Supporting Information to:

Structural Revision of Two Flavanonol Glycosides from *Smilax glabra*

Xiang Zhou¹, Qiang Xu¹, Jian-Xin Li², Ting Chen¹

Affiliation

¹ State Key Laboratory of Pharmaceutical Biotechnology, School of Life Sciences, Nanjing University, Nanjing, P.R. China
² Institute of Medicinal Chemistry, School of Chemistry and Chemical Engineering, Nanjing University, Nanjing, P.R. China

Correspondence

*Ting Chen*
State Key Laboratory of Pharmaceutical Biotechnology
School of Life Sciences
Nanjing University
Nanjing 210093
People’s Republic of China
Tel.: +86-25-8368-6786
Fax: +86-25-8359-7620
chenting.nju@163.com

HPLC-UV Analysis

LC-UV data were obtained with a Shimadzu 20A series HPLC system consisting of two pumps (LC-20A Solvent Delivery Unit), a column oven (CTO-10ASVP), an SPD detector (SPD-M20AV Photodiode Array Detector), and an LCsolution Work Station.
HPLC conditions: YMC-pack Pro C18 column (5 μm, 150 × 4.6 mm; YMC Co., Ltd.); solvent system: methanol–water (42:58, v/v); flow rate: 1.0 mL/min; injection volume: 5 μL; detection wavelength: 291 nm.

The mobile phase was degassed by ultrasonication and was filtered through a 0.22-μm membrane filter (ADVANTEC; Tokyo Roshi Kaisha). Before sample analysis, the column was stabilized with the mobile phase for at least 30 min. Under the analytical conditions above, the retention times of neoastilbin, astilbin, neoisoastilbin, and isoastilbin were 7.5, 8.2, 10.8, and 11.8 min, respectively.

**Preparative HPLC**

The standards of the four compounds were prepared with a Waters 600 HPLC system consisting of a Waters 600 Controller, a Waters 2487 Dual λ Absorbance Detector, a Waters 600 Pump, and an Empower Work Station.

HPLC conditions: Develosil ODS-5 column (5 μm, 250 × 20 mm; Nomura Chemical); solvent system: methanol–water (30:70, v/v); flow rate: 5.0 mL/min; detection wavelength: 291 nm.

**X-ray single-crystal analysis**

The x-ray analysis was performed with a Bruker Smart Apex CCD area detector. Suitable crystals of neoastilbin (neosmitilbin) were grown from H2O–methanol as needles. Intensity data were collected at 0 °C on a Bruker Smart Apex CCD area detector using MoKα graphite monochromated radiation (λ = 0.7073 Å) with phi and omega scans. The structure was solved by direct methods using the SHELXTL system, which was used for all further calculations. The H-atoms were refined isotropically and the non-hydrogen atoms anisotropically using weighted full-matrix least-squares. Crystal data for neoastilbin (neosmitilbin): C21H22O11·4(H2O); orthorhombic; space group P2; a = 7.1402(8), b = 14.8802(17), c = 22.775(3) Å, α = 90.0°, β = 90.0°, γ = 90.0°, V = 2419.8(5) Å³, Z = 4; observed reflections [1
Further details of the crystal structure investigation can be obtained from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, United Kingdom, by quoting the full journal citation (deposition number: CCDC 622556).

**Fig. 1S** HPLC diagram of the reaction products and four standards (the compound names in parentheses were mistakenly determined).
**Fig. 2S** $^1$H-NMR spectra of 4 isomers (the compound names in parentheses were mistakenly determined).