

A New Pyrone from *Elaeagnus Glabra*Yang T.L.¹, Liu C.M.², Li W.J.³, Li H.T.⁴, Liu S.L.^{5*} and Chen C.Y.^{6*}

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ABSTRACT

A new 2-pyrone, elaeagpyrone (1) was isolated from the leaves of *Elaeagnus glabra* (Elaeagnaceae). The structure of the new 2-pyrone was elucidated by chemical and physical evidence.

Keywords

Elaeagnus glabra, Elaeagnaceae, 2-pyrone.

Introduction

There are about 90 species of *Elaeagnus* around the world. The majority are native to the temperate and subtropical regions in Asia, of which nine species can be found in Taiwan [1]. Many species of *Elaeagnus* are considered as folk medicinal plants, e.g., *E. umbellata* [2], *E. pungens* [3], *E. angustifolia* [4] and *E. multiflora* [5]. Triterpenoids, steroids and flavonoids have been isolated from several species of *Elaeagnus* e.g., *E. ungens* [6], *E. umbellata* [6], *E. bockii* [7], *E. orientalis* [8] and *E. pungens* [9]. In the course of screening for biologically and chemically novel agents from Formosan elaeagnaceous plants, *E. glabra* Thunb. was chosen for further phytochemical investigation. *E. glabra* Thunb. is an evergreen shrub or small tree with alternate leaves, inhabits East Asia and is reported to have anti-cancer, anti-bacterial, procoagulant, anti-asthmatic and anti-diarrheal effects [10]. These observations provide useful information for potential chemopreventive drug design. Previously, we isolated 16 compounds, including four flavonoids, six benzenoids and six

steroids from the leaves of this plant [11]. In this paper, we report the isolation and structural elucidation of this new 2-pyrone.

Elaeagpyrone (1) was obtained as a white amorphous powder and its molecular formula was deduced as C₈H₈O₄ by HRESIMS (m/z 191.0318 [M + Na]⁺; calc. 191.0320). The UV spectrum of 1 showed intense absorption bands at 205 and 252 nm, which were typical of a pyrone skeleton [12]. The IR spectrum of 1 exhibited absorption bands at λ 1720 and 1640 cm⁻¹, indicating two carbonyl groups, respectively. The structure was confirmed from the ¹H NMR spectrum, which contained signals at δ 6.37 (1H, dd, J = 9.2, 1.0 Hz, H-4), 7.82 (1H, d, J = 9.2 Hz, H-3) and 7.84 (1H, d, J = 1.0 Hz, H-6) on the pyrone ring, δ 3.39 (2H, s, H-7) and δ 3.75 (3H, s, OCH₃). The ¹³C NMR and DEPT experiments for 1 showed eight resonance lines consisting of one methyl, one methylene, three methines, and three quaternary carbons (including two carbonyl signals at δ 159.7 and 174.5). The structure of 1 was also confirmed by 2D NMR experiments. A COSY correlation was observed between H-3 and H-4. The HETCOR experiment showed that the carbon signals at δ 117.9 for C-3, 147.1 for C-4, 153.8 for C-6, 30.5 for C-7 and 52.3 for OCH₃ were correlated

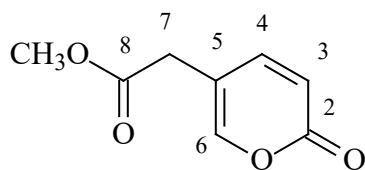
to the proton signals at δ 7.82 for H-3, δ 6.37 for H-4, δ 7.84 for H-6, δ 3.39 for H-7 and δ 3.75 for OCH₃, respectively. The above results support the structure of 1, as a new 2-pyrone, methyl ester of 2-pyrone-5-acetic acid, which we name elaeagpyrone.

Experimental

General: IR, Hitachi 260-30 spectrophotometer; 1D and 2D NMR, Varian (Unity Plus) NMR spectrometer (400 MHz, using CDCl₃ as solvents for measurement); Low-resolution ESI-MS, API 3000 (Applied Biosystems); High-resolution ESI-MS, Bruker Daltonics APEX II 30e spectrometer; Silica gel 60 for CC and precoated silica gel plates (Merck) were used for TLC, visualized with 10% H₂SO₄.

Plant Material: The specimen of *E. glabra* Thunb. was collected from Fuxing Township, Taoyuan City, Taiwan in March, 2014. A voucher specimen was identified by Professor Fu-Yuan Lu (Department of Forestry and Natural Resources College of Agriculture, National Chiayi University) and was deposited in the School of Medical and Health Sciences, Fooyin University, Kaohsiung, Taiwan.

Extraction and Isolation: The leaves (1.4 kg) of *E. glabra* were air dried and extracted repeatedly with MeOH (5 L \times 3) at room temperature. The combined MeOH extracts (15.6 g) were then evaporated and further separated into 10 fractions by column chromatography on silica gel (4.2 kg, 70-230 mesh) with gradients of n-hexane/CH₂Cl₂/MeOH. Fraction 9 (2.4 g) was subjected to silica gel chromatography, eluting with CH₂Cl₂-MeOH (70:1), and enriched gradually with MeOH, to obtain four fractions (9-1-9-4). Fraction 9-2 (0.3 g) was further purified on a silica gel column using CH₂Cl₂/MeOH mixtures to obtain elaeagpyrone (1) (6 mg).



Elaeagpyrone (1): White amorphous powder; IR (neat) ν_{\max} : 1720 (C=O), 1640 (C=O) cm⁻¹; UV/Vis (CH₃CN): λ_{\max} (log ϵ): 252 (2.65), 205 (2.21) nm; MS (ESI): m/z (%): 191 [M + Na]⁺; HRMS-ESI: m/z [M + Na]⁺ calcd for C₈H₈O₄Na: 191.0320; found: 191.0318; ¹H NMR (400 MHz, CDCl₃): 3.39 (2H, s, H-7), 3.75 (3H, s, OCH₃) 6.37 (1H, dd, J = 9.2, 1.0 Hz, H-4), 7.82 (1H, d, J = 9.2 Hz, H-3), 7.84 (1H, d, J = 1.0 Hz, H-6); ¹³C NMR (100 MHz, CDCl₃): 30.5 (C-7, CH₂), 52.3 (OCH₃, CH₃) 117.9 (C-3, CH), 124.0 (C-5, C), 147.1 (C-4, CH), 153.8 (C-6, CH), 159.7 (C-

2, C=O), 174.5 (C-8, C=O).

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