

Hydration of Thujopsene to Widdrol

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Hydration of thujopsene was first reported by Tanaka and Yamashita¹⁾, who obtained an alcohol $C_{15}H_{26}O$, m. p. $120\sim 122^{\circ}C$, $[\alpha]_D +115.3^{\circ}$ (yield 6%) under mild Bertram-Wahlbaum condition.

In the course of studies on the acid catalyzed isomerization of thujopsene, it was found that thujopsene gave another alcohol which was identified as widdrol by direct comparison with the sample obtained by Erdtman and Thomas²⁾.

A mixture of oxalic acid (50 g.), water (50 cc.), ethanol (600 cc.) and thujopsene (100 g.) was refluxed for 10 hr. The highest boiling fraction of the reaction products (b. p. $124\sim 129^{\circ}/5\text{mmHg}$, 25 g.) solidified on standing and repeated recrystallization of this material from petroleum ether and sublimation under reduced pressure gave widdrol, m. p. $98^{\circ}C$, $[\alpha]_D +105^{\circ}$ ($c=0.87$), $C_{15}H_{26}O$ (Found: C, 81.02, H, 11.79%). The hydrocarbon fraction (b. p. $105\sim 114^{\circ}C/5\text{mmHg}$, 65 g.) was treated under the same condition as mentioned above and further 4 g. of crude widdrol was obtained. The hydrocarbon fraction (b. p. $104\sim 111^{\circ}C/5\text{mmHg}$, 41 g.) recovered from this reaction mixture underwent the same treatment further to give the product; b. p. $113\sim 114^{\circ}C/5\text{mmHg}$, n_D^{20} 1.5022, d_4^{20} 0.9251, $[\alpha]_D +22.2^{\circ}$ (neat), I. R. bands $1638, 888\text{ cm}^{-1}$ (17 g.).

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