

*Picris echoides*

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(Received 20 June 2000)

Investigation of the constituents of the aerial parts of domestic plant species afforded the sesquiterpene lactones, guaianolides jacquilenin ( ), 11- $\beta$ -jacquilenin ( ), achillin ( ) and eudesmanolide telekin ( ). The chemical identification of the two monoterpene glucosides (–)-chrysanthenol- $\beta$ -D-glucopyranoside ( ) and its 6'-acetate is also reported. The guaianolide achillin ( ) and the two monoterpene glucosides and were isolated for the first time from this plant species. Isolation was achieved by column chromatography and the structures were established mainly by the interpretation of their physical and spectral data, which were in agreement with those in the literature.

: , sesquiterpene lactones, monoterpene glucosides.

## INTRODUCTION

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3–6

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# Serbian Chemical Society active member

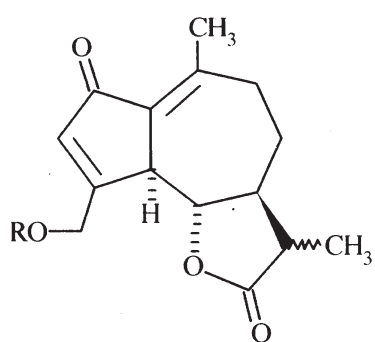
7,8

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## RESULTS AND DISCUSSION

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7,8,12

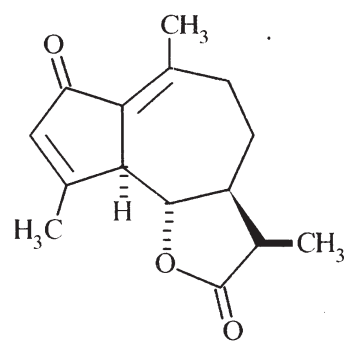


**1.** R = H, 11 $\beta$ H

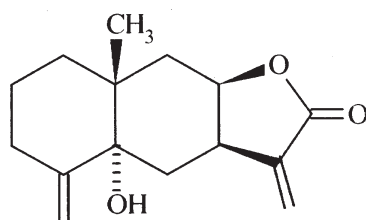
**1a.** R = Ac, 11 $\beta$ H

**2.** R = H, 11 $\alpha$ H

**2a.** R = Ac, 11 $\alpha$ H



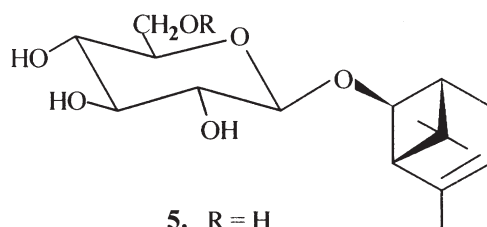
**3.**



**4.**

Scheme 1. Sesquiterpene lactones from the plant species

TERPENE LACTONES AND GLUCOSIDES



5. R = H

6. R = Ac

Scheme 2. Monoterpene glucosides from the

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## EXPERIMENTAL

Aerial parts of *Telekia speciosa* has traditional name Grgusa (family *Compositae*, tribe *Astereae*)<sup>1,19</sup> were collected in the vicinity of Mt. Rudnik in July 1995 and 1996 and authenticated by Dr. P. Marin, from the Department of Botany at the Faculty of Biology, University of Belgrade. A voucher specimen has been deposited in the herbarium of this Department.

Air-dried, finely ground aerial parts of *Telekia speciosa* (2.5 kg) were extracted for 48 h at room temperature with petrol-Et<sub>2</sub>O-MeOH, 1:1:1 (10 liters). After concentration at reduced pressure in a rotary evaporator, the residue was dissolved in boiling MeOH (1 liter) and cooled to the room temperature. The supernatant liquid was decanted from the waxy precipitate and then concentrated giving 32 g of dark greenish oil as residue. This residue was prefractionated by column chromatography on silica gel. The three fractions were collected after elution with solvents: petrol-Et<sub>2</sub>O, 1:3, 1:1; Et<sub>2</sub>O, and finally with Et<sub>2</sub>O-MeOH, 6:1, fraction 3. The fractions 1, 2 and 3 were subjected for further repeated CC on silica gel, respectively.

Fraction 1 (18 g) contained mainly the pigments, waxes and wide spread sterols. Elution was begun with petrol-Et<sub>2</sub>O, 9:1 and then gradually increasing the polarity by addition of Et<sub>2</sub>O, giving the lactone 1 with petrol-Et<sub>2</sub>O (8 mg, recrystallised from hexane-ether). It was isolated as colorless needles, melted at 133–134 °C and  $[\alpha]_D^{20} = +115$  (c 1, CHCl<sub>3</sub>), same as the literature,<sup>14</sup> EIMS,  $m/z$  (rel. int. %): 246 (M<sup>+</sup>, 47), 231 (6), 217 (23), 203 (25), 190 (15), 91 (100) and the other spectral data corresponded to those published. The polar fraction 2 (5.2 g), contained the material eluted with Et<sub>2</sub>O, previously, was further submitted to CC, starting with petrol-Et<sub>2</sub>O (1:1). The two main fractions were selected after the inspection by NMR and in the next step fractionated by HPLC (RP-18, MeOH-H<sub>2</sub>O, 3:2, 150 bar), respectively. The first fraction could not be separated by HPLC containing mixture of the epimers 2 and 3. Acetylation (Ac<sub>2</sub>O, 2 h, 70 °C) it gave the acetates, which were separated by silica gel CC, with petrol-Et<sub>2</sub>O (2:1) affording the 4, with  $[\alpha]_D^{20} = +105$  (c 0.7, CHCl<sub>3</sub>) (12 mg) and 5, with  $[\alpha]_D^{20} = +44$  (c 0.1 CHCl<sub>3</sub>) (8 mg), as colorless gummy materials. Their IR, NMR and MS data corresponded to those published. The second fraction, after separation by HPLC yielded lactone telekin (6, 13 mg), as the white crystals.

The more polar fraction 3 (2.5 g) was separated by silica gel CC (Et<sub>2</sub>O-MeOH, 7:1) yielding the (–)- 7-chrysanthenol-β-D-glucopyranoside (7, 18 mg), gum with  $[\alpha]_D^{20} = -44$  (c 0.8, MeOH); IR ( $\nu_{\max}$  cm<sup>-1</sup>): 3400, 1368, 1210, 1070, 1040, 1025, 760. EIMS  $m/z$  (rel. int.): 296.1624 (M-H<sub>2</sub>O)<sup>+</sup> (8), 268 (4), 192 (8), 190 (20), 175 (16), 149 (22), 134 (33), 119 (82), 93 (59), 91 (100), 55 (67). Calc. for C<sub>16</sub>H<sub>26</sub>O<sub>6</sub>-H<sub>2</sub>O, M<sup>+</sup>, 296.1618. The compound 8 (10 mg) was dissolved in 10 ml of methanol and 20 ml of 6 % HCl was added. The solution was heated on a water bath for 45 min, cooled and extracted several times with water. The solvent was removed and the remaining substance identified as (–)- 7-chrysanthenol and glucose was also detected in the water solution.<sup>16</sup> Elution with Et<sub>2</sub>O-MeOH, 7:1 as solvent mixture also afforded the (–)- 7-chrysanthenol-β-D-glucopyranoside-6'-acetate (9, 11 mg), gum with  $[\alpha]_D^{20} = -126$  (c 0.5, MeOH); IR ( $\nu_{\max}$  cm<sup>-1</sup>): 3400, 1725, 1436, 1365, 1250, 1215, 1080, 1030, 750. EIMS  $m/z$  (rel. int.): 296.1624 (M-HOAc)<sup>+</sup> (20), 192 (38), 190 (29), 175 (42), 149 (90), 134 (38), 119 (100), 107 (48), 105 (31), 93 (55), 91 (80). Calc. for

$C_{18}H_{28}O_7$ -HOAc,  $M^+$ , 296.1618. NMR  $^{13}C$ - and  $^1H$ - spectral data corresponded to those published.<sup>18</sup>

The authors are grateful to the Ministry of science and technology of the Republic of Serbia for financial support.

# ИЗВОД

## СЕСКВИТЕРПЕНСКИ ЛАКТони И МОНОТЕРПЕНСКИ ГЛУКОЗИДИ БИЉНЕ ВРСТЕ *Picris echinoides* L.

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Из надземног дела домаће биљне врсте (фамилија *е*), корова познатог под именом Гргуша, изолована су и идентификована четири сесквитерпенса лактона, као и два монотерпенса глукозида. Идентификовани лактони су гвајанолиди јаквиленин ( ), 11- -јаквиленин ( ) и ахилин ( ) као и еудесманолид телекин ( ). Монотерпенси глукозиди (–)- -кризантенол- $\beta$ -D-глукопиранозид ( ) и његов 6'-ацетат, су такође изоловани из истог екстракта. Гвајанолид ахилин ( ) и монотерпенси глукозиди ( , ) су по први пут изоловани из ове биљне врсте, док су преостала три лактона изолована више пута из европске биљне врсте.

(Примљено 20. јуна 2000)

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