Identity of Viminalol Acetate

Sir:

In their work on substances occurring in *Sarcostemma viminale*, Torrance and Marais (1) showed that this plant contained, in addition to β-amyrin and friedelin, a compound which they called viminalol acetate, and which they stated appeared to belong to the lupeol series. In the present investigation it is shown unequivocally that the compounds described by Torrance and Marais (1) as viminalol and viminalol acetate were in all respects identical with α-amyrin and α-amyrin acetate.

In the previous communication (1) the best melting point reported after chromatography on alumina for viminalol acetate, referred to as *B*, was 160–162°. Thin-layer chromatography, however, showed that this material was not pure. After repeated chromatography of this substance on aluminum oxide using benzene as solvent, a product was finally obtained with a melting point of 200–215°, which gave a single spot on thin-layer chromatography. A pure sample of *B* was eventually obtained by hydrolysis of *B*, in ethanolic potassium hydroxide, to the alcohol. The alcohol was crystallized from 95% ethanol until a constant melting point of 180–182° was obtained. Thin-layer chromatography of this alcohol gave a single spot. This pure alcohol was then acetylated, using pyridine and acetic anhydride in the cold, to give a product which, after crystallization from alcohol, gave a chromatographically pure substance of melting point 216–219°. This melting point remained constant after recrystallization.

A comparison of these melting points with that of α-amyrin 186° (2, 3) and of α-amyrin acetate 226° (2, 3) suggested the possibility of viminalol acetate being identical with α-amyrin acetate.

A number of derivatives of viminalol acetate were prepared and their melting points compared with the corresponding derivatives of α-amyrin and α-amyrin acetate described in the literature. From the physical constants given in Table I for viminalol acetate, α-amyrin acetate, and their derivatives it can be seen that viminalol acetate was in fact α-amyrin acetate.

TABLE I.—SUBSTANCES AND MELTING POINTS

Substance	M.p., °C.
α-Amyrin acetate	216–219°; lit., 226° (2, 3)
Viminalol acetate	216–219°
α-Amyrin	182°; lit., 186° (2, 3)
Alkaline hydrolysis product of viminalol acetate	180–182°
α-Amyrone	126° (2, 3)
Chromic acid oxidation of hydrolysis product of viminalol acetate	124–127°
α-Amyrenonal acetate	278° (2, 3)
Chromic acid oxidation product of viminalol acetate	278–280°
α-Amyrene	115° (2, 3)
Clemmensen reduction product of viminalol	108–111°

Furthermore, no depression in the melting points of α-amyrin and α-amyrin acetate were obtained on admixture with viminalol and viminalol acetate, respectively. (Table I.)

Thin-layer chromatography at room temperature (22°) using silica gel (G nach Stahl) and a solvent system of ethyl acetate–cyclohexane (2:8) on α-amyrin, viminalol, α-amyrin acetate, and viminalol acetate gave R_f values of 0.53 for α-amyrin and viminalol and 0.94 for α-amyrin acetate and viminalol acetate.

Finally, comparison of the infrared spectra of viminalol and viminalol acetate with authentic samples of α-amyrin and α-amyrin acetate showed that the substance previously described (1) as an unknown triterpene acetate, *viz.*, viminalol acetate is identical with α-amyrin acetate.

(1) Torrance, J. D., and Marais, J. L. C., *J. Pharm. Sci.*, **52**, 439(1963).
(2) "Elsevier's Encyclopedia of Organic Chemistry," vol. 14, Elsevier Publishing Co., New York, N. Y., 1940.
(3) Simonsen, J., and Ross, W. C. J., "The Terpenes," vol. IV, Cambridge University Press, Cambridge, England, 1957.

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