## PHYTOCHEMICAL NOTES.

## No. 112. The Sterols of Achillea Millefolium.\*

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In the course of the chemical investigation of milfoil by Katherine Graham (1) a sterol melting at 134–135° had been isolated. Its acetate melted at 123–124°. However, the melting points were not very sharp. A study of the original literature on sterols seemed to indicate that the product might be a mixture rather than a chemical individual.

In order to resolve this sterol mixture, if such it should prove to be, into its components, the acetates were brominated according to Windaus and Hauth (2). When, however, upon standing no separation had taken place, crystallization was induced by the addition of a little alcohol. From the crystals thus obtained, the ether-soluble material was removed by washing. The insoluble residue melted at 203°. Apparently, it may be regarded as the tetrabromide of stigmasterol acetate. The amount obtained was too small to render debromination practicable.

As for the melting point, Windaus and Hauth (2) record  $211^{\circ}$  as the melting point of stigmasterol acetate tetrabromide. However, melting points of  $205^{\circ}$  (3),  $208^{\circ}$  (4) and  $210^{\circ}$  (5) are also recorded. Moreover, H. Sandqvist and J. Gorton (6), as late as 1930, report  $203^{\circ}$  as the melting point.

Before attempting the debromination of the ether-soluble brominated sterol acetate, the technique (2) was tried out on the corresponding cholesterol derivative. The attempt was unsuccessful because the zinc dust suspended in the glacial acetic acid coagulated. When absolute alcohol was substituted for glacial acetic acid, the experiment was successful. With this substitution in the technique, the ether-soluble brominated sterol acetate was successfully debrominated. After several recrystallizations, the debrominated acetate melted at 127.5°. Upon saponification the free sterol was obtained melting at 138°. These constants agree with those of sitosterol. Apparently, therefore, the sterol isolated from milfoil is a mixture of stigmasterol and a sitosterol in which the latter predominates.

## REFERENCES.

(1) Graham, K., JOUR. A. PH. A., 22, 819 (1933).

(2) Windaus and Hauth, Ber., 39, 4381 (1906).

(3) Nabenhamer, F. P., and Anderson, R. J., J. A. C. S., 48, 2972 (1926).

(4) Power, F. B., and Salway, A., J. Chem. Soc., 105, 212 (1914).

(5) Beschke, Erich, Ber., 47, 1853 (1914).

(6) Sandqvist, H., and Gorton, J., Ibid., 63, 1935 (1930).

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These officers will be installed at the next annual meeting of the Association which will be held in Dallas, Texas, the time to be announced later.

\* From the Laboratory of Edward Kremers.