

## A Note on the Isolation of Solamargine from *Solanum nigrum*

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In the course of an investigation of alkaloids occurring in different species of the plant genus *Solanum* two alkaloidal glycosides were chromatographically detected in *S. nigrum*. The one with the highest  $R_F$ -value was subsequently isolated and identified as solamargine, an alkaloidal glycoside first isolated from *S. marginatum*<sup>1</sup>.

The isolation procedure reported by Kuhn and Löw<sup>2</sup> was followed. The green, oily residue obtained by this procedure was dissolved in water-saturated 1-butanol and chromatographed on a column of wet-packed alumina (Alcoa). Elution with water-saturated 1-butanol gave a yield of 650 mg alkaloid mixture from 2.93 kg of fresh plant tops.

Using the same developing solvent and technique as Kuhn and Löw<sup>2</sup> for paper chromatography two alkaloid spots with  $R_{\alpha}$ -solanine = 1.04 and  $R_{\alpha}$ -solanine = 1.72 were obtained (travelling distance of solvent 31.2 cm. The  $R_F$ - and  $R_{\alpha}$ -solanine-values depend highly upon both experimental conditions and travelling distance of solvent).

When the alkaloid mixture, dissolved in water-saturated 1-butanol, was rechromatographed on a column of wet-packed alumina (Alcoa) and eluted with water-saturated 1-butanol the alkaloid residue consisted mainly of the alkaloid component with the highest  $R_F$ -value. Repeated crystallization from ethanol-water (1:1) gave a chromatographically pure compound, m.p. 304–310°C (decomp., corr.),  $[\alpha]_D^{25} = -99^\circ$  (methanol,  $c$  0.531). (Literature values for solamargine<sup>1</sup>: m.p. 301°C (evacuated tube, decomp.),  $[\alpha]_D^{20} = -105^\circ$  (methanol)). Potentiometric titration of the glycoside with 0.02 N perchloric acid in glacial acetic acid gave an equivalent weight of 914 (calc. 868 for anhydrous solamargine).

At this stage of the investigation a review article was published by Schreiber<sup>3</sup>.

In his paper a footnote was added in proof stating that two chromatographically detected alkaloids,  $\gamma$ - and  $\delta$ -solanigrine, from *S. nigrum*<sup>4</sup> were identical with solasonine and solamargine, respectively. No analytical results were submitted.

The aglycone was isolated by acid hydrolysis of the glycoside following the procedure used by Briggs *et al.*<sup>1</sup> The aglycone melted at 199–200°C,  $[\alpha]_D^{25} = -98^\circ$  (methanol,  $c$  0.481). No melting point depression was observed when mixed with authentic solasodine (Literature values for solasodine<sup>1</sup>: m.p. 197.5–198.5°C,  $[\alpha]_D^{20} = -97.1$  (methanol)). The infrared spectrum of the aglycone was identical with the spectrum of solasodine.

The acidic mother liquor remaining after hydrolysis of the glycoside and precipitation of the aglycone was by means of paper chromatography shown to contain D-glucose and L-rhamnose. Aliquots were chromatographed on Whatman No. 1 paper at 23°C. Aniline hydrogen phthalate was used as detecting reagent.

The solvent mixture 1-butanol-ethanol-water (8:1:2) revealed spots with the following  $R_F$ -values: 0.44 L-rhamnose, 0.16 D-glucose.

The solvent mixture ethyl acetate-water-pyridine (2:2:1) revealed spots with the following  $R_F$ -values: 0.48 L-rhamnose, 0.27 D-glucose.

The results were controlled by parallel runs of pure monosaccharides alone and in mixtures.

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