Transtaganolides A-D: Novel Metabolites from *Thapsia transtagana*

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Experimental Section

General Experimental Section. Melting points are uncorrected and were measured in Reichert-Jung apparatus. Optical rotations were determined in a Perkin Elmer Model 341 digital polarimeter. IR spectra were measured in a Perkin Elmer Spectrum BX spectrophotometer. ¹H (1D, DQF-COSY, 1D-NOESY, 2D-NOESY) and ¹³C (1D, gHSQC, HMBC) NMR spectra were recorded in Varian Inova 400 spectrometer. Mass spectra were recorded at the Mass Spectra Facilities of Universidad de Santiago de

Compostela (Spain) in a Micromass Autospec spectrometer. Solvents were distilled prior to use, and spectral grade solvents were used for spectroscopic measurements. TLC was performed on plates precoated with silica gel F₂₅₄ (Merck, Germany).

Plant material. Roots of *Thapsia transtagana* (300 g) were collected in Bouznika (SW Morocco) and a voucher of the plant is deposited in the Departamento de Ciencias y Recursos Agrícolas y Forestales of the Universidad de Córdoba.

Isolation. The roots were grounded and extracted with CH₂Cl₂ in a Soxhlet apparatus yielding 24 g of an oily residue, which was purified by column chromatography with increasing polarities of EtOAc/hexanes mixtures. The 1:4 EtOAc/hexanes-eluted fraction was subjected to further column purification yielding 12 mg of hapolonolide (1). The 1:3 EtOAc/hexanes-eluted fraction yielded after further purification by column chromatography, thapsitranstagin (2) (1.5 g), transtaganolide A (48 mg), transtaganolide B (25 mg), transtaganolide C (60 mg), and transtaganolide D (48 mg).

Transtaganolide A (1): colorless crystals, mp 175-177 °C; $[\alpha]_D^{25}$ -44.8 (*c* 0.16, CHCl₃); IR (NaCl) $\nu_{\max}$ 2941, 1780, 1670, 1236, 997, 756; ¹H NMR (CDCl₃, 400 MHz) and ¹³C NMR (CDCl₃, 100 MHz) data, see table 1; HRCIMS *m/z* 358.1415 (calcd. for C₂₀H₂₂O₆, 358.1416); CIMS *m/z* (rel. int.) 359 (6) [M+1]⁺, 358 (17) [M]⁺, 314 (10), 225 (100), 197 (84), 183 (27), 155 (57).

Transtaganolide B (2): colorless crystals, mp 171-172 °C; $[\alpha]_D^{25}$ -25.8 (*c* 0.12, CHCl₃); IR (NaCl) $\nu_{\max}$ 2934, 1780, 1668, 1166, 998, 756; ¹H NMR (CDCl₃, 400 MHz) and ¹³C NMR (CDCl₃, 100 MHz) data, see table 1; HRCIMS *m/z* 358.1412 (calcd. for C₂₀H₂₂O₆, 358.1416); CIMS *m/z* (rel. int.) 359 (3) [M+1]⁺, 358 (12) [M]⁺, 314 (5), 225 (100), 197 (79), 183 (22), 155 (46).

Transtaganolide C (3): amorphous solid; $[\alpha]_D^{25}$ -10.0 (*c* 0.19, CHCl₃); IR (NaCl) $\nu_{\max}$ 2971, 1758, 1667, 1619, 1457, 1334, 1232, 1175, 1050, 1002, 969, 915, 753; ¹H NMR (CDCl₃, 400 MHz) and ¹³C NMR (CDCl₃, 100 MHz) data, see table 1; HRCIMS *m/z* 344.1621 (calcd. for C₂₀H₂₄O₅, 344.1624); CIMS *m/z* (rel. int.) 345 (13) [M+1]⁺, 344 (8) [M]⁺, 301 (100), 300 (83), 285 (29), 273 (27), 269 (73).

Transtaganolide D (4): amorphous solid; $[\alpha]_D^{25}$ -14.2 (*c* 0.07, CHCl₃); IR (NaCl) $\nu_{\max}$ 2986, 2925, 1756, 1668, 1619, 1455, 1333, 1267, 1236, 1194, 1177, 1104, 1008, 954, 915, 756; ¹H NMR (CDCl₃, 400 MHz) and ¹³C NMR (CDCl₃, 100 MHz) data, see table 1; HRCIMS *m/z* 344.1625 (calcd. for C₂₀H₂₄O₅, 344.1624); CIMS *m/z* (rel. int.) 345 (13) [M+1]⁺, 344 (5) [M]⁺, 329 (14), 301 (100), 300 (70), 285 (22), 273 (28), 269 (72).

transtaganolide A

transtaganolide A

transtaganolide B

transtaganolide B

transtaganolide C

transtaganolide C

transtaganolide D

transtaganolide D