Short Communications

A Steroidal Glycol, 22-Hydroxy-cholesterol from *Narthecium ossifragum* Huds.

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In the course of an investigation of the pigments of the flowering parts of *Narthecium ossifragum*, — a tiny lily supposed to be the cause of a sheep disease of photodynamic nature, — there was, during the chromatographic separation of the crude hypophasic carotenoids also isolated a colourless compound (I), which, from the following summary of its reactions, will be seen to be one of the two stereomeric forms of the hitherto unknown 22-hydroxy-cholesterols, m.p. $186^{\circ}_{\text{corr.}}$, $[\alpha]_D^{20} - 39$, CHCl_3 (*Anal.* Calc. for $\text{C}_{27}\text{H}_{46}\text{O}_2$: C 80.5; H 11.52. Found: C 80.0; H 11.83). The glycolic nature is demonstrated by the formation of a diacetate, m.p. 101° , $[\alpha]_D^{20} - 33$ (*Anal.* Calc. for $\text{C}_{31}\text{H}_{50}\text{O}_4$: C 76.5; H 10.35. Found: C 76.5; H 10.50), and a dibenzoate, m.p. 256° , $[\alpha]_D^{20} - 9$ (*Anal.* Calc. for $\text{C}_{41}\text{H}_{54}\text{O}_4$: C 80.6; H 8.91. Found: C 80.6; H 8.83). Catalytic hydrogenation furnished a dihydroglycol (II), m.p. 176.5° , $[\alpha]_D^{20} + 18$; diacetate, m.p. 76° , $[\alpha]_D^{20} + 14$; dibenzoate, m.p. $215-17^{\circ}$, $[\alpha]_D^{20} + 16$.

Careful oxidation of (I) with chromium trioxide gave a compound (III) of m.p.

$142.5-43.5^{\circ}$, $[\alpha]_D^{20} - 58$. The I.R. spectrum of this oxydation product indicated an unsaturated hydroxy-ketone. The found data were close to those given by Cole and Julian¹ for 22-keto-cholesterol, (m.p. $142-43^{\circ}$, $[\alpha]_D^{20} - 55$). Dr. Cole had the kindness to furnish a sample of this substance with m.p. $142-43^{\circ}$, $[\alpha]_D^{25} - 60$, and supposed by Dr. Cole to be a purer one. Mixed m.p. confirmed the identity of the two substances. Mixed m.p. of the acetate of (III) with the acetate of 22-keto-cholesterol, also kindly provided by Dr. Cole, furnished additional evidence for the identity.

The dihydroglycol (II) was oxidised with chromium trioxide to a diketone, m.p. 148° , $[\alpha]_D^{20} + 16$, which must be the previously unknown cholestane-3,22-dione. Reduction by the Clemmensen method afforded cholestane, m.p. 80° , undepressed on admixture with cholestane prepared from cholesterol.

The saponins of *Narthecium ossifragum* are presently being investigated. They seem to contain no 22-hydroxy-cholesterol. Their sapogenins have an ordinary number of oxygen atoms.

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1. Cole, W., and Julian, P. L. *J. Am. Chem. Soc.* 67 (1945) 1369.

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