ISOLATION OF A NEW COMPOUND FROM LAVANDULA STOECHAS LINN

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(Received March 11, 1967)

A new crystalline substance, lavanol, $C_{30}H_{46\text{-}48}O_3$, m.p. 280-84°C, $[\alpha]_D^{33}$ +25°C (THF) has been isolated from *Lavandula stoechas*.

Lavandula stoechas Linn. (locally called ustukhuddus) has been used as a drug in the indigeneous system of medicine for the treatment of chest affection and to relieve biliousness. It has been considered to be cephalic, deobstruent and carminative and used in chest affections. Muslim physicians considered it to be the broom for cleaning the brain and giving it strength. [1]

Hahn *et al.* [2] isolated three sterols, besides camphor and a hydrocarbon nonacosane, from the petroleum ether extractive of the drug material. The sterols were reported to have m.p. 204-205°C, $[\alpha]_D^{29}$ +39.6°; m.p. 135-36°C $[\alpha]_D^{24}$ −23.5°; m.p. 268–70°C, $[\alpha]_D^{26}$ +67°.

Isolation of a smooth muscle relaxant principle identified as 7-methoxycoumarin has been reported. [3] A new compound, designated as lavanol, different from the sterols reported by Hahn *et al.*, has now been isolated from the ethanol extractive of the dried plant (stem) material of *Lavandula stoechas Linn.* Lavanol melts at 282–284°C, and has $[\alpha]_D^{33}$ +25° (THF). It gives no absorption in the UV spectrum above 220 mμ. In the IR spectrum it absorps at 3520, 3450, 1720 and 1700 i cm-1 (KBr pellet) thus indicating the presence of an alcoholic function in addition to carbonyl groups. It analyses for $C_{30}H_{46\text{–}48}O_3$.

On acetylation it gives a mono-acetate, $C_{32}H_{48}O_4$, which in the IR spectrum gives peaks at 1740i, 1720, 1700i and 1275 cm-1 thus showing the presence of an acetate group (1740i cm-1) in addition to the carbonyl functions present in the original molecule.

Lavanol on chromic acid oxidation yields a product which gives a positive Zimmerman reaction.

Experimental

Unless otherwise stated IR and UV spectra were taken in KBr pellet and ethanol respectively

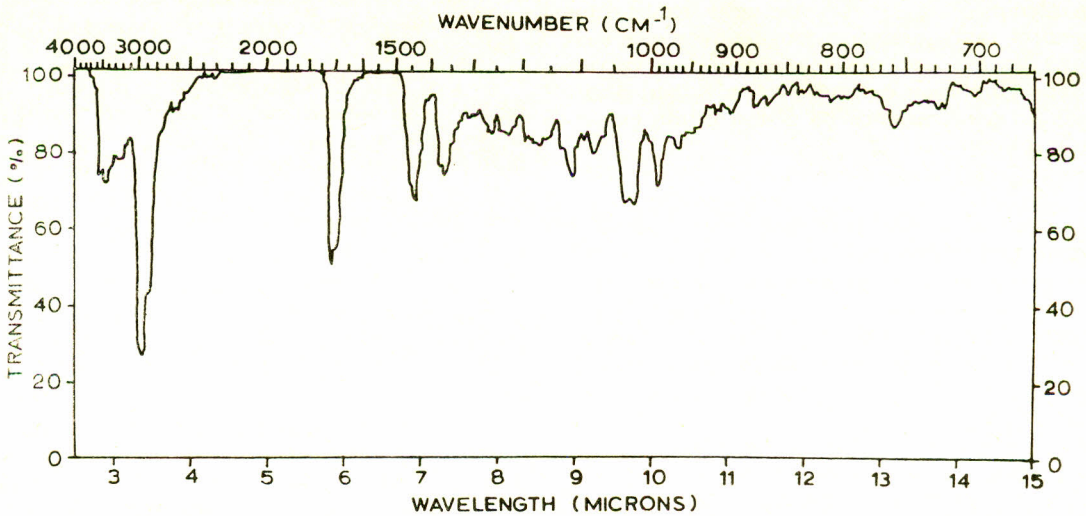

Fig. 1.—I R spectrum of lavanol.

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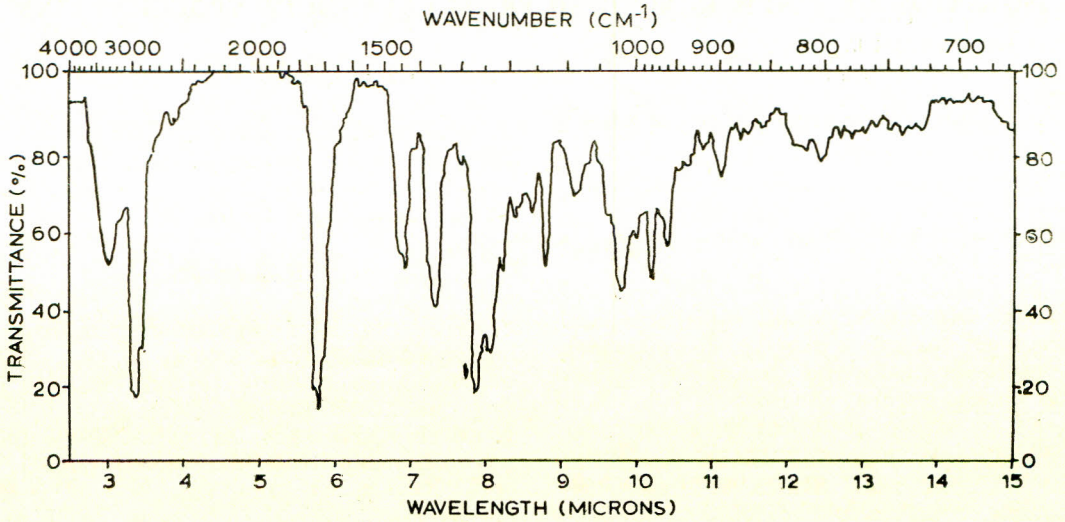

Fig. 2.—I R spectrum of lavanyl acetate.

and optical rotations were taken in ethanol. Analyses were carried out by Alfred Bernhardt Ltd., West Germany, and the Microanalytical Section of this laboratory.

Isolation of Lavanol.—The dried drug material (dried stems, leaves, flowers and seeds) was purchased from the local market. The material (500 g) was percolated with rectified spirit (1.5 l.) after soaking for 10 days at room temperature. This extraction was repeated three times by soaking in alcohol (1.5 l.) for one week each time, finally three extractions at room temperature with alcohol (1.5 l.) over 2-week period was carried out. The combined extracts were distilled under reduced pressure, whence 78 g of a crude extractive was obtained.

The residual solid was first triturated with petroleum ether with slight warming and filtered. This process was repeated until most of the petroleum ether soluble portion was leached out. The same procedure was followed with ethyl acetate and lastly chloroform after which the insoluble residue was crystallised from ethanol giving a crystalline substance (6.5 g). This was further purified by column chromatography over neutral alumina (90 g) and eluted with chloroform, followed by 50% ethanol in chloroform. After removal of the solvent from the elutes the residue crystallised from ethanol to give pure *lavanol*, m.p. 280–284°C; $[\alpha]_D^{33}$ +25° (in THF). (Found: C, 79.16; H, 10.14; O, 10.64%. $C_{30}H_{46}O_3$ requires C, 79.23; H, 10.20; O, 10.55%. $C_{30}H_{48}O_3$ requires C, 78.89; H, 10.59; O, 10.51%.)

On two dimensional thin layer chromatography on silica gel plate with cyclohexane–ethyl acetate (1:1) followed by elution on the second dimension with ethanol–benzene (8:1) only one spot could be detected on developing with a mixture of acetic anhydride, absolute ethanol, concentrated sulphuric acid (5:5) spray,[4] followed by heating in an oven. The R_f value in the first solvent was 0.741 and in the second solvent 0.441 respectively.

Lavanyl Acetate.—Lavanol (300 m) was refluxed with acetic anhydride (8 ml) and pyridine (4 drops) for 4 hours. The solvent was removed under reduced pressure and the residue triturated with water and filtered. The residue crystallised from ethanol to give *lavanyl acetate*, m.p. 278–80°C, $[\alpha]_D^{30}$ +44°. (Found: C, 77.33; H, 9.67; O,13.14; O-acetyl, 9.35%. Mol. Wt. 474. $C_{32}H_{48}O_4$ requires C, 77.37; H, 9.74; O, 12.88; O-acetyl (for one), 8.5%. Mol. Wt. 496.7; $C_{32}H_{50}O_4$ requires C, 77.06; H, 10.1; O,12.83%.)

Oxidation of Lavanol.—Lavanol (2.0 g) was dissolved in acetone (30 ml) and chromic acid solution prepared according to Jones *et al.*[5] was added to it in slight excess the excess chromic acid was destroyed by the addition of a few drops of methanol. The mixture was diluted with water and potassium carbonate was added (in

excess of neutralisation requirement), and extracted with ether containing a small amount of acetone. The ethereal layer was dried (Na_2SO_4) and evaporated to give an amorphous solid. This was chromatographed over neutral alumina (8 g) and eluted with ether, followed by ether–ethanol (5:1). On concentration of the ether–ethanol elute crystals were obtained of *oxolavanol*, m.p. 275-78°C, $[\alpha]_D^{31.5}+90°$ (Found: C, 78.94; H, 10.05; O, 10.67%. $C_{30}H_{46}O_3$ reqiures C, 79.24; H, 10.2; O, 10.56%.)

Oxolavanol gave an *oxime*, m.p. 250°C (Found: C, 72.00; H, 10.03; O, 15.08; N, 2.74%. $C_{30}H_{47}O_3N \cdot 1.5H_2O$ requires C, 72.54; H, 10.07; O, 14.50; N, 2.82%).

Oxolavanol gave a 2,4-*DNPH derivative*, from dilute hydrochloric acid solution of 2,4-dinitrophenylhydrazine and alcoholic solution of oxolavanol, as orange red crystals, m.p. 224–26° (decomp.) (Found: C, 67.76; H, 7.77; O, 15.88; N, 8.36%. $C_{36}H_{48}O_6N_4$ requires C, 68.34; H, 7.65; O, 15.17; N, 8.87%.)

Acknowledgement.—The authors wish to thank Dr. Ahmad Kamal, Director, Central Laboratories, for his interest. One of us (M.A. Khan) wishes to thank Dr. N.H. Khan and Dr. A.H. Khan for their help and encouragement during earlier stages of the work.

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