Two novel iridoids from *Scrophularia buergeriana*

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**Abstract**

Two new iridoid-related compounds with a new carbon skeleton, buergerin F (1) and G (2) were isolated from the roots of *Scrophularia buergeriana* Miq., and their structures were determined by spectroscopic means as well as by X-ray crystallographic analysis. The $^1$H, $^{13}$C NMR and EI-MS data of 1 and 2 are given. © 2000 Elsevier Science Ltd. All rights reserved.

In an extension of a study of the chemical constituents of *Scrophularia buergeriana* Miq., 1 two new-skeleton iridoids, buergerin F (1) and G (2) were isolated and identified.5,6 The $^1$H and $^{13}$C NMR spectral data of compounds 1 and 2 and X-ray structure of 2 are given.

![Structures of compounds 1 and 2](image)

Compound 1 was obtained as an oil. The IR spectrum of 1 showed no absorption of any hydroxy group. The molecular formula was determined as C$_9$H$_{14}$O$_3$ by HRMS (M$^+$: 170.0941, calculated: 170.0943), along with $^1$H and $^{13}$C NMR spectral data (Table 1). The IR and $^{13}$C NMR spectra of 1 showed that no unsaturated bond existed in 1. In view of the degree of unsaturation, three rings were required. The $^1$H NMR spectrum of 1 showed a tertiary methyl signal at $\delta$ 1.50 (3H, s) and a methine signal at $\delta$ 4.47 (1H, dd, $\text{J}=6.3$, 1.5 Hz) linked to oxygen. The $^{13}$C NMR and DEPT spectra of 1 showed the existence of five methene carbons, a quaternary carbon linked to oxygen and an acetal carbon at $\delta$ 108.6(s). $^1$H-$^1$H COSY experiments indicated the presence of three segments as follows:

A) $\text{O-CH}_2-\text{CH}_2$  
B) $\text{O-CH}_2-\text{CH}_2$  
C) $\text{O-CH}_2-\text{CH}_2$  

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Furthermore, the three segments A, B and C were connected by the $^2J_{^1H-^3H}$ correlations shown in HMBC spectrum. The most plausible structure of 1 is indicated in Fig. 1. The relative stereochemistry, with C-9 and O-11 in the cis-configuration is the more stable and was confirmed by the NOESY spectrum (Fig. 1) in which cross peaks are shown between H-1 and H-7$\alpha$, H-9$\beta$ and H-10.

Compound 2 was obtained as colorless prismatic crystals. The HRMS of 2 showed an M$^+$ ion at $m/z$ 184.0736 (calculated: 184.0735) indicating its molecular formula to be $C_{9}H_{12}O_{4}$, in agreement with $^1H$ and $^{13}C$ NMR spectral data. The NMR data for 2 showed it was similar in structure to 1, but the left ring was a γ-lactone moiety confirmed by IR ν (KBr) cm$^{-1}$: 1765, $^1H$ and $^{13}C$ NMR spectral data (Table 1). Accordingly, compound 2 was identified as 3-one-buergerinin F.

In order to complete the structural elucidation of 2, an X-ray crystallographic analysis was performed. The relative stereoscopic view of the molecule is shown in Fig. 2.

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Fig. 2. X-Ray structure of 2

References

4. X-Ray analysis was performed on a Rigaku AFC7R diffractometer with graphite monochromated Mo-Kα radiation and a 12 kW rotating anode generator.
5. Plant material, Scrophularia buergeriana Miq. was collected from Changbai mountains in Jilin province, China, in 1996.
6. Extraction and isolation. The air-dried roots were extracted with 60% EtOH at room temperature. A part of the petrol soluble fraction was chromatographed on a silica gel column with petrol:EtOAc (7:1) and then purified with CH₂Cl₂:acetone (3:1) to give compound 1. Compound 2 was obtained by recrystallization from the mixed solvent (petrol and acetone) after column chromatography on silica gel with petrol:EtOAc (4:1).
7. Buergerinin F (1). Yellow oil. [α]_D^25 +40.67 (c 0.431 CHCl₃). IR ν (KBr) cm⁻¹: 1388, 1234. EI-MS: m/z 170 M⁺ (14), 110 (100), 84 (99). ¹H and ¹³C NMR (CDCl₃): see Table 1.
8. Buergerinin G (2). Colorless prismatic crystal. Mp:152–154°C. [α]_D^25 +47.71 (c 0.509 CHCl₃). IR ν (KBr) cm⁻¹: 1765, 1230. EI-MS: m/z 184 M⁺ (25), 80 (100). ¹H and ¹³C NMR (CDCl₃): see Table 1.