

A Note on Tigogenin from *Digitalis Lanata**

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DURING the phytochemical investigations of the fresh leaves of *Digitalis lanata* for its cardiac glycosides, large quantities of aqueous extracts were made available. Previous investigations (1) had shown that such aqueous extracts from other *Digitalis* extracts contained saponins that could be isolated in a fair state of purity. By following these techniques a considerable quantity of a crude saponin mixture was obtained. At the time of this investigation no particular interest was manifested on our part as to the exact nature of the saponins *per se* by virtue of the difficulties that might be experienced in their purifications. Studies on the nature of the sapogenin were therefore initiated.

EXPERIMENTAL

Isolation of the Crude Saponins.—The isolation and partial purifications of the saponins from *Digitalis lanata* were carried out essentially according to the techniques described by Appel and Gisvold (1).

Hydrolysis of Saponin.—The less butanol-soluble saponin was dissolved in 20 cc. of ethanol and 40 cc. of water to which 3.44 cc. of concentrated sulfuric acid and 5 cc. of *n*-amyl alcohol was added. The mixture was refluxed for two and one-half hours. Upon cooling a white solid separated that was crystallized twice from methanol and once from acetone, m. p. 196–198°. Reported (2) m. p. for tigogenin is 203–204°.

Sapogenin (Tigogenin) Acetate.—Although the acetate of tigogenin is not very satisfactory because of its melting point, it was prepared for infrared studies. The acetate prepared in the usual way from acetic anhydride and sodium acetate was recrystallized twice from ethanol, m. p. 194–196°. Mixed melt with the original sapogenin was 174–177°.

Homogeneity of the Sapogenin.—The application of paper chromatography was used to establish the homogeneity of the sapogenin. Propylene glycol was used as the stationary phase on Whatman No. 1 paper. Benzene-chloroform (9:1) was used as the mobile phase with a running time of sixteen hours. The paper after drying was sprayed with a 25 per cent solution of trichloroacetic acid in chloroform. It was then heated for five minutes at 90° by means of a battery (four) of infrared lamps. When examined under an ultraviolet lamp, only one fluorescent spot was observed.

Infrared Studies.—A sample of the sapogenin and its acetate were examined for their infrared spectra through the courtesy of Dr. R. Norman Jones of the National Research Council, Canada. The spectra show a very close resemblance to the curves for tigogenin (3) and tigogenin acetate (3)

REFERENCES

- (1) Appel, R. M., and Gisvold, O., *THIS JOURNAL*, **43**, 215 (1954).
- (2) "Merck Index", Merck and Co., Inc., Rahway, N. J., 1952, p. 959.
- (3) Jones, R. N., Katzenellenboger, E., and K. Dobriner "Collected Infrared Absorption Spectra of Steroid Sapogenins," 1953. National Research Council of Canada and Sloan-Kettering Institute for Cancer Research.

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