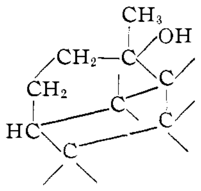
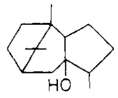
dehydration gave the diphenylethylene ester (XVII), m.p. 154–155°, $\lambda_{max.}$ 227 mμ (ϵ 11200), (*Anal.* Calcd. for $C_{26}H_{30}O_2$: C, 83.38; H, 8.07. Found: C, 83.17; H, 7.91). After considerable experimentation we found that XVII can be oxidized to IV with ozone in acetic acid solution at 25°. Ketoester IV had an infrared peak at 5.79 μ, which is characteristic for a cyclopentanone and ring B is thus established to be five-membered. Analysis of the facts provided thus far leads to part-structure XVIII for patchouly alcohol.

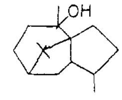


XVIII

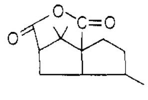
We turned next to a reëxamination of the patchoulene (V) described previously by Treibs,[2] from which information as to the size and attachment of ring C was forthcoming. Dehydration of II with sulfuric acid in ether solution at 35° gave a liquid hydrocarbon which is predominantly V as shown by its oxidation with osmium tetraoxide to a saturated diol (XIX), m.p. 122.8–123.5°, (*Anal.* Calcd. for $C_{15}H_{26}O_2$: C, 75.58; H, 11.00. Found: C, 75.37; H, 10.77). Cleavage of XIX with lead tetraacetate produced diketone XX, m.p. 95–96°, infrared max. 5.90 μ, (*Anal.* Calcd. for $C_{15}H_{24}O_2$: C, 76.22; H, 10.24. Found: C, 76.12; H, 9.96) which gave a negative iodoform test. The formation of a cyclohexenone (VI), m.p. 79.5–80°, $\lambda_{max.}$ 260 mμ (ϵ 11000), infrared max. 6.04 μ (*Anal.* Calcd. for $C_{15}H_{22}O$: C, 82.51; H, 10.16. Found: C, 82.44; H, 10.27) by base catalyzed aldolization of XX is only possible with a δ-diketone and V consequently must be a substituted cyclopentene. We have confirmed the finding of Treibs[2] who reported the formation of homocamphoric acid on ozonization of V and conclude that structure V proposed by this author is correct.



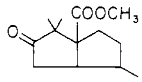
I



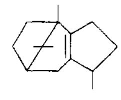
II

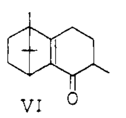


III

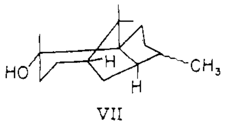


IV


V



VI



VII

It had previously been assumed that V contains the carbon skeleton of patchouly alcohol and this thesis had led to the erroneous structure I. The observations presented above clearly show that in the acid catalyzed dehydration of II a rearrangement had occurred. We have no degradative evidence for the location of the remaining methyl group and the structural argument rests solely on the formation of guaiazulene on dehydrogenation of II.[2] The reactions outlined above and others to be presented in a later paper provide evidence for the stereoformula VII of patchouly alcohol.

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DEPARTMENT OF CHEMISTRY G. BÜCHI
MASSACHUSETTS INSTITUTE OF TECH. R. E. ERICKSON
CAMBRIDGE 39, MASS.

RECEIVED FEBRUARY 6, 1956

PREPARATION OF ANHYDROUS ETHYLENIMINE: A CORRECTION

Sir:

It has been called to our attention[1] that the preparation of anhydrous ethylenimine by our published procedure[2] can lead to a violent reaction. This is the result of an unfortunate error in the quantity of water specified. In the interest of safety we should like to submit the following corrected procedure.

Anhydrous Ethylenimine.—A solution of 694 g. (6 moles) of 2-chloroethylamine hydrochloride in 1400 ml. of water was added with stirring to 600 g. (15 moles) of sodium hydroxide in 900 ml. of water. Solution temperatures were maintained below 50°. The resulting solution was then heated at 50° for 2 hours, after which time it was distilled at slightly reduced pressure (b.p. 30–35°) until 680 ml. of distillate had been collected. The cooled distillate was mixed with 350 g. of sodium hydroxide, and the ethylenimine was distilled at atmospheric pressure through a one-foot column packed with glass helices. A 50% sodium hydroxide solution was run in slowly at the top of the column during the distillation. The yield of ethylenimine was 187 g. (73%), b.p. 56–57°. The pot temperature did not exceed 70° during the distillation. The ethylenimine was stored over a few pellets of sodium hydroxide. Yields may be improved somewhat by increasing the efficiency of the ethylenimine removal operation as well as the condensation of the low-boiling distillate.

AMERICAN CYANAMID COMPANY
STAMFORD LABORATORIES V. P. WYSTRACH
RESEARCH DIVISION F. C. SCHAEFER

RECEIVED FEBRUARY 16, 1956

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(1) Personal communication from Professor Hakon Lund, Aarhus University, Denmark.

(2) V. P. Wystrach, D. W. Kaiser and F. C. Schaefer, THIS JOURNAL, **77**, 5915 (1955); specifically "Anhydrous Ethylenimine," p. 5917.