, d, $J = 2.0$ Hz, H-6), 6.29 (1H, d, $J = 2.0$ Hz, H-8); ^{13}C NMR (125 MHz, CD_3COCD_3): δ 180.8 (s, C-4), 164.4 (s, C-7), 163.0 (s, C-5), 158.2 (s, C-9), 157.6 (s, C-4'), 153.4 (d, C-2), 130.3 (d, C-2', 6'), 123.1 (s, C-1'), 122.1 (s, C-3), 115.1 (d, C-3', 5'), 105.2 (s, C-10), 99.0 (d, C-6), 93.6 (d, C-8).

dihydrodaidzein (21)

White needles; MF: $\text{C}_{15}\text{H}_{12}\text{O}_4$, MW: 256; ^1H NMR (500 MHz, CD_3COCD_3): δ 7.75 (1H, d, $J = 8.5$ Hz, H-5), 7.14 (2H, d, $J = 8.5$ Hz, H-2', 6'), 6.80 (2H, d, $J = 8.5$ Hz, H-3', 5'), 6.58 (1H, dd, $J = 8.5, 2.0$ Hz, H-6), 6.41 (1H, d, $J = 2.0$ Hz, H-8), 4.63 (2H, d, $J = 6.0$ Hz, H-2), 3.85 (1H, t, $J = 6.5$ Hz, H-3); ^{13}C NMR (125 MHz, CD_3COCD_3): δ 190.3 (s, C-4), 164.5 (s, C-7), 163.5 (s, C-9), 156.8 (s, C-4'), 129.6 (d, C-2', 6'), 129.2 (d, C-5), 127.0 (s, C-1'), 115.3 (d, C-3', 5'), 114.3 (s, C-10), 110.5 (d, C-6),

102.5 (d, C-8), 71.8 (t, C-2), 50.9 (d, C-3).

luteolin (22)

Yellow crystals; MF: C₁₅H₁₀O₆, MW: 286; ¹H NMR (500 MHz, Pyr): δ 13.78 (1H, br, 5-OH), 7.90 (1H, d, *J* = 1.5 Hz, H-2'), 7.53 (1H, dd, *J* = 8.5, 1.5 Hz, H-6'), 7.29 (1H, d, *J* = 8.5 Hz, H-5'), 6.91 (1H, s, H-3), 6.73 (2H, d, *J* = 1.5 Hz, H-6, 8); ¹³C NMR (125 MHz, Pyr): δ 184.0 (s, C-4), 167.1 (s, C-2), 166.1 (s, C-7), 164.1 (s, C-5), 159.8 (s, C-9), 153.0 (s, C-4'), 149.0 (s, C-3'), 124.2 (s, C-1'), 120.8 (d, C-6'), 118.1 (d, C-5'), 115.9 (d, C-2'), 106.3 (s, C-10), 105.2 (d, C-3), 101.2 (d, C-6), 96.1 (d, C-8).

3,7,4'-trihydroxyflavone (23)

Light-yellow needles; MF: C₁₅H₁₀O₅, MW: 270; ¹H NMR (500 MHz, CD₃COCD₃): δ 8.17 (2H, d, *J* = 9.0 Hz, H-2', 6'), 8.02 (1H, d, *J* = 9.0 Hz, H-5), 7.05 (1H, d, *J* = 2.0 Hz, H-8), 7.02 (1H, d, *J* = 9.0 Hz, H-3', 5'), 7.01 (1H, dd, *J* = 9.0, 2.0 Hz, H-6); ¹³C NMR (125 MHz, CD₃COCD₃): δ 172.2 (s, C-4), 162.5 (s, C-9), 159.0 (s, C-4'), 157.1 (s, C-7), 144.6 (s, C-2), 137.1 (s, C-3), 129.3 (d, C-2', 6'), 126.6 (d, C-5), 122.9 (s, C-1'), 115.4 (d, C-3', 5'), 114.7 (d, C-6), 114.3 (s, C-10), 102.3 (d, C-8).

ferulylaldehyde (24)

Light-yellow oil; MF: C₁₀H₁₀O₃, MW: 178; ¹H NMR (500 MHz, CD₃COCD₃): δ 9.65 (1H, d, *J* = 7.5 Hz, H-9), 7.58 (1H, d, *J* = 16.0 Hz, H-7), 7.40 (1H, s, H-6), 7.22 (1H, d, *J* = 7.5 Hz, H-3), 6.93 (1H, d, *J* = 8.5 Hz, H-2), 6.67 (1H, dd, *J* = 16.0, 8.5 Hz, H-8), 3.94 (3H, s, 5-OMe); ¹³C NMR (125 MHz, CD₃COCD₃): δ 193.0 (d, C-9), 153.2 (d, C-7), 150.0 (s, C-4), 148.0 (s, C-5), 126.5 (s, C-1), 126.1 (d, C-8), 123.9 (d, C-3), 115.4 (d, C-2), 110.7 (d, C-6), 55.5 (q, 5-OMe).

sinapaldehyde (25)

Yellow amorphous powder; MF: C₁₁H₁₂O₄, MW: 208; ¹H NMR (500 MHz, CD₃COCD₃): δ 9.65 (1H, d, *J* = 7.5 Hz, H-9), 7.57 (1H, d, *J* = 16.0 Hz, H-7), 7.09 (2H, s, H-2, 6), 6.70 (1H, dd, *J* = 16.0, 7.5 Hz, H-8), 3.92 (6H, s, 5-OMe, 3-OMe);

^{13}C NMR (125 MHz, CD_3COCD_3): δ 192.9 (d, C-9), 153.5 (d, C-7), 148.2 (s, C-3, 5), 139.4 (s, C-4), 126.4 (d, C-8), 125.3 (s, C-1), 106.5 (d, C-2, 6), 55.9 (q, 5-OMe, 3-OMe).

syringic acid (26)

Light-yellow needles; MF: $\text{C}_9\text{H}_{10}\text{O}_5$, MW: 195; ^1H NMR (500 MHz, CD_3COCD_3): δ 7.35 (2H, s, H-2, 6), 3.90 (6H, s, 3, 5-OMe); ^{13}C NMR (125 MHz, CD_3COCD_3): δ 166.8 (s, COOH), 147.5 (s, C-3, 5), 140.7 (s, C-4), 120.7 (s, C-1), 107.2 (d, C-2, 6), 55.8 (q, 3, 5-OMe).

3,4-dihydroxybenzoic acid (27)

Colorless crystals; MF: $\text{C}_7\text{H}_6\text{O}_4$, MW: 154; ^1H NMR (500 MHz, CD_3COCD_3): δ 7.54 (1H, d, $J = 1.5$ Hz, H-2), 7.48 (1H, dd, $J = 8.0, 1.5$ Hz, H-6), 6.91 (1H, d, $J = 8.0$ Hz, H-5); ^{13}C NMR (125 MHz, CD_3COCD_3) δ 167.4 (s, COOH), 150.0 (s, C-4), 144.8 (s, C-3), 122.7 (d, C-6), 122.1 (s, C-1), 116.6 (d, C-2), 114.8 (d, C-5).

β -sitosterol (28)

White crystals; MF: $\text{C}_{29}\text{H}_{50}\text{O}$, MW: 414; ^1H NMR (CDCl_3 , 500 MHz): δ 5.37 (1H, m, H-6), 3.55 (1H, m, H-3), 0.69 (3H, s, H-18); ^{13}C NMR (CDCl_3 , 125 MHz): δ 140.8 (s, C-5), 121.7 (d, C-6), 71.8 (d, C-3), 56.8 (d, C-14), 56.1 (d, C-17), 50.2 (d, C-9), 45.8 (C-24), 42.3 (t, C-4, s, C-13), 39.8 (t, C-12), 37.3 (t, C-1), 36.5 (s, C-10), 36.2 (d, C-20), 34.0 (t, C-22), 31.9 (t, C-7, d, C-8), 31.6 (t, C-2), 29.2 (d, C-25), 28.3 (t, C-16), 26.1 (t, C-23), 24.3 (t, C-15), 23.1 (t, C-28), 21.2 (t, C-11), 19.84 (q, C-26), 19.4 (q, C-19), 19.1 (q, C-27), 18.8 (q, C-21), 12.0 (q, C-18), 11.9 (q, C-29).

Original spectra of compound **1**.

^1H NMR:

^{13}C NMR:

HSQC:

HMBC:

IR Spectrum:

Sample : NQY-16		Frequency Range : 399.246 - 3996.32		Measured on : 05/03/2014	
Technique : KBr压片	Resolution : 4	Instrument : Tensor27		Sample Scans : 16	
Customer : 140305IR7	Zerofilling : 2	Acquisition : Double Sided, For			

HREIMS Spectrum:

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -10.0, max = 120.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

14 formula(e) evaluated with 1 results within limits (up to 51 closest results for each mass)

Elements Used:

C: 0-200 H: 0-400 O: 5-7

NOY-16

12:49:00 05-Mar-2014

Voltage EI+

KIB
M140305EA-04AFAMM 15 (1.378)

Autospec Premier

P776

1.25e3

Minimum: ~10.0
Maximum: 120.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula		
300.0634	300.0634	0.0	0.0	11.0	5546629.5	C16	H12	O6