

**Supporting Information to:**

**Cytotoxic Chalcones and Flavonoids from the Leaves of *Muntingia calabura***

Jih-Jung Chen<sup>1</sup>

Hsinn-Hsing Lee<sup>1</sup>

Chang-Yih Duh<sup>2</sup>

Ih-Sheng Chen<sup>3</sup>

**Affiliation:** <sup>1</sup> Graduate Institute of Pharmaceutical Technology, Tajen Institute of Technology, Pingtung, Taiwan, R.O.C.

<sup>2</sup> Institute of Marine Resources, National Sun Yat-sen University, Kaohsiung, Taiwan, R.O.C.

<sup>3</sup> School of Pharmacy, Kaohsiung Medical University, Kaohsiung, Taiwan, R.O.C.

**Correspondence:** Dr. J. J. Chen

Department of Pharmacy

Tajen Institute of Technology

Pingtung

Taiwan 907

R.O.C.

Fax: +886-8-762-5308

E-mail: [jjchen@ccsun.tajen.edu.tw](mailto:jjchen@ccsun.tajen.edu.tw)

Dr. I. S. Chen

School of Pharmacy

Kaohsiung Medical University

Kaohsiung

Taiwan 807

R.O.C.

Fax: +886-7-3210683

E-mail: [m635013@kmu.edu.tw](mailto:m635013@kmu.edu.tw)

## Physicochemical data

*2',4'-Dihydroxy-3'-methoxydihydrochalcone (1)*: Colorless needles from *n*-hexane-EtOAc, m.p. 86 – 88 °C. UV (MeOH):  $\lambda_{\max}$  (log  $\epsilon$ ) = 211 (4.04), 229 (sh, 3.71), 286 nm (3.82); (KOH): 210 (4.04), 254 (3.54), 336 nm (4.09); IR (KBr):  $\nu_{\max}$  = 3390 (br, OH), 1626  $\text{cm}^{-1}$  (C=O); EI-MS:  $m/z$  (rel. int.) = 272 ( $M^+$ , 57), 254 (10), 168 (9), 167 (100), 152 (18), 140 (6), 91 (17); HR-EI-MS:  $m/z$  found: 272.1038 [ $M$ ] $^+$ , calcd. for  $C_{16}H_{16}O_4$ : 272.1043;  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  = 3.05 (2H, t,  $J$  = 7.9 Hz, H- $\beta$ ), 3.25 (2H, t,  $J$  = 7.9 Hz, H- $\alpha$ ), 3.99 (3H, s, OMe-3'), 6.34 (1H, s, OH-4',  $\text{D}_2\text{O}$  exchangeable), 6.49 (1H, d,  $J$  = 8.5 Hz, H-5'), 7.22 (1H, t,  $J$  = 7.5 Hz, H-4), 7.24 (2H, d,  $J$  = 7.5 Hz, H-2 and H-6), 7.31 (2H, t,  $J$  = 7.5 Hz, H-3 and H-5), 7.44 (1H, d,  $J$  = 8.5 Hz, H-6'), 12.91 (1H, s, OH-2',  $\text{D}_2\text{O}$  exchangeable);  $^{13}\text{C-NMR}$  ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  = 30.3 (C- $\beta$ ), 39.8 (C- $\alpha$ ), 60.8 (OMe), 106.5 (C-5'), 114.3 (C-1'), 126.3 (C-4), 126.4 (C-6'), 128.4 (C-2 and C-6), 128.6 (C-3 and C-5), 134.1 (C-3'), 140.8 (C-1), 155.0 (C-4'), 156.5 (C-2'), 204.3 (C=O).

*(-)-3'-Methoxy-2',4', $\beta$ -trihydroxydihydrochalcone (2)*: Colorless amorphous solid.  $[\alpha]_{\text{D}}^{25}$ :  $-34.7^\circ$  ( $c$  0.07, MeOH); UV (MeOH):  $\lambda_{\max}$  (log  $\epsilon$ ) = 210 (4.06), 230 (sh, 3.76), 288 nm (3.86); (KOH): 207 (4.25), 254 (3.62), 338 nm (4.11); IR (KBr):  $\nu_{\max}$  = 3365 (br, OH), 1620  $\text{cm}^{-1}$  (C=O); EI-MS:  $m/z$  (rel. int.) = 288 ( $M^+$ , 48), 270 (35), 255 (16), 168 (11), 167 (100), 152 (22), 140 (10), 77 (10); HR-EI-MS:  $m/z$  found: 288.0994 [ $M$ ] $^+$ , calcd. for  $C_{16}H_{16}O_5$ : 288.0998;  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  = 3.29 (1H, dd,  $J$  = 17.4, 3.2 Hz, H- $\alpha$ ), 3.37 (1H, dd,  $J$  = 17.4, 8.8 Hz, H- $\alpha$ ), 3.57 (1H, br s, OH- $\beta$ ,  $\text{D}_2\text{O}$  exchangeable), 4.00 (3H, s, OMe-3'), 5.34 (1H, dd,  $J$  = 8.8, 3.2 Hz, H- $\beta$ ), 6.41 (1H, br s, OH-4',  $\text{D}_2\text{O}$  exchangeable), 6.50 (1H, d,  $J$  = 9.0 Hz, H-5'), 7.31 (1H, br t,  $J$  = 7.2 Hz, H-4), 7.39 (2H, br t,  $J$  = 7.6 Hz, H-3 and H-5), 7.40 (1H, d,  $J$  = 9.0 Hz, H-6'), 7.44 (2H, br d,  $J$  = 7.6 Hz, H-2 and H-6), 12.67 (1H, s, OH-2', exchangeable).

*(2S)-(-)-5'-Hydroxy-7,3',4'-trimethoxyflavanone (3)*: Yellow amorphous solid.  $[\alpha]_{\text{D}}^{25}$ :  $-15.2^\circ$  ( $c$  0.08,  $\text{CHCl}_3$ ); CD (MeOH): nm ( $\Delta\epsilon$ ) = 328 (+2.9), 301 ( $-3.5$ ), 236 (+6.6); UV (MeOH):  $\lambda_{\max}$  (log  $\epsilon$ ) = 211 (4.41), 230 (sh, 4.14), 282 (4.18), 310 nm (3.87); (KOH): 209 (4.54), 255 (3.97), 339 nm (4.26); IR (KBr):  $\nu_{\max}$  = 3395 (br, OH), 1662 (C=O), 1595, 1504, 1458  $\text{cm}^{-1}$  (aromatic ring C=C stretch); EI-MS:  $m/z$  (rel. int.) = 330 ( $M^+$ , 61), 312 (8), 297 (11), 277 (9), 196 (47), 181 (100), 153 (27), 137 (18), 69 (35); HR-EI-MS:  $m/z$  found: 330.1100 [ $M$ ] $^+$ , calcd.

for C<sub>18</sub>H<sub>18</sub>O<sub>6</sub>: 330.1103; <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 500 MHz): δ = 2.80 (1H, dd, *J* = 17.0, 2.6 Hz, H-3β), 3.01 (1H, dd, *J* = 17.0, 13.5 Hz, H-3α), 3.84 (3H, s, OMe-7), 3.90 (3H, s, OMe-3'), 3.92 (3H, s, OMe-4'), 5.36 (1H, dd, *J* = 13.5, 2.6 Hz, H-2), 6.51 (1H, d, *J* = 2.5 Hz, H-8), 6.59 (1H, d, *J* = 2.0 Hz, H-2'), 6.62 (1H, dd, *J* = 9.0, 2.5 Hz, H-6), 6.72 (1H, d, *J* = 2.0 Hz, H-6'), 7.87 (1H, d, *J* = 9.0 Hz, H-5).

*Muntingone* (**4**): Yellowish powder from MeOH, m.p. 166 – 168 °C. UV (MeOH): λ<sub>max</sub> (log ε) = 210 (4.34), 234 (sh, 4.02), 274 (4.13), 317 (3.81), 369 nm (3.80); (KOH): 210 (4.56), 278 (4.06), 286 (4.06), 320 (sh, 3.80), 379 nm (3.63); IR (KBr): ν<sub>max</sub> = 3250 (br, OH), 1656 (C=O), 1602, 1501, 1452 cm<sup>-1</sup> (aromatic ring C=C stretch); EI-MS: *m/z* (rel. int.) = 296 (M<sup>+</sup>, 79), 279 (11), 267 (12), 253 (17), 167 (34), 149 (100), 71 (24); HR-EI-MS: *m/z* found: 296.0683 [M]<sup>+</sup>, calcd. for C<sub>17</sub>H<sub>12</sub>O<sub>5</sub>: 296.0679; <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 500 MHz): δ = 3.89 (3H, s, OMe-10), 5.28 (2H, s, H-5), 6.38 (1H, d, *J* = 2.0 Hz, H-9), 6.50 (1H, d, *J* = 2.0 Hz, H-11), 7.21 (1H, br d, *J* = 8.0 Hz, H-4), 7.47 (2H, m, H-2 and H-3), 7.81 (1H, br d, *J* = 8.5 Hz, H-1), 12.54 (1H, s, OH-8, D<sub>2</sub>O exchangeable); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz): δ = 55.8 (OMe-10), 68.4 (C-5), 92.5 (C-11), 97.9 (C-9), 106.4 (C-7a), 121.8 (C-1), 124.6 (C-2), 124.8 (C-12b), 128.8 (C-3), 131.5 (C-4), 131.8 (C-4a), 135.9 (C-6a), 147.8 (C-12a), 156.4 (C-11a), 162.3 (C-8), 165.4 (C-10), 175.4 (C-7).

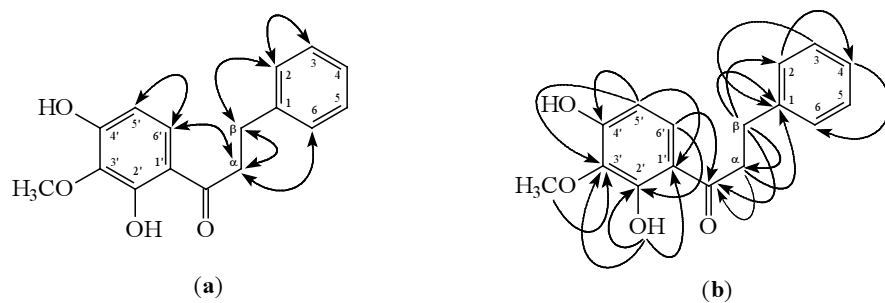


Fig. 1 Significant NOESY (a) and HMBC (b) correlations of 1.

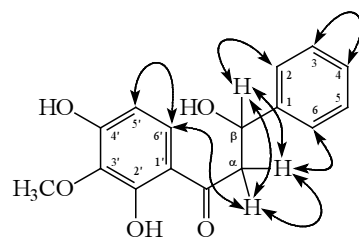


Fig. 2 Significant NOESY correlations of 2.

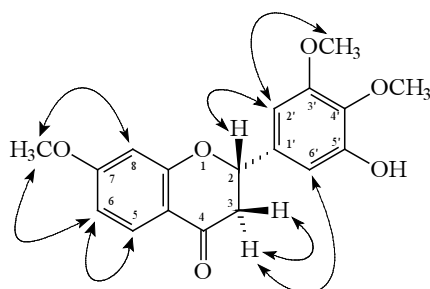


Fig. 3 Significant NOESY correlations of 3.

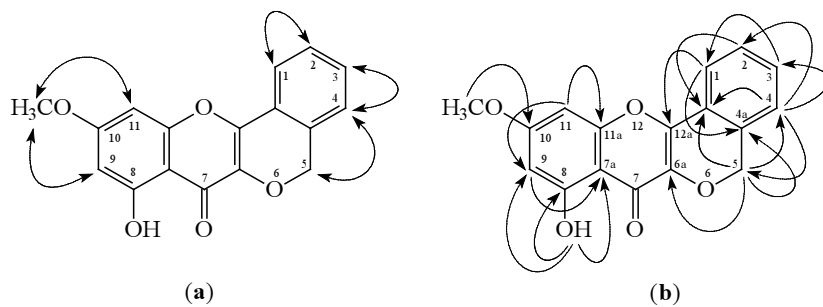


Fig. 4 Significant NOESY (a) and HMBC (b) correlations of 4.