Chemical Study of *Cestrum parqui*

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Solasonine and digitogenin were isolated and identified in the acid extract from *Cestrum parqui*.

THE BARK of *Cestrum parqui* L'Herit., a Chilean *Solanaceae*, is frequently used as an infusion for the treatment of common cold.

In connection with a systematic study on saponins of certain typical Chilean species (1–4), now under way in this laboratory, it appeared of interest to study *Cestrum parqui* in order to elucidate the presence or absence of alkaloids and sapogenins which have been reported repeatedly in current literature. Canham and Warren (5) have reported that *Cestrum parqui* contains two sapogenins, gitogenin and digitogenin. However, the authors failed to isolate any alkaloids. Mercier and Chevalier (6) had previously reported the presence of parquin. Several other investigators (7–10) had failed to isolate either parquin or any alkaloids.

This is a chemical study of the acetic acid extract of dried, pulverized, and benzene-defatted plant. This extract yielded a water-soluble fraction containing a saponin and a glycosidic alkaloid. The alkaloid was identified as solasonine. The saponin was hydrolyzed and its sapogenin identified as digitogenin. The benzene-soluble fraction yielded two products to be reported in a coming paper.

EXPERIMENTAL[1,2]

Isolation of Solasonine.—Stems and leaves of *Cestrum parqui* collected in April 1959 near Concepción, Chile, were dried at 80–90°. Nine kilos of the dried plant was ground and extracted in a Soxhlet extractor with benzene to exhaustion. The defatted plant material was dried at 80° and acid-soluble constituents were extracted with 5% acetic acid solution (11). This extract was then heated and precipitated with 5% ammonia solution. Subsequent filtering was extremely slow. The solid fraction was washed with diluted ammonia and dried at 90° to yield 184.5 Gm. The crude glycosidic alkaloid was partially purified by continuous extraction with ethanol.

The ethanol-soluble constituents were purified through repeated crystallization from ethanol-dioxane-water (4:4:2), yielding 31.5 mg. of solasonine; m.p. 260–264°. The infrared spectrum was identical to that of an authentic specimen.

Anal. (12).—Calcd. for $C_{45}H_{73}NO_{16}$: C, 61.1; H, 8.3; N, 1.6. Found: C, 61.2; H, 8.4; N, 1.6.

The melting point of the alkaloid was undepressed upon admixture with an authentic specimen, m.p. 264–266°.

Isolation of Digitogenin.—The alkaline filtrate from the precipitation of the alkaloid was concentrated and acidified with hydrochloric acid to render it approximately 2.5 *N*. The mixture was then refluxed for 9 hours and poured into water and ice. The precipitate was collected on a filter, washed with water, and dried to 90°; yield 195.3 Gm. The sapogenin was then extracted with petroleum benzin (b.p. 65–75°) in a Soxhlet extractor.

The extract was purified and recrystallized from ethanol to yield 8.55 Gm. of white needles, m.p. 264–267°. This product was chromatographed over 240 Gm. alumina Light H (13). Exhaustive elutions with benzene, chloroform, and ethanol yielded 31 fractions of 60 ml. each, which represented various degrees of purity of the same product. Paper chromatograms (14) of all eluted fractions indicated the presence of a single steroid. Fractions 8 to 18 were mixed to yield 355 mg. of digitogenin. This product was recrystallized from ethanol yielding white needles, m.p. 285–288°. This product did not show ultraviolet absorption and its infrared spectrum shows evidence indicating the presence of a steroid.

Anal.—Calcd. for $C_{27}H_{44}O_5$: C, 72.3; H, 9.9. Found: C, 72.0; H, 9.9.

Digitogenin acetate.—A solution of digitogenin (50 mg.) in pyridine (0.2 ml.) containing acetic anhydride (0.4 ml.) was boiled for 1 hour. The solid obtained by pouring the mixture into water was washed successively with 2 *N* hydrochloric acid, 5% sodium hydrogen carbonate solution, and water. Needles were obtained after recrystallization from ethanol; m.p. 233–235°.

Anal.—Calcd. for $C_{33}H_{50}O_8$: C, 69.0; H, 8.8. Found: C, 69.4; H, 9.1.

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[1] Melting points are uncorrected.
[2] We are indebted to Dr. Alfred Bernhardt of the Max Planck Institut for the microanalysis of the samples.